THE INHOMOGENEOUS MICROSTRUCTURE AND DEFORMATION OF SIMILAR AND DISSIMILAR AL-ZN CONTAINING Mg FRICTION STIR WELDS

by

JESSICA HISCOCKS

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Abstract

The magnesium-based aluminum-zinc alloys have excellent stiffness to weight ratios, and may be combined by friction stir welding to expand the possible applications. The high aluminum alloy AZ80 in particular has the advantage of being relatively stiff but still extrudable. However limited friction stir welding research is available for this alloy and extrapolation from the extensive work in aluminum alloys is impractical due differences in precipitation behaviour, and magnesium’s high plastic anisotropy and tendency to form strong textures during friction stir welding.

This work investigates the correlations between local friction stir welded microstructures, textures, residual strains, and the local deformation behaviour based on strain mapping during tensile tests. Covering bead-on-plate and butt configurations, joining of similar and dissimilar materials, and a range of processing conditions, many findings of interest for deformation modelling and industrial applications are presented.

Synchrotron x-ray diffraction study of an entire friction stir weld was used to determine texture, residual strain and dislocation density data from a single experiment. A number of unique findings were made, mainly related to the asymmetric distribution of properties both between sides of the weld and through the depth. Particularly in the case of strain measurements, features not detectable at coarser measurement
spacing or by line scan are presented and compared for multiple processing conditions.

Investigation of the longitudinal material flow during welding showed that even when periodicity in grain size, precipitate distribution, or texture was not observed, periodic changes in texture intensity resulting from compaction of material behind the tool were present, providing evidence that movement of nugget material remained periodic.

Strain localisation and fracture behaviour were found to be completely different between good quality similar and dissimilar friction stir welds. For similar magnesium friction stir welds, higher heat input was shown to improve mechanical performance by reducing the residual strain, while for dissimilar friction stir welds, deformation behaviour was found to be more sensitive to the final material distribution in the friction stir weld nugget. For dissimilar welds, even minor changes to the material flow were shown to have a major impact on the tensile performance.
Acknowledgements

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List of Abbreviations and Symbols

**AS**: Advancing Side - The side of a friction stir weld where the direction of tool rotation and material movement are opposite.

**Å**: Angstrom - A unit of length = $1 \times 10^{-10}$ m.

**AZ**: Aluminum-Zinc - A series of magnesium alloys with varying aluminum and zinc additions.

**ASTM**: American Section of the international association for Testing Materials - An international standards organization.

**BCC**: Body-Centered Cubic - A crystallographic arrangement of atoms whereby atoms are not close packed.

**β**: Beta (magnesium intermetallic phase) - Mg$_{17}$Al$_{12}$

**B**: Texture component in aluminum or magnesium created by shear deformation at elevated temperature.

**BM**: Base Material - The region of the starting material unaffected by the heat or deformation of welding.

**BOP**: Bead On Plate - A weld made on the surface of a single continuous plate of material.

**C**: Single Crystal Stiffness Tensor - A tensor relating the elastic stress to the elastic strain in three dimensions. or **C**: Texture component in aluminum created by shear deformation at elevated temperature.

**CCD**: Charge-Coupled Device - A two-dimensional detector of signals such as X-rays or visible light.

**CRSS**: Critical Resolved Shear Stress - The critical stress at which deformation begins for a given slip system and direction.
\(d\): The spacing between two parallel planes of the crystallographic lattice.

\(d_0\): The spacing between two parallel planes of the crystallographic lattice that are only subject to chemical effects and not residual strain effects.

**DIC**: Digital Image Correlation - A method of measuring strain using digital images taken of the sample at different stages of deformation and computationally correlating them.

**DOE**: Department Of Energy - American government agency funding research.

**DRX**: Dynamic Recrystallisation - Recrystallisation of microstructure occurring concurrently with deformation at an elevated temperature.

**E**: Young’s Modulus - The slope of the elastic portion of a tensile stress-strain curve.


**ED**: Extrusion Direction

**EDS**: Energy-Dispersive X-ray Spectroscopy - A method of compositional analysis frequently available in SEM.

\(\varepsilon\): Strain

**FCC**: Face-Centered Cubic - A crystallographic arrangement of atoms whereby all layers are close packed, and stacked in a pattern that repeats every three layers (ABCABC).

**FEI**: Company name of a manufacturer of SEM equipment

**FSW**: Friction Stir Welded, Weld, or Welding - A joining method using a non-consumable rotating tool with a wider shoulder and narrower pin.

**FWHM**: Full Width at Half Maximum - A measure of peak shape applied to curves fit to diffraction data, and frequently used to quantitatively compare peak broadening
by taking the peak width at half the maximum value.

**GS**: Grain Size

**HAZ**: Heat Affected Zone - The material outside the TMAZ where the material was subjected to elevated temperatures but no significant applied forces.

**HCP**: Hexagonal Close Packed - A crystallographic arrangement of atoms whereby all layers are close packed, and stacked in a pattern that repeats every two layers (ABAB) [1].

**MCT**: Micro-Computed Tomography - An analysis technique using multiple X-ray images taken from slightly different angles to reconstruct a 3D image of the sample density.

**MTEX**: An open-source analysis package for texture data running in *Matlab*.

**ND**: Normal Direction - Upwards through the weld. The shorter dimension of the sheet that is perpendicular to the WD.

**NSERC**: Natural Sciences and Engineering Research Council - Canadian government agency funding research in Natural Sciences and Engineering.

**ODF**: Orientation Distribution Function - A method of representing texture by creating a continuous function from discrete measurements.

**ν**: Poisson’s ratio - The ratio of change in dimension perpendicular and parallel to an applied elastic load.

**Q**: The scattering vector - The direction in which strain is measured during a diffraction measurement.

**RS**: Retreating Side - The side of a friction stir weld where the direction of tool rotation and material movement were the same.

**RPM**: Rotations Per Minute
$S$: Compliance matrix - The inverse of the stiffness tensor $C$.

**SEM**: Scanning Electron Microscope

$\sigma$: Stress

$\theta_b$: The Bragg angle of a specific crystallographic plane.

**TD**: Transverse Direction - The longer dimension of the sheet that is perpendicular to the WD.

**TEM**: Transmission Electron microscope

**TMAZ**: Thermo-Mechanically Affected Zone - The region outside the nugget interfaces where material was affected by heat and deformation.

**UTS**: Ultimate Tensile Stress - The greatest stress undergone by the sample during tensile testing.

**WD**: Welding Direction
Chapter 1

Introduction

The perfect material for automotive applications will be lightweight, ductile, elastically stiff, and easily weldable among other virtues. While this ideal alloy has not yet been developed, the higher content magnesium alloys show many of these desirable features, and are worthy of more extensive development than has been done to date.

Friction stir welding is a solid-state joining technique with several advantages over fusion techniques when used with magnesium alloys. The work presented here is an investigation of the best practices for dissimilar friction stir welding of high to low alloying content magnesium alloys, combinations which can be made with the aim of optimizing ductility and strength across a single welded part while minimizing weight.

Work done included metallography, texture measurements by multiple methods, residual strain measurements, hardness testing, and tensile testing with simultaneous local strain mapping, all done on welds with a range of compositions and processing parameters. A focused attempt was made to map properties in 2D across the weld normal-transverse section in high resolution wherever possible, to produce a deeper understanding than that achievable from linear measurements.

Using the resulting body of data, detailed microstructural and mechanical analyses
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were performed with an emphasis on drawing connections between the property-performance relationships in specific regions of the welds. The results presented here provide detailed information on the underlying mechanical mechanisms that govern deformation of magnesium friction stir welds, and practical information to aid in the systematic development of better dissimilar welds and selection of processing conditions.

1.1 Motivation

To meet the increasingly restrictive emission requirements in the automotive industry, weldable lightweight alloys are essential. In comparison to aluminum alloys, magnesium offers the potential for greater weight reduction in exchange for reduced mechanical properties, making it ideal for weight-critical applications. For example, the magnesium alloy AZ80 offers a weight reduction of about 26 % for parts of the same yield strength as the common aluminum alloy AA6061, in exchange for lower ductility [1, 2] and higher cost [3].

The AZ series alloys are magnesium-based with aluminum and zinc additions. As alloy content increases from 3 % aluminum in AZ31 to 8 or 9 % in AZ80 or AZ90, there is an increase in mechanical strength at the cost of ductility reduction. This alloy series is fairly mature and used in industry; hence it is a good target for development that will expand applications.

Much of the experimental data and conclusions presented in this thesis is based on work in AZ80, which can be both wrought and precipitation hardened to be relatively stronger than the other AZ alloys [4]. In comparison to AZ91 which has the highest aluminum content in this series and is cast, AZ80 may be forged or extruded resulting
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in both greater strength and ductility [5].

Friction stir welding is a solid state joining technique offering multiple advantages for use with materials that are challenging or impossible to weld successfully by fusion methods [6]. The high Al containing Mg alloys (AZ80 and AZ91) are one example of materials unsuitable for fusion welding due to the brittle intermetallic formed along the grain boundaries [7], however friction stir welds of good quality have been successfully made [8, 9].

In comparison to fusion welding techniques, friction stir welding results in a finer microstructure [10], lower residual stress [11], greater energy absorption on impact [12] and generally better mechanical properties [13, 14]. In terms of practical application, friction stir welding is also cheaper and better suited to automation [10] than fusion welding techniques.

By using friction stir welding to join dissimilar alloys such as AZ31 and AZ80, parts may be manufactured that have properties locally tailored to the anticipated loading. Although this broadens the potential applications while maintaining the characteristic light weight advantage, limited information is available on this topic in magnesium alloys.

While there exists a wide body of research into aluminum-based dissimilar welds, much of the research is not applicable to the magnesium-based AZ alloy series. Most fundamentally, this is due to the differences in plastic anisotropy stemming from the different crystallographic nature of aluminum (an FCC or Face-Centered Cubic material) versus magnesium, an HCP (Hexagonal Close-Packed) material. As a result, research in aluminum-based friction stir welds will not address critical behaviours of magnesium friction stir welds such as the tendency to form strong shear textures,
and highly inhomogeneous deformation under transverse loading. Extrapolating from dissimilar aluminum-magnesium friction stir welding research is also difficult due to the formation of other brittle intermetallic phases [15] which are not a concern when joining two dissimilar AZ series alloys. Consequently, deformation and failure in magnesium friction stir welds is substantially different from that in aluminum-based alloys, and more specific studies in the magnesium alloys involved is required.

1.2 Organization of Thesis

The current thesis work is of the folio type, containing four manuscripts preceded by a literature review included as Chapter 2. For an explanation of the terms and concepts discussed in this preamble, please see the literature review or the glossary. Note that in some cases work is duplicated between the literature review of the overall thesis and that of the included manuscripts.

The first published work, *Influence of magnesium AZ80 friction stir weld texture on tensile strain localisation* [8] is included as Chapter 3. The major focus of that publication is correlation of microtextures within and surrounding the weld nugget to the strain localisation and fracture behaviour of similar AZ80 welds made at a range of processing conditions. The tendency for similar friction stir welds to fail at the advancing side of the weld as opposed to the retreating side is shown to be related to the local textures.

The second published work, *Formation mechanisms of periodic longitudinal microstructure and texture patterns in friction stir welded magnesium AZ80* [16] is included as Chapter 4. This publication contains a detailed investigation of the microtexture and microstructure of periodic flow in the nugget of AZ80 friction stir
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welds, and contrasts the shear textures formed with those present in aluminum. The decreased tendency for magnesium-based alloys to form the periodic ‘onion ring’ structure commonly observed in the nugget of aluminum-based friction stir welds is explained, and the local texture changes resulting from periodic material flow are discussed.

The third work *Distribution of residual stresses in 2D across AZ80 friction stir welds at different processing conditions* (publication pending) [17] is included as Chapter 5. This publication contains an extensive residual stress analysis of several AZ80 welds created at varying friction stir welding parameters. The magnitudes and distribution of residual stresses are compared, and correlations to weld thermal distribution, local texture, and tensile behaviour are discussed.

The fourth and final work *Strain localisation and failure of dissimilar magnesium AZ series friction stir welds under transverse load* (publication pending) [18] is included as Chapter 6. The sole publication of this work addressing dissimilar welds, this work focuses on the effect of changes to the material combinations, tool wear, and other processing conditions on the mechanical performance and progression of strain localisation in the weld.

This thesis then concludes in Chapter 7 with a summary of the most notable contributions of the four publications, and several suggestions for further work.

1.3 Contributions

Some general results of the current project included measurements of the changes in weld profile, tensile properties, hardness measurements, and microtextures in similar welds made at a range of processing conditions [8]. Also included in this work are
transverse tensile measurements for friction stir welds made at varying processing conditions, tooling state, location relative to the friction stir weld start, alloying combinations, and layout [18]. These findings are applicable to future researchers for confirmation or comparison of results, and to industrial applications work to give an idea of the process limits.

Notable new results include the first high resolution 2D map of texture measurements spanning an entire magnesium friction stir weld [8] explaining why welds fail preferentially at the advancing side interface. High resolution residual strain mapping showed changes in residual stress with processing condition well account for the changes in transverse tensile properties [17]. Combined with detailed information on the progression of strain localisation and failure in similar and dissimilar welds [18], these results are valuable as input to deformation modelling of friction stir welded joints.

Several results pertinent to the development of friction stir welding methods for industrial application were found. In particular, strain localisation measurements showed that dissimilar friction stir welds will be far more sensitive to the welding parameters involved than similar welds [18]. It was also shown that thermal transients at start of a weld result in a mechanical transient, and in some cases different microstructures as well [18], a finding with strong implications for manufacturing applications.

1.3.1 Contributions to Manuscripts

This information applies to the manuscripts of Chapters 3 through 6. All welding was carried out off-site under the direction and supervision of Gerlich (currently at
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University of Waterloo). Welds were received in the as-welded condition labelled with the welding parameters used. Tool geometry and wear information was also courtesy of Gerlich.

Planning of experiments on the welded and base metal samples was done by Hiscocks with input from Daymond and Diak. Hiscocks carried out all experiments with the exception of initial synchrotron scans which were performed by Cochrane and Skippon as directed by Hiscocks. Later synchrotron scans (such as the calibration comb) were done by Hiscocks. All data analysis was performed by Hiscocks, with the exception of synchrotron experimental geometry calibration (e.g. detector distance and beam center) which was done by L. Balogh. Result interpretation and manuscript writing was done by Hiscocks with contributions from Gerlich, Daymond, and Diak.
Chapter 2

Literature Review

2.1 Magnesium Fundamentals

2.1.1 Assumed Background Knowledge

This work assumes working knowledge of basic material topics including the crystallographic lattice, grains, texture, and basic texture measurement concepts such as pole figures. In all cases, plane and directions in the crystallographic lattice are described using Miller-Bravais notation (see [1] for a brief introduction), and basic knowledge of this system is assumed. Directions for all welded samples are referenced as the ND (Normal Direction), TD (Transverse Direction) and WD (Welding direction) all of which are orthogonal.

2.1.2 Magnesium Unit Cell

Magnesium falls into the hexagonal close-packed (HCP) category of metals, where the unit cell is commonly represented as shown in Figure 2.1a. For pure magnesium at room temperature the length of the unit cell in the [0001] direction is 5.2108 Å as compared to the ⟨1120⟩ axis length of 3.2099 Å [19].
These unit cell dimensions are affected by the substitution of solute atoms, with the addition of aluminum or zinc atoms in dilute quantities causing a reduction in the dimensions of the unit cell axes proportionate to the concentration [19]. This phenomenon can be used to calculate composition based on measurements of the unit cell dimensions as described in Section 2.5.2.

Figure 2.1: Adapted from [20]. Schematic of an HCP unit cell with (a) the [0001] direction, one ⟨11\bar{2}0⟩ axis, and one ⟨\bar{1}00⟩ prismatic axis indicated. (b) Basal and prismatic planes shaded and labelled with the appropriate Miller-Bravais indexes.

Of particular interest to the current work are the (0001) basal plane and the {10\bar{1}0} prismatic plane family shown in Figure 2.1b. These planes are perpendicular, and thus when used in combination for analysis provide a unique representation of the HCP unit cell orientation, an approach used in multiple cases in this work.

2.1.3 Elastic versus Plastic Deformation

Up to the yield stress, materials deform in a completely recoverable fashion as the atomic bonds stretch elastically. Appropriately referred to as ‘elastic deformation’ these strains are completely recovered on removal of the applied stress [1] and may be measured as discussed in Section 2.5.4. At applications of stress greater than the yield,
magnesium begins to also deform plastically by means of slip and mechanical twinning processes, which involve permanent atomic displacements and are not recoverable [1]. These deformation mechanisms are introduced here briefly and form the basis for the deformation and failure processes discussed in Section 2.4.

Elastic deformation of magnesium

For any unit cell or other homogeneously oriented volume, we can describe the relationship between stress and strain within the elastic regime with the single crystal stiffness tensor (represented by $C$). The greater the crystal symmetry, the fewer unique parameters are required to define this tensor. From Figure 2.2a, we can see how the stiffness tensor for an HCP single crystal relates the stress to the strain using five unique parameters while the isotropic single crystal stiffness tensor requires only three [21], shown in Figure 2.2b.

