Neutron and Synchrotron X-ray Residual Stress Mapping of 7XXX Aluminium Alloy Aerospace Welds

Thesis

How to cite:

For guidance on citations see FAQs.

© 2003 The Author

Version: Version of Record

Copyright and Moral Rights for the articles on this site are retained by the individual authors and/or other copyright owners. For more information on Open Research Online’s data policy on reuse of materials please consult the policies page.

oro.open.ac.uk
NEUTRON AND SYNCHROTRON X-RAY RESIDUAL STRESS MAPPING OF 7XXX ALUMINIUM ALLOY AEROSPACE WELDS

Vadim A. Stelmukh

Doctor of Philosophy

OPEN

2003

Author no. T1497151
Date of submission 8 July 20
Date of award 5 February 2015
Neutron and Synchrotron X-ray Residual Stress Mapping of 7XXX Aluminium Alloy Aerospace Welds

ABSTRACT

It has been long appreciated that residual stresses can exert significant influences on fatigue and fracture behaviour. Diffraction methods offer the engineering community the ability to obtain macro residual stress profiles, which are needed for designing safety critical parts. Because of their penetrability, neutron and synchrotron X-rays can be used to measure the strain tensor in the interior of engineering components. Despite the fact that diffraction techniques are well developed by scientists, it will take some time before engineers can use them as routine tools for obtaining the experimental data which are convertible into stress distributions with anticipated precision.

This work deals with high-strength Al-Zn-Mg-Cu alloys, which are widely used by the aerospace industry and have recently been found to be weldable due to significant progress in the development of new welding processes and automation of existing ones. There are several problems specific to stress measurement in aluminium alloy welds: variation in grain size, preferred orientations and the associated spatial resolution and speed at which the diffraction data can be collected. Based on the findings of preliminary examinations, experimental strategies are proposed to overcome these difficulties for the alloys studied with a view of obtaining usable diffraction data. They include a hybrid neutron/synchrotron diffraction technique designed to optimize the mapping of the full 3D stress tensor for the whole weld cross-section and the use of hard X-rays for effective
triaxial strain measurements in highly textured zones of the welds. These and other procedures developed in the present work are also tested by the author on welds fabricated using the most advanced joining technologies: Metal Inert Gas, Variable Polarity Plasma Arc and Friction Stir welding. The role of “elastic stress-free” reference measurements, as a means of intrinsic separation of macrostrains from microstrains, is outlined. It has been shown that accurate macrostrain determination requires the reference measurements to be made under conditions identical to the bulk measurements.

The longitudinal stress distribution in the Metal Inert Gas weld is found to be characterized by the presence of distinct maxima in the regions of the heat affected zones bordering the double-V shaped fusion zone. The through-thickness asymmetry of the stress field is assumed to be responsible for the residual stress relaxation that occurs on the thinning to size of the weld, unambiguously determined in this thesis. The experimental data obtained for the Friction Stir welds with neutron diffraction shows that the welding speed has a significant influence on the magnitude and spatial distribution of the residual stresses: the specimen made at lower welding speed exhibits lower residual stress, which is accompanied by a lowering of the minimum hardness and a widening of the heat affected zone. The effect of heat inputs associated with the above welding processes on the development of residual stresses, and possible stress reducing measures are discussed in the light of the results obtained.
This thesis is submitted for the degree of Doctor of Philosophy of The Open University. It is an account of the research performed in the Department of Materials Engineering of the Faculty of Technology between December 1999 and May 2003 under supervision of Prof. Lyndon Edwards and Dr. Mike Fitzpatrick. The work reported is original and has been performed without collaboration. None of this work has been submitted for a degree or other qualification at this University or any other institution. Where the work of other authors has been included in the text, this has been acknowledged and its source given in the References at the end of the thesis.

Certain parts of the research conducted for this thesis have been published as the following journal and conference papers:


In addition, the results and findings referred to or detailed in this thesis contributed to the following conference papers:


ACKNOWLEDGEMENTS

It has been a privilege for me to conduct this research in The Department of Materials Engineering, where I met many people inspiring and enabling my work. First and foremost I am indebted to Professor Lyndon Edwards for supervising my work, for his continued encouragement and helping me to make this thesis readable. I would like to thank Javier Santisteban and Michael Fitzpatrick for their technical assistance and helpful discussions. I would also like to thank Peter Ledgard, Tim Gough and Gordon Imlach for their invaluable help in the preparation of the experiments, Stanley Hiller for his genuine interest in this work, moral support and facilitating preliminary examination of my specimens. I am very grateful to Naomi Williams (former manager of the Electron Microscopy suite) for her help in introducing me to the EBSD technique and a number of the stress relieving “racquet-ball” sessions, which I enjoyed so much.

Thanks are due to the authorities and staff of the European Synchrotron Radiation Facility (ESRF, Grenoble), Institute Max Von Laue-Paul Langevin (ILL, Grenoble), Laboratoire Leon Brillouin (LLB, CEA-Saclay) in France and the ISIS facility in the UK for approval and allocation of synchrotron and neutron beam time, and their support and assistance during the experiments, in particular from Francois Fauth and Andrew Fitch of ESRF, Thilo Pirling of ILL, Monica Ceretti of LLB, and Mark Daymond and Jude Dann of ISIS.

I express my gratitude to the Cranfield University Weld Engineering Research Centre and the Edison Welding Institute for providing the welds, to Airbus (UK) and the UK’s Engineering and Physical Sciences Research Council for their support via the grant “Weld processing design & durability of welded aircraft assemblies”, to The Open University for awarding me a grant for this research and to The Institute of Solid State Physics (Russian Academy of Sciences) for providing me with a world-class training, which predetermined the success of the present endeavour.

Finally, I wish to thank my wife, Inna, who has been a continual source of inspiration to me since 1986, when she became the most important part of my hectic life.
# TABLE OF CONTENTS

## INTRODUCTION

1. LITERATURE OVERVIEW

1.1 Origins of macro residual stresses 4
1.2 Residual stresses in welded joints 4
1.3 Welding of high strength aluminium alloys 9
  1.3.1 Metal inert gas welding 9
  1.3.2 Variable Polarity Plasma Arc welding 10
  1.3.3 Friction stir welding 11
1.4 Sin$^2\psi$ method for the elastic stress determination 14
1.5 Bulk measurements and $\sigma_0$-problem 17
1.6 Microstresses and their contribution to residual stress measurements 19
1.7 Measurements of Al-alloy welds 27

2. THE PRIME EXPERIMENTAL METHODS 32

2.1 Neutron diffraction as a strain mapping technique 32
  2.1.1 Instruments at steady-state sources 34
  2.1.2 Time-of-flight instrument 39
2.2 Synchrotron X-ray strain mapping 45
  2.2.1 Advantages and disadvantages of the technique 45
  2.2.2 BM16 (ID31) beamline and instrument configuration 49
3 SPECIMENS AND THEIR PRELIMINARY CHARACTERIZATION

3.1 Welds and preparation of test-pieces

3.2 Reference specimens and their measurement conditions

3.3 Hardness profile measurements

3.4 Optimizing strain mapping by the use of EBSD
   3.4.1 EBSD specimens and experimental details
   3.4.2 Results of the EBSD measurements

3.5 Planning neutron diffraction experiments

4 EXPERIMENTS USING LABORATORY X-RAYS

5 RESULTS

5.1 Studying an effect of mechanical treatment on the residual stress field in the MIG weld

5.2 Influence of the welding speed on residual stresses associated with friction stir welding

5.3 Residual stresses generated in a 12.6mm thick plate after friction stir welding
   5.3.1 Measurements of the “as-received” test-piece on ENGIN at ISIS
   5.3.2 Residual stress mapping in the friction stir weld on G5.2 at LLB

5.4 Full stress tensor determination in a highly textured Al alloy plate (VPPA welded) using synchrotron X-ray diffraction
6 DISCUSSION

6.1 Applicability of the crystallite-group method to residual strain mapping and the erroneousness of stress calculations performed

6.2 Reference measurements as a means of intrinsic separation of macrostrains from microstrains

6.3 The TOF data obtained and their use in the stress calculations

6.4 The use of synchrotron X-rays for assessment of the macrostress condition in the reference specimens and possible corrections of the stress tensor

6.5 The stress distributions obtained and their relevance to the microstructural characteristics and welding parameters

CONCLUSIONS

APPENDIX A

REFERENCES
GLOSSARY OF SYMBOLS AND ABBREVIATIONS

$A_{\text{hkl}}$ Anisotropy factor (dimensionless)

$A_j$ Atomic weight (g/mol)

AS Advancing Side (in FS welds)

$a, a_0, a_{\text{ref}}$ Lattice parameter ($\text{Å}, \text{nm}$)

$a(hkl)$ Lattice parameter derived from lattice spacing $d_{hkl}$ ($\text{Å}, \text{nm}$)

C Diffractometer constant ($\text{m}/\mu\text{s}$)

$C_{ijkl}$ Tensor of elastic stiffness (GPa)

D Depth below specimen's surface (mm)

$D_L$ Long diagonal of the synchrotron X-ray gauge (mm)

$d, d_0, d_{\text{ref}}, d_{hkl}, d_L$ Interplanar lattice spacing ($\text{Å}, \text{nm}$)

E Young's modulus (GPa)

EBSD Electron BackScatter Diffraction

EDM Electro Discharge Machining

$\varepsilon$, $\varepsilon_{ij}$ Strain, Components of strain tensor (dimensionless or microstrain)

F Download force (N)

FCC Face Centered Cubic

FE Finite Element

FS Friction Stir

FZ Fusion Zone

FWHM Full Width at Half Maximum (deg.)

$\varphi$, $\psi$ Azimuth and pole angles of the measurement direction $\overline{m}$ (deg.)

$\varphi_1$, $\phi$, $\varphi_2$ Euler angles (deg.)

GCP Geometrical Centre Position

GTA, GMA Gas Tungsten Arc, Gas Metal Arc

$\gamma$ Distortion (deg.)
h Planck’s constant (J·s)
H Heat input (J/mm^2)
(hkl), {hkl} Miller’s indices for lattice plane, all equivalent lattice planes
[hkl], <hkl> Miller’s indices for lattice direction, all equivalent lattice directions
HAZ Heat Affected Zone
k Neutron attenuation coefficient (cm^{-1})
L Distance of neutron flight (m)
LD Longitudinal Direction (welding direction)
λ Wavelength (Å)
MMA, MIG Manual Metal Arc, Metal Inert Gas
m Neutron mass (kg)
\overline{m} The measurement direction vector within the specimen-system
\textit{m}_j Mass fraction (dimensionless)
MUD Multiples of Uniform Density
μ X-ray absorption coefficient (cm^{-1})
N_A Avogadro’s number (mol^{-1})
n Rotation frequency (s^{-1})
η Coefficient of friction (dimensionless)
ν Poisson’s ratio (dimensionless)
ODF, f(g) Orientation Distribution Function
PAW Plasma Arc Welding
PM Parent Material
PV Pseudo-Voigt
P_{hkl} Multiplicity factor for {hkl} planes (dimensionless)
PSD Position Sensitive Detector
PWHT Post Welding Heat Treatment
\overline{q} Wave vector
Q  Power (W)
R  Radius (mm)
RB, LB  Right Bank, Left Bank (of the detectors on ENGIN)
RD, TD, ND  Rolling, Transverse, Normal Direction
RS  Retreating Side (in FS welds)
ρ  Material's density (g/cm³)
$s_i^m$, $s_2^m$  Macroscopic elastic constants (MPa⁻¹)
$S_{11}$, $S_{12}$, $S_{44}$  Components of compliance tensor in the Voigt's notation (MPa⁻¹)
$\sigma$, $\sigma_{ij}$  Stress, Components of stress tensor (MPa)
$\sigma_1$  Peak broadening in the TOF technique (µs)
$\Sigma_c$, $\Sigma_i$, $\Sigma_a$  Coherent, incoherent, absorption neutron cross-section (cm², barn)
t  Specimen’s thickness (mm)
TOF  Time Of Flight (µs)
TMAZ  Thermo-Mechanically Affected Zone (in FS welds)
θ  Diffraction angle (deg.)
$v_i$, $v_d$  Incident, diffracted beam apertures (mm)
VPPA  Variable Polarity Plasma Arc
V  Welding travel speed (mm/s)
w  Horizontal width of the incident beam (mm)
$XEC$, $s_1(hkl)$, $s_2(hkl)$  X-ray Elastic Constants (MPa⁻¹)
Z  Atomic number
INTRODUCTION

Welding is currently being considered as a cost-effective alternative to mechanical fastening for future aircraft metallic members and components [see e.g. Scott & Williams 1999], e.g. for very large aircraft (VLA) wing construction. Welds are generally more rigid than riveted joints providing a better transfer of load between parts of aircraft structures. However, there are a number of problems associated with the formation of reliable high quality joints of high strength precipitation hardened aluminium alloys used in airframe manufacturing. For example, hot cracking during welding and poor weld microstructures usually result in lower integrity and performance. Despite the highly successful welding process developments that have occurred in recent years [see e.g. Mendez 2000], their application demands radical changes in aircraft design and construction techniques, and could have profound effects on the fatigue durability and damage tolerance of the aircraft. Current designs of civil aircraft reflect the fracture and fatigue performance of mechanically fastened joints. Joints and structure have co-evolved to optimise damage tolerance performance, particularly with respect to issues of fail safety multiple load paths and residual strength. Use of welds will require re-examination of designs, and the way in which cracks grow within the welded fabrication in order to maintain fail safe, crack arrest features. The prediction of fatigue crack location, trajectory and of how cracks will interact with weld microstructures in the heat affected zones and residual stress fields are thus of paramount importance. In order to obtain this knowledge accurate reliable ways must be devised to determine the residual stresses, which can be just as detrimental as applied stresses.

Experiences show that often the residual stresses can only be characterized on the basis of measurements. Diffraction of neutrons and less penetrating X-rays is currently regarded as the most accurate non-destructive and non-contacting technique capable of measuring strain/stress fields deep within engineering components [see e.g. Daymond and Edwards 2000, Withers and Webster 2001]. The crystal lattice acts as an “atomic strain gauge”; so
that a change in interplanar atomic distances can be a direct measure of the elastic component of the strain. Provided the material's elastic constants are known, the elastic strain measured along a number of directions can be used to determine the full stress tensor. In order to design critical parts with sufficient reliability, an engineer is generally interested in macroscopic averages of the stresses (residual stresses of type-I). These are difficult to obtain from diffraction data unless appropriate diffraction conditions and sampling volumes are used and contributions of inter-granular stresses (residual stresses of type-II) in the measured changes in interplanar distances are taken into account [see e.g. Hauk 1986].

The main topic of this thesis is the use of neutron and synchrotron X-ray diffraction for residual strain/stress measurements around joints of 7XXX series aluminium alloys produced by the most advanced welding technologies. These alloys exhibit both precipitation dissolution and growth at various temperatures [see e.g. Nicolas & Deschamps 2002]. The thermal history imposed during welding and post-welding processing usually leads to a variation in the degree of supersaturation of the Al-based matrix with alloying elements (e.g. Zn, Mg, Cu) across the weld, which may result in a correspondent distribution of the so-called \( \delta_0 \), the "stress free" interplanar spacing [see e.g. Krawitz and Winholtz 1994] that is necessary for strain calculations. This distribution is often superimposed with the contribution from the intergranular stresses, which are likely to be present in the rolled plates before welding and those distributions can be also be modified during thermo-mechanical deformation associated with a given welding process and post-welding treatments. The Al alloy plates studied here are characterized by a strong preferred orientation (texture). In general, there are a number of variables that can affect the texture in aluminium alloys. It is well known that the presence of particles, as an example, is an important factor influencing the deformation and re-crystallization texture. Depending on the type of precipitates (shearable, non-shearable) various types of deformation texture were reported, even within the same series of Al-based alloys [see e.g.
Dutkiewicz & Bonarski 1997]. In order to select the most advantageous diffraction peaks and define appropriate conditions for the strain scanning experiments, it is important to characterize the texture and microstructure for different strain measurement positions across the welds. The results of this preliminary assessment facilitated the development of a number of the experimental strategies. Together with the allowances made for the above-mentioned effects these strategies allowed the author to perform fast strain measurements using different diffraction techniques and instruments (each characterized by its advantages and limitations) enabling an accurate determination of the residual stress profile around the candidate welds.
1 LITERATURE OVERVIEW

1.1 Origins of macro residual stresses

Residual stresses/strains are defined as stresses/strains that exist in a body under uniform temperature conditions in the absence of any externally applied load [Macherauch & Kloos 1987]. There are at least four ways in which macro residual stresses can arise in engineering components [Withers & Bhadeshia 2001]: through the interaction between misfitting parts within an assembly, and through the generation of chemical, thermal, and plastically induced misfits between different regions within one part. The chemically generated stresses can develop due to volume changes associated with chemical reactions, precipitation, or phase transformation. Thermally generated residual stresses are often the consequence of non-uniform heating or cooling operations. Coupled with the material constraints in the bulk of a large component this can lead to severe thermal gradients and the development of large internal stresses. Mechanically generated residual stresses are often a result of manufacturing processes that produce non-uniform plastic deformation. They may develop naturally during processing or treatment, or may be introduced deliberately to develop a particular stress profile in a component. In practice, the resultant stress distributions may be caused by combination of the above mechanisms. In any free standing body stress equilibrium must be maintained, which means that the presence of a tensile residual stress in the component will be balanced by a compressive stress elsewhere in the body. An example is the rapid cooling of aluminium alloys from the heat treatment temperature, which leads to surface compressive stresses, balanced by tensile stresses in the bulk of the component. A comprehensive explanation of how the correspondent stress distribution forms is proposed in [Noyan & Cohen 1987].

1.2 Residual stresses in welded joints

It is well known that residual stresses in welds are caused by the hindered contraction of the heated zones [Nitschke-Pagel & Wohlfahrt 2002]. The welding process generates an
inhomogeneous temperature field with hot and cold zones in the material. During cooling the hot zones attempt to shrink proportional to their current temperature and their thermal expansion coefficient, but this shrinkage is hindered by the cooler zones in the joint. This restraint is the reason why tensile residual stresses develop with decreasing temperature. In many engineering alloys, resultant tensile stresses approaching yield values are often observed [see e.g. Nitschke-Pagel & Wohlfahrt 2002, Webster 1993]. What is more, the material’s mechanical properties vary with temperature. Microstructural changes and possible phase transformations occurring during welding in a given region will also alter its yield strength. At some critical temperatures the corresponding values are exceeded creating a plastic deformation non-uniformly distributed across the weld. The latter is regarded as one of the main sources responsible for the generation of complex residual stress distributions in welds and other industrial components [see e.g. Noyan & Cohen 1987].

There have been numerous attempts to predict residual stresses in welds using finite element (FE) models [Cañas et al. 1996, Enzinger & Cerjak 2002, Sjöström 1992, Preston et al. 1999]. Residual stress profiles as shown in Fig. 1.1 are expected for thin plates with single-pass weld, where material does not undergo phase transformations in the solid state and there are no significant temperature gradients in the through-thickness direction. It appears to be in good agreement with experimental results obtained for AlMg4.5Mn Gas Tungsten Arc (GTA) [Nitschke-Pagel & Wohlfahrt 2002] and Al-5083-O Gas Metal Arc (GMA) [Cañas et al. 1996] welds. An excellent qualitative description of what an element of material in the vicinity of a butt weld experiences during the weld thermal cycle is given in [Easterling 1983]. As the temperature increases the initial expansion of the element is restrained by material further away from the heat source, which generates compressive stresses in the element, as shown schematically in Fig. 1.2. The elastic portion of the stress-strain curve is non-linear because of the decrease in elastic modulus. At some critical temperature (point 2), the yield stress of the element is exceeded, and further heating results in a decline in stress as the metal becomes softer. At point 3 the element’s stress is practically zero and considerable plastic strain may occur. At the peak temperature reached
Figure 1.1: Residual stresses associated with fusion welding [Nitschke-Pagel & Wohlfahrt 2002]. LD and TD stand for longitudinal and transverse directions respectively, which are perpendicular to the normal (or the plate’s through-thickness) direction (ND).

in the element the net strain is represented by vector $1 \rightarrow 4$. On cooling, as the temperature of the element decreases, resistance by adjacent, hotter material initially reverses the plastic strain pattern until a temperature is reached at which the element’s flow resistance increases, which start generating tensile stresses in the element (point 5). Further decrease in temperature is then accommodated by the element in the form of elastic tensile stresses until it cools down completely (point 6). Depending on the distance from the heat source, different levels of tensile stress are achieved across the welded joint. The pattern of the longitudinal residual stresses ($\sigma_{\text{LD}}$) in the butt weld thus appears as in Fig. 1.1. The presence of compressive stresses further away from the weld’s centre is predetermined by the balance of forces, $\sigma_{\text{LD}}(y_k, z_k)dA_k$, acting along the longitudinal direction (LD) over the whole TD - ND section, i.e. the following condition $\sum_{k=1}^{N} \sigma_{\text{LD}}(y_k, z_k)dA_k = 0$ holds, where
\( dA_k \) is the TD-ND cross-sectional area of one of the elements and their number \( N \) is big enough.

