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γ'' variant-sensitive deformation behaviour of Inconel 718 superalloy

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Highlights

● This study reveals, for the first time, the micro-mechanism of deformation of γ'' strengthened superalloys using neutron diffraction.
● Inconel 718 samples with different γ'' variant distributions were prepared by different stress ageing heat treatments.
● The γ'' variant-sensitive deformation behavior was studied by interpreting the stress-lattice strain curves.
● Compressive-aged sample shows larger yield strength compared to the tensile-aged sample.
● The difference in strengthening arises from the interactions between dislocations and different γ'' variants.
Abstract

Strengthening in Inconel 718 superalloy is derived from dislocation interaction with \( \gamma'' \) precipitates, which exist in disk-shaped three possible orientation variants with their \{100\} habit plane normal to each other. The interactions between dislocations and \( \gamma'' \) precipitates vary according to the \( \gamma'' \) orientation variants, which makes the deformation behaviour complicated and difficult to reveal experimentally. In this work, \( \gamma'' \) variant distributions of Inconel 718 samples were tailored by ageing heat treatment under either tensile or compressive stress. The \( \gamma'' \) variant-sensitive deformation behaviours were then studied by \textit{in situ} tensile tests via neutron diffraction at room temperature. It is demonstrated that yielding first takes place in grains oriented with \(<110>\) parallel to the loading direction. An identical lattice strain response to applied stress of both the \( \gamma \) matrix and the \( \gamma'' \) precipitates was observed during yielding, suggesting that dislocations shearing through the \( \gamma'' \) precipitates is predominant at this stage. Variations in yield strength for samples with different \( \gamma'' \) variant distributions were observed, which can be attributed to different strengthening that arises from interactions between dislocation and different \( \gamma'' \) variants.

Keywords: Ni-base superalloys; Precipitation strengthening; Neutron diffraction; Lattice strains; Plastic deformation

1. Introduction

Inconel 718 (IN718) has been one of the most widely used Ni-base superalloys in aeroengines, nuclear, and oil & gas industries for decades [1, 2], and has become a popular candidate for applications in additive manufacturing nowadays [3–6]. Its popularity is attributed to its excellent mechanical properties at elevated temperatures up to 650 °C, remarkable good weldability, and machinability plus relatively low cost.
IN718 derives strengthening from the presence of γ' and γ'' precipitates that are embedded in the γ matrix [7]. The γ matrix phase displays a disordered face-centred cubic (FCC) structure, the γ' phase possesses an L12 ordered structure and the γ'' phase has a D022 body-centred tetragonal (BCT) structure [1]. It has been suggested that the major strengthening phase in IN718 is the γ'' owing to its larger volume fraction compared to the γ’ phase [8–10].

The γ'' phase maintains a cube-cube orientation with the γ matrix, or \{100\}_γ//\{100\}_γ and [100]_γ//<100>_γ [1]. The γ'' phase has a relatively large lattice misfit of ~3% with the γ along the c-axis, while a smaller misfit of < 1% with the γ along the a-axis. The different lattice misfits consequently result in the occurrence of disk-shaped γ'' precipitates in three orientations with the disk plane normal to each other [11].

Misfit between the phases not only determines the morphology of the precipitate but also plays an important role in affecting the coherency strengthening in IN718 [8]. During deformation, the localized stresses in the precipitate-matrix system will interact with dislocation motions and significantly affect the deformation behaviour. Understanding the deformation mechanisms of precipitation strengthening materials is necessary for predicting the mechanical properties and achieving optimum performance. For precipitation hardening Ni-base superalloys, strengthening is achieved by impeding the motion of dislocations by the coherent precipitates. At room temperature, dislocation shearing or bowing around the coherent precipitates are the dominant strengthening mechanism. For the γ''-Ni-base superalloys, such as IN718, the deformation mechanisms are complicated due to the tetragonal crystal structures of the γ''. Oblak et al. [8] suggested the appearance of quadruplets 1/2<110> dislocations which shear the three γ'' variants and restore the order of the γ'' structure in an underaged IN718 sample. Chaturvedi and Han [10] reported that the dislocations
appeared in pairs in underaged IN718 samples. Sundararaman et al. [9] proposed a transition of deformation mechanism from shearing by dislocations movements to shearing by the passage of true crystallographic deformation twins when the $\gamma''$ precipitates exceed a critical size ($\sim$10 nm). Recent studies using the phase-field simulation technique uncovered a variety of sophisticated deformation mechanisms that involve unique interactions between dislocations and an individual $\gamma''$ variant [12–14]. Although these investigations show inconsistency in the observed deformation mechanisms, clearly, they all suggest that the deformation mechanism is variant dependent. Indeed, recent studies have shown that the mechanical property of IN718 can be enhanced by tailoring the $\gamma''$ variant distribution [15–18].

Thanks to the developments of neutron sources and strain-scanning beamlines, *in situ* neutron diffraction has become a powerful complementary method to microstructure observations in studying the elastic properties and deformation mechanisms of crystalline materials [19, 20, 29, 21–28]. In contrast to transmission electron microscopy (TEM) observation, which only provides post-mortem microstructures on very small material volumes, neutron diffraction reveals the evolution of $hkl$ plane-specific lattice strains sampled from a relatively large gauge volume ($\sim$ tens of mm$^3$) during testing. For multi-phase materials, the phase-specific lattice strains can be obtained, provided that the diffraction patterns from the constituent phases are distinguishable.