While all HCP materials are elastically isotropic radially about the [0001] axis [22], they are generally not isotropic in all directions. Several authors have proposed measures to quantify this anisotropy based on the elastic stiffness tensor (values for magnesium shown as Figure 2.2c). Tromans [22] proposed using the ratio of the elastic moduli along and perpendicular to the [0001] axis in the unit cell, which for magnesium gives a ratio of 1.117. Tomé [24] proposed quantifying the deviation of the tensor from an isotropic version of the tensor, resulting in values ranging from 1.03 to 1.24 for magnesium, and concluded that this material could be regarded as nearly elastically isotropic. Alternatively, we may use computational methods to calculate the isotropic version of the magnesium elastic tensor (i.e. determine the value for Mg polycrystal with equal texture in all directions) shown as Figure 2.2d which can be
2.1. MAGNESIUM FUNDAMENTALS

\[
\begin{bmatrix}
\sigma_1 \\
\sigma_2 \\
\sigma_3 \\
\sigma_4 \\
\sigma_5 \\
\sigma_6
\end{bmatrix}
= 
\begin{bmatrix}
C_{11} & C_{12} & C_{13} & \cdots & C_{14} & C_{16} \\
C_{21} & C_{22} & C_{23} & \cdots & C_{24} & C_{26} \\
C_{31} & C_{32} & C_{33} & \cdots & C_{34} & C_{36} \\
\vdots & \vdots & \vdots & \ddots & \vdots & \vdots \\
C_{61} & C_{62} & C_{63} & \cdots & C_{64} & C_{66}
\end{bmatrix}
\begin{bmatrix}
\varepsilon_1 \\
\varepsilon_2 \\
\varepsilon_3 \\
\gamma_4 \\
\gamma_5 \\
\gamma_6
\end{bmatrix}
\]

\[
C_{ij} = C_{ji}
\]

\[
C_{66} = \frac{C_{11} - C_{12}}{2}
\]

The elastic stiffness tensor \( C \). Dots represent zero components. a) Adapted from [21]. Equation showing how the single crystal anisotropic HCP elastic stiffness tensor relates the stress and strain of a crystal b) Adapted from [21]. The single crystal isotropic elastic stiffness tensor c) Values from Tromans [22] in MPa. The single crystal anisotropic magnesium elastic stiffness tensor. Note that values are defined in terms of the single crystal axes, with 1 parallel to the [2 ¯1¯10] axis, 2 parallel to the [01¯10] axis and 3 parallel to the [0001] axis. d) The simulated isotropic polycrystal stiffness tensor for magnesium in MPa, computed from c) using MTEX [23] and the Hill method by assuming an even texture in all directions.

seen to be quite similar to the single crystal anisotropic magnesium elastic stiffness tensor shown as Figure 2.2c, with all these approaches indicating magnesium is nearly elastically isotropic.

For a graphical approach, we may invert any stiffness matrix (represented by \( C \)) to calculate the compliance matrix (represented by \( S \)), and then plot the directional
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elastic stiffness ($E_{hkl}$) using the equation

$$\frac{1}{E_{hkl}} = (1 - L^2)^2 S_{11} + L^4 S_{33} + (1 - L^2)L^2 (2S_{13} + S_{44})$$  \hspace{1cm} (2.1)

where $L$ is the cosine of the angle between the direction of interest and the [0001] axis [25]. For an isotropic material the resulting figure would be a sphere. From Figure 2.3 which shows this plot for the values in Figure 2.2c, it can be seen that the elastic stiffness of magnesium is indeed radially symmetric and not significantly anisotropic, explaining the assertion commonly found in literature that pure magnesium may be treated as an elastically isotropic material without significant error [26, 22, 24].

![Figure 2.3: Directional elastic stiffness ($E_{hkl}$) for pure magnesium, calculated from equation 2.1 and values from Figure 2.2c.](image)

Plastic deformation of magnesium

**Magnesium slip** In magnesium at room temperature, the three easiest slip systems to activate are basal, prismatic, and 2nd order pyramidal [27], with the slip planes and directions shown in Figure 2.4.

Only the pyramidal slip system, the most difficult of these three systems to activate [28], allows for slip along the [0001] direction, and so slip perpendicular to
2.1. MAGNESIUM FUNDAMENTALS

Figure 2.4: Redrawn from [20]. Key slip systems in magnesium a) Basal, b) Prismatic, c) 2nd order pyramidal

[0001] happens easily at low stresses while that parallel to [0001] is difficult to activate. Plastic deformation behaviour in the magnesium unit cell is therefore highly anisotropic. Consequently, in any case where a magnesium polycrystal has developed a strong orientation preference (texture) there will be significant changes in macroscopic deformation with changes in the orientation of the applied force.

Magnesium twinning  In addition to slip, another deformation mode frequently seen in magnesium is mechanical twinning, where a portion of a grain reorients by a homogeneous shear transformation [29] in response to an applied stress. The reoriented portion of the grain is called the twin, the portion of the grain remaining in the original orientation is called the parent, and there will exist a specific crystallographic relationship between them. In contrast to slip which will operate with equal ease in either direction, twinning is polarised [30] and directional.

The two most commonly observed twinning orientation relationships in magnesium are extension and contraction twinning [31], called this due to the net increase of the unit cell along the [0001] direction in the first case, and the net contraction along the [0001] direction in the second case [32].
While extension twinning shear results in a net extension along the [0001] direction, it is a minor contributor to the macroscopic deformation of the material. Even if the entire volume of a single crystal twinned, less than 7% elongation in tension would occur [20]. One way in which twinning makes a significant contribution to macroscopic deformation despite the low volume fraction stems from the reorientation of a section of the grain to make previously difficult to activate slip systems more favourable [20]. In addition, the abrupt change in orientation across the twin boundary and change of planes available forms a barrier to slip as discussed in detail by Christian and Mahajan [30].

Due to the six-fold HCP crystal symmetry, there are six potential variants of each type of twin in a magnesium grain. Crystallographically these are identical, but within a real material each variant will have a different orientation relative to the macroscopic sample axes and any applied stresses. A pole figure showing an example parent grain and the six associated extension twin variants is shown as Figure 2.5b.

### 2.1.4 Schmid Factor and Deformation Mode Selection

For each slip and twinning system, there is a critical stress at which plastic deformation begins. This minimum stress required to activate a system when resolved along the slip plane and direction is called the critical resolved shear stress (CRSS). For magnesium, Chapuis and Driver [28] report values of 4 MPa for basal slip, 18 MPa for prismatic, and 40 MPa for pyramidal slip at 25 °C. Correspondingly, at room temperature in magnesium basal slip predominates [20] as it is far easier to activate than prismatic slip, which in turn is somewhat easier to activate than pyramidal slip [28], as mentioned in Section 2.1.3. Similarly, with a reported CRSS of 11 MPa at 25
°C, extension twinning is much more easily activated than contraction twinning (63 MPa at 100 °C)[28].

For a single crystal the fraction of the applied stress that will be resolved along a given slip or twinning system for a macroscopic applied stress may be calculated using the Schmid factor, which depends on the orientation relationship between the unit cell and the applied stress. Theoretically, in cases where the Schmid factor multiplied by the applied stress exceeds the CRSS of a given system, that system will be activated [20] for plastic deformation. Due to the low CRSS of basal slip, this system has been observed to be active even in orientations with Schmid factors as low as 0.035 [33].

The Schmid factor is calculated with the equation $\cos(\phi)\cos(\lambda)$, and has a maximum possible value of 0.5 [1]. In this equation $\phi$ and $\lambda$ represent the angle from the applied stress to the slip plane normal and slip direction respectively [1], as shown graphically in Figure 2.5 at left.

Figure 2.5 at right shows the result of Schmid factor calculation for each of the six possible variants of an extension twin for a magnesium crystal subject to stresses in the TD, presented on a pole figure. Note that for twinning which is polarised, a negative Schmid factor indicates the stress resolved along the twinning direction is negative, and thus extremely unfavourable.
Figure 2.5: (left) Redrawn from [1, pp. 160]. Schematic of Schmid Factor parameters. The red arrow indicates the resolved shear stress along the slip plane and direction resulting from the larger applied stress of the blue arrow. (right) Author’s work. (0001) Pole figure for a single grain showing the Euler angle of the parent (Bunge notation) and the Schmid factor of the six possible extension twin variants for a stress applied in the sample TD. Note that one value is negative.
2.2. THE MAGNESIUM AZ SERIES ALLOYS

2.2 The Magnesium AZ Series Alloys

The magnesium based AZ series alloys use additions of aluminum and zinc. Aluminum is the principal alloying element, and is used in additions of up to 10 % [34] due to the low density and price combined with advantageous effects on strength, corrosion resistance [35], and casting behaviour [2]. Minor zinc additions are used to facilitate age hardening, as zinc decreases the solubility of aluminum in magnesium and thereby encourages precipitate formation [36]. A comparison of the tensile properties for a few alloys of the AZ system produced through various methods is included as Table 2.1.

AZ31 is the most commonly used magnesium alloy sheet and plate material [34], and further aluminum additions increase tensile strength at the cost of ductility. From Table 2.1 extruded AZ31B with a nominal aluminum content of 3 wt% has significantly lower yield and UTS than those of extruded AZ80A, with a nominal aluminum content of 8 wt%. Wrought AZ alloys have superior mechanical properties to cast [37]. Comparing the properties of two similar alloys manufactured with different methods, the properties of Sand Cast AZ81A are inferior to those of Extruded AZ80A, as shown in Table 2.1. While AZ91 is widely used in cast form due to its low density and good mechanical properties [38], it is not generally used in wrought form [5] due to manufacturing issues stemming from the high aluminum content. In contrast AZ80 can be successfully extruded, and shows relatively high strength combined with reasonable ductility.

In comparison to commonly used aluminum alloys such as AA6061-T6, extruded AZ80-F offers a weight reduction of about 26 % for parts of the same tensile yield strength in exchange for a reduction in ductility [1, 2] and higher cost [3].
Table 2.1: Summarised from [2]. Effect of alloy content and manufacturing method on tensile mechanical properties for selected magnesium AZ alloys. Note that the suffix A, B or C after the alloying element percentages indicates a compositional specification, while the suffix -T4 is solution heat treated, -T6 is solution heat treated and age hardened, and -F is as-extruded [5].

<table>
<thead>
<tr>
<th>Alloy and condition</th>
<th>Yield (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation in 50 mm (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand Cast AZ81A-T4</td>
<td>83</td>
<td>275</td>
<td>15</td>
</tr>
<tr>
<td>Sand Cast AZ91C-T6</td>
<td>145</td>
<td>275</td>
<td>6</td>
</tr>
<tr>
<td>Extruded AZ31B-F</td>
<td>200</td>
<td>260</td>
<td>15</td>
</tr>
<tr>
<td>Extruded AZ61A-F</td>
<td>230</td>
<td>310</td>
<td>16</td>
</tr>
<tr>
<td>Extruded AZ80A-F</td>
<td>250</td>
<td>340</td>
<td>11</td>
</tr>
</tbody>
</table>

The dimensional flexibility of the extrusion process combined with the additional possibilities available through joining of material combinations make AZ80 a promising avenue for research, and so the majority of the work of the current project focuses on friction stir welding of extruded AZ80. For the dissimilar welding portion of this project, AZ31-AZ80 and AZ61-AZ80 joints were examined (see Chapter 6).

### 2.2.1 Precipitation

Extruded AZ80 contains the body centered cubic Mg$_{17}$Al$_{12}$ precipitate [39], which is the only precipitate of interest for age hardening in this alloy series. As long as the Al:Zn ratio of the material is greater than 3:1, zinc additions will not cause new compounds to form [35], and the Zn will substitute for some Al atoms in the Mg$_{17}$Al$_{12}$ precipitate [35].

The kinetics of age hardening in this alloy are far slower than in many aluminum age hardenable alloys. For example, AZ91 will reach peak hardness after 10 hours at 200 °C [36], while the common age hardenable aluminum alloy AA6061 reaches
peak hardness after 1 hour at a similar temperature [40]. Similarly, AA6061 will undergo measurable age hardening at room temperature over 30 days [40], while in AZ91 even after 652 days in the temperature range of 20-50 °C the age hardening response was found not to be significant and no evidence of precipitation was found [36]. As a result, precipitate distributions and fractions observed in the AZ series alloys at room temperature may be considered a static situation.

The weight percentage of this precipitate following ageing treatment has been reported to be 10±2 volume % in AZ80 as measured by diffraction [26], and as-extruded material is anticipated to have significantly less. In extruded AZ80 this Mg$_{17}$Al$_{12}$ precipitate is typically visible in bands oriented in the extrusion direction [41, 14], and Yang et al. [9] reported localised regions with a supersaturated Al content of over 20 %.
2.3 Properties of Friction Stir Welds and the Effects of Processing Parameters

2.3.1 Friction Stir Welding Process

Friction stir welding is a solid state process suitable for joining of several alloys that cannot be fusion welded due to the poor qualities of the resulting microstructure [6]. When the magnesium AZ series of alloys used for the current project are fusion welded, the equilibrium compound Mg$_{17}$Al$_{12}$ (β phase) solidifies along the grain boundaries creating brittleness. As the solid state nature of friction stir welding avoids this issue [7], it is a strongly preferable to fusion techniques for this alloy series.

Friction stir welding offers additional advantages including a finer microstructure in the welded area [10], and often improved tensile, bend, and fatigue properties [13] as compared to fusion welding techniques. In terms of processing convenience, no filler metals or shielding gases [42] are required for friction stir welding, and the process is well suited for automation [10]. As compared to linear or rotational friction welding techniques, friction stir welding has the dual advantages of being more independent of part geometry [6] and size [43], making friction stir welding better suited for large part production than other frictional welding methods. Friction stir welding was used by NASA for manufacture of the space shuttle external tank, and the standard deviation of mechanical test results was reported to be lower than for fusion techniques [10], an important consideration for estimating inspection intervals of critical parts.

The most basic friction stir welding tool shape is that of a narrow pin and a wider shoulder. During friction stir welding the frictional heat generated by the non-consumable rotating pin softens the material locally upon contact causing it to become plastic and move with the tool. Insertion of the pin continues until the wider tool
shoulder meets the material, and the rotating tool is then moved along the material joint in the welding direction.

A schematic of the friction stir welding process is shown as Figure 2.6a and includes the directions referenced in the current work. Friction stir welding is inherently an asymmetrical process because the tool is simultaneously rotating and progressing forwards as shown in Figure 2.6b. We define the advancing side (AS) of the weld as that where the pin rotates towards the welding direction and the retreating side (RS) as that where the pin is rotating away from the welding direction in conjunction with the base material. Base material is introduced to the weld at the front of the tool, and moves towards the RS first.

Figure 2.6: (left) Schematic of the friction stir welding process showing the tool rotating counter-clockwise as it progresses forwards. (right) The red helix shows the path of a point on the tool shoulder, demonstrating that the AS and RS are inherently asymmetric. On the AS the tool shoulder and material move in opposite directions, while on the RS they move in the same direction.

For the current work, the friction stir welding configurations of interest are butt welds, where two plates of equal thickness are abutted along one edge which is then welded over, and bead on plate (BOP) welds, where the weld is made along the surface of a single sheet of material. BOP welds are generally done for experimental
purposes to simplify material clamping and fit considerations with the aim of making the process more repeatable.

The friction stir welding work available in the AZ magnesium series alloys is limited, and particularly sparse in the AZ80 alloy which has several advantages for industrial application as listed in Section 2.2. As a result, in some cases selections from the significant body of literature on friction stir welding in aluminum alloys are discussed here instead, accompanied by an explanation of the impact of alloy change on the results described.

### Friction Stir Welding Processing Parameters

The two most critical friction stir welding processing parameters are the tool rotation rate and the tool rate of advancement along the joining path, also referred to as the welding speed [6]. The tool axis of rotation is generally angled between 1-3° from the plate normal away from the welding direction to encourage the retention of the plasticised material and provide consolidation force [44]. Friction stir welding rotational rates and welding speeds reported in the literature for AZ80 vary widely, from 400-1723 RPM and 32-100 mm/min [45, 9] and excessive or insufficient values of these parameters will result in defects as discussed in Section 2.3.8.

Welds made at different processing conditions may be compared in terms of the heat input, a ranking which increases with increasing tool rotational rate and decreases with increasing welding speed [46, 6]. Care should be taken to mentally separate heat input (which is the energy provided by the friction stir welding tool) from weld temperature, the resulting thermal state of the weld. While the two are directly proportional at lower heat inputs, at higher values of heat input the material
2.3. FRICTION STIR WELD PROPERTIES

Welding direction is out of the page. (Left) magnified view of the AS interface, showing the nugget at right and the TMAZ adjacent to the interface. (Right) characteristic areas of the weld, and a box showing the area from which the view at left was taken. HAZ not optically visible or marked.

softens to the point where the shear motion of the tool generates less heat, and heat input increases have little impact on nugget temperature. The range of processing parameters used for the current experimental work is towards the colder end of the viable processing window, and so it can be assumed that increases in heat input will result in increases in temperature.

**Characteristic Areas of the Weld**

Typical microstructural regions of a friction stir weld are shown as Figure 2.7. Within the welded region are the shoulder flow area near the surface, the nugget or stir zone below, and the swirl flow at the base of the weld. The size and shape of these regions vary with processing conditions [47], and the tool geometry [48].

Moving away from the nugget center there will be a critical isotherm where the flow stress of the material is higher than the local state of stress, resulting in the formation of an interface marking the boundary between the nugget and the thermomechanically affected zone (TMAZ) [49]. The TMAZ may be observed microstructurally from the
grain boundaries, which are generally distorted and elongated as they are drawn towards the nugget, visible in the magnified section at the left of Figure 2.7. As a result of the asymmetry between the AS and the RS, the interface between the weld and the TMAZ is sharper and more clearly visible on the AS than on the RS, as may be seen from Figure 2.7. The shape of the interface is dependent on the degree of material flow through the shoulder nugget and swirl zones, which changes with the processing parameters. Work by multiple authors [47, 50] has shown that changes in processing parameters may alter the interface surrounding the weld from being basin shaped at low heat inputs (similarly to Figure 2.7) to having a sharp inflection point between the nugget and shoulder flows at high heat inputs (similarly to Figure 2.9).

