Effect of Prior Plastic Strain on the High Temperature Creep Deformation and Damage Response of Type 316H Stainless Steel

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Preface

This thesis is submitted for the degree of Doctor of Philosophy of The Open University, United Kingdom. The research described herein was conducted in the School of Engineering & Innovation, Faculty of Science, Technology, Engineering and Mathematics between October 2016 and September 2021 under the supervision of Dr Hedieh Jazaeri, Prof. P. John Bouchard and Dr Catrin Davies. This work is original to the best of my knowledge. Information from the published work of others has been acknowledged and a list of references are given in the bibliography. This work has not been submitted in whole or part for any other degree at any other university. Part of this work has been published at conferences as listed below:

- Measurement of creep deformation and cavitation in a novel hourglass test specimen using 3D-DIC and SANS, Neutron & Muon Science and User Meeting, 29th April-01st May 2019, Warwick University, United Kingdom. (poster presentation)

- Measurement of creep deformation and cavitation in a novel hourglass test specimen using 3D-DIC and SANS, ISIS Student Meeting 2018, 29th-30th September 2018, Abingdon, United Kingdom (oral presentation)

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- Simulation and measurement of creep strain in 316H stainless steel using 3D-DIC and SANS, Neutron & Muon Science and User Meeting, 25th-27th April 2018, Warwick University, United Kingdom. (poster presentation)
Abstract

Creep damage in ductile alloys is associated with creep deformation, crack growth and starts with the nucleation and growth of cavities. Under sustained high temperature and stress conditions, growing cavities can start to coalesce leading to microcracking and ultimate failure of a component. This mechanism can limit the lifetime of power plant components operating at high temperature. Many engineering components enter service in a cold worked or prestrained condition as a result of manufacturing processes such as bending, forging, welding etc. Such pre-conditioning alters the creep resistance of the material significantly. Its effect on the creep deformation properties of a structure during service, and creep damage response can be advantageous for some materials but disadvantageous for others. Hence it is crucial to understand the effects of prior plastic strain when assessing the lifetime and safety of power plant components, for example in the context of nuclear power generation. The research set out in this thesis aims to examine the effect of prior plastic strain on subsequent creep deformation behaviour and development of damage in AISI type 316H austenitic stainless steel, a material widely used in the fleet of Advanced Gas Cooled reactors operated by EDF Energy in the UK.

A novel cylindrical hourglass shaped test specimen was designed for the research where a constant applied load provided a variation in uniaxial stress and associated creep strain rate along the hourglass gauge length. A further innovation in this PhD work involved exploiting the potential of 3D digital image correlation (3D-DIC) for measuring spatially resolved creep deformation along the hourglass gauge section over long duration creep tests at high temperature of 550°C. The scope of testing included load-controlled creep tests carried out on 5 samples where 0, 4, 8, 12 and 16% of prior tensile plastic strain was introduced at room temperature. The prestraining was carried out on cylindrical samples before the hourglass shape was machined, ensuring a uniform level of prior plastic strain was present along the gauge section prior to creep experiments. It was found that prior plastic strain increased the creep resistance of the as-received material. Increasing plastic
strain decreased the creep strain rate and creep ductility. On the other hand, it resulted in an increase in time to failure.

After creep failure at the maximum stress location, small angle neutron scattering (SANS) was utilised to investigate changes in creep cavitation damage as a function of applied stress, level of creep strain and prior plastic strain at room temperature. Two sets of experiments were performed using the D11 instrument at the ILL reactor source (France) and the SANS2D instrument at the ISIS spallation source (UK). Very similar scattering results were obtained from the two instruments. Furthermore, SANS data from the instruments were analysed using two independent analysis routes; a maximum entropy method (MAXE) and a Monte Carlo algorithm (McSAS). Since SANS is an indirect method for measuring creep cavitation, the microstructure of the specimens was also investigated using qualitative scanning electron microscopy (SEM) in order to interpret and verify the SANS cavitation observations. The SANS investigations revealed a strong correlation between the volume fraction and number density of creep cavities with applied stress and creep strain. Furthermore, an increasing number density of small creep cavities as a function of prior plastic strain was observed and verified by qualitative SEM studies. This is new evidence that prior plastic strain, induced at room temperature, introduces specific cavitational damage in Type 316H stainless steel. The macroscopic damage calculation based on the stress modified ductility exhaustion model revealed that the majority of damage for the series of prestrained specimens is caused by plastic hole growth as a consequence of inducing prior plastic strain rather than due to creep related diffusion processes.
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<th>Description</th>
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<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
</tr>
<tr>
<td>AGR</td>
<td>Advanced Gas Cooled Reactor</td>
</tr>
<tr>
<td>AISI</td>
<td>American Iron and Steel Institute</td>
</tr>
<tr>
<td>ASME</td>
<td>American Society of Mechanical Engineers</td>
</tr>
<tr>
<td>BF</td>
<td>Bright Field</td>
</tr>
<tr>
<td>CF</td>
<td>Contrast factor</td>
</tr>
<tr>
<td>CGF</td>
<td>Cavity Growth Factor</td>
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<tr>
<td>DIC</td>
<td>Digital Image Correlation</td>
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<tr>
<td>DF</td>
<td>Dark Field</td>
</tr>
<tr>
<td>DSA</td>
<td>Dynamic Strain Ageing</td>
</tr>
<tr>
<td>EBSC</td>
<td>Electron backscatter diffraction</td>
</tr>
<tr>
<td>EDM</td>
<td>Electro-discharge machining</td>
</tr>
<tr>
<td>BCC</td>
<td>Body centred cubic</td>
</tr>
<tr>
<td>FCC</td>
<td>Face centred cubic</td>
</tr>
<tr>
<td>FE</td>
<td>Finite Element</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
</tr>
<tr>
<td>GB</td>
<td>Grain boundary</td>
</tr>
<tr>
<td>GBS</td>
<td>Grain boundary sliding</td>
</tr>
<tr>
<td>HS-AFM</td>
<td>High Speed Atomic Force Microscope</td>
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<tr>
<td>HVEM</td>
<td>High Voltage Electron Microscopy</td>
</tr>
<tr>
<td>ILL</td>
<td>Institut Laue-Langevin</td>
</tr>
<tr>
<td>LVDT</td>
<td>Linear variable differential transformer</td>
</tr>
<tr>
<td>MAXE</td>
<td>Maximum Entropy</td>
</tr>
<tr>
<td>McSAS</td>
<td>Monte Carlo Small Angle Scattering</td>
</tr>
<tr>
<td>MLI</td>
<td>Mean Linear Intercept</td>
</tr>
<tr>
<td>MSF</td>
<td>Multiaxial Stress Factor</td>
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<tr>
<td>OM</td>
<td>Optical Microscopy</td>
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<tr>
<td>PD</td>
<td>Ploydispersity</td>
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<tr>
<td>SANS</td>
<td>Small angle neutron scattering</td>
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<td>SAXS</td>
<td>Small angle X-Ray scattering</td>
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<td>Abbreviation</td>
<td>Full Form</td>
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</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>SLD</td>
<td>Scattering length density</td>
</tr>
<tr>
<td>SMDE</td>
<td>Stress modified ductility exhaustion model</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>TOF</td>
<td>Time of Flight</td>
</tr>
<tr>
<td>USDFLD</td>
<td>User defined field</td>
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<tr>
<td>UTS</td>
<td>Ultimate Tensile Strength</td>
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Nomenclature

- $\epsilon_e$: Elastic strain
- $\epsilon_p$: Plastic strain
- $\epsilon_{cr}$: Creep strain
- $\epsilon_{min}$: Minimum creep strain
- $\sigma$: Stress
- $\epsilon$: Strain
- $\epsilon_t$: Total strain
- $\epsilon_{pr}$: Primary creep strain
- $\epsilon_{sec}$: Secondary creep strain
- $\epsilon_{ter}$: Tertiary creep strain
- $\sigma_1$: Maximum principal stress
- $\sigma_y$: Yield stress
- $\sigma_{vm}$: von Mises stress
- $\sigma_H$: Hydrostatic stress
- $\lambda$: Wavelength
- $C(D)$: Volume fraction of cavities
- $V(D)$: Volume fraction of scatterers
- $N(D)$: Number density
- $N_a(D)$: Areal number density
- $n$: Stress exponent in Norton Power Law
- $A$: Material constant in Norton Power Law
- $q$: Scattering vector
- $S_v$: Grain boundary surface area
- $N$: Number of intercepts
- $d_{hkl}$: Inter planar spacing
- $F$: Force
- $\epsilon_f$: Creep strain at failure
- $t_r$: Time to rupture
- $Q_c$: Activation energy for creep
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<tr>
<td>$T_m$</td>
<td>Melting temperature</td>
</tr>
<tr>
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<td>Creep damage per cycle</td>
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<tr>
<td>$d_c$</td>
<td>Mass</td>
</tr>
<tr>
<td>$\bar{l}$</td>
<td>Average grain size</td>
</tr>
<tr>
<td>$L$</td>
<td>Average spacing of cavities</td>
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<tr>
<td>$\gamma$</td>
<td>Surface tension</td>
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<tr>
<td>$t_{cr-0.2%}$</td>
<td>Time to reach creep strain of 0.2%</td>
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<td>Radius</td>
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<td>Volume</td>
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<td>Number of particles</td>
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<td>Average Volume</td>
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<td>$\Delta \rho$</td>
<td>Change in scattering length density</td>
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<td>$V_{Sph}$</td>
<td>Volume of spheres</td>
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<tr>
<td>$D$</td>
<td>Diameter</td>
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<td>bound coherent scattering length density</td>
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<td>$M$</td>
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1 Introduction

1.1 Background

Tackling climate change is one of the great challenges of our time and it is largely an energy challenge. The energy sector is responsible for about 75% of greenhouse gas emissions which are the driving factors for an increase of the average global temperature [1]. Therefore, a focus on how energy is produced and consumed plays a significant role to reduce global carbon dioxide emissions [2]. Achieving net-zero emissions within the first half of the century is one of the most ambitious goals of the 2015 Paris Agreement to limit net global warming below 1.5°C [3]. The UK became the first country of the group of seven states (G7) as parliament passed legally binding goals to reduce net emissions of carbon gas by 100% by 2050 relative to 1990 levels [3, 4]. Significant reductions in decarbonising the UK economy have been achieved so far and the country could reduce the amount of total greenhouse gas emissions to 43.1% lower than in 1990 [5]. However, the global energy demand keeps continuing to grow, driven by an increasing world population and a concurrent growth in prosperity and living standards in developing countries. In their published Energy Outlook for 2019, the U.S. Energy Information Administration (EIA) estimates that the world energy consumption will rise by nearly 50% between 2018 and 2050 driven by countries predominantly in Asia with strong economic growth [6]. New strategies have to be developed to meet these demands by providing an affordable solution for a carbon-free baseload electricity supply.

The UK government pledged to decarbonise all sectors of the economy in its Clean Growth Strategy in 2017 [7]. To meet this goal the nuclear innovation and advisory board (NIRAB) recommends establishing a cost-competitive build programme for Gen III+, Small Modular Reactor (SMR) and Advanced Modular Reactor (AMR) [8]. However, any investments into nuclear energy have to be substantial when set against a reality where most nuclear plants face closure. This is significant since nuclear power currently provides one of the few forms
to generate baseload low carbon electricity and therefore needs to be a crucial part of the future energy mix that is becoming more reliant on renewable but intermittent energy sources such as from solar and wind. To make a renewed case for nuclear energy new reactor designs that are more energy-efficient and safe have been developed over the last decades.

Figure 1.1: The evolution of nuclear power [9]

Figure 1.1 shows the evolution of different reactor types over the last 70 years which highlights that we are currently on the brink of the transition to the new Generation IV reactor types. Some of the most promising versions of new types of fission reactors are outlined in Table 1.1. It shows that the majority of them reach temperatures significantly higher than 500°C. These conditions are very much within the temperature regime where creep is, next to corrosion and fatigue, a major degradation mechanism that can have a significant impact on the structural integrity of components.
Table 1.1: Overview of five Generation IV reactor systems [10]

<table>
<thead>
<tr>
<th>System</th>
<th>Neutron Spectrum</th>
<th>Coolant</th>
<th>Outlet Coolant Temperature</th>
<th>Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>VHTR very-high-temperature reactor</td>
<td>Thermal</td>
<td>Helium</td>
<td>900-1000 °C</td>
<td>250-300 MWe</td>
</tr>
<tr>
<td>SFR sodium-cooled fast reactor</td>
<td>Fast</td>
<td>Sodium</td>
<td>550 °C</td>
<td>30-150 MWe</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>300-1500 MWe</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1000-2000 MWe</td>
</tr>
<tr>
<td>GFR gas-cooled fast reactor</td>
<td>Fast</td>
<td>Helium</td>
<td>850 °C</td>
<td>1200 MWe</td>
</tr>
<tr>
<td>LFR lead-cooled fast reactor</td>
<td>Fast</td>
<td>Lead</td>
<td>480-800 °C</td>
<td>20-180 MWe</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>300-1200 MWe</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>600-1000 MWe</td>
</tr>
<tr>
<td>MSR molten-salt reactor</td>
<td>Fast/thermal</td>
<td>Fluoride salts</td>
<td>700-800 °C</td>
<td>1000 MWe</td>
</tr>
</tbody>
</table>

The most common reactor type in the UK today is the Advanced Gas-cooled Reactor (AGR), currently operating at 7 twin reactor power stations. The first AGRs, in Hinkley Point, Somerset and at Hunterston, began their commercial operations in 1976 and the latest, at Heysham and Torness, in 1988. Each reactor unit is linked to a 660 MW turbine generator. Some of the older AGR nuclear power stations have reached the point where further life extensions are considered not to be viable and are operated with decommissioning in mind [11]. However, to meet a continuously growing demand of electricity in the UK, EDF Energy has extended the operating lives of all Advanced Gas-cooled Reactors (AGRs). In order to extend the lifetime of the plants, a reliable lifetime assessment procedure is required that ensures a safe and commercially viable continued operation. As part of this, EDF Energy has intensified the investigation of the creep properties of Type 316H stainless steels which are used in a range of structural components [12]. Austenitic stainless steels such as AISI type 316 are considered to be a structural material of choice in many
of the advanced nuclear systems due to their high temperature properties, corrosion resistance and are relatively easy to weld [13]. Type 316H stainless steel is of great significance and widely used in the UK’s AGRs which operates at temperatures in the range of 500 to 650 °C. One major concern for components operating at such elevated temperatures, is structural failure as a consequence of creep deformation over time. In addition, austenitic steels such as AISI Type 316 stainless steel might also have the potential to be used in fusion power plants. In their review on their potential use, Sahin et al. [14] found that austenitic steels can play an important role as a structural integrity material for future fusion reactors due to their excellent mechanical properties, huge technical database and fission reactor experience. It underlines the importance to study these types of materials.

1.2 Aims and Objectives

As a consequence of creep deformation, intergranular cavities nucleate, grow and coalesce which ultimately causes failure in ductile alloys such as Type 316H stainless steel. This process is referred to as creep damage and is one of the main limiting factors in a components lifetime operating at high temperature and under high stresses. Since creep failure after prolonged operation remains a huge safety and not at least financial concern, the aim to better understand creep degradation and to quantify creep damage is of huge importance. It is a necessity for developing new materials as well as to evaluate the structural integrity for life-extension plans. The fabrication process of a component, such as welding forging or cold rolling, can lead to an alteration of its mechanical response and deformation behaviour, such as creep properties. Most material creep properties are derived usually only from uniaxial constant load tests and are used for a range of different compositions and varying pre-loading histories. Subsequently, the model predictions especially for complex stress and temperature configurations have to be extrapolated. Secondly, short timescale laboratory experimental data are commonly used to fit creep models and to predict the long term structural integrity of components. In order to realise this, very high load stress conditions are chosen. However, this alters the creep response of materials with a relatively
low yielding strength, such as Type 316H stainless steel.

The main aim of the thesis is to investigate the influence of tensile plastic strain introduced at room temperature on subsequent creep deformation properties in Type 316H stainless steel. Since prior plastic strain can be beneficial for some materials (Type 316H stainless steel [15]) but might have a disadvantageous effect on the subsequent creep behaviour for others (Nickel-based C263 superalloy [16]) it is significant to fully understand its implications. In nuclear power plants where components are often used in prestrained conditions, it is of great importance to determine any change in creep response due to prior cold work. Furthermore, working with prestrained samples enables to distinguish the damage induced by creep from that induced by plastic loading. The main objectives of the thesis are:

- To investigate the effects of tensile plastic prestrain introduced at room temperature on the creep deformation properties of AISI type 316H austenitic stainless steel
- To examine the applicability of 3D digital image correlation (3D-DIC) to monitor creep curves spatially resolved as a function of stress in a novel cylindrical hourglass test specimen
- To investigate creep cavitation damage using small angle neutron scattering (SANS) and scanning electron microscopy (SEM)
- To compare two different software models that are used to analyse for fitting the SANS data
- To assess the stress modified ductility exhaustion model to determine the macroscopic creep damage for Type 316H stainless steel
- To assess the influence of prior cold work (tensile prestrain at room temperature) on creep deformation in terms of the multiple region stress modified ductility exhaustion (SMDE) model for Type 316H stainless steel, developed by EDF Energy [12]
1.3 Outline of the thesis

The thesis consists of 8 chapters in total.

- **Chapter 1** provides a general introduction to the thesis.

- **Chapter 2** provides a literature review on the most relevant research about creep cavitation in Type 316H stainless steel. It includes creep deformation and failure, precipitation, cavity nucleation and cavity growth models and a review on experimental methods to study creep cavities in Type 316H stainless steel.

- **Chapter 3** explains the experimental methods used in this thesis and covers the instruments, experimental set-ups, test procedures and data analysis methods used throughout the thesis.

- **Chapter 4** characterises the Type 316H stainless steel material used for the test programme presented in this thesis and covers the microstructure and mechanical properties. The design for a novel hourglass specimen is presented where 3D Digital Image Correlation (3D-DIC) is used to measure the variation in creep deformation as a function of stress.

- **Chapter 5** provides the results of an extensive creep testing programme on test samples prestrained at room temperature to different amounts. The changes in the creep response are quantified. Furthermore, the creep ductility as a function of stress and total inelastic strain is linked to the stress modified ductility exhaustion model used by EDF Energy.

- **Chapter 6** describes the creep cavitation damage analysis of the crept specimens using techniques such as small angle neutron scattering and scanning electron microscopy. A link between changes in the amount of prestrain and the type of cavitation damage is made.
• **Chapter 7** provides an overall discussion of the PhD thesis and evaluates the key findings of the work critically.

• **Chapter 8** draws together conclusions of the thesis and addresses potential starting points for future work.
2 Literature Review

2.1 Overview

The chapter aims to introduce the concepts and theories outlined in the thesis and to provide a critical review on the current state of knowledge with regard to creep cavitation in AISI type 316H stainless steel. The literature review begins with a brief introduction to stainless steels and creep deformation mechanisms. Cavity nucleation as well as cavity growth are explained in more detail. The effect of prior plastic strain induced at both room temperature and elevated temperature and its effect on the creep properties is outlined together with an overview about macroscopic methods to determine the damage in Type 316H stainless steel. Lastly, with small angle neutron scattering and scanning electron microscopy the most common quantification methods for microstructural features in Type 316H stainless steel are described. Published works relating to the effect of prior plastic strain on subsequent creep as well as on the experimental methods are reviewed to highlight the research gaps.

In the UK’s advanced gas cooled reactors, AISI type 316H stainless steels are an important material and used as parts of the heat exchanger units as well as for steam tubes. A schematic sketch of an AGR with its components and materials is illustrated in Figure 2.1. With working temperature in the range of 500°C-650°C of the reactor coolant gas, components in the advanced gas cooled reactors must provide a very high creep resistance. Due to its good corrosion resistance, creep and fatigue properties around these elevated temperatures Type 316H stainless steel is widely used. As illustrated in Figure 2.1 Type 316H stainless steel is used in steam tubes or in heat exchanger units.
2.2 Austenitic Steels

Austenitic steels preserve a face-centred cubic crystal (FCC) structure at room temperature and possess high ductility within a very large temperature range. They have the strength of mild steels with a 0.2% proof stress of around 200 MPa at room temperature. Unlike ferritic steels, austenitic steels cannot harden due to heat treatment but change their characteristics significantly with cold working. Moreover, they have good cryogenic properties and good oxidation resistance at elevated temperatures. Due to these good characteristics, austenitic steels are used in many applications such as roofing, cladding or heat exchanger ovens. A variety of alloying elements are used in Type 316H SS that have an influence on the properties as well as on the crystal structure:

- Chromium and Nickel are the main components that are added to improve the properties in terms of structural integrity for applications involving high temperature and corrosion resistance. Chromium is added primarily to improve corrosion resistance and prevent oxidation processes. It creates a self-adherent chromium oxide $Cr_2O_3$ surface layer. Chromium itself has a body-centred cubic (BCC) structure. Besides,
Chromium contributes to the precipitation of carbides and sigma phases in austenitic steels [17,18].

• Nickel on the other hand, is used as an austenite stabilising element which improves the ductility as well as the fracture toughness properties of steel.

• Niobium and Titanium are added as minor elements to improve the creep strength by precipitate strengthening due to forming of second phase particles. Both elements reduce the depletion of chromium along the grain boundaries since they are even stronger carbide former than Chromium [19].

• Molybdenum is added to improve the creep resistance due to solid solution hardening. It also facilitates the precipitation of second-phase particles such as carbides and for long term ageing, the growth of sigma and laves phases [20].

• Nitrogen is added mainly to promote the chromium diffusion and to reduce the rate of chromium carbide precipitation \( M_{23}C_6 \). It also promotes an austenite structure and adds mechanical strength due to solid solution hardening [19].

• Manganese promotes the solubility of nitrogen in the austenite phase and adds to the mechanical strength of the alloy [21].

• Carbon is another element that promotes an austenite structure and provides mechanical stability through solid solution and precipitation hardening. Since it reduces the chromium content by precipitating carbides, it can reduce the corrosion resistance for the material especially when the carbon content is quite high as it is in Type 316H stainless steel [19].

Within the 316 series of stainless steels, several different grades do exist. Compared to Type 316N and 316L, 316H stainless steel has a relatively high grade of carbon that increases the hardenability and reduces the ductility of the material.
2.2.1 Precipitation in Type 316H stainless steel

Type 316H stainless steel contains several alloying elements in a supersaturated solution that tend to form precipitates under creep conditions (temperature and time-dependent) at energetically preferential sites. Grain boundaries are preferable sites because of their high vacancy diffusion rate that accelerates the movement of solute atoms and because they can accommodate a structural mismatch between precipitates and the lattice [22,23]. Precipitates can also nucleate at twin boundaries, and sites that are associated with a high density of pinned dislocations [22]. Even vacancies and dislocations can be preferred sites for precipitation to occur [23].

Time temperature-transformation/precipitation diagrams show the sequence of precipitation for different alloys exposed to temperature and time. In Type 316H stainless steel different secondary phases such as α-ferrite, carbides and intermetallic phases can nucleate and grow after ageing time and service [22,24,25]. This is illustrated in the TTT diagram in Figure 2.2. The kinetics are relatively complex and depend on factors such as service stress, temperature and local composition of the component. It has been shown by several groups that precipitates can alter the creep properties such as ductility and creep strain rate [26–28]. Dyson stated that the level of trace impurities affects the nucleation rate of cavities. The following equation describes the relationship between the density of cavities and precipitates [29]:

\[ N_c = fN_p(1 - \exp^{-ke}), \tag{2.1} \]

where \( N_p \) is the number density of grain boundary precipitates, \( N_c \) is the number density of cavities, \( f \) is a function of interfacial energy and maximum principal stress, \( k \) describes the cavity interaction and \( \epsilon \) is the strain. Furthermore, it was mentioned by Seah et al. [30] that surface segregation of impurities can affect the cavity generation rate. According to Seah et. al there two hierarchies of active impurities. Stress relief cracking acts through
surface segregation, whereas creep embrittlement acts through grain boundary segregation.

Figure 2.2: Time temperature-transformation precipitation diagram for Type 316 stainless steel solution treated at a temperature of $1260^\circ C$ for 1.5 hours and subsequently water quenched [18].

A typical temperature-time precipitation diagram of a solution annealed Type 316 stainless steel is illustrated in Figure 2.2. The different phases will be described in Section 2.2.1.1. Weiss et al. investigated the effect of the solution treatment temperature on the precipitation kinetics [18]. They found that the temperature has a significant effect on the kinetics of the precipitation of second-phase particles. This can be contributed to a change in the grain microstructure that becomes finer with higher temperatures. Figure 2.3 illustrates the microstructural changes in Type 316 stainless steel as a function of temperature, cold work and amount of carbon of the solution treated samples. Weiss et al. observed the evolution of coarser particles around grain boundaries due to higher ageing temperatures. Furthermore, they found that the deformation faults such as dislocation pile-ups caused by cold work act as a nucleation site for small precipitates.
Figure 2.3: Effect of solution heat treatment temperature, cold work and carbon content on the kinetics of precipitation in Type 316L austenitic steel after ageing for 1500 hours. The precipitates are arranged in a decreasing order [18].

It has been reported that the nucleation and growth of carbides and intermetallic phases can be altered by elastic-plastic strain from loading prior to creep. Morris et al. found in an analysis of creep deformed Type 316 stainless steel specimens, creep tested at temperatures between 525°C and 900°C, that the strain induced from loading affects the dislocation density that is linked with the intragranular precipitation density [31,32]. This is shown in Figure 2.4, where Morris et al. applied different amounts of loading strain at a temper-
ature of 625°C to Type 316 stainless steel and subsequently annealed the specimens at a temperature 625°C for a time of 400 h in the absence of stress [31].

Spruiell et al. [33] noted that very high levels of 30–50% of prior cold work lead to a significant increase in the rate of precipitation in Type 316 stainless steel, predominantly the σ phase (will be covered in Section 2.2.1.1). Spruiell et al. postulated that the high amount of cold work leads to a grain boundary recrystallisation that provides preferable nucleation sites. Weiss et al. [18] found that cold work of less than 20% in solution treated steel results in significant clusters of stacking faults whereas even higher deformations cause an increase of deformation micro-twins. They noted that subsequent ageing results in accelerated precipitation of σ phase particles. Even creep fatigue can lead to the precipitation of second phase particles. Cheng et al. found evidence that the same level of precipitation of second phase particles occurs in a creep fatigue sample (12 hours) tested at 560°C compared to a solution treated sample aged for 1000 h at 560°C [34].

Recent modelling work on how creep performance can be influenced by the evolution of
second phase particles in Type 316H stainless steel has been done by Hu et al. [35]. The phenomenon of multiple secondary creep regimes was attributed to thermal solute drag and phase transformation on dislocation movement and recovery. Other groups such as Xiong et al. worked on a model that describes the precipitation kinetics in Type 316H stainless steel [36]. Their estimations on precipitate nucleation and growth were matched with experimental data from actual plant material. Figure 2.5 shows the predicted size distribution and relative frequency of $M_{23}C_6$ carbides after ageing of 91597 hours. Figure 2.5 a) shows that the estimated size distribution varies greatly with temperature. Figure 2.5 b) shows the growth of carbides over time for a range of different temperatures. The growth of carbides develops a lot quicker with an increase in temperature.

![Figure 2.5](image)

**Figure 2.5:** Modelling of the a) size distribution of $M_{23}C_6$ second phase particles with different radii and b) mean radius of carbides in Type 316H stainless steel at various temperatures. The plot on the left also includes the size distribution of carbides based on experimental data from plant material [36].

### 2.2.1.1 Carbide precipitation

- $M_{23}C_6$ **Carbide** is the most abundant precipitate formed in Type 316H stainless steel. It consists of predominantly chromium carbide. However, the chromium can be substituted with Fe, Mo or Ni whereas B and N can substitute C. For Type 316H stainless steel with its relatively high carbon content, precipitates can form
after a couple of minutes at temperatures higher than $650^\circ C$. Since the diffusion rates of chromium and carbon are higher at grain boundaries, carbides tend to form intergranular precipitates resulting in chromium depleted zones.

- $M_6C$ Carbide is commonly known as the $\eta$ carbide, formed in stainless steels containing Mo, W or Nb. It is formed after long ageing times in a process where $M_{23}C_6$ absorbs Mo and transforms to $M_6C$.

![Illustration of the evolution of carbides in the grain boundary area creating chromium depleted zones that can facilitate corrosion](image)

**Figure 2.6:** Illustration of the evolution of carbides in the grain boundary area creating chromium depleted zones that can facilitate corrosion [21].

### 2.2.1.2 Intermetallic phases

The three main intermetallic phases formed in steel are $\sigma$, $\chi$ and Laves phase.

- **Sigma phase:** is an intermetallic phase that forms in Fe-Cr high alloying systems. Other elements such as Ni, Mo, Mn and Si can dissolve and replace Fe and Cr. Similar to carbides sigma phases are commonly observed along grain boundaries and at twin boundaries [37]. It is a hard precipitate and can cause embrittlement in the alloy. It can precipitate from delta ferrites as well as from austenites where the latter process is about 100 times slower, see Figure 2.2. Compared to carbides it takes several hundreds to thousands of hours to form [38].

- **Laves phase:** is an intermetallic phase formed from Fe and other elements that exhibit a hexagonal crystal structure such as Mo, Nb, and Ti. Laves phase are stable
above temperatures of $815^\circ C$. Similar to sigma phase it takes long times to nucleate, see Figure 2.2. Minami et al. observed its formation after 1000 h in a temperature range between $625 - 800^\circ C$ [39].

- Chi phase: is an intermetallic phase very similar to the sigma phases with the difference that carbon tends to dissolve. Minami reported its precipitation time after ageing of 25000 h at a temperature between $700 - 850^\circ C$ [39].

### 2.3 Creep deformation

At room temperature, most metals deform in a way that is dependent on stress but is independent of time and the deformation occurs instantaneously. Below their yielding point, they exhibit a single, time-independent relationship between stress and strain described by Hooke’s law $\sigma_0 = E\varepsilon_0$. However, with an increase in temperature materials get thermally activated and components start to elongate under a fixed stress in a time-dependent deformation process even if the stress is below the yielding point of the material. This deformation process is called creep and can be understood as a thermally activated time-dependent accumulation of irrecoverable strain ultimately leading to failure of the material. The total strain $\varepsilon_{tot}$ of the material is the sum of both elastic strain $\varepsilon_{el}$ and inelastic strain $\varepsilon_{in}$ [40].

$$\varepsilon_{tot} = \varepsilon_{el} + \varepsilon_{in},$$ \hspace{1cm} (2.2)

In general, creep is theoretically possible at all temperatures but for most metals and alloys only becomes significant at temperatures close to or higher than $0.4T_m$, where $T_m$ is the melting temperature. At these temperatures, atoms become thermally activated which allows a time-dependent rearrangement of the structures [41].
2.3.1 Creep Deformation Stages

Following Andrade, there are three stages in a typical creep curve tested under a constant load and temperature: primary, secondary and tertiary creep [42]. A typical creep curve with the different creep regimes and the related creep damage is depicted in Figure 2.7.

![Creep Curve Diagram](image)

**Figure 2.7:** Schematic representation of a typical creep curve with the evolution of creep damage, after [43].

In the primary creep region, the creep strain rate \( \dot{\varepsilon} = \frac{d\varepsilon}{dt} \) is decreasing with an increasing strain or time. Thermal recovery processes such as redistribution of lattice defects advances deformation processes and work hardening processes strengthen the resistance of the material. The creep strain rate decreases and strain hardening is the dominating process. One way of explaining the relatively fast initial creep strain rate is to think of a certain density of mobile dislocations that are free to glide. As they start to move they become pinned and the total number of mobile dislocations reduces. This phenomenon is understood as hardening or reducing strain [19].

During the secondary creep region, the strain rate (creep rate) remains constant. The rate
at which pinned dislocations are released by climb or cross-slip is equal to the rate at which mobile dislocations are being pinned by obstacles such as precipitates. However, the total number of dislocations increases substantially due to the dislocation creation mechanisms. Hence, during the latter part of the second regime, isolated voids are starting to form and begin to orientate [44]. The creep behaviour in the secondary or stationary creep stage can be described by a power law equation. This relationship can be used for materials at a range of stresses and temperatures that deform by dislocation creep.

In the tertiary creep region, an increase of the strain rate (or decrease in the flow stress) is observed which leads to fracture at the end of this stage [44]. The increase in creep rate arises from an increasing stress due to cross section reduction facilitated by the evolution of microstructural damage that includes cavity coalescence [45]. Voids in the grain boundaries are starting to link up and form micro-cracks when they join together which increases the stress exponentially [46]. Once started, this leads to a positive-feedback in which augmented stress leads to a significant increase in strain. In this regime, failure of the component does ensue quickly. Another failure mechanism that has been reported is based on a softening caused by changes in the microstructure. This occurs mainly as a result of an accumulation of the dislocation density [47, 48]. Instabilities in the material can also lead to necking of the material for example due to recrystallisation of the grain structure.

2.3.2 Creep mechanisms

In polycrystalline materials, two main creep mechanisms describe the creep process during the secondary stage. Diffusional controlled creep and dislocation creep which is also known as power-law creep. Frost and Ashby created deformation maps for a variety of materials and grain sizes within the secondary stage [49]. The case for Type 316H SS with a grain size of 50µm is illustrated in Figure 2.8. The governing creep mechanism for a certain material under constant stress at a given evaluated temperature can be estimated with such a deformation mechanism map. These deformation maps are unique for each metal
or alloy and summarise the creep behaviour for a certain grain size. This helps to choose a specific alloy for a certain high-temperature application. A different grain size changes the zones of the different mechanisms. In the case of diffusional creep, for example, an increase of the grain size has a decreasing effect on the creep rate.

![Deformation-mechanism map for Type 316H stainless steel with a grain size of 50µm](image)

**Figure 2.8:** Deformation-mechanism map for Type 316H stainless steel with a grain size of 50µm [49].

The creep rate depends on several factors, such as the chemical composition, microstructure and grain size. Precipitation hardening or solution strengthening can impede dislocation movement whereas the grain size has a huge influence on the diffusional processes within the material [50]. For Type 316H stainless steel, which is commonly used at 550°C (see Figure 2.8) it can be observed that power law creep is the dominant creep mechanism at higher stresses and temperatures or even below the yield strength of a material.

### 2.3.2.1 Dislocation creep

In creep, dislocations can move by glide in a slip plane or climb triggered by thermal activation. At higher stresses and temperatures dislocations may climb out of their glide
planes due to absorbing or emitting of vacancies. This allows dislocations that are stopped by obstacles to continue their gliding. The dislocation glide determines the strain but the strain rate is controlled by the dislocation climb process. Therefore, the secondary creep rate is controlled by lattice diffusion processes [51]. The mechanism can be described by a power-law relationship:

\[ \dot{\epsilon}_s = A \sigma^n \exp \left( -\frac{Q_c}{RT} \right), \]  

(2.3)

where \( A \) is a material constant, \( Q_c \) is the activation energy, \( \sigma \) is the applied stress, \( R \) is the universal gas constant and \( n \) is the power law exponent [52]. An exponent larger than 5 is common for austenitic steels [53]. A stress exponent of around 3 indicates that the creep mechanism is viscous glide creep where the glide of dislocations is slowed down by solute atoms within the matrix. The process of dislocation glide along slip planes to pass obstacles is illustrated in Figure 2.9. In power law creep the process usually involves the climb of dislocations out of their glide planes by absorption or emission of vacancies cite [51]. This usually happens if a dislocation is blocked by an obstacle and tries to circumvent it. The climbing process usually determines the creep strain rate. The movement of the dislocation can also be impeded by solute atoms which cause the sliding process to happen at a slower strain rate.
2.3.2.2 Diffusion creep

At low stresses and temperatures diffusion creep becomes dominant which occurs by diffusion of atoms along the grain boundaries or through the lattice. It can be expressed with an Arrhenius type expression for the case of uniaxial loading:

\[ \dot{\epsilon}_s = A\sigma \exp\left(\frac{-Q_c}{RT}\right), \]  

where \( A \) is a material constant, \( Q_c \) is the activation energy, \( \sigma \) is the applied stress and \( R \) is the universal gas constant \[52\]. In theory, diffusion creep mechanisms can be related to a stress exponent of \( n = 1 \) \[54–56\]. However, it should be mentioned that several reports are proposing higher values of the creep exponent, up to about three. This was investigated for MgAl and MgZn alloys by Spigarelli et al. \[57\]. If the activation energy is sufficiently high, at a temperature of around 0.6 \( T_m \) and a stress below the yield limit, the diffusion trajectory...
is along the grain boundaries and results in grain elongation in the direction of stress and creep strain. This phenomenon is termed Coble Creep. At even higher temperatures at around \(0.8 T_m\) diffusion through the lattice occurs which is referred to as Nabarro-Herring creep. At these high homologous temperatures, particles that diffuse through the lattice can cause the grains to grow and elongate. Both processes, with the associated movement of atoms and vacancies, are depicted in Figure 2.10.

**Figure 2.10:** Illustration showing the vacancy and atom flow either by a) lattice diffusion through the grains or by b) diffusion along the grain boundary [51].

### 2.3.3 Creep dependency on stress and temperature

Stress as well as temperature both have significant effects on the shape of the creep curve. As shown in Figure 2.11 an increase in temperature results in an increase of creep strain rate but also in a reduction of creep rupture time. With temperatures higher than \(0.5T_m\) secondary as well as tertiary creep becomes more dominant whereas at lower temperatures primary creep dominates and low creep ductilities can be observed [50]. Increasing the stress while keeping the temperature constant results in a similar effect. The creep strain rate increases and the time to rupture usually drops. The amount of stress can also have a significant effect on the resulting creep ductility which will be discussed in more detail later in Section 2.7. The creep strain rate at failure always depends on the material, the examined stress range and the testing temperature. From a creep curve, several properties
can be deduced, such as the duration of the creep stages, the minimum creep rate, the time to fracture as well as the strain at failure.

![Illustration of the stress and temperature dependence of typical creep curves.](image)

**Figure 2.11:** Illustration of the stress and temperature dependence of typical creep curves.

Figure 2.12 illustrates the dominant creep deformation mechanism and the dependence of the creep constant $n$ to a change in stress and temperature for Type 316H stainless steel. With a decrease in stress, the stress exponent $n$ becomes smaller. It illustrates that at low stresses the dominant creep mechanism is diffusion controlled whereas power law creep dominates the intermediate section with dislocation creep as the main mechanism. Within the power law section, there is another transition of the creep exponent that indicates the power law breakdown at very high stresses. This transition indicates a change from climb controlled to glide controlled flow. The transition is very well reported for a variety of alloys, such as Type 316H SS [49].
Figure 2.12: Illustration of different creep deformation modes versus shear stress dependence [49].

2.3.4 Creep strengthening mechanisms

Austenitic steels can develop quite significant precipitation hardening due to carbonitrides and from fine intermetallic compounds during long term service at elevated temperatures [19]. Strain hardening in Type 316H stainless steel results from interactions of dislocations with precipitates, grain boundaries and other dislocations. The most common ways in which steels can be strengthened are:

- solid solution strengthening
• precipitation hardening

• dislocation hardening

Solid solution strengthening can be achieved by adding substitutional solute atoms such as Mo and W that have a relatively large atomic size number compared to the parent lattice. It usually involves heating the material to a suitable temperature before quenching it by rapidly cooling to ambient temperature. Depending on the size of the added elements they can either replace a solvent atom and become substitutional or if they are small in size they can occupy interstitial sites between two solvent atoms. This difference in size between solute and solvent atoms can cause a distortion in the lattice structure and local stress fields can be created. Stress fields are compressive when the solute atoms are relatively large and tensile when they are small. Subsequently, solute atoms can act as an obstacle to dislocation motion [58, 59]. However, it should be noted that in creep resistant steels the effect of solid solution hardening compared to other strengthening methods such as precipitation strengthening is negligible [19].

Precipitation hardening is one of the most important mechanisms to strengthen steels at elevated temperatures. Steels usually contain precipitate particles in the matrix as well as on the grain boundaries as discrete particles of different phases in solid states [19]. As described earlier in Section 2.2.1, typical precipitates in 316H stainless steel are carbides such as $M_23C_6$ where M represents the metallic element and C denotes the carbon atom, or intermetallic precipitates such as the Laves phase $Fe_2(Mo,W)$, $FeCr\sigma$ phase, $\chi$ phase $Fe_{36}Cr_{12}Mo_{10}$ etc. [60]. Precipitates can nucleate and grow along grain boundaries and within the grains but are more likely to nucleate in defect regions such as grain boundaries or around intragranular dislocations [60,61]. If the precipitation is intragranular the additional force that is required for a dislocation to cut through a small precipitate or to bow if the precipitates are impenetrable can cause strain hardening [62]. The latter process is called Orowan bowing and its strength depends on the particle dispersion within the matrix [63]. A small mean inter-particle spacing causes a large strengthening of the material [64]. As illustrated in Figure 2.13 a dislocation loop is left at each particle after
a dislocation has passed which causes a build-up of dislocation loops creating a wall of dense tangles of dislocations. At elevated temperatures, the particles tend to grow and coalesce which causes changes in the particle dispersion and hence the Orowan bowing. This subsequently changes the mean inter-particle spacing and reduces the strengthening effect with larger spaced dispersion of particles.

![Diagram](image)

**Figure 2.13:** Schematic drawing of a bowing dislocation through a line of particles by either a) Orowan mechanism or b) general climb mechanism [19].

An increase in plastic strain generally results in an increased density of dislocations either due to emission of dislocations from high angle boundaries, from the condensation of vacancies or due to the Frank Read mechanism [65]. If an FCC structured polycrystalline sample is pulled in tension to a small amount of strain, it has a certain amount of mobile dislocations that can glide freely in one slip system. When an increase in load dislocation movements suddenly starts to occur in a range of slip planes this causes the dislocations to tangle and to the formation of dislocation cell type sub-structures. The walls of these dislocation substructures have a very high density of dislocations and have been pointed out as a source of back stress, referred to as the Bauschinger effect since they impede the free movement of dislocations [66]. This can result in a lowered creep strain rate and an increase in the creep resistance of a material. With an increase in dislocation density the amount of mobile dislocations decreases which impedes further movement.

Another frequently encountered obstacle for mobile dislocations is called forest dislocation junction and can be understood as intersections with dislocations on different slip planes.
The diffusion of atoms and vacancies can be enhanced by thermal activation which subsequently results in higher mobility of dislocations since obstacles can be overcome more easily.

2.4 Creep failure

Creep failure (rupture) at high temperatures occurs either based on transgranular or intergranular fracture [41]. Both failure modes are illustrated in Figure 2.14 where fracture was produced by static bending tests in sulphur doped nickel polycrystalline samples.

![Figure 2.14: Shows the typical fracture modes produced by a static bending test for a sulphur doped nickel polycrystalline sample - left: typical transgranular fracture; right: typical intergranular fracture [67].](image)

Figure 2.15 illustrates the fracture mechanisms for Type 316H stainless steel at various stress and temperature based on a map by Langdon and Miller [68]. Transgranular creep fracture which is often referred to as ductile creep fracture, can be associated with a relatively high elongation accompanied by a reduction in area or necking. The location of the cavities are commonly around inclusions within the crystal matrix [69,70]. The failure of the specimen results from cavity nucleation and growth quite similar to micro-void coalescence for dimple rupture causing transgranular cracking.
At lower stresses and elevated temperatures, creep fracture can be caused by cracks formed at triple junctions due to grain boundary sliding. The deformation of the grain boundaries is due to dislocation glide and climb activity [71]. Intergranular fracture is a mode that is characterised by cavity growth on grain boundaries that are perpendicular to the tensile stress and cause fracture. Continuous diffusion of vacancies causes cavity growth resulting in creep failure with low ductility. Small or no reduction at all in the loading area of the test specimen at fracture is another significant attribute of this creep-brittle fracture mode [70]. This failure mode is associated with lower stresses compared to transgranular failure. At very high stress and strain rates, fracture occurs as a result of microstructural...
instabilities leading to necking which is associated with plastic deformation rather than creep. An illustration of the different failure modes is given in Figure 2.16.

![Figure 2.16](image)

**Figure 2.16:** Schematic drawing of the three dominant fracture mechanism for creep conditions at high temperature; (a) intergranular creep fracture due to voids and wedge cracks; (b) growth of cavities by power law creep both transgranular as well as intergranular; (c) rupture due to dynamic recovery or recrystallisation which is often associated with necking [19].

### 2.4.1 Creep failure mechanism in 316H stainless steel

Figure 2.17 provides an overview of the failure modes that have been observed for Type 316H stainless steel as a 2D plot over time to rupture and applied stress [72]. It also shows the conditions of interest to EDF Energy which covers a stress range of 100-300 MPa at a temperature range of 500 – 550°C and a stress range of 10-100 MPa at 650°C. From observations by EDF Energy and several other groups, it has been established that failure of solution treated Type 316H stainless steel components seems to be associated with second phase particles. In the stress range of 150-200 MPa cavitation appears at times beyond ≈ 10,000 hours. Only with an increase in temperature can intergranular cavitation be observed at lower stress ranges [72].
This can be explained with faster growth of precipitates that act as stress raisers and therefore facilitate the nucleation of grain boundaries even at low stresses. If stresses become even larger and grain boundary sliding can’t be impeded by second phase particles wedge cracks at triple junctions form. While the process of creep cavitation is heavily associated with second phase particles the formation of wedge cracking is more intrinsic [71]. Chen and Argon noted that although wedge cracking has been considered a separate phenomenon there is evidence that it is the result of the coalescence of growing cavities around point junctions if stresses are relatively low. At higher stresses it becomes the result of grain boundary sliding [73]. Evidence for their assumptions provided by SEM micrographs is depicted in Figure 2.18.
As illustrated in Figure 2.17, transgranular creep failure in the solution treated 316H stainless steel begins at stresses above 150 MPa at a temperature of 500 – 550°C. For stresses below 150 MPa grain boundary sliding is quite pronounced. The expected mode of failure here is caused by intergranular cavitation and triple point cracking.