In more complex situations, the residual stress distribution is difficult to predict unless an accurate model of the phase transformations occurring during welding and their influence on possible hindered volume expansion [Nitschke-Pagel & Wohlfahrt 2002] and local plastic behaviour is developed and incorporated into a FE code used for the calculations. In addition, the quality of such calculations depends strongly on the availability of the temperature and microstructure dependent material properties as well as on the achievable accuracy in defining boundary conditions for the parameters like heat input and heat transfer to the cooling medium.

An example of a calculated stress profile [Lin 2002] that inadequately simulates the experimentally detected distribution [Edwards et al. 2002] is shown in Fig. 1.3. Here the

Figure 1.2: Schematic illustration of the variations in stress-temperature and stress-strain during a weld thermal cycle. Point 6 refers to the final residual stress and strain after the element has cooled to ambient temperature (after [Easterling 1983])

An example of a calculated stress profile [Lin 2002] that inadequately simulates the experimentally detected distribution [Edwards et al. 2002] is shown in Fig. 1.3. Here the
FE modelling is complicated further by the fact that a double-pass welding process in a rather thick plate (12 mm) was under consideration. Until now the most encouraging results obtained for precipitation-hardening material are only for a single pass AA2024 GTA weld [Preston et al. 1999]. Thus, development of models still requires accurate measurements of material properties and residual stresses to validate and refine them.

Figure 1.3: The LD stress distributions for the midline of the cross-section in the middle of a dog-bone specimen cut from a double-pass MIG welded AA2024 plate obtained in synchrotron X-ray experiment [Edwards et al. 2002] and calculated by means of multi-purpose FE code MARC [Lin 2002].
1.3 Welding of high strength aluminium alloys

In the present work residual stresses associated with Metal Inert Gas, Variable Polarity Plasma Arc and Friction Stir welding of high-strength aluminium alloy plates have been studied and therefore it is appropriate to describe the main specifics of the above welding techniques.

1.3.1 Metal inert gas welding

First patented in the USA, GMA welding became popular in the UK from about 1952. Now it is frequently referred to as Metal Inert Gas (MIG) welding. MIG is an attractive alternative to Manual Metal Arc (MMA) welding, offering high deposition rates and high productivity [Easterling 1983]. In the MIG process (see schematic shown in Fig.1.4), the arc is formed between the end of a small diameter wire and the workpiece. The filler wire is continuously fed and serves as a consumable electrode, melting off as a spray of droplets at high welding currents and as discrete drops at lower welding currents. The arc and weld pool are protected from the atmosphere by a gas shield (argon for aluminium alloys). This enables bare wire to be used without a flux coating (required by MMA). However, the absence of flux to 'mop up' surface oxide places greater demand on the welder to ensure that the joint area is cleaned immediately before welding. This is usually done using a wire brush, for relatively clean aluminium alloy plates, or even a hand grinder. It is worth noting here that this treatment generates a surface stress state that will conceal the level of residual stresses generated by the welding if laboratory X-rays are used for the stress measurements.

The current applications of MIG in automatic welding benefit from its low cost, while the high reliability of MIG welds in manned airplanes is yet to be achieved [Mendez 2000].
1.3.2 Variable Polarity Plasma Arc welding

Variable Polarity Plasma Arc (VPPA) welding is one of the latest variations of Plasma Arc Welding (PAW), which uses a constricted arc between a nonconsumable electrode and the weld pool. This constricted arc is called plasma, which is the fourth state of matter. In PAW, the current is transferred from a tungsten electrode inside the welding torch though the orifice to the workpiece and back to the power supply (see Fig. 1.5). The tungsten electrode is connected for direct-current as the negative electrode. The heat intensity of the plasma is high enough to operate in a keyhole mode, when the plasma jet penetrates through the workpiece and forms a hole known as a keyhole. Surface tension forces the molten parent metal to flow around the keyhole to form the weld obviating the necessity for using any filler metal. Thus, the keyhole method provides for full-penetration single-pass autogenous welding.

The VPPA process is being developed by the aerospace industry for welding thicker sections of aluminium alloys [Cary 1998]. It employs a variable current waveform, where the positive part of the cycle (when the tungsten electrode is positive) provides a cathodic cleaning of the aluminium workpiece, while the negative portion (when the tungsten electrode is negative) provides the desired penetration and molten metal flow. With a special power source, which actually consists of two power sources connected together by
an electronic switch, the negative and positive half-cycles can each be varied with respect to current and time. The VPPA waveform is coupled to the plasma torch so that the keyhole method is possible. The concentrated heat of VPPA causes significantly less angular distortions than other fusion processes [Mendez 2000].

![Diagram of an electronic switch and VPPA waveform coupling to the plasma torch](image)

**Figure 1.5**: Transferred arc mode (a) and process diagram (b) for PAW using the keyhole method (after [Cary 1998]).

### 1.3.3 Friction stir welding

Invented at The Welding Institute (Cambridge, United Kingdom) [Thomas 1991], Friction Stir (FS) welding is believed to be a viable technique for joining aluminium alloys that are difficult to fusion weld [Thomas & Nicholas 1992, Mahoney *et al.* 1998, Williams 2001]. A schematic illustration of the weld process is shown in Fig. 1.6 [Mahoney *et al.* 1998].

To FS weld a butt joint, a specially designed cylindrical tool is rotated and plunged into the joint line (Fig. 1.6b). The tool has a small diameter entry probe with a concentric larger diameter shoulder (Fig. 1.6a). When the rotating entry probe makes contact with its surface, the heat created softens a column of metal around the probe as well as a small region of material underneath the probe (Fig. 1.6c). As the probe penetrates beneath the surface, part of this metal column is extruded above the surface. The depth of penetration is restricted by the tool shoulder and length of the entry probe. When the shoulder contacts the metal surface (Fig. 1.6d), its rotation creates additional frictional heat and plasticizes a
cylindrical metal column around the inserted probe. During welding, the metals to be joined and the tool are moved relative to each other such that the tool tracks the weld interface (Fig. 1.6e). The rotating tool provides a continual hot working action, plasticizing metal within a narrow zone while transporting metal from the leading edge of the tool to its trailing edge. FS welding has been found to be a solid-state joining process [Murr et al. 1998] with the weld completed without creation of liquid metal. A moving column of stirred hot metal consumes the weld interface, disrupting and dispersing aluminium surface oxides. The weld cools, not solidifies, as the tool passes, forming a defect-free weld. The FS welding process is asymmetric with different deformation conditions occurring on the “advancing” side (AS) and “retreating” side (RS) of the joint. The welds studied in this work were prepared using the tool rotated with left-handed motion that uniquely determines the position of the above sides, as shown in Fig. 1.6e.

Figure 1.6: Macrograph of the welding tool inside the joint (a) and schematic illustration (after [Mahoney et al. 1998]) of the friction-stir-weld process (b-e). RS and AS indicate the “retreating” and “advancing” side, respectively.
Although it is generally agreed that the FS welding produces small residual stresses in high-strength aluminium alloys when a typical welding speed of 100 mm/min is used [e.g. Jata et al. 2000], they have to be measured as research and development are still being undertaken to produce good quality and fatigue resistant joints at higher speeds. For example, it has been shown [Bussu & Irving 2003] that crack growth behaviour in FS welded 2024-T351 plates is dominated by the residual stress, whereas microstructure and hardness changes have a minor influence.
1.4 Sin²ψ method for the elastic stress determination

Measurement of the interplanar lattice spacing, \(d\), or lattice parameter, \(a\), in various specimen orientations is the basis of residual or applied stress determination by diffraction methods. If the stress-free spacing, \(d_0\), is known, precise measurement yields the strain
\[
\varepsilon = \frac{(d - d_0)}{d_0}
\]
for a given direction. It is possible to establish the strain state from any six measurements of independent strain [Allen et al. 1985, Winholtz & Cohen 1998]. In practice, it is highly recommended to make more measurements and extract the strain tensor, \(\varepsilon_{ij}\), from the data by a least-squares fit [Lorentzen & Leffers 1992]. The complete three-dimensional stress state of the irradiated volume is represented by the stress tensor, \(\sigma_{ij}\), which can be obtained by conversion from the strain tensor, provided the elastic stiffnesses for the material are known. Both strain and stress states are described by symmetrical tensors of 2nd rank:

\[
\varepsilon_{ij} = \begin{pmatrix}
\varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\
\varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\
\varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33}
\end{pmatrix}
\quad \sigma_{ij} = \begin{pmatrix}
\sigma_{11} & \sigma_{12} & \sigma_{13} \\
\sigma_{21} & \sigma_{22} & \sigma_{23} \\
\sigma_{31} & \sigma_{32} & \sigma_{33}
\end{pmatrix}
\]

(1.1)

where only 6 of the 9 components are independent because of the symmetry (\(\varepsilon_{ij} = \varepsilon_{ji}\) and \(\sigma_{ij} = \sigma_{ji}\)). For each tensor, there is a system of co-ordinates in which off-diagonal elements vanish. In this case the diagonal elements are called principal stresses. Thus, each stress state can be characterized by 3 principal stresses and the directions of the principal stresses with respect to a reference system.

The elastic stiffnesses form the 4th rank tensor, \(C_{ijkl}\). The use of the Voigt notation, \(C_{ijkl} = C_{mn}\), allows its representation to become handleable. The number of independent components reduces with increasing symmetry of the crystal lattice [Nye 1985]. For the cubic system, commonly occurring in many metals, \(C_{mn}\) shows the following arrangement with only three independent components:
The above representation is also applicable to the elastic compliances $S_{ijkl}$, which is the inverse tensor of the stiffness tensor, i.e. $S = C^{-1}$. For a given $[hkl]$ direction, the Young’s modulus, $E_{hkl}$, can be defined by considering a uniaxial stress state of a single crystal as follows [Allen et al. 1985]:

$$\frac{1}{E_{hkl}^l} = \frac{\varepsilon_{hkl}^{ll}}{\sigma^l} = S_{11} - 2\times \left( S_{11} - S_{12} - \frac{S_{44}}{2} \right) \times A_{hkl},$$

(1.3)

where $\varepsilon_{hkl}^{ll}$ is the strain in the direction of the applied load, $\sigma^l$, and $A_{hkl}$ is the anisotropy factor, which for cubic systems has the following form:

$$A_{hkl} = \frac{h^2k^2 + k^2l^2 + h^2l^2}{(h^2 + k^2 + l^2)^2}$$

(1.4)

In practice of the stress measurement, the strain, $\varepsilon_{\psi\psi}$, is measured in a direction $\overline{m}$ that is in the specimen co-ordinate system is usually determined by the angles $\phi$ and $\psi$ (see Fig.1.7). By applying Hooke’s law for elastically isotropic material ($S_{11} - S_{12} - S_{44}/2 = 0$) the following relation between $\varepsilon_{\psi\psi}$ and $\sigma_{\psi}$ can be obtained [Hauk 1997]:

$$\varepsilon_{\psi\psi} = 1/2s_2^m \left[ \sigma_{11} \cos^2 \varphi \sin^2 \psi + \sigma_{22} \sin^2 \varphi \sin^2 \psi + \sigma_{33} \cos^2 \psi \right]$$

$$+ 1/2s_2^m \left[ \sigma_{12} \sin 2\varphi \sin^2 \psi + \sigma_{13} \cos \varphi \sin 2\psi + \sigma_{23} \sin \varphi \sin 2\psi \right]$$

$$+ s_1^m \left[ \sigma_{11} + \sigma_{22} + \sigma_{33} \right]$$

(1.5)

where $s_2^m$ and $s_1^m$ are the macroscopic elastic constants, which can be expressed via Young’s modulus, $E$, and Poisson’s ratio, $\nu$:

$$1/2s_2^m = \frac{1+\nu}{E} \quad s_1^m = \frac{-\nu}{E}$$

(1.6)

For texture free material, only a small portion of all crystallites within the irradiated volume contributes into the registered intensity of the diffraction peak and their number
depends on the multiplicity factor, \(P_{\text{hkl}}\), for the lattice planes, \{hkl\}, used. As the elastic behaviour of this collective may differ from the macroscopic behaviour due to the elastic anisotropy of the crystals, the constants \(s_2^m\) and \(s_4^m\) in Equ.1.5 must be replaced by the \{hkl\} dependent constants \(s_2(hkl)\) and \(s_4(hkl)\). These constants are either calculated using different models describing the interaction between grains in a polycrystalline aggregate [Hauk 1997] or determined from lattice strains observed experimentally for a given material subjected to external load.

Figure 1.7: Definition of the measurement direction by angles \(\varphi\) and \(\psi\).

In some cases (e.g. when using laboratory X-rays) a bi-axial stress state can be assumed \((\sigma_{33}=0)\) due to low penetration of the incident beam. In addition, strain measurements can be done on a certain azimuth. These conditions simplify the Equ.1.5 and for \(\varphi=0\) one can obtain the following formula:

\[
\varepsilon_{0,\psi} = s_1(hkl)[\sigma_{11} + \sigma_{22}] + 1/2 s_2(hkl)[\sigma_{11}\sin^2 \psi + \sigma_{13}\sin 2\psi]
\]

(1.7)

If shear components are negligibly small \((\sigma_{13} \rightarrow 0)\), dependence of strain (or d-spacing) must be linear. Thus, the principal stress can be found directly using a slope of the d-vs-\(\sin^2 \psi\) dependence:

\[
\sigma_{11} = \frac{1}{1/2s_2(hkl) \frac{\partial d_{\psi,\psi}}{\partial \sin^2 \psi}}
\]

(1.8)
In this case an approximate value of stress-free spacing, $d_{0}^{\text{approx}}$, is sufficient and this is the main advantage of the X-ray diffraction technique for residual stress measurement. In fact, only relative positions of measured peaks are needed to obtain $\frac{\partial d_{\phi,\psi}}{\partial \sin^{-1} \psi}$ by a linear fit and for a value of stress to be calculated.

1.5 Bulk measurements and the $d_0$-problem

Using a similar approach and replacing conventional X-rays by neutrons or synchrotron X-rays of high energies, it is possible to obtain information on strains/stresses from within the bulk of materials. The regression analysis $d$-vs-$\sin^{2} \psi$ is usually replaced by three strain measurements at $(\phi=0, \psi=0)$, $(\phi=0, \psi=90)$ and $(\phi=90, \psi=90)$ under an assumption that these directions are the principal strain/stress directions. This can often be inferred from the nature of the process responsible for the generation of the residual stresses and the sample geometry. Moreover, even if incorrect principal stress axes directions were chosen, the stress components along these axes are still correct [Winholtz & Krawitz 1997], as the shear stress components make no contribution to the measured strain, $\varepsilon_{\phi,\psi}$, along the specimen coordinate axes (see Equ. 1.5).

It should be noted, however, that the measurement of triaxial stresses requires an accurate stress-free lattice spacing as in the interior of a bulk solid there is no guaranteed condition like $\sigma_{33}=0$. One of the possible solutions to the $d_0$-problem would be annealing of a reference sample to make it essentially stress-free. However, the stress-free reference value can vary with position in the specimen because of inhomogeneous response to thermo-mechanical treatment within a specimen and additional annealing may alter the original $d_0$-variation. Precipitation hardened aluminium alloys exhibit both precipitation dissolution and growth at various temperatures achieved during welding (e.g. [Nicolas & Deschamps 2002]). Therefore, an Al-based solid solution will contain a different amount of alloying elements at each measurement place across the weld that may lead to
significant spatial variation in $d_0$ [Albertini et al. 1997]. If not taken into account, this variation can introduce errors to the elastic strain measurements. For example, a 0.5% increase in copper content in unstressed Al-Cu alloys may cause the measured strain to be underestimated by 600 $\mu$strain, which is far beyond the tolerance limit [Preismeyer 1992, Preismeyer et al. 1994]. In this situation measurements of the “stress-free” $d$-spacings have to be performed on small cubes covering the whole weld’s section. They can be removed either from the specimen under investigation or from a companion specimen. If $d_0$ cubes are fully bathed in a gauge volume, defined by the cross section of the incoming and outgoing beams, macro residual stresses, which may still be present in the pieces, are averaged to zero [Krawitz & Winholtz 1994]. However, microstresses will still remain [Noyan 1987]. For example, the sensitivity of reference peak angles to orientation in the parent material 5083 aluminium alloy found in [Smith et al. 1988] is likely to be caused by presence of microstresses in a nominally “stress free” block (also see the next Section). It is evident that such an effect needs to be accounted for in residual stress calculations. If filings are used instead of cubes in order to average microstresses to zero and one wishes to avoid annealing them for the reason mentioned above, their state will not be stress-free, but a hydrostatic pressure. This can be exemplified by the results obtained in [Hauk et al. 1990]. In this case the values obtained can be corrected by imposing force and moment equilibrium. Nevertheless, it is recommended in a recent draft standard for residual stress measurement using neutron diffraction [VAMAS TWA 20 Draft Standard 2001] that experimental methods are used where possible, and that equilibrium is employed mainly as a check for consistency. When residual stresses are derived assuming the bi-axial stress condition for a thin plate specimen [Albertini et al. 1997, Oosterkamp et al. 2000, Wang X.-L. et al. 2000], the redundant information coming from the through-thickness measurement allows one to obtain $d_0$ directly using the following relation [Albertini et al. 1997]:

\[\text{Equation} \]

18
where \( d_{LD} \), \( d_{TD} \), and \( d_{ND} \) are the lattice spacings obtained with the same (hkl) peak for each orthogonal measurement direction (see Fig. 1.3). However, this method will not deliver a correct \( d_0 \) if the bulk measurements are affected by the plastic deformation that usually occurs during welding and may also be present in rolled plates [e.g. Prime & Hill 2002] before welding. One of the most practical solutions to the \( d_0 \)-problem has been used in this work, as described and discussed in Sections 3.2, 5.1 and 6.2 [see also Stelmukh et al. 2002]. Another one is based on the approach described in [Barret & Massalski 1980] and explores the pulsed neutron transmission diffraction technique to measure a thin weld’s cross-section subjected to external uniaxial load [Steuwer et al. 2002]. Unfortunately, the latter technique is not suitable for highly textured materials and aluminium alloys, which are poor neutron attenuators. Based on the above description, one may notice that the information on the "stress-free" d-spacing (often called \( d_0 \)) available in the open literature is somewhat ambiguous, as no clear definition for this term has been given so far. In Section 5.1, the reference d-spacing, \( d_{ref} \), or lattice parameter, \( a_{ref} \) will be introduced (also explained in detail in Section 6.2) to distinguish it from \( d_0 \) (or \( a_0 \)). The exact meaning of the latter may, however, be different from that assumed by other authors.

1.6 Microstresses and their contribution to residual stress measurements

Although the primary aim of the present work was to map macrostresses in Al-alloy welds, it is necessary to give a definition of the microstresses already mentioned above and overview their effect on the residual stress measurement. In a polycrystalline material, stresses may strongly fluctuate from grain to grain or even within a single grain. Usually, engineers are only interested in averages over macroscopic distances, i.e. macrostresses. However, diffraction methods never give macrostresses directly. In general, a
superposition of the following stress components of a phase $\alpha$ will determine diffraction peak shifts [Hauk & Nikolin 1988]:

$$\sigma^\alpha = \sigma^L + \sigma^I + \left(\sigma^{II}\right)^\alpha + \left(\sigma^{III}\right)^\alpha$$ (1.10)

where $\sigma^L$ is the applied stress, if a sample is subjected to external loading. The other three components represent three classes into which residual stresses (RS) can be grouped, according to the length scale over which the RS are nearly homogeneous [Macherauch & Kloos 1987]:

- **Type I RS**, $\sigma^I$, is the volume average of the position-dependent stresses $\sigma(x,y,z)$ over all crystallites and phases within the considered volume that is large enough to represent the macroscopic material. This stress is equilibrated within the whole body.

- **Type II RS**, $\sigma^{II}$, are nearly homogeneous across microscopic areas, within one grain or several parts of a grain of a material and defined as the mean deviation from the macrostress level. These stresses may also contribute to the peak broadening.