Outside the TMAZ is the heat affected zone (HAZ) which is characterised by only heat input with no plastic deformation [6], and may not be optically visible. Beyond the HAZ is the base material (BM), which by definition has not been affected by the welding process.

Material Flow Path

Chen et al. [51] have shown that the macroscopic plastic flow in joints made by friction stir weld can be divided into three regions: a top zone, dominated by the shoulder forces; a central nugget zone, dominated by pin and thread movements; and finally the swirl zone at the weld root formed by flow under the pin (see Figure 2.8a). Marker insert flow studies in AA6061 by Xu and Deng [47] have shown that within a broad set of processing conditions the shoulder flow is continuous (with material along the joint interface redeposited in a linear fashion behind the tool), while the flow in the nugget zone is periodic, with material from the joint interface deposited in regular arcs behind
2.3. FRICTION STIR WELD PROPERTIES

Figure 2.8: (Left) Reproduced from [51]. Cross section of an AA5083 friction stir weld made such that the flow from the shoulder flow, nugget, and swirl zones has not consolidated. The lower of the two indicated voids is classified as a wormhole defect. (Right) Reproduced from [52]. View downwards behind the fractured pin of a stop motion experiment. AA5083 BOP friction stir weld made at 312 mm/min and 710 RPM showing the fractured pin at top right, and multiple successive layers of periodic material deposits behind the pin. If welding had continued, these would be consolidated into the weld nugget by the rotation of the pin.

the tool. Flow in the swirl zone seems to be somewhat intermediate between the two states, with unstable periodicity at some processing conditions and near continuous behaviour at others. Xu et al. [47] have also shown that the extent to which any one of these three material streams contributes to the finished cross section of the weld varies with the processing conditions, and that the extent of periodic nugget flow is more dependent on the RPM than the welding speed.

Krishnan [53] showed the material periodically deposited in the nugget zone takes the form of successive hemispherical layers which when sectioned on the transverse plane reveal a concentric ‘onion ring’ feature. This is in agreement with the results of multiple stop motion friction stir welding experiments (e.g. [51]) and other investigative techniques [54] which consistently show nugget material moving around
the pin in distinct layers (see Figure 2.8b). In 2XXX and 5XXX series aluminum alloys the onion ring structure has been found to consist of variations in grain size and particle distribution \[55, 56\] in addition to texture change. In all cases where texture measurements of an onion ring pattern were performed in an aluminum alloy, periodic texture changes were reported \[57, 58, 59, 60, 61\] in the 1XXX, 2XXX and 6XXX series alloys used.

While an onion ring structure marked by periodic variations in grain size and particle distribution may be developed due to deformation during the welding process, work by Colligan \[62\] in AA6061 has shown that it may also be formed from inhomogeneous starting material as the tool threads cyclically draw in material from different depths of the base material plate. Attallah et al. \[55\] have shown that using 2XXX or 5XXX series aluminum plate with bands of intermetallic particles will result in a banded friction stir welded nugget containing variations in grain size and particle distribution, while a homogeneous plate used at the same processing conditions will result in a homogeneous nugget without optically visible onion rings.

In contrast to friction stir welding in Al based alloys, onion ring banding reported in the AZ alloy series of Mg alloys is generally attributed to local compositional changes \[9, 45\] caused as the $\beta$ intermetallic phase is affected by the temperatures and shear deformation of material during friction stir welding. Depending on the intermetallic volume fraction and heat input conditions either the precipitate becomes dissolved into the matrix causing local composition changes and different local etching behaviour, or at high RPM it forms liquid films which can lead to cracking or degradation of mechanical properties \[45\]. Since tapered pins produce greater heat input compared to cylindrical pins \[63\], formation of onion rings will be favoured by
tapered tools, as can be noted by comparison of the microstructures produced by Borle et al. [45] and Yang et al. [9]. An alternate type of banding in AZ61 friction stir welds was created with a cooled fixture by Lee et al. [64], who found alternating bands of fully recrystallised fine grains and partially recrystallised coarser grains in the nugget, presumably resulting from cyclic variations in shear strain such as those modelled by Xu et al. [47].

The work of Gratecap et al. [54] studied periodic flow around the friction stir welding tool in detail, using a range of materials, processing conditions and tool geometries. It was determined that in all cases periodic flow of material had occurred in the nugget region [54], indicating that periodic flow in the nugget is a natural consequence of the friction stir welding geometry. Gratecap et al. [54] conclude that in a defect-free friction stir weld separate layers of material are compressed together behind the pin. This result is supported by work of Yan et al. [56], who measured the tool oscillatory motion and forces, and found that forces in the downward and welding direction were cyclic with a period approximately equal to that of the tool advance per rotation. As this occurred even in cases where the tool oscillatory motion was intentionally minimised, it supports the idea that the material behind the tool is compressed into place rather than just moved radially.

Along with the periodic tool flow, there is some evidence that undeformed material is drawn into the friction stir welded nugget without significant deformation. For example, Fonda et al. [58] published optical micrographs indicating that material at the RS interface was periodically drawn into the nugget during friction stir welding. Similarly, EBSD work of Prangnell and Heason [65] on a stop-motion friction stir weld in AA2129 showed that in several regions of the nugget, large unrefined grains
had been drawn from the RS into the nugget and were only partially fragmented.

2.3.2 Strain Rates and Precipitate Dissolution

As discussed in Section 2.2.1, AZ80 precipitation is limited to the Mg$_{17}$Al$_{12}$ intermetallic, for which there is no measurable growth at room temperature [36]. Under static conditions, time for solutionisation of this precipitate can be lengthy and a typical solutionising heat treatment for AZ80 is 24 h at 415 °C [66]. During friction stir welding, dissolution of the precipitate is accelerated by the shear deformation of the tool, which Chang et al. [67] report to be in the range of 5-50 s$^{-1}$ for friction stir welding conditions of 90 mm/min and 180-1800 RPM. Yang et al. [9] report that for AZ80 friction stir welds made at 400-1200 RPM and 100 mm/min SEM measurements showed that the majority of the Mg$_{17}$Al$_{12}$ precipitate present in the base material was dissolved in the nugget. The level of aluminum in solution was found to be increased in the nugget as compared to the base material, and TEM work confirmed that the Mg$_{17}$Al$_{12}$ precipitate was dissolved or broken up within the nugget [9]. Feng et al. [68] also reported that most of the Mg$_{17}$Al$_{12}$ phase was dissolved into the matrix during friction stir processing, with corresponding increases in the content of Al in solution. However, in this case fine Mg$_{17}$Al$_{12}$ particles were found to be distributed along the grain boundaries. In contrast, for thixomoulded AZ91, for four different processing conditions friction stir welding was found to completely dissolve the Mg$_{17}$Al$_{12}$ precipitate in the nugget based on optical microscopy and TEM of this region [69]. Due to the slow precipitation kinetics of Mg$_{17}$Al$_{12}$, and the short cooling time for a friction stir welded nugget (on the order of 60 s [70]) these observations on the distribution of Mg$_{17}$Al$_{12}$ precipitate are anticipated to be reflective of the final distribution of this
precipitate in the welded nugget.

Consequently, it is expected that in the case of higher content AZ series alloys, precipitates will be present in the base material, partially or completely dissolved within the nugget, and of an intermediate state in the TMAZ.

2.3.3 Mechanical Properties and Hardness

Table 2.2 shows a comparison of literature values for AZ80 friction stir welds tested under transverse tension. There is a large variation between UTS of the cast and extruded base materials, with the rolled sheet having an intermediate value. While friction stir welds of cast material have a greater improvement on the mechanical properties (i.e. higher joint efficiency) the overall UTS remains inferior to that of the friction stir welds created in extruded material, and the rolled sheet is an intermediate case.

Comparing the set of friction stir welds made in extruded sheet, the weld created at 800 RPM had superior UTS and elongation, while that made at 1200 RPM had both relatively low value of UTS and high scatter in UTS. This may indicate the higher heat input of this condition triggered formation of a defect, or a liquid film of Mg\(_{17}\)Al\(_{12}\) precipitate in the nugget as discussed by Borle et al. [45]. In general, we expect degradation in material properties as either the cold or the hot end of the defect-free processing window is approached, and microstructure becomes more prone to defect formation.
Table 2.2: Tensile properties for selected AZ80 friction stir welds in MPa, where joint efficiency is defined as $(100 \times \text{weld UTS}/\text{BM UTS})$

<table>
<thead>
<tr>
<th>Alloy</th>
<th>RPM</th>
<th>Welding Speed (mm/min)</th>
<th>Yield (MPa)</th>
<th>Weld UTS (MPa)</th>
<th>Elongation (%)</th>
<th>Joint Efficiency (%)</th>
<th>BM UTS (MPa)</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cast AZ80</td>
<td>400</td>
<td>100</td>
<td>134.6</td>
<td>188.8</td>
<td>3.0</td>
<td>156</td>
<td>120.9</td>
<td>[68]</td>
</tr>
<tr>
<td>Extruded AZ80</td>
<td>400</td>
<td>100</td>
<td>167.0 ± 1.4</td>
<td>291.6 ± 3.9</td>
<td>10.2 ± 0.8</td>
<td>88.4</td>
<td>330 ± 3.4</td>
<td>[9]</td>
</tr>
<tr>
<td>Extruded AZ80</td>
<td>600</td>
<td>100</td>
<td>159.8 ± 1.0</td>
<td>287.3 ± 1.1</td>
<td>8.6 ± 0.4</td>
<td>87.1</td>
<td>330 ± 3.4</td>
<td>[9]</td>
</tr>
<tr>
<td>Extruded AZ80</td>
<td>800</td>
<td>100</td>
<td>160.8 ± 0.5</td>
<td>304.5 ± 1.9</td>
<td>11.5 ± 0.3</td>
<td>92.3</td>
<td>330 ± 3.4</td>
<td>[9]</td>
</tr>
<tr>
<td>Extruded AZ80</td>
<td>1200</td>
<td>100</td>
<td>159.4 ± 0.3</td>
<td>273.7 ± 16</td>
<td>7.2 ± 1.6</td>
<td>82.9</td>
<td>330 ± 3.4</td>
<td>[9]</td>
</tr>
<tr>
<td>Rolled AZ80</td>
<td>500</td>
<td>50</td>
<td>123</td>
<td>220</td>
<td>3.77</td>
<td>75.8</td>
<td>290</td>
<td>[71]</td>
</tr>
<tr>
<td>Rolled AZ80</td>
<td>750</td>
<td>75</td>
<td>131</td>
<td>228</td>
<td>4.94</td>
<td>78.6</td>
<td>290</td>
<td>[71]</td>
</tr>
</tbody>
</table>
2.3.4 Texture and Periodicity

As mentioned in Section 2.3.1, the material in the nugget shears around the pin as it is moved into place. Texture change during friction stir welding is dominated by this shear deformation [57, 60], which at high magnitudes causes formation of the $B$ shear fiber in aluminum with close packed $\langle 110 \rangle$ directions along the shear direction and $\{112\}$ planes aligned with the shear plane and at lower magnitudes causes formation of the $C$ fiber [73]. Using this information, the shear plane, direction, and a rough estimate of strain magnitude may all then be extracted from texture data in aluminum. Investigations of aluminum texture banding in AA2195 have shown it to be consistent with deposition of material layers oriented with $B$ fiber at one surface, $C$ fiber at the interior, and then the opposite direction of $B$ fiber at the final surface of the deposited layer [59].
For magnesium, shear strain causes formation of the $B$ shear fiber, with the (0001) basal plane aligned with the shear plane and some weak preferential alignment of the $\langle 11\bar{2}0 \rangle$ axis in the slip direction [73, 13]. The macroscopic shear strain during friction stir welding involves rotation around the shoulder near the top of the weld and the pin lower in the weld [74], and so the three dimensional texture distribution in magnesium can be visualised as a surface created by revolving the AS interface line around the axis of the tool, with basal planes tangent to this surface [13]. It should be noted that in magnesium the positive and negative $B$ shear fiber are identical, and no other shear textures are formed at lower shear strain magnitudes [75]. Due to this limitation, shear textures in magnesium may only be used to determine the plane of shear, and neither the direction nor the magnitude of strain can be extracted. To date, no investigation of periodic texture changes along the welding direction has been reported for magnesium friction stir welds.

When these shear strains are combined with the temperatures generated during friction stir welding, dynamic recrystallisation (DRX) occurs in the weld nugget [67, 69, 42]. While Chang et al. [67] have reported that DRX will result in reduction in intensity of the texture as new grains with different orientations are nucleated, the intense textures formed during magnesium friction stir welding [13] indicate that shear strain is by far the dominant influence on texture formation in a magnesium friction stir weld. In essence, shear deformation applied with a constant vector is only capable of intensifying texture in magnesium, and any decrease in the texture intensity means that less shear strain was applied, nucleation of new grains occurred following shear, or other strain modes such as compressive were active.

EBSD pole figures from multiple publications confirm basal plane distribution in
2.3. FRICTION STIR WELD PROPERTIES

a magnesium friction stir weld can be visualised as a surface created by revolving the AS interface line around the axis of the tool [13, 72] as shown in Figure 2.9. From the maximum intensity values shown at the bottom of Figure 2.9 it can also be seen that the texture is more intense in the stir zone than in the BM, TMAZ or HAZ [72].

When combined with the plastic anisotropy inherent to magnesium based alloys, this intense texture makes the texture distribution of primary importance for the mechanical behaviour under load. Given the nugget texture, for any stress applied transverse to the friction stir weld there will be basal planes on either side of the nugget which are oriented such that the Schmid factor reaches the maximum possible value [76]. As basal planes have the lowest CRSS of the magnesium deformation systems [28] this will result in low yield regions on either side of the nugget, and is one of the reasons for strain localisation and failure in this area as discussed in Section 2.4.

There is evidence in the literature that this idealised truncated cone view of texture is a simplification of the true texture distribution. For example, texture measurements of regions along the centerline of an AZ61 friction stir weld show asymmetry both at the mid thickness and root of the weld [13]. Neutron diffraction measurements across the midline of an AZ31 friction stir weld which incorporate information from a much larger measurement volume likewise show asymmetric diffracted intensity [77], indicating that the texture measured was asymmetric.

2.3.5 Thermal Distribution

Work in AA5083 by Lombard et al. [78] showed that the thermal input during friction stir welding has a critical influence on the resulting maximum residual stress. Prior to
2.3. FRICTION STIR WELD PROPERTIES

Discussing the distribution of residual stresses in a friction stir weld in Section 2.3.6, it is therefore productive to have a good understanding of the typical temperature distribution of the material immediately following passage of the tool. To this end, several studies using a wide range of measurement methods are discussed to provide a full picture of the typical thermal profile of a friction stir weld, and how this would be expected to change with processing parameters and material.

Moving vertically through the weld, multiple studies agree that the highest temperature will be under the tool shoulder. This includes three studies of friction stir welding nugget thermal distribution in AA6061; the work of Woo et al. [79] using in-situ neutron diffraction, the work of Kandaswaamy [80] using thermocouples adjacent to the nugget, and the work of Fehrenbacher [81] who used a friction stir welding tool instrumented with two thermocouples. Similarly, for magnesium AZ31 work by Yu et al. [82] using simulations in conjunction with experiments showed the highest temperature was reached under the shoulder, adjacent to the top of the threaded pin.

![Figure 2.10](image.png)

Figure 2.10: Reproduced from [81, Fig. 37]. Experimentally measured temperatures around the circumference of a friction stir welding tool during welding of AA6061. (left) shoulder near the pin, (right) pin, near the tip

Work by Huetsch et al. [83] using instrumented AZ31 base material showed that the temperature distribution is asymmetric around the pin, and higher on the AS, a result supported by numerous other works. For example, Kandaswaamy [80] measured
the temperature in AA6061 at a distance of 2.5 mm from the shoulder and the pin, as well as 1.3 mm beneath the pin passage and found that in descending order, the temperatures were shoulder AS, pin AS, shoulder RS, under pin centerline, and finally pin RS. This order was consistent over the course of multiple trials and (within the range tested) unaffected by changes in the RPM, welding speed, or tool shoulder diameter [80]. Using a friction stir welding tool with integrated thermocouples under the shoulder and on the side of the pin, Fehrenbacher [81] was able to precisely locate the highest temperature around the tool circumference as being on the AS approximately 60° from the direction of tool movement, as shown in Figure 2.10. Fehrenbacher [81] attributed the higher temperature at the AS to higher shearing and velocity gradients there, as well as the introduction of cooler new material which enters at the front of the tool and flows around the RS first.

The above spatial distribution is in agreement with the work of Riahi and Nazari [84] who used experimental data in conjunction with computer simulations to generate a three dimensional map of the temperature distribution of an in-process friction stir weld, shown as Figure 2.11.

![Simulated Heat Distribution](image)

Figure 2.11: Reproduced from [84]. A two dimensional cross section of the simulated heat distribution during friction stir welding of AA6061.

Comparing the relative impact of lower welding speed and higher rotational rate on the temperatures at the shoulder and the pin, Kandaswaamy [80] found that in
AA6061 the most significant factor was the welding speed, followed by the shoulder diameter, and finally the RPM. In contrast, Fehrenbacher found that for the same alloy RPM generally had a more significant impact on temperature than welding speed [81, p.54-56], although in some cases the effects of welding speed could dominate over those of RPM [81, p. 82].

As can be seen in Figure 2.12a, the influence of changes in RPM and welding speed on the temperature near the shoulder diminishes as the solidus temperature is approached. This makes it possible to reconcile the dominance of welding speed proposed by Kandaswaamy [80] to the dominance of RPM proposed by Fehrenbacher [81], as the work of the former was conducted within a much cooler processing window.