2.5 Effect of prestraining on subsequent creep properties

Nuclear power plant components are often plastically deformed during manufacture and may subsequently be welded to get them into the required shape (e.g. boiler tube bifurcations in AGR plant). Prior deformation, whether it is hot or cold work deformation, plays an important role in strengthening the materials by producing dislocation barriers. To optimise the component’s lifetime and performance, a deep understanding is needed to quantify the response of the material to prior-cold work. The effect of prior cold work on
subsequent high-temperature behaviour has been investigated since the 1960s. Due to the sensitivity of materials at elevated temperatures to many factors such as material composition, microstructure, sample geometry etc. conflicting outcomes have been reported [74]. Willis and Wilshire, for example, found that tensile prestrain at room temperature has an enhancing effect on the creep resistance of austenitic Type 316H/L stainless steels. Zhang and Knowles on the other side showed a reversed effect in their work on Nickel-based C263 prestrained in tension [16]. Studies from Garofalo et al. have also shown that prestrain in tension can lead to a beneficial effect on creep resistance and creep life [75]. Cold work such as prestrain on a material can have the effect of using up some of the mobile dislocations that would be available which leads to a reduced primary creep rate. On the other side, secondary creep is controlled by climb which preferentially occurs on so-called jogs. Jogs are created in most cases not just due to thermal movements but mainly due to plastic or creep deformation. So if jogs facilitate climb, this would increase the secondary creep rate. By pinning down mobile dislocations and creating jogs the effect of pre-strain can go either way dependent on various factors such as material temperature and stress.

Several authors working on austenitic stainless steels found an impeding effect of prestraining on the creep behaviour at elevated temperatures [15,76,77]. Ohashi et al. investigated the effect of pretension on subsequent creep behaviour in Type 316 stainless steel [76]. The pretension was conducted at a temperature of $650^\circ C$ to levels of 1%, 2% and 3% plastic strain. The creep test followed at the same temperature of $650^\circ C$. Figure 2.19 shows the creep resistance effect for the first 100 h of a test implemented at a constant stress of 140 MPa and a temperature of $650^\circ C$. There is a significant reduction in creep strain after minimal amounts of pretension.
Kikuchi et al. similarly observed, in their tests on Type 304 stainless steel, that a small amount of pre-straining at high temperatures increased the creep strength of the material [77]. The pretension was carried out at a temperature higher than the creep test temperature. They found that the creep strength of Type 304 stainless steel can be improved by small amounts of prestrain at high temperatures and that the initial deformation stage within primary creep was heavily influenced by the amount of prestrain. A characteristic decrease of primary creep and primary creep strain rate was found with an increased amount of prestrain.
Wilshire and Willis investigated the effect of prestrain in tension at room temperature and at a creep temperature of 575°C for samples of Type 316H as well as 316L stainless steel [15]. They divided their specimen into three distinct groups, partially solution-treated Type 316H, a full solution treated batch of Type 316H and a full solution treated group of Type 316L stainless steel. The solution treatment was done for 1 hour at 1050°C followed by water quenching. Their partial solution treated samples were taken from power plant components produced in a partial solution treated condition. The results from their creep tests for fully and partially solution treated specimens are shown in Figure 2.21. Similar to Kikuchi et al. [77], as illustrated in Figure 2.20, they could observe a significant drop in creep strain rate and creep strain. However, they tested creep samples after prestraining at room temperature. Wilshire and Willis even found a different outcome of prior cold work for partially solution treated and fully solution treated 316H SS. While the prior shows a decrease in creep strain, creep strain rate as well as time to fracture with an increase in prestrain, fully solution treated samples exhibit an increase in time to rupture with an
increase in prestrain. This is explained due to a non-homogeneous carbide distribution in the partial solution treated samples. The authors suggest that the grain boundary zone of the microstructure remains unchanged [15].

![Figure 2.21](image)

**Figure 2.21:** a) constant stress creep curve for partially solution treated Type 316H stainless steel prestrained in tension at room temperature (RT) to various degrees and creep tested at a stress of 450 MPa at 575°C; b) constant stress creep curve for fully solution treated 316H stainless steel prestrained at RT to various degree and creep tested at a stress of 450 MPa at 575°C [15].

Work on material pre-conditioning effects on the creep behaviour of Type 316H stainless steel was picked up again by Mehmanparast et al. [78, 79]. They investigated creep crack initiation and creep crack growth behaviour in Type 316H SS at a temperature of 550°C. Their results for the creep properties are illustrated in Figure 2.22 where the creep strain over time is plotted for different levels of pre-compression (PC). With an increase of material pre-compression, there is a decrease in creep ductility. They investigated material that had been pre-compressed to 4%, 8% and 12%. They have concluded that the lowest creep ductility is accompanied by the highest creep cavity growth in the 12% PC material. And also, that an increase in pre-compression in an ex-service material leads to a higher density of creep cavitation and subsequently lower creep ductilities and an increase in cavity
growth rate. The creep rupture behaviour that is strongly dependent on intergranular failure due to an increase in cavitation along boundaries becomes the dominant creep failure mechanism [78].

Auzoux et al. investigated the behaviour of relaxation cracking in Type 316H-L stainless steel over a long period beyond 100,000 hours. Auzoux observed that to achieve a certain microcrack density a significantly lowered creep deformation is sufficient if prior cold work in tension is present. Hardening due to cold work is responsible for relaxation cracking [80].

Figure 2.22: Creep curves for Type 316H stainless steel AR, 4%, 8% and 12%, precompressed (PC) materials tested at 300 MPa and at a temperature of 550°C [78].
2.5.1 Microstructural changes from prestraining

Dyson and Loveday confirmed a strong correlation between prestraining in tension and cavity density in their work on Nimonic 80A, illustrated in Figure 2.23 [81]. They examined TEM thin foil samples of material that had been plastically deformed at room temperature in uniaxial tension, torsion and compression.

![Figure 2.23: Variation of number density of cavities with changes in effective plastic strain at room temperature for three states; (●) tension, (○) torsion and (×) compression [82].](image)

Dyson and Loveday found an increase of prestrain-induced cavitation on the grain boundaries parallel to the applied stress. Growth at these boundaries that experience the highest tensile stress indicates that growth by diffusion is the dominating mechanism [82]. Loveday et al. investigated the effect of prestrain on the subsequent creep properties in IN597, another Nickel-based superalloy, and observed, similar to [82], an increase of cavity nucleation and growth as a consequence of prior tensile plastic strain [83]. They could observe an increase in microcracks as a consequence of prior plastic strain and suggested that these microcracks provide the potential sites for growth of cavities. As illustrated in Figure 2.24, creep damage in prestrained IN597 samples is characterised by the appearance of small
cavities at transverse grain boundaries to the loading direction. The non-prestrained samples of IN597 showed coalescence of creep cavities but with much larger spacings than in the prestrained samples.

![Figure 2.24](image.png)

**Figure 2.24:** a) intergranular creep damage in prestrained IN597, characterised by many small r-type cavities along transverse grain boundary - creep stress 300 MPa at 750 °C, rupture time 22 h; b) intergranular creep damage in non-prestrained IN597 that consists of wide cracks due to coalescence of cavities, cavities have larger spacing - creep stress 300 MPa at 750 °C, rupture time 208 h, loading direction upwards [83].

Shiozawa and Weertman [84] found that prestraining in tension in astroloy at room temperature results in significant grain boundary damage predominantly between carbides and matrix in the form of microcracking. They noted that prestraining introduces residual stresses that affect the creep fracture behaviour. Based on a theoretical model assuming an elliptical inclusion the stress distribution was calculated. It was suggested that the residual stress builds up around carbide and grain boundary. This has a significant outcome for the creep properties of the material resulting in a creep enhancing effect by an order of a
magnitude [84]. In their microscopic studies using TEM, they found that the substructure of grains as well as the amount of precipitation of carbides changes with the amount of pretension. An explanation of the decrease of primary creep strain rate is an evolution of dislocation subgrain structures. Another argument for the decrease in primary creep rate can be made from an accelerated precipitation of carbides. Etienne et al. reported carbide nucleation and growth in particular on grain boundaries, subgrain boundaries and dislocations [85]. In various studies, it was pointed out that the dislocation density can be altered significantly through an increased plastic strain. The dislocation density can be described by the Orowan equation [86]

\[ \dot{\epsilon}_{cr} = \rho_m b v, \]  

where \( b \) describes the Burger’s vector length, \( v \) is the average velocity of the dislocations and \( \rho_m \) is the mobile dislocation density. A high plastic strain before creep leads to an increase in dislocation density. If this cannot be resolved in the creep process due to thermal recovery, the mobile dislocation density is increased this leads to a reduction in creep strain rate. When the mobile dislocation density is not affected by prior plastic strain it does not lead to a decrease in creep strain, which was observed by [16,87]. Prestraining can also have an effect on the lattice structure when it produces high angle grain boundaries as reported by [88]. A greater number of high angle boundaries can lead to an increasing nucleation rate of dislocations which subsequently might lead to dislocation pinning and material hardening. On the other side, they can lead to an increase in dislocation annihilation rate due to an acceleration of edge dislocation in dipole configuration due to grain boundary diffusion [88]. Li et al. found that for a high density of high angle boundaries, the softening mechanism due to dislocation annihilation becomes the dominant contribution leading to a creep enhancing effect [88]. These two effects are competing within the creep process and can cause different resulting behaviour.
2.5.2 Temperature effect of prestraining

A temperature effect in the effect of prestrain was found by Kikuchi et al. in Type 304 stainless steel, by Cairney et al. in Ti-52 Al and by Rong in single-crystal Ni$_3$Al \cite{77, 89, 90}. It was found that prestraining at high temperature was leading to a different creep behaviour than prior prestrain at room temperature.

![Figure 2.25](image)

**Figure 2.25:** Effect of pretension on creep rate versus creep strain for specimen prestrained to 1\% at various temperature above the creep test temperature (650\,°C, 750\,°C). The creep tests were carried out at a temperature of 600\,°C and at a stress of 240 MPa \cite{77}.

As shown in Figure 2.25, prestraining of Type 304 SS at an increasing temperature has a decreasing effect on the creep resistance. Kikuchi et al. explained this behaviour by dynamic strain ageing which occurs in a temperature range between 300 – 600\,°C and leads to a maximum in work hardening within that range. Another explanation comes from Cairney et al. who found that samples prestrained at high temperatures evince Kear-Wilsdorf locks that are shorter in length resulting in a decrease in softening compared to samples prestrained at room temperature \cite{89}. Furthermore, it was mentioned by Goods and Brown that prestraining at room temperature makes it more susceptible to cavitation and formation of the microcracks in the material. This was reported for Fe-Fe$_3$C and Cu-
SiO$_2$ systems. At higher temperatures, the dislocation mobility is still very high which makes cavity nucleation due to stress concentration from dislocation pinning a lot less likely [91].

### 2.6 Cavity nucleation

Several authors assert that cavities often nucleate on grain boundaries that are transverse to the tensile stress and that nucleation of cavities can be associated with the maximum principle stress that is acting on the grain boundary [92–95]. Cavities can be associated with second phase particles in alloys and usually do not occur in high purity materials. Yavari and Langdon found that cavitation occurs in lower purity metals such as 99% Al whereas it is non-existent in high purity 99.999% Al [96]. In austenitic steels, cavities mainly nucleate where carbides are present [17,46,95,97,98]. These particles often impede the grain boundary sliding which causes a stress concentration around them. Pre-nucleated voids are likely to exist in ex-service materials that are tested under high loads at elevated temperatures [17]. According to Riedel the concentration of stress leads to a local decohesion which nucleates a cavity [46]. Cavity nucleation can be explained by two main mechanisms. Either cavitation damage due to interface fracture or as a process of thermally activated vacancy clustering. Both mechanisms have in common that they need very high local stresses. It was claimed by Dyson and other authors that cavities are nucleated throughout the component’s lifetime and that the number density of cavitation damage is proportional to the creep strain for several engineering alloys [29,46,99].

Kassner and Hayes categorise the nucleation theories into several different schemes where four of them are illustrated in Figure 2.26. It can be summarised that cavity nucleation is controlled by [44]:

- nucleation from vacancy condensation at high stress region
- grain boundary sliding leading to cavitation from ledges
• cavity nucleation from dislocation pile-ups

• cavity formation from a particle-obstacle which slows down the sliding and causes stress concentration

![Figure 2.26](image)

**Figure 2.26:** Different ways of cavity nucleation, (a) grain boundary sliding leading to cavitation damage from ledges, (b) cavity nucleation from vacancy condensation at triple junctions, (c) cavity nucleation from dislocation pile-ups (Zener-Stroh mechanism), (d) formation of cavity from particle obstacle in conjunction [44].

### 2.6.1 Cavity nucleation due to thermal activation

Based on the works of Greenwood, Raj and Ashby developed the theory of cavity nucleation by thermally activated vacancy formation [100, 101]. According to them, the nucleation process can be considered as the condensation of atomic vacancies and thus makes the nucleation of voids mostly stress controlled. Derived from their theory, a certain threshold stress and an incubation time for cavity nucleation can be calculated. According to Raj et al., the incubation period is determined by the number of vacancies contained in a cluster.
of a critical size. An increase in stress can lead to even smaller critical clusters growing due to a higher influx of vacancies. If the applied stress exceeds a certain threshold stress $\sigma_n$, mostly all nucleation sites within the material are activated. However, if the applied stress is smaller it takes a longer time to nucleate any cavities. Furthermore, cavity growth is inhibited because sintering is energetically more favourable than growth. Riedel noted that the stress that is necessary for a stable cavity nucleation and growth, due to a diffusion of vacancies, has to be 30-300 times greater than stresses at which nucleation of voids was investigated already [46]. The cavitation in pure metals is observed at applied stresses below 10 MPa or even at 1 MPa in copper bicrystals [53, 102]. An explanation for this observation might be, that the local stress compared to the applied stress may be enhanced by grain boundary sliding at second-phase particles, dislocation pile-ups or by slip bands. Riedel found that a stress concentration of at most 20 for an applied shear stress during grain boundary sliding can be expected due to hard second phase particles [103].

![Figure 2.27:](image)

(a) (b)

Figure 2.27: (a) stress concentration sites in a single phase polycrystal; (b) at second-phase inclusion particles [99].
Explanations for stress concentration in a polycrystalline material can be found in Figure 2.27 either for a single-phase sample or when second-phase particles are present. When a polycrystal is subjected to tensile stress the response of the specimen is homogeneous linear elastic deformation. When the shear stresses are relaxed due to grain boundary sliding strain localisation at irregularities such as grain boundaries does occur. Raj and Ashby deduced that an incubation time is required for the nucleation process that is quite short, in the order of a few microseconds [104]. According to them, cavity nucleation can be considered instantaneous once the threshold stress is reached. Evans suggested that the increase in the number density of cavities that is observed by several other authors is indeed an artefact and all cavities are nucleated instantaneously. Since the sliding of grain boundaries which determines the growth varies from grain boundary to grain boundary cavities become detectable at different times which leads to the false conclusion that nucleation happens continuously [46, 53]. This approach, however was criticised by Dyson. It was pointed out that in materials where cavities grow by diffusion they reach the detectability very quickly [29].

![Diagram](image)

**Figure 2.28:** Volume of critical cavities for a fixed value of $r_c$ for homogeneous as well as heterogeneous nucleation, one can obtain a decreasing critical volume of void formation and therefore a higher probability of stable nucleation of cavities [104].

According to Raj and Ashby, cavities can have different shapes based on their location of nucleation. This can be at a two-grain junction, three-grain junction, four-grain junction or at the interface of an inclusion that is present [104]. This is illustrated in Figure 2.28. One can further distinguish voids that nucleate at inclusion free grain boundaries and voids that are formed in boundaries that contain inclusions. From Figure 2.28 it can be found that the critical size for a void to become stable around a second phase particle is a lot
smaller than at a location between two grains.

2.6.2 Cavity nucleation due to grain boundary sliding

Grain boundary sliding at second phase particles and triple points leads to high local stresses [103]. One would expect that if grain boundary sliding is the dominant mechanism for cavity nucleation, damage would occur due to shear mostly along grain boundaries oriented at 45° concerning the maximum principal stress. However, it was found by several authors that cavities are observed mainly at boundaries transverse to the applied stress [73, 82]. Furthermore, since cavities are predominantly spherical rather than plate-like in shape, this suggests that grain boundary sliding might not be the dominant mechanism for cavity growth. Even in bicrystals on boundaries perpendicular to the applied stress and hence with an absence of shear stress and grain boundary sliding cavities could be found [105]. It appears that grain boundary sliding is not a necessary condition for the evolution of cavity nucleation. It was suggested by Harris et al. that to initiate cavity nucleation a grain boundary sliding displacement rate is necessary that is dependent on the size of the cavities. To nucleate smaller cavities a higher grain boundary sliding (GBS) rate is required. Cavity nucleation happens if the GBS rate exceeds the longitudinal sintering rate of the decohesions [106, 107]. Based on this work Sandstrom et al. proposed a nucleation model based on GBS. Cavity nucleation happens if a particle or sub-boundary corners of one side of a sliding grain boundary interact with a sub-boundary corner on the other side of the sliding grain boundary [108]. The model results in a cavity nucleation rate that is strongly correlated to the creep strain rate. Their model predictions of cavity number per unit grain boundary area as a function of creep strain were verified using several quantitative metallography studies in Type 304 and 347 stainless steel [109, 110].
2.6.3 Cavity nucleation due to dislocation pile-ups

It was shown by Dyson that cavities could be observed in creep samples of Nimonic 80A that were prestrained at ambient temperature [29, 82]. Since thermally activated cavity nucleation does not explain this, the Zehner-Stroh mechanism was used as an interpretation [111]. The mechanism involves a pile-up of dislocations at grain boundaries with a second phase particle or an obstacle present. These observations were verified by Kassner et al. in their creep experiments on high purity silver at room temperature [112]. According to their observation, cavity nucleation occurs continuously and not in a short period as proposed by Raj and later by Evans [53, 104]. Several other authors found that cavity growth and cavity nucleation occurs continuously over the whole creep life [29, 113–115]. Dyson explains cavity nucleation as a product of localised grain deformation instead of grain boundary sliding. According to him engineering alloys with their dense fraction of boundary second phase particles result in too low levels of stress intensification by purely sliding [29]. During creep, an increment of strain produces a range of voids with a certain distribution of spherical radii. This is illustrated in Figure 2.29.

Figure 2.29: A distribution of cavities with only those becoming stable where the radii exceeds a certain radius \( r > 2\gamma/\sigma_1 \) [29].

Only cavities with a radius \( r \) larger than \( 2\gamma/\sigma_1 \) become stable. \( \sigma_1 \) is in the direction of
the maximum principal stress. The maximum principal stress explains a higher density of cavitation damage on grain boundaries perpendicular to the applied stress regardless that the decohesions are completely random [116]. It was proposed by Lim that stress concentration can be caused due to dislocation pile-ups at intersections of subgrains and grain boundary dislocations [116]. If a dislocation pile-up model is considered there is no incubation time for cavities since this is a steady-state phenomenon of secondary creep. Lim et al. found that the stress concentration is large enough to trigger cavity nucleation and that it is thermodynamically feasible for cavities to nucleate at subgrains within the polycrystal structure of copper [116].

2.6.4 Smallest observed cavities

The critical size of a nucleus has not yet been determined experimentally but the prediction is around 2 to 5 nm which is very challenging to detect [117]. SEM can monitor stable creep cavities down to a minimum size of about 20 nm [118]. Using TEM it is possible to detect even smaller cavities as small as around 3 nm [119]. For Type 316H Stainless Steel Chen et al. calculated it to be 20 nm [120]. The critical size of a cavity strongly depends on the uniaxial stress state of the sample, the change of the grain and interphase boundary energies due to local composition and the anisotropic behaviour of strain within the material. As explained before, cavities at second phase particles have a higher probability of nucleation with a much lower nucleation barrier and the critical radius to become stable can be quite different from voids without the presence of second phase particles [26].

2.7 Cavity growth models

As mentioned earlier, creep fracture in austenitic steels is dominated by either wedge-type cracking at grain boundary triple points or caused by nucleation and growth of creep cavities at grain boundaries. Cavity nucleation is caused by three main mechanisms that include vacancy accumulation, grain boundary sliding or dislocation pile-ups as described
earlier in Section 2.6.1, 2.6.2 and 2.6.3 [29,101,103]. Morris and Harries found that the ductility of Type 316 stainless steel depends on the state of the intergranular precipitates during the creep process. In general, high ductility is related to short rupture times and high stress levels whereas a low creep ductility is expected to be low at low stress levels and for long creep rupture times [31,32,121].

Holdsworth proposed a ductility model that exhibits four regimes above a certain temperature $T_c$ at which creep occurs [122]. At high stresses, the ductility is relatively high with a transgranular failure mechanism due to plastic hole growth. It is followed by a transition region where the ductility decreases due to an increase in void nucleation and growth along grain boundaries. Regime III is a lowered plateau where constrained cavity growth is the dominant mechanism. In regime IV the ductility slowly increases due to significant inhibition of void nucleation and growth. The ductility model proposed by Holdsworth is depicted in Figure 2.30. In the right illustration, it can be found that the mechanisms vary quite significantly with a temperature change. The model was fitted to X19CrMoVNbN steel but Holdsworth admits that there is high complexity in fitting it to multi-temperature creep data collections.
Figure 2.30: (a) Schematic illustration of the creep ductility regimes for a ferritic steel; (b) and experimental evidence for a range of different temperature [122].

Dyson contributed significant work to identify creep mechanisms, mathematical representations of the damage or mechanisms that describe the damage evolution [123,124]. Based on his work Hales proposed a different regime model that reflects on the creep strain rate and its effect on a change in creep ductility which has been received with more acceptance [125,126]. The main mechanisms leading to cavity growth are [124,126].

- plastic hole growth
- diffusion controlled growth
- constrained cavity growth

The dominating mechanism is dependent on several factors such as material properties, temperature and stress level. The growth of cavities is caused by plastic deformation at very high stress and strain rates. At lower stress levels, the vacancy diffusion along boundaries becomes more and more dominant. In the following section, each mechanism is described more in detail. The illustration on the left side in Figure 2.31 depicts the three different regimes and the effect of inelastic strain rate on the creep strain at failure.
The right illustration shows the experimental data of Type 316H stainless steel of the three regime model \([127, 128]\). Here, the creep ductility is plotted against the normalised stress, applied stress over the yielding stress, at \(500 - 700^\circ C\) for materials with different heat treatment. For the temperature range from \(600 - 700^\circ C\) the three regimes are quite distinct. At a temperature of \(550^\circ C\) where the applied stress is significantly higher than the yielding point of the material, there is a lot of scattering in the data. Nevertheless, Mehmanparast assumes a similar progression for further calculations to predict the creep crack growth \([127, 128]\). Further experimental evidence for the validity of the model for 316 and 347 stainless steel can be found in \([129]\) and for T91, T92, as well as T122 in the work of Kimura et al. \([130]\).
to migrate and the voids to grow [126]. The diffusion process, as well as the shape of the cavity, determines the growth rate according to:

\[
\dot{r} = B_1 \dot{\epsilon},
\]

where \( r \) is the cavity radius and \( B_1 \) a constant that gives the cavity spacing \( \lambda \) at failure as:

\[
\lambda/2 = C\sigma_1^{-m}
\]

where \( C \) and \( m \) are material constants and \( \sigma_1 \) is the maximum principal stress [133]. Both can be combined to give the equation for uniaxial creep ductility as:

\[
\epsilon_{f,uni} = \frac{C\dot{\epsilon}_{in}}{A\sigma_1^{1+m}},
\]

It is worth noting that Beere and Speight proposed a model for a cavity growth rate that is not just a function of stress but of the cavity size and creep strain rate as well [134]. Needham and Gladman showed that an exponent greater than unity should be used to raise the stress. Spindler expressed that a general form of ductility given by the stress raised to the power \( n \) and the inelastic strain rate provides a good method to fit the creep data for steel [12]. The strain at failure can be represented as:

\[
\epsilon_f = A_1 \frac{\Delta G_{AC}}{RT} \epsilon_{in}^{m_1} \sigma^{-m_1},
\]

where \( A_1, n_1, m_1 \) and \( \Delta G_{AC} \) are material constants. \( \Delta G_{AC} \) is the difference of the activation energy of cavity nucleation and cavity growth. It was proposed by Hales to replace \( n_1 \) with the stress exponent \( \frac{n-1}{n} \) to give a theoretical value to the equation [126].
2.7.0.2 Creep Constrained Cavity Growth

Dyson proposed that void growth is constrained by the creep rate of the surrounding material which becomes significant especially for low stresses [135]. The rate of increased volume by the diffusive material is balanced by the deformation rate of the surrounding area or the rate of supply of vacancies at the adjacent grain boundary. This phenomenon causes a redistribution and reduction of the strain rate at the grain boundaries and subsequently constrains cavity growth. The inelastic strain at failure under these constrained conditions has been calculated by Dyson [135]:

\[ \epsilon_f = \frac{\lambda \pi}{6d}, \]  

(2.10)

where \( \lambda \) is the cavity spacing and \( d \) is the grain size. The strain at failure is therefore independent of strain rate and stress. If one takes into account equation 2.7 where the cavity spacing depends on the stress the equation for the creep ductility transforms to

\[ \epsilon_f = \frac{C \pi}{3d} \sigma_1^{-m}. \]  

(2.11)

From experimental data on the stress and temperature dependence of the creep behaviour the ductility can be fitted accordingly to the following equation

\[ \epsilon_f = A_2 \frac{\Delta G_C}{RT} \sigma_1^{-m_2}, \]  

(2.12)

where \( A_2, m_2 \) and \( \Delta G_C \) are material constants.

2.7.0.3 Plasticity

Cavities can grow by plasticity under very high strain-rate conditions simply as a result of the plastic deformation of the surrounding area. The mechanism is very similar to
McClintock’s model for cavity growth in a plastic field and gives a void growth rate of [114,126]

\[ \dot{r} = B_1 \dot{\epsilon} \]  

which by integration leads to a creep strain failure of

\[ \epsilon_f = \frac{\lambda}{2B_1} \]  

where \( \lambda/2 \) is the half mean cavity spacing and \( B_1 \) is a material constant. The growth of intragranular cavities is independent of the inelastic strain rate as well as on stress and the creep failure strain and can be represented by a constant failure strain \( \epsilon_u \). It should be noted that for all models failure is considered when void growth reaches from an initial radius \( r_0 \) to the half mean cavity spacing \( \lambda/2 \) [126].

### 2.7.1 Ductility exhaustion approach

For austenitic steels the R5 procedure uses a ductility exhaustion approach to calculate creep damage [136]:

\[ d_{R5}^c = \int_0^t \frac{\dot{\epsilon}_c}{\epsilon_f^*(\dot{\epsilon}_c, T)} dt, \]

where \( \dot{\epsilon}_c \) is the derivative of the creep strain, \( \epsilon_f^* \) is the corresponding multiaxial creep ductility, a function of both, creep strain rate and temperature. Failure of a component occurs if the damage parameter \( d_c \) reaches unity. To calculate the creep damage of a component the multiaxial creep ductility needs to be determined which is simply the product of the uniaxial creep ductility and a multiaxial strain factor \( MSF (\epsilon_f^* = \epsilon_f \cdot MSF) \) that considers void growth models as a function of triaxiality of stress. The \( MSF \) factor can be based
on either physical models or empirical observations. A physical process that considers a
direct micromechanical calculation of the grain boundary cavity growth using power law
creep was developed by Cocks and Ashby [137]:

\[ \frac{\bar{\epsilon}_f}{\epsilon_{f,uni}} = \frac{\sinh[2/3(n-0.5)]}{\sinh[2(n-0.5)/n+0.5]}. \] (2.16)

where \( n \) is the creep strain exponent, \( \sigma_1 \) is the maximum principal stress, \( \sigma_h \) is the hy-
drostatic stress, \( \sigma_{eq} \) is the equivalent or von Mises stress and \( \epsilon_{f,uni} \) is the uniaxial creep
ductility. Another approach based on a semi empirical model that is a further development
of the Rice and Tracey model and considers the effect of multiaxial stress on the cavity
growth has been developed by Spindler [138, 139]. It includes a factor that takes cavity
nucleation into account and can be described by:

\[ \frac{\bar{\epsilon}_f}{\epsilon_{f,uni}} = \exp[p(1 - \frac{\sigma_1}{\sigma_e}) + q\left(\frac{1}{2} - \frac{3\sigma_h}{2\sigma_e}\right)], \] (2.17)

where \( p \) and \( q \) are material constants that define if either cavity nucleation or cavity
growth is the dominant mechanism. Spindler could show that cavity growth alone was
not accountable for the change in creep ductility based on different creep strain rates.
The cavity nucleation is driven by the diffusion of vacancies under stress whereas the void
growth is dominated by power law creep of the matrix.

2.7.1.1 Stress modified ductility exhaustion approach

The stress modified ductility exhaustion model is, compared to the ductility exhaustion
model, an improved method that simply includes the effect of stress on creep damage by
treating the ductility as a function of stress and strain rate.

\[ d_{c}^{SM} = \int_{0}^{t} \frac{\dot{\epsilon}_c}{\bar{\epsilon}_f(\dot{\epsilon}_c, \sigma, T)} dt, \] (2.18)
Here, the inelastic strain at failure is a function of maximum principal stress, temperature and von Mises inelastic strain rate. It was developed by Spindler based on recent studies of creep damage calculations in Type 316H stainless steel [127,131]. Spindler could show that creep damage calculations based on inelastic strain rather than simply creep strain leads to better life-time predictions. Spindler notes that for creep tests conducted beyond the 0.2% it is not possible to distinguish time-independent plastic strain from time-dependent creep strain. In these cases, additional plastic strain accumulates during a creep test which subsequently makes it impossible to distinguish damage caused by plastic strain from that caused due to creep [12,129]. The von Mises inelastic strain at failure can be calculated as the product of the uniaxial inelastic strain at failure and a cavity growth factor (CGF).

\[
\bar{\epsilon}_f(\dot{\epsilon}_{in}, \sigma_1, T) = \epsilon_f(\dot{\epsilon}_{in}, \sigma_1, T) \cdot CGF
\] (2.19)

The model treats the ductility as a function of stress and strain and uses the same three failure mechanisms proposed by Hales for cavity growth that are considered in the R5 method [126,136]. There is an upper shelf failure accounting for very high inelastic strain rates where cavity growth is driven by plastic instability resulting in transgranular fracture. With lower inelastic strain rates, a transition region is observed resulting in sharply lowered inelastic strains at failure. The cavity growth here is triggered by diffusional controlled mechanisms. At even lower strain rates there is a lower shelf regime where the inelastic strain at failure is independent of the strain rate but shows a dependence on stress. This stress-dependent region is controlled by a constrained cavity growth. Both, the transition as well as the stress dependent region, are causing intergranular fracture. Spindler adds that these models should not be considered as mechanistic models but rather empirical ones [12]. A schematic illustration of the stress modified ductility exhaustion SMDE model can be found in Figure 2.32.
The three different regions leading to different failure mechanisms can be used in the following set of combined equations in case of a multiaxial inelastic strain at failure:

\[
\bar{\epsilon}_f = \text{MAX} \left\{ \text{MIN} \left[ \epsilon_u \exp \left( \frac{1}{2} - \frac{3\sigma_m}{2\sigma_c} \right) A_1 e^{\frac{\Delta G_{\text{AC}}}{RT} n_1} (\bar{\epsilon}_i \nu')(\sigma_1)^{m_1} \frac{\sigma_c}{\sigma_1} e^{\exp \left( \frac{1}{2} - \frac{3\sigma_m}{2\sigma_c} \right)} \right] \right. \\
\text{MIN} \left[ \epsilon_u \exp \left( \frac{1}{2} - \frac{3\sigma_m}{2\sigma_c} \right) A_2 e^{\frac{\Delta G_{\text{AC}}}{RT} (\sigma_1)^{-m_2} \frac{\sigma_c}{\sigma_1}} e^{\exp \left( \frac{1}{2} - \frac{3\sigma_m}{2\sigma_c} \right)} \right] \right\}
\] (2.20)

To account for the multiaxial state of stress Spindler simply adds the void growth model proposed by Hull and Rimmer [138].

### 2.8 Experimental methods to determine creep cavity damage

There is a range of experimental methods available to study creep damage. These methods can be categorised as either destructive or non-destructive. The benefits and drawbacks of
each method have been reviewed by Bouchard and Rist as well as by Sposito et al. [141, 142]. Figure 2.33 illustrates the spatial resolution of each of the measurement techniques compared against the length-scale of creep damage.

The present literature review focuses on the two measurement techniques which are used in this project to study submicron cavitation: small angle neutron scattering (SANS) as well as scanning electron microscopy (SEM).

2.8.1 Relevant small-angle neutron scattering studies

Since its discovery in the 1930s, where small angle neutron scattering was firstly observed by Guinier in experiments on Al-rich alloys, it has been used as a technique to investigate metals and alloys. The existence of very small but coherent precipitates was first observed as Guinier-Preston zones [143–145]. Since neutrons are relatively sensitive to differences in
the neutron scattering length density of different elements, minor changes in the composition of alloys, precipitate evolution and cavitational damage can be detected with neutrons. The small-angle scattering technique has therefore been used extensively to study features such as phase separation, precipitate growth and dissolution etc. Before synchrotron radiation sources became more accessible Al-rich alloys were found to be one of the few materials that could be studied with X-rays. For neutron scattering and its preferred attributes for heavy atoms and bulk structures, many other systems could be investigated [145]. Even though SANS doesn’t reveal a direct image, it has the huge advantage of being statistical accurate due to a large sampling volume. In contrast to X-Rays, very small absorption of neutrons allows an examination of most alloys up to a few mm thick. While a technique such as TEM, that covers a comparable spatial resolution, can characterise the size and the shape of individual cavities quite precisely it struggles to sample large enough areas. This makes it challenging to extrapolate statistical information of a larger collection of cavities. As mentioned earlier every small variation in the scattering length density results in a detectable small-angle scattering signal. In metals and alloys, it is therefore possible to investigate the effect of dislocations and grain boundaries. The effect of dislocations on the small-angle neutron scattering signal was reviewed by Schmatz and was found to be negligible. Even for a high density of dislocations, the cross-sections are so small that small defects such as cavities or pores or precipitates disguise their existence in the scattering pattern [146].

One of the first small-angle neutron scattering experiments to study the effect of grain boundary cavitation was done by Saegusa et al. [147]. In their investigation of a polycrystalline copper sample that had been fatigued in reverse bending at a temperature of 405°C it was reported that small cavities with a size smaller than 100 nm form at grain boundaries. Anisotropic scattering was reported and led to the conclusion that the voids were cuboidal [147]. With the success of these early experiments, other studies followed that concentrated further on creep fatigued copper. In the following study the SANS signal of a heavily fatigued copper single crystal was found to be similar to an unfatigued polycrys-
talline copper sample. It was concluded that changes in the scattering intensity within a creep fatigue process can be assigned to grain boundary cavitation. Furthermore, the void growth over time was analysed. It was found that after a rapid growth at the beginning, the increase slows down [148]. In a further study on creep fatigued polycrystalline copper, it was reported that the process of cavity nucleation was indeed continuous during creep fatigue. Page et al. analysed the data from the viewpoint of a classical two-phase model with a copper matrix and voids and found cavities in a size range of 10-90 nm at a volume fraction of around $5 \times 10^{-7}$. With such small features for scattering objects, it was proven that SANS is powerful as a technique to measure the size distribution as well as the nucleation and growth of cavities at grain boundaries. Yang and Weertman used the SANS data from copper samples at $405^\circ C$ at a stress of 27.6 MPa crept for various times to validate the, at the time commonly used, cavity growth model by Hull and Rimmer that was later corrected by Speight and Beere [132, 134, 149]. They also compared the experimental data to models that couple plasticity with vacancy division as developed by Chen and Argon as well as Martinez and Nix [73, 150]. They found good agreement between the SANS data and the predictions made by Beere and Speight for creep times below $1 \times 10^4$ seconds. The diffusion-plasticity model, however, was found to overestimate the cavity growth rate based on the SANS results and later confirmed by density change measurements. This is illustrated in Figure 2.34.
Figure 2.34: Size distribution of small voids in copper samples crept at 405°C under a stress of 27.6 MPa for several times of (a) $1.1 \times 10^4$ s; (b) $2.2 \times 10^4$ s; (c) $4.3 \times 10^4$ s; (d) $7.7 \times 10^4$ s. In each plot, the SANS experimental data is shown together with modelling results based on a grain boundary vacancy diffusion model, a diffusion coupled model with enhanced plasticity (Chen and Argon) and the Martinez and Nix model [149].

Their calculations of the size distribution was based on the assumption that voids with a diameter below $1 \mu m$ are spherical which was confirmed by High Voltage electron mi-
crosscopy (HVEM) on fatigued as well as crept copper samples [151]. In Figure 2.35 their observation on the change in number density as well as on cavity growth is illustrated.

![Figure 2.35: Size distribution of small voids in copper samples crept at 405°C under a stress of 27.6 MPa for several times [149].](image)

Yoo et al. studied the distribution of creep cavities in nickel by fatigue deformation at elevated temperatures using SANS and HVEM. Similar to Page et al. [151] it was found that the maximum cavity size from HVEM is larger than the estimated cavity size determined by SANS. Both Yoo and Page used the gyration radius to calculate the mean size of the largest cavities [151,152]. This approach, however tends to be not precise enough since the definition of the Guinier region for large voids is dependent on the smallest angle at which SANS measurements can be made. Another possible explanation for the discrepancy can also stem from errors in the sample preparation process. Due to etching effects, the size of cavities can appear much larger than their true size.

Several groups have studied the quantification of grain boundary cavitation using small-angle neutron scattering in austenitic steels. Boeuf et al. were the first to investigate creep cavities in Type 304 stainless steel using small angle neutron scattering [153]. While for studies in copper the scattering can be attributed to cavities only, commercial alloys such
as Type 304 SS and 316H SS contain many precipitates of second phase particles that contribute to the scattering signal. Yoo et al. deployed a method using post-creep solution heat treatment at a temperature of 1100°C for AISI 304 stainless steel which was followed by rapid quenching. The idea here was to remove any second phase particles that add to the scattering signal while leaving all cavities. The resulting signal was attributed to just cavitational damage [152,154].

![Figure 2.36: Size distribution of carbides in the (a) as crept, (b) control sample in the plot and (c) the post creep annealed sample. The dashed lines represent the net stress-induced damage [152].](image)

It was argued, based on the results presented in Figure 2.36, that the net scattering as shown by curve e results mainly from carbides rather than creep cavities. The same idea was picked up by Page et al. who conducted SANS investigations on Type 304 stainless steel as well as on alloy 800 and found an increase in the gyration radius and a decrease in integrated intensity [155]. While Yoo et al. reported an increase in scattering from the creep process, Page et al. reported a decrease for both materials. Since neither creep cavitation nor further precipitation can account for the decrease in the signal it was argued that the Type 304 SS used in the previous studies by Yoo et al. was solution treated whereas the material used by Page was in a fully precipitated condition. Carbide coarsening was reasoned to
explain the decrease in scattering signal. Their argument is not satisfying and it has to be noted that the degree of information provided by scattering on post creep annealed samples is impaired by the uncertainty about changes in the cavity size distribution due to solution treatment. It might be that in the process small cavities sinter and disappear or precipitates fill the voids during annealing. This effect was recently studied in high-purity FeAu and FeAuBN alloys [156]. It was reported that Au precipitates form at dislocations as well as on grain boundaries where the cavity density is relatively high. With deposits filling the voids after a post creep anneal the scattering signal becomes a lot weaker due to a change in the net scattering length density.

A different approach, first made by Boeuf et al. in Type 304 stainless steel, to distinguish cavities from precipitates was to conduct measurements on undeformed samples aged after solution annealing treatment [153]. The scattering intensity, attributed to $M_{23}C_6$ carbides only, was later subtracted from each position of the crept sample. This follows the idea that the characteristics of any precipitation morphology aren’t affected by the applied stress. The net result in scattering was attributed to creep induced cavities only. The authors noted that after the solution treatment of AISI type 304 stainless steel, carbon is supersaturated. They further assume that the average dimensions of the carbides are the same with and without stress which is justified by their TEM observations. It has to be noted that this is contrary to observations by Swindemann et al. where stress was found to accelerate the growth of $M_{23}C_6$ in the steel matrix [157]. Boeuf et al. reported a decrease in the volume fraction of cavities with an increasing distance from the failure position. Based on their instrument limitations only cavities in a size range of 1-100 nm could be studied. Changes in the volume fraction for different ageing times and distances from the failure position are illustrated in Figure 2.37. This result is in accordance with density measurements carried out by Matera et al. who studied the deformation-induced cavitation in austenitic stainless steels [158].
Fuller et al. carried out SANS studies to investigate the nucleation and early stages of cavity growth of creep cavities in Type 304 stainless steel [159]. Their investigations in the early 1980s supported theories on creep cavitation with experimental evidence. In their studies, a heat-treated but unstressed reference sample was placed in the furnace together with each creep specimen and the SANS signal was subtracted later to attribute the neutron scattering to just cavities. In their work, they primarily focused on the Guinier analysis to determine the cavity diameter at different stresses and after different times in the furnace. They found an increase in the average cavity diameter from around 30 nm to around 80 nm for specimens tested at 145 MPa and 124 MPa. A doubling of creep time at the same stress was found to double the average cavity size which is in agreement with the Chuang-Rice model that predicts a constant cavity growth rate [160]. The average diameter was found to be higher at a higher stress after 750 h. The change in size was indifferent after the final time of 1500 h. This, however, might just indicate that the cavity growth at this stage is too large to fall into the q-range that is provided by the scattering.
In 316H stainless steel first studies were carried out by Withers and Owen and by Bouchard et al. [161, 162]. A possible link was tried to make between a microstructural damage investigation using SANS and the ductility exhaustion model. Bouchard et al. used the V12 double crystal diffractometer as well as the SANS experiment at the LLB PAXE to characterise cavities in a relatively wide size range of $R = 10$-2000 nm. It was found that the creep damage at rupture was about 0.35 for cavities in a range of 10-100 nm. There was also a large increase in the number density of cavities reported during tertiary creep. Based on their monitored linear increase of number density of cavities with an increasing creep strain a constant cavity nucleation rate for the whole creep life was assumed [162]. Another more in-depth analysis to link creep damage with investigations from SANS was carried out by Bouchard et al. [163]. Here, the authors tried to quantify the creep cavitation damage around a crack in a stainless steel pressure vessel to validate a commonly used crack initiation model based on the ductility exhaustion approach [136]. Using the LOQ instrument at the ISIS neutron spallation source they were able not just to predict

Figure 2.38: SANS study for two 304 stainless steel samples tested at two different stresses. The average cavity diameter is plotted as a function of time [159].
the polydisperse distribution of scattering particles but also to estimate the grain boundary damage due to cavities based on the Porod data. Using this approach, a detailed damage map around the crack could be drawn, compared and validated using quantitative metallography. A peak of 1.2 for the damage could be found close to the crack that is in relatively good agreement with the crack initiation model [163]. The contour map is illustrated in Figure 2.39.

Figure 2.39: Spatial distribution of creep grain boundary damage based on the Porod data from a small angle neutron experiment carried out by Bouchard et al. [163].

In a study carried out by Jazaeri et al. the creep cavitation damage in a stainless steel weldment around a crack in a steam heater header material was observed. Similar to Bouchard et al. the SANS results were validated using quantitative metallography [163]. The SANS data revealed a decrease in volume fraction of cavities for growing distances perpendicular to the crack. Using the D11 instrument at the ILL a size range of cavitation damage from 5 nm to 600 nm could be observed. The main amount of cavities was found to be around a mean diameter of 100-300 nm as illustrated in Figure 2.40 [164].
Further investigations were made to monitor the fractional size distribution of cavities at different instants of creep life at a temperature of 550°C and at a stress of 320 MPa, as given in Figure 2.41 [165]. It was found that the evolution of cavities develops throughout the whole creep life confirming a steady nucleation process rather than instantaneous cavity nucleation after a certain incubation time. However, since the material is ex-service header material it may well be that microvoids were pre-present in the material, making it difficult to determine the onset of nucleation. Jazaeri et al. found two main distributions of cavities [165]. A population of small cavities with a size range from about 20 nm to 60 nm and a population of larger cavities with a mean size of around 200 nm. While the fractional size distribution was found to be relatively consistent for the larger cavities an increase was observed for the small-sized cavities throughout creep life.

**Figure 2.40:** a) Measurement positions and b) volume fraction of cavities in the vicinity of the crack in a steam header [164].
2.8.2 Relevant microscopy studies to determine creep damage

Scanning electron microscopy is heavily used as a complementary technique to SANS to examine creep cavitation at the nanometre scale. However, the preparation process may influence the apparent extent of creep cavities, especially at lower size scales. Silveira and Le May described the sample preparation process to study voids in 1\textit{Cr} − 1/2\textit{Mo}. They reported that an extended polishing and etching process is necessary to reveal creep cavitation. At the same time, they found that the number of steps influences the number and especially the size of voids. They concluded that voids can be created simply by the metallographic preparation [166].