However, challenges remain in studying $\gamma''$ when using neutron diffraction due to its low volume fraction, three variants distribution, and diffraction peaks overlapping. To tackle the difficulties, the stress-induced variant selection (SIVS) method was applied in this work. The $\gamma''$ precipitates experience SIVS when either a tensile or compressive stress was applied to IN718 during the ageing heat treatment stage. During
ageing, the applied stress interacts with the misfit strain of the $\gamma''$ particles and leads to a change in strain energy, which can be simplified as [30]:

$$\Delta E = -\sigma \times \varepsilon_c \times \cos^2 \theta \times V$$  \hspace{1cm} (1)

where $\sigma$ is the applied stress, $\varepsilon_c$ is the transformation strain along the $c$-axis, $\theta$ is the angle between the $c$-axis and the stress axis, and $V$ is the volume of the $\gamma''$ variant, as illustrated in Fig. 1. In an individual grain, if a variant’s $\theta$ is smaller compared to that of the other variants, this variant will be promoted in the case of tensile stress applied or suppressed when a compressive is applied. In terms of the extent of variant selection, it is a function of grain orientation, sign and magnitude of applied stress, ageing temperature, and duration of ageing. For example, the grain with its [111] parallel to the stress axis will experience no variant selection, as the $\theta$ is identical for all three variants. While the [100] oriented grain is the most vulnerable to the variant selection, as $\theta$ is 0° for one variant and 90° for the remaining two. The detail study of variant selection related to temperature, stress, and grain orientation can be found in Ref. [16]. Following the SIVS ageing heat treatment, the samples with selected variant distributions were taken to ENGIN-X Time-of-Flight neutron diffractometer for in situ tensile experiments. The benefits of selected $\gamma''$ variant distributions in the current work are twofold: first, it makes the $\gamma''$ diffraction peak discernible from neutron diffraction. For a sample without variant selection, two diffraction peaks of gamma double prime exist due to the different lattice parameters along $a$- and $c$-axes. In contrast, for a variant selected sample, the gamma double prime diffraction peak includes all the intensity that arises from the precipitates’ volume in these grains and has higher intensity [30–32]. And second, it makes the contributions of different $\gamma''$ variants in strengthening distinguishable, and thus, their respective effect is studied.
Fig. 1. Illustration of the angle $\theta$ between stress axis and $c$-axis of the $\gamma''$ precipitate [30].

2. Methodology

2.1. Sample preparation

In the current work, polycrystalline IN718 alloy with a nominal chemical composition listed in Table 1 was used. The alloy was manufactured via vacuum induction melting, electro-slag re-melting, and vacuum arc re-melting processes. The ingot underwent high-temperature homogenization with $1160 \, ^\circ C / 24 \, h + 1190 \, ^\circ C / 72 \, h + \text{air cooling}$, before radially forging into a bar of 220 mm diameter. Rod specimens were extracted from the forged IN718 bar and then solution heat-treated at $980 \, ^\circ C$ for 1 h followed by forced-air cooling to room temperature (RT). Rod specimens were machined to test bars with M12 thread heads, 42 mm gauge length, and 8 mm gauge diameter. Finally, test bars were subjected to ageing heat treatment at $790 \, ^\circ C$ for 5 h either with 300 MPa tensile or compressive stress applied, which resulted in $\gamma''$ variants selected.

Substantial research has shown that during ageing heat treatment when the platelet precipitation happens, the applied stress will have an impact on the selection of the orientation variant of the precipitates. These studies have proposed that the variant selection results from the different interaction energies between an applied stress and
transformation strains of different variants [8, 33–36]. The favoured growth and coarsening happen to the variant that has the most reduction in strain energy. The volume fraction of the selected variant is increased at the expense of the other variants. The benefits of selected $\gamma''$ variant distribution in the current work are twofold: first, it makes the $\gamma''$ diffraction peak discernible from neutron diffraction [30–32], and second, it makes the contributions of different $\gamma''$ variants in strengthening distinguishable, and thus, their respective deformation behaviour during tensile loading can be studied.

**Table 1** Chemical composition of IN718 alloy (in wt.%).

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Nb</th>
<th>Ti</th>
<th>Al</th>
<th>Mo</th>
<th>Fe</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.023</td>
<td>18.05</td>
<td>5.42</td>
<td>0.91</td>
<td>0.48</td>
<td>2.90</td>
<td>18.00</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

2.2. Microstructure characterization

Microstructure characterization focusing on the $\gamma''$ variant distribution after ageing heat treatment was performed on the cross-section of the test bars. The characterization was carried out using a JSM-7800F (JEOL, Japan) scanning electron microscope (SEM) equipped with an electron backscatter diffraction detector (EBSD). The FE-SEM samples were electro-polished at 20 V in an electrolyte solution of 20 % H$_2$SO$_4$ + 80 % CH$_3$OH, followed by etching in a solution of 150 mL H$_3$PO$_4$ + 10 mL H$_2$SO$_4$ + 15 g CrO$_3$. Crystal orientation mappings were first performed, then secondary electron imaging mode was used to characterize the $\gamma''$ variant distribution in the grains oriented with $<110>$, $<100>$, and $<111>$ closely parallel to the loading direction.

2.3. In situ neutron diffraction experiment

The *in situ* neutron diffraction tensile deformation experiment was performed using the ENGIN-X time-of-flight neutron diffractometer at ISIS neutron pulse source, UK.
The bar sample was horizontally mounted on a stress rig (INSTRON, 100 kN) with its longitudinal direction at 45° relative to the incident neutron beam. Two banks of detectors were located at $2\theta = \pm 90^\circ$, allowing $d$-spacing along the longitudinal and transverse directions to be measured simultaneously. To maximise the diffracted signal, a large nominal gauge volume (NGV) defined by the incident beam slit (10 mm in height and 4 mm in width) and the radial collimator (4 mm) was used. Since the NGV was not fully immersed in the sample, the sample was carefully positioned to ensure the centroid of the NGV lay on the axial line of the sample to minimise pseudo-strain effects. Tensile loading was applied under stress control mode with a stress rate of 10 MPa/s and held at each measurement point. Diffraction measurement was performed at every 200 MPa with an acquisition time of 20 min during the elastic regime. Measurement points were increased by reducing the load steps to every 50 MPa when the applied load reached 800 MPa, which is around the yield point. The experimental setup is illustrated in Fig. 2.