Comparing the temperatures measured near the shoulder and pin shown in Figure 2.12a and b, as temperatures increase, the disparity in temperature between the shoulder and the nugget decreases. In other words, increasing heat input from high RPM and slow welding speeds promotes an even temperature distribution. For example, for the lower heat input welding condition of 1700 RPM and 500 mm/min the
temperature difference between shoulder and pin is approximately 41 °C, as opposed to less than a degree when heat input is increased with processing conditions of 1700 RPM and 100 mm/min [81].

Asymmetry of the weld thermal profile has also been found to be affected by processing conditions. Work by Fehrenbacher [81] found that in AA6061 temperatures varied by 5-30 °C around the tool circumference, with higher variations at higher travel speeds [81]. This agrees with the work of Huetsch et al. [83] in AZ31 who used simulation work in conjunction with thermocouple measurements to determine the effect of three different welding speeds on the temperature distribution, with results shown graphically in Figure 2.13. Huetsch et al. [83] reported that as the processing speed increased the temperature distribution became increasingly asymmetric as the importance of material flow around the pin decreased and the importance of extrusion phenomena increased.

Looking farther away from the friction stir welding tool, Figure 2.13 shows that the zone of material at over 320 °C decreases with increasing welding speed until there is virtually no trailing heated material behind the tool shoulder. This is due to the constant thermal conductivity combined with the increasing feed of cold material into the weld, which will result in decreasing volumes of heated material [83].

Golezani et al. [85] used measurements from a thermocouple inserted below a sheet of AA7020, and measured friction stir welds made at 100 mm/min, and a range of rotational rates. They reported the peak temperature increased with increasing RPM and heat input, from 209 °C at 400 RPM and 100 mm/min to 311 °C at 1000 RPM and 100 mm/min. The time from passage of the welding tool to temperature decreasing below 100 °C was under 25 s for 400 RPM and about 85 s for friction stir
2.3. FRICTION STIR WELD PROPERTIES

Figure 2.13: Reproduced from [83, Fig. 2], with alternate units. Based on modelling of thermocouple input. Comparison of (top) 200 °C and (bottom) 320 °C isotherms for friction stir welding at various parameters.

welds made at 1000 RPM [85]. These measurements were made farther away from the center of the nugget and subject to thermal losses from the material backing, and so are unsurprisingly cooler than those reported by others such as Fehrenbacher [81] and Khandkar et al. [70], who are in close agreement. Khandkar et al. [70] used instrumented AA6061 sheet during friction stir welding and reported a peak temperature of 460 °C and that time from peak welding temperature to below 100 °C was just over 60 s for a friction stir weld made at 390 RPM and 141.8 mm/min. In comparison, for AZ31 Commin et al. [11] reported a maximum friction stir welding nugget temperature of approximately 400 °C.

Kandaswaamy [80] found that for all tests, the temperature at the start of the weld
was greater than that at steady state due to the dwell effects, while Fehrenbacher [81, p. 53] found that it was lowest at the start and increased to a steady state condition, which was supported by the work of Darras [86, p. 61-62] over several rotation rates and welding speeds in AA5052. Yu et al. [82] used simulation work to estimate the travel distance to steady state thermal conditions in AZ31, which was determined to be 8 cm for friction stir welding parameters of 66 mm/min and 600 RPM.

It is expected that different materials with different thermal properties will show different temperature distributions during friction stir welding. As the thermal diffusivity of the material increases, the spatial distribution of temperature within the friction stir weld should become more homogeneous. This is supported by the work of Fehrenbacher [81] who reported that the amplitude of temperature change during one revolution decreased as the thermal diffusivity increased for a series of aluminum alloys, as shown in Figure 2.14.

Work by Lee et al. [87] gives us thermal diffusivity values of $5.7 \times 10^{-5} \text{m}^2\text{s}^{-1}$ for AZ31, and $4.7 \times 10^{-5} \text{m}^2\text{s}^{-1}$ for AZ61 at this temperature. Work by Popescu et al. [88] gives a thermal diffusivity of $3.9 \times 10^{-5} \text{m}^2\text{s}^{-1}$ for AZ80 continuing the trend of decreasing thermal diffusivity with increasing aluminum content and leading us to predict a less homogeneous temperature distribution in this alloy as compared to either AZ61 or AZ31. As the thermal diffusivity for AZ61 is similar to that of AA5083 shown in Figure 2.14, we would expect a similar interface temperature amplitude (i.e. change in temperature during one rotation of the tool pin) for AZ61, a more homogeneous temperature distribution for AZ31, and a less homogeneous temperature distribution for AZ80.
2.3. FRICITION STIR WELD PROPERTIES 40

Figure 2.14: Annotated from [81], to add AZ alloy markers. Effect of material thermal diffusivity on the change in temperature radially around the pin for two depths. Thermal diffusivity for two magnesium alloys shown with dashed lines (AZ80 off scale to left).

2.3.6 Residual Stress

The current section presents general trends in spatial distribution of residual stress across friction stir welds in various materials. Magnitudes reported are discussed only briefly as they are influenced by the size of the sample being examined [89, 90]. Supporting materials theory and general methods of measurement for residual stress are discussed in Section 2.5, while the residual stress calculations made during the current project are included in Chapter 5.

As a general description, residual stresses are the self-equilibrating stresses that exist internally to a material body in the absence of any applied stresses [91]. Residual stresses exist on several length scales. Microstresses in the material extend over a length scale of a few atomic distances (Type III) or the scale of the material grain...
size (Type II) [92, p. 47]. Type I macrostresses extend over the scale of many grains [92, p. 47], and are the primary concern of the current work.

An example of the development of residual macrostresses reproduced from Hosford [48] is shown as Figure 2.15. Visualise a structure of three carbon steel bars originally of identical dimensions connected by rigid blocks at each end. We begin by heating the center bar to about 600 °C, shown as line A-B in the figure, during which the bar is undergoing elastic compressive strain due to thermal expansion. At B the bar yields in compression and begins to deform plastically. With further temperature increases the yield strength of the material decreases and so lower elastic compressive stresses can be supported by the bar, and it continues to deform plastically as it continues to expand.

When cooling begins at C the bar begins to reduce in length, and so from C-D it first unloads from compression and then loads again in tension elastically. At D the yield in tension is reached, and tensile plastic strain begins. From point D-E the temperature continues to decrease and the yield strength in tension increases, so more elastic strain is supported by the bar, up until point E. Point E represents the maximum possible residual stress in tension for the current situation, and further temperature increases during the thermal cycle would have had no effect on the final residual strain state of the bar. By plotting the line E-B’, parallel to the line A-B we can determine that for this part geometry and material the minimum temperature increase that results in development of this maximum residual stress is approximately 300 °C. If the bar was severed while under this residual tensile stress, elastic tensile residual strain would be released, and the central bar total length would now be lesser than the adjacent bars.
2.3. FRICTION STIR WELD PROPERTIES

For both fusion and friction stir welds, residual stress is mainly caused by inhibited thermal contraction of the weld bead or nugget during the cooling of the weld [93], much in the same way the central bar was inhibited from contracting as it cooled in the example above. These macroscale thermal residual stresses combine with applied stresses [91, p. 8110], improving or worsening the fracture resistance [91, p. 8151] and so a good understanding of the distribution of these macrostresses is a prerequisite for understanding failure in a friction stir weld. Residual stresses, particularly those in the TD [94] also influence distortion of the weld as illustrated schematically in Figure 2.16 [91, p. 8110, 8123].

During the process of friction stir welding, the material around the tool is heated
and plasticised, in some cases reaching the solidus temperature of the material [81](see Figure 2.12). At this high temperature, thermal expansion effects are readily accommodated within the nugget as the yield strength of the material is low. As the hot material of the recrystallised weld cools it undergoes thermal contraction, and at some point the yield strength of the material is high enough that instead of being accommodated by plastic strain, the thermal contraction generates elastic residual strains instead. In other words, the incomplete relaxation of the elastic strain from the thermal contraction leaves residual stresses in the material [92]. At room temperature these are typically distributed as shown in Figure 2.16.

In addition to thermal effects, residual stresses from fusion welding are due to solidification, other phase transformations, and material movement such as that between two butted plates [91, p. 8122] in addition to contributions from mechanical
deformation [95]. For AZ magnesium alloys, neither solidification nor phase transformation effects are expected to occur during cooling of the nugget following friction stir welding. While the mechanical deformation in the nugget of a friction stir weld does impact the residual stress state, Woo et al. [95] confirmed that the deformation from the pin was a less significant factor in the final residual stress than the heat input from the shoulder. Friction stir welds made for research purposes are frequently done along the surface of a continuous plate to avoid material movement concerns, as was done for the current work. This leaves inhibited thermal contraction of the nugget during cooling as the main source of residual stresses in a friction stir weld [93].

As discussed in Section 2.3.5 the thermal profile of the weld is asymmetric, not constant through the depth, and also affected by the material thermal diffusivity. Given the correlation between thermal and residual stress distribution, it is therefore to be expected that the residual stress distribution will also be asymmetric and vary through the depth of the weld. This makes the limitations of working with the line scan techniques frequently used for residual stress measurements obvious.

Residual stress measurements in the AZ series of magnesium alloys are mainly limited to the work in AZ31 by Woo et al. [96, 97, 93] with no apparent works for AZ80 or other high alloy-content materials in this series. Based on the thermal diffusivity disparity between AZ31 or AA6061 where residual stress measurements are available, and AZ80, it is also likely that the residual stress distribution will have some significant differences.

Reported transverse residual stress results for friction stir welds show significant variation, and several examples are shown as Figure 2.17. In most cases peak stresses
Figure 2.17: Transverse residual stress and strain profiles for a range of friction stir welded alloys. a) Reprinted from [78]. TD residual stress in several AA5083 friction stir welds made at different welding speeds and RPM. b) Reprinted from [97] TD residual stress (in red) in an AZ31 friction stir weld made at 600 RPM and 58.2 mm/min c) From [94, Fig. 4.8]. TD residual stress map for half of an AA6061 friction stir weld made at 1250 RPM and 282 mm/min. d) Reprinted from [98]. TD residual stress at two depths in a stainless steel 304L friction stir weld made at 102 mm/min and 500 RPM.

are aligned with the edge of the tool shoulder’s passage, where the highest temperatures are recorded during friction stir welding [84]. In some cases, the maximum transverse residual stress is reported to be tensile underneath the tool shoulder (e.g. [78, 97, 99] or Figure 2.17 a,b), while in others this peak residual stress is reported to be compressive (e.g. [94, 100] or Figure 2.17c). Typically, in the center of the friction stir welded nugget the residual stress approaches zero.

Woo et al. [93] suggest that in aluminum the underlying mechanism for this central reduction in residual stress is microstructural softening from precipitate dissolution and other sources limiting the maximum residual stress the region can support. This
softening along the weld centerline in aluminum may be seen by hardness testing (e.g. [96]). In contrast, for magnesium AZ31 Woo and Choo [96] found that hardness was essentially constant between the nugget and the base material, and instead attributed this decrease in residual strain to a local reduction in the yield stress due to the texture change across the friction stir welded nugget. A conflicting conclusion was drawn in the work by Webster et al. [101] in AA7108 friction stir welds which found that texture and residual strain were not closely coupled, and that the intensity distribution from texture did not align spatially to the residual strain distribution.

Residual stresses vary through the sample thickness, and Figure 2.17c shows a sample with fairly minor changes through the friction stir welded depth. In contrast, other samples show significant differences between measurements made at different depths, for example Figure 2.17d. These differences in transverse residual strain distribution are expected to be a function of the temperature distribution of the friction stir weld which will be a function of the thermal properties of the weld material, tool, and backing plate, as well as the processing conditions used. The location of measurement with respect to the weld ends is also expected have an influence, as the transverse residual strain measured along the centerline of the weld is at a tensile maximum halfway from the weld start as shown in Figure 2.16c.

The work of Lombard et al. [78] provides line scans of residual stress in the WD and TD at a wide range of processing conditions. This work provides evidence that higher heat input decreases residual stresses, and that there is decreasing difference between the AS and RS peak value as the feed rate increases [78].

Information on residual stresses in the normal direction is limited, and as the temperature gradient horizontally in a friction stir weld (see Figure 2.11) is greater
than that in the normal direction, we would expect the residual stress in the normal
direction to be lower than that in the transverse direction, making it less of a practical
concern. The work of Woo [94] showed the distribution of residual strains measured
across the nugget to be similar between the ND and TD direction, with a peak
(compressive) value under the edge of the shoulder’s passage, approaching zero in
the nugget center (see Figure 2.18 b). These peak compressive strains in the ND
below the shoulder were greater than those measured in the TD, which were also
compressive (see [94]). In terms of absolute magnitude, both the ND and TD residual
strains were reported to be lower than the peak tensile residual strains measured in
the longitudinal direction [94]. Woo [94] also reported that variation in ND residual
strain was greater through the depth of the friction stir weld than variation in TD
residual strain through the depth of the nugget. This implies that heat transfer in the
ND varied more vertically through the friction stir welded nugget than heat transfer
in the TD, which is sensible given the presence of a backing plate below the weld
which generally acts as a heat sink.

A similar residual stress distribution was reported by Sutton et al. [102], with
a peak compressive residual stress under the shoulder, trending to zero along the
centerline (see Figure 2.18 a). In contrast to the work of Woo [94] (see Figure 2.18
b), the residual stress was found to be more compressive at the top of the weld than
the root.
2.3.7 Tool Geometry and Wear

As mentioned in Section 2.3.1, the most basic form of a friction stir welding tool is a threaded cylindrical pin with larger shoulder. Work by Padmanaban and Balasubramanian [103] has shown that in AZ31, friction stir welds made with a threaded cylindrical pin had superior joint tensile efficiency and fewer defects as compared to those made with an unthreaded cylindrical tool. They attributed this to the dual impacts of increased vertical motion of material and heat generation in the weld caused by the threads [103]. As the tool undergoes wear, the pin reduces in length, and features which promote stirring are eroded [104]. Since the threads of the pin force downward motion of the softened material [58] and improve material flow around the pin [103], their erosion contributes to the formation of defects at the weld root [104] particularly void-type defects such as wormholes.
2.3.8 Defects

Friction stir welding defects result from the selection of unsuitable tool geometry and welding conditions [105]. Excessive heat input will lead to defects such as excessive flash formation, surface galling and nugget collapse [106], as the material becomes excessively soft and escapes from under the tool. Insufficient heat input will cause the formation of voids inside the weld or lack of bonding in the stir zone [42, 106], as the material becomes insufficiently formable to rejoin behind the tool. Wormholes are a common lack-of-fusion defect resulting from insufficient temperature in the weld and lack of consolidation forces [106]. This defect forms low in the weld at the junction of the advancing side flow and the swirl flow [49], as seen in Figure 2.8. As conditions move farther away from ideal, the wormhole may even become large enough to form a continuous tunnel at the root of a friction stir weld.

Defects in a friction stir weld can cause a drastic reduction in mechanical properties. Chen et al. [105] found the tensile strength of an AA5456 friction stir weld decreased from 95 % that of the base metal to 73 % with the presence of a groove defect in the weld surface. Non-destructive inspection of welds for internal defects can be costly in terms of time and expertise, while destructive testing carries the additional cost of sampled parts. It is thus desirable to design a friction stir welding process that is insensitive to minor variations in processing parameters so that the parts become consistent and reliable, decreasing the frequency of costly product testing.
2.3.9 Welding of Dissimilar Alloys

In the magnesium AZ series alloys dissimilar friction stir welding research is limited, and consists mainly of the paper by Liu et al. [14] who investigated an AZ31-AZ80 friction stir weld. The authors reported that fewer defects occurred when the material with greater plastic deformability was placed on the RS of the weld [14], which was also found in the current work 6.

Liu et al. [107] created dissimilar friction stir welds joining cast ZK60 (Mg, Zn 5.2 %, 0.5 % Zr) on the AS to hot rolled solutionised AZ31 at 800 RPM and 100 mm/min, and found the resulting textures to be essentially those of a similar friction stir weld. The tensile UTS and elongation of the friction stir weld was found to be lower than either of the composing materials, while the yield strength was intermediate to the two base materials used.

Similarly, the work of Liu et al. [14] reported UTS of approximately 235 MPa for dissimilar friction stir weld made from extruded AZ80 and AZ31, which is below the UTS for both extruded AZ31 and extruded AZ80 listed in Table 2.1, and confirms loss of properties during dissimilar friction stir welding can be quite significant.
2.4 Strain Localisation and Failure

From comparison of the transverse mechanical properties shown in Table 2.2 to the base material properties shown in Table 2.1, it is evident that friction stir welding generally causes loss of mechanical properties in comparison to the base material. This section discusses the microstructural basis for this loss of properties, and how the strain localisation during transverse deformation leads to failure in AZ series FSW.

In friction stir welds, failure from transverse tensile loading almost invariably occurs at the interface between the advancing side of the nugget and the TMAZ [108, 11, 109]. Multiple works have attributed failure along this interface to either an abrupt texture change resulting in non-uniform plastic deformation [108, 50] or the texture in this specific location causing local yielding [6, 110, 72, 50], and it is likely that these are both contributing factors.

At the beginning of transverse tensile testing, the friction stir weld deforms homogeneously before strain localises in bands at the AS and RS interface [83], while deformation in the center of the nugget remains low [72, 111]. These high strain areas at the sides of the weld align with regions where orientation is favourable for extension twinning and basal slip [72], as discussed in Section 2.3.4.