Chen and Argon were the first to study creep cavities in austenitic stainless steels using cryogenic fracturing. Here a brittle fracture is caused by cooling the sample down to a temperature of about −196°C, using liquid Nitrogen. The brittle fracture follows along the grain boundaries and allows to study the 3D-morphology of creep cavitated grain boundary structures without any further metallographic preparation. However, the spatial
resolution is limited and it was argued that the fracture follows the boundaries that are heavily covered with cavities since they provide the weakest structure in the material. The concern is that this might not be representative of the grain boundaries somewhere else in the material. Chen and Argon also established an approach where the creep tested samples are exposed to an additional post-creep strain after a finished test to cause grain boundary sliding and to observe creep cavities that are very small and would normally be closed in the polishing process [73]. Using this process they were able to observe cavities as small as 200 nm. The huge advantage cryogenic fracture provides is that it is not limited to 2D imaging. For SEM imaging this can only be resolved by the usage of serial sectioning of a sample that can be stacked to form a 3D microstructure. Slater et al. performed a multiscale investigation of creep cavitation using multiple techniques to link micro, macro and nanoscale features. In their work, they deployed FIB-SEM to reconstruct a slice through the material and to distinguish creep cavities from precipitates such as carbides and ferrites [98]. A typical cavitated area of grain boundaries in Type 304 stainless steel revealed by cryogenic fracture is illustrated in Figure 2.42 [73]. Since the technique covers a 3D morphology of a sample the resolution is limited compared to a flat surface SEM analysis. Subsequently, the technique reflects more on the cavity growth behaviour rather than the critical nucleation process. Furthermore, it gives information on the shape of relatively large cavities. In the case of elongated cavities, the authors could conclude that grain boundary sliding plays a significant role in terms of void growth.
Further tests on cryogenic fractured samples were later carried out by Jazaeri et al. in Type 316H stainless steel weldments. They could confirm an increase in the cavitated area on intergranular surfaces with a decrease in distance to the crack [164]. Further important metallographic studies in Type 316H stainless steel were undertaken by Bouchard et al. to study the cavitation damage around a crack in a pressure vessel. After the quantitative analysis of around 1200-1500 cavities a distribution of cavity sizes could be established and linked to results from small-angle neutron scattering. The quantitative measurements in Type 316H stainless steel were also conducted by Jazaeri et al. and a change in area fraction of the cavity damage on grain boundaries as a function of distance to the crack could be monitored. Several tests in welded samples creep crack growth specimen as well as in interrupted creep tests were undertaken and the area fraction of the cavity damage established. These studies confirmed a population of small cavities with a diameter below 100 nm and a population of larger cavities with a mean diameter of around 200 nm, as illustrated in Figure 2.43 [164, 165, 167]. However, uncertainty remains whether these features are indeed cavities and not second phase particles.
2.9 Rationale for this work

As laid out in the literature review, it has been established that cold work conditions as a consequence of manufacturing processes, such as bending welding or forging, can be beneficial or detrimental for the lifetime of power plant components operating at high temperature. There are quite a few publications investigating the effect of prior plastic strain on the subsequent creep behaviour in Type 316H stainless steel and the macroscopic creep deformation changes caused by prior plastic strain are well understood. For Type 316H stainless steel most studies have concluded that the creep resistance is enhanced by prior plastic strain, both in tension and compression. However, for Type 316H stainless steel, most creep tests are carried out in a way where the load that is applied is exceeding the yield strength of the material and subsequently plastic strain as a consequence of the loading procedure is induced into the material. Limited work has been published that aims to separate cavitation damage caused by plastic strain from that induced by creep. Tests on prestrained samples were found to be a good approach to separate both types of damage.
in 316H stainless steel. Furthermore, little is known about the type of creep cavitation
damage that occurs as a consequence of prior plastic strain in Type 316H stainless steel. It
is also unclear if cavity nucleation and growth occurs due to prestrain and to what extent
it is already present in the as-received material prior to creep tests.

The published work on SANS highlights its potential to provide quantitative information on
second phase particles and creep cavity damage in metals, such as austenitic steels. SANS
can contribute valuable details on parameters such as the size distribution, volume fraction,
number density or nucleation rate of submicron creep cavities. However, as outlined earlier
it is quite challenging to study polycrystalline materials with a complex microstructure
where second phase particles are present. To distinguish the scattering contribution of
creep cavities from second phase particles, two approaches have been outlined. Another
way to verify SANS findings is to use complementary techniques such as SEM or TEM,
which are both widely used to study creep cavitation in austenitic steels. This research
aims to establish a best practice to quantify creep cavitation damage and to separate
second phase particles from cavities.
3 Experimental Techniques and Data Analysis Procedure

3.1 Overview

In this chapter, the experimental techniques are presented that were applied to investigate the creep properties of the as-received material. In particular, experimental techniques including tensile and creep testing using 3D-DIC strain monitoring will be presented. Lastly, neutron scattering as well as scanning electron microscopy as techniques to characterise creep related cavity damage are described.

3.2 Tensile Testing

Mechanical tests were carried out at the Open University high temperature lab to characterise tensile and creep deformation properties. Uniaxial tensile tests were executed at both, room temperature and high temperature of 550°C according to BS EN ISO 6892-1:2016 and BS EN 6892-2:2011 respectively [168, 169]. The tests were conducted on an Instron 8862 machine with a load capacity of 100 kN. The machine consists of an electromechanical actuator, a load cell, a pull rod and a detachable furnace. The instrument is controlled over a computer by INSTRON’s Bluehill Universal software. Besides controlling the machine, the software can be used to record data and allows tests to be carried out in modes of displacement, strain and load control.

For the measurement at high temperature, an INSTRON 2632-054 extensometer was used to quantify the strain directly from the surface of the specimen. The gauge length of the extensometer, which is the distance between the two quartz rods, is 12.5 mm and can measure a displacement of up to 20% in tension and 10% in compression. To achieve a better grip on the surface of the specimen, the quartz rods have grooves at their ends.
Before each test the extensometer was calibrated with a micrometre. For the high temperature tests an electric resistance split furnace was used. The temperature was controlled by three heating zones inside the furnace. To ensure a uniformly heated specimen two N type thermocouples were attached to the sample surface at the bottom and the top right beneath the shoulder and close to the grip of the sample. A water cooling system had to be used to prevent the pull rods from overheating. Furthermore, an additional fan was placed next to the extensometer to avoid overheating.

### 3.3 3D-DIC Creep Test System

A three dimensional digital image correlation (3D-DIC) strain monitoring system has been developed at the Open University that can be used to measure the displacement on complex sample surfaces and in all three dimensions. Complex surfaces are non-planar surfaces such as on cylindrical test specimen. In addition, with a system of two cameras, rigid body motion of the sample can be detected and removed, allowing more accurate calculation of strain from displacement data, which will be further described in Section 3.3.5.

#### 3.3.1 Creep Rig

The creep rig uses a Severn Thermal Solutions Furnace, built into a standard lever-arm load frame designed by Phoenix. The lever arm ratio of the creep rig was 10:1 and had to be kept horizontal for the creep tests in order to maintain the lever arm ratio. Before each creep test, the load was calibrated using a load cell at room temperature to an accuracy of about 1%. The target stress based on the sample gauge section was converted into a target load. Weights were added until the required target load was achieved on the load cell. After calibration of the load, the load cell was replaced with the actual sample. Afterwards, a couple of pictures were obtained with the mid hourglass gauge section in focus. After this adjustment, the settings of the cameras stayed untouched for the whole creep experiment. As a further step, the 3D-DIC system was calibrated for which the sample was removed
again and replaced with a calibration plate. The calibration will be described in Section 3.3.3. During heating of the creep rig, a preload of 20 N was used to maintain a horizontal lever arm of the creep rig.

3.3.2 Temperature Measurement

A Severn Thermal Solution Furnace with a maximum operating temperature of $1100^\circ C$ was used for all creep tests carried out for the PhD work [170]. It has an open top and bottom to accommodate thermocouples and pull rods. The furnace has three different heating elements implemented in the bottom, middle and top zone that can be controlled individually using a Eurotherm control system. Each heating element has its own thermocouple embedded in the furnace to control and adjust the target temperature. To reduce heat losses from the furnace, ceramic fibre insulation was used to seal the bottom and the top of the furnace. The furnace had a custom built rectangular window aperture (99 mm x 40 mm x 1.25 mm) giving two cameras a field of view on the specimen. The long side of the window was perpendicular to the length of the specimen. The specimen is fixated over its threaded ends with the two pull rods in the middle of the furnace, which is illustrated in Figure 3.1. The heating process starts prior to loading. After the desired temperature was achieved, an additional six hours is given to ensure that the temperature stabilised and was homogeneous within the specimen. Two 1 mm diameter holes were drilled 2 mm deep at the top and the bottom of each specimen right below the shoulder and the grip section. Type N thermocouples were used to monitor the temperature throughout the whole creep test. The thermocouples were connected to a Picolog data logger, so that temperatures could be recorded at every 10 seconds throughout the test. With 3D-DIC as an optical solution to measure strain it was imperative to monitor the surface gauge length visually. Therefore, the third thermocouple recommended by ASTM-139, and usually placed in the middle of the sample, was discarded. Although one thermocouple was removed it was ensured that the temperature fluctuation was kept stable within $\pm 1^\circ C$. Prior to each measurement, every thermocouple was calibrated against a reference thermocouple at the
specific working temperature of $550^\circ C$ prior to each test.

### 3.3.3 Optical system

Figure 3.1 depicts a sketch of the 3D-DIC high temperature creep system at the Open University. The main components are a twin Nikon D810 DSLR camera system, the furnace, the loading frame and the fibre optic cable that transfers the flash light from the camera onto the surface of the sample.

![Figure 3.1: Schematic setup of the 3D-DIC strain measurement system at the high temperature creep lab at the OU.](image)

The resolution of each camera was 7360 by 4912 pixels. The two cameras are positioned at an angle of $20^\circ$ to each other. An angle between $20^\circ$ and $30^\circ$ has been recommended by other groups [171]. However, no adjustments have been made to further optimise the setting. Both cameras are equipped with 200 mm macro lenses (Nikkor 200 mm micro f4 IF-ED) that provide sufficient zoom capabilities. The Nikon camera has an infrared filter which was used as a filter to block blackbody radiation of the sample. The cameras
use the maximum aperture of f32 to maximise the depth of field. The main camera that is triggering the flash has a shutter time of 0.5 s while the slave camera has a much longer shutter time of 2 s. It was found that the flash sync delays the shutter time of the reference camera to which both cameras are synced to take pictures at the same time. Due to the slight deviation in the flash sync this approach ensures that each camera records an illuminated picture. However, it also increases the inaccuracy between the two pictures since one camera records a picture with a longer illumination time. With a heat flux that is fluctuating constantly, the images from both cameras tend to have a slight variation which decreases the resolution. The settings of the two cameras were adjusted to ensure that the level of illumination was roughly similar.

In order to carry out 3D-DIC, a calibration of the optical system was necessary. A calibration plate, a plate with black dot sizes of 1 mm and known spacings between the dots, was placed in front of a white background in the plane of the actual specimen position. The calibration of the 3D-DIC system was performed with 10-15 pictures taken of the calibration plate translated to several distances away from the camera sensor. The calibration of the DIC system eliminates parameters such as the camera lens distortion and determines the magnification of the image of the specimen or the scale factor. DIC is designed for use with special cameras equipped with a CMOS sensor and a monochromatic light source. However, it has been shown by Forsey and Gungor that colour DSLR cameras can be used with a bicubic spline based demosaicing method that converts camera raw files into monochrome images [172]. By this means, the high resolution of DSLR’s can be utilised to achieve a high level of accuracy. The laboratory was equipped with an air condition system that ensures a constant room temperature of $20 \pm 1^\circ C$. The room light was switched off and blinds on the windows used to keep out sun light thereby providing good stable contrast in the pictures.
3.3.4 Sample Preparation

A speckle pattern was created on the surface of the samples, using a silica ceramic based VHT FlameProof paint. This paint is particularly useful for long term tests at elevated temperature because it prevents oxidation. The speckle pattern was created by three layers of paint. First, a white primer was sprayed on the gauge area to enhance the durability of the paint. This was followed by a second coat of white paint which creates a matte surface that inhibits direct reflections. Subsequently, black paint was used to create the random speckle pattern. Following the instructions of the manufacturer, the paint was tempered at three different temperatures at $120^\circ C$, $225^\circ C$ and $315^\circ C$ for 30 mins respectively, each time followed by a cooling time of 30 mins.

Historically, VHT FlameProof paint was used at the OU for test specimen with EDM flat surfaces and showed good results. This was observed in former tests carried out by Sakanashi et al. where it was found that paint embrittlement is not an issue for flat EDM machined samples even for high plastic strains above 20% [173]. However, for test specimens with the cylindrical hourglass design (as later described in Section 4.3.2), problems with the adherence of the paint were observed. During the loading process prior to the creep experiment, due to substantial plastic strain some of the paint flaked off of the test specimens. Due to its hourglass geometry the sample had to be machined resulting in a very smooth surface of the test specimens. It is believed that a rougher surface increases the possibility for the paint to stick on the sample. The cylindrical hourglass design, used for all creep tests throughout this work, however causes flaking off of the paint. Figure 3.2 shows an example of a specimen prior to and after loading. In the right picture some of the paint is visibly detached from the sample surface.
Figure 3.2: A picture of the test specimen a) right before and b) right after loading. A substantial amount of the randomised black speckle pattern flaked off of the sample.

To address this problem, tests on hourglass dummy samples were conducted. There are several factors contributing to the adhesiveness of the paint on the sample:

- cylindrical hourglass geometry
- roughness of the surface of the test specimen
- thermal treatment of the paint
- thickness of the applied coating

Since the cylindrical hourglass geometry can not be changed, tests were conducted where the roughness of the sample surface was modified. Different etchants, such as Nitric acid as well as Hydroflourid acid, were used to increase the surface roughness to improve the
grip of the paint. One other approach was tested by applying the paint to the specimen but without carrying out the thermal tempering process, for curing the paint, prior to the test. It was believed that this thermal heat treatment prior to creep tests creates a paint embrittlement and is not really necessary since creep tests are carried out at relatively low temperatures. The VHT FlameProof paint is designed to withstand temperatures of up to $1093^\circ C$. However, these two approaches were found to be not successful. The thickness of the applied coating on the other hand, was found to have the most profound effect. It could be established that the best results could be achieved by creating coatings that are as thin as possible. The paint is applied with consumer spray cans and substantial practise is needed to create such very thin coatings on the cylindrical samples. One further point that adds to the complexity is that the samples need to be rotated for a complete coating. Ultimately, an automated system should be used for applying the paint, while rotating the cylindrical test specimen to ensure a thin uniform coating. Even though some of the paint was flaking off, it was possible for the DIC-software DaVis, for all creep tests carried out for this PhD work, to find a converging solution and to perform the image correlation. The amount of speckle that were still attached to the sample was found to be sufficient. The degradation of the paint happens only after loading and no further degradation was observed during creep. This indicated that oxidation is not an issue for creep tests carried out at a temperature of $550^\circ C$ on the as-received material.

3.3.5 DIC Analysis procedure

Images were taken after applying loads in increments to separate the deformation of the specimen due to loading from the creep deformation. After loading, the frequency at which images were taken was set to 5 minutes. This interval was kept for 100 hours to capture the whole primary creep curve. Then, the frequency was reduced to an image every 30 minutes. The creep deformation was monitored closely and when a transition between secondary and tertiary creep was detected, the frequency was increased again to 5 min to capture any changes in strain more closely.
All acquired RGB images were converted to grey scale using an in-house MATLAB script that uses a bicubic spline algorithm, described in more detail by Forsey and Gungor [172, 174]. The converted pictures were imported into the DIC analysis software DaVis [175]. To perform a 3D-DIC analysis the camera system has to be calibrated in a way where the relationship between the sensor position and world space is determined. In its simplest form it is a scaling between pixel and mm distance that is unaffected by rigid body movement. Figure 3.3 illustrates the stereovision system principle used for the 3D-DIC technique. It is a depth perception system that functions like human eyes. Each camera transforms a 3D object point into a point of a 2D image. To recalculate a 3D object point, the intersection of the two projected rays has to be determined. Using this method it is possible to recover real world three dimensional positions of points of an object [176]. Using two cameras the sample geometries as well as displacements in all three dimensions can be calculated. After calibration, the 3D-DIC system can calculate the surface shape, displacement and strain on the specimen. Rigid body movements in z-direction can therefore be accounted for and won’t give artificial strains which would be the case for 2D-DIC.

![Figure 3.3: Principle of the stereovision system principle used for 3D-DIC [176].](image)

The subset size which gives the width and height of the subset square in the reference image, can have a significant impact on the measurement accuracy of the DIC analysis.
For each subset size, an average grey value is calculated. The DIC correlation method calculates the displacement of a certain subset, using the Lucas Kanade method [177]. Over a pyramidal approach, pixels are binned for large displacements for a search over a large area [178].

Some uncertainty in the strain analysis rises from fluctuations from heated air currents within the furnace resulting in a constant changing air refractive index. To reduce the noise from heat fluctuations a time averaging approach was found to be effective. For all creep tests carried out and as recommended by [179], an average of three pictures is taken as the reference image for the initial condition that defines the position of the seeding points. After this smoothing process, the displacement calculations were carried out by the DAVIS software. This of course works only if the deformation process is slow, which is the case for the creep process. The step size is the distance between subset centres. For a robust correlation analysis, the features in each subset size have to be adequately distinctive to distinguish one subset from other subsets. Therefore, if the speckle size is too large, the subset size usually has to be increased for a better correlation analysis [180].

In order to increase the spatial resolution, a small subset size is beneficial. After calibration a field of view of usually around 19-25 mm by 4-6 mm and a scaling factor of approximately 4.25 \( \mu m/pixel \) was given by the optical system. With an hourglass gauge section of 28 mm only a part of it could be analysed with the 3D-DIC technique. The field of view that is given by the DIC system is always slightly different for each creep test. With a subset size of 31 pixels and a steps size of 8 pixels a spatial resolution of 0.034 mm could be calculated. The field of view is illustrated in the left picture of Figure 3.4 as the red box which illustrates that DIC is able to measure about one half of the hourglass gauge section. Due to its cylindrical shape it is not possible to get the whole width of the specimen in focus.
Figure 3.4: Shows typical field of view, here about 4.5 mm by 19 mm from one of the creep test specimens. The picture on the left side depicts the random speckle pattern on the sample surface. The right picture illustrates the displacement field after a certain amount of creep time. The mid-length of the specimen is indicated by the blue dotted line.

The displacement results that were calculated by the DIC software were exported as txt files. The method to calculate the strain from the displacement values will be described briefly here. A more detailed version can be found in [179]. An in-house built MATLAB script, developed by Dr Alex Forsey at the OU, was used to determine the strain on the surface of the sample by differentiating the displacement vectors. The calculated displacement field is really sensitive to noise in the signal caused by thermal fluctuations, chromatic aberration from the furnace window etc. and requires the use of an averaging approach over a larger region to produce good statistics. The deformation vector field was sampled to 50 equal sized rectangular regions with a sample length of 1 mm across the gauge length, which is illustrated in Figure 3.5. The strain was averaged across the width of the specimen for each region along the length of the gauge section. To increase the spatial
resolution an overlap of 75% within each region was utilised. 10% of the sample width at each edge of the sample was excluded to prevent noise from less constrained vectors in the displacement field. The displacement vector field is firstly separated into vectors in the x- and y-directions and fitted using a first order polynomial by a least squares approach. Any outliers between the measured displacement and fitted result above a certain threshold were removed. The gradients of the polynomials in the x- and y-directions for each time step were used to calculate the Lagrange strain on the surface.

![Diagram showing the spatial strain averaging procedure to extract creep data along the gauge section](image)

**Figure 3.5:** Diagram showing the spatial strain averaging procedure to extract creep data along the gauge section [179].

### 3.3.6 Reducing noise of the creep strain data

Since the creep strain rate for the as-received material is very low for all conducted creep tests, the signal to noise ratio is relatively poor and a robust fitting procedure had to be established. To reduce the noise from the 3D-DIC data, a series of filtering algorithms had to be used to determine creep properties such as the creep strain rate. In the 3D-DIC raw data, the creep strain rate can fluctuate between several orders of magnitudes continuously,
as depicted in Figure 3.7. A Savitzky-Golay filter with the length of 101 data points was found to be suitable and applied for smoothing the data without distorting the signal tendency [181]. The filter achieves this through convolution of the raw data by fitting sub sets of adjacent data points with a low-degree polynomial (3rd order), using the method of linear least squares. The smoothing for one example of a 3D-DIC raw data set with four creep curves taken from different points along the hourglass gauge section is illustrated in Figure 3.6. The plot highlights that the filter for smoothing the raw data works relatively well over the whole creep time. The length of the filtered window over which the data is convoluted was set to values in the range between 101 to 151 data points. For creep tests with a good signal to noise ratio the filter window length could be reduced. The divergence between raw creep deformation data and the fitted functions was kept at a minimum.

![Figure 3.6](image-url)

**Figure 3.6**: Raw Data from 3D-DIC test of five different positions along the hourglass gauge section and the related smoothed data using a Savitzki-Golay filter for a) the whole creep time until rupture and b) 500 h.

In order to obtain the creep strain rate accurately, another filtering method had to be deployed. In addition to the Savitzky-Golay filter, a univariate spline method was also used for smoothing the data [182]. It relies on constructing piecewise polynomial functions
between pairs of known data points. For this work, a cubic spline method was chosen that fits a third degree polynomial between knots. The method gives a fitting function that passes through known data points and interpolates in between them. The algorithm works really well and was found to be a lot less susceptible to oscillation which is a general problem for global polynomial fitting approaches. A comparison between the creep strain rate obtained from the 3D-DIC raw data and the creep strain rate calculated from the smoothed data is depicted in Figure 3.7. It can be found that the improvement for calculating the creep strain rate is quite significant. In Figure 3.7 the creep strain rate from one exemplary stress position of the mid-hourglass gauge section was depicted together with a) the creep strain rate processed using the Savitzky Golay filter and the univariate spline method. Figure 3.7 b) shows the creep strain data processed with just the Savitzky-Golay filter and using a combination of Savitzky Golay and Univariate Spline method. It has to be mentioned, that filtering of the creep data has to be carried out with caution. There is always the possibility of over-smoothing the data which would lead to false conclusions on the creep strain rate. This is particularly important for DIC data, where the level of noise is higher compared to other displacement measuring techniques (using extensometers or strain gauges).
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Figure 3.7: The creep strain rate calculated (a) from the raw 3D-DIC data and the smoothed data processed using a combination of Savitzky-Golay and the Univariate Spline filtering method and (b) the same creep strain rate data but filtered using the Savitzky-Golay filter and a combination of Savitzky-Golay and the Univariate Spline filtering method.

3.4 Microscopy Analysis

3.4.1 Optical microscope

To study the microstructure of the as-received material, such as grain sizes, slip bands etc., a Leica DMI 5000M light microscope was used. The microscope was applied in the bright field mode where features such as grains appear bright and irregularities such as precipitates, slip bands, inclusions etc. appear dark. Micrographs were usually taken at the smallest magnification of 50 to maximise the applicable field of view.

3.4.2 Scanning electron microscope

Scanning electron microscopy is a commonly used technique to conduct microstructural investigations at very high resolutions. With a frequency of the electron beam that is much
higher than for visible light, magnifications of almost a magnitude larger can be achieved. Electrons are generated in an electron gun due to thermal emission of a tungsten filament triggered by a field emission cathode. Throughout a series of electromagnetic lenses the electron beam can be focused and narrowed down onto a certain region of interest on the sample area. The SEM chamber is kept under vacuum to prevent any particle collisions with the electron beam. When the incoming electron beam hits the sample surface it can cause scattering which emits electrons. Detectors placed at certain positions around the sample can monitor the intensity of these emitted electrons for a certain position and create an SEM image. In this work, a Zeiss Supra 55VP high-resolution FEGSEM was used. In addition to a secondary detector, a detector for backscattering electrons was deployed. While the first gives a better contrast for topographical images, the backscattering detector has its strengths if information about the composition is required. Compared to the optical microscope the depth of field from the SEM is about two magnitudes higher and causes images to appear in a three dimensional way. All SEM images obtained for this PhD work were taken with an energy of 10 keV.

3.4.3 Sample Preparation

Sample preparation was performed using conventional mounting, grinding and polishing techniques. The grinding procedure, with successively finer grades of SiC papers of up to 4000µm, was conducted on a Buehler semi-automatic grinding machine. The grinding procedure was followed by polishing using diamond suspensions in a range from 9µm to 2µm. A vibro polisher (ATA Saphir Vibro) was used for the final polishing grades of 1µm and 0.25 µm. Each polishing step was conducted for about 60 min at a frequency of 108 Hz and an Amplitude of 100 using a diamond suspension. Between the polishing steps the sample was carefully cleaned using a ultra sonic cleaner to avoid decontamination from the different grades of diamond suspension. The procedure was refined in a range of trials to remove any scratches but to keep the carbide morphology untouched.
3.4.4 Cryogenic Fracture

Cryogenic fracture tests were conducted on the used material. For this technique, a brittle fracture with distinct facets is generated. A (3 mm x 1 mm x 30 mm) large matchstick was extracted along the gauge length of the specimen. To achieve that, notches were introduced at an interval of 5 mm along the gauge section, perpendicular to the creep ruptured surface. The matchsticks were fixed with a specially constructed jig and then cooled down with liquid nitrogen to about $-196^\circ C$. After about 5 min the specimens were taken out and instantly hit with a hammer to create brittle fracture.

3.5 Small angle neutron scattering

One method to quantify microstructural features within bulk samples is the use of small angle neutron scattering (SANS). Using neutrons has the main advantage of high penetration in metallic materials such as stainless steel. The interaction is not related to the electron cloud as it is in SAXS (small angle X-ray scattering) but to scattering with the nucleus. Thus, the scattering cross section varies randomly with the atomic number and is not proportional as it is for X-rays. SANS is a technique to investigate features inside inhomogeneous materials across a relatively wide size range, from 1 nm up to about $1\mu m$. Defects that have a different average scattering property from that of the surrounding matrix can be detected. It can be applied to study volume average information. In a conventional small angle neutron scattering experiment the beam is collimated by two pinholes. The beam that is hitting the sample is scattered at a small angle $\theta$ out of the direct beam and is detected on a 2D-detector. An illustration of a typical small angle scattering set up is illustrated in Figure 3.8. Due to a beam stopper right behind the sample the transmitted beam can be masked out and does not superimpose the actual scattered signal. The difference of the incoming wave vector $k_0$ and the scattered wave vector is defined as

$$q = |k - k_0| = \frac{4\pi}{\lambda} \theta, \quad (3.1)$$
where \( \lambda \) is the wavelength of the neutrons and \( \theta \) is the angle between incoming and scattered particle. The approximation \( \sin \theta \approx \theta \) can be used for very small angles. This leads to

\[
\theta = \frac{\lambda}{D_s} = \frac{\lambda q}{4\pi} = \frac{R_D}{L},
\]

where \( L \) is represented by the distance of the detector from the sample and \( R_D \) as the distance from the transmitted beam to the point at which the scattered beam is recorded.

\[ \theta = \frac{\lambda}{D_s} = \frac{\lambda q}{4\pi} = \frac{R_D}{L} \]

**Figure 3.8:** Schematic for SANS experiment.

Furthermore, one can relate the length scale \( D_s \) of the scattering object to the scattering angle and the scattering vector using Bragg’s equation. That leads to detectable objects in a range from a few \( nm \) up to about \( 1 \, \mu m \). The relationship between \( q \) and the size of an object can be estimated by the approximate relationship \( D \approx \frac{2\pi}{q} \).

In most of the cases it is possible to describe the distribution of scattering objects inside a material with the help of a form factor \( F(q) \), a structure factor \( S(q) \) and a contrast factor \((\rho_1 - \rho_0)^2 \). \( F(q) \) represents the interference of scattered neutrons from different parts of the same object. It also describes the size and shape of scattering objects. The structure factor \( S(q) \) describes the interference of neutrons scattered by different objects. With a system that has no inter-particle correlations \( S(q) \) becomes 1. The macroscopic cross section \( \frac{d\Sigma}{d\Omega} \) of the coherent scattering signal can be written as

\[
\frac{d\Sigma}{d\Omega}(q) = \frac{N}{V} (\rho_1 - \rho_0)^2 V_p^2 F(q) S(q),
\]  

(3.2)
where $N$ is the number of atoms in a sample volume $V$ which is the number density.

Figure 3.9: Overview over all the different observations within the whole q-range [183].

Figure 3.9 shows features that can be studied from a typical SANS curve. The features vary with scattering angles $\theta$. At a very low q-regime it is possible to determine the volume or size of the particle using the Guinier approximation [184]. In the intermediate q-regime one can determine the shape and dimension of the object. For example, needle shaped particles exhibit a cross section that varies with $q^{-1}$ whereas disk shaped particles a $q^{-2}$ variation. Finally, at the very high q-region one can determine the specific surface to volume ratio of the scatterers by applying a Porod analysis [185].

3.5.1 SANS instruments SANS2D and D11

Two different SANS instruments have been used to analyse microstructural features in stainless steel samples tested in this project. SANS2D at the ISIS facility and D11 at the Institute Laue Langevin (ILL). While for SANS2D neutrons are generated from a pulsed spallation source, the neutrons at D11 are produced from a reactor source. In a reactor source, like at the ILL, neutrons are generated from nuclear fission of U-235. The released hot neutrons are cooled down with moderators and can be made monochromatic
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via diffraction using a crystal monochromator. In a spallation source, such as at ISIS, neutrons are produced in a process where high energy protons hit a tungsten target and a flux of neutrons is released. Compared to the reactor source where the flux is continuous the spallation source produces a very high pulse of neutrons at a certain frequency. The polychromatic beam at SANS2D at ISIS is analysed through a time of flight technique where the velocity of different neutrons is measured and used to determine the wavelength and the polychromatic distribution.

3.5.2 SANS2D instrument

SANS2D is a time of flight instrument where neutrons are produced at a frequency of 10Hz. A white beam of neutrons is generated with an incident wavelength of 2 to 14 Å. This relates to a scattering wavevector \( q \) to range from 0.00156 Å\(^{-1}\) to 0.4 Å\(^{-1}\). Due to the time of flight technique this relatively broad \( q \)-range can be achieved at a sample detector distance that is quite short compared with other SANS instruments. A sketch of the SANS2D setup can be found in Figure 3.10.

Two detectors are used for each measurement with the second detector at a distance of 12 m behind the sample and a sideways offset of 150 mm vertically and 200 mm horizontally to extend the \( q \)-range at a given detector distance. Using this set up scattering particles within the size ranges of 5-400 nm can be measured. For each sample a transmission scan
needs to be conducted, to adjust for the absorption of the neutrons inside the material. A series of measurements using a blend of hydrogenous and predeuterated polystyrene has to be carried out by the instrument scientist to calibrate the instrument. Based on the calibration, the absolute macroscopic cross section, in $cm^{-1}$, can be determined from each measurement point as a function of the scattering vector. Based on these specifics, a data reduction was carried out using Mantid, a software used at various neutron instruments around the world [187]. The data reduction procedure for an instrument with a white beam of neutrons has to be more advanced since it has to include wavelength dependencies of the incoming neutrons, the sample transmission and the direct beam function which is the efficiency of the main detector compared to the incident beam. Further details about SANS2D can be found in [186,188]. The black and green line in Figure 3.11 illustrate the scattered neutron captured by the front and back detector respectively. The red line shows the merged data from both detectors. It can be seen that there are large fluctuations in the data in the overlap $q$-region between both detectors in a $q$-range from $4 \times 10^{-2} \, \text{Å}^{-1}$ until about $1 \times 10^{-1} \, \text{Å}^{-1}$.
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3.5.3 D11 at the ILL

In Figure 3.12 a schematic of the D11 instrument at the ILL is illustrated. The polychromatic neutron beam from the cold source is monochromated by a velocity selector before reaching the sample. The D11 instrument allows the user to change the wavelength in a spectrum from roughly 4.5 Å to about 40 Å. The monochromatic beam passes a neutron guide to collimate the beam and hits the sample with a neutron flux of roughly $1 \times 10^7$ n/cm$^2$. From the schematic drawing it can be seen that after the neutron beam hits the sample, transmitted and scattered neutrons are monitored by a 96 by 96 cm detector inside an evacuated tube. The He$^3$ detector is moveable between distances of 1.1 m and 39 m which relates to a q-range from $3 \times 10^{-4}$ Å$^{-1} \leq q \leq 1$ Å$^{-1}$. This wide range of distances makes the instrument suitable for mapping heterogeneities within a wide size range between 1 and 1000 nm.

Figure 3.11: Neutron scattering intensity versus q measured at the SANS2D instrument.
The scattering was measured at three distances of 1.5 m, 8 m and 28 m at a wavelength of 6 Å. 6Å was chosen because it provided the highest flux of neutrons per measurement. The three distinct detector positions enabled measurements within a q-range of \(1.76 \times 10^{-3} \text{Å}^{-1} \leq q \leq 0.4210 \text{Å}^{-1}\). Scattering objects within a diameter range of 7 nm to about 360 nm could be detected. At a detector distance of 1.5 m from the sample, the incoherent scattering which is constant with q, was measured. The signal to noise ratio here has to be relatively high for the maximum entropy analysis to perform well. Similar to measurements at the SANS2D instrument, a transmission scan had to be obtained from each sample position. In addition, scattering from a 1 mm thick water sample with a calibrated scattering was measured at each detector position to normalise the data and to obtain absolute cross sections at each condition. With the In-house software LAMP the data from each detector position could be reduced and merged together, which can be found in Figure 3.13. This process needs careful analysis and experience by the instrument scientist who knows the changes in efficiency for each detector position. With a much finer q-resolution on the D11 instrument that provides 5 times as many data points compared to the SANS2D instrument the data is a lot more robust and a lot less noisy.
3.5.4 SANS sample preparation

All SANS measurements were conducted on 1 mm thick specimen extracted from a longitudinal cut from mid-thickness of the crept test specimens. The samples were positioned in a custom built sample holder which was placed in the beamline and each sample position was carefully aligned with a laser system that projects the centre of the neutron beam. The coordinates for each measurement position were recorded and later used in a script file to run the measurements remotely. The dashed box in Figure 3.15 shows a schematic of the aperture that was used to define the gauge section for each measurement. Figure 3.14 shows an illustration of the sample holder used at the experiments at SANS2D and at the ILL. The loading capacity for the sample holder was large enough for all the tested specimens. This entails that the samples did not had to be switched and the alignment had to be measured only once during the experiments.
Figure 3.14: Sample holder used for SANS experiments at ILL and SANS2D. The first five samples on the left are the creep ruptured samples, used in the current research project.

A picture of one of the cut specimens can be found in Figure 3.15. In general, as can be seen from the markings in Figure 3.15, the first five points close to the fracture position were 1.5 mm apart from each other. A letter box aperture with the dimensions of 2 by 5 mm was chosen to increase the spatial resolution. For further points the distance between each point was increased to 2 mm. Lastly, reference points in the far field region were selected in the grip and the shoulder section of the specimen.
3.5.5 Contrast Factor

Changes in scattered intensity in the material due to precipitation or creep cavity damage are proportional to the square of the difference in neutron scattering length densities (SLD). The SLD depends on the local average scattering length of the atoms as well as on the number density. In an alloy with multiple components the scattering length density over any suitable volume becomes the sum of all products divided by the sample volume $V$ and can be calculated using:

$$\rho = \frac{\sum_i b_i n_i}{V} \quad (3.3)$$
where \( b_i \) is the scattering length and \( n_i \) the number density of the nuclear component \( i \).

The volume \( V \) can be calculated using:

\[
V = \frac{M}{\rho_{\text{bulk}} N_A}
\]  

(3.4)

where \( M \) is the molar mass, \( N_A \) is the Avogadro constant and \( \rho_{\text{bulk}} \) is the density of the sample. The contrast factor \( \Delta \rho^2 \) between neutrons and the change in potential that the neutron interact with, depends on differences in scattering length density between steel matrix and cavities. Since the contribution of precipitates to the scattering signal is eliminated with the subtraction of the signal from the reference points. The contrast factor can be obtained by applying:

\[
CF = \Delta \rho^2 = (\rho_i - \rho_j)^2
\]  

(3.5)

Table 3.1: Chemical composition of 316H-SS cast CD25520 and neutron scattering length density as well as contrast factor for each element [190].

<table>
<thead>
<tr>
<th>Element</th>
<th>Volume %</th>
<th>Volume in cm(^3)</th>
<th>( b_i ) in (10^{-12} \text{cm})</th>
<th>Scattering length density (sld) in cm(^{-2})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co</td>
<td>0.41</td>
<td>1.78E-23</td>
<td>0.77</td>
<td>2.69E8</td>
</tr>
<tr>
<td>Mn</td>
<td>1.37</td>
<td>1.23E-23</td>
<td>-0.37</td>
<td>-4.16E8</td>
</tr>
<tr>
<td>Mo</td>
<td>2.11</td>
<td>1.55E-23</td>
<td>0.67</td>
<td>9.15E8</td>
</tr>
<tr>
<td>N</td>
<td>0.054</td>
<td>2.88E-23</td>
<td>0.94</td>
<td>1.76E7</td>
</tr>
<tr>
<td>P</td>
<td>0.036</td>
<td>2.82E-23</td>
<td>0.51</td>
<td>6.5E6</td>
</tr>
<tr>
<td>S</td>
<td>0.0009</td>
<td>2.57E-23</td>
<td>0.28</td>
<td>9.79E5</td>
</tr>
<tr>
<td>C</td>
<td>0.069</td>
<td>8.86E-24</td>
<td>0.67</td>
<td>5.18E7</td>
</tr>
<tr>
<td>Si</td>
<td>0.41</td>
<td>2.00E-23</td>
<td>0.42</td>
<td>8.56E7</td>
</tr>
<tr>
<td>Cr</td>
<td>16.95</td>
<td>1.20E-23</td>
<td>0.36</td>
<td>5.14E9</td>
</tr>
<tr>
<td>Ni</td>
<td>10.089</td>
<td>1.09E-23</td>
<td>1.03</td>
<td>9.49E9</td>
</tr>
<tr>
<td>Fe</td>
<td>69</td>
<td>1.18E-23</td>
<td>0.94</td>
<td>5.49E10</td>
</tr>
<tr>
<td>316H-SS matrix</td>
<td>100</td>
<td>1.86E-22</td>
<td>-</td>
<td>7.05E10</td>
</tr>
</tbody>
</table>
From the listed parameter in Table 3.1 one can find the contrast factor that is necessary to calculate the cavitation damage in the as-received material. $b_i$ is the bound coherent atomic scattering length over any volume $V$. The units of the contrast factor are usually given in reciprocal squared units such as cm or Angstrom ($10^{10} \text{cm}^{-2} = 10^{-6} \text{A}^{-2}$). To obtain the volume fraction of cavities from the measured signal on each point one has to subtract the signal from the reference points on each sample and divide the result by $49.71 \times 10^{28} \text{m}^{-4}$ which refers to the squared difference of the neutron scattering length density between steel matrix and cavities, see Equation 3.7.

### 3.5.6 Analysis of SANS Data

For each SANS experiment, the biggest challenge is to perform a sufficient data correction and analysis. Most fitting methods use a standard least square optimisation between the measured data and a model pattern, that is defined by some model parameters. It is particularly challenging to apply these standard model distribution forms which usually include lognormal, Gaussian or Schultz distribution, to metals and alloys were such distributions can not be assumed a priori. Any assessments of the microstructural features need to be done with microscopy imaging using e.g. SEM to help with the prior assumptions. To obtain accurate results from the SANS data, such as the size distribution and volume fraction of scatterers, two independent software methods where applied and compared for this work. A maximum entropy method (MAXE) as well as a Monte Carlo software (McSAS) were deployed to study creep cavities in the as-received material. Both software methods have been applied repeatedly for SANS experiments in metals and alloys and were found to be particularly useful for polydisperse distributions of scatterers [164, 165, 191, 192]. Both will be explained in more detail in the following sections.
3.5.7 Maximum Entropy Method (MAXE)

After the data reduction of the scattering results, using MANTID or LAMP the data can be deconvoluted using a maximum entropy algorithm (MAXE). An essential part of the overall data fitting was done using the maximum entropy method. MAXE can be applied if one assumes spherical shaped scatterers and that the density of precipitates and cavities is small enough that inter-particle interferences can be neglected [193]. MAXE calculates the volume weighted size distribution $V(D)$ over a range of diameter. This is a smooth function defined such that $V(D)$ multiplied with $\delta(D)$ gives the volume fraction of spherical scatterers with a diameter $D + \delta D$. The summation over a range of $D$ gives the total volume fraction. MAXE usually gives a good approach to determine the absolute volume fraction of scatterers from fitting the data. The MAXE routine was developed at Harwell in FORTRAN and has been re-written in C++ at the Open University. The benefit of the maximum entropy method is that it is independent from empirical distribution models but derives the particle size distribution from a maximum entropy configuration. The theory is described in more detail in several other publications [193–195].

3.5.8 MAXE Settings

The settings used in MAXE need to be chosen carefully. Since the main analysis for this work was done using this software a quick explanation of the settings will be presented. Every parameter needs to be adjusted to give the best visual fit to the data and has to be kept fixed for the whole data analysis for the results to be comparable.

An important parameter is the error factor as it defines the $\chi^2$ criterion. This factor establishes the deviation between measured data and fitted results. A higher error factor allows for the convergence to be reached earlier. Secondly, the number of bins can be modified in the software. This is an adjustment on how much the distribution of scattering particles is partitioned. An increase results in a finer sampling of the resulting histogram at the expense of a rise in uncertainty about the volume fraction of each bin. The number
of iterations can also be altered. This simply gives a limit number after which the analysis is aborted if the convergence criterion isn’t reached. Changing the maximum particle diameter gives a boundary in which the distribution is shown only. This has to match with the q-range of each instrument and each setting. The contrast factor is the difference of the squared neutron scattering length density from the sample matrix and the scattering phase. In Section 3.5.5 it is explained how it is calculated for the as-received material that is used for this project. The flat background can also be adjusted to account for the incoherent scattering or for changes in the density of the sample. Lastly, the user can change the lower and upper q-range limits within the raw data. In the following Table 3.2 the parameters are summarised.
Table 3.2: Advanced settings and defaults that can be adjusted in MAXE

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Error Scaling Factor (EF)</td>
<td>It determines the convergence criterion or the least square value $\chi^2$ at which the fit is terminated and considered a success. Larger error factors result in larger deviation between measured and fitted data data</td>
</tr>
<tr>
<td>Number of bins</td>
<td>The number of partitions in the distribution. By increasing the number a finer sampling of the distribution is enabled but the uncertainty will be increased.</td>
</tr>
<tr>
<td>Max of Iterations</td>
<td>The amount of iterations that is implemented. If convergence isn’t reached within this number the fitting process is aborted. Increasing this number increases the optimisation time.</td>
</tr>
<tr>
<td>Contrast Factor</td>
<td>Determines the contrast factor between matrix and scattering phase. It is calculated from the difference of the squared neutron scattering length density ($\rho_1^2 - \rho_0^2$)</td>
</tr>
<tr>
<td>Max particle diameter</td>
<td>The distribution is constrained by this parameter and it is used as a limit where the fitted distribution cuts off</td>
</tr>
<tr>
<td>Units conversion factor</td>
<td>Introduces a factor for the volume fraction of scatterers. The parameter was unchanged and left at 1 for the whole analysis of the project.</td>
</tr>
<tr>
<td>Flat Background</td>
<td>A flat background is introduced that accounts for the contribution from incoherent scattering</td>
</tr>
<tr>
<td>First and last data point</td>
<td>Leaves the user the possibility to partition the data by changing the q-range from the raw data</td>
</tr>
</tbody>
</table>

In Figure 3.16 the effect of changing the error factor as a fitting parameter is depicted. Five different error factors have been chosen to demonstrate the effect on the fitting accuracy and the subsequent changes of the polydisperse distribution of scatterers. An error factor of 1 fits the data best but gives a really abrasive distribution of scatterers which is illustrated in Figure 3.17. An error factor of 1 also creates difficulties in reaching convergence of the
solution which is one of the reasons it has not been used for the SANS analysis in this work. In general, the fitting procedure works well within the the low and mid-q section for all five error factors. In the high q-region at around 0.06-0.1 Å however, a larger deviation between scattering signal and fitted results for larger error factors can be observed.

![Intensity versus q plot for different error factors](image)

**Figure 3.16:** Changes in the deviation between experimental and fitted result for five different error factors. a) shows the Intensity versus q plot for the whole q-section \((1.54 \times 10^{-3} \text{ Å}^{-1} \leq q \leq 4.210 \times 10^{-1} \text{ Å}^{-1})\). b) shows a section within the q-range from \(1.0 \times 10^{-2} \text{ Å}^{-1} \leq q \leq 4.210 \times 10^{-1} \text{ Å}^{-1}\).

This relatively small deviation between fitted result and experimental data yields an increasing peak at around 30 nm that can be observed in Figure 3.17 which shows the change in volume fraction of scatterers. The calculated volume fraction of the first peak at 20 nm \((q \approx 0.03 \text{ Å})\) for an error factor 2 is about 50% times higher as the volume fraction for an error factor of 5. In addition, there is a shift in the peak from 20 nm at an error factors of 2 to around 35 nm for an error factor of 5, as illustrated in Figure 3.17. An error factor of 1 results in three distinct high peaks at 10 nm, 20 nm and 35 nm for scatterers with a diameter of less than 50 nm which is illustrated in Figure 3.17 b) . With an increasing error factor the peak shifts towards higher diameter values. It could also be observed that
the whole distribution broadens for small error factors. The green line which indicates
an error factor of 5 reaches a volume fraction of zero at smaller sized scatterers already.
Choosing the right error factor always depends on the user and is highly subjective. Therefore, in an analysis of the SANS data very careful examination of the fitting results of each measurement point is necessary.

![Graph showing volume fraction of scatterers V(D) for different error factors.](a)

![Graph showing details of the fit for small spherical scatterers.](b)

**Figure 3.17:** Volume fraction of particles (V(D)), obtained from MAXE for four different error factors. a) shows the deviation between experimental and fitted result for the Intensity versus q plot. b) shows details of the fit for small spherical scatterers.