**Fig. 2.** Schematic illustration of *in situ* neutron diffraction tensile deformation experiment in ENGIN-X, ISIS neutron source, UK.
3. Results

3.1. Selected variant distribution

Inverse pole figures (IPF) viewed along the z-axis which is consistent with the loading direction, are shown in Fig. 3(a, e) for the tensile-aged (hereinafter TA sample) and compressive-aged (CA sample) samples, respectively. Fig. 3(b–d) shows the γ'' variant distribution in grains labelled with ‘b’, ‘c’, and ‘d’ in Fig. 3(a), which are oriented closely to <111>, <100>, and <110> directions, respectively. Similarly, Fig. 3(f–h) shows the variant distribution in the CA sample. The γ'' variants are represented with the different colours shown in the insets with their orientation relationship with the matrix. For the <111> oriented grain shown in Fig. 3(b), the three variants are present in approximately equal quantities. For the <100> oriented grain in Fig. 3(c), only the variant with the disk-plane perpendicular to the observation direction is present. For the <110> oriented grain in Fig. 3(d), two orientationally equivalent variants are present. For the <111> oriented grain shown in Fig. 3(f), the three variants are present in approximately equal quantities as in Fig. 3(b). For the <100> oriented grain in Fig. 3(g), two variants whose disk-plane is normal to each other and parallel to the observation direction are present. For the <110> oriented grain Fig. 3(h), only the variant whose disk plane is parallel to the observation direction is present. From all six micrographs, the averaged long axis of the γ'' precipitate is measured to be 56 ± 5 nm, with no significant difference between grains or the two samples. Besides the γ'' precipitates, smaller spherical particles in diameters less than 30 nm are observed, which are believed to be the γ' precipitates. According to Refs. [38, 39], the volume fraction of the γ'' phase is generally not greater than 15%, while the micrographs show a large density of γ'' precipitates with its volume fraction looking very high. This is because the
micrographs taken after electropolishing have eliminated the gamma matrix and may have exposed multi-layers of precipitates.

![Image](image_url)

**Fig. 3.** (a) IPF along the z-axis on the cross-section of the TA sample, (b)–(d) γ” variant distribution in grains selected from locations indicated in (a), (e) IPF on the cross-section of the CA sample, (f)–(h) γ” variant distribution in grains selected from locations indicated in (e). Insets show the orientation of existing γ” variants (indicated as either coloured ellipse or circle) with habit planes lying on different faces of the grey cube according to the {100}<100>γ, and [100]γ/[100]γ’ relationship with the matrix phase.

3.2. **Diffraction pattern and fitting**

Diffraction patterns obtained by bank 1 detectors at RT before loading for both the TA and CA samples are shown in Fig. 4. One can see the four major peaks (\{111\}, \{200\}, \{220\}, and \{311\}) from the γ phase. Two peaks from the δ phase are also noticed in the patterns. Comparing the two patterns, it is noted that peaks from the γ” phase (\{004\}, \{204\} and \{116\}) are captured in the pattern for the TA sample, but are not apparent in the pattern for the CA sample. \{112\} γ” peak presents at the right shoulder of the \{111\} γ peak in both patterns.
Fig. 4. Diffraction patterns obtained along the loading direction at room temperature (RT) before loading both tensile-aged (TA) and compressive-aged (CA) samples. Small \{116\}, \{204\}, and \{004\} \gamma'' peaks are discerned in TA sample but not in CA sample.

The differences between the two diffraction patterns related to the \gamma'' phase are attributed to the different \gamma'' variant distribution in the two samples. For example, Fig. 5 illustrates the diffraction on \textless 100 \textgreater oriented grains in the two samples. In the \textless 100 \textgreater oriented grains of the TA sample, only the variant with the disk-plane perpendicular to the longitudinal direction is present. Diffraction from this variant contributes to the \{004\} \gamma'' peak, which is located at the right of the \{200\} \gamma peak due to the larger lattice parameter of the \gamma'' phase than that of the \gamma phase. In contrast, in the \textless 100 \textgreater oriented grains of the CA sample, only the variants with its disk-plane parallel to the longitudinal direction are present. Diffraction from these variants contributes to the \{200\} \gamma'' peak. Since the lattice parameter of the \gamma'' phase is very close to that of the \gamma phase along its \alpha-axis, the \{200\} \gamma'' peak overlaps with the \{200\} \gamma peak. Similar diffraction happens to the \textless 110 \textgreater and \textless 311 \textgreater oriented grains.
Fig. 5. Schematic illustration of diffraction from an <100> oriented grain in TA and CA samples, respectively, viewing along the transverse direction of the bar sample. Non-overlapping {004} γ'' peak is contributed from the variant with a c-axis parallel to the diffraction vector while the {200} overlapping peak is from the variant with an a-axis parallel to the diffraction vector.