Yang et al. [50] transversely loaded AZ31 friction stir welded samples in tension to varying degrees and found that near the yield strength of the material, extension twinning was present along the interface between the stir zone and the TMAZ, in the location where failure occurs the most frequently. While similar reports of extension twinning distribution were made by Xin et al. [72], they attributed the strain localisation to the extensive basal slip which had occurred along the interface.
Yang et al. found that in AZ31 friction stir welds at lower stress levels almost no twins were present in the nugget area, but that between 75 and 95 % of the UTS many contraction and double twins initiated and grew at the center of the nugget [50]. Despite the formation of twins in the nugget, this area undergoes virtually no macroscopic contraction [111], confirming that twinning is unlikely to be a significant contributor to necking, and highlighting the inhomogeneous nature of deformation in a friction stir weld. After fracture, the lack of necking outside the nugget area shows that most of the base material deforms only in an elastic fashion [83].

One exception to this common fracture mode is AZ91 friction stir welds where the base material is cast, as the coarse structure with intermetallic particles and shrinkage defects will cause the sample to fail outside the weld [38].

2.4.1 Differences in Fracture due to Precipitate Growth

It is worth noting that fracture behaviour is quite different in age hardenable aluminum alloys, which have much more rapid precipitation kinetics as discussed in Section 2.2.1. During friction stir welding of age hardened aluminum alloy, the added heat input from welding inevitably causes overaging and thereby fracture in the HAZ.

For example, Mahoney et al. [112] reported that in an AA7075-T651 friction stir weld, fracture occurred in the HAZ where overaged precipitates triggered failure, and similar results have been reported in other age-hardenable aluminum alloys [6]. This confirms that while some research in aluminum is applicable to magnesium friction stir welds (e.g. material flow paths discussed in Section 2.3.1), strain localisation and failure behaviour is not included in this, and specific magnesium alloy work is required.
2.5 Transmission Diffraction Theory

2.5.1 General Diffraction Theory

For diffraction to occur, the incoming beam must have a wavelength of the same order as the spacing between the crystallographic planes of the material. To accomplish this, diffraction experiments are typically performed with a beam of neutrons at a reactor, or with photons in the form of X-rays which may be produced either on the laboratory scale or in higher energy form at the testing facility of a synchrotron.

The selection of lab scale X-rays, synchrotron X-ray diffraction, or neutron diffraction determines the maximum depth of material that may be probed, the signal intensity, and the possible Bragg angles [21]. These factors in turn dictate which experimental geometries are feasible. For magnesium, the depth of material at which the signal is attenuated by half varies from 0.1 mm for lab scale X-rays, to about a 100 mm for neutrons [113, pp. 41], to about 17 mm for synchrotron X-rays [114] of the type used for the current experiments. As lab scale X-rays are restricted to surface analysis, they will not be discussed further here.

For a given experimental facility, the length of time per measurement can be decreased by using a larger diffracting volume at the cost of reduced spatial resolution. The diffracting volume is the area of the sample that is both illuminated by the beam, and capable of diffracting onto the detector within any aperture limits.

Neutron diffraction in magnesium typically uses volumes of 2 mm$^3$ [97] and a measurement time on the order of 20 min per peak [115], while the current experiments used volumes less than 0.02 mm$^3$ and 3 s per measurement, with corresponding effects on the maximum possible spatial resolution and equipment time requirements.
For synchrotron and neutron diffraction experiments the vast majority of the incident beam will continue through the sample unaffected. This undiffracted portion of the beam (referred to as the transmitted or direct beam), may be used for experimental control, but is generally not used for measurement purposes. To diffract, a crystallographic plane of atoms must be oriented with the Bragg angle $\theta_b$ relative to the transmitted beam. $\theta_b$ can be calculated from Bragg’s law; $n\lambda = 2d\sin\theta_b$, where $n$ is the order of the reflection ($n=1$ used here), $\lambda$ is the wavelength of the incoming beam, $d$ is the spacing between parallel lattice planes and $2\theta_b$ is the angle between the transmitted beam and diffracted beam [92], which is shown schematically in Figure 2.19. As each crystallographic plane or order of reflection has a different $d$ spacing, it has a different associated Bragg angle $\theta_b$.

Figure 2.19: Adapted from [116, Fig. 3-4]. Common parameters in diffraction experiments shown schematically, exaggerated scale within sample. This experimental setup is referred to as a $2\theta$ type experiment. $Q$ represents the normal of the diffracting planes and the direction along which strain is measured.

While there are many possible diffraction experiments, for the current discussion we shall assume that in all cases the incoming beam is collimated with a known
monochromatic wavelength, and $\theta_b$ is measured experimentally, allowing for calculations of $d$ to be made\cite{113}. Nominal values of $d$ spacing found in the literature (e.g. \cite{19}) can be compared to the measured values to identify the crystallographic index of the diffracting planes. Two types of monochromatic diffraction experiments are the common $2\theta$ type illustrated in Figure 2.19, and the low angle transmission setup of the current work which uses a constant 90 degree incident angle. A detailed comparison of these methods is made in Section 2.5.5, with particular attention to the advantages of each approach.

Figure 2.20: Redrawn based on \cite[Fig. 3-12]{116}. Schematic of Debye-Scherrer diffracted cones from a transmission experiment. Wireframe box represents a sample with a diffracting volume composed of many grains. For each cone, the half-apex angle is $2\theta_b$.

In cases where the diffracting volume contains multiple grains with varying orientations, each diffracting plane for each grain will generate one diffracted point, so a single grain may generate one, multiple, or no diffracted points on the detector depending on the orientation. Given a sufficiently large number of grains with varying orientations within the diffracting volume, the resulting diffracted beam will take the
2.5. TRANSMISSION DIFFRACTION THEORY

form of a continuous cone with a half-apex angle of $2\theta_b \ [113]$. As this will happen for a number of lattice planes simultaneously, multiple nested cones will form as shown in Figure 2.20. The circle or ellipse that results on intersection of this cone with a detector is called a Debye-Scherrer ring [113]. Depending on the experimental geometry the full ring or only a portion may be captured. If the diffraction volume is 10-100% greater than the grain size [91, p. 8161], a large number of diffraction spots will appear, and the Debye-Scherrer rings will be smooth in appearance, while coarser grained samples will result in visible spots around the ring [117, p. 308]. Figure 2.21 shows the concentric Debye-Scherrer rings captured using low-angle transmission experiments with synchrotron X-rays for two measurement locations, one in the finely-grained weld and one in the base material which has coarser grains. The disparity in grain size between the two regions is clearly visible in the diffracted data image, particularly in the outer rings.

Figure 2.21: Author’s work. Diffraction patterns from (left) base material (right) stir zone. The innermost ring is diffraction on the $\{10\bar{1}0\}$ plane family, and the next larger is from the $(0002)$ plane. Note the smoother rings at right indicating finer grain size, and the highly textured nature of the material in both locations.
The three most significant microstructural factors in terms of influence on the Debye-Scherrer rings are chemical changes (which cause changes in diameter of the ring), texture changes (which affect the distribution of intensity around the ring circumference), and residual strains. While hydrostatic residual strains cause changes in the diameter of the diffracted rings, deviatoric strains cause eccentricity of the diffracted ring, and so the two can be independently determined if the diameter of the ring when only under chemical effects \( (d_0) \) is known. Analysis methods and deconvolution of these effects are discussed in detail below.

While temperature changes of the diffracting material would also cause changes in the diameter of the Debye-Scherrer rings, all of the work of this project was conducted at room temperature, and so that topic is not addressed in this work. In brief, the effects of temperature are analogous to those presented for chemical changes in terms of anisotropy, intergranular effects, and texture dependencies.

Table 2.3: Change in magnesium unit cell axis length for solid solutions of varying content at 25 °C. Values extracted from \[19\].

<table>
<thead>
<tr>
<th>Pure Mg unit cell length in ( \langle 11\overline{2}0 \rangle )</th>
<th>Pure Mg unit cell length in [0001]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Mg</td>
<td>3.2099 Å</td>
</tr>
<tr>
<td>Change along ( \langle 11\overline{2}0 \rangle ) per added atomic % in Åx10^{-4}</td>
<td>Change along [0001] per added atomic % in Åx10^{-4}</td>
</tr>
<tr>
<td>Added Al</td>
<td>-41.1</td>
</tr>
<tr>
<td>Added Zn</td>
<td>-48.1</td>
</tr>
</tbody>
</table>

2.5.2 Chemical Changes

Compositional changes away from pure magnesium will result in changes to the dimensions of the crystallographic unit cell introduced in Figure 2.1, which may be measured by diffraction. For the current project, the two major magnesium alloying
elements are Al and Zn, which will both cause decrease in the dimensions of the magnesium unit cell when substituted for magnesium atoms as shown in Table 2.3.

Under equilibrium conditions at room temperature only about 1 atomic % [118] of either Al or Zn is in solid solution, with any greater alloy content forming the \( \text{Mg}_{17}\text{Al}_{12} \) precipitate described in Section 2.2.1. However, under non-equilibrium conditions greater concentrations of Al and Zn may retained in the matrix as a solid solution.

**Measuring composition by diffraction** Given experimental measurements of the Debye-Scherrer rings in a strain free sample, the calculated \( d_0 \) spacing of the unit cell may be used in conjunction with literature values such as those of Table 2.3 to calculate the average composition of the diffracting volume which could easily be less than 0.01 mm\(^2\) in the case of synchrotron diffraction. This method has been commonly used to determine composition of binary alloys [116, pp. 388] and allows for much finer sampling than typical chemical methods which require samples in the neighbourhood of several grams.

This approach has two major limitations, namely that hydrostatic strains must be accounted for as discussed in Section 2.5.7, and that the maximum number of elements that can be measured is limited by the crystallographic symmetry of the sample.

Compared to compositional measurements made by the \( d \) spacing method described above, electron based techniques such as EDS (Energy Dispersive X-ray Spectroscopy) have finer maximum resolution in exchange for a far smaller depth of penetration. For example, in magnesium EDS is limited to a depth of about 5 \( \mu \text{m} \) at 20 keV [119]. Although EDS has the advantage of simultaneously extracting
and categorising data from multiple elements in a qualitative fashion, accurate quantitative results require calibration targets. While requiring good surface preparation, EDS is also more easily available for research purposes as it is routinely installed in most modern scanning electron microscopes.

Two advantages of the $d$ spacing method as compared to destructive chemical analysis methods are the low volume of material required, and the ability to differentiate between elements precipitated and those dissolved in the matrix. As such, when investigating the precipitation or dissolution of phases by diffraction, the $d$ spacing method would be a valuable complement to the technique of checking for the presence of diffraction peaks. While the presence of diffraction peaks associated with a precipitating phase is a definitive indication of the presence of a coherent phase, at low quantities the peaks will be low in intensity and therefore difficult to separate from background noise. By combining the two techniques, the simultaneous decrease in solutionised phase and increase in the intensity of the diffracted precipitate peaks could be used to more precisely detect the onset of precipitation.

2.5.3 Texture Measurements and Methods

Texture is the distribution of crystallographic orientations for a specific region [113], and both affects and is affected by the mechanical deformation of a material. Bulk texture of a material is commonly measured by diffraction methods such as X-ray or neutron diffraction. At any X-ray or neutron beam wavelength, the majority of grains will not diffract, and so it is necessary to rotate the sample axes with respect to the incoming beam to determine the complete sample texture. From measurements of the diffracted intensities at multiple angles for multiple planes, the complete texture
distribution may be calculated using freely available software (e.g. \[23\]). The experimental methods involved will vary with the equipment to be used, and a detailed discussion of how this process differs between the $2\theta$ diffraction method (which uses point detectors) and the transmission method of the current work (which uses area detectors) is available in He [117].

When a full Debye-Scherrer ring is captured on the detector, the distribution of the measured intensity around the circumference of the ring provides information on the distribution of those planes that are in the diffraction condition. An isotropic texture will show equal intensity around the circumference while samples with a preferred texture will instead show lower intensity in some directions to the point of having gaps in some rings [113]. From this distribution a rough estimate of the texture intensity and orientation may be extracted. In the case of $2\theta$ type diffraction experiments strong textures can cause issues when the selected portion of the Debye-Scherrer ring is of insufficient intensity for valid diffraction peak measurement. Examining the two innermost rings of Figure 2.21, it is evident that in both the base material and the weld a strong texture has been developed as is typical of Mg alloys (see Section 2.3.4), and that there are multiple directions where $2\theta$ type diffraction peak measurements would be quite challenging.

Comparing texture measurement capability, EBSD data provides a complete orientation for each measurement point while synchrotron data provides only the orientation of the grains that are at the Bragg angle with respect to the transmitted beam. For the current synchrotron experiment Bragg angles are small and only an incident angle of 90° was used, and so the texture information is limited to the fractional subset of grains with a plane normal essentially perpendicular to the incoming beam.
While it may be theoretically possible to reconstruct this limited information from a series of planes into a full texture, it would be a complex computational undertaking.

While EBSD is restricted to a small penetration depth (<0.01 mm [119]), the high intensity synchrotron beam can penetrate through several mm of magnesium alloy with ease. A smaller diffraction depth means that EBSD has the disadvantage of high sensitivity to surface preparation procedures and artefacts as compared to the synchrotron method, in exchange for a far greater resolution and finer measurement spacing. As consequence, much finer details can be extracted from EBSD scans, which permits examination of trends across interfaces, and multiple measurement points within a single material grain. The span of coverage from both methods is also different and testing a grid of points spanning a sample several mm in area is far more straightforward in synchrotron work than EBSD.

2.5.4 Residual Strain

As discussed in Section 2.3.6, residual stresses are internal balancing forces between regions of a friction stir weld and have measurable effects on the performance of the welds. This section describes the basic theory of how residual stress affects diffraction, the influence of material properties, and a high level overview of the experimental approaches used in this project.

While the discussion of Section 2.3.6 referred to residual stresses, in actuality only residual strains of the material are actually measurable [92] by diffraction. In the direction of local tensile forces these residual strains manifest as increases in the crystallographic spacing of the lattice planes (represented by $d$), while in the direction of local compressive forces, values of $d$ are decreased [116] as compared to the natural
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lattice spacing of the unstrained material \((d_0)\). These microscopic lattice strains may be measured by diffraction, and converted to stress values.

Visualisation of residual strain and stress Consider the simplest possible (and somewhat unrealistic) situation of a material and experiment that meets the following conditions. Keep in mind that references to the ‘material element’ refer to the entire diffracting volume, and not just the particular grains or crystallographic planes that diffract. Let us consider a material element where

- Elastic properties are isotropic
- No intergranular strain is present
- Texture intensity is equal in all directions
- All residual strains are elastic and not plastic

The effect of each of these conditions on the diffraction behaviour is discussed later in this section.

For a material element of this ideal situation, stress is a continuous function in 3D. We can visualise this elastic stress as an ellipsoid centered at the origin, shown schematically as Figure 2.22. The three major ellipsoid axes are the directions of principal stresses in the material element. The ellipsoid may be represented mathematically as a second-degree tensor, and when we define the three axes of the stress tensor (Figure 2.22a) to be parallel to the stress ellipsoid major axes \((\sigma_{I-III})\) in Figure 2.22b), all non-diagonal elements of the tensor will be zero indicating no shear stresses are present in the tensor [120].

The process flow for calculating stress from diffraction measurements of \(d\) spacing is shown in Figure 2.23. To calculate the full stress ellipsoid we must first calculate
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Figure 2.22: a) Equation for the 3x3 second rank stress tensor. b) Reproduced from [117, Fig. 9.1]. The stress ellipsoid and principal stresses $\sigma_{I-III}$, which are independent of the sample axes ($S_{1-3}$).

the fully defined strain ellipsoid based on experimental measurements of the $d$ spacing in various orientations, which may be visualised as sections through a ‘$d$ spacing ellipsoid’. A minimum of two sections through the $d$ spacing ellipsoid or six point measurements are required from the same or different diffracting planes unless assumptions are made to simplify the problem [121]. This $d$ spacing ellipsoid is then converted to strain using the equation $(d - d_0)/d_0$, where $d$ is the lattice spacing calculated from the experimental data and $d_0$ is the spacing of the lattice when it is completely free of strains, both applied and residual. The unique stress ellipsoid of the element can then be calculated from the unique strain ellipsoid of the material element using the elastic stiffness tensor $C$ [91, p. 8114]. While $C$ is a 4th rank tensor, it may be reduced to a 6x6 matrix for materials [22] (shown for an isotropic material in Figure 2.2a).

For any given diffracting volume, there will be one stress and one strain ellipsoid [121], and multiple $d$ spacing ellipsoids (one for each crystallographic plane), not all of which will be recorded during the experiment. The experiential data shown in Figure 2.21 is effectively a section through several nested $d$ spacing ellipsoids all centered about the same point. Under the current set of assumptions, the stress,
strain, and $d$ spacing ellipsoids will be the same shape scaled proportionate to each other with the principal axes oriented identically relative to the sample axes, and centered at the origin. We assume the origin for the ellipsoids is identical to the center of the diffraction volume, which is essentially true in magnesium due to the low linear absorption coefficient, but would be a poor assumption in, for example, copper [21, p. 30,121].

### 2.5.5 Two Experimental Approaches

Two approaches to a diffraction experiment are shown as Figure 2.24, which schematically shows the resulting diffracting volumes and scattering vectors. When we conduct a $2\theta$ scanning diffraction measurement such as that shown in Figure 2.24a and locate
the diffraction peak maximum, we have located one point on the surface of a $d$ spacing ellipsoid relative to the sample axes, as shown schematically at the left of Figure 2.23. The requirement for a minimum of six point measurements [92] at multiple angles is in effect creating a series of point measurements in 3D space to which this ellipsoid may be fitted, and accuracy is increased by additional measurements [92, p.114]. The minimum number of required point measurements decreases to three if they are made along the principal axes of the ellipsoid, and many experimental works simplify measurements by assuming that these principal strain axes will be aligned with the macroscopic loading applied [25]. Winholtz and Krawitz [122] used neutron diffraction measurements of a hoop weld in steel to show that the principal stress directions were generally not aligned with the sample axes. This work provided strong evidence that assuming the principal axis directions align with the sample axes is a practice of questionable validity, at least in welds.