- **Type III RS**, $\sigma^{III}$, are inhomogeneous across submicroscopic areas of a material, several atomic distances within a grain. The respective average of $\sigma^{III}$ is equal to zero and this stress is virtually impossible to measure by the diffraction techniques except for very coarse-grained materials [Reimers 1992]. Hence, the presence of $\sigma^{III}$ is not much a problem for the evaluation of strain data, as it does not contribute to the peak shifts. Its contribution in the peak broadening can be used to monitor changes in the density and the arrangements of dislocations after plastic deformation, heat treatment or recrystallization. However, there is no explicit relationship between peak broadening and $\sigma^{III}$, as stress and $d_0$ gradients in the gauge volume, grain interaction stresses and small grain size also contribute to peak broadening.
Angle brackets on the microstresses in the above equation indicate that they are averages over those grain groups within the diffracting volume, which contribute to the intensity of the peaks used for the stress measurements. The use of the brackets to denote average macrostress will depend on real macrostress gradients in a component and the size of gauge volume over which strains are sampled.

Microstresses emerge from incompatibilities on a microscopic scale [Pintschovius 1992]. They occur if the elastic properties of the material vary from grain to grain. When external loading is applied to a polycrystalline aggregate consisting of hard and soft particles, the hard particles are loaded more than the soft ones. The most obvious reason for a variation in elastic response is the presence of different phases. Whilst macrostresses balance over different macroscopic regions of a body, the microstresses must balance locally [Noyan 1983]. For a two-phase material, microstresses must be of opposite sign and obey the following equation:

\[
\left\langle \sigma^\alpha \right\rangle^\alpha (1 - f) + \left\langle \sigma^\beta \right\rangle^\beta f = 0
\]

(1.11)

where \( f \) is the volume fraction of phase \( \beta \).

However, even within one phase, elastic anisotropy can lead to considerable microstresses. Although elastic anisotropy is relatively small for aluminium (~10%), it is significantly stronger for the austenite in steels, where the ratio of Young's modulus for the hardest \(<111>\) and softest \(<100>\) direction reaches a factor of 1.6, as can be seen from the \(E_{hk}/E_{100}\) dependences in Fig. 1.8. These were produced using X-ray Elastic Constants (XEC) calculated according to the model of Eshelby-Kröner [e.g. Hauk 1997].

After strong plastic flow, the variation of the elastic properties from grain to grain ceases to be the main cause for microstresses. Instead, microstresses develop on the scale of the grain size because distinct crystallographic orientations of grains deform plastically by different amounts since the angles which the fundamental slip directions make with the applied stress axis are different [Noyan & Cohen 1987]. For this reason, the face-centered
cubic (FCC) materials, which slip along <110> directions in {111} planes, are plastically anisotropic.

Figure 1.8: Anisotropy of Young's moduli for aluminium and austenite, both with face centred cubic structure. This is derived from the XEC $s_1$ and $1/2s_2$ given in [Hauk 1997]. Open circles are plotted for the (hkl) peaks used in this work for the bulk measurements.

In this work we deal with 7XXX series (Al-Zn-Mg-Cu) aluminium alloys, which, to a first approximation, can be considered as nearly single-phase materials. In fact, the volume fraction of minority phase(s) may reach 5% [e.g. Bigot et al. 1997]. Thus, intuitively, macrostress can be given as:

$$\sigma_{\text{macro}} = (\sigma^a - \sigma_{\text{int}}) \cdot (1-f) + \sigma^b \cdot f$$  \hspace{1cm} (1.12)

where $\sigma_{\text{int}}$ stands for a microstress comprising the grain interaction and intergranular stresses in Al-based matrix. This is schematically illustrated in Fig.1.9.

Unfortunately, it is virtually impossible to investigate minority phases separately if the abundance of a phase is less than 10%. Moreover, the average diameter of particles in high strength aluminium alloys does not exceed 10 nm, even after overageing [Nicolas & Deschamps 2002], which usually causes a significant broadening of the corresponding
diffraction peaks [see e.g. Klug and Alexander 1974]. However, plastic anisotropy of an aluminium-based matrix is of higher importance, particularly in the materials with a strong texture [Pintschovius et al. 1987]. The experimental results obtained in [Pang et al. 1998] indicate that intergranular stresses can be significant and can add a large uncertainty in the macroscopic residual stress measurements. In order to detect or even to characterize the grain interaction stresses, it is necessary to make measurements using various (hkl) reflections. The texture-weighted average of the residual stresses can be a reliable measure of the macroscopic stresses as intergranular stresses integrated over all (hkl) must average to zero. Alternatively, (hkl) peaks with high multiplicity can be used, as these peaks sample grains with very different orientations and hence are relatively little influenced by intergranular stresses. Many examples of the relevant investigations are given in [Hauk 1997].

Figure 1.9: Different components of stress. Curly lines represent $\sigma^{III}$, while $\sigma^{e}_{\text{macro}}$ is macrostress corrected for the effect of elastic anisotropy, which is rather small for aluminium.
A rate-independent self-consistent model [Hutchinson 1970] was used in [Clausen et al. 1998] to calculate the intergranular stresses developed during tensile deformation of initially texture-free FCC polycrystals (aluminium, copper, austenitic steel). For most crystallographic planes the lattice strain/applied stress relation deviated very significantly from linearity. For the elastically anisotropic materials (copper and steel) the \{311\} planes came close to linearity, i.e. the (311) reflection was most representative of the overall stress in the directions considered. For aluminium the \{311\}, \{331\} and \{531\} planes were about equally close to linearity. Recently, the calculations for austenitic steel have been repeated [Clausen & Leffers 2003] for plain-strain deformation in order to check the generality of the conclusion on the proper selection of reflection drawn in [Clausen et al. 1998]. For lattice strains in the three main directions (311) has again been found to be the best reflection. However, complete lattice strain pole figures have shown that the (111) and (422) reflections are the best overall choices. The authors also conclude that the \{111\} planes are by far the best choice, if the stress condition (biaxial or uniaxial) is unknown (see also Table 1.1 summarising the results of the work).

<table>
<thead>
<tr>
<th>(hkl)</th>
<th>Plane strain</th>
<th>Tension</th>
<th>(hkl)</th>
<th>Plane strain</th>
<th>Tension</th>
</tr>
</thead>
<tbody>
<tr>
<td>(111)</td>
<td>103</td>
<td>55</td>
<td>(331)</td>
<td>155</td>
<td>178</td>
</tr>
<tr>
<td>(200)</td>
<td>511</td>
<td>397</td>
<td>(420)</td>
<td>176</td>
<td>172</td>
</tr>
<tr>
<td>(220)</td>
<td>258</td>
<td>197</td>
<td>(422)</td>
<td>81</td>
<td>90</td>
</tr>
<tr>
<td>(311)</td>
<td>161</td>
<td>112</td>
<td>(531)</td>
<td>150</td>
<td>182</td>
</tr>
</tbody>
</table>

Table 1.1: Largest deviation from zero lattice strain (in units of microstrain) after 2% plastic strain and unloading of austenitic steel for the eight reflections considered for plane strain and tension. Calculations were made for a “specimen” consisting of 10,000 grains of initially random orientations [Leffers 2003].

There are several ways to estimate the influence of microstresses on the observed peak shifts [Pintschovius 1992]. After converting strains into stresses one should check the
balance of forces. Since residual stresses are self-equilibrating stresses, the resultant force and moment produced by them must be zero. If the balance of forces is fulfilled within experimental error, the calculated residual stresses are likely to be macrostresses. However, this requires the measurement of complete strain distributions across the whole specimen that might be impossible when dealing with large samples or real industrial components. The linearity of the \( d_{hkl} \)-vs-\( \sin^2 \psi \) distributions can also be checked. Intergranular stresses often give rise to non-linearities, in particular for the \( (hkl) \) peaks with low multiplicity [Hauk & Nikolin 1988]. However, moderate non-linearities are also expected in materials with \( \sigma^1 \) only because of elastic anisotropy [Döller & Hauk 1978]. For cubic systems the use of the \( (h00) \) and \( (hhh) \) diffraction peaks should result in linear \( d\)-vs-\( \sin^2 \psi \) distributions even in the case of elastic anisotropy if microstresses are negligibly small [e.g. Van Acker et al. 1994]. With the time-of-flight (TOF) neutron diffraction technique, described in Section 2.1.2, the lattice parameter, \( a(hkl) = d_{hkl} \sqrt{h^2+k^2+l^2} \), calculated for different \( (hkl) \) peaks can be plotted as a function of the stiffness anisotropy [Bourke et al. 1992]. Linear dependences, like those presented in Fig.1.10, may suggest the absence of microstresses at the measurement location. Unfortunately, both methods may be difficult to use when dealing with highly textured materials for the following reasons. It is obvious that \( (h00) \) or \( (hhh) \) peaks of acceptable intensity will not be possible to register at more than a few \( \psi \) orientations due to small multiplicity factors for the relevant planes. The TOF measurements might be impractical to perform as well, as they are time consuming when trying to obtain statistically significant positions of the \( (hkl) \) peaks corresponding to small \( d \)-spacings, due to the form of the Lorentz factor for TOF data [Buras & Gerward 1975].

It is well known that stacking faults are another source of the peak shifts [Warren 1990]. Commercial Al-based alloys are likely to be characterized by a large stacking fault energy because of the relatively small content of alloying elements [Schulthess et al. 1998], and hence there will be no defects of that kind.
Figure 1.10: Lattice parameters calculated from the positions of the strongest (hkl) peaks obtained for stainless steel tube. Experimental values represented by stars are found by fitting the whole TOF spectrum and plotted against the anisotropy factor averaged over all <hkl> directions [Allen et al. 1985]. The dependences are close to linear, indicating small, if any, microstresses at the measurement locations. Other experimental details can be found in [Edwards et al. 2001].

To conclude this part it may be said that allowances for the presence of different stresses in engineering materials are difficult to make when trying to identify exclusively macro residual stresses.
1.7 Measurements of Al-alloy welds

The number of publications with experimental results on residual stresses in Al-alloy welds is very limited as all the measurements required for obtaining the full strain tensor are generally quite time consuming. With neutrons, for example, these measurements in an Al-based material may typically take an hour or so per point. Consequently, area mapping could be prohibitively costly in its use of sources unless large sampling volumes are used [Webster 1993, Smith et al. 1988, Smith & Webster 1997, Owen et al. 2003], some assumptions are made regarding the stress condition of a test-piece [Wang X.-L. et al. 2000, Albertini et al. 1997, Oosterkamp et al. 2000, Webster et al. 2001] or data are being “under-collected” [Webster 1993].

Only transverse stresses are reported for a 200x12x44 mm³ section of a multi-pass double-V MIG weld [Webster 1993] that was prepared by transverse cutting a 44 mm thick AA5083 plate. High residual stresses approaching yield values were detected (see Fig. 1.11). The near surface results, just below the surface at a depth of 2 mm in the TD-ND measurement area, exhibit a relatively low but erratic pattern within the fusion zone. The residual stresses rise smoothly through the edges of the weld to reach maximum tensile values at positions about 5 mm on both sides of the weld centreline before decreasing at greater distances. This distribution is in marked contrast to that obtained for the midline of the measurement area, where the residual stresses are compressive, with the maximum at the centre of the weld. This stress distribution is believed to be expected from force balance considerations. The sectioning would have substantially relaxed the longitudinal stress [Webster 1993] that was confirmed in earlier work [Smith et al.1988]. It is worth noting, however, that [Webster 1993] gives no information on how the strain data obtained using both (311) and (222) Bragg reflections were combined to derive the residual stresses.

A neutron diffraction study of residual stresses in 6061-T6 aluminium FS welds manufactured using different welding speeds is reported in [Wang X.-L. et al. 2000]. The longitudinal stress in 6mm thick 200mm square coupons was found to exhibit a double-
peak profile across the weld centre-line, with tensile maxima located in the middle of the heat-affected zone (see Fig. 1.12). The weld made at low welding speed exhibits lower residual stress. The maximum tensile values amount to 53 % and 73 % of the yield strength of the parent plate, which is 276 MPa at room temperature. For both coupons, the stress data obtained did not reveal either evidence of through-thickness dependence or apparent asymmetry with respect to the weld centre line. For both specimens, the transverse stress was small at all locations. It should be noted, however, that the residual stresses were derived assuming the bi-axial stress condition ($\sigma_{ND}=0$).

Figure 1.11: Transverse residual stresses across the double-V MIG weld derived from strain scans at depths of $x=2$ and 22 mm (which are the distances from the weld's surface along ND). $y$-coordinate is coincident with the strain measurement direction (i.e. TD). The numbered bands correspond to the positions of individual weld beads [Webster 1993].
Figure 1.12: Longitudinal residual stress in AA6061-T6 friction stir welds for specimens prepared with a welding speed of 279 mm/min (a) and 787 mm/min (b). The corresponding hardness data are also plotted [Wang X.-L. 2000].

The synchrotron strain scanning technique was used to map the longitudinal stress in a 6 mm thick AA7108 plate FS welded in T79 condition [Oosterkamp et al. 2000]. A rather high welding speed, 600 mm/min, was used to manufacture this weld. In spite of a strong texture usually observed in 7XXX series alloys (see e.g. [Dutkiewicz & Bonarski 1997]), all directions were measured with the (311) reflection. The highest tensile stresses were observed in the stirred zone, which is commonly referred to as the nugget (see also section 3.4.2). On the advancing side, tension showed a local minimum near the top surface with the maximum close to the mid-depth. On the retreating side, a high tensile region extends down from the top surface, also referred in the present work as the “shoulder side” surface. Near the root, the pattern is roughly symmetrical, whereas at the topside, the pattern is
more asymmetric with the highest tensile regions being associated with the retreating side. The transverse stress field is found to be much weaker than the longitudinal field, however exhibiting through-thickness variation and advancing to retreating side gradients. It is worth noting here that the ND measurements were not made through the plate, as follows from [Webster et al. 2001] published subsequently, and the average peak position for the reference specimen was used for calculation of the residual strains. There is a principal difference between these results and the work referred to previously [Wang X.-L. et al. 2000]: the presence of a distinctive stress maxima within the nugget area (see Fig. 1.13), which may not have appeared had allowances been made for variation in the stress-free lattice spacing across the weld (see e.g. Fig. 5.24). The other obvious explanation to the apparent discrepancies could be the fact that different materials were under consideration, 7XXX [Webster et al. 2001] and 6XXX [Wang X.-L. et al. 2000] alloys. It should be noted, however, that in [Webster 2001] variation in the "stress-free" d-spacing was assessed from measurements on a similar plate, which was not welded. In both investigations, the in-plane stress condition was assumed rather than measured. It seems that this assumption may not be valid for one of the welds studied.

![Graph showing longitudinal mid-plane strains in AA7108-T79 friction stir weld](image)

Figure 1.13: Longitudinal mid-plane strains in AA7108-T79 friction stir weld [Webster 2001].
A combination of diffraction methods was used to provide non-destructive information about the residual stress field in TIG-welded 2024 Al alloy [Owen et al. 2003]. The most interesting result of this work is that the magnitudes of the tensile longitudinal stress decrease along the plate (see Fig. 1.14) due to progressive heating up of the plate ahead of the arc during welding. It is quite clear that this effect should be taken into account when selecting the section that can be used for the reference specimen preparation. That is the reason that stress free parameters were measured on matchsticks cut from another weld prepared under nominally identical conditions. The longest dimension of the matchsticks was in the LD. It should be noted, however, that steady-state conditions were not achieved in this particular process presumably because the welded plates were rather short, 160 mm.

![Graph showing stress variation](image)

**Figure 1.14:** The variation in longitudinal and transverse stresses with distance from the weld centre line, for transverse line scans at 50 (solid) and 110 mm (open symbols) from the beginning of the plate in the weld direction [Owen et al. 2003].
2 THE PRIME EXPERIMENTAL METHODS

The measurement techniques used in this work are founded upon the diffraction of radiation by a crystalline lattice. X-rays is electromagnetic radiation which is diffracted by elastic scattering of the incident waves by the shell electrons of the atoms. Particle beams, like electrons and neutrons, can also be considered as waves of radiation, with their wavelength given by the de Broglie relation:

\[ \lambda = \frac{h}{\sqrt{2mE_{\text{kin}}}} \]  \hspace{1cm} (2.1)

where \( \lambda \) is wavelength, \( h \) is Planck’s constant and \( m, E_{\text{kin}} \) are mass and kinetic energy of the particles respectively. When X-ray, electron or neutron radiation interacts with crystalline material specific diffraction (hkl) peaks can be observed provided that its wavelength is of the same order of magnitude or smaller than the distances between atomic planes \( d_{\text{hkl}} \). W. H. Bragg [1913] and his son Sir W. L. Bragg showed that the angle \( \theta \) at which diffraction occurs depend on both the wavelength \( \lambda \) and interplanar distances \( d_{\text{hkl}} \).

The Bragg's law can be written as:

\[ 2d_{\text{hkl}}\sin \theta = \lambda. \]  \hspace{1cm} (2.2)

This Chapter addresses the use of neutron and synchrotron X-ray diffraction for strain mapping and describes some particulars of the instrumentation and the methodology deployed to accomplish the main experimental program of this thesis.

2.1 Neutron diffraction as a strain mapping technique

The main reason to use neutrons is their penetration depth. For many common engineering alloys, neutrons penetrate about 3 orders of magnitude more than laboratory X-rays of comparable wavelength [Hutchings & Windsor 1987]. The neutron attenuation coefficient, \( k \), for an alloy is given by [Hutchings & Windsor 1987]:

\[ k = \rho N_A \sum_j \frac{m_j \Sigma_j}{A_j} \]  \hspace{1cm} (2.3)
where $\rho$ is the alloy’s density, $N_A$ is Avogadro’s number, $A_j$ and $m_j$ are respectively the atomic weight and mass fraction of the element $j$, and, finally, $\Sigma_j$ is the total neutron cross-section for each element, comprised by its coherent ($\Sigma_c$), incoherent ($\Sigma_i$) and absorption ($\Sigma_a$) cross-sections. As an example, the data used to calculate the 50% transmission thickness for one of the Al-based alloys studied in this work are summarized in Table 2.1.

X-ray absorption data are also given for comparison.

### Table 2.1

<table>
<thead>
<tr>
<th>Element</th>
<th>Density (g/cm³)</th>
<th>Atomic Weight (g/mol)</th>
<th>$m_j$ (cm³/g)</th>
<th>$\rho/n$ (1/cm)</th>
<th>$H_j$ (1/cm)</th>
<th>Number Density (mol/cm³)</th>
<th>Neutron cross-sections ($10^{-24}$ cm²)</th>
<th>$\Sigma_j$</th>
<th>$\Sigma_i$</th>
<th>$\Sigma_a$</th>
<th>$\Sigma_j$</th>
<th>$\Sigma_i$</th>
<th>$\Sigma_a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A7050</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>26.98</td>
<td>0.892</td>
<td>47.7</td>
<td>119.6</td>
<td>0.0903</td>
<td>1.495</td>
<td>0.008</td>
<td>0.198</td>
<td>1.794</td>
<td>0.00968</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>63.55</td>
<td>0.023</td>
<td>48.6</td>
<td>3.14</td>
<td>0.0010</td>
<td>7.485</td>
<td>0.550</td>
<td>3.241</td>
<td>11.277</td>
<td>0.00016</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>24.31</td>
<td>0.023</td>
<td>38.2</td>
<td>2.42</td>
<td>0.0026</td>
<td>3.631</td>
<td>0.080</td>
<td>0.054</td>
<td>3.768</td>
<td>0.00013</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zn</td>
<td>65.38</td>
<td>0.062</td>
<td>54.4</td>
<td>9.48</td>
<td>0.0027</td>
<td>4.054</td>
<td>0.077</td>
<td>0.952</td>
<td>5.086</td>
<td>0.00051</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>144.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.0051</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**X-ray 50% transmission thickness, mm**: 0.05

**Neutron 50% transmission thickness, mm**: 76.61

### Table 2.1: Calculated 50% transmission thickness for laboratory X-rays with $\lambda = 1.5418$Å, corresponding to CuKα radiation, and neutrons of the same wavelength. Mass absorption coefficients for X-rays were taken from [International Tables for Crystallography 1974]. Neutron cross-sections are from [Sears 1992].

There are two main types of neutron sources for the production of intense neutron beams: steady-state nuclear reactors and accelerator-based spallation sources. In reactors the neutrons are released by fission of $^{235}$U, whereas in spallation sources neutrons are obtained via the disintegration of heavy nuclei (Ta, W, Pb, U etc) by bombardment with high-energy protons. In both cases the energy of the “as-generated” neutrons is too high to be used for diffraction experiments. That is why these neutrons have to be moderated through repeated collisions with atoms of a moderator such as graphite or heavy water (D₂O). Moderated neutrons with energies in the range between 1 and 100 meV have
wavelengths comparable with atomic spacings in crystals. In order to deliver polychromatic beams of neutrons to the location of scattering instruments without significant loss of flux neutron guides are often utilized. An example of a typical neutron spectrum so obtained is shown in Fig. 2.1. One can recognize that such a spectrum will predetermine the intensity of diffraction peaks obtained at a selected neutron wavelength (energy). In the following sections, description of the neutron instruments and methods used for strain scanning experiments in this work will be given.