In Figure 3.18 the effect of changing the number of bins is depicted. By using a maximum particle diameter of 4000 Å a binning factor of 400 results in a subdivision of the volume fraction over a ΔD of 10 Å. A higher number of bins results in an insignificantly larger volume fraction for small particles. For the subsequent analysis, smoothing the data with 400 bins was applied.
**Figure 3.18:** Comparison of fitting results obtained from MAXE for four different binning factors ranging from 50 to 400.

Figure 3.19 illustrates changes of the fitting results if the background or signal from the incoherent scattering is changed. In Figure 3.19 a) it can be found, that the scattering signal, which is illustrated as the blue line, shows an upswing towards higher q. As a common practise for this work, the last three or four points were therefore, always disregarded as an artefact. If the last three measurement points are included this changes the background MAXE calculates from 0.0481 to 0.04815. For the following investigation, the background value was modified by ± 0.01 but this resulted in insignificant changes in the volume fraction.
Figure 3.19: Volume fraction of particles \( V(D) \), obtained from MAXE for four different error factors. a) shows the deviation between experimental and fitted result for the Intensity versus \( q \) plot. b) shows the volume fraction as a function of mean diameter for spherical scatterers.

Figure 3.20 displays the changes in volume fraction caused by partitioning the scattering data to part of the \( q \)-range that is \( 6.3 \times 10^{-3} \AA^{-1} \leq q \leq 4.21 \times 10^{-1} \AA^{-1} \). This relates to scattering particles within a diameter range of 1.5 nm to about 100 nm. This variation was investigated to determine if better fitting can be achieved by partitioning the data into two subsets. The black line shows the fit when the whole \( q \)-range \( 1.76 \times 10^{-3} \AA^{-1} \leq q \leq 4.210 \times 10^{-1} \AA^{-1} \) is analysed. The red line on the other hand, shows the results from fitting over a \( q \)-range where the minimum \( q \) value is changed to \( 6.3 \times 10^{-3} \AA^{-1} \) from \( 1.76 \times 10^{-3} \AA^{-1} \). A partitioned \( q \)-range results in a cut off (at 100 nm) of the polydisperse distribution which is highlighted by the red line in Figure 3.20. Within a range from 5 nm to 100 nm the volume fraction calculated from the partitioned \( q \)-range is slightly higher and broader compared to that using the whole \( q \)-range. However, since the fitting result does not improve significantly using a subset, the whole \( q \)-range ought to be used.
Figure 3.20: MAXE fitting results which shows effect of partitioning of the data input. The black curve covers the whole q range of $1.76 \times 10^{-3} \text{Å}^{-1} \leq q \leq 4.210 \times 10^{-1} \text{Å}^{-1}$ and the red curve covers a subset of the q range from $6.3 \times 10^{-3} \text{Å}^{-1} \leq q \leq 4.210 \times 10^{-1} \text{Å}^{-1}$ (only up to 100 nm diameter particle size).

Figure 3.18 and Figure 3.19 highlight that the number of bins and the background value do not affect the resulting volume fraction and size distribution. The data fitting is most significantly changed by the error factor, as illustrated in Figure 3.17.

3.5.9 McSAS

An independent SANS data analysis method that is a Monte Carlo based approach called McSAS for analysing uncorrelated scattering objects was investigated. The software package has been used widely for analysing small angle scattering (SAS) data [191, 196, 197]. A brief explanation of the software is given below but a more detailed description can be found in Bressler et. al [192]. McSAS uses a Monte Carlo algorithm to extract odd-shaped parameter distributions from the scattering data. This is particularly applicable
for polydisperse distributions of scatterers that are very common in metals and alloys. The software identifies the absolute volume fraction of scatterers as a function of size. In contrast to MAXE, it allows the user to work with a variety of scattering form factors which includes spheres, ellipsoids, rectangular objects, oblate nano particles etc. Prior information about the shape of scatterers or the scattering length densities of the phases within the sample matrix needs to be provided by other techniques such as microscopy. Once the initial parameter have been chosen, the software computes an intensity for random values chosen for the fitting parameters. Similar to MAXE, a $\chi^2$ is calculated as an error between the model and the measured data, weighted by the uncertainty of the measured data [198]. Through a Monte Carlo iteration process the fitting parameters are changed by replacing one of the scattering objects by another similar shaped object. The software evaluates if a reduction in $\chi^2$ is reached. If $\chi^2$ reaches a certain threshold limit the fitting process is eliminated. The graphical user interface (GUI) of McSAS is similar to MAXE, in its adjustable parameters. Parameters such as the error factor, a flat background level, number of bins or the number of iterations can be adjusted in both methods. It is worth noting that the units used in McSAS are different from MAXE. While the intensity used by MAXE is in $(cm(sr^{-1}))$ the intensity of the scattering data in McSAS has to be entered in $(msr^{-1})$. This means that the intensity has to be multiplied by a factor of 100. Furthermore, in McSAS the units of q are in $nm^{-1}$ rather than $Å^{-1}$. Thirdly, McSAS needs as an input the uncertainty estimate $\sigma(I)$ in $(m(sr^{-1}))$. Therefore particular attention should be given to conversion of the units since most data that is exported from neutron scattering instruments such as SANS2D and D11 are given in $(cm(sr−1))$ and $Å^{-1}$ for the intensity and the q-range respectively. As an output, McSAS gives the results of the fit versus q as well as the results of the volume weighted size distribution as a function of the radius of the data. Furthermore, McSAS calculates the uncertainty over each bar in the size distribution. If the number of bins is increased, more detail about the development of the distribution is revealed but the uncertainty as well as the observability limit is increased as well. For the distribution the user can choose between a number weighted or a volume weighted distribution. While the former gives a certain number of particles
within dimension bracket $D + \Delta D$ the latter relates to a certain mass or volume fraction. However, according to [192] if broad sized distributions are shown for small sized scatterers in a dispersed mixture as a number weighted distribution the values and uncertainties of the small sized scattering particles vary significantly and become excessively large. The distribution can be presented with linear or logarithmic scaling dependent on the expected results from the scattering data.
Table 3.3: Advanced settings and defaults that can be adjusted in McSAS [192].

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Convergence criterion</td>
<td>The least-squares value at which the converging solution is found. For data with a somewhat good error this can be set to larger values.</td>
</tr>
<tr>
<td>Number of bins</td>
<td>Gives the number with which the smooth function $V(D)$ is divided. A higher number of bins results in larger uncertainty limits and observability limits.</td>
</tr>
<tr>
<td>Number of repetitions</td>
<td>Sets the parameter for the number of independent optimisations. If the parameter is increased this results usually in improved uncertainty estimates on the results.</td>
</tr>
<tr>
<td>maxIterations</td>
<td>The amount of implemented iterations. If a convergence is not reached within this number the fitting process is aborted. Increasing this parameter increases the optimisation time.</td>
</tr>
<tr>
<td>Scaling</td>
<td>McSAS gives the option to show the results on a linear as well as logarithmic X or Y-scale.</td>
</tr>
<tr>
<td>Neutron scattering length density (sld) difference</td>
<td>The sld difference between sample matrix and scattering phase can be chosen. It is given in units of Å$^{-1}$.</td>
</tr>
<tr>
<td>Max particle diameter</td>
<td>The distribution is constrained by this parameter and it is used as a limit where the fitted distribution cuts off</td>
</tr>
<tr>
<td>Number of contributions</td>
<td>The number of contributions whose integrated sum comprises the intensity of the model. Most models can be fitted using a number of contribution of 300 [192].</td>
</tr>
<tr>
<td>Flat Background</td>
<td>A flat background is introduced that fits model and experimental data and accounts for the contribution from incoherent scattering.</td>
</tr>
</tbody>
</table>

The graphical user output from McSAS using a set of SANS2D data is presented in Figure 3.21. McSAS gives exports the experimental data and the fitted function as well as a histogram showing the distribution of the relative volume fraction $V(D)$ over the radius.
of the scattered particles. In the picture on the left, the red line shows the fitted function, the black line shows the experimental data and the green line shows the flat background. McSAS estimates for the contribution of incoherent scattering. McSAS gives the uncertainty estimates as well as the observability limits over each bin, which is shown as the red dotted line in the plot on the right side in Figure 3.21. If the number of bins is increased this usually results in larger uncertainty estimates and a larger observability limits. The standard deviation over each bin rises with the diameter of the scatterers. It is illustrated in Figure 3.21 that small scatterers have a smaller standard deviation. For larger scatterers the uncertainty rises. The McSAS analysis presents the selected q-range and calculates the total value (total volume fraction) as well as the mean of the distribution, the variance, the skew and kurtosis. The distribution shown in Figure 3.21 is the volume weighted size distribution of spherical cavities plotted over 50 bins as a function of the radius.

**Figure 3.21:** Graphical output from McSAS showing the best fitted result of the intensity over q in the left plot as well as the volume weighted sized distribution that is associated with the particular fit of the data in the plot on the right side. The volume fraction calculated by McSAS is given as a function of the radius. MC in the title bar refers to Monte Carlo.
3.5.10 Analysis procedure

A common procedure for analysing data from SANS experiments is to subtract the scattering intensity of measurement points from a reference point prior to the data fitting procedure. However, this approach increases the noise significantly in a way such that MAXE sometimes failed to find a convergent solution. An alternative approach was adopted where the absolute volume fraction of scatterers was calculated before subtracting the reference data and dividing by the contrast factor. The results from the two approaches are depicted in Figure 3.22 and give very similar results in a size range of 20 nm to about 400 nm. Both methods show a narrow peak for small scatterers and a more broader peak for larger ones. However, it is apparent that calculating $V(D)$ first before subtracting the fitting results, gives a disproportionate high peak at around 5 nm that is not detectable in the other two plots in Figure 3.22 from the MAXE as well as the McSAS analysis. This highlights that data fitting prior to subtracting the scattering results from the reference points can cause artefacts. It is therefore recommended, if possible, to apply fitting after subtracting the SANS signal of the reference points from the measurement points.
3.5.11 Simulated Scattering Data using SasView

SasView is a SANS analysis software package which was originally developed by the University of Tennessee as part of the NSF Data Analysis of Neutron Scattering Experiments (DANSE) project and is now maintained by an international collaboration of scattering facilities. The software was used to generate a set of simulated scattering data to determine the rigour of the MAXE and McSAS fitting analysis. The set of parameters generated in SasView are listed in Table 3.4. In the simulated data, polydisperse spherical scatterers with a Gaussian distribution and a PD of 0.1 have been chosen. PD is defined as the ratio of standard deviation $\sigma$ and the mean $\bar{x}$. SasView generates a 1D scattering intensity, which is illustrated in Figure 3.23, as described by Guinier et al. [184]:

$$I(q) = \frac{\text{scale}}{V} \cdot \left[ V(\Delta \rho)^2 \cdot \frac{3 \sin(qr) - qr \cos(qr))}{(qr)^3} \right]^2 + \text{background} \quad (3.6)$$

Figure 3.22: Comparison of SANS2D analysed with MAXE and D11 data analysed with McSAS.
where scale is the volume fraction, \( V \) is the volume of the scatterer, \( r \) is the radius of the spherical scatterer and background is the background level. Furthermore, the difference in the scattering length density for the solvent and the scatterer is incorporated in the contrast factor (CF) \((\Delta \rho)^2\).

Table 3.4: Set of demonstration data set generated in SasView with the results of the fitting calculated using MAXE and McSAS.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>SASView Simulated</th>
<th>MAXE Result</th>
<th>McSAS Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scattering length Density (Sld) Scatterer</td>
<td>1E-6 A(^{-2})</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Sld Solvent</td>
<td>0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Polydispersity</td>
<td>Gaussian</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PD = (\sigma/\bar{x})</td>
<td>0.1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total Volume Fraction of scatterers (scale)</td>
<td>(1 \times 10^{-2})</td>
<td>1.0028 (\times 10^{-2})</td>
<td>1.006 (\times 10^{-3})</td>
</tr>
<tr>
<td>Background</td>
<td>(5 \times 10^{-3})</td>
<td>4.919 (\times 10^{-3})</td>
<td>4.98 (\times 10^{-3})</td>
</tr>
<tr>
<td>Mean Diameter</td>
<td>200 nm</td>
<td>206.3 nm</td>
<td>208.8 nm</td>
</tr>
</tbody>
</table>

The 1D scattering intensity \( I \) versus \( q \) based on the parameters in Table 3.4 and the results from the fitting using MAXE and McSAS are illustrated in Figure 3.23. It could be found that both software packages find a good approximation for the fitting function that matches the simulated data. In SASView it is possible to export the intensity \( I \) versus \( q \) data but the standard deviation from the intensity is not given. Therefore, the quality of the fitting result has to be evaluated by eye.
Figure 3.23: Simulated data generated in SasView of 1D scattering intensity versus q from spherical scatterers with a Gaussian distribution with the fitting results from McSAS (a) and from MAXE (b).

The results from both methods of the smooth function $V(D)$, which is defined such as $V(D) \cdot \Delta D$ gives the total volume fraction of scatterers, are illustrated in Figure 3.24. It can be found that the distribution function from MAXE is matched reasonably well by McSAS. The total volume fraction of scatterers is $1.0028 \times 10^{-2}$ and $1.006 \times 10^{-2}$ calculated by MAXE and McSAS respectively. This is relatively close to the simulated volume fraction or scale factor of $1.00 \times 10^{-2}$. 
The analysis of the simulated data showed that the mean diameter from the MAXE and McSAS result is slightly shifted compared to the mean diameter of the simulation data which was set to 200 nm. This is illustrated in Figure 3.25 where the distribution function of the normalised volume fraction of scatterers is plotted. The shift can be explained by the difference between the numeric mean and the volumetric mean. Whilst the numeric mean implies a certain number of $N$ particles within $D$ and $D + \Delta D$ nm a volumetric mean implies a certain volume or mass fraction within $D$ and $D + \Delta D$ nm. MAXE and McSAS calculate the volumetric mean whereas the SasView simulated data relates to the numeric mean. The shift becomes more significant with a larger standard deviation of the polydisperse distribution of the simulated data since the number-weighted and volume-weighted distributions are no longer identical. In the case that is shown here, the shift
between simulated and calculated mean diameter is 6.3 nm and 8.8 nm for MAXE and McSAS respectively (listed in Table 3.4). Tests were also carried out for a set of simulated data that represent scatterers with a mean diameter smaller than 200 nm. The results of this analysis showed similar matching results.

Figure 3.25: Comparison of the normalised volume fraction of scatters of the SasView simulated data number weighted distribution) and fitted results from McSAS and MAXE (volume weighted distribution) which shows a slight shift in the Sasview simulated data.

3.5.12 Analysis of fitting data

In the as-received material the scattering signal originated from features such as precipitates, cavities and with a lesser intensity from grain boundaries and dislocations. In order to separate the absolute volume fraction of cavities, the macroscopic cross section from each sample position had to be subtracted from measured results from the grip area were the sample has experienced temperature but since it is stress free the amount of cavitation
can be neglected. It is assumed that the evolution of carbides is homogenous along the whole sample. \( C(D) \) which is the volume fraction distribution of spherical cavities can be calculated according to the following equation:

\[
C(D) = \frac{V(D) - V(D)_{\text{Ref}}}{CF},
\]

where \( V(D) \) is the volume fraction of scatterers, precipitates and cavities. \( V(D)_{\text{Ref}} \) is defined as the volume fraction of scatterers at the reference position. \( CF \) defines the contrast factor which is the squared difference of the neutron scattering length density of the steel matrix and the cavities. The contrast factor is calculated from the difference of the squared neutron scattering length density \( \rho_1^2 - \rho_0^2 \). The scattering length density (SLD) of cavities \( \rho_0^2 \) equals zero which entails that the total contrast factor results from the chemical composition of the steel matrix \( \rho_1^2 \), which has been previously explained in Section 3.5.5. The fitting methods MAXE and McSAS provide a size distribution function of scattering particles over a certain diameter range. The results of the volume fraction can be used to determine other parameters such as the number density or mean diameter of scatterers. The number density of spheres for example can be calculated using:

\[
N(D) = \frac{C(D)}{V_{\text{mean}}},
\]

where \( V_{\text{mean}} \) denotes the average volume of the spherical cavities \( V = \frac{4}{3}\pi r^3 \). For each measurement point on the sample, the number density can be calculated according to Equation 3.8. If one wants to calculate the mean diameter of scatterers with a diameter of less than 60 nm it was found to be best practice to fit these peaks to a Gaussian function and to deduce the mean diameter from these results. Since the distribution of larger sized particles is a more fluctuating function with usually several small peaks it was found to be best practice to calculate the mean diameter for the broader size distributions for scatterers with diameters between 60 and 400 nm, by a method of moments and direct summation [165].
For a further analysis, parameters such as the number density of cavities per grain boundary area, as well as the spacing of creep cavities per grain boundary area were calculated. Based on these metrics of the total volumetric number density, the areal density of cavities in the grain boundary area can be estimated using:

\[ N_a(D) = \frac{N(D)}{S_v}, \]  

(3.9)

where \( S_v \) denotes the grain boundary surface area, the ratio of grain boundary surface area and grain volume. Equation 3.9 can be applied when it is assumed that all cavities lie on grain boundaries and are uniformly distributed. A simplified estimation is given in the ASTM standards where the surface area can be calculated using:

\[ S_v = \frac{2}{\bar{l}}, \]  

(3.10)

where \( \bar{l} \) is the average grain size [199]. The average grain size of the as-received Type 316H stainless steel was measured and established to be roughly 80 \( \mu m \), as later described in Section 4.3.1. This is a simplification under the assumption that the grain size does not change due to prestrain and creep. The average spacing of cavities can be obtained from the number of cavities per unit grain boundary area [108]:

\[ L = \frac{1}{\sqrt{N_a(D)}}. \]  

(3.11)

3.6 Summary

This chapter has described the experimental techniques used and the data analysis techniques applied for the purpose of determine the tensile properties, the creep deformation properties using 3D-DIC and the creep cavitation damage of Type 316H stainless steel as a function of plastic strain and applied stress at a temperature of 550\(^\circ\)C. A description
of the operating principles of 3D-DIC to measure the strain on complex sample surfaces in all three dimensions has been outlined. Procedures have been presented to improve the adhesiveness of the coating on the sample. Furthermore, the method of strain smoothing, implemented to analyse the creep strain data without eliminating essential features of the raw data, has been described. In addition to SANS data analysis using MAXE, an approach using the independent software package McSAS has been assessed including its possibilities to determine the volume fraction and distribution of scattering particles of crept samples of AISI Type 316H stainless steel. The data analysis procedure for the neutron scattering data has been described in detail and an approach outlined that defines the best setting for each software package. The capabilities of MAXE and McSAS to fit a simulated demonstration set of spherical scatterers has been analysed. It was found that the results from both methods are almost identical which gives confidence in the rigour of both methods to study creep cavitation in Type 316H stainless steel. This supports the conclusions drawn from the analysis of the scattering data from more complex samples.

1. The measurement procedures for using 3D-DIC to measure creep deformation at high temperature have been presented.

2. The sample preparation procedure used for 3D-DIC has been described. In addition, an analysis on improving the adhesiveness of the coating on the hourglass specimen has been given.

3. The methods and settings of two independent software methods have been outlined and discussed for best analysis practise of SANS measurement data.

4. Two independent software methods (MAXE and McSAS) were used to fit a set of simulated scattering data which proved the rigour of both methods to analyse SANS data.

5. Imaging techniques such as OM and SEM have been described as methods to determine creep cavitation in the as-received material. Furthermore, a procedure to study creep cavities on samples which have been fractured using the cryogenic method has
been outlined.
4 Creep Testing On A Novel Cylindrical Hourglass Specimen

4.1 Introduction

Multiple creep strain curves using 3D-DIC on a novel cylindrical hourglass specimen (CHS) made of the as-received material (Type 316H stainless steel) were measured. That was followed by evaluating creep damage as a function of stress using SANS on a slice of the cylindrical hourglass specimen. The creep test was conducted at the OU high temperature creep laboratory on a load controlled setting carried out at a temperature of 550°C. The creep strain on the surface of the sample was mapped using the non-contact technique of 3D-DIC, which has been described in Chapter 3. The results of the DIC measurement could be correlated to the calculated stress at each position along the length of the hourglass section. Also, the extended results from this experiment were correlated to that measured using SANS.

4.2 As-received Material

The material utilised for this work was provided by EDF Energy and is representative of plant material in its chemical composition. Since actual plant material is always short in supply and has inherited properties that can add complexity to the experimental work this virgin cast of Type 316H stainless steel was selected to be tested by EDF and its partner universities for the high temperature material testing programme. The as-received material is more suitable for systematic studies and can be more reliably compared between different HTC partners. The chemical composition of the as-received material can be found in Table 4.1 and confirms the specified conditions for Type 316H stainless steel in ASTM A479/479M-17 [200]. The high amount of carbon in Type 316H stainless steel that accompanies a loss in ductility is usually offset by an increasing amount of manganese.
Table 4.1: Chemical composition of as received 316H stainless steel material (Cast CD25520) measured by ELG Haniel Metals.

<table>
<thead>
<tr>
<th>Cast No</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Cu</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>CD25520</td>
<td>0.069</td>
<td>0.41</td>
<td>1.37</td>
<td>0.36</td>
<td>0.009</td>
<td>16.95</td>
<td>2.11</td>
<td>10.08</td>
<td>0.41</td>
<td>0.054</td>
</tr>
</tbody>
</table>

The as-received material, as illustrated in Figure 4.1, was received as a 250 mm long bar with a 90 mm diameter and was sectioned from an original 20 m long bar with a weight of 994 kg. The original bar was manufactured with electric arc melting and was vacuum degassed. This was followed by hot forging and a solution annealing at 1060°C for 4 hours. After solution annealing, the bar was water quenched [201].

![Image of the cylindrical bar of the as-received material.](image)

Figure 4.1: Image of the cylindrical bar of the as-received material.

### 4.3 Characterisation and sample design of as-received material

#### 4.3.1 Grain Size Measurement

A 5 mm disk was cut off from the top of the test bar and different pieces were cut to enable grain size measurements on several positions through the cross section of the as-received
bar. A sketch that illustrates the sections where grain size measurements were performed is shown in Figure 4.2.

![Figure 4.2: Sketch through the cross section of the as-received bar showing where grain size measurements were performed.](image)

The grain sizes were measured from longitudinal sections of the bar. The measurements were performed using the Leica Application Suite software and by applying the mean linear intercept (MLI) method described in ASTM E112-13 [199]. From each section, six positions were picked and then measured by the software. The MLI method was performed in vertical as well as horizontal orientations to smoothen outliers. The average grain sizes were measured by dividing the length of the lines by the amounts of intersections.

**Table 4.2:** Grain size calculations for different sample positions along the cylinder measured in perpendicular to the axial direction.

<table>
<thead>
<tr>
<th>Position</th>
<th>Sample Section</th>
<th>Specimen Ave grain size (µm)</th>
<th>ASTM #</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centre</td>
<td>1</td>
<td>82.5 ± 3.73 µm</td>
<td>4.25 ± 0.142</td>
</tr>
<tr>
<td>Edge</td>
<td>5</td>
<td>79.4 ± 4.4 µm</td>
<td>4.35 ± 0.18</td>
</tr>
</tbody>
</table>

The data in Table 4.2 reveals that the difference in grain size from the centre of the bar to the edge of the bar is around 3 µm. The average grain size was measured to be 80.9 µm with an ASTM grain size number of 4.3. The difference between the maximum and
minimum grain size in all of the conducted measurements was found to be less than 16 µm. Overall, the grain morphology was relatively similar overall tested areas. This implies that the as-received material has a homogeneous grain size distribution, so the test specimen could be freely extracted from different places in the bar. Twinning was observed which is believed to be caused by the solution annealing and subsequent water quenching within the manufacturing route of the material. The number of twin boundaries was found to be relatively even distributed over the whole scanned section with a slight dominance at the outer part of the cylinder. The results confirm the findings from Wood [201]. Two images, taken with the optical microscope, illustrate the grain sizes at the centre of the sample in Figure 4.3 and at the edge of the sample in Figure 4.4.

Figure 4.3: Optical micrograph of the as received material at the centre of the bar (Section 1).
Figure 4.4: Optical micrograph of the as received material at the edge of the bar (Section 5).

Figure 4.5 shows some of the twin boundaries that were observed in sample location 1 in the centre of the cylindrical bar.

Figure 4.5: Optical micrograph of the as received material showing examples of twin boundaries.
4.3.2 Sample Design

A new cylindrical test specimen was designed for the present research with a length of 85 mm and a diameter of 8 mm was extracted longitudinally from the as-received material. Out of this cylindrical specimen an hourglass shape was machined. The CHS design is illustrated in Figure 4.6. The length of the hourglass gauge section is 28 mm. In the mid-section the diameter narrows down to 6 mm from 8 mm in the straight cylindrical section. The overall radius of the hourglass gauge section is 100 mm.

![Figure 4.6: Technical drawing of the novel hourglass test specimen design.](image)

The same hourglass specimen design was used for all 3D-DIC measurements throughout this research project and hence the length of the hourglass section was determined by the window size of the furnace which is 40 mm in height and 99 mm in width. Since the edges of the window cause artefacts in the analysis due to chromatic aberration, the hourglass gauge section had to be designed to be shorter than 40 mm. Two 1 mm sized holes were drilled about 35 mm away from the midpoint of the sample to insert thermocouples. Holes were found to be the best solution to ensure good thermal contact by minimising the chances of slipping. The specimen has a 6 mm diameter shoulder and M12 screw ends.

Finite element analysis (FEA) has been used to predict the difference between peak stress and strain and its minimum at the straight section of the cylindrical sample. The initial idea behind the design was to have a certain limit in the stress- as well as strain gradient to optimise it for a subsequent investigation using small angle neutron scattering. If the radius of the hourglass section becomes more and more shallow not much creep strain variation can be observed. The concern here was that little changes in creep strain reduce the
likelihood of significant changes in cavitation density that can be measured using SANS. On the other hand, steep changes in creep stress and strain variation can cause large variation of creep cavitation damage in a short section of the gauge area, which again may not be accurately measurable using SANS. The initial finite element analysis was based on creep data of a parent Type 316H stainless steel material from a different cast that included strain hardening data to simulate the elastic-plastic response of the specimen and data from the R66. The creep response of the material was modelled by a user defined field USDFLD subroutine. Based on these estimations, a design was chosen where the peak stress falls to about 54\% from the mid-hourglass section to the straight section of the cylindrical sample.

### 4.3.3 Tensile Properties

The tensile properties were obtained from a test conducted at room temperature. Figure 4.7 shows a dimensioned technical drawing for the type of test specimen used, a uniaxial cylindrical specimen with a gauge diameter of 8 mm, a gauge length of 43 mm and a total length of 85 mm. The same sample design has been used for the tensile tests conducted at room temperature and at 550°C.

![Figure 4.7: Design of specimen used for tensile tests at room and elevated temperature.](image)

The displacement rate of the tensile test was 1 mm/min and the test was carried out until rupture of the specimen. The material properties such as the 0.2 \%, 1 \% proof stress as well as the ultimate tensile strength (UTS) and the failure strain $\epsilon_f$ are shown in Table 4.3.
Table 4.3: Tensile properties for as received material for test properties at room temperature and at 550°C.

<table>
<thead>
<tr>
<th>Test Name</th>
<th>Temperature</th>
<th>E (GPa)</th>
<th>$\sigma_{0.2%}$ (MPa)</th>
<th>$\sigma_{1%}$ (MPa)</th>
<th>UTS (MPa)</th>
<th>$\epsilon_f$ %</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR-OU-550</td>
<td>550°C</td>
<td>153</td>
<td>144</td>
<td>175</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>AR-OU-RT</td>
<td>RT</td>
<td>168</td>
<td>257</td>
<td>297</td>
<td>609 MPa</td>
<td>66.7</td>
</tr>
<tr>
<td>AR-Wood-550 Test</td>
<td>550°C</td>
<td>149</td>
<td>147</td>
<td>167</td>
<td>462</td>
<td>55.5</td>
</tr>
</tbody>
</table>

Properties such as Young’s modulus $E$ and the $UTS$ were calculated using the engineering stress and strain response of the specimen whereas, the $\sigma_{0.2\%}$ and $\sigma_{1\%}$ proof stress are based on calculated true stress and strain values. Both values were found based on the intersection of the true stress and strain curves with parallel lines to the linear elastic part with an offset of 0.2% and 1% respectively. The engineering stress and strain results from the tensile tests conducted at room temperature and at 550°C are illustrated in Figure 4.8 a) and Figure 4.8 b) respectively. The tensile test at room temperature has been carried out until failure of the specimen, whereas the test at 550°C has been conducted up to 20%, the gauge limit of the high temperature extensometer. The procedure on how the tensile tests at room temperature and at 550°C were conducted has been described in more detail in Section 3.2. The drop in the room temperature stress and strain curve at an engineering strain of 20% can be explained because the extensometer reached its limit and had to be taken out. Afterwards, the measurement had to be restarted again. Any further monitoring of the displacement was done using the slow strain rate machine. The data had to be stitched to reproduce the whole stress and strain curve. The tensile test conducted at a temperature of 550°C showed evidence of dynamic strain ageing (DSA). It is an effect that has been observed in austenitic steels for tensile tests within a temperature range between 273°C and 800°C strain rate [202]. While the deformation is generally not affected by the strain rate at room temperature, this changes at higher temperature where the flow stress is influenced by the strain rate [203]. Figure 5.3 illustrates that the effect of dynamic strain ageing starting to occur at a true strain of around 1%, which corresponds to a stress of about 175 MPa. The effect of dynamic strain or Portevin-Le Chatelier effect
describes the discontinuous yielding. The large strain pulses that are significant for the DSA effect can lead to internal microstructural damage [204]. Only one tensile test at a temperature of 550°C has been carried out at the OU. However, the results have been compared with two tests conducted at Imperial College London which showed a good agreement.

\[
\sigma_{\text{true}} = \sigma_{\text{nom}}(1 + \epsilon_{\text{load}}) \tag{4.1}
\]

as well as:

\[
\epsilon_{\text{true}} = ln\left(\frac{l}{l_0}\right) = ln(1 + \epsilon_{\text{eng}}) \tag{4.2}
\]

**Figure 4.8:** Tensile test results (or stress-strain curve) of as received material performed at a) room temperature at the OU, and b) at 550°C (AR-OU-550), as summarised in the Table 4.3.
4.4 3D-DIC Creep Test on CHS Specimen

This section presents a short description of the experimental setup and the results from the 3D-DIC test. The plastic strain from loading together with the creep strain is illustrated in the subsequent Figures.

4.4.1 Experimental Methods

Creep properties from a static load creep test have been obtained using the non-contact 3D-Digital Image Correlation deformation monitoring technique which has been described in detail in Section 3.3. A sketch of the set up of the 3D-DIC experiment can be found in Figure 3.1. With the design of the cylindrical hourglass specimen, as shown Figure 4.6, it is possible to measure multiple creep curves within just one specimen. A high temperature creep test was performed to measure the creep properties at a temperature of 550°C. The sample was placed in the middle of the furnace to give an optimal view of the hourglass gauge section. The specific design of the hourglass specimen leads to a stress range from about 200 MPa to 355 MPa engineering stress when a maximum load of 10,043 N is applied. The stress decreases roughly by 54% from its maximum value. With a slight discrepancy due to the machining process, a diameter of 5.97 mm instead of 6 mm was measured in the mid-section of the hourglass gauge length. Prior to the creep test, a silica ceramic-based paint was used to create a randomised speckle pattern on the surface of the hourglass gauge length. A combination of a white primer with a finish of black speckles helps in two ways. Firstly, it increases the contrast and secondly, it reduces any effects of oxidation. These oxidation effects are minimal for stainless steels but considering the long duration of each creep test, it was found to be useful. As described in Section 3.3.4, etching prior to coating the sample was found not to improve the grip of the paint and therefore the creep tests were carried out without further etching. Type N thermocouples were used in the test to monitor the temperature throughout the whole creep test. It was ensured that the temperature was kept stable between ±1°C. With the camera system the primary, as
well as the tertiary creep regime, were scanned more frequently than the secondary creep regime. The interval within primary as well as tertiary creep was 5 min and changed to 30 min within secondary creep. Since there is no integrated feedback within the system, the pictures were analysed regularly to monitor the different stages. The hourglass specimen was illuminated using a fibre optic light cable attached to the flash unit of one of the DSLR cameras. For the whole creep test, a total of 2566 pictures on each camera were recorded and analysed using the DaVis software to compute the displacement vectors [175]. The loading part was separated from the creep part to distinguish time-dependent creep from time-independent plastic strain.

4.4.2 Results

4.4.2.1 Plastic strain from loading

After applying each dead weight to the specimen, a picture was taken and the plastic strain from loading could be recorded. It was possible to monitor the surface strain over a specimen area of approximately 18 mm by 6 mm. The maximum loading strain $\epsilon_{yy}$ in the tensile direction was found to be 14.5% with a maximum transversal strain of $-6\%$. The position with the smallest diameter within the gauge length was defined to be at 0 mm in the x-axis in all subsequent Figures where the strain is plotted against the position. The tensile and transversal strain from loading prior to creep is illustrated in Figure 4.9. The high loading strain in tensile direction which acts as prior hot work is changing over the hourglass gauge section based on the corresponding diameter. At a distance of 10 mm away from the mid-section, the plastic strain in tensile direction is still about 7% with a transversal strain of around $-2.8\%$. 

Figure 4.9: Average strain from loading across the length of the hourglass gauge section in a) longitudinal and b) transversal direction.

An example of the finite element analysis used to calculate the strain from loading is illustrated in Figure 4.10. Figure 4.10 a) shows the FE calculation of LE22 which is the sum of elastic and plastic strain in loading direction. From that simulation, a line plot has been exported over one half-length of the test specimen, up until the straight section, which is illustrated in Figure 4.10 b). The data from the FE simulation is depicted together with the experimental results from the hot loading strain in tensile direction. For the FE-modelling one quarter of the specimen has been modelled to safe computational time. It can be found that the 3D-DIC result is in good agreement with the predicted loading strain. The elastic-plastic properties of the material were used from tensile testing conducted at 550°C and at a strain rate of 0.0004 s\(^{-1}\) carried out by Wood to characterise the material [201].
Figure 4.10: Illustrates a) FE-modelling of the plastic and elastic strain behaviour over one half length of the test specimen and b) and the line plot in y direction of the elastic and plastic strain simulated with ABAQUS and the measured strain using 3D-DIC.

During loading, some of the paint flaked off from the sample. The specimen experienced significant loading strain since the experiment is conducted well beyond the yield strength of the material, which was measured to be at roughly 145 MPa at a temperature of 550°C. As described in Section 3.3.4 many factors can contribute to this. The sample couldn’t be machined using wire electro-discharge machining (EDM) which often creates a rough matt surface helping the paint to stick to the sample. Secondly, the paint needs to be applied as a very thin layer which often is tricky to realise using customary spray cans. The inhomogeneity of the paint in terms of its thickness due to manual application promotes the detachment of the paint. Nevertheless, the reconstructed displacement field was not significantly influenced by missing speckle points. It was still possible for the DIC software DaVis 8.4 to find a converging solution.
4.4.3 Creep test results

Using 3D-DIC on the cylindrical hourglass specimen, a total of 50 creep curves, covering a wide start of test stress-range, could be spatially resolved, which is a great advantage of using this sample design and the technique. Each creep region was sampled over 0.5 mm length with an overlap of 75% to increase the spatial resolution. Figure 4.11 shows the creep strain evolution over the whole time span up to failure at 851 hours. It was possible to monitor three distinct creep zones, primary, secondary and the tertiary creep regime.

![Creep deformation curves](image)

**Figure 4.11**: Creep deformation curves at six different positions along the gauge length of the cylindrical hourglass specimen measured by 3D-DIC, covers a true stress variation 404-284 MPa up to creep failure at 851 h.

One can obtain a significant variation across each creep curve. While some of the curves reach the tertiary regime, it can be observed that some parts of the hourglass gauge section stayed within secondary until rupture. The highest creep ductility was found to be in the middle of the sample where true stress after loading is the highest with 404 MPa. At this position, a creep ductility of approximately 4.0% was measured as illustrated in Figure 4.11.
Figure 4.12: Creep deformation curves at six different positions along the gauge length of the cylindrical hourglass specimen measured by 3D-DIC, covers a true stress variation 404-284 MPa up to time of 200 h.

In Figure 4.12, the primary creep regime (up to 200 h) is plotted for the same stress regions as in Figure 4.11 along the hourglass gauge length. Creep curves from six positions were selected and plotted in this figure spanning a stress range from around 404 MPa to about 284 MPa. A large variation in primary creep strain could be observed. While at the highest stress a creep strain of around 0.3% is reached after 200 h, the amount of strain decreased to about 0.05 % for a stress of 284 MPa. It can also be found that the creep strain behaviour for 404 MPa is very similar to the creep behaviour of 394 MPa, which is about 2 mm away from the central position with the maximum stress. In general, it could be observed that the creep strain evolves quite similarly around the mid-hourglass gauge section where the stress variation is minor. However, a clear trend can be observed. The amount of creep strain reduces with stress based on increasing diameter of each position. Both Figures, 4.11 and 4.12, show that it is possible to spatially resolve creep strain rates along different positions and changes in creep strain even for relatively small amounts of about 0.05 % creep strain.
Figure 4.13 shows the obtained creep strain in both, the longitudinal $\epsilon_{yy}$ and transversal direction $\epsilon_{xx}$ along the hourglass gauge length. There is a variation in creep strain from around 4% to about 0.1% in an area $\pm$ 5 mm away from the failure position in the middle of the hourglass section. The creep strain in the transverse direction drops to approximately -1.6% in the mid-hourglass gauge section. It was found that the creep strain at failure for the as-received material is relatively low. The creep ductility for ex-service 316H stainless steel has been found larger. However, it is not straightforward to compare because there is always a huge scatter in creep ductility data and since there is a strong stress dependency [205]. With the possibility of monitoring strain in transverse direction, it is possible at each time during the creep test to calculate the true stress of the specimen.

![Figure 4.13: Average strain in a) longitudinal and b) transversal direction across the hourglass gauge length using 3D-DIC data from creep test performed at 550°C.](image)

In Figure 4.14 the accumulated creep strain as a function of time and position over the hourglass gauge section is illustrated. Up until a time of around 500 hours, the creep curves around the middle show a very similar evolution. As mentioned earlier, the position with the maximum stress is marked at the position 0 mm. Just within the last 300 hours, the creep strain over the mid-section of the sample is accelerating.
Figure 4.14: 3D surface plots showing the time and spatial resolved evolution of creep strain along the hourglass section of the cylindrical specimen using 3D-DIC data from the creep test performed at 550°C.

4.4.4 Creep strain rate results

With creep experiments in general, it is quite challenging to determine the steady-state creep regime. In 3D-DIC, there is significant fluctuation from thermal currents, adding a huge scatter on the creep strain rate. To distinguish the onset and end of the steady-state creep regime a scatter band of +20% was added to the point where the minimum creep strain was found. The intersection between creep strain rate and the area around the minimum creep strain rate defines the three regimes as illustrated in Figure 4.15. The
black dashed line is tangent to the minimum creep strain rate. The 20% scatter band is indicated by the red area and the green section symbolises the steady-state creep range. To calculate changes of the steady-state creep rate, an average value in this section was determined. This approach has been adopted from a report on creep behaviour on Type 316L stainless steel conducted at the KIT [206]. It was found that this margin led to different time ranges for the steady-state creep based on the position within the hourglass gauge length. Regions with lower stresses spent longer fractions of their creep time in secondary creep whereas, for positions at higher stresses tertiary creep became more and more dominant. For each curve, the minimum creep strain rate was averaged over the whole steady-state section to minimise any potential errors.

![Figure 4.15: Determination of characteristic creep regimes from the creep strain curve from the mid-section of the hourglass gauge length with a true stress of 404 MPa.](image)

The creep strain rate of the position in the mid-section of the hourglass gauge length is illustrated in Figure 4.15. At this stress position, it could be found that the creep strain rate starts at around \(2 \times 10^{-4} \text{ h}^{-1}\), slows down to about \(3 \times 10^{-6} \text{ h}^{-1}\) and then rises to around \(2.8 \times 10^{-3} \text{ h}^{-1}\) right before creep failure. Figure 4.15 highlights the typical bathtub shape that could be observed. With this approach and a scatter band of 20% added to the
minimum creep strain rate, a secondary or steady-state regime could be estimated in a time frame from around 180 hours to about 360 hours as highlighted in green in Figure 4.15. For this particular creep curve, it can be stated that the dominating creep mechanism is tertiary creep with a life fraction of around 58% (\((851-360)/851=58\)%) spent in this regime. The time of minimum creep strain rate was determined to be at around 260 hours which translates roughly to \(t/t_f=0.31\) of the normalised creep life. The normalised creep life is simply the ratio of creep time and time to failure. 3D-DIC in combination with the novel hourglass design of the specimen allowed determination of multiple creep curves from just one test specimen. A selection of creep curves were examined to calculate the creep strain rate as a function of creep time to time to failure (\(t/t_f\)), as illustrated in Figure 4.16. The creep strain rates all start within the order of around \(1 \times 10^{-4} \, h^{-1}\) to \(1 \times 10^{-5} \, h^{-1}\), based on the position within the hourglass gauge section. For a stress range of around 400 MPa to about 320 MPa, the minimum creep strain rate varies within the order of a magnitude between \(3 \times 10^{-5} \, h^{-1}\) and \(2 \times 10^{-6} \, h^{-1}\). The data reveals that the time of the minimum creep strain rate is relatively similar for all curves at around \(t/t_f=0.35\), shown for a stress range of 404 MPa to 322 MPa, as illustrated in Figure 4.16.
For positions with true stresses less than 320 MPa, it was found to be challenging to determine the minimum creep strain rates. This was due to large fluctuations in the raw data and the associated uncertainty. These curves had to be excluded from any further analysis of the creep strain rate. A steady-state creep strain rate could be established and fitted to Norton's creep law of the form of Equation 5.1:

\[ \dot{\epsilon} = A \sigma^n \]  

(4.3)

where \( A \) denotes a time-dependent constant and \( n \) is the creep exponent. The parameters were found to be \( A = 1.717 \times 10^{-29} \) and \( n = 8.96 \). Since it was not possible to establish the creep strain rate for the whole hourglass gauge length a selection had to be made. For this analysis, creep strain curves within an applied stress range between 404 MPa and 328 MPa were selected, see Figure 4.17.
Figure 4.17: Minimum creep strain rates within an applied stress range between 404 MPa and 328 MPa fitted to the Norton creep law.

4.5 SANS experiments

4.5.1 Experimental Methods

The SANS technique (as described in great details in Section 3.5) was used to measure creep damage in the crept CHS sample. The small-angle neutron measurements were conducted at the ISIS neutron source at the Rutherford Appleton Laboratory on the SANS2D instrument. Before carrying out the neutron scattering experiments, the thickness of the sample was measured precisely, using a caliper with an accuracy of ± 0.02 mm, to adjust for absorption corrections in the data reduction. The failed specimen was sectioned into a 1 mm thick longitudinal slice, taken from the middle of the hourglass test specimen. A thickness of 1 mm has been found optimal for Type 316H stainless steel \cite{164,165}. With thicker samples, the effect of multiple scattering becomes more likely, making it impossible
to quantify the cavitation damage. Creep cavities were measured at various measurement points along the hourglass section. A rectangular-shaped aperture with the dimensions 2 mm by 5 mm was used to scan each measurement position. From both sides of the creep failed hourglass specimen, 20 measurements were taken in total. The first five positions close to the failure position were at a distance of 1.5 mm to each other. After that, the subsequent distance was increased to 2 mm. This is illustrated in Figure 4.18 where the important measurement points used for the analysis are marked. In addition, two more samples have been analysed using SANS. As a second sample a stripe of the as-received Type 316H stainless steel material was used as a pristine reference material which hasn’t seen any prior creep strain or elevated temperature. As a third, a cylindrical sample with an hourglass shape was used to measure the effect of hot plastic strain on cavitation damage. The idea to test a sample that was only plastically deformed, was to probe to which extent plastic strain causes cavities, that might be pre-existent in the material, to grow. This sample is to be referred to as the plastically deformed cylindrical hourglass (PCHS) sample in the following sections. The specimen was kept in the furnace for 24 hours at a temperature of 550°C. Then, the same load of 10,043 N as applied to the CHS sample, was used. Right after applying the load, the temperature was turned off and the sample was taken out of the furnace.
For each measurement, an acquisition time of 60 min was used. Points within the grip region of the hourglass sample were used as reference measurements. Here, it can be assumed that the specimen has experienced a negligible stress compared to the hourglass gauge section and therefore cavitation damage can be neglected. The SANS data from each measured point were analysed using MAXE, as described in more detail in Section 3.5.7, that calculates the volume fraction of scattering objects as a function of diameter, assuming a spherical structure of the scattering objects. This is a good approximation to a range of cavity shapes. If for example, ellipsoidal cavities are distributed randomly inside the material they average out to spherical objects.
4.5.2 SANS Results

The results from the MAXE fitting procedure, using results from one exemplary sample position obtained from the SANS2D instrument, are illustrated in Figure 4.19. The background, which represents the amount of inelastic or incoherent scattering, is illustrated as the red dashed line and was calculated to be approximately 0.042. The orange line shows the absolute experimental scattering data plotted over the q-range. It can be found that the standard deviation, which is illustrated by the orange vertical line gets relatively large at around 0.07-0.1 Å\(^{-1}\) on the x-axis. This is roughly the region where the signal from both detectors is merged and artificial spikes can be anticipated. The blue line illustrates the fitted result from the MAXE analysis. The fitting works reasonably well up until 0.03 Å. A q-value of 0.03 translates roughly to a diameter of 25 nm using the relation \(q \approx 2\pi/d\), where \(d\) is the diameter of the scattering particle. MAXE assumes scattering from spherical and polydispersed scatterers that are distributed randomly inside the material.
Figure 4.19: The orange line represents the measured absolute macroscopic cross section plotted against the scattering length vector $q$. The blue line shows the MAXE fit with a calculated background of 0.0428.