When we perform a low angle transmission experiment as shown in Figure 2.24b and measure sets of full diffracted Debye-Scherrer rings on the 2D detector we are examining one cross section of the $d$-spacing ellipsoid for each diffracting plane as shown schematically at the left of Figure 2.23. This ring will take the form of a circle, or if deviatoric strains are present, an ellipse. The plane on which the ellipsoid is sectioned will be a function of the incoming beam angle and the Bragg angle ($\theta_b$) for the diffracting plane from which the ellipse was generated. The section of the $d$ spacing ellipsoid measured is calculated from a cone with the apex at the origin, centered about the incoming beam axis, with half apex angle of $90-2\theta_b$ [117, p. 21], as shown in Figure 2.25a. It should be noted that while the ellipsoid is centered at the origin, there is no requirement for the resulting diffracted ellipse to be centered on the
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Figure 2.24: Adapted from [91, p. 8166], a) A 2θ type experiment. Note that the diffracting volume (in green) defined by the apertures (in black) does not extend through the sample, making a series of through thickness measurements possible. A point detector (triangle) is used to measure the diffracted intensity at several adjacent locations to define the curve shown below. b) Low angle transmission diffraction experiment, which uses a CCD area detector. Note that each diffracting plane will have a unique scattering vector Q.

beam, indeed this is unlikely to be the case unless a principal strain axis is aligned with the incoming beam. Consequently, there is no requirement for the diffracted rings to be concentric. This offset from the beam center will become increasingly evident as the Bragg angle increases, and the section of the ellipsoid moves away from the origin. For practical analysis purposes, if the goal is to extract strain, texture or chemical information, it is acceptable to center the ellipse data, however if the goal is to define a 3D stress ellipsoid, centering operations will invalidate the data.

As compared to the 2θ scanning method, low angle transmission diffraction has several advantages including higher measurement speeds and fewer potential measurement errors, as there is no rotation of the sample or changes in the length of the beam path which may cause changes in background intensity. The transmission approach also has the capability to determine the magnitude and orientation of strains relative
to the sample axes for multiple crystallographic planes with one measurement. Separating the effects which cause change in diameter of the ring (such as chemical and hydrostatic strains) from deviatoric residual strains can be easily done, as described below. One major disadvantage of the transmission approach is that changes in strain along the beam path cannot be measured. As the transmission approach was used for the current set of experiments, all further discussion will focus on this technique.

### 2.5.6 Determining a Complete Ellipsoid

To completely define any ellipsoid, a minimum of two independent sections must be made, although data quality is improved by additional sections [121]. One approach to achieving independent sections is to rotate the sample as shown in Figure 2.25b which has the major disadvantage of introducing new material into the sampling volume. This approach also has precise alignment requirements [121] negating some of the advantages of choosing the transmission diffraction approach over the $2\theta$ method. Alternately, parallel sections of the $d$ spacing ellipsoid may be sampled simultaneously by using multiple diffracting rings with different Bragg angles, as shown in Figure
2.25c. These rings come from the diffraction of different lattice planes, or higher order diffraction from the same plane (i.e. different values of n in the Bragg equation). For example, in the case of the (0002) plane in magnesium, second order diffraction from (0004) is frequently observed. While the same crystallographic plane (0002) is diffraeting in both cases, the scattering vector Q at which strain is being measured differs (see Figure 2.24), and these are thus independent strain measurements.

While the parallel section approach has the advantages listed above, in synchrotron work with short incoming beam wavelengths the resulting change in Bragg angles between diffraeting planes will be minor and the resulting \( d \) spacing ellipsoid sections will be close both to each other and to the \( d \) spacing ellipsoid origin, rendering this approach less feasible. In effect, multiple sections of the ellipsoid nearly through the origin perpendicular to the incoming beam will be made, and extrapolating the minor diameter changes to out-of-plane ellipsoid dimensions will result in high measurement error. This approach is therefore best suited for cases with wavelengths of 0.5-1.5 Å [121]. Alternatively, parallel sections may be made by changing the incoming beam wavelength which is likely the simplest approach but not available at all test facilities and outside the scope of the current work.

2.5.7 Deconvoluting Effects on \( d \) Spacing

The \( d \) spacing, and thus dimensions of the diffracting ellipsoid are a function of both the chemical composition and the strain state. Before calculating the strain ellipsoid, the chemical and strain effects must be deconvoluted. The strain state may be considered as the sum of the hydrostatic and deviatoric components. The mean of the principal strain values is the hydrostatic component, and non-directional, while
the variations from the mean with direction are the deviatoric component.

If there are only hydrostatic strains and chemical effects in a diffracting volume, the $d$ spacing ellipsoid will become a sphere and the diffracted Debye-Scherrer ring will form a circle on the detector. If the elastic strain has deviatoric components, cross sections of the $d$ spacing ellipsoid will be an ellipse with the long axis located parallel to the most tensile direction in-plane. The mean of the principal axis lengths of the $d$ spacing ellipsoid will give the radius of a sphere representing the hydrostatic and chemical effects on the $d$ spacing. By comparing the $d$ spacing ellipsoid radius to the sphere, we can identify locations where the ellipsoid is smaller than the sphere radius, and deviatoric strain is compressive. In the reverse situation deviatoric strain is tensile.

Similarly, even if the full $d$ spacing ellipsoid is unknown, the in-plane deviatoric strains may be calculated directly from the diffracted data. This is done by comparing the ellipse fitted to the diffracted points to a circle fit to the same points, shown graphically as Figure 2.26a. The circle represents the mean value of the $d$ spacing due to both chemical and in-plane hydrostatic effects, while the ellipse includes chemical, in-plane hydrostatic and in-plane deviatoric effects. The deviatoric strain components on the diffracting plane may then easily be calculated by comparing the ellipse to the circle, in other words the deviation from the average strain on the diffracting plane for all angles. At angles where the fitted ellipse radius is less than the circle radius, deviatoric strain is compressive and at angles where the ellipse is greater than the circle radius deviatoric strain is tensile, shown as Figure 2.26b. Looking at Figure 2.26, it is evident that the in-plane deviatoric strains will sum to zero. Note that regardless of the deviatoric strain on a plane, the absolute strain may be tensile.
or compressive depending on the state of the hydrostatic strain, which cannot be extracted without additional data.

Figure 2.26: Author’s work. a) Polar plot of the deviatoric strain, sample TD axis at 0°, sample ND axis at 90°. Blue dots represent transmission diffraction measurements from \{10\bar{1}0\} (note the radial axis (Å) expanded to show detail). The black line shows the fitted ellipse, the purple line the fitted circle. Dotted red lines show the ellipse major and minor axes. The circle represents the $d$ spacing effects of chemistry and in-plane hydrostatic strain, while the ellipse represents these plus the additional effect of in-plane deviatoric strains on $d$ spacing. Points where the ellipse extends outside the circle represent in-plane deviatoric tensile strains while points inside represent compressive strains. b) Plot of deviatoric residual strains in percent calculated from the circle and ellipse fits at left, tensile in blue, compressive in red.

To deconvolute the hydrostatic strain from the chemical effects requires creation of a separate strain-relieved sample which has been sectioned to relieve the macroscopic strain, from which values of $d_0$ the strain free lattice spacing [21, p.206] may be determined. The measured value of $d_0$ containing only chemical effects is compared to the radius of the circle fit to the diffracted data, which contains both hydrostatic and chemical effects. The difference between the two values is the in-plane hydrostatic strain component of that measurement location.
Complication #1 Anisotropic single crystal elastic properties

If a single crystal is truly isotropic, then the single and polycrystal properties will be identical and independent of texture and loading axis. However this is an unusual case, and most materials are anisotropic to some extent.

Given an anisotropic single crystal, the polycrystal may only behave in a more isotropic fashion [25, p. 438], because as the crystal orientations become more evenly distributed so do the material properties. If we assume a magnesium volume which has equal texture in all directions and a sufficiently large number of grains, the polycrystalline material element as a whole will behave in a fully isotropic fashion [91, p. 2412]. As described in Section 2.1.3, magnesium is nearly elastically isotropic as a single crystal, and therefore as a polycrystal will act in an even more isotropic manner.

To return to our ellipsoid visualisation, provided the texture is isotropically distributed and grains are small, changing the single crystal elastic properties from isotropic to anisotropic will not impact the macroscopic polycrystal stress and strain ellipsoids. The stress, strain, and \( d \) spacing ellipsoids for the polycrystal will continue to have the same principal axes and to be the same shape scaled proportionate to each other.

Complication #2 Intergranular effects of single crystal anisotropy

One consequence of single crystal elastic anisotropy is that even if the overall polycrystal response is isotropic, each grain of the material volume reacts slightly differently to the applied stress. This causes scatter in the elastic response of each grain [21], resulting in intergranular stresses, and fractionally different spacing in the lattice planes
between adjacent grains [116]. These intergranular stresses, and other stresses on the length scale of a few grains are referred to as type II microstresses, while stresses on length scales less than the grain size are referred to as type III microstresses [21].

Type II microstresses will not occur with a material that is completely isotropic, and increase in magnitude as the mechanical, chemical, and thermal anisotropy of the single crystal increases. If the size of the diffracting volume is greater than that of the grain size (as is the case for the current work), increased scatter in the distribution of type II stresses, or increases in the magnitude of type III stresses will result in broadening of the diffraction peak, which may be quantified by the FWHM (full width at half peak maximum, see Figure 2.24).

In contrast, if the average value of the type II stresses undergoes a consistent shift (i.e. an increase or decrease in the average value), it will be reflected in a shift of the diffraction peak center. Peak shifts are not possible with type III strains, because they equilibrate over a length scale far shorter than the diffraction volume.

In summary, peak broadening may be caused by the following effects:

- compositional heterogeneity [92, p.80]
- intergranular effects from elastic [92, p.80] and plastic [21] anisotropy
- intergranular effects from thermal anisotropy [92, p.80]
- intergranular effects from chemical anisotropy
- plastic misfits [92, p.80] such as those around coherent particles
- size of the diffracting domains [92, p.80]
- type III strains (within grains) including dislocations [92, p.80]
- instrumental broadening [92, p.80]

Experimentally, peak broadening increases in FWHM are observed as an increase
in the thickness of the diffracted rings for transmission experiments, and increase in the diffraction peak width for $2\theta$ type experiments. In terms of our previously discussed visualisation, FWHM increases will cause the ‘surface’ of the diffracting ellipsoid to become thicker.

**Complication #3 Texture**

Highly textured samples will have ‘gaps’ in the surface of the $d$ spacing ellipsoid where there is insufficient material oriented to meet the diffraction condition and locate the ellipsoid surface. Accounting for these gaps on the varying crystallographic planes is a critical part of experimental setup for $2\theta$ type diffraction measurements, and can pose significant measurement difficulties [97]. However, small angle transmission experiments are mostly insensitive to gaps in the Debye-Scherrer ring, as the entire 2D ellipse on the detector is fitted simultaneously [124], making the fit an over defined problem mathematically. In the rare case where an entire ring has insufficient diffracted intensity, post-processing analysis may be switched to another ring on the detector since several are usually captured simultaneously.

In cases of a perfectly elastically isotropic material, a preferred orientation will have no impact, save on the angles at which measurements may be made experimentally. However, while it is more convenient to work with isotropic assumptions, in truth all single crystals are anisotropic [91, p. 2411], and so all polycrystals are as well, to a degree which depends on the texture of the material [91, p. 2411]. Given that perfectly uniform textures in HCP materials are difficult to achieve, in many cases the sample will have at least some degree of anisotropy.

For a material element which is both elastically anisotropic and textured, the
polycrystal elastic stiffness tensor $C$ is now dependent on the texture of the material element. To calculate local values of the stiffness tensor, which are required if accurate conversions between stress and strain are to be made, a full texture must be known for each measurement point. This complicates the experiment significantly because neither small angle transmission nor $2\theta$ type diffraction measurements are well suited to generate a complete texture which is a necessary input to this calculation. If both the texture and the single crystal tensor are known, the polycrystal elastic stiffness tensor may be calculated computationally [23].

Referring back to our ellipsoid visualisation, if the material is both anisotropic and has a preferred orientation, the principal axes of the stress ellipsoid and the strain ellipsoid may now potentially be different [121]. To determine if this is the case, examine the polycrystal elastic stiffness tensor. If any of the components which were zero in Figure 2.2b have become non-zero, the principal axes between the polycrystal stress and strain ellipsoids will no longer be identical. An example of this case is shown as Equation 2.2, which was calculated by combining the anisotropic Mg stiffness tensor of Figure 2.2a with an experimentally measured texture using the Hill method [21]. Note that normal strains ($\varepsilon_{1-3}$) will now result in shear stresses ($\sigma_{4-6}$), which was not the case with the either the isotropic tensor shown in Figure 2.2d or the anisotropic HCP tensor shown in Figure 2.2c. However, all components which were originally zero are all far smaller than the original HCP stiffness tensor, indicating this effect is minor. The principal axes of the strain and $d$ spacing ellipsoids will however remain identical.
When combined with strong texture, single crystal anisotropic effects (such as elastic, chemical, or thermal) will cause even a sample free of residual strains to show behaviour consistent with the presence of deviatoric residual strains. Separating these effects from those caused by residual strains is a complex process which would require point-by-point complete texture measurements.

In a material with low elastic anisotropy such as magnesium, or one with a random texture this will be an insignificant effect provided strain remains below the elastic limit.

Figure 2.27: Adapted from Gharghouri [26, p. 61]. Strain on two crystallographic planes in AZ80 under monotonic tension. Solid line indicates loading, dashed line indicates unloading. Note that plastic strains are first measured around 100 MPa, shown in the deviation from linearity.
2.6. DIRECTION OF STUDY

Complication #4 Plastic strain

Plastic strains do not directly result in changes in the lattice spacing [91, p. 8161]. Figure 2.27 shows that for AZ80 the lattice spacing for both planes increases linearly with the applied stress until the yield strength of the least stiff (0002) plane is reached. At this point the (0002) plane deforms plastically, and the load of this plane is shifted onto the stiffer {10\bar{1}0} planes. Note that although the single crystal elastic stiffness of the (0002) plane is greater than that of the {10\bar{1}0} plane, this particular test was conducted in a polycrystal with the tensile load applied perpendicular to the majority of the (0002) planes [26]. The results shown here will change with loading direction due to changes in the resolved stress on the crystallographic planes of the diffracting volume, as may be seen in the original work of [26].

The presence and arrangement of high numbers of dislocations produced during plastic deformation do however disrupt the crystal lattice as Type III strains which may cause diffraction peak broadening, as mentioned briefly in Section 2.5.7.

Once the elastic limit is exceeded, the 3D functions which represent the stress and strain are no longer simple ellipsoids [121], and the relationship between the stress and the strain becomes more complex.

2.6 Direction of Study

Mishra and Ma [6] state in their notable review paper that the technology of friction stir welding has outpaced the fundamental understanding of the microstructure-property relationships involved. Although published over a decade ago, this still remains true as friction stir welding is extended into increasingly complex applications. The focus of the current work is on improving this fundamental understanding
2.6. DIRECTION OF STUDY

by investigating the connections between the asymmetry of the texture and residual strain distribution in a friction stir weld, and the resulting impacts on the highly localised deformation process.

Despite the idealised view of the texture distribution in a friction stir weld (e.g. [13]), the process of forming a friction stir weld is inherently asymmetric. Evidence of this asymmetry generally comes from line scans with point measurements widely spaced with respect to the abrupt changes in structure that occur at the friction stir weld interfaces (e.g. [77]). Deformation in friction stir welds has also been shown to be asymmetric (e.g. [109]) with failure occurring preferentially at advancing side.

To truly understand this behaviour, we must fill many gaps in the literature with regards to the two dimensional spatial distribution of texture. Similarly, we must enhance our understanding of the magnitude and distribution of residual strains spanning a friction stir weld nugget, and understand how these are affected by changes to the processing parameters.

Once our understanding of the microstructure is improved, we must better understand the impact of these microstructural changes in terms of physical deformation of similar welds before finally investigating the area of application, dissimilar welds.
Chapter 3

Influence of Magnesium AZ80 Friction Stir Weld texture on Tensile Strain Localisation

Previously published as [8], with notes added for later thesis version marked with an (*)

J. Hiscocks, B. J. Diak, A. P. Gerlich and M. R. Daymond

Abstract

Synchrotron diffraction was used to construct the first 2D texture maps of entire magnesium AZ80 friction stir welds and showed that basal slip is favoured along most of the advancing side interface, and to a lesser extent on the retreating side interface. Zones of grains optimally oriented for basal slip are known to be a major contributor to strain localisation leading to failure during transverse tensile tests. Profilometry results confirm that the basal plane orientation dominates strain localisation. Micro-texture maps traversing the weld interfaces were used to describe the material flow within the weld using scatter from the ideal shear texture fibre. The current results are highly applicable to modelling the strength and ductility of these joints under transverse loading.
3.1. INTRODUCTION

Friction stir welding (FSW) is a solid state process in which a non-consumable tool consisting of a wider shoulder and a narrower pin is rapidly rotated and inserted into the workpiece causing the contacted material to soften from frictional heating. The tool shears this softened material around the pin and traverses the workpiece at a selected welding speed leaving a seam of hot worked material behind. Heat input to the weld increases with the rate of tool rotation and decreases with increasing welding speed, and so these factors have a direct impact on the microstructure, texture, and mechanical properties of the weld. On one side of the weld the material flowing around the tool is moving in the opposite direction to the base material and creates a sharp advancing side (AS) interface, while at the other a more diffuse retreating side (RS) interface is created. This interface separates the recrystallised [6] magnesium alloy of the nugget from the thermomechanically affected zone (TMAZ) that surrounds it.