![Figure 2.1: Spectrum of moderated neutrons obtained from the reactor “Orphée” at LLB (Saclay, France) for the cold-neutron guide G5. The moderator is liquid hydrogen [The Reactor and the Neutron Sources, 2002]. Dashed vertical lines indicate the recommended range of wavelengths [Ceretti 2000]. See the text for other details.](image)

2.1.1 **Instruments at steady-state sources**

The instruments using fixed wavelength sources of neutrons used in this work were G5.2 [Ceretti *et al*. 1995] of Laboratoire Leon Brillouin (Saclay, France) and D1A [Pirling & Wimpory 1997] of the Institute Laue-Langevin (Grenoble, France). The first component in such instruments is a monochromator (see Fig. 2.2). Its function is to select from the polychromatic beam neutrons that have a wavelength within a relatively narrow set band and to direct these neutrons onto the sample. The way in which this wavelength is selected
on G5.2 is different from that on D1A. As only (002) and (004) reflections from a single crystal of pyrolytic graphite are used to monochromate the neutron beam on G5.2, the selection of a particular wavelength is achieved via continuous change of the monochromator take-off angle, $\theta_M$. That means that the whole instrument has to be rotated about the monochromator axis. When using the (002) reflection, $\theta_M$ is within the range from about 46 to about 97 degrees for a recommended range of wavelengths from 2.6 to 5Å. On D1A, the take-off angle is fixed at 122° in order to get the highest angular resolution [Margaça 1992] and, hence different reflection planes of a Ge monochromator are used for obtaining a set of discrete wavelengths, which are listed in Table 2.1 together with other parameters for both diffractometers.

![Schematic of experimental set-up for the strain scanning on G5.2](image)

**Figure 2.2: Schematic of experimental set-up for the strain scanning on G5.2**

<table>
<thead>
<tr>
<th>Monochromator</th>
<th>Specimen (Al-based, Fm3m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crystal</td>
<td>$2\theta_M$</td>
</tr>
<tr>
<td><strong>G5.2</strong></td>
<td></td>
</tr>
<tr>
<td>Graphite</td>
<td>59.6</td>
</tr>
<tr>
<td>Graphite</td>
<td>79.3</td>
</tr>
<tr>
<td><strong>D1A</strong></td>
<td></td>
</tr>
<tr>
<td>Ge</td>
<td>122</td>
</tr>
<tr>
<td>Ge</td>
<td>122</td>
</tr>
<tr>
<td>Ge</td>
<td>122</td>
</tr>
<tr>
<td>Ge</td>
<td>122</td>
</tr>
</tbody>
</table>

*) Approximate values. More accurate wavelengths are usually found by calibration.

Table 2.2: Parameters used to select the wavelength on G5.2 and D1A for strain scanning experiments.
The instruments are equipped with either linear (on G5-2) or 2D (on D1A) position sensitive detector (PSD) to enable a complete diffraction peak to be measured simultaneously. The PSD's angular position, $2\theta_{\text{det}}$, remains fixed during the measurements with a selected $(hkl)$ peak (see the last column of Table 2.2). Calibration parameters allowing translation from the PSD's channel numbers into values of diffraction angle are found by several measurements of the diffraction peak at different $2\theta_{\text{det}}$.

Strain mapping requires the sample under study to be placed on a computer-controlled x-y-z-ω table (see Fig. 2.1), which is used to align the sample and move any point of interest into the gauge volume. The gauge volume is defined by the intersection of the incident and diffracted beams. The height and width of the volume may be chosen by adjusting the cross-section of the incident beam using neutron-absorbing cadmium ($\Sigma_a = 2520$ barns [Sears 1992]) slits. The depth of the volume can be controlled by limiting the size of the detector aperture. In order to provide the possibility to measure the strain in three orthogonal directions, it is necessary to dismount the sample, and manually reorient it, since engineering samples are rarely suited for use in a Eulerian cradle. In order to avoid manual sample handling mislocating the gauge volume within the sample, special holders can be prepared to satisfy the high accuracy requirements necessary for neutron strain scanning [Webster & Wimpory 2002]. The location of the sample surfaces with respect to the beam centerline should be determined regularly by measuring the variation in integral peak intensity as the gauge volume enters and leaves the sample. The latter method has been used by a number of researchers for years [Ezeilo 1992, Brand & Prask 1994, Wang & Edwards 1996], as it is difficult or sometimes impossible to precisely determine specimen surface position using only theodolites and telescopes, due to large and irregular sample dimensions and/or complicated experimental set-ups [Wang, D.Q. et al. 2000]. An example of typical scans obtained in this work for a 7XXX aluminium alloy plate is shown in Fig. 2.3. Regardless of the experimental geometry (transmission or reflection), the surface position was always found as a centre, $x_0$, of a simple sigmoidal fit,
\[ y = a_2 + (a_1 - a_2) \cdot [1 + \exp\left(\frac{x - x_0}{dx}\right)] \], to the data. Due to the small attenuation of neutrons by aluminium (\(\mu \sim 0.1\text{mm}^{-1}\)) the difference between surface positions given by this method and that obtained with the program “SURF V1.5”, described in [Wang, D.Q. et al. 2000], is less than 0.1mm.

Figure 2.3: Experimental scan obtained in reflection geometry for an Al alloy plate and two fits based on different models.

It should be noted, however, that the above method is not always possible in specimens which possess a through-thickness texture variation as described in detail in section 3.4.2. The most reliable way of finding the surface position of an Al-alloy plate would be to perform similar scans using a piece of aluminium (or other suitable material) of well-defined dimensions, preferably with a strong and well-known texture, attached to the specimen (see Fig. 2.4). This proved to be a very practical method when dealing with machined plates, where the texture variation is further complicated by the presence of deformed surface layers.
Figure 2.4: Precise determination of the surface position of a machined Al alloy plate. A complex through-thickness intensity variation (a), schematic of the experimental set-up (b) and intensity scan (c) for the piece with constant texture attached to the plate as shown in (b).

In order to calculate d-spacings and strains, diffraction peak positions are usually found by fitting experimental data to analytical peak-shape functions. The pseudo-Voigt approximation of the Voigt function is the most widely used for X-ray and constant wavelength neutron data [Thompson et al. 1987]. The pseudo-Voigt function, \( PV(x) \), is simply a linear combination of Gaussian and Lorentzian components in the ratio \( \eta/(1-\eta) \), where \( \eta \) is the pseudo-Voigt mixing parameter (after [Enzo et al. 1988]):

\[
PV(x) = l_p[\eta G(x) + (1 - \eta)L(x)]
\]  

(2.4)

where \( G(x) = \exp(-\ln 2 \cdot x^2) \) and \( L(x) = (1 + x^2)^{-1} \) with \( x = (2\theta - 2\theta_0)/\omega \), \( 2\theta_0 \) is the peak maximum position, \( 2\omega \) the full width at half maximum (FWHM), \( l_p \) the intensity at the peak maximum. This function describes a diffraction peak quite well, as it is essentially symmetrical at the high diffraction angles, \( 2\theta \sim 90^\circ \), usually used in neutron strain scanning experiments. However, constant-wavelength neutron diffractometers have much lower resolution than constant-wavelength X-ray instruments. That is why the peak shapes are nearly Gaussian (\( \eta \rightarrow 1 \)) because of a dominant instrument contribution (see Fig. 2.5). The use of constant background is of particular importance when dealing with “under-collected” data [Webster 2003].
Figure 2.5: The observed (circles), calculated (line) and difference (bottom) profiles for the (111) peak from Ge powder measured on G5.2.

2.1.2 Time-of-flight instrument

ENGIN is a specialized diffractometer based on the PEARL beam line at the pulsed neutron source ISIS, Rutherford Appleton Laboratory, UK. It was designed to meet a number of important requirements for strain scanning in real industrial components [Johnson et al. 1997]:

- Strain within samples could be measured up to ±50µstrain;
- Gauge volumes within samples could be resolved down to <2mm³;
- Samples could be scanned up to ±130mm (x, y, z);
- Samples could be positioned to ±0.1 mm;
- Samples could weigh up to 250 kg.

A schematic of the instrument is shown in Fig. 2.6. Here the incident beam is collimated by a pair of slits made from sintered boron carbide (B₄C). With 2-5 mm wide slits its divergence is about 0.2° [Wang 1996]. To focus the diffracted beams, there are two collimators, each containing 41 gadolinium oxide coated Mylar foils. The collimators
define the third dimension of the gauge volume to ~ 1.4 mm [Wang & Edwards 1997, Withers et al. 2000]. Diffracted beams are detected by two banks. Each detector bank contains 135 detectors arranged in three rows of 45 detectors stacked above each other, behind the collimators. Each detector collects neutrons at a slightly different scattering angle within an approximate 20 range from 81.4 to 98.6° in the horizontal plane. Together with the divergence of the diffracted beam in vertical plane (±11.3°) this effectively acts as angular oscillations of the sample allowing a relatively large number of grains to be measured.

The data from individual detectors are combined by the focusing routine, which accounts for the small difference in path length for each detector. This is because, in the time-of-flight (TOF) method used on ENGIN and described in detail elsewhere [Windsor 1981], the detectors record the time of arrival of the neutrons generated by a spallation source and slowed down by a moderator. Neutrons with the highest velocity (or energy) will take the smallest time to arrive at the sample, and subsequently after scattering, at the detector, followed by those with progressively smaller velocity (or energy). As each pulse

Figure 2.6: Schematic of ENGIN
contains a continuous spectrum of energies, neutrons of many wavelengths are always available for diffraction so permitting the measurement of the whole diffraction spectrum at a fixed orientation of the sample and detector banks. A typical TOF spectrum is shown in Fig. 2.7 together with the result of Rietveld refinement [Rietveld 1969] and corresponding residuals. The TOF axis can be converted to the d-spacing axis using a linear relationship, which is derived from the de Broglie equation (see Equ. 2.1) and Bragg’s law at $2\theta = 90^\circ$:

$$d = \frac{h \cdot \text{ToF}}{\sqrt{2} \times m \times L},$$

where $h$ is the Planck’s constant, $m$ - the neutron mass and $L$ - the distance of neutron flight. In practice, the diffractometer constant, $C = \frac{h}{\sqrt{2} \times m \times L}$, is routinely determined by the measurement of a standard with a well-known lattice parameter. Silicon powder is usually used to calibrate ENGIN.

The most important problem in applying the Rietveld method to pulsed neutron source data is the peak shape and its wavelength dependence [David & Jorgensen 1993]. The neutron pulse from the moderator is highly asymmetric. The leading edge is very sharp, because the first neutrons to emerge are almost coincident with the high-energy neutron pulse hitting the spallation target, while the trailing edge decays according to the moderator size and temperature. The other contribution to the resolution function is Gaussian and determined by the instrument design. That is why the peak shape function widely used for full profile refinement and also installed in General Structure Analysis System (GSAS) [Larson & Von Dreele 2000] is based on a convolution of separate rising and falling exponentials that represents the time dependence of the initial neutron pulse with a symmetric Gaussian term. Within the GSAS environment, the function used to fit the TOF spectra of the present work is an extension of the above convolution to include the Lorentzian broadening usually exhibited by most samples [Larson & Von Dreele 2000]. This function contains 21 profile coefficients, 5 of which are non-zero and usually obtained
from diffraction data for the standard sample. Two coefficients, which are worth refining together with lattice parameter, are $\sigma_1^2$ and $\gamma_1$, as they may be different for the sample under study due to the slit size used for its measurements (i.e. the instrumental broadening $\sigma_1^2$ may vary) and strain broadening contribution (i.e. $\gamma_1$ may vary). GSAS also allows one to use a definite advantage of the TOF data: different (hkl) peaks can be fitted individually, yielding valuable information regarding the presence of microstresses. An example of the fit to an individual peak is shown in Fig. 2.8. Here $\sigma_1^2$ was also refined. For relatively weak peaks, this may result in peak shifts, equivalent to $\approx 100 \, \mu$strain (see Fig. 2.9). The purple line shows residuals for the fits.

The presence of two detector banks enables two directions in a sample to be measured simultaneously. For flat samples, like butt-welded plates, the right bank (RB) of the detectors can be used for measurements in transmission, whilst the left bank (LB) – for measurements in reflection (see Fig. 2.6). Though manual sample handling is still required to set up measurement in the third direction, the measurements in reflection are performed twice, so the actual locations of the gauge volume within the sample can be verified by making comparison between two sets of data. It should be noted, however, that the RB on ENGIN is characterized by better resolution and smaller acquisition time for obtaining a statistically significant value of the lattice parameter than the LB. This is clearly illustrated by Fig. 2.10, where the lattice parameter of aluminium powder is plotted against measurement time assuming that the neutron current is 180 $\mu$amps/hr. Similar plots produced for typical places within the sample allow one to determine an optimum measurement time.
Figure 2.7: Time-of-flight neutron diffraction spectrum obtained from Si standard that is used to calibrate ENGIN. The spectrum is fitted using GSAS [Larson & Von Dreele 2000]. Residuals for the fit are shown by the purple line.

Figure 2.8: The (220) peak from a highly textured Al-alloy fitted using GSAS.
Figure 2.9: Fits to the (111) peak performed with fixed $\sigma_1$, equal to that obtained for the silicon standard, (a) and by refining $\sigma_1$ (b).

Figure 2.10. Lattice parameter of Al-powder at different measurement times. The measurements were performed with a 1.5x20 mm$^2$ incident beam.

ENGIN was used here to measure three strain tensor components across 12.6 mm thick FS and MIG welds and to perform a part of the hybrid experiment designed to obtain a full strain tensor for the machined sample of the MIG weld. Unless stated otherwise, all lattice parameters were found using a structure-free approach [Le Bail et al. 1988] where the intensities of the reflections are simply adjusted to fit the observed ones.
2.2 Synchrotron X-ray strain mapping

When high energy electrons are deflected by strong magnetic fields, they emit electromagnetic waves called synchrotron radiation. Covering the whole spectral range from infrared to hard (short wavelength) X-rays, the light produced by a storage ring comes in the form of a fine and very intense beam. Synchrotron X-ray diffraction has recently emerged as an exceptionally powerful technique for strain mapping in industrial components, particularly in Al-based materials [Korsunsky et al. 1998, Oosterkamp et al. 2000, Webster et al. 2000], due to the following circumstances. In 1995 synchrotron sources of the third generation were created, which are now generating synchrotron X-ray beams that are up to a trillion \((10^{12})\) times more intense than those produced by X-ray tubes [see e.g. Krawitz 2001]. Three hard X-ray facilities were built. One of them is the European Synchrotron Radiation Facility (Grenoble, France), where a part of the experimental work reported here was done. Unlike conventional X-rays, synchrotron X-rays are emitted with a wide range of energies, allowing a beam of any energy to be produced using appropriate monochromatization. Starting from energies above 25 keV the penetration depth in aluminium becomes more than 1 mm, permitting bulk strain measurements. This is because X-ray absorption strongly depends on the wavelength (or energy) of the incident beam [Noyan & Cohen 1987]:

\[
\mu = \rho \cdot K \cdot Z^4 \cdot \lambda^3, \tag{2.6}
\]

where \(\rho\) is the density of the absorber, \(Z\) is the atomic number and \(K\) is a proportionality constant. The corresponding dependence for aluminium \((Z=13)\) is shown in Fig. 2.11.

2.2.1 Advantages and disadvantages of the technique

The combination of a high intensity incident beam with low absorption facilitates incredibly fast measurements in Al-based materials if performed in transmission (Fig. 2.12a). Typically they take only tiny fraction of time needed for the equivalent measurements with neutrons.
The main disadvantage of the technique, however, is that measurements in reflection on flat specimens are difficult to make, preventing one from obtaining a full strain tensor. This is because the small wavelengths, characteristic of synchrotron X-rays, lead to grazing diffraction angles for the most useful reflections. As a result large X-ray paths \((2\times D/\sin\theta)\) are unavoidable (see Fig. 2.12b) and through-thickness strain scanning in reflection appears to be impractical due to extremely high absorption. In addition, accurate determination of the strain tensor for a measurement location requires that effective shift of the gauge centre is taken into account, as the detected X-rays are not contributed evenly from the whole instrumental gauge. This is important for near surface measurements [Webster et al. 1998] or even for bulk measurements when the latter are performed in other common alloys, based on a more absorbing element than Al (e.g. steels). The necessary correction will be difficult to make when dealing with components exhibiting through-thickness variations of preferred orientations. Figure 2.13 shows an example of the shifts expected for aluminium \((\mu_{\text{Al}}=0.154 \text{ mm}^{-1})\) and iron \((\mu_{\text{Fe}}=2.852 \text{ mm}^{-1})\) plates with a
constant texture. Whilst for bulk measurements (GCP ≥ 0.5) in Al this shift is rather small, for Fe the origin of the detected signal is heavily weighted towards the surface [Withers & Webster 2001].

\[
\mathbf{q} \parallel \text{any "in-plane" direction}
\]

\[
L_{TR} = \frac{t}{\cos \theta} = \text{const}
\]

\[
L_{RE} = \frac{2D}{\sin \theta}
\]

Figure 2.12: Synchrotron X-ray flight paths for measurements in transmission (a) and reflection (b).

Figure 2.13: Effective shift of the sampling volume versus geometrical centre position (GCP) of the instrumental gauge. Arbitrary unit (a.u.) represents a portion of the shortest gauge diagonal. When whole gauge is inside the plate (GCP ≥ 0.5), the shift is constant (After [Kang 1996]). Calculations are made for a slit size of 0.5 mm that is much smaller than the absorption length for Al.
Another consequence of the small diffraction angles is the diamond shaped gauge volume. This affects spatial resolution, which will be poorest in the normal (or through-thickness) direction (ND) when performing strain measurements in transmission (see Fig. 2.12a). The length of the gauge that determines the resolution is given by:

\[ D_L = \left( \frac{v_i}{2\sin\theta} + \frac{v_d}{2\sin\theta} \right) \]  

(2.7)

where \( v_i \) is the incident aperture width and \( v_d \) the width of the diffracted beam aperture (Fig. 2.14). If identical apertures are used (\( v_i = v_d = v \)), the resolution becomes proportional to the longest diagonal of the gauge’s cross-section. For transmission mode, the effective gauge centre is coincident with the geometrical one and the peak intensity is proportional to the gauge cross-section, which has a simple form:

\[ \text{Area} = \frac{v^2}{\sin2\theta} \]  

(2.8)

Close inspection of the above formula and absorption dependence (Equ. 2.6) will lead one to the conclusion that there is a certain range of plate thickness where the same (hkl) peak cannot be measured faster by switching to the higher energies if the resolutions in the directions perpendicular to the scattering vector \( \vec{q} \) (strain measurement direction) are to be kept unchanged.

Figure 2.14: Elongation of the gauge volume observed at diffraction angles typical for synchrotron X-ray diffraction.
2.2.2 BM16 (ID31) beamline and instrument configuration

The instrument used for strain mapping in the MIG weld was based on the BM16 bending-magnet beamline at ESRF (it has recently been moved to the undulator beamline ID31 and used to measure a VPPA weld; undulators, the most advanced class of magnetic Insertion Devices (ID), enhance photon flux by a factor of 10 or more over Bend Magnet (BM) sources [e.g. Corbett & Rabedeau 2002]), where high angular resolution (starting from 0.006°) is routinely obtained using the parallel beam geometry with a monochromator/analyser combination [Hodeau et al. 1998]. Though the instrument may be operated over a wide X-ray energy band [Webster et al. 2001], X-rays of the highest energy (~ 40 keV) were selected to perform the measurements in transmission mode. The instrumental peak shape on BM16 is found to be fitted well [Masson et al. 2001] with a modified Pseudo-Voigt function convoluted into the asymmetry function described in [Finger et al. 1994]. However, as the synchrotron diffraction peaks used here were seen at relatively high diffraction angles, from 15 to 22 deg., they were symmetrical enough to be fit with the PV profile function. This function was characterized by relatively small \( \eta \) that is attributed to the effect of analyser crystals that precede the parallel scintillation counters [Masson et al. 2001]. In fact, a Lorentzian function (\( \eta = 0 \)) is also adequate for finding peak positions. It will suffice to say that the corresponding difference can be much smaller than an error of the fit with either symmetrical profile function. Both types of fit to the same peak are exemplified in Fig. 2.15. The most important function of the analyser crystals, as far as the strain measurement is concerned, is that they eliminate the anomalous surface effect characteristic of other instruments [see e.g. Webster et al. 1996, Wang 1996].