Figure 4.20 illustrates the volume fraction from all scatterers plotted against the diameter. Here, the results have been obtained from 4 measurement points on a strip of the pristine sample of the as-received material. The illustration shows a peak at around 40 nm diameter and a more broadened peak with a mean diameter at around 160 nm. Some variation of the volume fraction from about $0.65 \times 10^{-5}$ to about $0.9 \times 10^{-5}$ could be observed at around 40 nm. The data reveals less scattering from larger scatterers and less variation in their changes in volume fraction. For the subsequent analysis, these four measurements from the pristine sample were averaged.
Figure 4.20: Volume fraction of all scatterers \( (V(D)) \) for four different positions measured on a 1 mm thick strip of the as-received material.

In Figure 4.21, the averaged distribution from all four points of the pristine sample is plotted together with the volume fraction from the reference measurement points of the crept CHS as well as from the PCHS sample. The reference measurements were taken from the grip area, where both samples have experienced a temperature of 550°C but with almost negligible stress. A shift of the narrow peak could be observed. While the peak of the CHS sample was found to be at approximately 30 nm the peak of the pristine or as-received sample shifted to about 40 nm. The data also reveals an increase of the narrow peak from \( 0.8 \times 10^{-5} \) for the pristine sample, to about \( 1.8 \times 10^{-5} \) on the PCHS and \( 2.4 \times 10^{-5} \) on the CHS sample. There is also an increase in \( V(D) \) on the broader peak for larger particles from around \( 0.3 \times 10^{-5} \) to about \( 0.6 \times 10^{-5} \). For the peak at 40 nm, the data reveals a clear increase in scattering just from the time the two samples have spent in the furnace. While the PCHS sample was under temperature for just about 24 h the time the CHS sample spent in the furnace was approximately 851 h. However, this
has to be taken with a caveat since the q-resolution within this area is relatively poor, see standard deviation in Figure 4.19, and there is the deviation between the fitting result and the absolute intensity from scattering in this q-region. The signal for large scattering particles, however reveals no further increase between CHS and PCHS sample. Only an increase of $V(D)$ between the pristine sample and the CHS and PCHS samples could be observed. All volume fractions are flattening out towards a particle diameter larger than 330 nm.

Figure 4.21: Comparison of volume fraction of all scatterers, $V(D)$, for as-received material and reference measurement positions in the CHS and PCHS samples.

Figure 4.22 a) and b) illustrate the SANS results for each side of the creep fractured cylindrical hourglass specimen. In Figure 4.22 a), points from the left side of the creep failed sample are shown, whereas Figure 4.22 b) depicts the size distribution of the other side of the creep failed sample. Again, two distinct distributions could be observed in both pictures. There is a narrow peak at a diameter at around 25 nm and a broader more flattened peak with a mean diameter of around 180 nm. A significant increase of the fractional size
distribution of scatterers towards the failure position and hence towards higher stresses and creep strain can be observed. This trend is similar for both peaks and hence for all sizes of scattering particles. The volume fraction of scattering particles reaches around $3 \times 10^{-5}$ for particles with a mean diameter of around 25 nm and $1.5 \times 10^{-5}$ for larger particles with a mean diameter of around 180 nm. Each side of the creep fractured hourglass sample reveals a significant stress dependency, i.e. pronounced variation in volume fraction, for large scatterers with a mean diameter $>50$ nm. The variation of scattering from small scatterers with a mean diameter $<50$ nm for the three largest stress levels on the other hand, is relatively similar. Here, a stress dependency of the volume fraction can be observed but is a lot less notable. This is illustrated in Figure 4.22 a) and b).

By subtracting the scattering intensity $V(D)$ of the reference points (at the grip end) and dividing with the neutron contrast factor $49.71 \times 10^{-28} \text{ m}^{-4}$, the fractional size distribution of cavities $C(D)$ can be determined, which is illustrated in Figure 4.23 (a) and (b). Similar to Figure 4.22, two distributions could be observed. A distribution of cavities with a
diameter of less than 50 nm and a broader distribution of cavities with diameters ranging from 50 nm to about 400 nm. The results of Figure 4.23 reveal that the volume fraction of cavities $C(D)$ is about two orders of magnitude smaller than the volume fraction of all scatterers $V(D)$ (illustrated in Figure 4.22), which indicates that precipitates contribute to the majority of scattering in the as-received material. With an increase in stress, the amount of scattering from cavities increases significantly. Similar to $V(D)$, a significant stress dependency of the volume fraction of cavities $C(D)$ can be observed.

![Figure 4.23: SANS results illustrating the fractional size distribution of cavities $C(D)$ for the a) right and b) left side of the CHS sample.](image)

Figure 4.24 illustrates the volume fraction of scattering from particles $V(D)$ as well as from cavities $C(D)$, in the PCHS sample. Data points for a stress range from about 439.51 MPa to about 205.47 MPa have been measured. Contrary to the results of the crept CHS sample, see Figure 4.23, the measurements showed no clear correlation of volume fraction and stress. The $C(D)$ function of larger cavities ($>50$ nm) in the PCHS sample does not reveal any distinct peaks and no systematic increase with stress can be noticed. The signal of cavities on the PCHS sample, illustrated in Figure 4.24, is relatively hairy and it is not
trivial to find a clear value for the mean cavity diameter. The data for the small as well as large population of apparent cavities shows no clear trend either. Figure 4.23, which illustrates the CHS sample, reveals a maximum in the volume fraction of large cavities $C(D)$ at $2.5 \times 10^{-7}$ whereas only $0.6 \times 10^{-7}$ is reached for the highest stress position for the PCHS sample. Since the volume fraction of cavities in the PCHS sample shows no correlation with stress and plastic strain and is much smaller compared to the volume fraction of the CHS sample, see Figure 4.23, it is believed that Figure 4.24 shows the amount of cavities or voids that are pre-existent in the material.

![Graphs](image)

**Figure 4.24:** SANS results illustrating the fractional size distribution of a) all scatterers $V(D)$ and b) cavities $C(D)$ for the PCHS sample.

The mean diameter for both, the CHS and PCHS sample have been calculated based on the maximum entropy fitting results for the SANS signals. To calculate the number density of the cavities, a mean diameter has to be determined, see Section 3.5.12. The size distribution was divided into two populations. The population of small cavities ($<50$ nm) was fitted to a Gaussian function. The mean diameter for the broader distribution of large scatterers (50 to 400 nm) was calculated by a method of moments and direct summation,
similar to what has been suggested by Jazaeri et al. [165]. The results are illustrated in Figure 4.25. For smaller cavities (< 50 nm) the mean diameter was found to be around 30 nm. There is very little deviation around this value and it was found to be relatively similar for both sides of the CHS sample as well as for the PCHS sample. The mean diameter of larger cavities (> 50 nm) on the crept sample CHS were found to be larger than the mean diameters of the PCHS sample. The mean diameter for large cavities on the CHS sample varies between 153-188 nm whereas the mean diameter of large cavities from the PCHS sample varies in the range from 151 nm to 170 nm, see Table 4.4. Both populations of cavities, did not reveal any correlation of stress with mean diameter.

![Graph showing mean diameter of cavities](image)

**Figure 4.25:** The mean diameter of cavities obtained from both halves of the CHS and the PCHS sample and plotted for two populations of cavities measured over a range of 200-450 MPa.

An overview over the calculated mean diameters of the CHS and PCHS samples is given in Table 4.4.
Table 4.4: Estimation of the mean diameter of creep cavities as a function of stress in the CHS and PCHS sample calculated from the MAXE results.

<table>
<thead>
<tr>
<th>Sample Pos.</th>
<th>Applied Stress (MPa)</th>
<th>Average cavity diameter small in nm</th>
<th>Average cavity diameter large in nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-1</td>
<td>421</td>
<td>22</td>
<td>174</td>
</tr>
<tr>
<td>CHS-2</td>
<td>398</td>
<td>22</td>
<td>173</td>
</tr>
<tr>
<td>CHS-3</td>
<td>372</td>
<td>23</td>
<td>179</td>
</tr>
<tr>
<td>CHS-4</td>
<td>348</td>
<td>24</td>
<td>181</td>
</tr>
<tr>
<td>CHS-5</td>
<td>327</td>
<td>26</td>
<td>188</td>
</tr>
<tr>
<td>CHS-6</td>
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<td>CHS-7</td>
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<td>CHS-8</td>
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</tr>
<tr>
<td>CHS-9</td>
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</tr>
<tr>
<td>CHS-10</td>
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</tr>
<tr>
<td>CHS-11</td>
<td>398</td>
<td>22</td>
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</tr>
<tr>
<td>CHS-12</td>
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<td>25</td>
<td>185</td>
</tr>
<tr>
<td>CHS-13</td>
<td>359</td>
<td>26</td>
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</tr>
<tr>
<td>CHS-14</td>
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</tr>
<tr>
<td>CHS-15</td>
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<tr>
<td>CHS-16</td>
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<td>175</td>
</tr>
<tr>
<td>PCHS-1</td>
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</tr>
<tr>
<td>PCHS-2</td>
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<td>30</td>
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</tr>
<tr>
<td>PCHS-3</td>
<td>416</td>
<td>29</td>
<td>161</td>
</tr>
<tr>
<td>PCHS-4</td>
<td>396</td>
<td>31</td>
<td>153</td>
</tr>
<tr>
<td>PCHS-5</td>
<td>365</td>
<td>31</td>
<td>157</td>
</tr>
<tr>
<td>PCHS-6</td>
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<td>28</td>
<td>170</td>
</tr>
<tr>
<td>PCHS-7</td>
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<tr>
<td>PCHS-8</td>
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<tr>
<td>PCHS-9</td>
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<td>31</td>
<td>153</td>
</tr>
<tr>
<td>PCHS-11</td>
<td>203</td>
<td>31</td>
<td>159</td>
</tr>
</tbody>
</table>

Figure 4.26 illustrates the integrated volume fraction $C(D)$ of cavities for the two different samples. The fractional size distribution of cavities is summed up for all cavities within a size range of 5-400 nm diameter. The green and red dots illustrate the integrated volume fraction from both halves of the failed crept CHS sample, see Figure 4.23. The black dots depict the fractional size distribution of cavities on the PCHS sample. An interesting
outcome could be observed. In a stress range from 200 to about 350 MPa, the integrated volume fraction of cavities stays relatively constant and similar in the CHS as well as in the PCHS sample. Beyond 350 MPa, a clear trend becomes detectable. While the integrated fractional size distribution of cavities is rising exponentially towards high stresses on the CHS sample, it stays relatively constant over the whole stress range for the PCHS sample with prior hot loading strain but without any further creep strain. The integrated volume fraction stays at around $1 \times 10^{-4}$ for the PCHS sample and rises to about $4 \times 10^{-4}$ for the CHS sample.

![Figure 4.26](image.png)

**Figure 4.26:** Comparison of the integrals of $C(D)$ for all creep cavities from the CHS and the PCHS specimen over a broad stress range of 200-450 MPa. The integrals were calculated for all cavities within 5-400 nm diameter size.

Figure 4.27 and 4.28 illustrate the number density for smaller cavities (<50 nm) and larger cavities (>50 nm) respectively. From both Figures, a comparison between the number density of the PCHS sample and the CHS sample can be drawn. An increase of the number density for the crept sample towards higher stress regions can be observed. Again
and similar to the integrated volume fraction, the number density stays relatively constant for the PCHS specimen. The number density for the small population of cavities in the CHS sample is rising with an increase in stress but seems to flatten out towards stresses above 400 MPa. On the other hand, the number density of the population of large cavities in the CHS sample rises exponentially with an increase in stress. For both samples (CHS and PCHS), a higher number density could be calculated for small-sized creep cavities compared to large cavities (>50 nm). Furthermore, the number density of small creep cavities on the CHS sample is significantly higher than for the PCHS sample. A significant increase in volume fraction (see Figure 4.26) as well as in number density (see Figure 4.28) of creep cavitation damage, especially for larger cavities (>50 nm) as a function of stress can be observed.

**Figure 4.27:** The number density \(N(D)\) of small cavities (<50 nm diameter) in the CHS and PCHS samples, measured by SANS.
Figure 4.28: The number density ($N(D)$) of large cavities (>50nm diameter) in the CHS and PCHS samples, measured by SANS.

4.5.2.1 Guinier Analysis

Another parameter of interest is the gyration radius of the scattering objects. A Guinier analysis was performed to get an estimation about the average cavity size and to compare with the results obtained using the maximum entropy analysis. Guinier showed that for randomly distributed monosized scatterers in a sample the scattering intensity follows:

$$I = I_0 e^{x p(-R_g^2 q^2/3)}, \quad (4.4)$$

where, $I_0$ is the scattering intensity at $q = 0$ and $R_g$ is the gyration radius of the spherical cavities [184]. If the cavities are assumed to be spherical the radius can be calculated according to:

$$R = \sqrt{5/3} R_g. \quad (4.5)$$
Since it is known that cavities are not monodispersed in the Type 316H stainless steel samples [164,165], the utility where the Guinier analysis can be applied is limited to a very narrow q-section close to zero. The Guinier area is illustrated in Figure 4.29, where the logarithmic scattering intensity is plotted against the squared scattering vector $q^2$. Since a regression line can only be fitted over the illustrated section, it can be concluded that the scatterers have to be polydispersed.

![Figure 4.29: Illustration of the Guinier region for position CHS-9 with a true stress of 430 MPa on the CHS sample. The logarithmic intensity is plotted over the squared scattering vector $q^2$. The red dots illustrate the scattering intensity of cavities.](image)

A regressional fit for one stress position (CHS 09) in the CHS sample is illustrated in Figure 4.30. The fitting was applied to sample points where the signal from the reference point was subtracted beforehand. So, the data only shows the scattering contribution from creep cavities. Using this fitting procedure, estimates about the gyration radius could be made. The calculated values for the cavity diameter can be found in Table 4.5. No clear trend in cavity growth towards higher stresses can be observed. The calculated diameter
scatters around an average diameter of around 162 nm. If compared with the population of large cavities, see the MAXE analysis in Table 4.4, the Guinier approach shows similar but slightly smaller results for the mean diameter of cavities larger than 50 nm. Since the Guinier analysis is a relatively simple approach to analyse a sample, it is constrained in its informative value especially for samples with polydispersed scatterers. It gives an estimate about the mean of the largest scatterers. However, this analysis discards the small peak measured with the MAXE algorithm. It is a rather simple approach to get a quick analysis of the largest scatterers within a material.

**Figure 4.30:** Best fit associated with one position CHS-9 with a true stress of 430.37 MPa on the CHS sample. The fit was done in the Guinier region as marked as in Figure 4.29.
Table 4.5: Estimation of diameter of creep cavities as a function of stress in the CHS sample using the Guinier approach.

<table>
<thead>
<tr>
<th>Sample Pos.</th>
<th>Applied Stress (MPa)</th>
<th>Aver. cavity diameter (nm)</th>
<th>Sample Pos.</th>
<th>Applied Stress (MPa)</th>
<th>Aver. cavity diameter (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-1</td>
<td>421</td>
<td>160</td>
<td>CHS-9</td>
<td>430</td>
<td>168</td>
</tr>
<tr>
<td>CHS-2</td>
<td>398</td>
<td>156</td>
<td>CHS-10</td>
<td>410</td>
<td>160</td>
</tr>
<tr>
<td>CHS-3</td>
<td>372</td>
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<td>CHS-5</td>
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<tr>
<td>CHS-8</td>
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<td>164</td>
<td>CHS-16</td>
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<td>170</td>
</tr>
</tbody>
</table>

4.6 Microstructural analysis

An analysis of the microstructure was carried out using a Zeiss Sigma 500 Field Emission scanning electron microscope. All measurements were performed with an accelerating voltage of 10kV and an aperture size of 30µm in the high definition backscattered diffraction (HDBSD) mode. Positions around the fracture surface have been examined to verify findings concluded from the SANS analysis with regard to the cavity size. The fracture micrographs shown in Figure 4.31 were taken from the CHS sample at longitudinal areas close to the creep fracture surface where large microcracks could be observed. The majority of those cracks tends to form perpendicular to the stress axis and were found to be intergranular. Figure 4.32 and 4.33 show some qualitative assessments of cavities and their dimensions at a distance of 1.5 mm away from the fracture surface (i.e. close to CHS-09). In both pictures, the dimensions of a selection of cavities are highlighted. They confirm the SANS analysis highlighting cavities with diameters of less than 175 nm. In Figure 4.32 and Figure 4.33, even smaller cavities can be found, but the resolution gets worse. Cavities with diameters of 27 nm and 35 nm could be observed and are highlighted in Figure 4.32 which also shows that the cavities surrounded by second phase particles. Figure 4.33 depicts a line of very small cavities below a microcrack of the size of several µm. The line of cavities follows the grain boundary. Another example of cavities is shown in Figure 4.34.
Here, cavities are found along grain boundaries at two triple junctions. Small micro cracks are visible at grain boundaries with angles of 45° and 90° towards the loading direction. Again, the cavities are surrounded by second phase particles, believed to be $M_{23}C_6$ carbides. The cavities exhibit an elongated shape, presumably caused due to the large plastic strain from loading and right before creep failure. In the middle of the grain boundary transverse to the loading direction, a line of scattered cavities is illustrated. Towards the triple points larger voids and cavities that are coalesced already can be found. Figure 4.35 illustrates some examples of intergranular as well as intragranular cavities surrounded by second phase particles at a distance of 5 mm away from the failure position.

Figure 4.31: HDBSD image showing the fracture region of the CHS creep ruptured sample tested at a maximum engineering stress of 360 MPa at a temperature of 550°C.
Figure 4.32: HDBSD image showing the size and facetted structure of an isolated cavity. The image is taken from the crept CHS with a true stress of 430 MPa sample at a 1.5 mm distance from the failure position.

Figure 4.33
HDBSD image showing the size and facetted structure of some cavities. The image is taken from the crept CHS with a true stress of 430 MPa sample at a 1.5 mm distance from the failure position.
Figure 4.34: HDBSD image showing the size and facettted structure of some cavities. The image is taken from the crept CHS with a true stress of 430 MPa sample at a 1.5 mm distance from the failure position.

Figure 4.35: HDBSD image showing examples of intragranular as well as intergranular cavities surrounded by second phase paricles captured at a distance of 5 mm away from the creep fracture surface.
4.7 Discussion

For this work, a novel hourglass test specimen was designed for high temperature creep testing optimised for the non-contact strain measurement technique 3D-DIC. To improve the design of the hourglass sample, an FE-analysis has been performed to determine the stress and strain distribution across the hourglass gauge section for different settings of length, diameter and radii. The specimen had to be designed in a way to allow a quantification of creep cavities along the hourglass gauge section in accordance with the restrictions set by the SANS analysis and the specifications of the creep furnace. A radius of 100 mm and an hourglass gauge section length of 28 mm were found to give good results. The design, illustrated in Figure 4.6, enabled the measurement and analysis of a variation of creep strain curves based on a single sample. Using 3D-DIC as a technique to measure multiple spatially resolved creep strain curves on just one specimen is beneficial for industries where the amount of testing material as well as time in general is limited. 3D-DIC also ensures that the creep response for different stresses is gathered using the same loading as well as the same temperature which is always challenging in conducting multiple creep tests on different rigs. Over the hourglass gauge length, 50 creep curves could be spatially resolved. This provided useful information on the creep strain as well as creep strain rate as a function of stress and time (see Figure 4.11 and 4.16). Furthermore, it was possible to monitor the transverse strain and therefore the reduction of area as a function of position across the hourglass gauge section which allowed a calculation of true stress for each of the creep strain curves.

The creep test on the novel hourglass sample design was conducted using a mid-length engineering stress of 360 MPa which induced significant plastic strain. The plastic strain from loading varied as a function of diameter along the hourglass gauge section and did have an effect on creep properties such as the creep ductility, the time to rupture or creep strain rate. The variation in plastic deformation acts like different rates of hardening along the hourglass gauge length which leads to a position-dependent variation in creep strength.
A higher loading strain is likely to increase the number of dislocations within the material. Subsequently, they become pinned by the precipitation of carbides during the early stages of the test and hence the material creeps more slowly initially as the dislocation movement is impeded. To distinguish damage caused by plasticity from purely creep effects, tests with an applied stress below yield need to be carried out to eliminate the damage effects of plastic strain from loading. The 3D-DIC revealed an increase in true stress over the whole creep time but in particular in the tertiary regime. It is still unclear if the increase in true stress is caused by an increase in creep damage or softening or even necking of the material. While necking is caused by an instability in the material causing high localised strain from material inhomogeneities or defects, softening is a rather slow time dependent process due changes in the microstructure. The increase in true stress, right before creep failure can lead to cavity growth caused by plastic strain rather than creep strain.

The results show that SANS is a really powerful technique to obtain volumetric information about the cavitation damage in metals as it provides a relatively high sensitivity which makes it particularly useful to track the early stages of cavity evolution. SANS does not require sample preparation in terms of repeated polishing as well as etching like other techniques. It provides information on the total number of cavities and precipitates. By using the Maximum Entropy data analysis method, it is possible to obtain information for highly polydisperse solutions of scatterers. However, there are some limitations for SANS that need to be pointed out. The application of SANS in complex alloys is quite challenging since it is difficult to distinguish scattering due to cavitation from scattering due to second phase particles. This is only possible if the assumption is made that the precipitation characteristics are not changing under stress and creep strain conditions for the treatment times inside a furnace. For a distinct determination of cavity damage, it has to be assumed that creep induced changes in the morphology of carbides do not contribute significantly to the signal from SANS. This approach has been recommended by Boeuf et al. as well as Jazaeri et al. in their SANS analysis on cavity damage in Type 304 and 316H stainless steel [153, 165]. By subtracting the signal of the reference points from
each measurement point, the effects of carbides on the SANS outcome could be minimised. However, it has been shown by Swindeman et al. that the dislocation structure can be changed due to creep strain and this offers more nucleation sites and accelerated growth for second phase particles [157]. Since this becomes more and more pronounced at times far beyond 1000 hours, it was neglected in this analysis. Nevertheless, all results need to be carefully verified by complementary techniques such as SEM or TEM.

The mean diameter for the small population of cavities on both halves of the failed crept sample CHS and for the PCHS sample is around 22-31 nm (see Figure 4.25). This is comparable to what has been found for Type 316H stainless steel by Jazaeri et al. [164, 165]. The crept cylindrical hourglass sample CHS, as well as the PCHS sample, show a similar mean diameter for the small population of cavities. The standard deviation was 1.5 nm for the CHS sample and 2.2 nm for the PCHS sample. Very minor cavity growth can be observed for the small population of cavities over the whole stress range. Following Raj’s nucleation theory, there is a theoretical minimum of stable sized cavities that can be calculated using \( r_{\text{min}} = \frac{2\gamma}{\sigma} \), where \( \gamma \) is the surface tension and \( \sigma \) is the applied stress. For Type 316H stainless steel, a surface tension in the order of 2.41 can be presumed [207]. As the applied stress in the gauge section of the hourglass test specimen varies, the minimum stable size of cavities would be different too. For positions with higher stress the minimum creep cavity ought to be smaller than for lower stress regions. At the highest stress level of around 420 MPa on the crept sample, a minimum stable cavity with a diameter of just 22 nm can be estimated. This is comparable to what has been observed in the SANS analysis, see Table 4.4. On the other hand, at a stress of just 200 MPa the minimum stable sized cavity diameter is expected to be as large as 42 nm. This variation could not be confirmed with the outcome of the SANS results. The mean diameter of cavities was found to be relatively stable around a mean diameter of 21-31 nm over the whole stress spectrum from around 430 MPa to about 200 MPa. However, it has to be noted that the q-resolution from the SANS2D instrument is relatively coarse and a large scatter is affecting the scanning results, especially in the high-q region. As illustrated in Figure 4.19 there are
large fluctuations in SANS2D at high q values at around 0.04 Å⁻¹ to about 0.1 Å⁻¹ which is corresponding with a cavity size range of around 15 nm to 60 nm. Furthermore, this is the area where there was a significant deviation between the scanning result and fitting results from the maximum entropy algorithm. Thus the level of uncertainty in the small scatterers results is high and only tentative conclusions can be drawn.

Two populations of scatterers were observed in all samples. The data always shows a narrow peak with a mean diameter of around 22-31 nm and a wider and flattened peak with a mean diameter of around 151-188 nm, see Table 4.4. Similar size distributions were reported earlier in Type 304 and 316H stainless steel samples after creep deformation [208, 209]. In the PCHS sample, which has not seen any creep deformation, the data reveals a relatively small volume fraction of cavities (see Figure 4.26). The idea to test the PCHS sample, was to investigate if plastic deformation causes cavities that might be pre-existent in the material to grow. The rate of plastic prestrain, induced at a temperature of 550°C, does not seem to change the amount in volume fraction of cavities which is constant over a relatively wide stress range of 203-440 MPa. No cavity growth could be observed as a consequence of prior plastic prestrain and the data does not suggest that plastic strain, applied at a temperature of 550°C, induces cavitation in the as-received material. In the CHS sample, there is evidence that the volume fraction for both distributions of the cavitational damage \( C(D) \) increases systematically with a rise in stress and creep strain. This suggests that cavitational damage is strongly related to stress and creep strain and occurs during the whole creep life triggered by their continuous nucleation. The number density of small cavities (<50 nm) on the crept sample shows a different behaviour than for large cavities (>50 nm) (see Figure 4.27 and Figure 4.28). While the number density is increasing towards higher stresses for the large population of cavities (>50 nm), it seems to flatten out towards higher stresses for cavities with a diameter smaller than 50 nm. There is the possibility that coalescence occurs which explains a stalling increase of the number density towards high stress regions (see Figure 4.27). As pointed out by Weertman et al., there is a likely possibility that SANS underestimates the volume fraction, as well as the average
size the for large populations of cavities [210]. If cavities grow larger than the minimum resolvable q range from the SANS2D instrument settings, the average size as well as the volume fraction are undervalued. The Guinier analysis is very limited and constrained in its informative value. However, it gives some trend that can be compared with other fitting approaches such as that done by the MAXE analysis. The results of the Guinier analysis confirmed the estimation of the mean diameter for large cavities, around 170 nm, made by the MAXE analysis, see Table 4.5. However, Deschamps et al. pointed out that for samples with a very polydisperse solution of scatterers the Guinier analysis can lead to wrong assumptions for the gyrational radius. An increase in polydispersity might lead to a shift in the calculation of the gyrational radius [211]. The SEM results, which showed evidence of creep cavitational damage, confirmed the findings from the SANS analysis which predicted cavities in a diameter range between 20 nm and 400 nm. Cavities with diameters of only 27 nm could be observed using SEM, see Figure 4.32. Both findings are in agreement with previous work on other 316H stainless steel material [164, 165].

4.8 Summary

The digital image correlation has been applied successfully to measure the spatial variation in creep strain along a cylindrical sample with a novel hourglass design. The following observations from the experiment were made:

1. A novel hourglass test specimen has been designed and creep tested at an elevated temperature of 550°C. The strain on the surface was measured using the non-contact measurement technique 3D-DIC.

2. 3D-DIC has been proven to be a suitable technique to measure the creep strain for long duration tests at high temperatures for complex shaped geometries. The local variations of plastic as well as creep strain at elevated temperatures could be spatially resolved.

3. 3D-DIC could be used to measure the creep strain both in longitudinal and transversal
directions. This enabled monitoring of the true stress along the hourglass gauge section for the whole creep process.

4. 3D-DIC enables measurements of spatially resolved creep strains. In the presented analysis 50 creep curves could be measured along the hourglass gauge section. The analysis showed that to obtain the creep parameters for a large stress variation only a small sample is required.

5. Two populations of creep cavities were observed at all positions along the gauge length of the cylindrical hourglass sample, tested at a maximum engineering stress of 360 MPa at a temperature of 550°C. A small population with a diameter range of 10-50 nm and another population of cavities with a diameter between 50-400 nm were observed.

6. An analysis using qualitative SEM images supported the SANS observations in terms of the dimensions of creep cavities. Spherical and facetted cavities with diameters between 27 nm and 200 nm could be found in the material. The cavities were found to be associated with second phase particles.

7. The volume fraction as well as the number density of creep cavities increased as a function of stress and creep strain along the hourglass gauge section of the CHS specimen. However, the number density seemed to flatten out towards points close to the fracture surface with very high stresses.

8. The volume fraction in the exclusive plastically deformed PCHS sample the volume fraction of creep cavities stayed constant over a stress range from 200 MPa to about 450 MPa.
5 Effects Of Prior Plastic Strain On Subsequent Creep

5.1 Introduction

The effect of plastic deformation on the subsequent creep behaviour is of theoretical and practical interest since many engineering components operating at high temperature enter service in prestrained conditions. Cold work can alter the creep deformation and creep damage properties of materials significantly and the phenomena needs to be characterised and understood to assess a component’s life time [15,74]. All the results presented here are based upon tests performed on the as-received material, as introduced in Chapter 4. The material has been distributed by EDF Energy to carry out fundamental studies and to gain more knowledge on its creep properties. The low yield strength of this material of around 150 MPa, as shown in Table 4.3 at a temperature of 550°C means that creep tests are often carried out using an initial stress above yield or at an even higher temperature to reduce the run time. This facilitates completion of creep tests within a feasible time period, which usually means months at a university or years at a dedicated laboratory. In these cases, it is not possible to distinguish between the amount of damage that has been induced due to plastic strain from loading and the time-dependent creep deformation. Material parameters such as the creep ductility and creep strain rate are significantly affected by the amount of prior cold work but its effects on cavity nucleation and growth are poorly understood.

In this chapter, the findings of experimental works investigating the effect of prior plastic strain in tension applied at room temperature on the subsequent creep properties such as rupture time, creep ductility and creep strain rate are presented. The main aim was to investigate how creep properties are altered and whether the influence of prior plastic strain on subsequent creep is preferable in the as-received material. Each creep test was carried out up until creep failure at a temperature of 550°C and at a maximum start of test stress of 305 MPa. The purpose of those studies is to better understand creep induced
cavitation as a failure mechanism in reactor components made from Type 316H austenitic stainless steel.

5.2 Experimental Methods

5.2.1 Overview

In order to measure the effects of prior plastic strain on the subsequent creep properties, a series of five creep tests were conducted on samples with an hourglass design, identical to the design described in Section 4.3.2 and illustrated in Figure 5.1. The possibility to measure multiple creep strain curves from just one sample has been utilised. In this series of tests however, cylindrical uniaxial samples were prestrained in tension to several amounts preceding the creep tests. These uniaxial cylindrical specimens were extracted from the as-received Type 316H stainless steel. After applying prior plastic strain on cylindrical specimens with an initial diameter of 9 mm and a length of 85 mm, an hourglass shape was machined so that each specimen had a unique and even amount of prior plastic strain. Each creep test was carried out on these cylindrical hourglass specimens up until creep failure at a temperature of 550°C and at a maximum start of test stress of 305 MPa. The procedure is illustrated in Figure 5.2.

Figure 5.1: Sample design of the an hourglass creep test specimen machined after prestraining to different amount of prior plastic strain.

Since the final hourglass design as shown in Figure 5.1 has a minimum diameter of 8 mm it had to be ensured that after prior plastic strain the reduction in diameter would be less
than 1 mm from the initial diameter (9 mm). A plastic Poisson’s ratio of 0.5 was assumed, since inducing plastic strain of up to 16% far exceeds the elastic ratio of the as-received material. For smaller strains an elastic Poisson’s ratio of 0.3 for Type 316H stainless steel can be assumed. The tensile tests were carried out on an Instron-8862 screw driven slow strain test machine with a calibrated 100 kN capacity load cell, as described in Section 3.2. The strain at room temperature was measured using an Instron 2630-100 series clip-on extensometer. The gauge length of the extensometer amounted to 12.5 mm. All tensile tests were conducted at a constant strain rate of $1.5 \times 10^{-4} \text{ s}^{-1}$. The applied stresses to achieve plastic true strains of 4%, 8%, 12% and 16% at room temperature are listed in Table 5.1.

![Uniaxial Specimen](image)

**Figure 5.2:** Procedure of inducing prior plastic strain on cylindrical specimens before machining an hourglass gauge section. a) The uniaxial cylindrical specimen with an initial diameter of 9 mm is depicted. b) Prior plastic was induced at room temperature to several amounts. c) After prior plastic strain was induced an hourglass shape was machined.
EFFECTS OF PRIOR PLASTIC STRAIN ON SUBSEQUENT CREEP

5. EFFECTS OF PRIOR PLASTIC STRAIN ON SUBSEQUENT CREEP

Table 5.1: True stress and strain properties of the 4%, 8%, 12% and 16% materials prestrained in tension at room temperature.

<table>
<thead>
<tr>
<th>Sample</th>
<th>True Strain in %</th>
<th>True Stress in MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-04</td>
<td>4</td>
<td>343</td>
</tr>
<tr>
<td>CHS-08</td>
<td>8</td>
<td>420</td>
</tr>
<tr>
<td>CHS-12</td>
<td>12</td>
<td>490</td>
</tr>
<tr>
<td>CHS-16</td>
<td>16</td>
<td>550</td>
</tr>
</tbody>
</table>

5.3 Results

5.3.1 Tensile test at 550°C

Prior to all tensile tests, a thermal heat treatment has been performed on all specimens in order to relieve any residual stresses trapped in the as-received material during its production (as described in Section 4.2) and to dissolve precipitates inside the material. For the solution annealing the specimens were kept inside a furnace at a temperature of 1060°C for one hour with a subsequent air cooling of the samples.

To establish an equal stress range in each specimen for creep testing, a tensile test at 550°C has been conducted to specify a maximum load that could be applied without causing any further strain from loading prior to the creep tests in three of the five test specimens. From a slow strain tensile test conducted at a temperature of 550°C, a 0.2% proof stress of around 150 MPa was monitored. This implies a significant strain from loading if one was to carry out conventional creep tests. A long term creep test below this stress and at a temperature of 550°C would result in a creep failure time of around 100,000 hours [212].

Inducing prior plastic strain in the as-received material has been chosen because it diminishes any further plastic strain from loading at relatively large stresses and therefore, allows to separate the damage generated by plastic deformation from damage caused due to creep deformation. The slow strain tensile test at high temperature was measured using an Instron 2632-054 high temperature extensometer. It has a gauge length of 12.5 mm.
and it is possible to measure strain in tension up to 20%. It is attached to the specimen with a combination of a ceramic and a simple spring. The ceramic legs of the extensometer have grooves to ensure a tight grip for the whole test. The test at 550°C showed evidence of dynamic strain ageing could. This has been discussed in Section 4.3.3. Figure 5.3 illustrates the results from the tensile test conducted at 550°C, where the true stress is plotted against the true strain. This gives an indication for the maximum true stress that can be applied for the samples with 8%, 12% and 16% plastic prior strain to stay below the yielding point. The yellow and red coloured arrowed lines in Figure 5.3 indicate the stress range that is covered with one creep test using the hourglass design. The engineering stress spans from 305 MPa to 165 MPa at the start of test. The red arrows highlight samples where the applied loading induced further plastic strain from loading, whereas the yellow lines indicate samples where no further plastic strain from loading is induced.

![Figure 5.3: True stress versus true strain from a slow strain rate test conducted at 550°C. The arrows are indicating the covered stress range within the hourglass section for the 8%, 12% and 16% samples. It highlights that samples with prior plastic prestrain to these degrees (yellow arrows) won’t experience any further plasticity from loading. The samples prestrained to 0% and 4% (red arrows) experience further plastic strain from loading.](image-url)
5.3.2 FEA and 3D-DIC results from loading

An elastic-plastic FE analysis was performed based on the tensile test data at a temperature of 550°C. Supported by that result, a load of 8.6 kN was chosen. This resulted in a start of test stress of about 305 MPa in the mid section of the hourglass gauge length. Figure 5.4 illustrates the FE-simulation of the stress distribution in tensile direction after loading which reveals a maximum true stress of 321.8 MPa at the beginning of the creep test. The stress and strain distribution was calculated using geometric non-linearity settings since a change in geometry was to be expected. For the FE-simulation a half width of the specimen was modelled and axis-symmetric effects applied to save computational time.

**Figure 5.4:** FE-simulation showing the S22 stress distribution in the y-direction by applying a total load of 8.6 kN corresponding to a maximum engineering stress of 305 MPa. The plot on the right side illustrates the S22 stress in the centreline of the specimen.

Figure 5.5 illustrates the maximum equivalent or von Mises plastic strain of 7.84% in
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the middle of the hourglass section after applying the load. These results were used to
determine the amount of load that could be applied which ensured that for samples with
8% or even higher pretension no further plastic strain from loading would be induced.

**Figure 5.5:** FE-simulation showing the equivalent plastic strain distribution in the y-direction
by applying a total load of 8.6 kN corresponding to a maximum engineering stress of 305 MPa.
The plot on the right side illustrates the equivalent plastic strain in the centreline of the specimen.

Figure 5.6 depicts the strain from loading from the 3D-DIC analysis along the hourglass
gauge section. It shows the strain that is caused due to loading at a temperature of 550°C
and at from a loading of 8.6 kN. The maximum strain value in the mid hourglass gauge
section of CHS-0 with 0% prior plastic strain amounts to 7.4%. This result illustrates its
close proximity to the FE-Result as shown in Figure 5.5. Around 16 mm away from the
centre position the strain has dropped to about 1%. Figure 5.6 illustrates that strain in
about 26 mm (-10 mm until about +16 mm) along the hourglass gauge section of the creep
test specimen can be monitored by 3D-DIC.
5.3.3 Creep test results

The creep strain was measured using 3D-DIC, as described in Section 3.3. All creep specimens were placed in the middle of the furnace to provide the best view on the hourglass gauge length. After switching on the furnace, a constant temperature of 550°C was kept for at least 24 h before any load was applied to ensure a uniform temperature distribution within the specimens and to stabilise the temperature within the furnace. Each creep specimen was subjected to a constant load that provided a start of test stress of 305 MPa in the middle of the hourglass gauge section. The samples were illuminated using a fibre optic light bundle linked to a strobe flash and triggered by one of the DSLR’s. Images were taken in a regular interval of 5 min for the first 100 hours to capture the primary

Figure 5.6: Total centreline strain in CHS-0 hourglass specimens with 0% prior plastic strain introduced by applying a total load of 8.6 kN corresponding to a maximum engineering stress of 305MPa. In addition, the results of the FE-simulation showing the equivalent plastic strain distribution in the y-direction.
creep regime more closely. After that, the frequency was lowered to 30 min for each camera. The acquired images were analysed using the DIC software package Davis [175]. Thermal currents within the furnace introduced a significant variability in the acquired 3D-DIC data. Therefore, careful data processing of the obtained deformation images was necessary to extract the strain data, as described in more detail in Section 3.3.5. As mentioned earlier in Section 5.2.1, creep tests were performed on 0%, 4%, 8%, 12% and 16% prestrained specimens at 550°C. The accumulation of creep strain in the CHS-0 specimen, without any further prior plastic strain, is illustrated in Figure 5.7. Figure 5.8 depicts the accumulation of creep strain for CHS-4 with 4%, CHS-8 with 8%, CHS-12 with 12% and CHS-16 with 16% prior plastic strain. In both Figures, 5.7 and 5.8, 3D surface plots are illustrating the variation in measured creep strain along the hourglass gauge length as a function of test duration for an applied stress of 305 MPa at 550°C. The creep strain could be monitored longitudinally along the hourglass gauge section over a section of not less than 22 mm in all creep tested specimens. The mid section of each hourglass test specimen was always set to 0 mm in each Figure.
Figure 5.7: 3D surface plots from 3D-DIC data analysis showing the variation in measured creep strain for CHS-0, without further prior plastic strain, along the hourglass gauge length as a function of test duration for an applied stress of 305 MPa at 550°C at mid-length of the specimen.

Figure 5.7 and 5.8 reveal that the region with the highest creep strain is always at the centre of the hourglass test specimen, which is labelled as 0 mm. Figure 5.8 illustrates that the accumulation of creep strain for the CHS-16 sample is concentrated to the mid section of the specimen with an accumulation in creep strain that is relatively narrow across the centre of the mid-section compared to CHS-4, CHS-8 and CHS-12 which exhibit a more broadened peak.
Figure 5.8: 3D surface plots from 3D-DIC data showing the variation in measured creep strain for a) CHS-4 with 4%, b) CHS-8 with 8%, c) CHS-12 with 12% and d) CHS-16 with 16% prior plastic strain as a function of test duration. The samples all crept until fracture at a temperature of 550°C and a net engineering stress of 305 MPa at mid-length of the specimen.

A summary of all the creep test results can be found in Table 5.2:
Table 5.2: Creep properties from series of tests conducted on specimens with a varying amount of prior plastic strain. The creep tests were conducted at a temperature of 550°C and a start of test stress of 305 MPa.

<table>
<thead>
<tr>
<th>Sample</th>
<th>CHS-0</th>
<th>CHS-4</th>
<th>CHS-8</th>
<th>CHS-12</th>
<th>CHS-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat treatment</td>
<td>SA-Furnace cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
</tr>
<tr>
<td>Prestrain at room temperature (%)</td>
<td>0</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td>16</td>
</tr>
<tr>
<td>Hot plastic strain from loading (%)</td>
<td>7.57</td>
<td>4.55</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>True stress (MPa)</td>
<td>324</td>
<td>314</td>
<td>305</td>
<td>305</td>
<td>305</td>
</tr>
<tr>
<td>Eng. stress (MPa)</td>
<td>305</td>
<td>305</td>
<td>305</td>
<td>305</td>
<td>305</td>
</tr>
<tr>
<td>$t_{cr-0.2%}$ (h)</td>
<td>165</td>
<td>723</td>
<td>1241</td>
<td>1259</td>
<td>1873</td>
</tr>
<tr>
<td>$\epsilon_f$ (%)</td>
<td>4.05</td>
<td>2.422</td>
<td>1.6</td>
<td>1.42</td>
<td>0.8</td>
</tr>
<tr>
<td>$t_f$ (h)</td>
<td>1088</td>
<td>1624</td>
<td>2619</td>
<td>2954</td>
<td>3080</td>
</tr>
<tr>
<td>mid-section $\epsilon_{min}$ (h⁻¹)</td>
<td>$2 \times 10^{-6}$</td>
<td>$1.3 \times 10^{-6}$</td>
<td>$2 \times 10^{-7}$</td>
<td>$2.6 \times 10^{-7}$</td>
<td>$2 \times 10^{-7}$</td>
</tr>
</tbody>
</table>

A comparison of the creep behaviour, monitored at mid-length of the hourglass gauge section, for all tested specimens is illustrated in Figure 5.9. Here, the creep strain is plotted against the time for specimens with a varying amount of prior plastic strain. The hot plastic strain from loading which has been monitored for the samples CHS-0 and CHS-04 with 0% and 4% prior plastic strain respectively, has been subtracted in order to compare only creep strain. It is referred to as hot plastic strain from loading because it was applied at a temperature of 550°C as a consequence of the loading procedure right before the start of the creep test. Figure 5.9 illustrates that an increase of prior cold work causes an increase in the time to rupture but a decrease in creep ductility. The creep ductility is around 4% for CHS-0 and reduces to about 0.8% for CHS-16. The creep ductility of CHS-16 reduces to roughly one fifth of the ductility measured without any prior plastic strain. The data reveals that the reduction in creep strain proceeds continuously as a function of prior plastic strain. A reduction in ductility and increase in time to rupture are detectable also for the CHS-0 and CHS-04 samples. In both samples, albeit the prior plastic strain induced at room temperature has not been large enough to fully suppress the
hot plastic strain component from loading at the start of the creep test, an alteration in the creep properties can be detected, as shown in Figure 5.9. The creep behaviour between the samples CHS-08 and CHS-12, with 8% and 12% prior plastic strain respectively, is relatively similar up to a time of around 2200 h. Afterwards, the creep curve of CHS-08 with 8% prior plastic strain accelerates and failure occurs at 2619 h a little earlier than for the CHS-12 sample where failure occurs at 2995 h. The creep ductility however, is relatively similar and reduces from 1.62% to about 1.4% only.

Both, the primary creep strain rate as well as the primary creep strain was significantly reduced with an increase of prior cold work. This is illustrated in Figure 5.10 where the primary creep strain is plotted against time, for the first 300 h of each test. The reduction of primary creep strain was lowered consistently as a function of prior plastic strain. After a
time of 300 h the CHS-0 specimen reaches about 0.22% strain which reduces to just 0.04% for CHS-16. Changes in primary creep strain are most noticeable between CHS-0 and CHS-04, where primary creep reduces from around 0.23% to about 0.08%. The reduction in primary creep becomes less significant between the CHS-08, CHS-12 and CHS-16 tests. Here, the amount of creep strain varies from about 0.06% to 0.04% after a time of 300 h. The data reveals that with an increase in prior plastic strain changes in the primary creep behaviour are getting less significant.

**Figure 5.10:** Constant stress creep curves during creep testing up to 200 h, from the mid-length hourglass section, at an engineering stress of 305 MPa for the as-received material prestrained at room temperature by 4,8,12 and 16% prior to creep testing at 550°C.
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Figure 5.11: Variation of the time to rupture in creep tests carried out at $550^\circ C$ for specimens prestrained to 4,8,12 and 16%. a) shows the time to rupture against prior plastic strain induced at room temperature and b) shows the time to rupture against the total strain (prestrain + hot strain from loading + creep strain).