Compared to aluminum alloys, magnesium offers the potential for greater weight reduction in exchange for reduced mechanical properties. The magnesium-aluminum-zinc (AZ) alloy system used in this work is currently in industrial use and thus a good subject for FSW development that will expand applications. Within this alloy series, higher alloy content materials such as Mg AZ80 offer age hardening capability and greater tensile and compressive strength in exchange for reduced ductility [34]. In comparison to Mg AZ91 which has the highest alloy content in this series and is cast, Mg AZ80 may be forged or extruded resulting in both greater strength and ductility.
3.1. INTRODUCTION

For joining of magnesium alloys FSW is superior to fusion welding techniques and results in a finer microstructure [10], lower residual stress [11], greater energy absorption on impact [12] and generally better mechanical properties [13, 14]. However there is still usually degradation of mechanical properties as compared to the base material, in particular ductility. Yang et al. [9] found that even in the most ductile of their FSW joints, the elongation to failure was reduced by about half as compared to the parent material. This is due to the strong texture developed, which when combined with the highly anisotropic nature of magnesium causes inhomogeneous deformation, strain localisation, and failure [111]. In deformation of magnesium at room temperature the critical resolved shear stress to activate basal slip is far lower than either prismatic (the next softest slip system), or extension twinning [28] and thus the distribution of basal planes is of primary importance, but has only been reported in small isolated areas of a FSW.

The idealised view of the FSW nugget texture generally described is that of basal (0001) planes parallel to a truncated cone centered on the tool path, while the prismatic {1010} planes are randomly distributed perpendicular to this [13, 72]. In this simplified texture description, basal planes are optimally oriented for slip at the sides of the weld nugget along the interfaces between the weld and the base material, leading to a tendency for strain localisation and failure to occur here [109, 110, 77, 111]. However, this idealised texture description is lacking, as evidence of asymmetry of the weld texture exists both in measurements [77, 13], and in the tendency for FSW in magnesium to preferentially fracture at the AS interface [111, 109]. Liu et al. [107] have reported that for an entire weld loaded in the transverse direction, fracture
3.1. INTRODUCTION

initiates at an inflection point on the AS interface where there is a triple junction between the flow that forms the shoulder, the nugget, and the TMAZ. Liu et al. [125] attributed failure at this inflection point to the local texture changes. Liu et al. [126] investigated the behaviour of samples where this triple junction was removed, and found that failure instead initiated at the RS interface. This was attributed to a disparity in texture between the AS and RS interfaces resulting in asymmetrical strain localisation. While these works link localised texture change and fracture, inflection of the AS interface is not a commonly reported feature and changes in material flow redistribute the texture. Other locations of abrupt texture transition may also exist, localising deformation and leading to failure.

The aim of the current work is to evaluate the asymmetrical features of texture across a FSW and relate this to the mechanical performance under transverse loads. Of specific interest is to quantify the extent and distribution of basal planes oriented for easy slip across an entire weld, which will allow for new insights into strain localisation. To this end, synchrotron examination was used which allows measurement over a much larger area than electron back-scattered diffraction (EBSD), with finer spatial resolution than either lab X-ray or neutron diffraction. This unprecedented coverage of a complete weld permits us to determine changes in texture between each microstructural region for a range of processing conditions. The resulting 2D texture maps show for the first time the distribution of all basal planes optimally oriented for slip across the entire cross section of a weld. This work was supplemented with multiple EBSD scans across the interfaces of the weld where texture transition occurs which also allowed insight into the material flow paths in the weld. To identify the distribution and severity of strain localisation interrupted tensile tests were performed,
3.2 EXPERIMENTAL PROCEDURE

and profilometry scans of the resulting samples were made.

3.2 Experimental procedure

A total of 12 friction stir welds 100 mm long were created under four processing conditions on an extruded sheet of Mg AZ80 oriented perpendicular to the sheet extrusion direction (ED). The four processing conditions used and the resulting relative heat inputs are shown in Table 3.1. All welds were performed on the surface of a continuous plate to remove potential issues such as weld line gaps and clamping variations. The extruded sheet was 6.3 mm thick by 116 mm wide by 287 mm extruded length manufactured by Morgo Magnesium, with composition in weight percentage of 7.75 Al, 0.65 Zn, 0.14 Mn, 0.09 Si, 0.005 Fe and balance Mg.

Table 3.1: Bead on plate FSW processing conditions. Heat input is ranked from 1 (greatest) to 4 (least).

<table>
<thead>
<tr>
<th>Welding speed (mm min$^{-1}$)</th>
<th>rev min$^{-1}$</th>
<th>Advance per rotation (mm)</th>
<th>Heat input</th>
</tr>
</thead>
<tbody>
<tr>
<td>88</td>
<td>820</td>
<td>0.107</td>
<td>1</td>
</tr>
<tr>
<td>88</td>
<td>583</td>
<td>0.151</td>
<td>2</td>
</tr>
<tr>
<td>148</td>
<td>583</td>
<td>0.254</td>
<td>3</td>
</tr>
<tr>
<td>248</td>
<td>583</td>
<td>0.425</td>
<td>4</td>
</tr>
</tbody>
</table>

All welds were made on a JAFO model FWR40J milling machine using a tool with a 10 mm diameter shoulder and 4 mm diameter cylindrical threaded pin 2.2 mm long. The tool threading was M4, and the direction of rotation was counter-clockwise as viewed from above. The tool was inclined to an angle of 2.5° from the vertical away from the welding direction (WD), which aids in retention and movement of plasticized material by the shoulder around the tool [6].

All weld cross sections were examined optically in the plane 30 mm from the weld
3.2. EXPERIMENTAL PROCEDURE

start. Samples were cold mounted in epoxy, ground, and polished with diamond paste lubricated with oil or 3:1 ethanol:glycerol. The diamond paste abrasives of sizes 9, 3 and 1 $\mu m$ on napless cloth were followed by a final polish with colloidal silica on a napped pad. Etching to reveal the material flow patterns was done by immersion in a solution of 1 g tartaric acid and 20 mL $H_2O$ for up to 20 s. Images were captured digitally and combined using the GIMP and Enblend software tools to give the results shown in Figure 3.3. The GIMP image software was then used to trace outlines of all 12 welds, as shown in Figure 3.4. Base material microscopy was conducted on three perpendicular planes with similar preparation procedures and a 1 mL acetic acid: 9 mL $H_2O$ etch for 5 s to reveal grain boundaries. Average grain size in the normal-transverse plane was measured from optical micrographs using the ImageJ [127] software.

A total of six transverse weld and three base material tensile samples were tested. As the WD was perpendicular to the ED the long tensile axis was parallel to the ED as shown in Figure 3.1. Base material samples were cut in the same orientation from the same plate outside of the welded area.

![Figure 3.1: Dimensions of a typical transverse weld tensile sample in mm. Sheet extrusion direction (ED) and FSW direction shown (WD).](image)

After cutting, the gauge section was manually polished and etched with the tartaric acid solution previously described to reveal the weld profile. The weld top surface was ground to remove projecting flash and weld grooves, following which the
3.2. EXPERIMENTAL PROCEDURE

opposite surface was ground away until it corresponded with the root of the weld. As the welds had slightly different depths (see Figure 3.4) this resulted in a slightly different thickness for each sample. A random speckle pattern was then applied over the gauge length to facilitate optical strain measurements. All transverse tensile tests were performed at room temperature on an Instron 8502 servohydraulic frame with a 250 kN load cell at a crosshead speed of $5.9 \times 10^{-3}$ mm s$^{-1}$ until failure. Strain data was collected by digitally imaging the sample at 5 second intervals and using the Istra 4D strain processing software. Following testing, the surface profiles across the gauge length of samples which failed in the grips were measured using a Keyence VHX-2000 digital microscope at a spatial resolution of $1.05 \mu m^2$ and a height resolution of approximately $\pm 5 \mu m$.

Figure 3.2: a) Schematic of transmission geometry based on Randle and Engler [113, pp. 196]. The direct beam is not shown. b) Diffraction pattern from the stir zone of a weld. The inner ring represents the prismatic planes while the next larger represents the basal planes from which the graphs in Figure 3.10 were generated.

Chinese

EBSD specimens were prepared from optically polished samples by ion milling
3.2. EXPERIMENTAL PROCEDURE

in an Hitachi IM4000 using Argon gas and an acceleration voltage of 4.5 kV with the beam inclined 3° to the surface for 30 minutes at 25 rev min\(^{-1}\). EBSD scans were performed in an FEI nanoSEM with a Bruker e-flash detector at an accelerating voltage of 20 kV and data analysis was done with the MTEX software module for MATLAB [23]. The interface scans shown in Figure 3.8 cover a minimum of 50 \( \times \) 1000 \( \mu \text{m}^2 \), and each scan was divided into a series of pole figures covering an area approximately 100 \( \mu \text{m} \) long to show the transition in texture from the nugget to the TMAZ. Grain size of the nugget center was calculated from area scans with a measurement spacing of 0.5-0.37 \( \mu \text{m}^2 \) covering a minimum of 3000 grains. Grain boundaries were defined as a misorientation of 10° or greater, and areas consisting of only one measurement point were omitted from these calculations.

Hardness testing was conducted using a MicroMaterials Vantage Nanotest platform with a Berkovich indenter. Following EBSD examination the samples were immersion etched for 10 s in a solution of 2 % nitric acid and 98 % anhydrous ethanol. The indenter force was increased to a maximum of 500 mN over 5 s, held for a dwell time of 15 s, unloaded over 1 s, and the resulting hardness values calculated. Residual indents were examined under an optical microscope and any data from non-uniform impressions was discarded.

Monochromatic beam transmission mode synchrotron experiments were conducted on the 1-ID beam line at the Advanced Photon Source, Argonne National Laboratory. Data was collected using a charge coupled device (CCD) as shown in Figure 3.2. This experimental setup is suitable for mapping samples that display variation in two dimensions without through-thickness variation. Transverse sections of the three welds with the greatest heat input were scanned in a grid pattern with 0.1 mm
3.2. EXPERIMENTAL PROCEDURE

Figure 3.3: Friction stir welds shown from greatest heat input (top) to least (bottom). AS to the left. Voids are present at the base of the lowest weld.

horizontal and 0.1-0.2 mm vertical spacing for a minimum of 2000 points per sample. Sample thicknesses were approximately 7.5 mm, with a beam defined by slits to an incident size of 0.05 x 0.05 mm² and a wavelength of 0.14410 Å. It should be noted
3.2. EXPERIMENTAL PROCEDURE

Figure 3.4: Comparison of weld profiles produced under four processing conditions. Colour coded with red as the condition of greatest heat input and blue the least.

that X-rays are scattered through the weld thickness, and thus the diffraction information from each point is an average of the entire $7.5 \times 0.05 \times 0.05 \ mm^3$ diffraction volume. Only those crystallographic planes oriented with the Bragg angle relative to the transmitted beam diffract X-rays [92] resulting in a series of concentric cones, each representing one lattice plane of the diffraction volume. When the diffracted X-rays contact the detector a series of concentric circles or ellipses called Debye-Scherrer rings are recorded from which information about the diffracting volume can be extracted [113] as shown in Figure 3.2. Texture information is represented by the distribution of intensity around the azimuth of the ring, with a randomly textured sample generating an even intensity and samples with a preferred texture showing unequal distribution in some directions. A complete texture cannot be readily determined by this method as any crystallographic planes not at the Bragg angle with respect to the beam will not diffract [113] and thus the vast majority of texture information is not gathered. In the case of the (0002) basal plane, the Bragg angle is $1.6^\circ$, and thus only the grain volumes with basal poles nearly in the ND-TD plane of the sample as per Figure 3.2 will diffract. Effectively this experimental geometry allows for detection of basal
3.3. RESULTS

planes optimally oriented for slip under transverse loading, which is taken advantage of in this work.

3.3 Results

The base material was found to have an equiaxed grain size of 35.4 $\mu m^2$, and an inhomogeneous microstructure with bands of both precipitates and other local compositional variation (as determined from local etching behaviour) extending in the ED. Identical features have been reported in other samples of extruded AZ80 by Yang et al. [9] who found bands of the $\beta$ phase precipitates as well as bands of locally elevated Al content.

Transverse weld micrographs are shown as Figure 3.3, with the dark banding of precipitates in the base material indicating the ED of the plate. Darker coloration in some areas of the nugget is due to regions or bands of finer grains which are more extensive in the lower heat input welds. Onion-ring type structures (*resulting from periodic microstructural changes along the weld length) were not observed under any processing condition. The lowest heat input condition produced voids at the root of the weld while no evidence of defects was found in any of the other welds. This type of defect is attributed to insufficient heat input resulting in insufficient material flow [49], and a similar pattern of void type defects in hot rolled Mg AZ31 at low heat inputs has been reported [42].

Traced outlines of the interface between the nugget and the TMAZ were made for all 12 welds and are shown as Figure 3.4. Welds with identical heat inputs were found to have similar profiles, but as heat input increased the welds increased in width and depth. The transverse weld images shown have a common basin-shaped profile and
Table 3.2: Summary of weld tensile test outcomes. Note the UTS, and reduction in area (RA) values for the samples fractured in the grips will be conservative.

<table>
<thead>
<tr>
<th>Heat input</th>
<th>0.2% Yield (MPa)</th>
<th>UTS (MPa)</th>
<th>RA</th>
<th>Fracture Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>203</td>
<td>312</td>
<td>17%</td>
<td>Fail at AS</td>
</tr>
<tr>
<td>1</td>
<td>187</td>
<td>306</td>
<td>19%</td>
<td>Fail in grips after yield</td>
</tr>
<tr>
<td>2</td>
<td>230</td>
<td>310</td>
<td>22%</td>
<td>Fail at AS</td>
</tr>
<tr>
<td>2</td>
<td>165</td>
<td>293</td>
<td>24%</td>
<td>Fail at AS</td>
</tr>
<tr>
<td>3</td>
<td>186</td>
<td>281</td>
<td>25%</td>
<td>Fail at AS</td>
</tr>
<tr>
<td>4</td>
<td>173</td>
<td>234</td>
<td>15%</td>
<td>Fail in grips after yield</td>
</tr>
</tbody>
</table>

increases to the heat input from decreasing the welding speed and increasing the rev min\(^{-1}\) were not found to change the overall profile morphology. This contrasts with the results of Yang et al. [9] where a similar range of conditions caused a transition from basin shape to round nugget, demonstrating the greater heat input from using a tapered FSW tool pin. Inflections of the AS weld interface such as those reported by Liu et al. [107] were not present.

Engineering stress-strain tensile curves for the base metal and welds are shown in Figure 3.5, and further details of the weld results are shown in Table 3.2. For the extruded base material the average 0.2 % offset yield and ultimate tensile strength (UTS) were calculated to be 246 and 356 MPa respectively, with an average area reduction of 36 % and elongation to failure of 18 %, similar to published literature values [2]. In all cases tested, FSW decreased the tensile performance relative to the base material, and results were close to those produced by Yang et al. [9] following similar processing. As the weld heat input increased, the transverse tensile UTS of the weld increased from 65 % to 87 % of the base material while the yield stress showed much more scatter in the results.
3.3. RESULTS

Figure 3.5: Engineering stress-strain curves of transverse tensile properties. Left, 3 base material tests; right, 6 FSW tests. (*In the left plot, unloading and reloading of one of the base material samples is visible due to temporary equipment failure. In the right plot, the 148 mm min$^{-1}$ and 583 (Heat input 3) sample has an extremely steep loading curve which is probably due to sample curvature stemming from high residual stresses. See Chapter 6 for details.

Figure 3.6: Colorised WD height map of a FSW made at 88 mm min$^{-1}$ and 820 rev min$^{-1}$ (heat input 1) following tensile testing to a stress of 306 MPa. Height ranges from a minimum value of 73 $\mu$m near the RS interface to a peak height of 188 $\mu$m in the weld nugget as indicated with a triangle mark. Fracture has initiated at the AS interface as marked by an X. Weld profile outlined, AS at left, RS at right.

Of the six weld samples tested, four failed along the interface between the AS of the weld and the base material following contraction in the WD at both interfaces. This is confirmation that for this tool geometry the AS of the FSW joint is the most probable fracture location. The remaining samples failed in the grips, indicating insufficient
grip length. Figure 3.6 is a surface profile from one of these samples, which shows that contraction along the WD was inhomogeneous and almost completely confined to the interior of the weld nugget. This contraction was most extreme at the RS where it was spread over a large area as compared to the AS where contraction of a slightly lesser extent occurred in a narrow band aligned with the AS interface. This localised contraction is a key contributing factor to failure which was observed to initiate along the AS interface (see location marked on Figure 3.6), in the same location as reported by Liu et al. [107] and Liu et al. [125].

Hardness results for Figure 3.7 are presented for two different processing conditions and show typical scatter in measurement due to the inhomogeneous nature of the material. For both samples the weld was found to be more homogeneous and have a higher average hardness than the base material which had an average hardness of 1.13 GPa. The 88 mm min$^{-1}$ and 820 rev min$^{-1}$ (highest heat input) sample had a greater average hardness of 1.18 GPa as compared to the 148 mm min$^{-1}$ and 583 (Heat input 3) sample with 1.15 GPa. The distribution of high and low hardness zones within the nuggets of the two samples are not similar, and heat effected zone softening around the nugget is not evident in either case.