Because of the large dimensions of the test-pieces involved in the study (280x280 mm\(^2\)), a special set-up was designed to operate the instrument in its strain-mapping mode, using some components of the positioning equipment developed by P. J. Webster and co-workers [Webster et al. 2002]. The main components of this set-up are indicated in a photograph shown in Fig. 2.16. One of the inner detectors (No.4) was used to record the
diffracted beam. Movements of slits and an XYZ translation system were computer controlled. Other parameters of the synchrotron scanning experiments are given in Sections 5.1 and 5.4.

Figure 2.15: Fits to the (222) peak using pseudo-Voigt (a) and Lorentzian (b). Measurements of AA7150 plate were made in TD.

Figure 2.16: Experimental set-up used for the synchrotron strain scanning experiment: 1 – Detectors; 2 - Box with anti “cross-talk” blades, filled with He; 3 – Analyzer crystals (Ge) in He atmosphere; 4 – Secondary slit; 5 – 2θ-table; 6 – Ω-table, not in use, however restricting the range of horizontal scanning; 7 – Test-piece, 8 – χ-drive used to set 0-2θ diffraction geometry; 9 – z-table and 10 – x-y table for the 3D translation of the test-piece.
3 SPECIMENS AND THEIR PRELIMINARY CHARACTERIZATION

3.1 Welds and preparation of test-pieces

A double pass MIG and a single pass VPPA welds were produced and supplied by the Cranfield University Weld Engineering Research Centre as part of an EPSRC funded joint project entitled “Weld processing design & durability of welded aircraft assemblies”. 12.6 mm thick 500 mm long AA7150 plates were welded in the W51 condition (solution treatment, quenching followed by stress relief by stretching (~2%) in the rolling direction). AA5039 filler wire was used to produce the MIG weld, whilst the VPPA weld was fabricated autogenously, using the “keyhole” technique (see Section 1.3.2 and e.g. [Woodward et al. 2000]). Travel speed of the heat source during the MIG process was about 7 mm/s. The VPPA welding torch velocity was 2 mm/s. A conventional peak aging treatment was applied to both specimens following welding, equivalent to T6 (i.e. heat treatment at about 120°C for 24 hours), that resulted in a T651 condition in the far field parent material (PM) and related conditions in the heat affected and fusion zones. The weld orientation in the resultant 280 mm wide specimens was parallel to the rolling direction (RD) of the parent plates.

Figure 3.1: Position of the test-piece in the specimen of the MIG weld. The missing weld section was used for the reference specimen preparation. Vertical dotted line indicates the trace of the stress measurement plane.
In order to perform residual stress measurements, 280 mm square test-pieces were cut off, as shown in Fig. 3.1 for the MIG weld. The MIG weld test-piece was then used in the measurements by neutron diffraction. Subsequently both test-pieces were reduced in thickness to 7 mm by machining from both sides in increments of 0.5 mm to simulate a likely aerospace manufacturing process. The machined test-piece of the MIG weld was measured again using a hybrid neutron/synchrotron diffraction technique described in Section 5.1. The triaxial stress field around the VPPA weld was measured using synchrotron X-rays.

Friction stir welds were prepared in the Edison Welding Institute (USA) using AA7050 alloy plates in T7451 condition. (T7451 is an overaged temper, specially developed by the aluminium industry to optimize corrosion resistance and mechanical properties [see e.g. Hill & Panontin 2002], and is widely used by aircraft manufactures. This consists of solution heat treatment at 475°C, quenching and stress relief to achieve the W51 condition followed by two-stage treatment: at 107°C for 6-8 hrs and at 163°C for 24-30 hrs [Aluminium and Aluminium Alloys 1993]). Fabricated in a single pass, these welds were of full penetration with the rotating tool probe sufficiently close to the bottom of the plate to include the entire butt joint. Three specimens were studied. The 12.6 mm thick specimen was subjected to a lengthy post-welding aging at 120°C. A similar treatment was applied to other specimens as was inferred from the corresponding hardness distributions shown in Section 3.3. The dimensions of the corresponding test-piece were 92 mm in the longitudinal direction (LD) and 306 mm in the transverse direction (TD). After a TOF experiment on ENGIN, its transverse dimension had to be reduced to 190 mm, as it was dictated by the geometry restrictions of the experimental set-up on the G5.2 diffractometer at Saclay. Other specimens, 6.3 mm thick, prepared by machining of the original 12.6 mm thick plates from one side before welding, were manufactured using substantially different welding speeds (specimens A and B in what follows). Unlike the 12.6 mm thick weld, these specimens were slightly distorted as shown in Fig. 3.2 (with $\gamma$ up to 0.3°, for the
specimen B produced using higher welding speed). Detailed welding parameters, such as tool rotation speed and the work-piece travel speed, are proprietary to the manufacturer. However, under the assumption that tool rotation frequency was fixed [Crompton 2000], the result of the measurements on semicircular features seen on the “shoulder side” areas of both welds (see Fig. 3.2) suggests that the welding speed for the specimen B was double that for A, as the spacing between the features, $s$, matches the distance moved by the tool in one rotation [Krishnan 2002]. The residual stress state in 210 mm square test-pieces was studied. Nominal compositions of the alloys used in fabrication of the welds are given in Table 3.1.

![Figure 3.2: The shoulder side areas of the FS welds and schematics showing the semicircular features and distortion of the welds characterized by angle $\gamma$.](image)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cu</th>
<th>Mg</th>
<th>Mn</th>
<th>Zn</th>
<th>Cr</th>
<th>Zr</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>7150</td>
<td>2.2</td>
<td>2.4</td>
<td>-</td>
<td>6.4</td>
<td>-</td>
<td>0.12</td>
<td>bal</td>
</tr>
<tr>
<td>7050</td>
<td>2.3</td>
<td>2.2</td>
<td>-</td>
<td>6.2</td>
<td>-</td>
<td>0.12</td>
<td>bal</td>
</tr>
<tr>
<td>5039</td>
<td>-</td>
<td>3.8</td>
<td>0.4</td>
<td>2.8</td>
<td>0.15</td>
<td>-</td>
<td>bal</td>
</tr>
</tbody>
</table>

Table 3.1. Nominal composition of the alloys used (wt %) [Aluminium and Aluminium Alloys 1993]
3.2 Reference specimens and their measurement conditions

For each test-piece of the MIG and FS welds, a 90 mm long comb-like reference specimen or individual rectangular pieces were prepared by Electro Discharge Machining (EDM) to be used for the $d_{\text{ref}}$ ($a_{\text{ref}}$) measurements (see Section 5.1 and Chapter 6 for a detailed definition). A photograph of one of the combs is shown in Fig. 3.3. The corresponding dimensions for each comb are given in Table 3.2. Note that for the VPPA weld the corresponding reference specimen was 120 mm long due to a much wider HAZ, as shown in Section 3.3.

![Figure 3.3: Reference specimen cut from the MIG weld.](image)

**Table 3.2: Parameters of the reference specimens indicated in Fig. 3.3 and their thickness.**

<table>
<thead>
<tr>
<th>Specimen of</th>
<th>Parameters of reference specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>t, mm</td>
</tr>
<tr>
<td>thick FS weld</td>
<td>12.6</td>
</tr>
<tr>
<td>thin FS welds (A and B)</td>
<td>6.3</td>
</tr>
<tr>
<td>MIG weld</td>
<td>12.6</td>
</tr>
<tr>
<td>VPPA weld</td>
<td>12.6</td>
</tr>
</tbody>
</table>

Unless specified otherwise, reference measurements were made at orientations corresponding to equivalent measurements in the bulk material (see also Chapter 6). A picture taken of the experimental set-up for the reference measurements for the 12.6 mm thick FS weld is shown in Fig. 3.4b. Here 21 $a_{\text{ref}}$-pieces, cut to cover all the measurement
locations from -43.5 to +43.5 mm within the cross-sectional area perpendicular to LD (fragment of the correspondent layout and orientations are shown schematically in Fig. 3.4a), were glued to a 1.5 mm thick Al sheet to form a "comb"-like construction, which could facilitate $a_{\text{ref}}$ mapping. Unfortunately, the LD and ND measurements were made only on 7 pieces (red-coloured in Fig. 3.4a) due to a shortage of beam-time available for the experiment.

Figure 3.4: Layout of the pieces cut from the 12.6 mm thick FS weld (a) and experimental set-up on ENGIN (b) used for the reference measurements. The "dash and dot" line in (a) indicates the position of the weld's centre.

### 3.3 Hardness profile measurements

Measurements of hardness were performed with a Vickers diamond using a "Miniload" Leitz Wetzlar test machine. Indentations were made by applying a load of 500 grams for about 20 seconds. 1.5 mm thick transverse sections of the 12.6 mm thick FS and MIG welds and the reference specimens for 6.3 mm FS welds and the VPPA weld were used to carry out the measurements. Their ND-TD surfaces' finish was as for metallographic examination, i.e. involving step-by-step grinding and diamond polishing (1μ in its final stage).
Maps of hardness for the 12.6 mm thick welds were produced using 1 and 2 mm steps in both directions, whilst hardness profiles for the specimens A and B were taken only at mid-depth at an interval of 1.5 mm. An interval of 3 mm was used to perform measurements along the through-thickness midline of the VPPA reference specimen. The results for the FS welds are shown in Fig. 3.5 and 3.6, whilst Figure 3.7 shows the midline hardness profiles for the MIG and VPPA welds.

Figure 3.5: Hardness profile for the 12.6 mm FS weld. The beginning of the through-thickness axis corresponds to a depth of about 0.3 mm below the “root” surface.
Figure 3.6: Mid-depth hardness distributions for the 6.3 mm thick plates FS welded using lower (a) and higher (b) welding speeds. Distribution for the 12.6 mm weld is also given in (c) to facilitate discussion.

Figure 3.7: Mid-depth hardness distributions for the MIG (a) and VPPA (b) weld. A 2D map for the whole cross-sectional area of the MIG weld was produced by Mr. S. Ganguly, part of which can be found in [Stelmukh et al. 2002].
3.4 Optimizing strain mapping by the use of EBSD

A number of experiments reported here were performed at a reactor neutron source, where a monochromator is used to produce a collimated beam of neutrons with a single wavelength $\lambda$. Although this wavelength could be varied as described in Section 2.1.1, for a polycrystalline material with cubic symmetry only a few reflections give an acceptable neutron count when using the preferred scattering angle close to 45 degrees. This geometry is desirable as it produces a similar spatial resolution for all three orthogonal measurement directions due to the rectangular-shaped gauge. As 7XXX alloys exhibit crystallographic texture (see e.g. [Dutkiewicz & Bonarski 1997, Chang et al. 1998]), preliminary assessment of the most advantageous reflections and sample orientations was necessary, since the above test-pieces were too large to be attached to an Eulerian cradle. Although electron backscatter diffraction (EBSD) is mainly used for the determination of point-to-point misorientation relationships and local textures [Schwarzer 2000], it can also be used to examine macrotexture, provided some information is available about how the material was processed. In addition, EBSD orientation mapping can be used to determine grain size distributions, and hence estimates can be made of the number of suitably oriented grains that would contribute to the intensity of a neutron diffraction peak. These combined advantages of the EBSD technique open up new opportunities for optimising strain measurements in engineering components exhibiting texture variation [Stelmukh & Edwards 2002].

3.4.1 EBSD specimens and experimental details

Systematic measurements were made only for the 12.6 mm thick FS weld. Its cross-section perpendicular to the LD was the main object of the investigation. In addition, there were several complementary specimens with the TD-ND and TD-LD acquisition surfaces prepared for specific purposes (find more on page 62). As the 7150 alloy was in a different condition from the 7050 alloy even before MIG welding, a couple of places in the HAZ
and the parent material were also measured. The EBSD specimens were prepared by cutting using EDM, gentle grinding, diamond polishing and subsequent etching.

Electron back-scattering patterns (EBSP) resulting from backscattered electrons were obtained using hardware from Jelen Technology Norway, mounted on a JEOL 820 scanning electron microscope (SEM) operating at 25kV. The specimen was inclined in the SEM at 70° to the incident electron beam. Figure 3.8b shows an example of EBSP from the 7050 alloy consisting of many intersecting, linear features those geometry and crystallographic information are equivalent [see e.g. Randle and Engler 2000] to Kikuchi bands [Kikuchi 1928]. The edge of each band is geometrically attributable to electrons that have been diffracted from a particular plane of atoms within the specimen (see Fig. 3.8a). The software used to acquire and analyze the EBSD bands (Channel 4) was produced by HKL Technology ApS in Denmark. Automated scans were carried out in the stage-scanning mode using 10-µm steps within a 1 mm square grid pattern. The location of several regions, each containing about 10000 measurement points, is indicated on the macrophotograph of the weld cross-section shown in Fig. 3.9f. The fraction of EBSD patterns that could not be indexed for any region was never higher than 17%.

Figure 3.8: Schematic of Kikuchi band formation (a) and an example of EBSP obtained from an Al-based alloy and its indexing (b).
The resulting texture data is presented here as pole figures generated by post processing software (Mambo, HKL Technology), using the equal-area projection method because of its advantages for examining population densities. For example, a uniform density of points on a pole figure will correspond to a random distribution of orientations in an isotropic material.

3.4.2 Results of the EBSD measurements

The parent material (PM) and all other zones of the joint affected by the welding process with the exception of the very centre of the weld are characterised by a strong texture. Pole figures obtained for PM (Figs 3.9 a and d) confirm that the Brass, <211> (110), texture is one of the strongest components [Maurice & Driver 1997]. As can be seen by comparing multiples of uniform density (MUD), the preferred orientation is sharper in the inner portion of the plate than in its outer portion. Virtually no change in the texture was observed in the HAZ, presumably due to the relatively small heat input used in FS welding, which is a solid-state process [Tang et al. 1998]. The major transformations occur in the thermo-mechanically affected zone (TMAZ). The central part of the zone, or so-called “nugget”, differs from the rest of the TMAZ, as here the material is believed to undergo dynamic recrystallization [Murr et al. 1998]. It was found to have a fine equiaxed grain structure on the order of 10 μ in diameter, which is slightly higher than reported in [Jata et al. 2000]. The typical pole figure presented in Fig. 3.9b contains a distinctive maximum, which moves over the pole figure’s area when changing the position of the acquisition region within the nugget. In the vicinity of the nugget, the parent material is also subject to significant mechanical deformation that causes rotation of elongated grains. The manner in which this rotation takes place is dependent on the proximity of the area of interest to the top surface of the weld. For the mid-point, it is essentially uplifting of the grains (Fig. 3.9e), whilst additional rotation in the LD-TD plane is observed in the upper region (Fig. 3.9c). The latter is due to the deformation action of the tool shoulder, which is
in direct contact with the top surface of the plates during welding. Overall, the EBSD results agree well with the diffracted neutron intensity distributions obtained in subsequent experiments [see also Stelmukh & Edwards 2002].

Because of rather big steps used in the EBSD scans only approximate parameters of the "pancake" microstructure in the parent material and HAZ were obtained. The average values found using the method of intercepts with the usual criterion for level of misorientation (>15°) are 20, 60 and 100 µ for the ND, TD and RD, respectively. The maximal detected length in RD was about 1500 µ on the specimen with the ND-LD acquisition surface. Figure 3.10 shows the microstructure as maps of orientations represented by co-ordinates (φ₁, φ, φ₂) of Euler space [Bunge 1999]. One of the longest grains is highlighted in Fig 3.10a.

Figure 3.9: (111) pole figures (a-e) obtained for the regions indicated on the macrophotograph of the weld cross-section (f). The acquisition surface was perpendicular to LD. A macrophotograph of the top surface of the weld is given in (g).
The above trends in through-thickness texture variation gave us the first indication that it might be possible to make surface stress measurements using the standard $\sin^2\psi$ method and laboratory X-rays. In order to confirm the feasibility of such measurements the EBSD experiment was also performed for the specimen with the acquisition surface perpendicular to the ND. A layer of less than 15µ was gently removed from the original surface during specimen preparation. As can be seen from Fig. 3.11a, MUD maxima have become much weaker and there are no orientations of zero MUD. Pole figures for the \{hkl\} planes with higher multiplicity factor, $P_{hkl}$, (see Figs. 3.11 b, c) show that the variation in intensity of the corresponding diffraction peaks are likely to meet one of the criteria of applicability of the $\sin^2\psi$ method, recently outlined in [Fitzpatrick et al. 2002]. No attempt, however, was made to infer any texture component from the data obtained. The average grain diameter in the LD-TD plane was found to be about 20 µ. The texture index remains nearly the same to a depth of approx.1mm below the plate’s surface, giving rise to the possibility of accurate wall scans in strain mapping experiments.

Figure 3.10: Maps of orientations for the middle of the parent plate: the ND-RD (LD) cross-section, also used to calculate microstructural parameters (a) and the ND-TD cross-section, measured using 2-µm steps (b).
3.5 Planning neutron diffraction experiments

Aluminium is rather a poor neutron scatterer ($\Sigma_c = 1.495$ barns [Sears 1992]). Therefore neutron strain mapping cannot be performed accurately and efficiently unless the most advantageous reflections are selected. Analysis of the pole figures for $\{111\}$ and other $\{hkl\}$ planes shows that $(111)$ is the only fcc aluminium reflection that could be used, if the range of neutron wavelength readily available on the G5.2 diffractometer (from 2.6 to 5.0 Å) is also taken into account. At the wavelength of 3.335 Å used in the experiment, the $(111)$ peak was seen at 2θ close to 91° and could be measured in the TD and LD-α directions, where $\alpha$ (approx. 19.5°) is the rotation angle about the plate’s normal (see Fig. 3.12). The LD strains were obtained later from the strain data for LD-α and TD using a formula derived from plane trigonometry, equivalent to the construction of Mohr’s circles [Timoshenko & Goodier 1970]. This simplified approach is based on the fact that aluminium possesses little elastic anisotropy [Hosford 1993]. The ND strains were subsequently obtained in a separate experiment where the $(220)$ peak was used. This rather non-standard solution was found after close examination of the neutron spectrum shown in Fig. 2.1. The wavelength used, about 2.14 Å, is outside the recommended range, which is thought to be defined by the fact that the real shape of the neutron flux spectrum for the wavelengths less than 2.6 Å is not very well known, as it tends to fluctuate. In addition, an
inevitable decrease (more than 30%) in the neutron flux was expected, partly because the (400) reflection from pyrolytic graphite had to be used for the beam monochromatization. However, the intensity of the (220) peak was considerably enhanced by the brass texture component for most of the measurement places located in the HAZ and PM. The same approach was used to design an experiment on the D1A instrument at ILL. It should be noted, however, that this instrument is characterized by a much wider range of wavelengths suitable for measurements of common engineering alloys. These were listed in Table 2.2 and other details of the experiment will be given in Section 5.2.

![Diagram of diffraction angles](image)

Figure 3.12: Definition of the LD-α measurement direction (parallel to q) relative to the orientation of the neutron sampling volume.

EBSD measurements for 7150 alloy plate resulted in slightly different values of MUD. In practice, this means that a similar strategy could be adopted when designing strain mapping experiments for both materials. Some measurement places in the fusion zones (FZ) of the MIG and VPPA welds may be difficult to measure using (hkl) peaks with low multiplicity as it has a casting structure with much larger crystallites (up to 1-2 mm) and the condition of having sufficient number of crystallites randomly oriented to give a good statistical average may not be satisfied. However, the FZ will occupy only about 15% of the measurement cross-sectional area, i.e. even less than the nugget in the FS weld (see Fig. 5.11b and 5.26a for comparison).
A map of the crystal orientations for the strongest peaks present on the TOF neutron spectra obtained from the middle of the AA7150 plate is shown in Fig. 3.13a. Together with the corresponding pole plot (see Fig. 3.13b), this map will be referred to in Chapter 6 to facilitate discussion.

Figure 3.13: Crystal orientations in 7150 aluminium (a) and the corresponding pole plot (b) obtained by integration over all orientations in the TD-ND plane.
EXPERIMENTS USING LABORATORY X-RAYS

X-ray diffraction experiments were conducted for the following purposes:

- To obtain a first approximation to the “elastic stress-free” d-spacing distributions by measuring reference specimens at constant \(\psi=0\), when \(\vec{q}\) is parallel to LD (\(d_L\));
- To estimate remaining ND stress on the surface of the reference specimen using the crystalline group method described in [Baron et al. 1987];
- To determine surface residual stresses by means of a standard \(\sin^2\psi\) technique [Noyan & Cohen 1987] and verify its consistence with the results of the bulk measurements.