Since the γ'' peaks are discernible in the TA diffraction pattern, diffraction peaks of both the γ and γ'' peaks were fitted by using Pseudo-Voigt functions [40–42]. γ' peaks were not considered to have large influences on the current peak fitting due to their low volume fraction and close lattice parameter to that of the γ phase. The non-overlapping γ and γ'' peaks were fitted as shown in Fig. 6(a–c), the respective γ-to-γ'' intensity ratio was then obtained. For the CA sample where the γ and γ'' peaks are overlapped, peak deconvolution using two peak functions was performed. The γ-to-γ'' peak intensity ratio was constrained to the value obtained from the non-overlapping peaks from the TA sample, leaving other fitting parameters free to fit. Such an approach worked well for the three peaks shown in Fig. 6(d–f) except for the {111}γ/{112}γ'' partially overlapping
peak. Diffraction peaks obtained at each stress increment were fitted using the same procedure. The fitting sensitivity to the constrained intensity ratio was checked by trying various values with $+/-10\%$ off. It resulted in a small shift of $d$-spacing of the $\gamma''$ peak, but the small shift was cancelled out when lattice strain evolution was considered. Therefore, the fitting method was considered reliable for the current work.

![Fig. 6](image)

**Fig. 6.** Fitting of diffraction peaks for (a)–(c) non-overlapping peaks for TA sample, and deconvolution results for (d)–(f) overlapping peak for CA sample, showing the accuracy of the fitting.

3.3. Elasticity of $\gamma''$

The $d$-spacing $d_{hkl}$ of each $hkl$ reflection for both the $\gamma$ and $\gamma''$ phases was obtained from fitting each $hkl$ reflection. The elastic lattice strain was calculated by:
where $d_{hkl}^0$ is the $d$-spacing for a specific $h\ell k$ reflection measured at the beginning of the test. The lattice strain against applied stress at the elastic regime was obtained and plotted in Fig. 7. The DEC of each $h\ell k$ reflection $E_{h\ell k}$ of both phases was obtained by linearly fitting each of the plots. The results are listed in Table 2 and Table 3.

\[
\varepsilon_{h\ell k} = \frac{d_{h\ell k} - d_{h\ell k}^0}{d_{h\ell k}^0}
\]

**Fig. 7.** Stress-lattice strain plots for each $h\ell k$ reflection of both the $\gamma$ and $\gamma''$ phases in the elastic regime. (a)–(e) for TA sample, (d)–(f) for CA sample. Diffraction elastic constants were obtained by linearly fitting to each of the plots. It is shown that the $\gamma''$ is stiffer along its $c$-axis ($<004>$ direction) than $a$-axis ($<200>$ direction), as well as stiffer than the matrix phase. Errors bars associated with the fitting uncertainties are about the size of the symbol and are not shown for clarity.

**Table 2** Diffraction elastic constant for each $h\ell k$ reflection of the $\gamma''$ phase, uncertainties associated with the linear fitting are given. $h\ell k$ reflections for the TA sample are indicated as (TA) while for the CA sample are indicated as (CA).
<table>
<thead>
<tr>
<th>$E^{hkl}$ (GPa)</th>
<th>215</th>
<th>246</th>
<th>227</th>
<th>214</th>
<th>164</th>
<th>225</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uncertainty (GPa)</td>
<td>13</td>
<td>22</td>
<td>24</td>
<td>18</td>
<td>2</td>
<td>22</td>
</tr>
</tbody>
</table>

Table 3 Diffraction elastic constant for each $hkl$ reflection of the $\gamma$ phase, uncertainties associated with the linear fitting are given.

<table>
<thead>
<tr>
<th>$hkl$ of $\gamma$</th>
<th>311 (CA)</th>
<th>311 (TA)</th>
<th>220 (CA)</th>
<th>220 (TA)</th>
<th>200 (CA)</th>
<th>200 (TA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E^{hkl}$ (GPa)</td>
<td>200</td>
<td>189</td>
<td>229</td>
<td>226</td>
<td>165</td>
<td>157</td>
</tr>
<tr>
<td>Uncertainty (GPa)</td>
<td>2</td>
<td>2</td>
<td>5</td>
<td>2</td>
<td>2</td>
<td>3</td>
</tr>
</tbody>
</table>

The $\gamma''$ phase possesses a BCT crystallographic structure, the DECs along the $a$-axis and $c$-axis are different when comparing the $E^{004}$ with the $E^{200}$ (225 GPa vs 164 GPa), bearing in mind that the {004} plane is normal to the $c$-axis, while the {200} plane is normal to the $a$-axis. The difference implies that the D0$_{22}$ $\gamma''$ is stiffer along its $c$-axis than along the $a$-axis, although the constraints imposed on each grain/phase of a multi-phase polycrystalline material mean that the DECs do not represent the intrinsic elastic properties. In contrast, $E^{204}$ and $E^{220}$ of the $\gamma''$ phase show no significant differences. By comparing the DECs of the $\gamma''$ with those of the $\gamma$ phase (Table 3), it is shown that $\gamma''$ is stiffer than the $\gamma$ matrix phase ($E^{116}_{\gamma''}$ vs $E^{311}_{\gamma}$ and $E^{004}_{\gamma''}$ vs $E^{004}_{\gamma}$).

### 3.4. Macroscopic stress–strain curves

The applied stress–strain curves for the two samples recorded during in situ neutron diffraction tensile experiment are displayed in Fig. 8. Note that the complete curves are not shown due to the extensometer slipping at large plastic strain, the fracture took place at an applied stress of 1289 MPa and 1342 MPa for the TA and CA samples, respectively. The CA sample exhibits a larger yield strength and tensile strength than
the TA sample. The variation in strengthening by different stress-ageing processes is confirmed by a tensile experiment performed in accordance with the ISO 6892-1 2011 standard, and also agrees well with those reported by Qin et al. [15]. The 0.2% yield strength and tensile strength are listed in Table 4. The 0.2% yield strength of the in situ neutron diffraction tested samples is higher compared to their counterpart tested in-house, this can be attributed to the step manner loading during the in situ experiment, although the tensile strength is similar. The microstructures of the two samples are similar in terms of grain size, γ” precipitate volume fraction and size, and presence of the δ, the major difference is the different stress-induced γ” variant distribution. Therefore, it is reasonable to speculate that the differences in mechanical performance are due to the different γ” variant distributions, details will be discussed in Section 4.2.