Figure 5.11 a) illustrates the increase of the creep rupture time as a function of prior plastic strain. The time to failure increases by a factor of about 3 if one compares CHS-0 and CHS-16. The change in time to rupture seems to increase relatively steep from 0% to 8% in prior plastic strain and seems to flatten for larger amounts in prior plastic strain at a time of around 3,000 hours. Figure 5.11 a) clearly shows that the gradient of time to fracture decreases. Figure 5.11 b) on the other hand, depicts the change in rupture time as a function of total strain, which is the sum of prestrain at room temperature, creep strain and hot loading strain in CHS-0 and CHS-04. It was found that there is a significant dip in the time to rupture as a function of total strain. CHS-08 has the lowest total strain with 9.6% but shows a significantly larger time to rupture compared to CHS-0 and CHS-04 with a total strain of about 11% and about 11.5% respectively. Figure 5.11 b) illustrates quite clearly that a significant increase in time to rupture correlates only with prior plastic strain induced at room temperature. CHS-04, which exhibits prior plastic strain induced at room temperature and hot plastic strain from loading, has a smaller total strain compared to CHS-0 but shows a significant increase in rupture time. CHS-0 has a larger total strain,
compared to CHS-4 and CHS-8, but exhibits the minimum value in time to rupture for the series of prestrained creep tested specimens. The data reveal that prior plastic strain has to be induced at room temperature in order to improve the creep time to failure.

Figure 5.12 shows the creep strain rates taken from the mid hourglass section of the 0%, 4%, 8%, 12% and 16% specimens. The creep strain rates have been obtained by numerical differentiation of creep strain and time. A continuous decrease in the minimum creep strain rate for all of the specimens could be detected. Both samples, CHS-0 and CHS-4, reach a minimum creep strain rate at a time of around 300-350 h and proceed into tertiary creep subsequently which becomes dominant as soon after the minimum creep strain rate is reached. On the other hand, Figure 5.12 illustrates that the creep strain rates for 8%, 12% as well as 16% exhibit a different behaviour. All of the three samples, all without any further hot plastic strain from loading, reach a minimum creep strain rate soon after start of the test that is followed by a time period with an almost constant creep strain rate that seems to increase with an increasing amount of prior plastic strain.
Figure 5.12: Variation of creep strain rates with time for five creep test specimens with a varying amount of prior plastic strain with an engineering stress of 305 MPa tested at a temperature of 550°C.

Only at the very end, an accelerated increase in creep strain rate sets in which indicates the tertiary creep regime. Furthermore, it can be observed that the time in tertiary creep seems to decline with a rise in prior plastic strain. The time spent in the secondary creep regime becomes significantly shorter for samples with lesser amount of prior plastic strain.

5.3.4 Comparison of creep strain at positions with same true stress

As shown in Table 5.2, a significant amount of hot loading strain for the samples CHS-0 and CHS-04 with 0% and 4% prior plastic strain was monitored which resulted in a change in true stress. This leads to a change in hardening effects inducing an altered creep behaviour. It is therefore more meaningful to compare positions in terms of their creep
properties at the same true stress. Using 3D-DIC it is possible to monitor the lateral changes in diameter for each area across the hourglass gauge section and to calculate the true stress at each time interval. The accumulation of creep strain over time for all tested samples at positions with true stresses of 305 MPa, 290 MPa, 270 MPa and 250 MPa is illustrated in Figure 5.13. The same trend that has been found in Figure 5.12 is still detectable. An increase of prior plastic strain has a creep resistance effect which can be observed for all four stress positions in all of the samples as it reduces the creep strain rate. The ductility as well as the time to rupture can’t be compared in Figure 5.13 since most curves exhibit positions where failure has not occured yet. For positions with true stresses of 270 MPa and at 250 MPa the data becomes significantly noisier because of a very low creep strain rate. For example, at 250 MPa the creep strain for CHS-8, CHS-12 and CHS-16 stayed almost below 0.1% for the entire creep life. With a reduction in creep strain the signal to noise ratio becomes worse, which can be observed in the scatter of the creep strain data. Figure 5.13 illustrates that CHS-0 and CHS-4 reached tertiary creep for all four stress positions. CHS-08, CHS-12 and CHS-16 stayed within secondary creep for low stresses of 270 and 250 MPa. CHS-16 only reached tertiary creep for the 305 MPa true stress position.
Figure 5.13: Constant stress creep curves taken from four different true stress positions with a) 305 MPa, b) 290 MPa, c) 270 MPa and d) 250 MPa for fully solution treated 316H stainless steel prestrained at room temperature to 0, 4, 8, 12 and 16% prior to creep testing at 550°C. The true stress was calculated at the end of each test.
The same effect of creep resistance from prior plastic strain can also be found in the primary creep behaviour which is illustrated in Figure 5.14. Figure 5.14 is similar to Figure 5.13 as it shows the same stress positions but provides a closer view of the early stage of creep, primary creep, up to 200 hours. A continuous decrease in primary creep can be observed for all four stress positions as a function of prior plastic strain. CHS-0 and CHS-4 showed a significant distinction in their primary creep performance, even though the prior plastic strain induced at room temperature was not large enough to eliminate all hot loading strain prior to creep. For positions with a true stress of 305 MPa and 290 MPa (see Figure 5.14), CHS-8 and CHS-12 exhibited a relatively similar primary creep behaviour. CHS-0 always showed the largest amount of accumulated primary creep while CHS-16 exhibited the lowest. CHS-16 accumulated a creep strain well below 0.05% for each stress position after a time of 200 hours. The data revealed, that the amount of primary creep decreases as a function of prior plastic work.
Figure 5.14: Constant stress primary creep curves over the first 200 h. The creep curves are taken from four different true stress positions with a) 305 MPa, b) 290 MPa, c) 270 MPa and d) 250 MPa for fully solution treated 316H stainless steel prestrained at room temperature to 0, 4, 8, 12 and 16% prior to creep testing at 550°C.
Whilst the trend and general creep deformation behaviour can be read from the 3D-DIC data, the primary creep results (Figure 5.14) show significant fluctuations owing to poor 3D-DIC resolution at low levels of strain. Figure 5.15 illustrates the variation in the creep strain rate for all tested specimens for four different true stress positions. The pattern is relatively similar to Figure 5.12. The creep strain rate reduces with the amount of stress and prior plastic strain. All samples illustrate primary creep at the beginning of each test where the creep strain rate decays. After the minimum creep strain rate is reached for CHS-0 and CHS-4 a tertiary creep behaviour followed subsequently without substantial time spent in the secondary creep regime. This again is markedly different to the overall creep strain rate of the samples with prior cold work and no further hot loading strain such as CHS-8, CHS-12, CHS-16. These curves showed a decaying primary creep after which the strain rate reaches a minimum. This minimum is followed by an accelerating creep strain rate that reaches an almost constant creep strain rate for a prolonged period of time. The constant strain rate period seems to extend as a function of prior plastic work. For CHS-08, CHS-12 and CHS-16 the tertiary creep regime becomes apparent only at the end of each test. For lower stresses of 270 MPa and 250 MPa it seems as if another minimum or dip in the creep strain rate is forming. It is a behaviour that has been observed for Type 316H SS earlier by Kubo et al. [213] and can be explained by an ever evolving microstructure of the material. It has to be noted that the overall creep strain rates for this batch of material are very low in general and a significant amount of noise had to be dealt with. This becomes more and more apparent for the overall creep strain rate at low stresses where the signal to noise ratio becomes a lot worse.
Figure 5.15: Variation of creep strain rates as a function of time from four different true stress positions with a) 305 MPa, b) 290 MPa, c) 270 MPa and d) 250 MPa for fully solution treated 316H stainless steel prestrained at room temperature to 0, 4, 8, 12 and 16% prior to creep testing at 550°C.
5.3.5 Comparison of secondary creep strain rate as a function of prior plastic strain

As mentioned by Webster et al., the Norton Law describes the steady state creeping behaviour as:

\[ \dot{\epsilon}_{sec} = A\sigma^n \]  

(5.1)

where \( n \) is the power-law stress exponent, \( A \) is a material constant that is dependent on the temperature and \( \sigma \) is the applied stress [50]. For CHS-8, CHS-12 and CHS-16 with 8%, 12% and 16% prior plastic strain respectively, the secondary creep properties have been determined by fitting the creep curves to a Norton Power Law. For each sample the slope and intercept of the regression line has been determined at a creep time of 1200 h.

\[ \text{Figure 5.16: The creep strain rate plotted against time for specimens prestrained to 8, 12 and 16\% prior to creep testing at a temperature of 550}^\circ\text{C.} \]
The variation of the creep strain rate for each of the three specimens prestrained to 8, 12 and 16% prior to creep testing at a temperature of 550°C, is illustrated in Figure 5.16. The area that shows a nearly constant creep strain rate is highlighted as a green box. For this section, it was feasible to fit the creep strain rates from the three samples with 8%, 12% and 16% prior plastic strain, over an engineering stress range from 250 MPa to 305 MPa to the Norton Power Law. CHS-0 and CHS-4 were excluded from the analysis since they experienced hot plastic strain from loading and there is rapid transition from primary to tertiary creep. Figure 5.16 highlights that the period of the constant creep strain is not at the minimum creep strain rate which arises at a time between 200 h and 400 h for all three tested specimens. As a consequence, the creep strain rate over the constant strain rate period was fitted against the Norton Power Law.

![Figure 5.17: Results from fitting to Norton Power Law for samples with 8%, 12% and 16% prestrained prior to creep testing at a temperature of 550°C. The fitting was applied at a time of 1200 h where the creep strain rate showed a constant behaviour.](image-url)
As illustrated in Figure 5.17, the creep strain rate along the hourglass gauge section was plotted against the true stress and fitted using a linear regression that minimises the residual sum of squares between the data points in the dataset and data points predicted by the linear approximation. For the analysis, data points with a true stress between 250 MPa and 305 MPa were used. The results of the fitting procedure are listed in Table 5.3 and are illustrated in Figure 5.17. Each dot in Figure 5.17 represents a stress position along the hourglass gauge section where the creep strain rate has been monitored at a time of 1200 h. It shows a steady increase of the creep strain rate as a function of stress. Figure 5.17 compares the three fitted functions. It highlights the differences in creep strain rate as a function of stress and prior plastic strain. It could be found that the amount of prior plastic strain has a decreasing effect on the creep strain rate. CHS-16 shows the lowest constant creep strain rate while CHS-08 exhibits the highest however, only for stress positions larger than 300 MPa. For a stress range between 260 MPa and 280 MPa CHS-08 and CHS-12 show a very similar progress of the creep strain rate as a function of stress. The slope of CHS-08 however is steeper and the creep strain rate rises quicker for larger stresses.

Table 5.3: The parameter from the Norton Power Law fitting procedure for CHS-08, CHS-12 and CHS-16 with 8, 12 and 16% plastic strain prior to creep testing at a temperature of 550°C.

<table>
<thead>
<tr>
<th>Sample</th>
<th>A (MPa$^{-n}h^{-1}$)</th>
<th>n</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-08</td>
<td>4.65 × 10$^{-37}$</td>
<td>12.37</td>
</tr>
<tr>
<td>CHS-12</td>
<td>3.49 × 10$^{-33}$</td>
<td>10.8</td>
</tr>
<tr>
<td>CHS-16</td>
<td>1.9 × 10$^{-34}$</td>
<td>11.16</td>
</tr>
</tbody>
</table>

5.3.6 Results of the stress modified ductility exhaustion model

The creep ductility of Type 316H stainless steel varies because of many mechanisms taking action on the microstructural level. Above a certain temperature $T_c$, damage accumulation can be the result of plastic hole growth, diffusion controlled cavity growth or constrained cavity growth as shown by [126, 132, 135]. An upper shelf ductility failure can be the
consequence of very high inelastic strain rates where cavity growth is driven by plastic instability resulting in transgranular fracture. At lower inelastic strain rates a transition to lower ductility is observed. The cavity growth here, is believed to be controlled by diffusional controlled mechanisms. Thirdly, there is a lower shelf regime where the inelastic strain at failure is seemingly independent from the strain rate but shows a dependence from stress. This stress dependent region is controlled by a constrained cavity growth which is limited by the creep of the surrounding grains [135]. Both, the transition as well as the stress dependent region are associated with intergranular fracture. A more detailed description of the failure modes has been presented in Section 2.7. The three different regions leading to different failure mechanisms can be described using the following set of combined equations:

\[
\bar{\epsilon}_f = \text{MAX} \left\{ \begin{array}{l}
\text{MIN} \left[ \epsilon_u \exp(\frac{1}{2} - \frac{3\sigma_m}{2\sigma_e}), A_1 e^{\frac{\Delta G_{AC}}{RT} (\dot{\epsilon}_{in})^{n_1}} (\sigma_1)^{-m_1} \frac{\sigma_e}{\sigma_1} \exp(\frac{1}{2} - \frac{3\sigma_m}{2\sigma_e}) \right] \\
\text{MIN} \left[ \epsilon_u \exp(\frac{1}{2} - \frac{3\sigma_m}{2\sigma_e}), A_2 e^{\frac{\Delta G_{AC}}{RT} (\sigma_1)^{-m_2} \frac{\sigma_e}{\sigma_1} \exp(\frac{1}{2} - \frac{3\sigma_m}{2\sigma_e})} \right]
\end{array} \right.
\]

The set of equations, earlier described in Section 2.7, which are the basis of the stress modified ductility exhaustion model (SMDE) can be used to calculate changes in ductility as a function of stress and strain rate [12]. To determine the damage one has to measure the instantaneous true inelastic stain rate as well as the true stress from available creep curves. 3D-DIC provides suitable data for determining the damage, using the SMDE model, because it allows a measurement of strain in the transverse direction. Therefore it is possible to determine the true stress over the whole time of the experiment. Equation 5.2 can be simplified since any triaxiality of stress can be neglected which results in:

\[
\bar{\epsilon}_f = \text{MAX} \left\{ \begin{array}{l}
\text{MIN} \left[ \epsilon_u, A_1 e^{\frac{\Delta G_{AC}}{RT} (\dot{\epsilon}_{in})^{n_1}} (\sigma_1)^{-m_1} \right] \\
\text{MIN} \left[ \epsilon_u, A_2 e^{\frac{\Delta G_{AC}}{RT} (\sigma_1)^{-m_2}} \right]
\end{array} \right.
\]
Based on the 3D-DIC data, the stress and strain rate at each time interval, throughout each creep test can be monitored. The set of equations (5.3) of the SMDE model selects which mechanism applies under the corresponding conditions of stress and inelastic strain. The SMDE model predicts that creep tests fails on the upper shelf, if the ductility, predicted by both stress dependent and transition region, are greater than the upper shelf ductility. The stress dependent region sets a lower limit of the applicability to the model which describes the transition region [12]. This lower limit is necessary since the equations for the transition region yield zero as the inelastic strain rate reduces. A ductility that reduces to zero, however is not realistic. After taking the logs of the uniaxial form of Equation 5.3, one can determine the creep damage using the ductility exhaustion model according to the following equation:

$$d_{SM}^c = \sum \left\{ \frac{\delta \epsilon_c}{\epsilon_{\text{exp}}} \left\{ \begin{array}{l}
\text{MIN}[\ln(\epsilon_u), \ln(A_1) + \frac{\Delta G_{AC}}{RT} + (n_1)\ln(\dot{\epsilon}_{in}) + (-m_1)\ln(\sigma_1)] \\
\text{MIN}[\ln(\epsilon_u), \ln(A_2) + \frac{\Delta G_{AC}}{RT} + (-m_2)(\sigma_1)]
\end{array} \right\} \right\}. $$

(5.4)

where $\delta \epsilon_c$ is the increment of inelastic strain between at each time interval. Equation 5.4 was used to evaluate creep damage for all creep tests of the as-received AISI type 316H stainless steel using material coefficients provided by EDF Energy, as listed in Table 5.4 [140]. These parameters are derived from constant load creep tests as well creep fatigue data on parent material tested at a temperature of $550^\circ C$. 
Table 5.4: Parameter for the SMDE model for Type 316H stainless steel parent material at a temperature of 550°C.

<table>
<thead>
<tr>
<th>Region</th>
<th>Parameter</th>
<th>Parent Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper shelf</td>
<td>$\ln(\epsilon_{upper})$</td>
<td>-0.8158</td>
</tr>
<tr>
<td></td>
<td>$\Delta G_{US}/R$</td>
<td>0</td>
</tr>
<tr>
<td>Stress dependent</td>
<td>$\ln(A_1)$</td>
<td>11.0192</td>
</tr>
<tr>
<td>region</td>
<td>$n_1$</td>
<td>0.497</td>
</tr>
<tr>
<td></td>
<td>$m_1$</td>
<td>-2.512</td>
</tr>
<tr>
<td></td>
<td>$\Delta G_1/R$</td>
<td>5266</td>
</tr>
<tr>
<td>Transition region</td>
<td>$\ln(A_2)$</td>
<td>18.29</td>
</tr>
<tr>
<td></td>
<td>$m_2$</td>
<td>-0.6363</td>
</tr>
<tr>
<td></td>
<td>$\Delta G_2/R$</td>
<td>-13600</td>
</tr>
</tbody>
</table>

Using the parameter presented in Table 5.4, the SMDE model can be depicted, as shown in Figure 5.18 where three different damage regions can be distinguished. Here, and for all following analysis, the upper shelf region is coloured green, whereas the transition region and stress dependent region are coloured red and blue, respectively. The upper shelf creep ductility was calculated to be 44.3% for the parent material. The illustration shows that the red transition region is intersected by the upper shelf region for large stresses and strain rates. On the lower end, the transition region is intersected by the stress dependent region. The SMDE model uses the total inelastic strain rather than just the creep strain. This takes the massive differences between strain rates during loading and creep into account.
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Figure 5.18: EDF Energy stress modified ductility exhaustion (SMDE) model that describes the creep ductility dependence on stress as well as inelastic strain rate and distinguishes three regions accounting for three different creep failure mechanisms in Type 316H stainless steel.

In this section, the damage calculation based on the SMDE model for the test series of prestrained creep specimens, as described in Section 5.3.3 is presented. The samples CHS-0 as well as CHS-4 with 0% and 4% prestrain respectively, exhibited hot loading strain. Subsequently both started in the upper shelf region where damage can be calculated based on the constant creep ductility. The exact strain rate due to loading has not been captured but strain happens almost instantaneous after applying each dead weight. Within the creep period, this changes. The changes of the different failure modes that a creep curve passes through can be retraced from Figure 5.19 where the creep curve of the mid-length of the hourglass gauge section of CHS-0 is plotted. Each failure region is colour coded based on the colours presented for each region in Figure 5.18, except for the turquoise which is changed to a dark blue colour tone. The creep strain is plotted against time normalised
with respect to creep failure time and presented as $t/t_f$. Based on the curve from mid-length of the hourglass specimen 90% creep life is spent in the stress dependent region of the model where creep cavity growth is constrained by the surrounding grains. For a short period from approximately 90% to about 98% the creep curve passes the transition region where cavity growth is controlled by diffusional processes. At larger creep strain rates towards the end of the test, at a creep strain of around 2.5%, upper shelf conditions prevail where the model indicates that damage is driven by plastic instability as well as necking. The model therefore predicts that for the mid hourglass region of CHS-0, damage, in terms of ductility exhaustion, is affected by plastic hole growth as well as creep driven processes such as diffusional cavity growth.

**Figure 5.19:** Creep curve from mid-length of the hourglass section of the CHS-0 test with SMDE model regions highlighted. The mechanisms associated with the coloured regions are detailed in 5.18.

A similar plot for the mid hourglass section of sample CHS-12 with 12% prior plastic strain, is illustrated in Figure 5.20. The model indicates that the predominant damage mechanism is again stress dependent until about 98% creep life when transition behaviour is reached.
during tertiary creep. The final creep strain rate, as a combination with stress, does not lead to creep damage on the upper shelf region. However, the last recorded data points might not be obtained immediately before failure since the 3D-DIC images were taken at a certain frequency and the creep failure process at the end was captured only every 30 min. The creep ductility close to creep failure could be in fact slightly higher.

![Figure 5.20](image)

**Figure 5.20:** Creep curve from centre of the hourglass section of the CHS-12 test with sections highlighted of each regime. The blue zone is the damage from the stress dependent region and red highlights the transition region.

The damage calculation based on Equation 5.4 for the mid-section of CHS-0 is illustrated in Figure 5.21. At each time interval, the damage was calculated using a ductility exhaustion approach where the prevalent true stress and inelastic strain rate determined the associated region of the SMDE model. In Figure 5.21, the total inelastic strain is plotted as the orange line and refers to the left side of the y-axis. The calculated creep damage refers to the right side of the y-axis. Both, total inelastic strain as well as creep damage are plotted versus creep time. The accumulation of damage based on each region could be obtained and was calculated to be around 0.33. A damage level of less than one seems confusing at
first but this has to be taken with respect to the overall creep ductility data of the parent Type 316H stainless steel material. It highlights the very low creep ductility of this cast of as-received material compared to a whole range of creep tests of the parent Type 316H stainless steel tested at the same temperature of 550°C.

![Inelastic Strain and Damage Calculation](image)

**Figure 5.21:** Total inelastic strain as well as damage calculation for the creep curve of the mid hourglass section of the CHS-0 sample without prior plastic strain tested at 305MPa and at a temperature of 550°C. The black dashed line represents the total damage which is the summation of damage over each region.

The total damage for CHS-0, as shown in Figure 5.21 consists of damage from four regions and is visualised as a pie chart in Figure 5.22. By adding the damage from loading 50.5% and from the upper shelf region from creep 8.7%, the damage caused by plastic hole growth accounts for 59.2% damage. The damage from the constrained cavity growth of the stress dependent region is 23.4% and 17.4% from the transition region where cavity growth is believed to be caused by diffusional processes. It is evident that, the majority of damage is driven by plasticity rather than creep processes.
Figure 5.22: Pie chart illustrating the amount of damage for each of the regions of the SMDE model in the CHS-0 sample.

The damage calculation, described for CHS-0 in this section was performed for each of the series of prestrained creep test specimens. Whilst the approach was relatively straightforward for the samples without any pre-tension a modification had to be taken into account for the samples CHS-04, CHS-08, CHS-12 and CHS-16 where prior plastic strain was induced at room temperature. This modified approach was applied for the loading process simply by changing the upper shelf ductility at 550°C, calculated to be 44.3%, to a strain at failure measured at room temperature of 55%. The total inelastic strain together with the total damage illustrated over the creep curve and as a pie chart are presented in Figure 5.23 for CHS-04 with 4%, Figure 5.24 for CHS-08 with 8%, Figure 5.25 for CHS-12 with 12% and in Figure 5.26 for CHS-16 with 16% prior plastic strain. It can be found that the amount of prior plastic strain reduces the amount of damage from the transition region. While CHS-04 showed 9.9% for the transition region this reduced to 0% for the CHS-16 specimen. Furthermore, it could be found that by comparing CHS-08, CHS-12 and CHS-16, the samples without any further plastic strain from loading, the total damage (illustrated as the black dashed lines in Figure 5.24, 5.25 and 5.26) increased as a function of prior cold work. While the damage of CHS-08 is around 0.24 this increases to about 0.36 for CHS-16. For CHS-16 however, it has to be noted that most of the damage comes from prior cold work rather than creep. The pie chart in Figure 5.26 shows a damage
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contribution of 85.7% from the pre-tension and only 14.3% from creep. The mid section of CHS-16 has not reached the transition region over the whole creep period and stayed in the stress dependent region all along. The percentage of damage from loading is continuously increasing as a function of prior plastic strain. While the damage from loading is 50.5% for CHS-0 this increases to 85.7% for CHS-16 with 16% pre-tension. As the damage from loading is increasing the damage from the two other regions is decreasing continuously. While CHS-08 shows a damage of 27.3% for the stress dependent region this reduces to 21.0% for CHS-12 and 14.3% for CHS-16.

Figure 5.23: a) Total inelastic strain as well as damage calculation for the creep curve of the mid hourglass section of the CHS-04 sample with 4% prior plastic strain tested at 305 MPa and at a temperature of 550°C. b) Pie chart illustrating the amount of damage for each of the regions of the SMDE model in the CHS-4 sample.
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Figure 5.24: a) Total inelastic strain as well as damage calculation for the creep curve of the mid hourglass section of the CHS-08 sample with 8% prior plastic strain tested at 305 MPa and at a temperature of 550°C. b) Pie chart illustrating the amount of damage for each of the regions of the SMDE model in the CHS-08 sample.

Figure 5.25: a) Total inelastic strain as well as damage calculation for the creep curve of the mid hourglass section of the CHS-12 sample with 12% prior plastic strain tested at 305 MPa and at a temperature of 550°C. b) Pie chart illustrating the amount of damage for each of the regions of the SMDE model in the CHS-12 sample.
Figure 5.26: a) Total inelastic strain as well as damage calculation for the creep curve of the mid hourglass section of the CHS-16 sample with 16% prior plastic strain tested at 305 MPa and at a temperature of 550°C. b) Pie chart illustrating the amount of damage for each of the regions of the SMDE model in the CHS-16 sample.

The calculated damage based on the SMDE model for all of the samples exhibited a damage fraction significantly less than 1. This implies that the ductility of the material tested was much lower than the properties embedded in the SMDE model. The results of all five tested specimens including 0, 4, 8, 12 and 16% prestrained prior to creep and tested at 550°C are listed in Table 5.5.

Table 5.5: Damage calculations based on the stress modified ductility exhaustion model for specimens with 0, 4, 8, 12 and 16% prior plastic strain creep tested at a maximum engineering stress of 305 MPa and at a temperature of 550°C.
5.4 Discussion

The creep process for Type 316H stainless steel is affected by the density and arrangements of dislocations within the steel matrix. The dislocations act as a barrier to plastic flow and subsequently the material becomes more creep resistant with a higher plastic pre-strain [75,214]. As shown in Figure 5.17 a significant stress dependence for the creep strain rate could be observed which indicates a predominant dislocation creep mechanism [31,49].

It is well known that in most metals and alloys a certain number of mobile dislocations exist, free to glide if a certain stress and temperature level is reached [19, 46]. With the process of strain hardening these mobile dislocations become pinned which leads to a lowered creep strain rate at the end of primary and beginning of secondary creep. Cold work induced at room temperature prior to the creep tests, depletes some of the mobile dislocations [15,215]. This subsequently leads to a slowed primary creep strain rate. It was mentioned by Wilshire and Willis that creep curves of most metals and alloys hardly go into a steady state creep after reaching the end of primary. Most of the time when a minimum creep rate is reached this is followed by tertiary creep to which an acceleration of creep strain rate is characteristic [15]. In the current study, this behaviour could be observed for specimens without or with less amount of prior plastic strain, such as CHS-0 and CHS-04, see Figure 5.12. According to findings by Wilshire and Willis as well as by Davies et al. creep parameters such as the time to rupture as well as the minimum creep strain rate are changing only when the prior plastic strain is large enough to eliminate any further hot loading strain prior to the creep experiments. This was not observed for the series of creep tests of the as-received material [15,214]. The loading strain in the mid section of the hourglass gauge length was around 7.57% for CHS-0 (without prior plastic strain) and about 4.55% for CHS-04 (with 4% prior plastic strain). Both specimens however, despite having significant plastic strain from loading showed differences in their creep properties, as illustrated in Figure 5.11 as well as in Figure 5.13. Both, the creep strain as well as the time to rupture are continuously changing for all conducted tests unbiased by the amount.
of initial hot plastic strain from loading. The prior plastic strain has also a decreasing effect on the primary creep strain as well as the primary creep strain rate. As highlighted in Figure 5.14, the primary creep strain is lowered and becomes significantly smaller as a function of prior plastic strain. Also, the amount of primary creep after a time of 200 hours for CHS-16 with 16% prior plastic strain is less than a fifth compared to CHS-0 without any prior plastic strain. The time to reach the minimum creep strain rate was, however, relatively similar for the mid-hourglass stress position in all of the samples and was observed at times between 200 h and 400 h. The period of tertiary creep was found to become less dominant with more and more induced prior cold work.

The creep curves of the Type 316H stainless steel samples with prior plastic strain exhibited a somewhat anomalous behaviour which has been reported by Morris et al. for 316 stainless steel [31]. The creep rate reduced promptly after loading to a very low creep rate, as can be seen in the dip in Figure 5.15. This period was followed by a constant creep strain rate. The period of constant creep strain rate was dependent on the amount of prior plastic strain. The higher the amount of plastic cold work, the longer the duration time of the constant period. The creep strain rate of this constant period was fitted to a Norton Power law and correlated with the amount of prior cold work as illustrated in Figure 5.16. Prior plastic strain reduces the creep strain rate here continuously. Subsequently, a period of an accelerated creep rate commenced shortly before creep failure of the specimen. For the mid-hourglass section it could be observed that the minimum creep strain rate was dependent on the amount of prior plastic strain so that samples with large amount of pretension exhibited a slower creep strain rate. The higher prior plastic strain is likely to increase the number of dislocations within the material. They become pinned by the precipitation of carbides during the early stages of the test and hence the material creeps more slowly initially as the dislocations movement is impeded. After the minimum in creep strain rate is reached an annihilation of dislocations could explain that the creep strain accelerates again. When creep carbides coarsen, the dislocations become more mobile as the pinning of dislocations by coarser carbides is less effective. This is accompanied with an increase in
creep strain rate. Figure 5.15 indicates a renewed primary creep behaviour for low stresses of 270 MPa and 250 MPa at times of around 2000 h and observed only for specimens with significant prior plastic strains such as 12\% and 16\%. This anomalous behaviour has been reported earlier by Kubo et al. [213] for long duration creep tests on SUS 316 stainless steel at time scales in the order of several thousand hours. The observations of the "renewed" primary behaviour, especially in the lowest stress tests can be explained similarly due to the precipitation of carbides on dislocations causing a reduction in the creep rate, followed by coarsening and subsequent unpinning of dislocations accompanied by an increase in creep rate.

The overall damage calculated, using the SMDE model, confirms the low creep ductility of the as-received material in comparison to the database upon which the SMDE model parameters were based. The total damage for all of the tested specimen never reached a value above 0.37, see Table 5.5. From all tested specimens, CHS-0, the sample without any further induced prior plastic strain, was the only one with a sufficiently large enough creep strain rate to reach the upper shelf region shortly before rupture as illustrated in Figure 5.19. However this has to be taken with a caveat since the final creep strain rate could only be estimated due to the limitations of using 3D-DIC. The last logged strain based on an image is changing for each 3D-DIC test and depends on the frequency of images being taken at the final part of the creep test. These frequencies in which pictures were shot varied for each of the tests from 10 min to about 30 minutes. Therefore, some of the changes in creep strain immediately before the end of creep life might not have been captured using the 3D-DIC analysis.

The macroscopic damage calculations based on the SMDE model suggest that most of the damage for load controlled creep tests are due to plastic hole growth either caused by loading before creep or due to an accelerated creep strain rate in the tertiary creep regime, see Table 5.5. With an accumulation in prior plastic strain, the contribution of damage from the upper shelf region where cavity growth is caused by plastic hole growth was found to be increasing. The amount of prior plastic strain had a diminishing effect on
the significance of both, the stress dependent region as well as the transition region with diffusional creep cavity growth. Nevertheless, and as illustrated in Figure 5.19 and 5.20 for the creep test specimens CHS-0 and CHS-12, respectively, the model predicts that the creep test specimens spent almost their whole life in either the stress dependent or transition region where the SMDE model suggests that cavity nucleation occurs [12]. However, the contribution of creep cavity nucleation and growth becomes less significant to the actual failure if prior plastic strain is induced. For CHS-16 with 16% prior plastic strain, the contribution from creep processes to the total damage is only 14.3%. According to the SMDE model, the main contribution of damage is a consequence of prior plastic strain and is pre-existent at the start of creep life. The tracking of the creep failure times, as illustrated in Figure 5.11 showed a clear distinction between the rupture times as a function of prior plastic strain induced at room temperature and total inelastic strain. While the latter does not show a clear dependency and even shows a dip in the time to failure, the former clearly exhibits a continuous increase of time to failure as a function of cold induced plastic strain. The SMDE model however does not reflect these significant changes regarding the temperature at which the prior plastic strain is induced. According to the SMDE model plastic strain induced at room temperature does not cause as much damage since the total inelastic strain at failure is larger compared to it at an elevated temperature of 550°C. The results however show the opposite with a significantly lowered creep ductility and an increased time to rupture if the strain is induced at room temperature. The damage parameter from the parent 316 H material might not reflect the prestrained material very well and a damage model with parameters based on material from the heat affected zone (HAZ) that has a significantly lowered creep ductility and lot of induced prestrain as a consequence of residual stresses might be a better choice.

5.5 Summary

3D-DIC has been applied to measure the time dependent variation in creep strain along an hourglass gauge section of samples with different amounts of prior plastic strain in tension
and the following observations made:

1. Prior plastic strain induced at room temperature increases the creep resistance of the as-received material. A decrease in creep strain rate as well as in creep ductility and an increase in time to failure of the as-received material was observed. Furthermore, the temperature at which the prior plastic strain is induced influences the time to rupture significantly.

2. Prior plastic strain alters the subsequent creep properties such as creep strain rate, creep ductility and time to rupture, even if the pre-tension is not large enough to fully eliminate any further hot plastic strain from loading. This observation is contrary to what has been reported in literature.

3. A stress dependence on the creep strain rate was monitored that indicates that dislocation creep is the dominant mechanism under the prevalent test conditions.

4. Prior plastic strain at room temperature was found to cause an anomalous behaviour on the subsequent creep response. The creep strain rate reached a minimum right after start of test followed by a constant creep strain rate. The length and the creep strain rate of this constant period is a function of the amount of cold work. This behaviour was observed only for samples where the prior plastic strain was large enough to annihilate any further hot plastic strain from loading such as in CHS-08, CHS-12 and CHS-16.

5. The secondary creep coefficients were determined for CHS-08, CHS-12 and CHS-16 with 8%, 12% and 16% prior plastic strain respectively, for the period with an almost constant creep strain rate. CHS-16 the sample with the largest amount of prior plastic strain exhibited the lowest creep strain rate. CHS-08 and CHS-12 showed a similar progress below a stress of 300 MPa. For larger stresses above 300 MPa CHS-08 showed the highest strain rate.

6. Using the stress modified ductility exhaustion model it could be found that most of the damage in Type 316H stainless steel in load controlled creep tests is due
to plastic hole growth and not from diffusion creep processes. With an increase in prior plastic strain the damage was found to be increasing. According to the SMDE model, for the series of prestrained specimens, the main contribution of damage is pre-existent in the material before start of creep.
6 Assessment Of Creep Damage For Prestrained Specimens

6.1 Overview

As described in the previous chapter, pretension from loading prior to creep reduces both, the creep strain rate and the creep ductility and increases the time to rupture of Type 316H stainless steel. As a consequence of creep deformation, ductile alloys such as the as-received material typically fail due to nucleation, growth and coalescence of cavities formed at grain boundaries and in the vicinity of secondary phase particles. The nucleation and formation of these voids, that eventually leads to cracking and failure, is referred to as creep damage. This chapter focuses on a characterisation of creep damage in the as-received material for the set of creep ruptured specimens with different amount of prior plastic strain induced. The creep specimens were tested at a temperature of 550°C, the operational temperature at the AGRs, and a maximum net engineering stress of 305 MPa. The amount of stress was chosen because it does not induce any further plastic strain from loading in three of the samples (CHS-8, CHS-12, CHS-16) in the series of prestrained specimens. Small angle neutron scattering (SANS) experiments, as well as scanning electron microscopy (SEM) studies were used to investigate creep cavitation. The SANS studies’ objective was to quantify creep cavitation damage as a function of prior cold work as well as creep strain and stress. If verified with other techniques such as SEM, SANS can provide quantitative creep damage information for example in terms of cavity size, total cavity volume, total number density, cavity spacing and nucleation rates. The validation of the SANS results for this PhD work is provided using qualitative scanning electron microscopy studies. SEM images were taken from samples prepared with a conventional metallographic procedure of repeated grinding and polishing, as described in Section 3.4.3. Scanning electron microscope imaging was also used to examine cryogenic fractured samples. This technique allows direct observation of grain boundary creep cavities without changing their morphol-
ogy by metallographic (including grinding, polishing, etching) procedures. Furthermore, brittle fracture at cryogenic temperatures reveals the inclination angle of the grain boundary facets as well as information about the size and distribution of cavities. Table 6.1 summarises the conditions and properties of test samples examined and presented in this chapter.

Table 6.1: Creep properties from series of tests conducted on specimen with a varying amount of prior plastic strain. The creep tests were conducted at a temperature of 550°C and a start of test stress of 305 MPa, where \( t_f \) is the creep rupture time, \( t_{cr-0.2\%} \) is the time to reach 0.2% creep strain and \( \epsilon_f \) is the strain at failure.

<table>
<thead>
<tr>
<th>Sample</th>
<th>CHS-0</th>
<th>CHS-4</th>
<th>CHS-8</th>
<th>CHS-12</th>
<th>CHS-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat treatment prior to prestrain and creep</td>
<td>SA-Furnace cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
<td>SA-Air cooled</td>
</tr>
<tr>
<td>Prestrain at room temperature (%)</td>
<td>0</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td>16</td>
</tr>
<tr>
<td>Test temperature (550°C)</td>
<td>550</td>
<td>550</td>
<td>550</td>
<td>550</td>
<td>550</td>
</tr>
<tr>
<td>Hot loading strain (%)</td>
<td>7.57</td>
<td>4.55</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Max. True stress (MPa)</td>
<td>324</td>
<td>314</td>
<td>305</td>
<td>305</td>
<td>305</td>
</tr>
<tr>
<td>Max. Eng. stress (MPa)</td>
<td>305</td>
<td>305</td>
<td>305</td>
<td>305</td>
<td>305</td>
</tr>
<tr>
<td>( t_{cr-0.2%} ) (h)</td>
<td>165</td>
<td>723</td>
<td>1241</td>
<td>1259</td>
<td>1873</td>
</tr>
<tr>
<td>( \epsilon_f ) (%)</td>
<td>4.05</td>
<td>2.422</td>
<td>1.6</td>
<td>1.42</td>
<td>0.8</td>
</tr>
<tr>
<td>( t_f ) (h)</td>
<td>1088</td>
<td>1624</td>
<td>2619</td>
<td>2954</td>
<td>3080</td>
</tr>
<tr>
<td>Stress range examined using SANS (MPa)</td>
<td>-</td>
<td>268.9-313.9</td>
<td>266.1-314.2</td>
<td>267.7-306.1</td>
<td>278.6-307.3</td>
</tr>
</tbody>
</table>

6.2 SANS experimental work

Small angle neutron scattering experiments have been conducted at the High Flux Reactor of the Institut Laue-Langevin using the D11 high-resolution diffractometer and at the ISIS spallation source (Harwell) using the SANS2D instrument. The D11 instrument allows
measurements of scattering objects in a size range from 5 nm up to 1 µm. Three distances between the detector and sample position at 1.5 m, 8 m and 28 m were utilised to measure a scattering $q$ vector in a range from 0.00167 to 0.426 Å$^{-1}$, which translates roughly to a detectability of scatterers between 5 nm and roughly 360 nm in size. While each measurement at distances of 28 m and 8 m was carried out for 30 min, the measurement time required for the closer 1.5 m scan was only 15 min. At D11, a monochromated neutron beam of a particular wavelength is used. A transmission scan, used for analysing the SANS data, was carried out on each sample position for 5 min. A wavelength of 6 Å was used for all measurements because it provides the highest neutron flux and gives the shortest possible measurement times. With a longer wavelength, the $q$-range can be extended, but this decreases the neutron flux. The data reduction was executed using the ILL facility software LAMP (Large Array Manipulation Program) [216]. This is a software package specifically designed to treat data obtained from neutron experiments at the Institut Laue-Langevin. The results from the three detector positions had to be first merged to create a single scattering curve. The reduced data were fitted and analysed using the same procedure as described in Section 3.5.6.

Measurements at the SANS2D instrument were conducted with an incident wavelength range between 4.2 and 12.5 Å resulting in a scattering vector range spanning 0.0157 to 0.42 Å$^{-1}$. Two detector positions were used for each measurement, with the furthest detector placed 12 m behind the sample. This yields a measurement range for heterogeneities from about 5 to 400 nm. For both experiments (D11 and SANS2D), a letter box aperture with dimensions of 2 by 4 mm was used. This resulted in a gauge volume of 8 mm$^3$ for each measurement point. At SANS2D, each sample point was measured for 50 min with one transmission scan of 5 min for each sample. All the following data fitting was done using the Maximum Entropy Method (MAXE) or a Monte Carlo algorithm (MCSAS). For both data fitting approaches, a structure factor for spherical shaped scatterers was assumed.
6.3 SANS Results

6.3.1 Neutron scattering pattern

The 1D data in Figure 6.1 was obtained by integrating the intensity that is collected from specific cells of the multidetector that have the same distance from the centre. The intensity at each distance from the centre is integrated over $2\pi$ to create a 1D plot. Figure 6.1 shows the nuclear scattering cross-section from two positions of the CHS-16 sample, with 16% prestrain, measured on the D11 instrument as well as on the SANS2D instrument. CHS-16-Reference refers to the grip area of the specimen where cavitation damage is negligible. The grip area is also the region where no cold work has been applied since the grip area was left unstrained. CHS-16-01, on the other hand, refers to the position closest to the creep fracture surface (about 1.5 mm away), that experienced the highest stress of about 307 MPa. Some measurements were carried out at both facilities to determine the SANS measurements reproducibility. On the D11 instrument, measurements at three different detector positions (1.5 m, 8 m and 28 m) were conducted to obtain about 360 scattering intensities across a q-range from 0.00167 to 0.426 Å$^{-1}$. At the SANS2D instrument, only 74 scattering intensities were recorded over a very similar q-range. This underlines that the q-resolution is significantly better for measurements on the D11 instrument. The results from both instruments, D11 and SANS2D, underline very similar scattering intensities for large parts of the q-range. Figure 6.1 illustrates that the scattering intensities from both instruments for two different stress positions in the CHS-16 sample are almost identical for q-values lower than 0.07 Å$^{-1}$. Only at larger q-values a difference from both instruments is notable. In this high q-area the scattering intensity is predominantly a consequence of inelastic scattering. For the results of the SANS2D instrument, this contribution is significantly higher than for the D11 data. As mentioned earlier, the better q-resolution from the D11 instrument leads to an improved accuracy, especially in this high q-region where the flux of suitable neutrons in a white beam (SANS2D) is inherently lower. Subsequently, the D11 data is more reliable. A higher background or contribution of inelastic scattering
leads to a lower measure of volume fraction and number density of scatterers.

Figure 6.1: Neutron scattering results (in log scale) from two points of the CHS-16 sample with 16% prior plastic strain (a) 16-01 about 1.5 mm away from the failure position and b) CHS-16-Reference in the grip area measured using the SANS2D and D11 instruments.

Since the q resolution on the D11 instrument is inherently better compared to the results from SANS2D, mostly D11 data obtained at the ILL was used for the analysis in this chapter. In a further series of plots the variation of the macroscopic scattering from the 12% and 16% prestrained specimens are shown in Figure 6.2. Various locations with 1.5 mm spacing for CHS-12 and 1.0 mm spacing for CHS-16 were measured along the length of both crept specimens. The data reveals the effect of creep on the scattering curves, \( \frac{\partial \Sigma}{\partial \Omega} \) versus \( q \). Both plots in Figure 6.2 exhibit an enhancement in scattering as a function of stress. Due to the hourglass sample design, the increase in distance from mid-length is associated with a decrease in stress and creep strain. The measurements across the hourglass gauge section revealed significantly larger scattering when compared to the grip region which were used as reference measurements. An increase can be observed over the whole q-range but is particularly apparent in the low q region. A rise in either precipitation
or cavity density can contribute to the rise in scattering intensity. As described earlier in Section 2.8.1, changes in the scattering signal due to a variation in dislocation density can be neglected in the data analysis. Schmatz et al. could show that even in samples with a high density of dislocations, defects such as cavities or precipitates dominate the scattering signal in SANS experiments [146]. Since both, precipitates and cavities mainly contribute to the scattering signal for Type 316H stainless steel, the data needs to be carefully looked at, to distinguish cavities from precipitates, to draw right conclusions. Therefore, SANS always requires validation with other complementary techniques such as SEM and cryogenic fracture which will be covered later in this chapter.

**Figure 6.2:** Differential scattering cross section (in log scale) measured at the D11 instrument and at different positions away from the fracture position. The specimens were prestrained to a) 12% and b) 16% and crept at a net engineering stress of 305 MPa and a temperature of 550°C until creep failure. The black lines show the scattering from the reference position.
6.3.2 Size Distribution and volume fraction

The significance of these scattering curves can be inferred by fitting models using the Maximum Entropy (MAXE) method that estimates the volume fraction and size distribution of scatterers. For simplification of the fitting, it was assumed that the number of equivalent spheres is equal to the total number of scatterers within the sample. So all scatterers are assumed to be spherical. This implies that if the cavity shape differs from a spherical structure, the distribution function calculated by MAXE might under or overestimate the cavity dimensions. However, since the sample is a complex alloy the number of spheres is not equal to the number of cavities within the sample. To exclude the contribution of precipitates to the measured scattering cross section, the reference data, measured within the grip region, had to be subtracted. The grip regions spent the same amount of time in the furnace but didn’t experience any strain during creep. It is therefore reasonable to assume that they are free of cavitation damage and all scattering can be assumed to be from precipitates. For a system of diluted particles such as cavities or $M_{23}C_6$ carbides in Type 316H stainless steel, the integral of the coherent cross section $\tilde{q}(0)$ gives the total volume fraction $V(D)$ of scatterers, that is defined by

$$V(D) = \frac{\sum N_p V_p}{V_{tot}}, \quad (6.1)$$

where $V_p$ is the volume of each scatterer, $N_p$ is the number of scatterers and $V_{tot}$ is the total volume of the sample. The integral of the coherent cross section can be defined by

$$\tilde{q}(0) = 4\pi \int_0^\infty \frac{d\Sigma}{d\Omega}(q)q^2dq. \quad (6.2)$$

The integral $\tilde{q}(0)$ can be obtained by extrapolating the cross section accordingly to the Porod law for high $q$ values and to the Guinier law for low $q$ values [143, 185]. It is worth pointing out that the equation is valid if the volume fraction $V(D)$ is smaller than $1 \times 10^{-1}$. 
which is the case for all tested samples [153]. To distinguish the contribution of carbides and cavities it is assumed that the precipitation does not change significantly under creep conditions which will be discussed in Section 6.3.3. It is a simplification that has been utilised by other groups [153, 159, 165]. In doing so the volume fraction of cavities can be deduced by decomposing the total integrated volume fraction of scatterers as

$$\tilde{q}(0) = \tilde{q}(0)_{Cavities} + \tilde{q}(0)_{M_{23}C_6}$$ (6.3)

Equation 6.3 underlines that the total scattering measured in Type 316H stainless steel is the sum of scattering from cavities and carbides. Therefore, the scattering contribution of cavities can be measured by subtracting data from the grip region. The calculation of the volume fraction is done by MAXE which provides the volume fraction of the scattering particles \(V(D)\).