The measurements were performed using CrK and CuK radiations. Corresponding wavelengths are listed in Table 4.1 together with the \((hkl)\) peaks used and other parameters.

<table>
<thead>
<tr>
<th>Radiation</th>
<th>(\lambda, \AA)</th>
<th>((hkl))</th>
<th>2(\theta), deg.</th>
<th>Penetration depths (mm) at (\psi = 0) and 45(^\circ)</th>
<th>X-ray spot size, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuK(_{\alpha1})</td>
<td>1.54060</td>
<td>422</td>
<td>(~137)</td>
<td>0.036 / 0.025</td>
<td>1-2</td>
</tr>
<tr>
<td>CuK(_{\beta})</td>
<td>1.39222</td>
<td>440</td>
<td>(~153)</td>
<td>0.050 / 0.035</td>
<td>1-2</td>
</tr>
<tr>
<td>CrK(_{\alpha1})</td>
<td>2.28970</td>
<td>311</td>
<td>(~139)</td>
<td>0.011 / 0.008</td>
<td>2-3</td>
</tr>
</tbody>
</table>

Table 4.1. Parameters of X-ray diffraction experiments.

Comb specimens and the 12.6 mm thick FS weld were measured on a Bruker D5005 X-ray Diffractometer equipped with a Eulerian cradle manufactured by Huber. The surface of the comb specimens was prepared by gentle grinding and polishing using a number of grinding papers and diamond water suspensions (1 \(\mu\) at final stage of the preparation). The diffraction peaks were recorded by a linear PSD in the Bragg-Brentano focusing geometry. In \(\sin^2\psi\) experiments, tilting of the specimens was performed transverse to the plane.
defined by the primary and diffracted X-ray beams. Transfer from positive to pseudo-negative values of $\psi$ was made via a 180° rotation of the specimen around the normal to its surface (i.e. using $\phi$-rotation). The beam spot location was defined with accuracy better than 0.3 mm over the whole range of $\psi$ and $\phi$ angles used. This was achieved through a thorough alignment of the rotation centre of the x-y-z table and the cradle. For the sake of clarity, a photograph taken of the experimental set-up is shown in Fig. 4.1. Individual peak positions were calculated using either the sliding gravity method [Pfeiffer 1994] available in the data processing software supplied by the diffractometer manufacturer or by fitting with the PV function and a linear background. Examples of the latter fits are given in Fig. 4.2. Here two coupled PV functions were used to simulate the $K\alpha_{1,2}$ doublet. Typical sets of diffraction patterns (see Fig. 4.3) obtained for different zones along the measured line exhibit moderate variation in the peak intensity. As can be seen from Fig. 4.4, $\sin^2\psi$ dependencies close to linear were observed for most measurement parts. This resulted in relatively small errors in the stress measurement ranging from 3 to 15 MPa.

![Figure 4.1: Photograph taken from the experimental set-up on the Bruker diffractometer: 1 - incident beam collimation system, 2 - PSD, 3 - goniometer, 4 - table with x-y-z-$\phi$ translations, 5 - its attachment to the open cradle, 6 - test-piece of the FS weld.](image-url)
Figure 4.2: Typical peaks obtained from the highly textured zones of the reference specimen using CuK radiation. Fits and difference curve are also shown.

Figure 4.3: Typical set of diffraction patterns obtained in a $\sin^2\psi$ experiment with the (422) peak. Every other pattern is shown to save space.

A number of experiments were also conducted using an X-ray stress analyzer (XStress3000) from Stresstech, design of which permits measurements from very large specimens and even real industrial components or assemblies. This measurement system is
based on a solid-state sensor technique and operates at very small distances between the X-ray source, detectors and acquisition surface (see Fig. 4.5a). This facilitates very fast measurements. From the options available in the software supplied with the instrument, a cross-correlation method was selected for the real time analysis of the results, as it is characterized by enhanced utilization of the collected data permitting more accurate determination of relative peak positions, particularly for asymmetric and/or distorted peaks [Tönshoff et al. 1981]. Because of the relatively small penetration of the CrK radiation (see Table 4.1), oscillations for $\psi$ ($\pm 3-6^\circ$) and $\varphi$ ($\pm 3-5^\circ$) were used to increase the number of diffracting crystallites. The observed $\sin^2\psi$ distributions were linear only for places near the welding zones (Fig. 4.5b) characterized by relatively high tensile stress (>40 MPa). Places with small stresses exhibited pronounced splitting suggesting the presence of shear components (Fig. 4.5c). A strong non-linear behavior (Fig. 4.5d), often combined with shear splitting, was found for many places in the PM. As a result the errors of linear approximation ranged from 2 to 25 MPa.

Figure 4.4: Lattice parameters calculated from $d_{422}$ and relative intensities vs. $\sin^2\psi$ for measurement places in the HAZ (a) and PM (b) of the 12mm thick FS weld.
Figure 4.5: Photograph taken of the FS weld set for the measurements with the XStress3000 (a) and typical $d_{311}$-vs-$\sin^2\psi$ distributions obtained for the LD stress measurements (b-d). See the text for the details.
5 RESULTS

5.1 Studying an effect of mechanical treatment on the residual stress field in the MIG weld

Three separate experiments were conducted to determine residual stresses in a 7150 MIG weld before and after the machining described in Section 3.1. Strain components in the original weld were mapped using spallation-source neutrons, whilst both neutrons and synchrotron X-rays were used for strain mapping in the machined test-piece. Schematics of all experimental set-ups, approximate gauge shapes and dimensions are given in Fig. 5.1. Relatively poor spatial resolution in ND could be tolerated when using synchrotron radiation, as moderate strain/stress gradients in this direction were found during the original weld investigation. Single peak scans on BM16 were made with the (422) and (222) peaks to determine the LD and TD strains respectively, employing an experimental strategy similar to that described in Section 3.5. Note that the TD strains in the 7 mm thick plate were measured twice, allowing comparison to be made between the two methods.

When performing the synchrotron X-ray experiment, 7 measurement lines (at -3, -2, -1, 0, +1, +2 and +3 mm from the through-thickness midline) were distributed over the cross-sectional area perpendicular to the weld seam. The measurements across the weld were taken using a 1-mm step. For the neutron diffraction experiments, the number of measurement lines had to be reduced, as individual count times per point were much lower. The corresponding layouts of the measurement places for both conditions, i.e. before and after machining, are shown in Fig. 5.2. In order to avoid having to make corrections for the anomalous surface effect that occurs [Harris & Withers 1995, Wang et al. 1996, Wang et al. 1998, Edwards 2003] when the gauge volume extends beyond the sample surface, the through-thickness positions in the machined test-piece were confined to a narrower range, ± 2.5mm. To calculate the strain tensor for each measurement place d-spacing distributions for the missing lines were found by linear interpolation of the synchrotron data (± 3, ± 2 → ± 2.5 and ± 1, ± 2 → ± 1.5).
Figure 5.1: Details of strain scanning experiments performed on ENGIN (a, c) and BM-16 (b) to measure the MIG weld in the “as-received” state (a) and after machining (b, c). Approximate dimensions of the neutron gauge volume (a, c) are given along the measurement directions and take into account the neutron beam divergence. Parameters and directions in brackets shown in (b) are for the TD measurements.
Figure 5.2: Layouts of the measurement locations: for the ND (TD) scans (a) and the LD (ND), TD (ND) scans (b) in the test-piece before (b) and after (a) machining to the thickness of 7 mm. Horizontal dashed lines show the surface positions of the welded plates. All distances are given in mm.

Typical neutron diffraction spectra obtained from the parent material and HAZ are shown in Fig. 5.3. They are plotted together with the corresponding fits and residuals.

Figure 5.3: Spectra obtained on ENGIN in TD (a) and ND (b).

To aid comparison of the two TD measurements, the relevant results are presented in Fig. 5.4 in terms of a pseudo-strain obtained using a constant reference value, the average d-spacing for the parent material at each side. Good comparability of the two diffraction
techniques was found on comparing the TD measurements. These and all subsequent maps were obtained using bilinear interpolation of several hundred individual values using Gsharp 3.2 software. The positive through-thickness co-ordinate refers to the 1st pass side of the MIG weld.

About half the maximum in the pseudo-strain variation observed in the weld fusion zone is due to variation in the composition of the Al-based solid solution present in the filler metal, which essentially defines how the reference d-spacing value changes across the weld. The latter was also supported by the results of X-ray microanalysis used to measure compositional variation across the weld. The lattice parameter calculated for the FZ using Vegard's law (see Table 5.1) agreed well with the value obtained by the TOF neutron diffraction technique from the corresponding reference specimen. Unfortunately, a similar estimate could not be made for zones outside the FZ, where the composition of the solid solution in the precipitation hardened material is impossible to measure without using ultrahigh resolution techniques because of extremely small distances between precipitates enriched in alloying elements (see e.g. [Nicolas & Deschamps 2000]).

<table>
<thead>
<tr>
<th>Detectable component</th>
<th>Cu</th>
<th>Error</th>
<th>Mg</th>
<th>Error</th>
<th>Mn</th>
<th>Error</th>
<th>Zn</th>
<th>Error</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic radius (R), Å</td>
<td>1.28</td>
<td></td>
<td>1.60</td>
<td></td>
<td>1.26</td>
<td></td>
<td>1.39</td>
<td></td>
<td>1.43</td>
</tr>
<tr>
<td>Measured content (C), at%</td>
<td>0.22</td>
<td>0.03</td>
<td>2.33</td>
<td>0.10</td>
<td>0.20</td>
<td>0.01</td>
<td>1.37</td>
<td>0.04</td>
<td>rem</td>
</tr>
<tr>
<td>Contribution to the change in average atomic radius (R0-R)/R0×C/100</td>
<td>-2.3E-04</td>
<td>-4.5E-05</td>
<td>2.8E-03</td>
<td>1.7E-04</td>
<td>-2.4E-04</td>
<td>-1.7E-05</td>
<td>-3.8E-04</td>
<td>-1.6E-05</td>
<td></td>
</tr>
<tr>
<td>Lattice parameter, Å</td>
<td>4.0572</td>
<td>0.0007</td>
<td>calculated using Vegard's law and lattice parameter for pure Al (4.0494Å)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4.0568</td>
<td>0.0002</td>
<td>obtained by fitting the whole neutron TOF spectra</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 5.1: Results of X-ray microanalysis used to calculate the “stress-free” lattice parameter, a₀, for the FZ. Metallic atomic radii are taken from [Alcock 1990]

As mentioned earlier (see Section 1.6) and discussed in Chapter 6, the measurements taken from both the test-pieces and reference specimens are generally affected by the presence of microstresses. In order to avoid any confusion in what follows, the d-spacings (lattice parameters) obtained from the reference specimens will be referred to as the
reference d-spacings, \( d_{\text{ref}} \) (lattice parameters, \( a_{\text{ref}} \)), or simply reference data, to distinguish them from so-called \( d_0 \) (\( a_0 \)) values, which correspond to the "stress-free" condition and are solely determined by the content of the alloying elements in a metal-based solid solution with cubic structure. Only in specific cases (see e.g. Table 5.1) will \( d_{\text{ref}} \) (\( a_{\text{ref}} \)) be equal to \( d_0 \) (\( a_0 \)).

The 3D map of reference data obtained for the orientation corresponding to the LD measurement direction in the test-pieces is shown in Fig. 5.5. The average d-spacing for the parent material was again used to calculate individual values of pseudo-strain.

Figure 5.4: Maps of pseudo-strain calculated using data obtained for TD in synchrotron X-ray (a) and neutron (b) diffraction experiments. For the sake of consistency only the (111) reflection of the TOF neutron spectra was analysed. Here and in all subsequent maps y coordinate refers to the distance (in mm) from the through-thickness midline.

Strain contour maps calculated for the 7 mm thick test-piece are shown in Fig. 5.6. Three separate maps of reference d-spacings, obtained for each measurement direction, were used in the calculations, as follows:

\[
\varepsilon_i = \frac{d_i - d_{\text{ref}}}{d_{\text{ref}}} \quad \text{and} \quad \varepsilon_j = \frac{a_j - a_{\text{ref}}}{a_{\text{ref}}},
\]

(5.1)

where \( i \) stands for LD, and \( j \) for TD or ND.
Figure 5.5: Reference data in terms of pseudo-strain obtained using synchrotron X-ray diffraction in LD. The (422) reflection was used.

Figure 5.6: Distributions of the LD (a), TD (b) and ND (b) strain in the machined test-piece of 7 mm thickness.

Strain maps were converted into stress maps using the reasonable assumption that the measurement directions were aligned with the principal stress axes, and Hooke’s Law:

\[
\sigma_i = \frac{E}{1 + \nu} \varepsilon_i + \frac{\nu \cdot E}{(1 + \nu)(1 - 2\nu)} (\varepsilon_{LD} + \varepsilon_{TD} + \varepsilon_{ND}),
\]  

(5.2)
where \( i \) stands for the LD, TD or ND. The elastic modulus, \( E \), was taken as 71GPa and Poisson’s ratio, \( v \), as 0.35. All three stress components obtained for the machined test-piece are presented in Fig. 5.7, while longitudinal stress maps for both conditions of the weld are compared in Fig. 5.8. The typical error in individual stress values, calculated using uncertainties of the peak positions for both the test-piece and the reference specimen, is about 10 MPa. The quality of the data used for the mapping can be assessed by examining Figs 5.9 and 5.10 where strain and stress distributions for the midline are given.

![Figure 5.7](image_url)

Figure 5.7: The LD (a), TD (b) and ND (c) residual stresses in the MIG welded plate after its machining to a thickness of 7 mm.

![Figure 5.8](image_url)

Figure 5.8: Longitudinal stress distributions for similar areas in the 7150 MIG weld before (a) and after (b) machining.
Figure 5.9: Midline strain corrected for the correspondent reference data (a) and stress (b) distribution for the original 7150 MIG weld. Here and in Fig. 5.10, the HAZs start at about 2 mm and end at about 25 mm from the weld centre-line.

Figure 5.10: Midline strain corrected for the correspondent reference data (a) and stress (b) distributions for the machined 7150 MIG weld.

The longitudinal stress distributions are characterized by the presence of distinct maxima in the HAZs close to the double-V shaped FZ. This can be seen by comparing the stress and integral intensity distributions shown in Fig. 5.11. As it was shown by measuring hardness (see Fig. 3.7 a), parent plate values were reached at approx. ± 25 mm from the weld centre-line, thus given us the outer boundaries of the HAZs. Using these hardness data and the reported yield strength for the parent material [Lin et al. 2003], the minimum yield strength estimate for the HAZ results in about 365 MPa, which is approx.
30% higher than the maximal tensile stress in the 12.6 mm thick specimen, found, however, at another location within the HAZ (compare Fig. 3.7a and Fig. 5.9b). This is likely to be an intended effect of the heat treatment applied to the specimen after the welding (see also Sections 3.1 and 6.5 for further details and explanation).

Figure 5.11: Maps of the LD stress (a) and amplitude of the (111) peak, measured in the TD, (b) obtained for the original weld using spallation-source neutrons.

Two maps of the FWHM values obtained in the synchrotron X-ray experiment are shown in Fig. 5.12. The average and maximal uncertainties, found by performing statistics on the FWHM standard errors for all the relevant peak fits, were approximately 5 and 8 millidegrees respectively. The peak width for a powder standard measured in similar conditions was found to be about 7 millidegrees. When making a qualitative estimate of the welding induced plastic deformation, the data for the reference specimen (Fig.5.12a) have obvious advantage over those for the test-piece: the former are mostly affected by variation in microstrain, while steep macrostrain gradients present in the test-piece must be responsible for the appearance of additional maxima (see Fig. 5.12b), thus giving one a false impression about the microstrain distribution.
5.2 Influence of welding speed on residual stresses associated with friction stir welding

This experiment was conducted using a part of the D1A diffractometer dedicated to residual stress measurements. 1 mm wide slits were used in order to obtain higher spatial resolution for the measurements in rather thin plates (6.3 mm). However, the large divergence of the incident beam led to the following approximate dimensions of the gauge volume: 2x2x2 mm$^3$ for the LD-α measurements and 2x2x10 mm$^3$ for the TD and ND measurements, and so are comparable to the spatial resolution of other experiments in this thesis. Single peak measurements were carried out with a step of 1 mm along the midline in each test-piece. The LD-α measurements in the test-piece B had to be performed twice in order to check their reproducibility, as an uncertainty in the through-thickness translation mechanism had been detected when analyzing peak intensity variation throughout the parent plates, known from its preliminary assessment (see Section 3.4.2). The experimental set-up used for the LD-α measurements is shown in Fig. 5.13. Note the reference specimen that was firmly attached to and accurately aligned with the test-piece to achieve conditions identical to those for the bulk measurements.
Figure 5.13: Experimental set-up for the LD-α measurements: 1) slit system, 2) test-piece, 3) reference specimen, 4) diffracted beam collimator 5) test-piece holder 6) x-y-z translation table.

Distributions of reference values obtained for the weld B using the same (hkl) peak at two different orientations of the “comb” specimen are shown in Fig. 5.14. In this and subsequent figures, AS indicates the advancing side of the welds.

Figure 5.14: The lattice parameter variations obtained at two orientations of the reference specimen, equivalent to the LD-α and TD measurement directions in the weld B. The measurements were performed using the (111) peak.
Longitudinal strains were calculated from the strains measured in LD-α and TD using the following relation:

\[ \varepsilon_{LD} = \left( \varepsilon_{LD-\alpha} - \varepsilon_{TD} \sin^2 \alpha \right) / \cos^2 \alpha \]  

(5.3)

Even at \( \alpha = 19.5^\circ \), the difference between \( \varepsilon_{LD} \) and \( \varepsilon_{LD-\alpha} \) can be appreciable, about 15% if uniaxial stress state is anticipated.

The LD and ND stress distributions are presented in Fig. 5.15. A significant increase in the LD stress (by up to 100 MPa) with welding speed is observed. There is a good correlation between the stress maxima and heat affected zones as can be seen by comparing the LD stress distribution with the macrograph of the cross-section of the weld B. For sake of clarity the transverse stresses are plotted in a separate graph (see Fig. 5.16).

![Figure 5.15: Longitudinal and normal stress distributions.](image)

The synchrotron X-ray measurements were also performed on BM16 at an interval of 1.5 mm along the through-thickness midline in the reference specimen for the weld B using the (422) peak. The peak width profile obtained from this is shown in Fig. 5.17.
The bulk stress data were supplemented by laboratory X-ray stress surface measurements performed on one side of the welded plates, unaffected by the machining. This side was in direct contact with the supporting plate during the FS welding (the underside in what follows). The longitudinal stress distributions obtained with the XStress3000 are presented in Fig. 5.18.
5.3 Residual stresses generated in a 12.6 mm thick plate after friction stir welding

5.3.1 Measurements of the “as-received” test-piece on ENGIN at ISIS

A limited 2-day experiment was conducted using the TOF technique on ENGIN to determine the residual stress for the mid-line of the 12.6 mm thick FS weld. The transverse and through-thickness strains were measured using a 10×2×1.7 mm³ gauge volume whilst the longitudinal strain was measured using a 2×2×1.7 mm³ gauge volume. Based on the results from the relatively fast measurement in the TD, which gave close to a symmetrical lattice parameter distribution, it was decided to perform other measurements mostly on the retreating side of the weld. The variation in the lattice parameter of the elastically unstrained material was assessed from measurements of 2.7×2.7×12.6 mm³ pieces at orientations corresponding to the strain measurements in the LD and ND (see photograph shown in Fig. 3.4). Because of the small gauge volume used for the a₀-measurements, 1.5×2×1.7 mm³, only 7 pieces were measured and other values were subsequently obtained by linear interpolation. Although the variation in reference lattice parameter for the TD could be different from those obtained for the LD and ND (see an example in the previous
Section), the corresponding variation for LD was used to calculate both the TD and LD strains. The residual strain distributions are shown in Fig. 5.19.

![Figure 5.19: $d_{ref}$ corrected residual strain distributions in the 12.6mm thick FS weld.](image)

The effect of possible assumptions about the reference lattice parameters, $a_{ref}$, for the TD can be seen in Fig. 5.20, where the longitudinal stress distributions are presented. The results of calculations with a constant reference lattice parameter are also added for comparison. In the latter case, $a_{ref}$ was taken as the average of the lattice parameters for the reference piece of the parent material measured at different orientations. The corresponding value, $0.40508 \pm 0.00001$ nm, was in good agreement with the result reported in [Pang et al. 1998] for the same alloy.