![Applied stress–strain curves for quasi-static in situ neutron tensile deformation and in-house laboratory tensile deformation of the two differently aged samples.](image)

**Fig. 8.** Applied stress–strain curves for quasi-static in situ neutron tensile deformation and in-house laboratory tensile deformation of the two differently aged samples. The ‘x’ marker shows the point where the extensometer slipped.

**Table 4** Summary of 0.2% yield strength and tensile strength of the two differently aged samples tested by in situ neutron diffraction experiment and in-house tensile experiment.

<table>
<thead>
<tr>
<th></th>
<th>0.2% yield strength (MPa)</th>
<th>Tensile strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TA - Neutron</td>
<td>990</td>
<td>1289</td>
</tr>
</tbody>
</table>

16
3.5. Lattice strain evolution

The \( \gamma \) phase \( hkl \)-specific lattice strain evolutions measured along the longitudinal are shown in Fig. 9(a, b). The stress-lattice strain curves give the lattice response to the applied stress during the tensile deformation, which reveals the deformation behaviour at the grain scale. The stress-lattice strain curves for the two samples are similar, showing that the 111 and 311 reflections maintain linearity through the deformation, while the 200 and 220 reflections possess nonlinearity at some stage. The nonlinearities are brought about by changes in the intergranular stresses. 220 reflections show reduced strain accumulation, while 200 reflections show increased strain accumulation after the onset of the nonlinearities. The onset of nonlinearity of 220 reflections characterizes the onset of crystallographic slip in the \(<110>\) oriented grains and therefore acts as an indicator of the onset of plasticity at the microscale. The onset of nonlinearity of 220 reflections for the TA sample is observed to be at 900 MPa, while for the CA sample, it is 1000 MPa. These onsets of nonlinearity match well with that of the macroscopic stress–strain curves in Fig. 8.

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
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</thead>
<tbody>
<tr>
<td>CA - Neutron</td>
<td>1095</td>
<td>1342</td>
</tr>
<tr>
<td>TA - In house</td>
<td>910</td>
<td>1245</td>
</tr>
<tr>
<td>CA - In house</td>
<td>1000</td>
<td>1322</td>
</tr>
</tbody>
</table>
**Fig. 9.** Evolution of $hkl$-specific lattice strain of the $\gamma$ phase along the longitudinal direction for (a) TA and (b) CA samples. The horizontal lines indicate the onsets of nonlinearity, which coincide with the 0.2% yield strength obtained from macro stress–strain curves. The error bars associated with uncertainties from peak fitting are about the size of the symbol and are not shown here for clarity.

Fig. 10 shows the lattice strain evolution of both the $\gamma$ and $\gamma''$ phases in the $<100>$ and $<110>$ oriented grains along the loading direction during deformation.

**Fig. 10.** Lattice strain evolution of the $\gamma$ and $\gamma''$ phases for (a) TA and (b) CA samples during tensile deformation. The horizontal lines indicate the onsets of nonlinearity. Error bars associated with uncertainties of peak fitting are about the size of the symbol in the elastic regime for both phases, and increase from 200 to 1200 microstrains in the
plastic regime for the $\gamma''$, 100 to 360 microstrains for the $\gamma$ phase. Error bars are not shown for clarity.

The lattice strain responses can be summarized: (i) In the $<110>$ oriented grains of the TA sample (green plots in Fig. 10(a)), the $\gamma''$ lattice strain increases similar to that of $\gamma$ in the elastic regime and at the early stage of plastic deformation up to 1100 MPa. With further loading, the $\gamma''$ phase experienced a larger strain accumulation compared to the $\gamma$ phase. (ii) Similarly, in the $<110>$ oriented grains of the CA sample (green plots in Fig. 10(b)), the $\gamma''$ lattice strain increases similar to that of $\gamma$ in the elastic regime and at the early stage of plastic deformation up to 1200 MPa. With further loading, the $\gamma''$ phase experienced a larger strain accumulation compared to the $\gamma$ phase. (iii) In contrast, in the $<100>$ oriented grains of the TA sample (blue plots in Fig. 10(a)), the $\gamma''$ lattice strain evolution separates from that of the $\gamma$ phase in the elastic regime, which is due to the different elasticity as pointed out previously. Both phases experienced increased strain accumulation during plastic deformation. (iv) In the $<100>$ oriented grains of the CA sample (blue plots in Fig. 10(b)), the $\gamma''$ lattice strain increases together with the $\gamma$ up to 1000 MPa. Then, the $\gamma''$ phase accumulates little strain increase between 1000 and 1200 MPa, followed by strain accumulation again until fracture. The $\gamma$ phase keeps accumulating elastic strain until fracture.

3.6. Peak width evolution and grain rotation

In addition to the lattice strain evolution, the peak broadening during tensile deformation provides important information to understand plastic deformation. The evolutions of normalized full width at half maximum intensity (FWHM) of the $\gamma$ and $\gamma''$ reflections from the $<100>$ and $<110>$ oriented grains of the two samples are shown in
Fig. 11(a, b). Peak broadening can have several origins associated with the microstructure of the sample, including nano-sized particles, dislocations, stacking faults, microstrain, and steep macrostrain gradient [40]. Peak broadening during plastic deformation can be attributed to the following reasons: (i) dislocations and stacking faults break the entity of a crystalline particle, leading to a smaller diffracting size and resulting in particle size broadening; (ii) dislocations and stacking faults generate type-III microstrain fields that give rise to broadening; (iii) grain interaction stresses caused by plastic anisotropy [40]. The dislocations and stacking faults related to peak broadening are grain orientation-specific since they may preferentially occur in specifically oriented grains during plastic deformation. Whilst, the grain interaction stresses peak broadening is not highly dependent on orientation. Therefore, the peak broadening can act as a qualitative indicator of plastic deformation.