### 6.3.3 Reference measurements

Table 6.1 shows the creep properties of the four tested samples. It highlights that the four samples spent a varying time under temperature within the furnace until fracture due to creep. While the creep time for CHS-4 accounts for 1624 h, CHS-16 has spent almost twice the time in the furnace with 3080 h under a temperature of 550°C. The data, illustrated in Figure 6.3, were analysed using the SANS2D measurements. The measurement on the D11 instrument of the reference point on CHS-04 was found to be inexplicably high and it is assumed that the scattering signal of this position was erroneous.
Figure 6.3: Size distribution function of the volume fraction of scatterers $V(D)$ from the reference (grip region) for four samples prestrained to 4, 8, 12 and 16% measured using the SANS2D instrument.

Figure 6.3 shows the scattering intensity of the reference (grip region) for each of the four samples. The reference (grip region) measurements of the four tested specimens show a very similar distribution of the volume fraction $V(D)$ of scatterers. All of them exhibit a similar pattern with a maximum peak at around 20-30 nm. This illustrates the existence of small scatterers. Furthermore, Figure 6.3 illustrates a broad relatively flat curve with a flattened peak at around 200 nm. The data shows that the time difference spent in the furnace had a negligible effect on the evolution of scattering particles. Since cavitation can be ruled out due to the absence of any strain at the grip end, presumably only second phase particles contribute to the scattering. The SANS data reveal that there is not much change in either volume fraction or growth of second phase particles.
6.3.4 Points with different amounts of prior cold work

In addition to the measurements within the grip region, data was obtained from the parallel gauge section above the hourglass section of each specimen, as illustrated in Figure 3.15. These points lie in the straight section of each specimen, around 24 mm away from the creep fractured surface. Based on the 3D-DIC data, it could be guaranteed that no additional creep strain was accumulating here during the experiments, as illustrated in Figure 5.8.

![Figure 6.4: Size distribution function of the volume fraction of cavities $C(D)$ from points with different amounts of prior cold work but no additional creep strain about 24 mm away from the creep failure surface.](image)

However, in contrast to the reference points (grip region), the straight section of the samples was exposed to different amounts of cold work from the prestraining process prior to the creep experiments, as described in Section 5.2.1. These measurements were done to investigate if cold plastic loading strain has any effects on a change in cavity or void evolution. The stress at these points, which will be referred to as S-points in the following
description, is about 165 MPa. The fitting results, illustrated in Figure 6.4, show a systematic increase of volume fraction of cavities $C(D)$ with an increase in prior cold work. The CHS-16 sample shows the highest amount of cavities whereas CHS-04 and CHS-08 show the lowest volume fraction. Figure 6.4 also illustrates a change in the volume fraction of cavities as a function of cold work over the whole diameter size spectrum. However, the most significant changes can be detected for cavities with a diameter smaller than approximately 75 nm.

6.3.5 Measurement points in the hourglass section

To obtain the volume fraction of only cavities within the crept region, the scattering intensity from the far field region was subtracted. The size distribution of the volume fraction $C(D)$ of creep cavities is illustrated in the following four plots of Figure 6.5. Here, measurements from the D11 experiment are illustrated. Each plot depicts the volume fraction of four points on each of the samples with 4 %, 8 %, 12 % and 16 % prestrain. The plots reveal the distribution of the volume fraction of cavities over a size range between 5-356 nm. The distance between each measurement point were kept to 1.5 mm. With an aperture size of 2 by 4 mm there is a slight overlap between each point which increases the spatial resolution. The 16% prestrained sample was the only specimen where a measurement point distance of 1 mm was chosen. This was done because the 3D-DIC data showed a very low creep ductility as well as a sharp decline in creep strain. It was believed that significant changes in the SANS signal would be found only close to the fracture surface.

In general, for each of the samples an increase of the volume fraction of cavities $C(D)$ could be observed that correlates to an increase in stress and creep strain. It can be found that the most significant change in volume fraction in each sample is between the first and second measurement point (close to the failure position). This is particularly apparent for the large sized population ($>60 \text{ nm}$) of cavities. For each of the samples it was tried to pick points with a similar distribution of true stress and as close to the creep fracture surface as possible. Figure 6.5 illustrates the SANS results conducted at D11 and Figure 6.6 from
SANS2D. Both experiments revealed a similar distribution of creep cavities.

**Figure 6.5:** SANS results, from D11, showing the distribution of cavities and their increase in volume fraction of cavities for four samples with a) 4%, b) 8%, c) 12% and d) 16% of pre-straining. The sample all crept until fracture at a temperature of 550°C and a net engineering stress of 305 MPa.
Figure 6.6: SANS results, from SANS2D, showing the distribution of cavities and their increase in volume fraction of cavities for four samples with a) 4%, b) 8%, c) 12% and d) 16% of pre-straining. The sample all crept until fracture at a temperature of 550°C and a net engineering stress of 305 MPa.
Two populations of cavities can be observed: a population of small cavities with a diameter size range between 15 and 60 nm and a population of larger cavities with a more broad distribution but lower volume fraction. The population of large cavities exhibits a diameter range between 60 nm and 400 nm. The volume fraction of cavities $C(D)$, as illustrated in Figure 6.5 and 6.6, are consistently positive which demonstrates that an appropriate scattering signal from the grip region has been subtracted. Furthermore, it could be observed that the volume fraction of the data from the SANS2D instrument converges to the x-axis for cavities with diameters close to 356 nm, whereas the D11 instrument revealed a volume fraction that converges to zero for larger cavities than that. The data from both instruments, SANS2D and D11, revealed a steady increase of the volume fraction as a function of stress. Furthermore, the data from both instruments revealed an increase in volume fraction for the population of small cavities ($< 60$ nm) as a function of prior plastic strain. With an increase of prior plastic strain it was found that the volume fraction of small cavities ($< 60$ nm) enhances.

To take a closer look at the data from both instruments (SANS2D and D11), Figure 6.7 compares the results for the CHS-16 sample, with 16% prestrain at four different stress positions along the hourglass gauge section. For consistency and similar to the previous analysis, both data sets have been analysed with the same error factor of 3 (as described in Section 3.5.8). An error factor of 3 fits the data from the D11 instrument better than the data from the SANS2D instrument. The distribution of the volume fraction from both instruments for large cavities is almost identical for CHS-16-02, CHS-16-03 and CHS-16-05. Only CHS-16-01 shows a different pattern. Here, the data from the D11 instrument revealed a peak shifted towards larger cavities. The SANS2D data reveals a peak in the volume fraction for small cavities ($< 60$ nm) that is higher compared to data from the D11 instrument for all four stress positions across the hourglass gauge section. Furthermore, the data from the D11 instrument shows a double peak for small cavities, whereas the SANS2D data reveals a Gaussian profile. For cavities smaller than 60 nm, the results from the SANS2D instrument reveal a less declining and more similar volume fraction at around
0.8 \times 10^{-6} for all four stresses, whereas the data of the D11 instrument reveal a continuous decline of the peak of the volume fraction as a function of stress. The double peak and the decrease in volume fraction highlights a good q-resolution of the D11 instrument that captures small sized scatterers.
Figure 6.7: SANS results, from the SANS2D and D11 experiments, showing the distribution of cavities and their increase in volume fraction of cavities for four different stress positions along the hourglass gauge section of the CHS-16 specimen with 16% prior plastic strain. The sample crept until fracture at a temperature of 550°C and a maximum net engineering stress of 305 MPa.
6.3.5.1 Mean diameter of cavities

The frequency distribution of the volume fraction of cavities was used to determine a mean diameter for each population of cavities. The mean diameter of the population of small cavities (<50 nm) was determined by fitting it to a Gaussian function. The mean diameter for the broader distribution of large cavities (50 to 356 nm) was calculated by a method of moments and direct summation, similar to an approach recommended by Jazaeri et al [165]. The results are depicted in Figure 6.8 which illustrates the mean diameter as a function of stress, based on the results from the D11 instrument. For small cavities (see Figure 6.8 a)) the data reveals a mean diameter between 20-30 nm for all four specimens, that is decreasing by about 2-3 nm over a stress range of 266 MPa to about 310 MPa. This shows a small dependency of the minimum stable size of cavities with an increasing applied stress. It confirms Raj’s nucleation theory that states that the minimum stable cavity size is inversely related to the applied stress and can be calculated by \( r = \frac{2\gamma}{\sigma} \), where \( \gamma \) is the surface tension and \( \sigma \) is the applied stress. Using this equation for the applied stresses of 266-310 MPa and a surface tension \( \gamma \) of 2.41, the minimum stable diameter size of cavities varies from 30-36 nm. These theoretical results are relatively close to what has been measured. CHS-4 with 4% prestrain exhibits the smallest cavities with a mean of just about 20 nm. For the population of large cavities CHS-4 exhibits the largest dimensions with mean diameters between 208-220 nm (see Figure 6.8 b)). The other three samples exhibit slightly smaller cavities ranging between 190 nm to about 210 nm. In general, for large cavities a growth of about 10-15 nm can be observed in a stress range of 266 MPa to about 310 MPa. The data reveals that the mean diameter of these large cavities is controlled by cavity growth and is proportional to the applied stress.
Figure 6.8: The mean cavity diameter measured by small angle neutron scattering at D11 of the two populations of cavities as a function of stress. The mean diameter is depicted for samples with 4%, 8%, 12% and 16% prestrain and a) cavities smaller than 60 nm and b) larger than 60 nm.

6.3.5.2 Total volume fraction and number density

In the following section, creep damage parameters such as the total volume fraction or number density of cavities have been calculated as a function of stress or distance towards the creep fracture surface as well as prior plastic strain. The procedure how to calculate the volume fraction as well as the number density of cavities has been described in Section 3.5.12. The data from D11 has been used because the D11 instrument has a better q-resolution and shows better fitting results for the analysis with MAXE. Figure 6.9 illustrates the integrated volume fraction over two parts of the distribution, for cavities smaller than 60 nm and for large cavities with a diameter larger than that (60-356 nm). It depicts an increase in volume fraction that corresponds to an increase in stress. Figure 6.9 also depicts the integrated volume fraction of cavities at points with a stress of just 165 MPa (S-Points), as described in Section 6.3.4. These points, in the straight section of the cylindrical sample, experienced only a varying plastic strain due to cold work but
no further creep strain. The small cavities at this constant stress of 165 MPa reveal an increase in volume fraction as a function of prior cold work. The CHS-16 results show the highest amount \((0.85 \times 10^{-4})\) followed by CHS-12 \((0.55 \times 10^{-4})\). The total volume fraction of creep cavities particularly at lower stresses, for CHS-4 and CHS-8, is relatively similar. Figure 6.9 b) shows the integrated volume fraction for cavities with a diameter larger than 60 nm. An increase in total volume fraction is visible, however changes with respect to the amount of cold work are not so significant. The gradient with which the volume fraction is increasing is relatively sharp. This shows that the population of large cavities \((> 60\text{ nm})\) is dominated by stress and fairly independent of prior plastic strain. Furthermore, the gradient of the total volume fraction between each position becomes larger towards higher stresses.

![Figure 6.9](image)

**Figure 6.9:** The total integrated volume fraction of cavities as a function of stress for a) cavities smaller than 60nm and b) cavities larger than 60 nm, measured by small angle neutron scattering. The total volume fraction is depicted for samples with 4%, 8%, 12% and 16% prestrain.

The number density of spherical cavities was calculated using the approach outlined in Section 3.5.12. There are two ways to calculate the number density. In the first approach, the volume distribution function needs to be distinguished between small \((< 60\text{ nm})\) and
large (> 60nm) cavities. From each distribution a mean diameter can be determined either by fitting to a Gaussian profile or by a method of moments and direct summation. The mean diameter of small cavities (< 60nm) was calculated by fitting the volume fraction to a Gaussian function. For calculating the mean diameter of large cavities (> 60nm), where the volume fraction fluctuates more, the method of moments and direct summation was applied. To calculate the number density, the total integrated volume fraction of each distribution of cavities needs to be divided by the mean diameter of each distribution. This gives two results for the number density N(D). One result for small cavities (< 60nm) and one for large cavities (> 60nm). However, it is also possible to calculate the ratio between volume fraction and spherical volume over each diameter section and to determine the number density N(D) as a function of diameter. This second approach has the benefit that a distribution of the number density function N(D) can be calculated, which is illustrated in Figure 6.10. If one calculates the summation of this number density distribution function (illustrated in Figure 6.10) over 0-60 nm and 60-356 nm two specific number density values can be determined. Both approaches have been followed and were found to give relatively similar results.
Figure 6.10: The number density distribution function $N(D)$ of cavities as a function of diameter for samples with a) 4%, b) 8%, c) 12% and d) 16% prestrain measured by small angle neutron scattering. The number density has been determined by calculating the ratio between volume fraction and spherical volume over each diameter section.
The data reveals, that for all four samples, the smallest cavities have the highest number density $N(D)$ which reduces sharply with an increase in size. For each case, the number density drops relatively sharply until about 60 nm. Furthermore, the rate of decline reduces until a diameter size of about 300 nm is reached. For the largest cavities, $N(D)$ drops sharply again. Following to these calculations, the number density could be integrated for each measurement point and distinguished between small and large sized cavities as illustrated in Figure 6.11. This is similar to the calculation of the total volume fraction (see Figure 6.9). The number density calculated for different true stresses is almost two magnitudes higher for small cavities with a radius below 60 nm compared to larger cavities with a diameter larger than 60 nm. For both populations, the number density rises exponentially with an increase in stress and creep strain. The results are very similar to the calculation of the volume fraction shown in Figure 6.9. However, the difference in magnitude between number densities of small and large cavities is greater than that in volume fraction, since in the calculation of the number density the volume fraction is divided by cavity size so the difference is amplified. For the small population of cavities, the integrated number density increases as a function of prior plastic strain with CHS-12 and CHS-16 showing the highest amount. The number density for the large population of cavities increases with stress but not so much with prior plastic strain. The progression of the number density of creep cavities as a function of stress was fitted to a stress dependent exponential function for each of the four specimens and for both populations of cavities. The results of the fitted functions for small cavities, which are shown in Figure 6.11, highlight the correlation between prior plastic strain and number density.
The results of the calculation of the number density of cavities per grain boundary area, as described in Section 3.5.12, are illustrated in Figure 6.12. The number density of cavities per grain boundary area (\(\mu m^{-2}\)) is rising as a function of stress. Similar to previous results, the CHS-16 specimen shows the highest density of cavities per grain boundary area followed by CHS-12. The lowest density of cavities has Sample CHS-8. The calculations for the cavity spacing reveal that the spacing decreases with stress and creep strain. They also show that the cavity spacing is below 100 nm for all samples and just above 50 nm for the CHS-16 sample, which shows the lowest average cavity spacing. These calculations provide an estimate on the density of creep cavitation that should be expected in scanning microscopy studies. However, they are just an assumption and the data can fluctuate quite significantly. For these calculations, only cavities larger than 15 nm are considered. This is in accordance with the diffusion controlled cavity nucleation model that predicts a minimum stable cavity size from the expression \(2\gamma/\sigma\), where \(\gamma\) is the surface tension and \(\sigma\) is the applied stress [101]. If this number of the minimum stable cavity diameter is changed the results alter quite significantly. If only cavities larger than 100 nm are considered, the
cavity spacing is increased by almost a magnitude. This has to be considered since most imaging studies discard cavities below 100 nm. The amount of prior cold work seems to increase the number density and subsequently decrease the cavity spacing per grain boundary area. The number density is increasing towards higher stresses while the cavity spacing is declining.

Figure 6.12: a) Number density of creep cavitation per grain boundary area and b) average cavity spacing over grain boundary area, using the D11 data. The specimens were prestrained to 4, 8, 12 and 16 % and subsequently crept until fracture at a temperature of 550°C and at a net engineering peak stress of 305 MPa.

6.3.6 McSAS vs MAXE

Since the distribution of scattering particles for each SANS measurement series is so distinctly recurring it was tried to establish that it was not merely the result of the MAXE software that was used to analyse creep damage in Type 316H stainless steel. The data up to this section has been analysed using the MAXE software as described in Chapter 3. As outlined earlier, it is not straightforward to analyse the scattering of particles in alloys...
where the distribution is highly polydisperse. Commonly used software packages such as SASView assume standardised distributions such as Gaussian, Schultz Zimmerman or log normal [217]. McSAS, based on a Monte Carlo algorithm, on the other hand enables, similar to MAXE, a fitting procedure without a priori knowledge about the distribution of scattering particles [192]. Figure 6.13 illustrates that the error between the raw data and the fitted curves is relatively small for the MAXE as well as McSAS analysis. The MAXE fitting shows a slight discrepancy at a \( q \) value of around 0.1 Å\(^{-1}\). However, this discrepancy is relatively minor.

![Differential scattering cross section as a function of the scattering vector q for the reference position of the sample CHS-16 using D11 data. The fitting result of the MAXE fit is shown in a) whereas b) illustrates the McSAS fitting.](image)

**Figure 6.13:** Differential scattering cross section as a function of the scattering vector \( q \) for the reference position of the sample CHS-16 using D11 data. The fitting result of the MAXE fit is shown in a) whereas b) illustrates the McSAS fitting.

In the following section the results of the distribution and volume fraction of cavities for the samples with 8%, 12% and 16% are illustrated in Figure 6.14, 6.15 and 6.16. The McSAS results are shown as a bar plot with 50 bins over the whole size range. The scattering data from the reference region was subtracted prior to the fitting. In addition, the volume fraction of scatterers \( V(D) \) had to be divided by the scattering length density of the steel.
matrix $7.05 \times 10^{-6} \text{ Å}^{-2}$ in order to calculate the volume fraction of cavities $C(D)$, as described in Section 3.5.5. It can be found that the results from both software packages give a very similar distribution of cavities. Even the double peak, for example in CHS-16-01 and CHS-12-09, can be found to be almost identical for both software packages. The volume fraction calculated by McSAS for small cavities is smaller but exceeds the MAXE results in the highest diameter region. Contrary to the MAXE results, the volume fraction in the McSAS plots is not decreasing as much towards larger sized cavities, i.e. greater than 300 nm. This is plausible since cavity growth does not stop at around 300 nm as could be assumed from the MAXE analysis. The McSAS distribution is quite volatile with some sections growing in volume fraction while others stay at the same level over the whole stress range. For points with high stresses, for example CHS-08-01 and CHS-12-09, the McSAS results reveal that the volume fraction from both distributions levels out with certain bars even extending the volume fraction of the small population of cavities. This is not the case for the MAXE results where the volume fraction of small cavities (< 60nm) is always larger than for large cavities. Both software packages show a growth in volume fraction as a function of applied stress and creep strain for all three samples. Between CHS-16-01 and CHS-16-03, there is only a distance of 2 mm but the volume fraction of the large population of cavities decreases quite significantly. In general, it can be found that the trend of size distribution calculated by the Monte Carlo approach is very similar to the fitting result given by the Maximum Entropy method. These good agreements support the assumptions made by analysing the SANS results when calculating the volume fraction and size distribution of cavities in steel samples. However, since SANS has its limitations to distinguish second phase particles from cavities all the results have to be supported by a qualitative microscopy using SEM which will be covered in Section 6.4.
Figure 6.14: Distribution of the volume fraction of cavities for sample with 16% pre-strain crept at 550°C and at a net engineering peak stress of 305 MPa. The volume fraction of cavities $C(D)$ is calculated with MAXE as well as the Monte Carlo Software McSAS based on the D11 data.
Figure 6.15: Distribution of the volume fraction of cavities for sample with 12% pre-strain crept at 550°C and at a net engineering peak stress of 305 MPa. The volume fraction of cavities $C(D)$ is calculated with MAXE as well as the Monte Carlo Software McSAS based on the D11 data.
Figure 6.16: Distribution of the volume fraction of cavities for sample with 8% pre-strain crept at 550°C and at a net engineering peak stress of 305 MPa. The volume fraction of cavities \( C(D) \) is calculated with MAXE as well as the Monte Carlo Software McSAS based on the D11 data.

The calculated mean diameter from both software packages are compiled in Table 6.2. The mean diameter calculated by McSAS estimates slightly larger mean diameters for the small
as well as the large population of cavities. Both software packages reveal an increase of
the mean diameter for the population of large cavities as a function of stress of about 6-18
nm over a stress range from 266 MPa to about 314 MPa. The mean diameter of small
cavities with a diameter less than 60 nm is decreasing with an increase in stress. These
changes are quite subtle for the MAXE results with only 2-3 nm and more significant for
the McSAS results with changes from 4-8 nm. These results seem to confirm the diffusion
driven cavity nucleation theory proposed by Raj where the minimum stable diameter of a
cavity is a function $2\gamma/\sigma$ [101]. Furthermore, Table 6.2 shows the total volume fraction of
the cavities calculated with MAXE and McSAS. It could be found that the volume fraction
calculated with McSAS is always slightly larger the the value calculated with MAXE. This
higher volume fraction comes from the larger cavities. McSAS does not calculate such a
significant drop in the volume fraction for large cavities with a mean diameter above 300
nm as MAXE does, as seen in Figures 6.14, 6.15 and 6.16. In McSAS the volume fraction
for large cavities sometime even rises, as can be seen for CHS-16-02 and CHS-16-03.
Table 6.2: Overview of the SANS results, such as mean diameter for both populations of cavities and total volume fraction, obtained from two independent methods (MAXE and McSAS).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Eng Stress</th>
<th>Prestrain</th>
<th>Diameter Small (&lt;60 nm) MAXE</th>
<th>Diameter Small (&lt;60 nm) McSAS</th>
<th>Diameter Large (&gt;60 nm) MAXE</th>
<th>Diameter Large (&gt;60 nm) McSAS</th>
<th>Total Volume Fraction MAXE</th>
<th>Total Volume Fraction McSAS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-08-01</td>
<td>314.2 MPa</td>
<td>8%</td>
<td>20 nm</td>
<td>24 nm</td>
<td>205 nm</td>
<td>210 nm</td>
<td>5.512e-4</td>
<td>6.282e-4</td>
</tr>
<tr>
<td>CHS-08-02</td>
<td>297.1 MPa</td>
<td>8%</td>
<td>22 nm</td>
<td>26 nm</td>
<td>196 nm</td>
<td>204 nm</td>
<td>3.429e-4</td>
<td>4.069e-4</td>
</tr>
<tr>
<td>CHS-08-03</td>
<td>282.5 MPa</td>
<td>8%</td>
<td>23 nm</td>
<td>29 nm</td>
<td>190 nm</td>
<td>207 nm</td>
<td>2.625e-4</td>
<td>3.240e-4</td>
</tr>
<tr>
<td>CHS-08-04</td>
<td>266.1 MPa</td>
<td>8%</td>
<td>23 nm</td>
<td>30 nm</td>
<td>190 nm</td>
<td>203 nm</td>
<td>2.032e-4</td>
<td>2.624e-4</td>
</tr>
<tr>
<td>CHS-12-09</td>
<td>306.1 MPa</td>
<td>12%</td>
<td>24 nm</td>
<td>28 nm</td>
<td>205 nm</td>
<td>212 nm</td>
<td>6.833e-4</td>
<td>7.663e-4</td>
</tr>
<tr>
<td>CHS-12-10</td>
<td>290.9 MPa</td>
<td>12%</td>
<td>26 nm</td>
<td>33 nm</td>
<td>194 nm</td>
<td>209 nm</td>
<td>4.466e-4</td>
<td>5.320e-4</td>
</tr>
<tr>
<td>CHS-12-11</td>
<td>278.7 MPa</td>
<td>12%</td>
<td>26 nm</td>
<td>36 nm</td>
<td>187 nm</td>
<td>210 nm</td>
<td>3.378e-4</td>
<td>4.225e-4</td>
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<tr>
<td>CHS-12-12</td>
<td>267.7 MPa</td>
<td>12%</td>
<td>26 nm</td>
<td>36 nm</td>
<td>187 nm</td>
<td>206 nm</td>
<td>2.986e-4</td>
<td>3.757e-4</td>
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<tr>
<td>CHS-16-01</td>
<td>307.3 MPa</td>
<td>16%</td>
<td>23 nm</td>
<td>26 nm</td>
<td>213 nm</td>
<td>211 nm</td>
<td>6.617e-4</td>
<td>7.019e-4</td>
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<tr>
<td>CHS-16-02</td>
<td>299.8 MPa</td>
<td>16%</td>
<td>24 nm</td>
<td>28 nm</td>
<td>207 nm</td>
<td>210 nm</td>
<td>5.279e-4</td>
<td>5.811e-4</td>
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<tr>
<td>CHS-16-03</td>
<td>297.0 MPa</td>
<td>16%</td>
<td>24 nm</td>
<td>29 nm</td>
<td>200 nm</td>
<td>208 nm</td>
<td>4.505e-4</td>
<td>5.063e-4</td>
</tr>
<tr>
<td>CHS-16-05</td>
<td>278.6 MPa</td>
<td>16%</td>
<td>24 nm</td>
<td>30 nm</td>
<td>195 nm</td>
<td>207 nm</td>
<td>3.676e-4</td>
<td>4.215e-4</td>
</tr>
</tbody>
</table>

6.4 Microstructural analysis using SEM

Microstructural analysis was carried out using a Zeiss Sigma 500 Field Emission Scanning Electron Microscope (FEGSEM) to obtain images and to qualitatively assess cavities. Three selected samples CHS-8, CHS-12, CHS-16 have been etched and polished accord-
ing to the preparation procedure described in Section 3.4.3. The samples were mainly examined utilising the high definition backscattered, the secondary or the InLens imaging mode with an accelerating voltage of 10 kV and an aperture size of 30 µm. In addition to the conventional SEM studies, a cryogenic fracture technique was used to reveal creep cavities. This has the benefit that it reveals the microstructure without the alterations that can be caused by etching and polishing which is required for conventional SEM investigations. The technique has been used by Chen and Argon to investigate creep cavitation damage for Type 304 stainless steel and more recently by Jazaeri et al. for Type 316H stainless steel [73, 218]. Similar to Type 304, Type 316H stainless steel develops grain boundary embrittlement when subjected to cryogenic temperatures. Subsequently, intergranular fracture that is free from dimples and tearing of the sample can be achieved. The cryogenic fracture experiments were conducted on matchstick test specimen. As illustrated in Figure 6.17, a slice with a thickness of 2.5 mm from the sample was extracted. At distinct distances of 3 mm, notches with a depth of 0.9 mm were inserted by EDM to support the initiation of cryogenic brittle fracture. The slice was fixed in a specially constructed jig and cooled down in liquid nitrogen for about 4-5 min to reach a temperature close to −196°C. After removing, the samples were fractured immediately.
6.4.1 Overview observation of the microstructure

The fracture micrographs of the cross section of the CHS-08 and CHS-16 samples close to the creep fractured surface are presented in Figure 6.18. For both prestrained samples large microcracks could be observed. The majority of those cracks tends to form perpendicular to the stress axis and intergranular. It can be observed in Figure 6.18 that these microcracks are relatively large close to the fracture surface and reduce in density with an increase in distance to the fracture surface. Since each crack length is varying quite significantly the information from taking the number of cracks as an indication of damage is limited. It is quite striking that although the vast majority of cracks is close to the fracture surface some large cracks are 1-2 mm away from the fracture surface. The length in which the microcracks are growing does not show a linear decrease with the distance and they are distributed relatively randomly. However, this can be misleading since close to the fracture the coalescence of short cracks to form long cracks is highly likely to have caused fracture of
the sample. Both samples have just experienced cold strain prior to the creep experiment and no further hot plastic strain from loading, as shown in Table 6.1. The final creep ductility reached 1.4% for CHS-12 and 0.8% for CHS-16 which is really low and makes occurrence of necking and plastic hole growth right before failure unlikely.

![Fracture region of (a) CHS-8 and (b) CHS-16](image)

**Figure 6.18:** Fracture region of (a) CHS-8 and (b) CHS-16, creep rupture samples tested at 305 MPa at a temperature of 550°C. The loading for both samples was applied in the direction as highlighted as in a).

### 6.4.2 Qualitative analysis of creep cavity dimensions

In order to verify the findings from the SANS results, qualitative SEM micrographs were obtained from the CHS-12 sample in order to analyse the dimensions of the creep cavities. Figure 6.19, 6.20, 6.21 and 6.22 were taken from various positions within the hourglass section of the sample with 12% prestrain. The vibro-polishing, as described in Section 3.4.3, revealed cavitation damage as well as precipitates around the grain boundaries. The boundary could be traced by following the cavities and carbides. For some grains, changes in the contrast helped to identify the boundary structure and to separate different grains. The analysis revealed that while some of the small cavities are relatively spherical and
equiaxed, the majority of cavities especially around precipitates show a facetted structure. Figure 6.19 illustrates this quite nicely with a number of elongated cavities around two large second phase particles that are presumably carbides. The length of the two largest cavities is around $1\mu m$ while the width is less than 100 nm. In the upper right corner of Figure 6.19 the dimensions of three cavities have been added. Further evidence of the facetted structures of cavities are depicted in Figure 6.20 and Figure 6.21. Each of these images, taken from the 12% prestrained sample, verify the sizes of cavities measured by SANS. Commonly observed dimensions are starting from 20 nm diameter to larger cavities with sizes of several hundreds of nm. Nearly all of them are surrounded by second phase particles that presumably facilitate their nucleation. These second phase particles could be found all over the sample even within the far field region. Here however, without any evidence of cavitation. The majority of the cavities as well as second phase particles was found intergranular with some exceptions of intragranular second phase particles.

![Figure 6.19: High Definition Back-Scattered electron Detector (HDBSD) image showing the size and faceted structure of cavities (appear black) nucleating at second phase particles in sample in the crept sample, 12% prestrained at a 3 mm distance from the failure position.](image-url)
Figure 6.20: HDBSD image showing the size and faceted structure of cavities (appear black) nucleating at second phase particles in the crept sample, 12% prestrained at a 3 mm distance from the failure position. The right figure is the close view of the area marked in the left figure.

Figure 6.21: HDBSD image showing the size and faceted structure of cavities (appear black). The cavity nucleation seems to happen at several points of the second phase particles. The image is taken from the crept sample, 12% prestrained at a 5 mm distance from the failure position.
6.4.3 Analysis of cavitation on grain boundaries

Figure 6.23 and 6.24 illustrate an interesting observation for the 16\% prestrained sample. Images at a distance of 3 mm and 5 mm from the creep fracture surface are depicted. They are recorded with the backscattered detector in the high resolution mode and hence an image pixel size of around 9 nm was achieved. This implies that cavity sizes as low as around 30 nm can be observed. Figure 6.23 and 6.24 reveal that cavity nucleation occurs relatively homogeneously around the grain. A very fine line of cavities with a diameter size range between 30-120 nm can be observed on each grain boundary. Almost all cavities are situated intergranularly on the grain boundaries. It also reveals that cavity growth happens only on specific grain boundaries with a certain grain boundary orientation relative to the maximum principal stress direction. In Figure 6.23 three different boundaries denoted as A, B and C are highlighted. Cavity growth and coalescence seems to happen only on boundaries A and C while the cavities on boundary B stay relatively small. With an angle
of $88^\circ$ towards the loading direction, Boundary A is almost perpendicular while boundary C is at $57^\circ$ to the loading direction. For both, it can be argued that there is cavity growth due to a relatively large grain boundary sliding at boundary C and high normal stresses on boundary A [73, 98]. On the other hand, the angle between loading direction and boundary B is beyond $80^\circ$ as well however, with far less indication of cavity growth. It seems that factors such as the crystallographic orientation of the adjacent grains has a significant contribution on the cavity growth. While small cavities in Figure 6.23 and 6.24 are between 30-100 nm the large ones easily reach sizes of around 150-300 nm similar to what was measured by SANS.

Figure 6.23: Distribution of intergranular creep cavities in the CHS-16 (16% prestrained) sample 3 mm away from the failure position. The Figure shows several grain boundaries where cavity growth and coalescence are observable. The cavity spacing is given for one particular region on the top right image.

The same behaviour is observable in Figure 6.24 which is 5 mm away from the creep fracture surface. Again coalescence and growth of cavities happens only on one specific grain
boundary that is almost transverse to the loading direction. Since the grain boundaries in Figure 6.23 and 6.24 are covered with a huge number of small cavities the cavity spacing was measured which is illustrated in both figures. It can be found that the density and interspacing of the small creep cavities is indeed decreasing with a further distance from the fracture surface that correlates with a decrease in stress and creep strain. However, these results can not be taken as quantitative evidence. Furthermore, the cavity spacing can not be determined where coalescence has already started and cavities have grown to a certain size. While the calculation from SANS predicted a cavity spacing of around 50 nm, illustrated in Figure 6.12, the spacing between cavities for these two examples is somewhat larger.

Figure 6.24: Distribution of intergranular creep cavities in the CHS-16 (16% prestrained) sample 5 mm away from the failure position. The cavity spacing is given qualitatively for one particular region.
6.4.4 Cavitation morphology and position using cryogenic fracture

Figure 6.25 shows that the extent of creep cavitation is changing with the distance from the fracture position. The series of images was taken from the sample with 8% prestrain prior to creep. Figure 6.25 a) to d) refer to distances of 2.5 mm, 5.5 mm and 15 mm and 35 mm respectively away from the failure position.
Figure 6.25: Scanning electron micrographs of fractured surfaces in the 8% prestrained sample, separated by cryogenic fracture. a) 2.5 mm, b) 5.5 mm, c) 15 mm, d) 35 mm away from the fractured surface.

A similar series of pictures for the 12% prestrained sample is illustrated in Figure 6.26. Here, micrographs are taken at distances of 4 mm, 8 mm, 12 mm and 33 mm away from the fracture surface. The cavity damage decreases with the distance. However, isolated
and scattered creep cavities and precipitates are found at each interval. Even the picture on the bottom right in Figure 6.26 which is captured at a distance of 33 mm shows cavities as well as second phase particles.

Figure 6.26: Scanning electron micrographs of fractured surfaces in the 12% prestrained sample, separated by cryogenic fracture. a) 4 mm, b) 8 mm, c) 12 mm, d) 33 mm away from the fractured surface.
The extent to which intergranular facets are cavitated is clearly decreasing. However, even at positions close to the creep fracture position many but not all facets are cavitated. It can be observed that the cavitation damage is very heterogeneously distributed. While some facets are completely cavitated, adjacent grains can be almost free of cavities. Using the cryogenic fracture method, there are limitations in resolution so that cavities with a size of less than 100 nm may not be resolved. It can only be assumed that they are indeed cavity free. This is quite apparent in Figure 6.27 where heavily cavitated facets are shown. Nonetheless, there is a section that seems to be undamaged. It could be that this is due to a change in the crystallographic orientation presumably due to twinning. The images show that the cavity shapes are more equiaxed than elongated. The cryogenic fracture also revealed spherical and facetted shaped creep cavities as illustrated in Figure 6.27. As mentioned earlier, cavities are heterogeneously distributed along the grain boundaries but it can be found that they are mainly concentrated around triple points where they increase in size and density which can be observed in Figure 6.27 and Figure 6.28. The enhanced picture on right side of Figure 6.28 reveals a number of small cavities within a small microcrack. It’s presumably a contribution of grain boundary sliding as well as cavity growth and coalescence that facilitates the development of these wedge crack structures similar to what has been observed in Type 304 stainless steel by Chen et al. [73, 98].
Figure 6.27: Observation of caveatted and non-cavitated facets adjacent to each other in 8% prestrain sample at 2.5 mm away from the fracture surface.

Figure 6.28: Cavity clustering on a) several facets in the CHS-08 sample 2.5 mm away from the creep fracture surface and b) a closer view of the area marked in the left hand side image.
6.5 Discussion

While there are a variety of techniques either destructive, semi-destructive or non-destructive to characterise microstructural changes in creep damaged metals and alloys, only a few are suitable to detect cavities at sub micron sizes. TEM and SEM are the most common techniques to study microstructural features of materials despite their dependence on metallographic preparation, which usually involves repeated polishing and etching that can alter the microstructure of the material. For example, it can cause precipitates to be removed from the sample and therefore change the apparent size and quantity of creep cavities. It also can enlarge cavities and therefore give an overestimation of the size of cavities. Also, it is limited in the sense that it only provides information for a two dimensional section of a small sampling area. This makes it usually quite challenging to obtain quantitative metallographic results [120, 163]. However, as a direct technique it is possible to obtain images featuring creep cavity damage. Small angle neutron scattering on the other hand, is classified as an indirect technique. The results, such as the volume fraction or number density of scatterers, can only be obtained from fitting models to the 1D or 2D scattering patterns. Furthermore, SANS has difficulties to distinguish between precipitates and creep cavities and therefore has to be supported by SEM or TEM interrogation of the microstructure. It has to be acknowledged that each technique has its limitations and a combination is necessary to make profound conclusions. For the SANS analysis, certain simplifications had to be made. It was assumed that all scatterers in the material in a diameter size range of 10-400 nm are spherical. Furthermore, similar to the work of Fuller et al. as well as Boeuf et al. [153, 159], who both studied cavities in Type 304 stainless steel, it was assumed that for the crept tested specimen the average dimensions and volume fraction of second phase particles is the same with and without creep conditions. This is not fully correct as pointed out by Swindemann et al. but provides the only possibility to distinguish second phase particles from creep cavities [157].

In the SANS analysis, two populations of cavities were observed in all crept specimens.
that had undergone cold plastic work prior to creep. The volume fraction as well as the
distribution of cavities calculated by MAXE has also been confirmed with another software
package McSAS that is designed to provide fitting results for metals and alloys. These
software methods are both suitable for analysing a high polydispersity of scatterers where
there is no prior knowledge about the distribution. A population of small cavities with a
diameter size range between 10 and 60 nm could be observed for the as-received material
after creep deformation. This confirms other studies on creep cavity damage carried out
on Type 304 and 316H stainless steel [153, 163, 165]. The fitting of the SANS data also
revealed a population of larger cavities with a mean diameter of around 200 nm, as shown
in Figure 6.8. Two populations of creep cavities have been observed in other studies on
the same type of steel by Jazaeri et al. [164, 165, 209]. Jazaeri et al. also carried out
quantitative metallographic work on Type 316H stainless steel and found a similar volume
fraction of intergranular creep cavities using SANS and SEM. Scanning electron microscopy
carried out on three samples with 8%, 12% and 16% prior plastic strain confirmed cavities
with dimensions close to that observed by SANS studies. The metallographic studies also
revealed precipitates and coalesced cavities larger than 400 nm that can not be resolved
with small angle neutron scattering. SANS has its limitations if a fraction of cavities
and precipitates grows out of the bounds of the minimum resolvable q-range to which
the instrument has been adjusted for. If this occurs the volume fraction and mean size
of cavities will be underestimated. A study by Bouchard et al. was carried out in the
past to extend the size range with ultra small angle scattering [162]. Each population of
cavities was found to increase in volume fraction, number density and to a certain extent
mean diameter as a function of stress and creep strain. This is a clear sign that creep
damage and fracture in Type 316H stainless steel is caused by the continuous nucleation
and growth of creep cavities. The continuous nucleation of creep cavities as a consequence
of accumulation of creep strain has been reported by [109, 115]. The volume fraction of
cavities for both populations increased systematically with an increase in stress for all
tested specimens with 4%, 8%, 12% and 16 % prior cold work, as illustrated in Figure 6.9.
In addition, it could be observed that changes in volume fraction are particularly large
at positions close to the fracture surface. The McSAS and MAXE results both showed a continuous cavity growth of up to 18 nm in diameter over a stress range from 266 to about 314 MPa (see Table 6.2). This highlights that cavity growth for large cavities is governed by stress. For positions further away with less stress and creep strain, changes in volume fraction are becoming smaller but are nonetheless observable. In addition, it could be observed that the number density for small cavities (<60 nm) enhances with a change in the amount of prior cold work. The results in Figure 6.11 suggest that prior plastic strain increases the nucleation sites of creep cavities with increasing number density for small cavities with a diameter of less than 60 nm. However, based on the SANS data, cavity growth was observed by only 15-18 nm over a stress range from 266-314 MPa. It raises the question if creep damage is determined by the large number density of small cavities or rather by cavities that have grown and eventually coalesce and cause fracture.

The samples prestrained to 12%, 16% show a higher fraction of small cavities, as illustrated in Figure 6.9. This was observed and verified in some of the SEM images, see Figure 6.23. For CHS-16, a fraction of small intergranular cavities with a diameter between 30-120 nm was observed. The extent of these small cavities exceeded the amount of small cavities found in the CHS-8 as well as CHS-12 samples. Dyson et al. have suggested that a large value of creep cavities per creep strain \( \frac{N_a}{d\epsilon} \) is associated with a small ductility [29]. This relation has been observed for the sample with 16% prestrain that showed the lowest creep ductility but yet the highest number density of nucleated creep cavities. It seems also reasonable to assume that cavity nucleation rather than cavity growth is influenced by prestrain prior to creep. This is illustrated in Figure 6.9 a) where it can be found that the volume fraction of cavities with a diameter <60 nm is increasing significantly with prior plastic strain.

The MAXE and McSAS analysis revealed that the mean diameter of large cavities (>60 nm) increased with the applied stress, as shown in Table 6.2. The mean diameter of cavities smaller than 60 nm on the other hand, decreased with an increasing stress, which confirms the diffusion controlled cavity nucleation that predicts a minimum stable cavity radius.
that is a function of $2\gamma/\sigma$, where $\gamma$ is the surface tension and $\sigma$ is the applied stress [101].

The McSAS analysis revealed a minimum stable diameter that varied in the tested stress range from 24 - 30 nm for CHS-08, 28 - 36 nm for CHS-12 and 26 - 30 nm for CHS-16. Raj’s equation of the diffusion controlled cavity nucleation model predicts a change in the minimum stable diameter ranging from approximately 30 nm to 36 nm. These results match very well with the results from the McSAS analysis, shown in Table 6.2. The MAXE results for the mean diameter of the small cavities are lower than what Raj’s theory predicts but are nonetheless relatively close.

The microscopy studies also revealed that cavities are nucleated predominantly along grain boundaries as well as around precipitates, triple junctions and locations where high stresses are present, see Figure 6.23, 6.24 and 6.27. Evidence of small and large cavities faceted around precipitates could be found, see Figure 6.19. It is reasonable to assume that these precipitates are in fact $M_{23}C_6$ carbides which are commonly found at very early creep stages and are known to facilitate the cavity nucleation since they can act as local stress raisers if they impede grain boundary sliding [219]. As mentioned by Raj et al. they can also reduce the nucleation barrier of stable cavity formation as they attract vacancies to relieve stresses from dislocation build ups close to them [94,104]. Most of the cavities found in the samples were in fact linked with these second phase particles along the grain boundaries. The faceted nature of some of the observed cavities might be a result of the anisotropy of their surface energy or a result of grain boundary sliding which causes faceted rather than spherical voids. The cryogenic fracture procedure revealed a predominantly heterogeneous distribution of creep cavities on intergranular facets in the samples CHS-08 and CHS-12, as described in Section 6.4.4. While some facets are heavily cavitated others are completely cavity free, even if they are adjacent. However, the 3D morphology of the fracture surface makes it challenging to resolve small features and in some cases it could not be verified if the facets were indeed free of cavities or if the cavities were too small to be resolved. However, cavity growth was observed towards triple points. A large number of cavities could be observed on surfaces close to the creep failure position where high stress and creep strain
were present. The fraction of cavitated facets decreases as the distance to the creep fracture surface increases. The far field measurements show facets with a very smooth surface, as illustrated in Figure 6.25 and 6.26. This verifies the findings from SANS where a change in number density could be found as a function of stress as illustrated in Figure 6.11. The inclination angle between loading direction and grain boundaries has a significant effect on the nucleation and especially growth of cavities. From the SEM images, see Figure 6.23 and 6.24, it could be determined that creep cavity growth happens predominantly but not exclusively on boundaries transverse to the loading direction. While the nucleation of cavities in CHS-16 is homogeneously distributed along the grain boundaries, cavity seems to occur only on specific grain boundaries. Cavity growth on boundaries with an inclination angle of 90° to the loading direction can be explained due to their orientation to the maximum principal stress, leading to dislocation pile ups causing cavities to nucleate and to grow. Some boundaries with inclination angles of around 45°, where shear stresses are highest, also show cavity growth and coalescence. Slater et al. found a high density of cavities on boundaries with such an inclination angle [98]. It is commonly accepted that cavity nucleation is promoted by grain boundary sliding [44,46]. The sliding in the presence of particles has to be large enough to stimulate cavity growth and counter sintering. This has been investigated by Harris et al. who determined the amount of sliding rate necessary to enable cavity nucleation [107,108].