TOF profiles of high statistical accuracy were obtained for both the test-piece and the reference specimen. This allows data for individual peaks to be obtained. These were used to investigate any possible effect of plastic anisotropy on macrostrain determination when using different approaches to $a_{ref}$ measurement. Longitudinal strain was calculated for positions in the PM, HAZ and nugget from individual reflections. Their transverse co-ordinates are given in the legend for the corresponding spectra shown in Fig. 5.21, where the analysed $(hkl)$ peaks are also indicated. Figure 5.22 shows the results plotted against the anisotropy factor, $A_{hkl}$ (see Equ. 1.4).
Figure 5.20: Longitudinal stress distributions obtained with different assumptions about variation in \( a_{\text{ref}} \) for TD, which was not measured, and with the constant reference value (0.40508 nm).

Figure 5.21: TOF spectra obtained from different zones of the FS weld. The measurements were made in the LD.
Figure 5.22: Longitudinal strain calculated for different measurement locations using the strongest (hkl) peaks: with single $a_{\text{ref}}$ (a), obtained by fitting the whole spectrum, or individual values of $a_{\text{ref}}$ obtained from the positions of the same (hkl) peaks (b). Stars in (b) represent the longitudinal strains shown in Fig. 5.19.

Similar calculations were made for several measurement places in the HAZ of the MIG weld which was characterized by high tensile longitudinal stress. The results for one of them (at -4.5 mm from the weld centre-line) are shown in Fig. 5.23

Figure 5.23: Longitudinal strain calculated using the strongest (hkl) peaks: with $a_{\text{ref}}$ obtained by fitting the same (hkl) peak (a) and the whole TOF spectrum (b). The strain value represented by star (c) was obtained using the lattice parameters determined by the whole spectra. The near-zero strain variation for Al powder (d) was calculated in analogy to (b) except that (220) was used instead of (422).
The above analysis was difficult to make for the other measurement directions, TD and ND, as the corresponding spectra contain fewer strong peaks because of the texture and the form of the Lorentz factor for the TOF technique [Buras and Gerward 1975] (see also Chapter 6).

5.3.2 Residual stress mapping in the friction stir weld on G5.2 at the LLB

Bearing in mind the expected asymmetry of the deformation conditions during the FS welding, through-thickness mapping of residual strains was performed using the same test-piece, except that it was shortened by 58 mm from both sides in the TD to fit on the G5.2 spectrometer (see Section 3.1). The experimental strategy enabling efficient measurements in the highly textured material was again deployed. However, as described in Section 3.4, two separate experiments had to be conducted in order to find a full stress tensor for each measurement location, as wavelength change procedure requires complete realignment of the G5.2 instrument. In addition, a single measurement with G5.2 takes much longer than that with D1A, so it was also impractical to map reference values. Shortage of these data for all measurement directions could lead to systematic errors in the stress determination similar to those shown in the previous section, that is up to ±15 MPa. Nevertheless, all strain calculations were based on the map of reference d-spacings obtained with laboratory X-rays for the LD direction using CuKα radiation and the (422) peak. This map, shown in Fig. 5.24, again in terms of pseudo-strain, was computed using the average d-spacing for the whole measurement area. It should be noted that in spite of the huge difference in the corresponding gauge volumes, the X-ray data obtained for the midline was in good agreement with the corresponding results of the neutron TOF experiment described above. For strain calculations, absolute lattice parameters for the reference were biased by -0.00015 nm, which was obtained by comparing the data with a known Al powder.
Figure 5.24: Map of reference d-spacings ($d_\perp$), in terms of pseudo-strain, for the 12.6 mm thick FS weld measured using laboratory X-rays at orientations corresponding to the LD bulk measurements. The positive through-thickness co-ordinate refers to the topside of the weld.

The layout of the bulk measurement positions is shown in Fig. 5.25. Although this grid is noticeably shifted towards the topside of the cross-sectional area, the protruded part of the gauge volume never exceeded more than 2% when taking measurements near the shoulder side.

Figure 5.25: Layout of measurement locations within transverse section situated in the middle of the test-piece. Horizontal dashed lines represent the surface positions of the welded plates.

The longitudinal stress map computed using the same approach as for the thin FS welds (see Section 5.2) is shown in Fig. 5.26 together with the (111) peak intensity pattern for the
measurement area. The position of this area relative to the test-piece’s surfaces can be easily seen, as the distance between horizontal axes of each map is set here to 12.6 mm, i.e. the plate’s thickness. The stress distributions for individual measurement lines are presented in Fig. 5.27.

Surface stress measurements on the underside of the test-piece were made twice using both CuK and CrK radiations, using the Bruker and Stresstech diffractometers, respectively. The shoulder side scan was performed just once, using CrK radiation. The results of these measurements are shown in Fig. 5.28 and 5.29, respectively. Note that Young’s modulus for the (311) planes used to convert slopes of the $\sin^2\psi$ dependences into stresses was taken as 69.2 GPa [Hauk 1997].

![Figure 5.26](image)

Figure 5.26: Maps of normalized intensity (N.I.) of the (111) peak measured in the LD-\( \alpha \) direction (a) and the resultant longitudinal stress (b).
Figure 5.27: Longitudinal stress distributions used to produce the map shown in Fig. 5.26. Numbers indicate the corresponding distances (mm) from the midline.

Figure 5.28: The LD stress distributions for the underside of the FS weld.
Figure 5.29: The LD stress distribution for the shoulder side of the FS weld
5.4 Full stress tensor determination in an AA7150 alloy plate (VPPA welded) using synchrotron X-ray diffraction

Triaxial strain scanning was performed on a 7 mm thick specimen of the 7150 VPPA weld, prepared as described in Section 3.1. This experiment was conducted using 45 keV X-rays to demonstrate that the use of the specific advantageous (hkl) peaks that occur in this highly textured alloy plate permits fast deep strain measurements in reflection as well as in transmission. The reflection measurements are made viable by the high intensity of (hh0) peaks which are enhanced by the presence of the strong Brass texture component as shown in Fig. 5.30. It has been found from the EBSD measurements (see Sections 3.4.2) and subsequently confirmed in neutron strain scanning experiments (see e.g. Fig. 5.22) that this component strengthens with depth. The (422) and (222) reflections were used to measure the longitudinal and transverse strains respectively. Schematics of the corresponding experimental set-ups and gauge dimensions are given in Fig. 5.31 a and b.

![Figure 5.30: Through-thickness intensity variation of the (440) peak measured in reflection mode. Data represented by the bold line were obtained from neutron diffraction experiments.](image)

Although the measurements were made along several lines distributed over the cross-sectional area perpendicular to the weld seam, here I will concentrate only on the results...
obtained for the through-thickness midline. Due to shortage of beam time the ND strains were not measured from the midline directly. Instead, the midline distribution was found as average of those obtained for the positions 2.5 mm below the plate’s surfaces. As can be seen from Fig. 5.32, where the relevant data are presented, this averaging is unlikely to affect accuracy of the subsequent strain/stress analysis. Data for the FZ are absent because the Brass texture component discontinues within this region, which approximately covers ±4 mm from the weld centre-line.

Figure 5.31: Details of synchrotron strain measurements on the VPPA welded plate performed in transmission (a) and reflection (b). Relative orientations of the gauge and the reference comb’s cross-sections (exactly to scale) when using the sin^2\psi method (c). Rectangular cross-section of the neutron gauge (2×2 mm^2) is also shown in (c) for comparison.
Three separate distributions of reference d-spacings, obtained for each measurement direction, were used in the strain calculations. The resultant strain distributions are shown in Fig. 5.33. A noticeable scatter in the values of the TD strain in the FZ is attributable to the reduced number of grains in the gauge volume and related difficulties with texture average when using the (222) reflection characterized by a relatively small multiplicity factor (P_{222}=8, whereas P_{422}=24). The LD, TD and ND strains were converted to stresses using formula 5.2. The result of these calculations is shown in Fig. 5.34.

Figure 5.32: Lattice parameter variation obtained from the ND measurements made at depths of 2.5 mm below the specimen’s surfaces. The corresponding neutron diffraction data for the midline (determined by the position of the (220) peak on TOF spectra) were biased by + 0.0005Å (i.e. equivalent to about 100 µstrain).

Following the approach described in [Hauk & Nikolin 1988], the sin⁻²ψ method was also utilized to monitor the variation in deviatoric stress component (σ_{LD}−σ_{ND}) across the weld in both the test-piece and a comb-like reference specimen. As can be seen from Fig. 5.31c, a relatively small portion of the gauge is contributed to by the same volume of the material at different ψ angles. However, both the ND and LD strain through-thickness strain
gradients are found to be small (see e.g. Fig. 5.35), so the error introduced into the analysis by this is small as well. An example of linear fits to the $\sin^2\psi$ data obtained is shown in Fig. 5.36.

Figure 5.33: Through-thickness midline strain distributions in the VPPA welded plate

Figure 5.34: Through-thickness midline stress distributions in the VPPA welded plate. The solid line with error bars is the “near-zero” variation of the corrected ND stress (see Chapter 6 for the details).
As already noted (see Section 1.4), the calculation of the deviatoric component (when \(\sigma_{33}=\sigma_{ND}\neq 0\)) does not require knowledge of the exact stress-free lattice parameter. Here, the following relation has been used for the calculations:

\[
\sigma_{LD} - \sigma_{ND} = \frac{1}{1/2s_{2(hkl)}} \frac{1}{a_0^{approx}} \frac{\partial a_0}{\partial \sin^2 \psi}
\]  

(5.3)

where the elastic constant, \(s_{2(hkl)} = 38.14 \times 10^{-6} \text{MPa}^{-1}\), is the same for the (422) and (440) planes, as the corresponding [hkl] directions in cubic materials are characterized by the same elastic anisotropy factor, \(A_{hkl}\). The results for each measurement position in one of the welded plates is shown in Fig. 5.37a and can be compared with those obtained from the triaxial stress data presented above (see Fig. 5.37c). Figure 5.37b gives the corresponding data for the reference specimen. In addition, the variation in \(\sigma_{ND}\) (see Fig. 5.37d) was assessed using the surface stress measurements made on the diamond polished ND-TD acquisition surface of the reference specimen using laboratory X-rays (CuK radiation) assuming that the \(\sigma_{LD}=0\) condition holds.

Figure 5.35: Longitudinal strain distributions along three lines of the measurement cross-sectional area (a). Error bars are given only for the through-thickness midline, as their size is much smaller than the symbols used. A 3D map of the LD strain is given in (b).
Figure 5.36: Typical $a$-vs.-$\sin^2 \psi$ dependencies obtained for the test-piece and the reference specimen. The lattice parameter, $a$, was calculated using the d-spacing defined by the positions of the (422) and (440) peaks.

As can be seen from Fig. 5.37a, non-linearity of the $a$-vs-$\sin^2 \psi$ dependencies, reflected in the stress error, is noticeably increased in the regions located at distances of more than 25 mm from the weld centre-line. This correlates well with the FWHM behaviour (Fig. 5.38).

Figure 5.37: Midline stress distributions for the weld (a, b) and reference comb (c, d) determined by the $\sin^2 \psi$ method (a, c, d) and from triaxial stress measurements (b).
Figure 5.38: The (422) peak width distribution
6 DISCUSSION

In the final chapter of this work possible systematic errors associated with macrostrain data collection and conversion of strains into stresses will be investigated. In this connection special attention will be given to the TOF data obtained. Advantages of synchrotron X-rays in terms of the stress state verification for the reference specimens will also be considered. The stress distributions presented in Chapter 5 will be related to the known microstructural changes occurring during welding. Conclusions will be drawn regarding the role of welding parameters and post welding treatments on the properties of the welded structures.

6.1 Applicability of the crystallite-group method to residual strain mapping and erroneousness of the stress calculations

This work deals with 7XXX alloy rolled plates. There are a number of reasons why the particular (hkl) reflections and orientations used were beneficial for macrostrain measurement in this highly textured material. In general, a gauge volume comprises different groups of grains characterized by nearly the same orientation, g (texture components). If the gauge dimensions are chosen correctly, the strain \( \varepsilon_k^g \) is constant within a \( k \)th group [Bunge 1999]. Strains for all \( N \) groups must be known in order to calculate the macrostrain for a given direction within the measured object:

\[
\varepsilon = \sum_{k=1}^{N} \varepsilon_k^g \cdot w_k , \quad (6.1)
\]

where \( w_k \) describes the volume fraction of the \( k \)th texture component. The above formula is a somewhat simplified representation of the macrostrain calculation for a polycrystalline material, which will include the orientation distribution function (ODF), \( f(g) \), defined by the following relation:

\[
\frac{dV}{V} = f(g) dg , \quad (6.2)
\]
where \( \frac{dV}{V} \) is the volume fraction of grains of a specified phase that have an orientation lying within the interval \( dg \) of the specified orientation \( g \), often represented by coordinates \( (\varphi_1, \phi, \varphi_2) \) of Euler space.

As may be inferred from the results of the texture measurements performed on the 7050 and 7150 plates (see Sections 3.4, 3.5), the Brass, <211> (110), texture is the strongest component within a wide range of through-thickness positions in the measurement cross-sections. This is valid for all zones except for the nugget in the FS welds or the fusion zone in both the MIG and VPPA welds (see e.g. Figs 5.11b and 5.26a). Moreover, it is not feasible to make allowance for any change in the volume fraction of other texture components for each position within the highly textured zones of the measurement cross-section. That is why those components were simply ignored, similarly to that assumed in the so-called crystallite-group method [Hauk 1997]. When the (4h 2h 2h), (h h h) and (2h 2h 0) reflections are used to measure the LD, TD and ND strains respectively, a large number of the same grains are sampled (note, however, Fig. 3.8d suggesting the rotation of the elongated grains about the RD). In addition, there will not be many measurement \((hkl)\) planes belonging to the other texture components known for the 7XXX alloys [see e.g. Dutkiewicz & Bonarsi 1997, Chang et al. 1998] that can contribute to the LD, TD or ND measurements. Moreover, it can be shown that Hooke's Law for isotropic solids (formula 5.2) and the constants \((E, v)\) used are suitable for the stress calculations when comparing their results, \(\sigma^{ko}_{LD}\), with those obtained using a generalized Hooke's law for an aluminium plate with the (011) [2 1 1] ideal orientation, \(\sigma_{LD}\). (The corresponding matrix and its coefficients used to calculate \(\sigma_{LD}\) and other stress components are given in Appendix A.)

In order to demonstrate this, Fig. 6.1 compares \(\sigma_{LD}\) with \(\sigma^{ko}_{LD}\) in terms of a possible systematic error, \((\sigma_{LD} - \sigma^{ko}_{LD}) / \sigma^{ko}_{LD} \times 100\%\), plotted against the following quantity:
\[ \Delta = \sqrt{\frac{\sigma_{TD}^2 + \sigma_{ND}^2}{2 \times \sigma_{LD}}} \]  

which may be described as the departure from the uniaxial stress condition:

\[
\begin{pmatrix}
\sigma_{LD}^0 & 0 & 0 \\
0 & 0 & 0 \\
0 & 0 & 0
\end{pmatrix}
\]  

(6.4)

derived from the corresponding strain matrix:

\[
\begin{pmatrix}
\varepsilon_{LD}^0 & 0 & 0 \\
0 & \varepsilon_{TD}^0 & 0 \\
0 & 0 & \varepsilon_{ND}^0
\end{pmatrix}
\]  

(6.5)

The shadowed area in Fig. 6.1 covers nearly all the most important situations (e.g. high tensile LD stresses in the HAZ) that were observed in the present work. As can be seen from the figure, the possible error associated with the use of formula 5.2 is rather small. In terms of absolute values, the difference \( \sigma - \sigma^{\text{iso}} \) for other stress components does not exceed the stress uncertainties associated with the determination of d-spacings and lattice parameters.

![Figure 6.1: Systematic error associated with the use of formula 5.2](image)
6.2 Reference measurements as a means of intrinsic separation of macrostrains from microstrains

To the extent discussed above, the calculated stresses are correct as long as macrostrains are separated from microstrains (see also Section 1.6). There are results obtained in this work suggesting that these strains cannot be determined accurately enough without using data for the reference specimens obtained in the conditions identical to those of the bulk measurements. This is important because the reference measurements, \( a_{\text{ref}} \), are generally affected by the presence of microstrain that can be expressed as follows:

\[
a_{\text{ref}}(hkl, \overline{m}) = a_0(c) + \Delta_p(hkl, \overline{m})
\]  

(6.6)

where \( a_0 \) is determined by the content of alloying elements in Al-based solid solution, \( c \), whilst \( \Delta_p(hkl, \overline{m}) \) is a \((hkl)\) and measurement orientation, \( \overline{m} \), dependent contribution caused by the plastic deformation. The effect of the measurement orientation is well illustrated by the results obtained with the same \((hkl)\) peak on the reference specimens for thin FS welds. As exemplified by the reference data distributions Fig. 5.14, this effect is particularly pronounced in the parent material.

Then the bulk measurement, \( a \), will be determined by \( a_{\text{ref}} \) and \((hkl)\) and the \( \overline{m} \) dependent contribution from the elastic strain, \( \delta_e(hkl, \overline{m}) \):

\[
a(hkl, \overline{m}) = a_{\text{ref}}(hkl, \overline{m}) + \delta_e(hkl, \overline{m})
\]  

(6.7)

Note that \( a \) and \( a_{\text{ref}} \) are meant to be the calculated lattice parameters from the corresponding \( d_{hkl} \)-spacings when single \((hkl)\) peaks are measured. Alternatively, these parameters are obtained by full-profile refinement, the result of which is determined by the positions and intensities of all detectable peaks (but see below), and the \((hkl)\) dependence in (6.7) is replaced with the corresponding range of TOF. Thus, in general, the strain \( \varepsilon \) can be calculated as follows:

\[
\varepsilon = \frac{a(h_1k_1l_1, \overline{m}_1) - a_{\text{ref}}(h_2k_2l_2, \overline{m}_2)}{a_{\text{ref}}(h_2k_2l_2, \overline{m}_2)}
\]  

(6.8)
An inspection of Equ. (6.6), (6.7) and (6.8) shows that $\varepsilon$ will be close to the elastic strain if the bulk and reference measurements are performed in identical conditions or $\Delta p$ is negligibly small. Often, there is no prior knowledge about the latter, although the use of the TOF technique may provide some evidence [see also Korsunsky et al. 2000]. Figures 5.22a and 5.23b exhibit a large variety in values of longitudinal strain that can be obtained when using a single $a_{ref}$ (taken in these cases as the result of the full-profile refinement). The scatter in the strain data is substantially reduced when strains are calculated with reference data for individual (hkl) peaks (see Fig. 5.22b and Fig. 5.23a). The results of the above analysis would have been of greater value had the reference measurements been made on a comb specimen cut exactly from the bulk measurement cross-sectional area. Unfortunately, that was not possible from a practical point of view. It is worth noting, however, that both the reference and the bulk measurements were taken from the positions where steady-state conditions were achieved, so that the corresponding weld heat inputs were very nearly equal at those positions.

6.3 The TOF data obtained and their use in the stress calculations

As far as the triaxial strain mapping of Al alloy welds is concerned, the neutron TOF technique has proved to be relatively time consuming (see e.g. Fig. 2.10), particularly for the LD measurements using the smallest gauge volume. In addition, for two measurement directions in a highly textured plate, TD and ND, there would be only little extra information when compared with that obtained from corresponding single peak experiments (e.g. those performed at the ESRF, LLB or ILL) using the (hhh) and (hh0) peaks respectively, as the refined value of the lattice parameter is mostly determined by the position of the most intense peak on a TOF pattern (see Fig. 6.2), which is (111) for the TD and (220) for the ND measurement direction (see e.g. spectra in Fig. 5.3). This agrees well with the results of the texture examination (see Chapter 3).
Figure 6.2: Fitted lattice parameter distributions obtained for the textured zones in the MIG weld in the TD (a) and ND (b). Measurements were made on ENGIN using the right bank of detectors in separate experiments (see Figs 5.1 a and c). Examples of the corresponding spectra were shown in Fig. 5.3.

By contrast, the TOF data for the LD strain measurements are worth analysing further. The (200) and (111) peaks are still the strongest ones (see e.g. the corresponding spectra in Fig. 5.21) despite the fact that the <211> direction is coincident with the RD for the bulk of the grains in the parent material and HAZ (see e.g. Fig. 3.13a in Section 3.5). This effect mostly arises from the form of the Lorentz correction for TOF data, $L = d^4 \sin \theta$ [Buras & Gerward 1975], which strongly enhances the scattered intensity for the large-$d$-spacing reflections [McCusker et al. 1999]. As the (211) reflection is forbidden by the structure-factor for the FCC structure, the Brass texture component is represented by the (422) peak.