In Fig. 11(a, b), it is shown that negligible peak broadening occurs in the elastic regime. The onset of noticeable peak broadening closely correlates with the onset of the nonlinearities of lattice strain evolution. The peak broadening of γ reflections is similar to each other but not as significant as the broadening of γ” reflections. For the TA sample (Fig. 11(a)), 204 γ” reflection broadens more significantly than the 004 γ” reflection. For the CA sample (Fig. 11(b)), both γ” reflections broaden significantly. The peak broadening associated with plastic deformation of specific γ” variants during tensile loading will be discussed in Section 4.1.

Grain rotation occurred during plastic deformation as a result of slips. The grain rotation can be observed by the evolution intensity of the γ reflections, as shown in Fig. 11(c, d). The intensity of each peak remained unchanged in the elastic regime, indicating that there is no grain rotation during elastic deformation. The onset of change in intensity coincides with the onset of the nonlinearities in Fig. 10 and the onset of
peak broadening in Fig. 11(a, b). For both samples, the intensity of 220 reflections decreases during plastic deformation whilst the intensities of 200 and 111 reflections increase. This indicates that the original <110> oriented grains rotate to other orientations, and <100> and <111> oriented grains increase due to the rotation of the other oriented grains.

**Fig. 11.** Evolution of normalized peak broadening of the $\gamma$ and $\gamma''$ phases in the {200} and {220} oriented grains for the (a) TA and (b) CA samples. And peak intensity evolution for (c) TA and (d) CA samples. Error bars for peak width associated with uncertainties of peak fitting are about the size of the symbol in the elastic regime and increase from 0.07 to 0.44 in the plastic regime for both phases. Error bars for peak intensity are about the size of the symbol. Error bars are not shown for clarity.
4. Discussion

4.1. Localised deformation mechanism

Various deformation mechanisms associated with the γ'' precipitate were proposed based on TEM observations or simulations: dislocation shearing (anti-phase boundary (APB) shearing, stacking fault (SF) shearing, micro-twinning shearing, etc.) and Orowan looping [8–10, 12–14]. Some of these deformation mechanisms may be active during deformation at different stages or in different γ'' variants. For example, Sundararaman et al. [9] observed dislocations associated with APB shearing and SF shearing in samples subjected to small deformation (2% plastic strain) and micro-twinning shearing in samples with large plastic deformation. By the combination of ab initio calculation and phase-field simulation, Lv et al. [13] discovered that a partial dislocation loop can be left around the precipitate belonging to certain variants sheared by dislocations.

Although the detailed interactions between dislocations and precipitates cannot be revealed by neutron diffraction, lattice strain response to the applied stress during deformation can act as a trace of the predominant deformation behaviour: (1) when dislocations from the matrix enter the precipitate and cut through it, the precipitate and the matrix deform jointly and would not carry extra loads. As a result, both phases accumulate little increase in lattice strain with increasing stress. This is the case for the <110> oriented grains in the two samples at the early stage of plastic deformation, during which the lattice strain of both phases goes straight up (green plots in Fig. 10(a, b)). Meanwhile, loads transfer from the yielding grains to those unyielded grains, i.e., <100> oriented grains, as shown in blue plots in Fig. 10(a, b) that experienced accelerated lattice strain accumulation. (2) With further deformation, dislocation shearing the precipitates becomes more difficult whilst dislocation bypassing becomes
the dominant process of deformation. When this happens, loads transfer from the matrix to the γ'' phase, as observed from the green plots in Fig. 10(a, b) at larger plastic deformation when the γ'' phase accumulates a larger lattice strain than the matrix phase. The transition from shearing to bypassing is yet unclear. Two possible mechanisms can be the candidates to account for the transition: a) although the shearing mechanism is predominant at the early stage, bypassing can be operating at the same time to a less extent given that the critical resolved shear stresses (CRSSs) for each deformation mode are not in much different and vary depending on precipitate size. The left-behind dislocation loops generate by bypassing accumulation and make the shearing more difficult and the transition can happen. b) According to Lv et al. [13], dislocation shearing can leave partial dislocation loops around precipitate. These partial dislocation loops can hinder the following dislocation from entering the sheared precipitate and bypassing becomes more favourable. (3) The lattice strain of both phases in the <100> oriented grains of the TA sample displays accelerated accumulation during plastic deformation until failure (blue square plots in Fig. 10(a)), indicating that both phases in these grains carry extra loads transferred from those yielded grains but preserve elastically deformed. This is also evident from the small peak broadening of the two phases shown in Fig. 11(a). In contrast, the lattice strain evolution of the two phases in the CA sample differs from each other at the onset of yielding (blue plots in Fig. 10(b)) accompanied by large peak broadening of the γ'' phase (Fig. 11(b)), suggesting that the γ'' phase is being yielded while the matrix remains unyielded in the <100> oriented grains. This acts as a sign of microtwinning that is confined in the γ'' precipitates yet does not propagate into the matrix. Intrinsic stacking faults can be common in large γ'' precipitates due to ultralow energy (~2.3 mJ/m²) [13, 43] and serve as a precursor of microtwinning [13]. Indeed, microtwinning has been reported as the deformation
mechanism by Sundararaman et al. [9] from their observations of a great majority of such microtwins in $\gamma''$ and an estimation of a lower CRSS than that for either dislocation shear or bypassing mechanisms. In addition, McAllister et al. [14] also observed microtwinning as a prominent deformation mode at elevated temperatures in commercially aged IN718. Both studies revealed that the microtwins are conserved in the $\gamma''$ but not extended into the matrix. It is interesting to note from the lattice strain evolution that the microtwinning occurs in the $<100>$ oriented grains where the disk plane of $\gamma''$ is parallel to the applied stress (Fig. 10(b)), yet does not happen in those $\gamma''$ with disk plane normal to the applied stress (Fig. 10(a)). The underlying mechanism related to the different behaviour remains unknown and is worth further investigation.