As shown by Petkov et al., even at transverse boundaries sliding can be existent if the adjacent grains are anisotropic [71]. While there is a discussion whether cavity growth on transverse boundaries is the result of an anisotropic nucleation or preferential growth of cavities, the SEM results of CHS-16 seem to give evidence that cavity growth is the result of preferential growth. This observation supports conclusions provided by Dyson et al. [82]. However not all transverse boundaries show signs of cavity growth. The crystallographic orientation of the adjacent grains also seems to influence cavity nucleation and growth. Around twin boundaries it could be observed that the density of cavities can be highly disparate. Dyson et al. found an increase in cavity nucleation in presence of prior plastic
strain in their studies of Nimonic 80A [82]. They suggested that colliding of slip bands with second phase particles can be linked to a higher density of cavities. A higher density of cavities can be found in the vicinity of softer grains where it is more likely for slip to occur. If the applied loading exceeds the macroscopic yield stress, softer grains start to deform by single slip. To maintain continuity the deformation becomes non homogeneous, causing strain gradients. Adjacent grains with different crystallographic orientations experience different strain which leads to local stress concentrations. If the local strain accumulation leads to slip bands, this raises the stress concentration locally even further. Slip bands can lead to an increase in decohesions and microcracks. Therefore, cavity nucleation is higher in grains with a higher Schmid factor surrounded by softer grains. The large scatter in growth for individual creep cavities has been investigated by Dzieciol et al. in copper [94,220]. He acknowledged local dislocation glide as a factor that adds significantly to the amount of cavity growth. Since dislocation glide occurs only on particular crystallographic planes this could cause heterogeneity of creep damage.

6.6 Summary

Creep cavity damage in a set of prestrained Type 316H stainless steel specimens crept at 550°C under a net maximum engineering stress of 305 MPa has been investigated using SANS and SEM. Parameters such as the volume fraction, number density and size distribution of cavities were measured quantitatively using SANS. Complementary, qualitative imaging was conducted using SEM using either a conventional preparation technique of the samples or a cryogenic fracture technique to verify some of the SANS observations.

1. Two populations of creep cavities were observed for all creep samples with a varying amount of prior plastic strain of 4%, 8%,12% and 16% and applied engineering stresses ranging from 305 MPa to 165 MPa. A population of small cavities with a diameter range of 10-60 nm and large cavities with a diameter between 60-400 nm were measured by SANS.
2. SANS data analysed using the Maximum entropy method (MAXE) were verified using an independent Monte Carlo simulation method (McSAS). A good correlation of size distribution and volume fraction is achieved.

3. An increase in number density of small creep cavities, as a function of prior plastic work, was observed in the SANS analysis and later confirmed qualitatively using SEM. A large number of nucleated and stable intergranular creep cavities were observed with cavity growth and coalescence only on particular grain boundaries.

4. The cavities are found to be associated with second phase particles and showed a predominantly spherical and facetted shape.

5. The results of the cryogenic fractured samples confirmed an increase in volume fraction of creep cavities with an increasing stress and creep strain. Furthermore, the images revealed a highly heterogeneous distribution of creep cavities with some facets heavily cavitated while adjacent grains cavity free. The cavities were found to grow in size and density towards triple points.
7 General Discussion

7.1 Overview

The discussion covers the main findings of this PhD work. The first part reviews how prior plastic strain influences the creep properties of the as-received material. The results of two characterisation techniques, SANS and SEM, are presented to provide an overview about the influence of stress and creep strain but also of prior plastic strain, on the evolution of intergranular creep cavitation. In Section 7.2.5, application of a stress modified ductility exhaustion model to estimate macroscopic creep damage from the creep properties is reviewed in the light of some of the SANS findings. The final part of the chapter part covers the applicability of 3D-DIC monitoring for long term creep tests under high temperature to measure spatially resolved creep strain curves using a novel hourglass specimen.

7.2 Discussion on key findings

7.2.1 Changes in creep deformation as a result of prior loading history

Investigations on the effects of prior deformation on subsequent creep behaviour are important since most components enter service in prestrained conditions either as welded components or due to fabrication processes such as cold rolling. As outlined in the literature review in Section 2.5, there is a general consensus of understanding the creep resistance effects of prior plastic deformation on the subsequent creep deformation in Type 316H stainless steel [15,76]. In general, the strain to failure as well as the creep strain rate reduces as a consequence of prior plastic tensile strain. This leads to an increase in the time to failure. For compressive prestrain, it could be found that the strain to failure as well as the creep strain rate reduces as well. However, contrary to tests in pretension, a decreasing time to failure could be observed [79].
The Ashby deformation map for Type 316H stainless steel predicts dislocation creep as the main deformation mechanism for all tested creep conditions in the present work [49]. The Orowan relation, which has been mentioned in Section 2.5.1, describes the restricted dislocation mobility as a consequence of prior cold work. According to the Orowan relation, the strain rate can be described using \( \dot{\varepsilon} = v_m b \rho_m \), where \( \rho_m \) is the density of mobile dislocations (a function of temperature and stress), \( b \) is the magnitude of the Burgers vector and \( v_m \) is the average velocity of the dislocations. The dislocation density changes significantly with the amount of plastic strain as pointed out by [15, 215]. The decrease in creep strain rate is usually attributed to an increase in dislocation density that impedes dislocation motion. Therefore, the creep rate decreases as a consequence of work hardening.

The heating process prior to a typical creep test removes some barriers to dislocation movement by thermal recovery, but does not fully recover material in its initial state. For materials where prestrain does not lead to an increase in creep resistance, as observed for example by [82, 90], it has been argued that the mobility of dislocations is not affected.

The influence of prior plastic strain on subsequent creep in the as-received material is presented in Chapter 5. Cylindrical samples were prestrained in tension to different amounts of 4 %, 8 %, 12 % and 16% at room temperature. After prestraining, an hourglass gauge section was machined. These samples were then creep tested with a maximum engineering stress of 305 MPa under load controlled settings. The findings of this series of tests are in good agreement with published work on prestrained Type 316H stainless steel [15]. In all creep tested samples where prior plastic strain was induced at room temperature, a reduction in creep ductility and creep strain rate with an increase in time to rupture was observed, see Figure 5.9. The plots of the creep strain rates, as illustrated in Figure 5.12, showed a somewhat anomalous behaviour. After loading, all samples exhibited a period of reducing creep rate that dropped to a minimum. Samples with sufficient prior plastic strain, that avoided further hot plastic strain from creep test loading (i.e. 8%, 12% and 16%), maintained a period of constant creep rate, after dropping to a minimum. The length of the constant strain rate period was increased by the amount of prior plastic strain. The
creep strain rate in this period could be fitted to a Norton Power law, see Figure 5.17, which showed a reduction of creep strain rate as a function of prior plastic strain. All samples exhibited the usual acceleration of creep rate shortly before failure. For samples with less prior plastic strain, there was no well-defined period of constant creep rate and the samples went to an extended tertiary regime soon after they reached the minimum creep rate. The anomalous behaviour of the very low creep strain rate followed by a constant creep strain rate made it impossible to fit the creep strain curves to the RCC-MR creep model which is generally used to characterise the creep properties in Type 316H stainless steel [221]. The creep strain rates (illustrated in Figure 5.13) at lower stresses (below 250 MPa) and only in a prestrained condition, showed a renewed primary creep strain rate. This behaviour has been observed for Type 316H stainless steel elsewhere, albeit for much longer duration times [213]. A possible explanation for this behaviour is that, as the tests start dislocations become pinned due to the precipitation of second phase particles causing a reduction in creep rate. The coarsening of carbides and subsequent unpinning of dislocations is accompanied by an increase in creep rate. Wilshire and Willis observed, for fully solution annealed material, that pretension changes creep properties only if the amount is large enough to fully eliminate any further plastic strain from creep test loading [15]. This observation was not confirmed in the present work, as illustrated in Figure 5.12 and 5.13. For increasing prior plastic strains a decreasing effect on the creep properties could be observed. Even the CHS-0 and CHS-4 specimens, both with significant amount of hot loading strain, showed a drop in creep ductility and creep strain rate. The reduction in creep ductility as a function of prior plastic strain, as illustrated in Figure 5.9, is believed to be a consequence of an accelerated cavity nucleation which will be further discussed in the next section.

7.2.2 Cavity nucleation as a result of prior plastic strain

Goods et al. studied the creep behaviour of silver samples implanted with cavities and found that creep life and strain at failure were significantly reduced compared to faultless
ones [115]. Dyson et al. showed that a large $dN_a/d\epsilon$, where $dN_a$ is an incremental change in the number of cavities per unit volume and $d\epsilon$ is an incremental change in strain, can be linked to a low creep ductility [29]. The failure at a lower creep ductility of the pre-strained material might be due to early nucleation of voids in the material as a result of the prior plastic strain induced at room temperature. Some theories to explain this behaviour are outlined in the following section.

There are three main principles that govern cavity nucleation. It can be caused by either grain boundary sliding, dislocation pile-ups or condensation of vacancies driven by diffusion processes [29,101,103]. There is agreement about prior plastic strain leading to an increased density of intergranular creep cavities [29,82]. Several theories explain this acceleration of cavity nucleation as a consequence of induced prior plastic work. The phenomenon was observed by Dyson et al. for Nimonic 80A as well as by Shiozawa et al. in Astroloy where samples pre-strained at room temperature were found to exhibit an increased density of cavity nuclei. Dyson for example, suggested that the colliding of slip bands with second phase particles can be linked to a higher density of creep cavities. An increased amount of slip bands in the vicinity of cavities as a result of plastic strain was also observed by Auzoux et al. in 316L(N) stainless steel [222]. Dyson et al. found also that a higher density of creep cavities can be linked to plastically softer grains where it is more likely for local slip to occur [82]. Grains that yield lower stresses are termed soft, whereas grains that yield larger stresses are termed hard. If the macroscopic yield stress is exceeded the grains start to deform by single slip. To maintain continuity across all grain boundaries the deformation becomes non-homogeneous and strain gradients are created. The likelihood of slip bands at grain boundaries leading to microcracks or decohesions, i.e. cavity nucleation, is higher in grains with a higher Schmid factor surrounded by softer grains. These microcracks are growing on boundaries that are parallel to the maximum principal stress direction and will shrink on those which are orthogonal. On the other hand, cavities in the vicinity of harder grains or normal to the applied stress axis that can carry higher stresses are more likely to exhibit cavity growth. The deformation incompatibility caused by an increase
in prestrain can also lead to local stress concentrations, which then again leads to cavity
nucleation around the grain boundaries. Shiozawa et al. concluded that intergranular
damage can be considered as a result of stress concentrations that cause nucleation due to
local decohesions [84]. Prestraining leads to an increase in grain boundary alteration as a
result of damage between carbides and matrix. Subsequently, the residual stresses around
these areas are enhanced significantly. If there is a tensile stress acting upon the specimen,
there are stress concentrations around these rather hard second phase particles leading to
dislocation pile ups that generate enough tensile stresses to exceed the cohesive strength
between matrix and particles. As a consequence, cavitation or even microcracks occur
that are associated with localised slip. Shiozawa argued that even if the load is removed
residual stresses maintain and cavities still form as a consequence of plastic strain around
the precipitates. Since the problem of reheat cracking is also triggered by slow strain rates,
residual stress concentrations and stress triaxiality, prior plastic strain can lead to a similar
effect [223]. As mentioned by Dhooge et al. reheat cracking has been found on unwelded
but prestrained components [224]. Local stress concentrations as a result of plastic strain
are more likely to occur in f.c.c. crystals with a low stacking fault energy (such as Type
316H stainless steel). Plastic strains in grains can cause misorientation changes that can
be visualised with EBSD mapping. The inelastic strain causes surface changes in the local
crystallographic orientation or misorientation which is a parameter that can be measured
using EBSD. According to Unnikrishnan et al., microcracks in Type 316H stainless steel
form between grains with a higher misorientation, which is linked to higher amount of
plastic strain [225].

Chen and Argon on the other hand, suggested that cavity nucleation is a consequence of
stress concentration due to grain boundary sliding [73]. Sliding along inclined boundaries
increases both cavity nucleation and cavity growth. In their scanning electron microscopy
studies on crept Type 304 stainless steel samples, they found an increased number of
facetted and ellipsoidal cavities as well as a higher density of creep cavities around triple
junctions, similar to what has been observed in Figure 6.27 and 6.28. Both observations
indicate that grain boundary sliding facilitates creep cavity nucleation and growth. Recently, it has been found by Petkov et al. that even transverse boundaries show signs of sliding if the adjacent grains are anisotropic [71]. It was long believed that boundary sliding on these perpendicular grains does not occur and therefore grain boundary sliding is not a necessary driving mechanism for cavity nucleation since cavities have been found on transverse boundaries [101,103]. The maximum amount of shear stresses can be found on planes with an inclination angle of 45°. In a study by Slater et al. on Type 316 stainless steel, inclined boundaries are found to exhibit a higher density of cavities. Cavity growth however, was found to be associated with grain boundaries perpendicular to the loading direction [98]. He et al. suggested that the rate of grain boundary sliding determines the nucleation rate of cavities [108]. They derived a model that suggests cavity nucleation as a consequence of the impeding movement of subboundaries and particles. It is an empirical model that links the proportional behaviour between cavity nucleation and creep strain.

7.2.3 Using SANS to determine creep cavitation in Type 316H SS

In Chapter 4 and 6, results of creep cavity measurements using small angle neutron scattering are presented. SANS was chosen to investigate creep cavities because it is very sensitive to scattering particles of sub-micron size and free of concerns regarding the metallographic preparation, which makes it ideal to study the evolution of creep cavitation as a function of stress and creep strain. Since SANS is sensitive even to small cavities with diameter sizes of just a few nm, this measurement technique can inform understanding of cavity nucleation and growth processes in polycrystalline materials. SANS can be used to determine cavity damage in bulk sample volumes that are orders of magnitude larger than what can be sampled using SEM or TEM methods. However, since it is an indirect measuring method and fitting of the scattering data is relatively complex, complementary methods must be applied to confirm its findings.

The fact that the dimension of scattering particles varies from a few to several hundreds
of nanometres adds to the level of complexity. Furthermore, since the distribution form of the scattering particles is not known a priori it cannot be assumed as a starting parameter. There are only a few software packages that analyse scattering data from polycrystalline samples and most of them offer only a limited amount of mathematical forms for the distributions (e.g. lognormal, Gaussian or Schultz-Zimmerman distribution). Consequently, few models are capable of fitting the scattering data from particles in metals and alloys correctly. In addition to MAXE, which has been commonly used to analyse SANS data in Type 316H stainless steel [164, 165, 218], an independent software method based on a Monte Carlo algorithm (McSAS) was used to fit the scattering data. Both methods avoid the need to define a specific distribution function to which the scattering parameters are fitted. McSAS has only recently been developed and since then applied mostly to SAXS data [191, 197]. The validity of both methods has been supported by fitting a set of simulated data, see Figure 3.23 and Figure 3.24. While the general shape of scatterers in MAXE is restricted to spheres, this can be extended to other shapes in McSAS, i.e. ellipsoids or rectangular scatterers. If the shape of scatterers is verified by complementary methods such as SEM or TEM, both software packages can derive a unique solution for the volume fraction and size distribution. The analysis of SANS data using the two independent software methods, McSAS as well as MAXE, revealed almost identical results for the size distribution and volume fraction of creep cavities, see Table 6.2. This gives confidence in the accuracy and rigour of SANS as a characterisation technique to determine creep cavitation in the austenitic Type 316H stainless steel.

In general, the SANS experimental data acquired in the present studies showed an increase in volume fraction as well as number density of cavities as a function of creep strain, time and stress. With similar experimental setups on the SANS2D instrument at the ISIS spallation source (Harwell) and on the D11 instrument at the Institut Laue-Langevin (Grenoble) in terms of the q-range, scattered particles over a diameter size range from 5 nm to about 356 nm (D11) and 400 nm (SANS2D) could be measured. The two instruments gave almost identical scattering results, as shown in Figure 6.1. Two distinct populations
of creep cavities could be established in all creep tested samples tested at various stresses, see Figure 6.9. A population of small cavities with a diameter in the range 15-60 nm and a population of large cavities with a broader distribution and diameter range between 60 and 356 (D11) or 400 (SANS2D) nm. Furthermore, an increase of volume fraction and number density with an increase in stress could be observed for all creep tested samples tested for various stress conditions.

The challenges in obtaining quantitative information on the nucleation and growth of cavities as a consequence of creep are relatively complex. It is necessary to distinguish any contribution to the neutron scattering coming from second phase particles, which is only feasible under the simplified assumption that changes of the precipitation density during creep can be neglected. Only then is it possible to obtain information on the total volume fraction of creep cavities. Raj’s diffusion controlled cavity nucleation theory predicts a minimum stable cavity radius that can be estimated from the expression \( 2\gamma/\sigma \), where \( \gamma \) is the surface tension and \( \sigma \) is the applied stress. The equation predicts a varying range for the minimum stable cavity radius along the hourglass gauge section [101]. With a surface tension of 2.41 \( N m^{-1} \), which is a reasonable value for Type 316H stainless steel according to Yu et al. [207], this would result in a change in the minimum stable diameter between approximately 36 nm and 30 nm for a stress range of 266 MPa to 314 MPa. At lower applied stresses, larger cavities become stable. The results for small cavities (< 60 nm), tabulated in Table 6.2, are in reasonable agreement with the predictions. Especially the McSAS results, were found to be really close to what the diffusion controlled cavity nucleation theory, proposed by Raj, predicts for the minimum stable diameter. The data analysed by MAXE and McSAS revealed an increase in size for small cavities (< 60 nm, see Table 6.2) with a decrease in stress that seems to confirm the equation of the minimum stable cavity size [101]. While the decrease of the mean diameter for the MAXE results are more subtle and are only about 2-3 nm, the McSAS results reveal a decrease in mean diameter with an increase of applied stress by approximately 4-8 nm in a stress range from 266 MPa to around 314 MPa, see Table 6.2. The minimum stable cavity size, as mentioned by Chen et
al., is challenging to verify due to limitations of direct experimental techniques such as SEM or TEM, due to alteration of the microstructure with sample preparation [120]. For the population of cavities with a diameter above 60 nm, the MAXE results revealed that the average cavity size grew by 15-18 nm with an increase in stress along the hourglass gauge length, as illustrated in Figure 6.8. For larger cavities, the mean diameter is controlled by cavity growth and is proportional to the applied stress. This accords with what has been observed in the literature with either unconstrained or constrained creep cavity growth that is stress dependent. While the unconstrained cavity growth is directly proportional to the maximum principal stress, unconstrained cavity growth is proportional to the shear stress raised to some power [46, 135]. The SANS results revealed that large cavities (> 60 nm) are growing by only small margins of 15-18 nm. An explanation for this rather small growth could be that if cavities grow and start to coalesce they scatter at q-values outside of the q-range to which the SANS experiments have been adjusted for. This implies an underestimation of the expected average size and volume fraction.

For the analysis of SANS using McSAS, it was established that the scattering data needs to be subtracted from reference data before the fitting process. MAXE on the other hand leaves the possibility to do the subtraction prior or after the fitting, aside from errors that were discovered for scattering in the high q-region from the D11 experiment, as described in Section 3.5.10. The scattering data gathered from SANS2D, possessed too large uncertainty in the high q-region for an analysis using the McSAS reference subtraction routine (see Figure 4.19). Thus, McSAS was only applied to analyse the D11 data. In order to use both software packages for Type 316H stainless steel, the measuring time per position has to be increased for future SANS2D experiments to resolve the instrument limitations. To verify the findings of the SANS results of the CHS-sample, described in Chapter 4, the mean diameter of cavities, calculated using MAXE was verified based on calculations of the Guinier radius. These results were found to be in good agreement, see Table 4.5. However, the calculation of the Guinier radius was not further utilised since its informative value apart from the mean diameter is rather limited.
Parameters such as the number density, volume fraction and cavity spacing were found to be influenced by initial prior plastic strain. The volume fraction as well as number density for small cavities with dimensions of less than 60 nm increased as a function of cold work (illustrated in Figure 6.9 and 6.11). This can be explained due to an increase in void density due to coalescence of piled up dislocations against hard particles or along slip planes [82]. Even at points with only prior plastic strain (S-Points) but no further creep strain an increased number of cavities could be measured, see Figure 6.4. These findings support the assumption of pre-existing cavities at the start of the creep test. The volume fraction as well as the number density of small cavities with a diameter of less than 60 nm was found to be increasing continuously with the onset of creep, which is shown in Figure 6.9 a). Here, the total volume fraction of small cavities changes not only due to stress but also as a function of prior plastic strain. A similar trend could not be confirmed for the population of creep cavities with diameters larger than 60 nm. Here, as illustrated in Figure 6.9 b) and Figure 6.11 b) the number density and total volume fraction increased only with a rise in stress as well as creep strain. The changes in creep ductility that have been observed for the prestrained specimen can be linked to changes in cavity nucleation [29]. The results confirm findings from Jazaeri et al. who showed that the number density of creep cavities is much larger for prestrained specimens compared to samples deformed under pure creep but at a higher temperature [165]. For the series of prestrained specimens, no signs of cavity growth, measured by the mean diameter, could be observed as a function of prior plastic strain, see Table 6.2. Prior plastic strain that promotes creep damage is therefore associated with cavity nucleation rather than cavity growth. Several studies showed that intergranular creep cavitation is a continuous process that is driven by creep strain [109, 115]. Recently, Jazaeri et al. found evidence of continuous cavity nucleation on their creep experiments in Type 316H stainless steel conducted at a temperature of 550°C and at an engineering stress of 320 MPa [165].

There are still uncertainties regarding the contribution to the scattering signal from second phase particles. This contribution might not be fully eliminated even when the scattering
signal from a reference region, the grip area where the temperature is similar but the stress level is negligible, is used. This approach was recommended by Fuller and Boeuf [153, 159] for their work on Type 304 stainless steel and later picked up by several other groups [164, 165] has its limitations since it assumes a similar carbide morphology for different stresses. Another possibility to eliminate contributions of second phase particles to the neutron scattering in Type 304 stainless steels was outlined by Yoo et al. [152]. Their approach was to use solution annealing after the creep experiments which showed a significant reduction in the scattering signal. Based on their findings Yoo et al. concluded that a majority of the scattering in Type 304 stainless steel crept samples comes actually from precipitation of second phase particles. The present author disputes this finding on the basis that the microstructure of the sample due to post creep solution annealing will be altered. It simply cannot be assumed that other particles do not diffuse into the cavities or voids while heating or cooling the sample. If this happens the neutron scattering length would be reduced significantly leading to a reduced neutron scattering. Furthermore, rapid cooling and corresponding thermal stresses could lead to sintering or shrinking of the cavities. Both explanations, as outlined by Fuller and Yoo [152,159], have their limitations and it remains challenging to interpret scattering data in polycrystalline samples where precipitation of second phase particles occurs.

7.2.4 Damage observed after induced prior plastic strain

Between tensile and creep tests, there is a transition in the failure modes from visco-plastic flow which causes ductile failure, to grain boundary diffusion which causes intergranular creep cavities to nucleate and grow [46]. The latter is accountable for a drop in the ductility. Based on the SEM images acquired in the present work, no apparent difference in the fracture mode could be found between crept samples where prestrain was induced and samples without prior plastic strain, see Figure 4.31 and Figure 6.18. The fracture surfaces and the reduction of area were quite similar. Intergranular cracks perpendicular to the loading direction could be found in both samples. For the present work, only a
qualitative metallographic approach was utilised (using FEGSEM) to verify the findings from SANS regarding the shape as well as size of creep cavities. The SEM images verified the specific size ranges inferred by SANS with small cavities with diameters of less than 60 nm and a second population of cavities with diameters larger than 60 nm, see Figure 6.20, 6.21 and 6.22. Jazaeri et al. carried out quantitative metallographic studies in Type 316H stainless steel to compare the volume fraction, size distribution as well as the dimensions of creep cavities with SANS observations and found a good agreement in general [164,165]. The imaging methods revealed spherical as well as irregular facetted type cavities predominantly located around second phase particles and along grain boundaries (illustrated in Figure 4.34 and 6.21). Investigations on the morphology of second phase particles in Type 316H stainless steel confirmed their irregular shapes and facetted structures which confirms previous studies [26,98]. The morphology of the precipitated second phase particles are illustrated in Figure 6.20 and 6.21. These images revealed multiple nucleation sites for creep cavities. Kim et al. showed that planar carbides exhibit a higher resistance to nucleate creep cavities which suggests that the morphology of the second phase particles is an important factor that influences creep cavity nucleation [226]. Burnett et al. suggested that models of creep cavity nucleation and growth that are neglecting the complexity of their facetted structures and their connection with second phase particles are oversimplified with regard to life time assessments [94].

The SEM studies on the longitudinal cross sections of the prestrained specimens proved that creep cavity damage increased steadily along the hourglass gauge section with an increase in stress due to nucleation, growth and coalescence of intergranular creep cavities, as illustrated in Figure 6.23 and 6.24. In addition, the cryogenic fracture studies revealed that the density of creep cavities varied with the distance from the fracture surface (i.e. the applied stress), see Figure 6.25 and 6.26. Furthermore, larger creep cavities seemed to be concentrated around triple junctions, suggesting that stress concentration due to grain boundary sliding is important [46,118].

A correlation between intergranular creep cavitation and precipitation of second phase
particles has been observed in literature before. Parker et al. found that the cavity density in copper is only influenced by prestraining if it was performed under poor vacuum conditions [215]. This indicates that if copper samples contain small oxides along the grain boundaries, prestraining promotes a significant increase in void formation which then results in significant reductions in both creep life and creep resistance. These findings are similar to what Fujiwara et al. found for testing Type 316 stainless steel solution annealed at a temperature of only 1000°C. Here, prestraining resulted in a significant increase in carbides, very similar in their behaviour to the oxygen particles in the copper studies, as they promoted void nucleation and growth. As outlined earlier, second phase particles can act as stress raisers due to their differences in their elastic constant which can lead to plastic deformation between the inclusions and the surrounding matrix. Burnett et al. could observe that boundaries where the highest number of cavities could be found were accompanied by second phase particles such as carbides [94]. Slater et al. found, in Type 316H stainless steel, the highest number density of nucleated creep cavities on boundaries at an angle of around 60° to the principal loading direction [98]. At such an inclination angle of the grain boundary to the loading direction shear stresses have the highest contribution. The SEM images in CHS-16 however, revealed a similar cavity nucleation rate across all grain boundaries, as illustrated in Figure 6.23 and 6.24. Only the cavity spacing decreased as a function of stress. The analysis also revealed, that only certain grain boundaries showed signs of cavity growth and coalescence whereas others exhibited relatively small cavities despite their boundary angle being at almost 90° to the loading direction, see Figure 6.23 and 6.24. In those SEM images coalesced or grown cavities could be observed along transverse grain boundaries with an inclination angle of 90°, presumably due to their orientation to the maximum principal stress leading to dislocation pile-up and at grain boundaries with a reduced inclination angle of 45 – 60° where grain boundary sliding due to shear stresses is dominant [98]. Both factors seem to contribute to cavity growth. Large cavities were found to have a more ellipsoidal shape compared to the small more spherical shape like cavities. This suggests that grain boundary sliding plays a role for cavity growth although it is unclear if growth is a result of coalescence of several small
cavities or simple growth of individual ones.

The crystallographic orientation of grains adjacent to cavitated boundaries presumably has an impact on the nucleation rate of cavities as well on cavity growth as the distribution of creep cavities has been found to be highly heterogeneous. Around the presence of twin boundaries it was observed that the number of creep cavities on adjacent grains was highly disparate with some facets completely cavity free while adjacent grain facets highly cavitated, see Figure 6.27. Hong et al. showed that the morphology and formation of intergranular carbides which are linked to the nucleation of creep cavities are dependent on the misorientation of the grain boundaries [97]. Jones et al. observed that the presence of $M_{23}C_6$ carbides was selective to the grain boundary plane type and that coherent twins could be indeed immune to precipitation which would explain almost free cavity facets at twin boundaries [227]. For this PhD work, no EBSD studies have been carried out to investigate the crystallographic orientation of grains in the vicinity of creep cavities but they should be considered for future work to build on this.

### 7.2.5 Creep damage measurements using the stress modified ductility exhaustion model

Parameters for the SMDE model were provided by EDF-Energy based on parent material of Type 316H stainless steel. For this PhD work, the model was applied on the set of creep fractured specimens where prior plastic strain was induced. The SMDE model predicts that most damage developed in creep tests using a load controlled setting is a consequence of plastic hole growth, either from loading or due to accelerated strain accumulation in the tertiary creep regime. The damage predicted by the SMDE model showed, for all tested samples, a total damage value significantly lower than 1, which reflects the very low creep ductility of the as-received material, see Table 5.5. According to the SMDE model, the prestrained specimens (apart from CHS-0), were tested either in the stress dependent region or transition region during all of their creep life which implies continuous cavity nucleation
and either constrained or diffusional cavity growth [12, 126]. The samples where prestrain was induced, all failed in the transition region apart from CHS-16 which stayed in the stress dependent region for the whole creep life. With an increase of prior plastic strain and a subsequent increase in damage from the upper shelf region (plastic hole growth) a reduced contribution of damage from the stress dependent region as well as from the transition region could be observed. This subsequently implies less contribution of constant cavity nucleation and diffusional as well as constrained cavity growth to the overall damage.

According to the model, prior plastic strain is the main source of damage by creating voids that are already existent at the start of creep, similar to what has been observed by other groups [29, 82]. The nucleation of these voids can either be a consequence of impeding movements of slip bands across hard second phase particles or grain boundary sliding leading to stress concentration, as outlined earlier. Table 5.5 exhibits an increase of damage as a consequence of prior plastic strain for samples without any further hot plastic strain from creep test loading. If one compares the three samples CHS-08, CHS-12 and CHS-16, it can be found that for a rise in prior plastic strain, an increasing amount of damage comes from plastic hole growth from the upper shelf region. Furthermore, the overall damage rises as a function of plastic strain. The overall damage from merely creep processes, i.e. the stress dependent region or the transition region reduces to around 32% for CHS-08, 26% for CHS-12 and only 14.3% for CHS-16. This shows the significant contribution of damage that the SMDE model attributes to the loading process. An increase in the number density of small cavities as a consequence of prior loading strain was confirmed by the SANS experiments. The proportion of damage that the SMDE model attributes to loading is somewhat larger than what the SANS data revealed. In Figure 6.9 and 6.11 it can be found that both, the total number density as well as the total volume fraction of cavities rises significantly from measurement points with a stress of 165 MPa, i.e. points with only prior plastic strain, to measurement points with the largest stress values close to the creep fracture surface. If one compares the volume fraction at the highest and lowest stress points it rises by a factor of about 3 for cavities with a diameter of less than 60 nm and by a factor of 5-6 for larger cavities. Subsequently, as the applied stress increases
along the hourglass gauge length, a significant increase in volume fraction, number density as well as a decrease in cavity spacing can be attributed to creep damage processes.

For a more in depth investigation of the SMDE model, strain controlled creep tests could channel damage accumulation within a targeted zone, preferably the transition region which represents plant conditions. Changes in intergranular creep cavitation could be investigated for samples creep tested in the stress dependent region where cavity growth is constrained and the transition region where cavity growth is diffusional. This type of testing is recommended for future investigations of the SMDE model, which is beyond the scope of this thesis.

### 7.2.6 Using 3D-DIC to measure strain in a high temperature creep test

3D-DIC has been successfully applied to monitor the surface strain on test samples having curved surfaces in long term creep experiments at high temperature. For this project, a novel cylindrical hourglass test specimen has been designed that allows measurements of creep curves at multiple stress levels from a single specimen. FE-analysis was carried out to determine the applied stress variation along the length of the cylindrical sample. The constraints for the design of the hourglass gauge section were determined by the load capacity of the creep test machine, the size of the access window, of the furnace for 3D-DIC monitoring and by the resolution of the neutron scattering experiment, as outlined in Section 4.3.2. This determined the radius of the hourglass gauge section. Steep changes in creep stress and strain were minimised since they cause large variation in creep damage within a short section. Along the hourglass gauge section multiple creep strain curves at different stress levels could be spatially resolved, see Figure 4.14. This special feature of measuring a multitude of creep curves reduces the number of creep tests required for characterising the creep behaviour of any material of interest and makes the material characterisation process more economical. For all creep tested samples, a total of 50 creep curves could be spatially resolved. However, this number is dependent on factors such as
fluctuation of displacement vectors and the resolution of the camera [179]. It is not a fixed number and needs to be adjusted for each set of tests. In general, it could be found that to exploit all of its capabilities, creep strain rates of at least $5 \times 10^{-7}$ per hour are preferred to analyse the creep strain rates accurately. The as-received material showed relatively slow creep strain rates that were further reduced by introducing prior cold work. These low rates precluded measurements of the creep strain rate to a satisfactory degree along the whole gauge length of some specimens. 3D-DIC allowed measuring creep strain not just in the longitudinal but also in the transverse direction. The strain in the transverse direction could be analysed to determine the reduction of area and thereby to calculate the true stress over the whole hourglass gauge section. The creep tests, which lasted from 851 hours to roughly 3000 hours, showed that the sample surface condition stayed stable under load control and a temperature of $550^\circ C$. No deterioration of the sample surface, which is normally a sign of oxidation, was observed. The high temperature paint applied was found to be in good condition even after creep times of about 3000 hours. However, applying large plastic strains, i.e. above 10%, lead to paint detached from the sample surface, as illustrated in Figure 3.2, which shows an example of a sample prior and after loading. It is believed that the geometry of the test specimen as well as the roughness of the surface of the material mainly contribute to the adherence of the paint on the sample. A series of tests established that the best way to avoid this is by creating coatings that are as thin as possible. As part of further future work it is recommended that an automated system should be designed for applying the paint while rotating the test specimen.

The scatter in the 3D-DIC data limits its accuracy. Origins of the data scatter include imperfections of the furnace window, which involves variations in thickness of glass and surface curvature, and variations of the refractive index of heated air in the furnace and between the camera lens and furnace window [179]. The main source of error however, comes from heat haze fluctuations. The 3D-DIC experiments showed that time-averaging techniques had to be applied to reduce the heat haze effects for strain calculations. This follows recommendations made by Sakanashi et al. for 2D-DIC experiments [179]. A
certain number of images, three to five were found to be ideal for filtering out the noise. However, the approach has its limitations and works only for experiments where the strain rate is relatively slow, which is the case for creep experiments. A common problem for DIC tests in general is that the strain to failure, especially in hourglass samples, is not straightforward to determine since pictures are taken only at a certain frequency. For long term creep tests, this frequency can be in the order of several minutes. Since failure in the tertiary regime is usually driven by plastic strain rather than creep and can occur quickly, the strain to failure might be underestimated by DIC. One of the benefits of using 3D-DIC rather than 2D-DIC is, as described in Section 3.3.5, is that the problem of the translation or rotation of the sample causing out of plane displacements is eliminated. The usage of the two camera stereo vision allowed capturing the deformation in all three dimensions.

One of the main disadvantages of using the hourglass specimen design is that it does not provide data on the creep rupture times and subsequently average creep strain rates for several stress positions within the hourglass gauge section. It is only possible to analyse the average creep strain rate for the position where the sample has failed. This limits the informative value that can be evaluated from one sample. For all other stress positions, rather than analysing the average creep strain rate, one has to analyse the minimum creep strain rate instead as a material defining parameter.

### 7.2.7 Limitations imposed on this work

The first creep experiment, described in Chapter 4, was tested at a relatively high stress due to time constraints by the awarded beam time at the ISIS spallation source with the first scattering experiment taking place only five month after starting the PhD work. Since applying for neutron beam time is highly contested and a lengthy process, a proposal for conducting a SANS experiment was submitted 6 month before starting the PhD already. Within the first five months of the PhD work, a sample design had to be conceived and a creep test up to failure had to conducted. Due to these limitations, a maximum engineering
stress of 360 MPa was applied to the hourglass specimen. These test conditions were chosen with an expected creep time to failure of about one month. The large stress, but also the material conditions in general with a yield strength of just over 140 MPa at a temperature of 550°C, see Table 4.3, lead to a significant amount of plastic strain from loading that superimposes the mere creep deformation. The geometry of the hourglass shape adds to the complexity of the experiments since it leads to varying plasticity based on the changing diameter. With these relatively large stresses that do not actually represent plant conditions there is a concern that damage within the sample is driven by plasticity rather than diffusion creep processes. The varying plastic loading strain which induces strain hardening along the hourglass gauge section affects the creep parameters significantly. A varying loading strain changes the density of dislocations across the hourglass section. Due to the precipitation of carbides they become pinned. As the creep time progresses the second phase particles coarsen and the dislocations become more mobile since the pinning of dislocations due to larger carbides is less significant. The informative value of experiments carried out under such high loads is therefore limited.

Due to limited time available to use laboratory facilities at the Open University as a consequence of Covid-19 restrictions, extensive quantitative microscopy could not be carried out as initially intended. An imaging analysis on positions measured by SANS for each creep tested specimen that quantitatively determines cavity sizes and volume fraction of creep cavities could not be carried out. Furthermore, it had been planned to use EBSD to link the location of intergranular creep cavities to the orientations of their adjacent grains. The EBSD analysis could have been used to investigate microstructural requirements that determine cavity nucleation and cavity growth. This however, was also not possible due to the limited access of laboratory facilities.
8 Overall Conclusion And Future Work

8.1 Overall conclusion

The first objective of the present research was to develop and harness 3D digital image correlation (3D-DIC) monitoring technology for determining the stress-dependent creep deformation behaviour of austenitic stainless steel using a variable gauge area test specimen under load control at high temperature. The second objective was to use the new test specimen design to investigate the high temperature creep deformation and damage response of AISI Type 316H stainless steel under tensile plastically pre-strained conditions. The research performed for this PhD has met both intentions and contributed to knowledge in the field as summarised below:

1. A novel tensile creep test specimen with a variable cylindrical cross section, and therefore applied stress, along the gauge length (hourglass shaped) has been designed.

2. A 3D-DIC system for monitoring the (length-wise) spatially dependent creep deformation of hourglass design test specimens at high temperature has been developed and successfully implemented for the first time (to the authors knowledge). Creep tests enduring up to 3,100 hours at 550°C have been performed and the time dependent creep deformation analysed as a function of stress (i.e. position along the specimen).

3. The 3D-DIC system monitors both, axial direction (loading) and transverse (Poissons) strains. The spatial resolution of the monitoring system is approximately 0.034 mm in y and x direction. 50 creep curves along the hourglass gauge section could be measured from one single specimen.

4. Introduction of 4%, 8%, 12% and 16% of plastic strain at room temperature prior to creep testing resulted in progressively longer times to rupture with reductions in creep strain rate and creep ductility for the same mid-length applied stress.
5. The measured minimum strain rates for the plastic pre-strained samples were found to correlate with applied true stress and the level of plastic pre-strain via a simple Norton power law. The time period of constant strain rate behaviour increased with level of plastic pre-strain.

6. The SANS measurements revealed two populations of creep cavities that were observed in all creep tested samples under various stress conditions. Small cavities with a diameter range of 10-60 nm and another population of larger cavities with a diameter range between 60-400 nm.

7. Two experiments at the ILL and the ISIS spallation source were used to carry out SANS studies in the as-received material with very similar results. For the first time, SANS results analysed using the maximum entropy method (MAXE) have been confirmed by repeating the analysis using an independent software package based on a Monte Carlo algorithm (McSAS). Both methods showed almost matching results of the size distribution and volume fraction of creep cavities that gave confidence in the rigour of using SANS as a method to quantify creep damage in Type 316H stainless steel.

8. The number density and volume fraction of small cavities with diameters less than 60 nm measured by small angle neutron scattering (SANS) was found to increase as a function of prior plastic strain. This is new evidence indicating that prior plastic strain introduces specific cavitation damage in the material.

9. Studies using the cryogenic fracture method showed a cavitation damage that varies with applied stress and distance to the fracture surface. These studies revealed a higher density of creep cavities around triple junctions. Furthermore, the images showed that creep cavity damage is highly heterogeneous with certain grain boundaries to be completely free of cavitation even close to creep rupture.

10. SEM studies revealed intergranular creep cavities as the main cause of failure for all tested stress conditions under prestrained as well as unprestrained conditions. The
SANS size distribution could be verified in a qualitative sense using SEM microscopy. In the SEM images small cavities with a characteristic size below 100 nm and larger creep cavities with dimensions of up to micron size were evident. In all microscopic studies cavities were associated with second phase particles and exhibited spherical as well as facetted structures.

11. The stress modified ductility exhaustion model revealed that damage in the series of creep tested specimen where prior plastic strain is induced is mainly a consequence of plastic hole growth rather from prestraining. Even though creep cavity nucleation happens continuously throughout creep life for all samples, this contributes only little to the overall damage.

8.2 Suggested future work

The studies carried out supporting this thesis have been investigated and demonstrated the potential for implementing 3D-DIC in long term high temperature tests to determine local creep deformation properties from a single test specimen. Procedures on how to use SANS for analysing creep damage measurements have also been established. There is wide scope for building on the work as outlined below.

1. Tests using strain controlled rather than load controlled settings for creep tests should be conducted to avoid cavity growth controlled by plastic hole growth rather than diffusion processes. A series of tests at varying constant strain rates could be used to further investigate and verify the SMDE model. A strain controlled creep test allows creep experiments to be conducted within one damage region, i.e. either the stress dependent region or the transition region which represents plant conditions.

2. The accuracy of the 3D-DIC technique could be further improved by replacing the furnace quartz glass with a sapphire glass window to reduce the effect of chromatic aberration. Furthermore a fan could be used to reduce the external heat flux that is a major source of fluctuations in the displacement data. The analysis approach (i.e.
averaging method, subset size or correlation method) to calculate the displacement data could be further improved and standardised.

3. Since creep cavitation damage has been observed to be relatively heterogeneous and the effects of grain orientation on cavity nucleation as well as on cavity growth are unclear, EBSD studies are recommended. These could be used to explore relationships between grain boundaries that are susceptible to creep cavity damage.

4. Alterations in the morphology of second phase particles due to stress and strain may significantly affect investigations of creep cavities in Type 316H stainless steel using SANS. The design of the hourglass sample is suitable to carry out an experiment where a load is applied at room temperature that leads to a varying plastic strain and a change in dislocation density. Subsequently, the load should be removed and the sample annealed at a temperature of 550°C. The experiment could be used to determine whether a change in dislocation density as a result of prior plastic strain affects the nucleation and growth of second phase particles within a time frame of some months. A similar experiment can be carried with the load applied at a temperature of 550°C.

5. A detailed TEM investigation where parts of the specimen are extracted using FIB could be used to verify conclusions made by SANS and SEM regarding the mean diameter as well as location of intergranular creep cavities. With the TEM studies dislocation structures could also be investigated to link changes in creep strain rate with changes in dislocation density. Subsequently, the creep strain rate behaviour that was observed for the series of prestrained samples could be further investigated.

6. The successful application of 3D-DIC to measure the surface strain on complex sample surfaces could be further extended to study the effect of stress triaxiality on creep ductility. This can be achieved by using a double notched sample with an hourglass gauge section in the middle. Since multiaxial loading is not possible to introduce in flat samples, a cylindrical sample and therefore the application of 3D-DIC is neces-
sary. Around each notch root, the creep cavitation damage can be determined using small angle neutron scattering and SEM and compared to a corresponding value measured along the hourglass gauge length where the creep strain can be determined using DIC.

7. Since neutron scattering experiments are only carried out within a diameter size range between 5 nm and 400 nm, additional usage of Ultra-High Resolution SANS could be applied to investigate cavities up to several micrometer. In addition, further quantitative work with the SEM could be carried out to verify findings from the neutron scattering.
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REFERENCES


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Figure A.1: Materials specification supplied by ELG Haniels.


B  Creep ductility trends

Figure B.1 depicts the creep ductility plotted against the normalised stress. The normalised stress is the ratio between applied stress and yield stress and characterises the amount of plastic strain that is induced in the sample as a consequence of loading. The model estimates the creep ductility trends based on an existing large data set of Type 316H stainless steel that covers a wide range of stress for material tested by EDF Energy at a temperature of 550°C. It also includes data from Type 316H stainless steel pre-compressed to several amounts [79]. Three different regions have been identified, very similar to the SMDE model. An upper and lower shelf where the creep ductility is constant and not dependent on stress. And a transition region that links the lower and upper shelf. In addition, in Figure B.1 the data from the series of creep tests conducted at the OU, with specimens prestrained in tension to 4%, 8%, 12% and 16% and described in Chapter 5 have been included. In Table B.1 the true stresses applied for each creep test are listed together with the $\sigma_{0.2}$ yield stresses obtained from the data of the tensile test at high temperature, see Figure 5.3.

Table B.1: Tensile and creep properties to calculate the normalised stress for CHS-0, CHS-04, CHS-08, CHS-12 and CHS-16 pretensioned to 4%, 8%, 12% and 16% respectively.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\sigma_{0.2}$ in MPa</th>
<th>$\sigma_{true}$ in MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHS-00</td>
<td>144</td>
<td>325</td>
</tr>
<tr>
<td>CHS-04</td>
<td>235</td>
<td>314</td>
</tr>
<tr>
<td>CHS-08</td>
<td>325</td>
<td>305</td>
</tr>
<tr>
<td>CHS-12</td>
<td>402</td>
<td>305</td>
</tr>
<tr>
<td>CHS-16</td>
<td>460</td>
<td>305</td>
</tr>
</tbody>
</table>

It could be found that CHS-08, CHS-12 and CHS-16 all fail close to the the lower shelf region. CHS-16 even falls below the estimated lower bound limit of 1%. An increase could be observed for CHS-0 and CHS-4 with 0% and 4% prestrain respectively. The creep ductility of CHS-0, the sample without prior plastic strain is somewhat comparable with the data of the EDF AR Database. The results obtained from this PhD work corroborate
the inferred trends from [79, 128]. The results from CHS-08, CHS-12 and CHS-16 with 8%, 12% and 16% prestrain respectively and normalised stresses lower or close to 1 can be found around the estimated lower bound limit of 1% for the ductility of Type 316H stainless steel that was estimated for a temperature of 550°C.

![Figure B.1: Comparison of the axial creep ductility data points for the AR and pre-compressed material with the estimated trends, compiled by [79]. In addition, the creep ductility data for the material tested at the OU in pretension was added.](image)

Figure B.1: Comparison of the axial creep ductility data points for the AR and pre-compressed material with the estimated trends, compiled by [79]. In addition, the creep ductility data for the material tested at the OU in pretension was added.