For example, an approximate ratio between the (200) and (422) peak amplitudes for Al powder can be estimated as:

$$\left( \frac{d_{200}}{d_{422}} \right)^4 \cdot \frac{P_{200}}{P_{422}} \cdot \exp \left[ -B \cdot \left( \frac{1}{2d_{200}^2} - \frac{1}{2d_{422}^2} \right) \right] \sim 15$$

(6.9)

where $P_{hkl}$ is multiplicity factor for a given reflection and $B$ is a quantity used in the Debye-Waller factor describing the effect of the temperature dependent lattice vibrations.
on the intensity of diffraction peaks [Sears & Shelley 1991]. For a highly textured material the above ratio will be substantially reduced. However, for 7XXX series alloys in the T651, T7451 and other overaged conditions, the (200) peak is also enhanced by the presence of an additional texture component, Cube (note the corresponding MUD value for RD in Fig. 3.13b), so that the (200) peak is still much stronger than (422). Figure 6.3 shows typical data obtained with these peaks and the whole spectra for the MIG weld.

Figure 6.3: Fitted lattice parameter distributions obtained for the midline across the MIG weld before its mechanical treatment. Full spectra and individual peaks were fitted using GSAS.

The practical outcome of the above consideration for measurements where the (200) peak dominates the spectrum is that the tensile longitudinal stress could be overestimated if average elastic constants are used in the calculations. Fortunately, Al possesses little elastic anisotropy and so the error introduced will be relatively small (less than 5%). It should be noted, however, that the above analysis exposes one serious misconception which may be cultivated amongst the engineering community using the neutron TOF technique for the measurements of macro residual stresses: as spectra contain many (hkl) peaks, average or mechanical elastic constants are used when converting strains into stresses. In fact, even for a material with relatively small texture, the position of the first peak may
predominately influence the refined value of the lattice parameter (e.g. for RD measurements performed on a Cu-based plate having the $\langle 2\bar{1} \bar{1} \rangle [111]$ texture component). Once again, attention can be drawn to the fact that there is a significant difference between absolute values of lattice parameter calculated from $d_{200}$ and $d_{422}$ (see Fig 6.3), which cannot be explained by elastic anisotropy. As was emphasized above, it is important to perform the corresponding reference measurements under identical conditions in order to obtain accurate macrostrain. Based on this discussion, one is led to conclude that measurements allowing for intergranular effects are of much higher practical importance for the strain/stress analysis in an Al-based component than knowledge of the exact elastic constants.

6.4 The use of synchrotron X-rays for assessment of the macrostress condition in the reference specimens and possible corrections of the stress tensor

As demonstrated by the measurements made on the VPPA weld, the main advantage of the synchrotron strain mapping technique over neutron strain mapping is the quality of the data and speed at which they are collected. As can be seen from Fig. 5.31c, where the LD-ND cross-section of the “comb” specimen is given, the synchrotron diamond-shaped sampling volume is usually immersed in each tooth during the reference measurements and there is no guarantee that the macrostress in the tooth is fully relieved. It should be noted, however, that even with the larger neutron gauge the macrostress is not averaged to zero either (see the corresponding gauge in Fig. 5.31c), unless small cubes are used for the measurements [Krawitz & Winholtz 1994; Edwards et al. 2001], which are difficult to handle in practice. Combined with the results of laboratory X-ray measurements and the bulk measurements, synchrotron diffraction allows application of the $\sin^2\psi$ method to many measurement positions in the reference specimen which is a much more suitable technique for this assessment of the reference data. As can be inferred from Fig. 5.37 c and d, the remaining stress in the reference specimen is relatively small, ranging from -15 to +15 MPa. On the
assumption that the uniaxial approximation for the stress condition \((\sigma_{LD} = \sigma_{TD} = 0)\) in each “tooth” of the reference specimen is valid, the stress tensor found from the triaxial strain measurements can be corrected to eliminate the apparent disagreement between the \(\sigma_{LD} - \sigma_{ND}\) distributions shown in Fig. 5.37 a and b. When applied, this correction results in a biaxial stress condition \((\sigma_{ND} = 0)\) for the region in the HAZ corresponding to the first maximum of the longitudinal stress distribution (see the solid line with error bars in Fig. 5.34). The highest tensile stress values, however, remain unchanged after this correction. The maximum tensile longitudinal stresses, ranging from 140 to 150 MPa, are about 25% of the yield strength of AA7150-T651 alloy, which is 570 MPa at room temperature (Lin et al. 2003). Starting from about ±22 mm, this stress declines rapidly with a distance from the weld centre-line, switching from tension to compression at 32-34 mm. This is near the outer boundaries of the HAZ (±37 mm), which were found by measuring hardness (see Fig. 3.7b). The transverse stress is comparatively small and tensile at all locations. The latter is valid for all the welds studied in this work and probably predetermined by the existing stress condition of the rolled plates (see e.g. Prime & Hill 2002) before welding. At the weld centre-line the value of stress is estimated to be about 40 MPa. This can be done using the reasonable assumption that the behaviour of \(\varepsilon_{ND}\) in the FZ is similar to that of \(\varepsilon_{TD}\) that was subsequently confirmed in the neutron diffraction experiment (compare Fig. 5.32 and 5.33).

6.5 The stress distributions obtained and their relevance to the microstructural characteristics and welding parameters

As can be seen from Fig. 3.6 and Fig. 3.7, in the FZ of the MIG and VPPA welds, hardness is generally lower than in the nugget of the FS welds. For the MIG weld, this is merely because the filler wire used as consumable electrode is a 5XXX alloy, which derives most of its strength from solution hardening [see e.g. Ashby & Jones, 1986]. This mechanism
gives a moderate level of strength, particularly when it is not enhanced by the contribution from work hardening (usually achieved by cold rolling). The solidification process during the VPPA welding, on the other hand, is probably characterized by a rather small cooling speed within a critical range of temperatures below the solidus, so it cannot act a solid solution treatment, which is, by contrast, effectively realized within the dynamically recrystallized zone during the FS welding. A significant hardening effect is usually observed after the post-welding heat treatment (T6) of FS welds. This is designed to recover the lost tensile strength of the weld nugget microstructure [Mahoney et al. 1998]. For the 12.6 mm thick weld and specimen B, the hardness values distinctly exceed those of the parent material (see Fig. 3.6). According to [Jata et al. 2000], who investigated the FS welding effects in the same alloy (7050-T7451), aging to the T6 treatment reduces the dislocation density in the nugget and results in fine precipitates. The hardness and microstructure, however, were found to be not truly representative of the T6 condition, because of the lack of solution heat treatment and quenching [see also Sato & Kokawa 2001]. For the specimens studied in the present work, the tensile strength of the nugget is expected to be even higher than that observed in [Jata et al. 2000]. It should be noted, however, that the post-welding heat treatment (PWHT) recovers a large portion of the tensile yield strength in the nugget at the cost of ductility, which typically drops from 6-15% to 3-4% [Jata 2000 et al., Mahoney et al. 1998]. In the MIG and VPPA welds, PWHT leads to similar hardening effects in the regions of the HAZ bordering the FZ, where the optimal conditions (achieved temperature and the cooling rate) are apparently realized (see Fig. 3.7). Considerable softening is observed in the middle of the HAZ for all welds. This is due to the coarse precipitates in the HAZ. As shown in [Jata et al. 2000], precipitates in the HAZ of the FS weld are about 5 times coarser than in the parent metal. The actual width of the HAZ and associated loss of hardness are dependent on the heat inputs typical for a given welding process and its parameters.
Let us consider the power \( Q \) and heat input \( H \) for the FS welding process. Assuming that Coulomb friction model is applicable to the hot working process, the following equations can be used [Oosterkamp et al. 2000]:

\[
Q = \eta \times F \times R \times \frac{2 \pi n}{60} \tag{6.10}
\]

\[
H = \frac{Q}{V \times t} \tag{6.11}
\]

where \( \eta \) is the coefficient of friction, \( F \) is the download force, \( R \) is the tool shoulder radius, \( n \) is the rotation frequency, \( t \) is the plate’s thickness and \( V \) is the welding travel speed.

As the heat input is inversely related to the welding travel speed, the specimen B, produced using higher speed (see Section 3.1) is characterised by a much smaller drop in hardness within the HAZ than specimen A (see Fig. 3.6 a and b). There is a certain limit below which hardness does not drop (about 100 Hv). In this case, the longer heating time associated with much lower welding speed widens the HAZ, as represented by the hardness profile for the 12.6 mm thick FS weld in Fig. 3.6 c. The extended HAZ results in a redistribution of residual stresses, with reduced peak stress. The lower minimum in hardness means lower yield strength, which further limits the development of tensile residual stresses. The longitudinal stress does not exceed 90 MPa in the 12.6 mm thick FS weld. In its RS (see Fig. 5.20), the maximum of the corresponding distribution is located near the TMAZ-HAZ boundary, which is about 10 mm from the weld centre-line. Size reduction of the test-piece in TD (see Section 3.1) led to a significant change in this distribution (see the "0.25 mm" plot in Fig. 5.27) with the appearance of a compressive stress state within the nugget region. Being aware of the uncertainty of the reference data discussed in Section 5.3, it is important that this observation was confirmed by the laboratory X-ray measurements (see e.g. Fig. 5.29) and could be expected from the balance of forces acting on the weld’s TD-ND cross-section (see also Section 1.2) after mechanical removal (see Section 3.1) of its significant part (about 38%), which probably was in compression. However, the considerable asymmetry in the stress distribution from the
bulk measurements, logically assigned to the different deformation conditions occurring at
the RS and AS of the weld [Stelmukh & Edwards 2000], is not supported by the surface
measurements. To such an extent, this asymmetry was not detected from the mid-depth
measurements on the thinner plates (see Fig. 5.15) and has not been reported by other
investigators [see e.g. X.-L. Wang et al. 2000]. This might be explained by the specific
shape of the welding tool’s probe used for the specimen preparation, which is confidential
to the manufacturer. In the 210 mm square specimens the maximum tensile longitudinal
stresses are much higher: about 150 MPa and 230 MPa for the specimens A and B
respectively. These values amount to 30% and 47% of the yield strength of 7050-T7451
alloy, which is reported to be 489 MPa at room temperature [Jata et al. 2000]. According
to the same investigators [Jata et al. 2000], who performed tensile tests in the TD using the
FS welded 7050-T7451 (T6) alloy exhibiting a hardness profile similar to that of specimen
A, the yield strength of their specimen was only 291 MPa. Admittedly, specific details of
the stress distributions may be influenced by the PWHT and there is no relevant
information on the intermediate stage (i.e. the “as-welded” condition). This creates
limitations for more credible analysis of other parameters obtained in the diffraction
experiments. For example, the peak width distributions shown in Figs. 5.12a, 5.17 and 5.38
may no longer reflect non-uniformity in plastic deformation generated during welding,
since it is well known [see e.g. Sachs and Van Horn, 1951] that increasing the
concentration of dislocations makes a metal more thermodynamically unstable, and
processes of recovery and recrystallization usually start earlier and take place faster in the
regions most affected by plastic deformation.

Based on the residual stress and hardness results obtained in this work for five
specimens (produced using three different welding processes), one may conclude that
higher heat inputs lead to smaller tensile residual stress. In the FS process, this can be
achieved by reducing the welding speed. However, from a microstructural point of view,
higher welding speed produces FS welds with improved mechanical properties, so long as
defects are not formed [Hashimoto et al. 1999]. In a number of works where mechanical properties and fatigue behaviour were studied [see e.g. North et al. 1999, Karlsson et al. 1999], the specimens were quite small so that the residual stresses are substantially relaxed due to specimen preparation. According to the author’s recent findings, the peak values of the LD stress in a 75mm×78mm×7mm Compact Tension Specimen (CTS) of the MIG weld are reduced to nearly half of the values observed in the 280 mm square specimen. To quantify the influence of the residual stress, mechanical testing results are needed for large welding pieces, similar to those used in the present study. Once the influence of the residual stresses is established, it should be possible to optimize welding parameters and/or find the most suitable welding process. This will be based on a compromise between the microstructure and residual stress, thereby leading to desirable properties of the welded structures. On other hand, a significant reduction of the longitudinal stress around the MIG weld was observed after the specimen’s machining to 7 mm (see Fig. 5.8). The stress redistribution is likely to be responsible for this reduction and caused by the original through-thickness asymmetry of the stress distribution (see Fig. 5.8 a). This effect could be employed for further stress reduction in the FS welds, where marked stress asymmetry is also present (see Fig. 5.26). Should the optimized technology (welding technique plus post-welding treatment) and its energy-consuming parameters prove to be cost-effective, there would be a great potential for a wide application of welding in the aerospace manufacturing industry to replace mechanical fastening.
CONCLUSIONS

1. Neutron and synchrotron X-ray strain scanning techniques have been utilized to determine residual stress fields in plates of high-strength precipitation hardened aluminium alloys joined using Metal Inert Gas (MIG), Variable Polarity Plasma Arc (VPPA) and Friction Stir (FS) welding techniques.

2. Preliminary assessment of different zones of the joints has been conducted in order to develop experimental strategies permitting effective measurements with different types of radiation and experimental set-ups. The latter were also optimized in order to accommodate large test-pieces.

3. It has been shown that the preferred orientation is much sharper in the inner portion of the plates than in their outer portion. These specifics proved to be favourable for measurements using the laboratory X-rays on the original surfaces and bulk measurements using synchrotron X-rays and neutrons.

4. Accurate strain determination requires “elastic stress free” measurements to be made under conditions identical to the bulk measurements. This is of particular importance for the regions exhibiting strong intergranular strain effects.

5. It has been shown that the variation in alloying elements content in Al-based solid solution across the weld is equivalent to about 500 and 1300 microstrain when this variation is determined by dissolution and growth of the precipitates and the use of AA5039 alloy as a filler wire for the MIG welding respectively.

6. The (422), (hhh) and (hh0) are found to be the most suitable reflections for the longitudinal (LD), transverse (TD) and through-thickness (ND) strain measurements respectively. This is predetermined by the texture observed, in which Brass is the main texture component. The (hhh) peaks can also be successfully used for the LD-19.5° measurements in the situations when the (422) peak is not available.
7. Hooke's Law for isotropic solids is found to be suitable for the stress calculations when comparing their results with those obtained using formulas derived for the (011) [211] ideal orientation in the crystallite group method.

8. The neutron Time-of-Flight (TOF) technique and a combination of neutron and synchrotron X-ray diffraction have been used to optimize residual stress mapping in 280 mm square test-piece of a double pass MIG weld before and after mechanical treatment. The intense flux of synchrotron X-rays permitted fast strain measurements in transmission, whilst neutrons were most appropriate for the measurements in reflection.

9. The longitudinal stress distribution in the MIG weld is characterized by the presence of distinct maxima in the heat affected zone (HAZ) close to the double-V shaped fusion zone (FZ). The through-thickness asymmetry of the residual stress field is assumed to be responsible for a significant decrease in the peak values of longitudinal stress after the mechanical treatment: from about 290 MPa in the original 12.6 mm thick specimen to about 220 MPa in the machined 7 mm thick specimen.

10. It has been demonstrated that the use of the reflections listed in conclusion 6 permits fast deep measurements in reflection as well as in transmission with synchrotron X-rays alone, enabling the most accurate determination of the full stress tensor over textured zones of the VPPA weld.

11. The experimental data obtained for the FS welds with neutron diffraction shows that the welding speed has a significant influence on the magnitude and spatial distribution of the residual stresses: the specimen made at lower welding speed exhibits lower residual stress, which is accompanied by a lowering of the minimum hardness and a widening of the HAZ.

12. The low stresses observed in the test-piece of the 12.6 mm thick FS weld were attributed to both the welding parameters used for its fabrication and the relatively small dimensions of the specimen used for the measurements. The through-thickness
asymmetry in the stress distribution obtained can probably be exploited for design of stress-reducing measures, such as the mechanical treatment applied to the MIG weld.
APPENDIX A

Generalized Hooke’s law states that:

$$\sigma_{ij} = C_{ijkl} \cdot \varepsilon_{kl}$$  \hspace{1cm} (1A)

where $C$ is the single-crystal stiffness tensor. Equation 1A can be alternatively expressed in terms of the elastic compliance $S=C^{-1}$:

$$\varepsilon_{ij} = S_{ijkl} \cdot \sigma_{kl}$$  \hspace{1cm} (2A)

For a cubic single crystal the strain-stress relationships are [see e.g. Hosford 1993]:

$$\varepsilon_{ijkl} = \sum_{i=1}^{3} \left[ S_{12} + S_0 (\alpha^2 \alpha_i^2 + \beta^2 \beta_i^2 + \gamma^2 \gamma_i^2) + \frac{1}{2} S_{44} (\alpha \alpha_i + \beta \beta_i + \gamma \gamma_i)^2 \right] \cdot \sigma_i$$  \hspace{1cm} (3A)

where $\alpha, \beta, \gamma$ denote the direction cosines between [hkl] and the cubic axes <100>, whilst $\alpha_i, \beta_i, \gamma_i$ are the direction cosines between the principal stresses $\sigma_i$ and the cubic axes. $S_{11}, S_{12}$ and $S_{44}$ are the single crystal compliances in the notation of Voigt and $S_0 = S_{11} - S_{12} - S_{44}/2$.

Equation 3A was the basis for the ($\psi, \phi$) orientation dependent relations for strain which can be derived [Hauk 1986] for a plate consistent of a single crystal, in which the principal strain/stress axes aligned with the LD, TD and ND parallel to the [211], [111] and [011] directions in the crystal respectively. This model is considered as a limitary idealization of a polycrystalline plate with a strong Brass texture (conventionally referred to as the (011) [211] ideal orientation in Chapter 6). A linear system of 3 equations in 3 unknown orthogonal stresses can be derived from Equ. 3A directly, noting that in this case $\alpha = \alpha_i, \beta = \beta_i$ and $\gamma = \gamma_i$, and solved using the following matrix representation:

$$\begin{bmatrix}
a_{11} & a_{12} & a_{13} \\
a_{21} & a_{22} & a_{23} \\
a_{31} & a_{32} & a_{33}
\end{bmatrix} \begin{bmatrix}
\sigma_{LD} \\
\sigma_{TD} \\
\sigma_{ND}
\end{bmatrix} = \begin{bmatrix}
\varepsilon_{LD} \\
\varepsilon_{TD} \\
\varepsilon_{ND}
\end{bmatrix}$$  \hspace{1cm} (4A)

with coefficients $a_{ij}$:

$$a_{11} = S_{12} + \frac{S_0}{2} + \frac{S_{44}}{2}$$
$$a_{12} = S_{12} + \frac{S_0}{3}$$
$$a_{13} = S_{12} + \frac{S_0}{6}$$

116
\[
\begin{align*}
    a_{21} &= S_{12} + \frac{S_0}{3} \\
    a_{22} &= S_{12} + \frac{S_0}{3} + \frac{S_{44}}{2} \\
    a_{32} &= S_{12} + \frac{S_0}{3} \\
    a_{31} &= S_{12} + \frac{S_0}{6} \\
    a_{33} &= S_{12} + \frac{S_0}{2} + \frac{S_{44}}{2}
\end{align*}
\]

The relevant calculations of Chapter 6 were made using elastic compliances for aluminium given in [Hosford 1993]: \(S_{11} = 15.82 \text{ MPa}^{-1}\), \(S_{12} = -5.73 \text{ MPa}^{-1}\) and \(S_{44} = 35.34 \text{ MPa}^{-1}\).

It is easily seen that for an elastically isotropic material \((S_0=0)\) the above matrix is transformed into the following equations:

\[
\epsilon_i = (S_{11} - S_{12}) \cdot \sigma_i + S_{12} \cdot (\sigma_{LD} + \sigma_{TD} + \sigma_{ND})
\]

which are equivalent to the equations 5.2.
REFERENCES


Crompton, J.S. (2000), Private communication


120


GSharp, Advanced Visual Systems, Waltham, Massachusetts, USA; (www.avs.com)

Harris, I.H. und Withers, P.J. (1995) "Residual strain measurement on ENGIN", PREMIS third-year report (Brite EuRam program contract No. BRE2-CT92-0156), ISIS


Leffers, T. (2003), Private communication

Lin, J. (2002), Private communication


Sachs, G. and Van Horn, K.R. (1951) in “Practical Metallurgy”, American Society for Metals, Metals Park, OH


Van Acker, K; Van Houtte, P.; Aernoudt, E (1994) “Determination of Residual Stresses in Heavily Cold Deformed Steel” in Proc. of 4th International Conference on Residual Stresses, Baltimore, Maryland, USA, SEM, pp.402-409


Webster, P. J. (2003) Private communication


Withers, P.J. and Webster; P.J. (2001) “Neutron and Synchrotron X-ray Strain Scanning”, Strain, 37, pp.19-33