The current work focuses on using the in situ neutron diffraction to reveal the deformation behaviour during tensile deformation in a relatively large gauge volume. Attempts to obtain dislocation structure micrographs via transmission electron microscopy (TEM) on the fractured samples were not successful. Challenges come from the very high dislocation density in the fractured samples as well as the multi-mechanisms that happened during deformation, micrographs showing vast dislocations do not help to unravel the micro-mechanism in a conclusive way. One way to tackle this would be to obtain micrographs on tensile samples interrupted at different plastic strains. In addition, the crystallographic orientation of the TEM foils concerning the loading direction has to be carefully selected to reflect the grain specific deformation behaviour. Nevertheless, the lattice strain evolution of both the matrix and precipitate phases bridges the gap between the micro and macro scale of deformation behaviour in a more representative manner thanks to the large gauge volume scanned and the selective characteristics of grains and phases.
4.2. \( \gamma'' \) variant-sensitive strengthening

It is shown in Section 3.4 that the yield strength of the CA sample is greater than that of the TA sample. Despite microstructures showing that the former possesses a slightly larger grain size, the major difference remains in the different \( \gamma'' \) variant distributions, which is the most likely reason accounting for the different levels of strengthening. Indeed, substantial studies have pointed out that the interaction between dislocations and \( \gamma'' \) precipitates is variant-dependent, although the detailed underlying mechanisms vary. Oblak et al. [8] and Chaturvedi et al. [10] observed pairs and/or quadruplets of \( 1/2<110> \) dislocations that interact with variants differently. A recent study using transmission electron microscopy found no evidence of such pairs or quadruplets \( 1/2<110> \) dislocations but the existence of \( 1/6<112> \) dislocations [12].

Phase-field simulation by the same group showed that the \( 1/6<112> \) dislocations, which are a result of \( 1/2<110> \) dislocation pair with different burgers vectors (i.e., \( 1/2[110] \) and \( 1/2[101] \)), are more likely the operative dislocations that shear the three variants uniquely [12–14], although the simulation did not take the coherency stress of the precipitates into account.

An estimation of the CRSS increment with variant dependence based on coherency strengthening was developed by Oblak et al. [7]. According to the coherency strengthening mechanism, the misfit stress fields that arise from the large tetragonal lattice misfit between the \( \gamma'' \) and \( \gamma \) will interact with dislocations [44]. The stress field of an ellipsoid is rather complex, making the description of the interaction between dislocations and particle stress field difficult. Oblak et al. [7] simplified the problem by assuming that the dislocation was a straight line that had average stress at the centre of the particle. Considering the \( \gamma'' \) phase whose misfit is tetragonal, there is a major strain axis (which is parallel to its \( c \)-axis) and a minor strain axis (along which the strain can
be neglected), an active dislocation does not interact equally with all the three \( \gamma'' \) variants. Instead, a stronger interaction occurs when the angle between the Burgers vector of the dislocation and the strain axis of the precipitate is smaller, and vice versa. The estimation successfully matched the experimental difference in CRSS increment between samples with different variant distributions [8], which was IN718 in single crystal form and aged with either a tensile or compressive stress along its [001] direction in a similar way to this work.

It is worthy to point out that the CRSS increment in the TA and CA samples in the form of a single crystal is opposite to our findings, which is that the TA single crystal sample gained a larger CRSS increment than the compressive one. The different findings between polycrystal (this work) and single crystal [8] can be attributed to their different active slip systems. In fact, the CRSS increment in the current work can be explained using Oblak’s equation, which will be shown as follows.

Fig. 12 schematically illustrates the active slip systems with different Burgers vectors and \( \gamma'' \) precipitates in the grain-oriented with [011] parallel to the loading direction. Note that for the TA sample, Fig. 12(a) shows the presence of only the [001] and [010] \( \gamma'' \) variants whose \( c \)-axis is 45\(^{\circ} \) to the applied load. Conversely, for the CA sample, Fig. 12(b) shows the presence of only the [100] \( \gamma'' \) variant whose \( c \)-axis is 90\(^{\circ} \) to the applied load. The four active slip systems, whose Schmid factors are the largest among the twelve slip systems, with different Burgers vectors are shown. For the TA sample, when the dislocations approach the \( \gamma'' \) precipitates, the dislocations whose Burgers vectors are \( \mathbf{b} = 1/2[\bar{1}01] \) and \( \mathbf{b} = 1/2[101] \) will have strong interactions with the [001] variant and weak interactions with the [010] variant, the reason is that their Burgers vectors are at 45\(^{\circ} \) to the major strain axis of the [001] variant and at 90\(^{\circ} \) to the [010] variant. For the interaction between dislocations of the other two slip systems and
precipitates, the strong/weak interactions swap for the same reason. In contrast, for the CA sample (Fig. 12(b)), dislocations of all the four slip systems will have strong interactions with the [100] variant since the Burgers vectors are all at 45° to the major axis of the precipitate. In other words, the dislocation/precipitate interaction ‘efficiency’ is doubled in the CA <110> oriented grains compared to the TA <110> oriented grains, contributing to a larger increment in the CRSS in the CA sample than that in the TA sample.

**Fig. 12.** Schematic illustrations of active dislocations (arrows in blue and red) and existing γ'' variants in grains oriented with [011] parallel to loading direction in the (a) TA sample and (b) CA sample.

According to the coherency strain strengthening mechanism, the increment in CRSS arose from dislocation shearing the γ'' particle is given by [8]:

\[
\Delta \tau = 1.7G|\varepsilon|^{3/2} \left[ \frac{h^2f \beta}{2br} \right]^{1/2}
\]

(3)
where $G$ is the shear modulus of the $\gamma$ matrix, $\varepsilon$ is the stress-free transformation strain along the normal of the $\gamma''$ precipitate, $b$ is the magnitude of Burgers vector of the dislocation shearing the $\gamma''$ precipitates, $f$ is the volume fraction of the $\gamma''$ precipitates, $R$ is the half of the major axis of the $\gamma''$ precipitates, and $h$ is the half-thickness of the ellipsoidal. $\beta = 1/2$ is for the half interaction efficiency in the TA $<110>$ oriented grains where 2 variants are present, or $\beta = 1$ is for the full interaction efficiency in the CA $<110>$ oriented grains where only 1 variant is present. $\varepsilon$, $R$, and $h$ have been measured and reported in our previous work [31]. $I'$ is derived from the intensity of $\{220\}$ $\gamma$ peak and $\{204\}$ $\gamma''$ peak from neutron diffraction of the TA sample with scattering structure factor taken into account:

$$\frac{I'}{I'} = \frac{(\lambda' f' \eta')^2 f' y}{(\lambda f \eta)^2 f y}.$$  

where $I$ is the intensity, $\lambda$ is the wavelength of the diffraction neutron beam, $\eta$ is the scattering density, and the detailed calculation is referred to in Ref. [30]. $G$ and $b$ are adopted from Ref. [8]. The parameters for the calculation are listed in Table 5.

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<td>70 GPa</td>
<td>0.254 nm</td>
<td>0.08</td>
<td>0.031</td>
<td>28 nm</td>
<td>6.1 nm</td>
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The increment in CRSS for the TA and CA samples was calculated to be $\Delta \tau_T = 213$ MPa and $\Delta \tau_C = 301$ MPa, respectively. The difference in $\Delta \tau$ between the two samples is $\Delta \tau_C - \Delta \tau_T = 88$ MPa, considering the Schmid factor $m = 0.408$ for these active slip systems, the difference in yield strength is $\Delta \sigma_{\text{yield}} = \frac{\Delta \tau_C - \Delta \tau_T}{m} = 216$ MPa. This calculated value is larger than the experimental difference (110 MPa from Table 4) by 96%. The discrepancy could be because the extent of variant selection was not that
complete in the <110> oriented grains. Evidence can be found with careful observation of Fig. 3(d, h), in which a very small portion of the residual variants can be found. In fact, the degree of variant selection is a function of ageing temperature, ageing time, stress applied, and grain orientation [16]. Therefore, the $\beta$ is probably slightly larger than 1/2 for the TA sample and smaller than 1 for the CA one. With $\beta = 0.6$ for the former and $\beta = 0.9$ for the latter, a value of $\Delta\sigma_{\text{yield}} = 129$ MPa is achieved, which agrees well with the experimental value.

Mechanical property is highly dependent on microstructure which is an outcome of the thermomechanical history. Among the sophisticated microstructures, the variant distributions have a direct effect on the coherency strengthening. The variant-dependent effect brings several implications. If such an alloy was aged under a high level of stress, which could happen in additively manufactured IN718 parts where localized residual stresses can be very large, the $\gamma''$ variant distribution could be profoundly inhomogeneous, resulting in an unexpected mechanical performance. On the other hand, mechanical performance can be improved by deliberately tailoring the $\gamma''$ variant distribution, i.e., direct ageing of cold-rolled IN718, during which a high level of residual stress is induced [17].

5. Summary

This work reports the deformation behaviour related to the $\gamma''$ strengthening phase of IN718 in situ for the first time. This in situ neutron diffraction work bridges the grain- and phase-specific micro-mechanisms and macro deformation behaviour via sampling a highly representative and relatively large gauge volume. In addition, the effect of the specific $\gamma''$ variant on the yield strength was studied via the coherency strengthening theory. The findings of this work are categorized as follows:
(1) The $hkl$-specific diffraction elastic constants (DECs) of the $\gamma''$ phase were measured. It is shown the $\gamma''$ phase is stiffer along its $c$-axis than along its $a$-axis.

(2) From the point of view of the stress-lattice strain evolution and diffraction peak width evolution, the plastic deformation first takes place in the grains oriented with $<110>$ parallel to the loading direction as the $\gamma$ matrix and the $\gamma''$ precipitates are jointly deformed, which can be attributed to dislocations shearing precipitates process at the early stage of plastic deformation. At the later stage, the deformation by dislocations bypassing the $\gamma''$ precipitates become predominant. In addition, the occurrence of microtwinning is suggested by the lattice strain deviation in the $<100>$ oriented grains of the compressive-aged sample.

(3) The compressive-aged samples display a larger yield strength compared to the tensile-aged sample. Analysis combining the coherency strengthening model and variant distributions in the two samples reveals that dislocation–precipitate interaction in the compressive-aged sample is more effective compared to that in the tensile-aged sample. The calculated variation in yield strength agrees with the experimental value, indicating the variant-sensitive dislocation–precipitate interaction accounts for the variation in strengthening.

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