Figure 3.7: Hardness values across welds made at left, 88 mm min$^{-1}$ and 820 rev min$^{-1}$ (highest heat input) sample; right, 148 mm min$^{-1}$ and 583 (Heat input 3) sample. Weld profile outlined, AS at left, RS at right.

In all cases there was significant grain refinement as compared to the base material.
The grain sizes were found to be consistent with the values measured in Mg AZ31 by Chang et al. [67] and Razal Rose et al. in Mg AZ61 [128] and increased monotonically with heat input. These results are in agreement with the work of Chang et al. [67] in magnesium who showed the recrystallised grain size to increase with the peak FSW temperature, a trend which has also been reported in aluminum by various authors [6].

To the authors’ knowledge the results published here are the first detailed measurements of how texture changes across multiple interfaces of a FSW in magnesium, and the first texture measurements of any type in FSW of Mg AZ80. While multiple interface scans were taken for each of three welding conditions, only one set of EBSD measurements is presented here, as all samples were found to be similar. Comparing Figure 3.8 to the measurements by Park et al. [13] in FSW of extruded Mg AZ61, the textures are consistent both in the base metal and at all areas inside the nugget showing basal planes located on an ellipsoidal flow surface with the prismatic planes distributed almost randomly at 90° to these. However, in Figure 3.8 there is an additional texture component in each (0002) pole figure from the nugget areas which appears as a diffuse arc of texture attached to the region of peak intensity. Comparing scan 1 to scan 4, and scan 3 to scan 5, it is evident these diffuse arcs are mirror images on either side of the nugget. In addition scan 5 exhibits one location (indicated by the red box) in which two variants of the usual nugget texture overlap, clearly forming an ‘x’ pattern in the {10$ar{1}$0} pole figures at the RS interface. This component was only observed in the sample processed at 148 mm min$^{-1}$ and 583 rev min$^{-1}$ (heat input 3). To the authors’ knowledge, neither of these two components has been previously reported.
Figure 3.8: Transverse-normal section of a FSW made at 148 mm min$^{-1}$ and 583 rev min$^{-1}$ (heat input 3) showing EBSD scan areas marked in blue. Each scan area was divided into a series of basal (0002) and prismatic \{10\bar{1}0\} pole figures showing transitions in texture across the interface between the nugget and the TMAZ. For scans 1-5, basal pole figure series is on top with the prismatic on the bottom, while for scan 6 basal is at the left and prismatic at the right. The pole figure overlaid on the weld nugget shows the basal texture of the blue box immediately below. AS at left, RS at right. For information about the red box, see text.

For each synchrotron measurement point the software Fit2D \cite{129, 130} was used to measure the azimuthal intensity distribution of the selected Debye-Scherrer ring
for 36 angular segments of 10° each. From these 36 segments the angle and magnitude of maximum intensity were used to calculate a vector, and the resulting grid of normalised vectors was plotted to create what is effectively the relative intensity and orientation of the basal poles as measured in the plane of the sample surface. The current results are the first 2D maps showing how the basal poles optimally oriented for slip are distributed across the complete span of a FSW joint, and how this distribution changes with processing.

Figure 3.10 clearly shows that the texture is not symmetric, confirming indications from previously published line scans e.g. [77, 13]. Comparison of the AS and RS interfaces shows that while there is a continuous reorientation of texture extending over 0.7 mm at the AS interface, the texture change across the RS interface is more abrupt and the intensity in the weld is greater, indicating the basal poles are more aligned in the ND-TD plane near this interface. The lack of symmetry in texture is an indication that material flow differs from the ideal ellipsoidal flow surface commonly portrayed. In all cases the texture was found to vary through the depth of the weld which is consistent with numerous models and experimental results c.f. [49, 131] which have shown that the material flow path also varies through the thickness of the weld.

The synchrotron texture results also show variation with processing conditions, particularly between the highest heat input sample and the others which is consistent with other works showing texture dependence on welding parameters [46]. The most inhomogeneous of the synchrotron textures in the weld nugget area is the highest heat input condition of 88 mm min$^{-1}$ 820 rev min$^{-1}$ (Figure 3.10). This corresponds well with the lobes in the nugget visible in Figure 3.3 and likely represents a change
in material flow path and the start of a corresponding change in the weld profile morphology. Comparing the three processing conditions shown in Figure 3.10, the texture of the weld created at 88 mm min$^{-1}$ 583 rev min$^{-1}$ (heat input 2) shows greater similarity to the weld created at 148 mm min$^{-1}$ 583 rev min$^{-1}$ (heat input 3) than to the weld created at 88 mm min$^{-1}$ 820 rev min$^{-1}$ (highest heat input).
3.4 Discussion

Onion ring structures have been previously reported in FSW of Mg AZ80 by Borle et al. [45] where a cylindrical tool and greater heat input than the current FSW
conditions were used, as well as by Yang et al. [9] where similar parameters to those of the current study and a conical threaded tool were used. In both cases, the onion ring structure was attributed to $\beta$ phase that dissolved during FSW. As it has been reported that a cylindrical tool will result in a decreased FSW nugget temperature as compared to a tapered pin [63] it is probable that the current combination of tool and welding parameters generated insufficient heat to dissolve the $\beta$ phase and create this structure.

In the current work, the maximum UTS resulted from the greatest heat input condition, and it is possible that further increases in heat input would result in additional increase in the UTS. However Yang et al. [9] have reported that in Mg AZ80 welds, the UTS increased with increasing rotation rate and then began to decrease. Work such as that by Razal Rose et al. [128] has shown that while the energy input from the FSW tool to the weld material increases monotonically as the welding speed decreases, the material temperature around the tool does not. Similar behaviours in other alloys have also been noted, for example Nakata [12] found that the tensile shear strength of an Mg AZ31 lap joint increased to a peak before decreasing as the dwell time increased.

These observations suggest that the maximum temperature in the weld nugget increases with heat input until the $\beta$ solidus is reached and this phase liquefies causing a decrease in shear forces during welding, and a corresponding decrease in temperature [45]. This liquid film has a negative impact on mechanical properties [9] and avoiding its formation becomes a more significant problem in this alloy series as increasing aluminum content causes corresponding increases in the $\beta$ precipitate volume fraction. As this volume fraction increases, the range of FSW processing parameters for quality
welds decreases \[12\] and the heat generated at any given processing condition reduces \[7, 45\].

This also suggests that measuring the temperature at the welding probe tip while altering the FSW parameters and selecting those that produce the highest welding temperature could prove a suitable method of determining the maximum UTS possible for a given alloy and tool geometry.

As the current hardness measurements are a full 2D map instead of the more common line profile they can be better compared to the synchrotron textures and EBSD measurements. It can be concluded that with the current test procedures texture does not correlate with hardness, as in multiple locations such as the base of the welds the texture is similar but the hardnesses are different. In addition, for locations near the base of the weld such as Figure 3.8, midway through Scan 6, basal slip would be favoured upon through thickness compression yet lower hardness values are not present.

Neither the tensile yield strengths nor the hardnesses act in accordance with the Hall-Petch equation as the higher heat input samples had both greater nugget grain size, higher yield strengths, and higher hardnesses than the lower heat input welds. This is strong evidence that grain boundary-dislocation interaction is not the dominant strengthening mechanism in the current study. The opposite result has been reported by Afrin et al. [108] and Chowdhury et al. [132] in AZ31, which may be attributable to the higher aluminum content and volume fraction of $\beta$ precipitate in the current welds.

In the current work, no twins due to FSW with the characteristic lenticular morphology were detected in the EBSD scans. In some locations, twin-like relationships
3.4. DISCUSSION

between adjacent grains were measured, which may indicate that twins formed during the welding process develop into independent grains due to deformation and grain growth during FSW. In Figure 3.8, all the (0002) nugget pole figures show a ‘comet & tail’ pattern consisting of a high intensity texture peak connected to a lower intensity texture arc. Rotating the texture results to align with the predicted flow of material around the tool based on the weld diameter and interface angle measured optically as per the procedure discussed by Fonda and Knipling [73] results in the revised set of pole figures shown as Figure 3.9. The high intensity portion of the nugget textures matches the $B$ fiber [73] which forms as a result of basal slip in response to the dominant shear force of the material flowing around the pin, and has been reported numerous times in literature (e.g. [13]). With this $B$ fiber now centered in the pole figures, the lower intensity arcs of texture become linear features representing the scatter in the shear forces, and thus the material flow path near the interfaces.

As material is laid down behind the tool, the direction of flow will be from the RS to the AS, or towards the left in the case of the figures shown, allowing construction of a vector representing the flow direction based on the shear scatter, several of which are shown as white arrows in Figure 3.9. The current findings are in agreement with the work of Chen et al. [51] who used the location of voids in an AA5083 FSW to locate the junctions between material flow paths. They reported a swirl zone near the root of the weld with material flow towards the AS and upwards (see line 6 vectors) located underneath the nugget zone where material flowed mostly to the AS, but also slightly downwards near the interface to meet the swirl zone (see line 3 vectors). They found voids at the intersection of these two flow paths where laps or voids were found in the two lowest heat input conditions of the current work, indicated by the
two red ‘x’. They also reported a void located at the triple junction between the nugget and the shoulder flow which was not observed in the current work, but may indicate slightly better flow or increased strain in the current welds. The current analysis procedure offers new methods for analysing the flow in a magnesium FSW and provides both different data from that accessible via marker insert studies and a new avenue to validate computational flow models. It should be noted that the current analysis is particularly suitable for lower symmetry materials such as HCP, but as it is based on EBSD data, any sort of periodic variation along the length of the weld will not be captured.

In addition to the standard nugget $B$ fiber, the pole figures marked with a red box in Figures 3.8 have a second texture component. Following rotation to the local plane of shear in Figure 3.9, this additional component can be seen to be similar in orientation to the base material. This implies that in this area some base material was rotated to the interior of the weld alongside the sheared material without itself undergoing sufficient forces to reorient it. Support for this theory can be found in the work of Fonda et al. [58] who presented optical results showing that in AA2195 at similar depths to those used here some drawing of material from the TMAZ into the nugget occurred near the RS interface.

Synchrotron textures gathered by the experimental geometry used here cannot be reconstructed into full orientation data as no out of plane textures are measured. Within this restriction, the synchrotron results of Figure 3.10 agree well with the EBSD scans shown in Figure 3.8.

As basal slip is the most easily activated slip mode at room temperature [28] and the greatest stress will be resolved onto basal planes with $45^\circ$ to the tensile axis [20]
it is of particular interest to visualise these easy basal slip regions, and determine how
the distribution varies with FSW parameters.

Figure 3.11: Transverse-normal vector plot of the [0002] direction and magnitude
(line length) of maximum intensity of those vectors at 40-50° to the
transverse axis. Highest heat input condition (88 mm min⁻¹ 820 rev
min⁻¹) is in red, heat input 2 (88 mm min⁻¹ 583 rev min⁻¹) is in
orange, and heat input 3 (148 mm min⁻¹ 583 rev min⁻¹) is in light
blue, AS at left, RS at right.

For Figure 3.11 all texture measurements without an angle between the basal
poles and the weld transverse direction of 40-50° were hidden and the three processing
conditions of Figure 3.10 were overlaid. The three conditions have strong similarities,
and the common features generally move outwards as the weld heat input increases,
reflecting the changes shown in Figure 3.4. Comparing Figure 3.11 to Figure 3.6,
it is evident that the zones where basal slip is favoured are well aligned with the
areas where contraction strain was most significant. As a result, the region of easy
basal slip distributed linearly along the AS and macroscopically oriented at 45° to
the tensile force has resulted in a narrow band of contraction ideally located and
oriented to favour failure at this interface, and initiating fracture here at the location
marked in Figure 3.6 where the band terminates. This indicates that even without a
visible inflection in the interface, the triple junction reported by Liu et al. [107] has
3.5. CONCLUSIONS

a strong influence on fracture. However, in cases where this area and the top of the weld have been removed, as in the work by Liu et al. [107], the AS contraction band can deform the entire thickness of the sample evenly, and failure shifts to the RS where it is triggered by the more extensive but diffuse region of basal slip here. These results provide underlying microstructural causes and distribution for the textural asymmetry reported by other authors (e.g. [77, 13]) and provide a full 2D picture of basal slip in a FSW.

3.5 Conclusions

Direction of shear material flow was measured by EBSD in the vicinity of the weld interfaces, and used to visualise the flow in the weld. Voids were found to be located at the intersection of two separate flow paths. In the locations measured, the flow is not only subjected to the dominant rotational force but also moving upwards or downwards vertically through the weld. This is evidence that the classical picture of a conical shear path centered on the pin is over idealised and inaccurate.

The synchrotron data presented spans the entire cross section of a FSW, and supports the EBSD data by showing that texture is not symmetrical across the weld, nor is it consistent through the depth of the weld. Further, the texture distribution changes with processing conditions and moves with the nugget interface.

The most critical aspect of this texture asymmetry between the AS and the RS with respect to transverse tensile behaviour is that the regions where basal slip is favoured are arranged along the AS interface at 45° to the tensile axis, terminating where the material flow from the shoulder meets that from the nugget. This texture distribution reported for the first time here, leads to greater probability of early
yielding, strain localisation, and subsequent fracture along the AS interface, and also explains the switch in fracture location to the RS when the top of the weld is removed due to the extensive but more distributed arrangement of easy basal slip at this side of the weld.

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Chapter 4

Formation mechanisms of periodic longitudinal microstructure and texture patterns in friction stir welded magnesium AZ80

Previously published as [16], with notes added for later thesis version marked with an (*)

J. Hiscocks, B. J. Diak, A. P. Gerlich and M. R. Daymond

Abstract

Many studies of friction stir welding have shown that periodicity of metal flow around the tool pin may result in the formation of periodic differences in microstructure and texture in the weld nugget area correlated with the weld pitch. The current work investigates the periodicity of magnesium weld microtexture in the nugget region and its association with material flow using optical and electron microscopy. Two welds created in AZ80 at different processing conditions are presented in detail, one illustrating periodic longitudinal texture change, and one showing for the first time that periodic variations in texture, grain size, or composition are not defining features
of periodic nugget flow. While nugget texture is dominated by shear deformation, it was found here to be affected to a lesser degree by compaction of material behind the welding tool, which led to reduction in intensity of the shear texture fiber. The decreased tendency for magnesium based alloys to form periodic patterns as compared to aluminum based alloys is explained with reference to the shear textures.

**keywords:** Friction stir joining, onion ring, shear texture, texture intensity, void, defect

### 4.1 Introduction

Friction stir welded (FSW) joints offer superior mechanical performance [6] in comparison to fusion welding techniques, with potentially fewer defects such as voids [10, 44], while being both cheaper and better suited to automation [10]. More aggressive light-weighting efforts by the automotive industry will require use and efficient joining of magnesium-based alloys. Alloys of magnesium with aluminum and zinc (AZ series) such as AZ31, AZ61, AZ80, AZ90 are fairly mature and used in industry. Mg AZ80 has the highest aluminum content in this series that can be both wrought and precipitation hardened to be relatively stronger than the other AZ alloys [4], and thus AZ80 is the focus of study in this work. This alloy contains approximately 12 % by volume [133] of the $\beta$ phase $\text{Mg}_{17}\text{Al}_{12}$ intermetallic precipitate, which in the case of extrusions is generally distributed inhomogeneously throughout the workpiece in strings aligned with the extrusion direction.

Chen et al. [51] have shown that the macroscopic plastic flow in joints made by FSW can be divided into three regions: a top zone, dominated by the shoulder forces; a central nugget zone, dominated by pin and thread movements; and finally the swirl
zone at the weld root formed by flow under the pin (see Figure 4.1a). Marker insert flow studies in AA6061 by Xu et al. [47] have shown that within a broad set of processing conditions the shoulder flow is continuous (with material along the joint interface redeposited in a linear fashion behind the tool), while the flow in the nugget zone is periodic, with material from the joint interface deposited in regular arcs behind the tool. Flow in the swirl zone seems to be somewhat intermediate between the two states, with unstable periodicity at some processing conditions and near continuous behaviour at others. Xu et al. [47] have also shown that the extent to which any one of these three material streams contributes to the finished cross section of the weld varies with the processing conditions, and that the extent of periodic nugget flow is more dependent on the RPM than the welding speed.

Krishnan [53] showed the process by which periodic flow deposited in the nugget zone takes the form of successive hemispherical layers which when sectioned on the transverse plane reveal a concentric ‘onion ring’ feature. In 2XXX and 5XXX series aluminum alloys this structure has been found to consist of variations in grain size and particle distribution [55, 56] in addition to texture change. In all cases where texture measurements of an onion ring pattern were performed in an aluminum alloy, periodic texture changes were reported [57, 58, 59, 60, 61] in the 1XXX, 2XXX and 6XXX series alloys used. This is in agreement with the results of multiple stop motion FSW experiments (e.g. [51]) and other investigative techniques [54] which consistently show nugget material moving around the pin in distinct layers.

While periodic variations in grain size and particle distribution may be developed due to deformation during the welding process, Colligan [62] has shown that they can also be caused in AA6061 as the tool threads cyclically draw in material from
Figure 4.1: Friction stir welds shown from greatest heat input a) to least d), sectioned along the ND-WD plane at the centerline of the weld, tartaric etch. Complete image is a composite of many stitched images aligned and blended. The top image shows the depths of the shoulder flow, nugget flow, and swirl flow with vertical white arrows, and the weld root with a horizontal line. Voids are present at the base of the lowest weld (*see inset 3b), which also shows the AS interface from the ND-TD plane of this weld rotated and overlaid (*as a white line). Magnified views of selected areas are shown as Figure 4.2 and 4.3. a) 88 mm/min, 820 RPM. b) 88 mm/min, 583 RPM. c) 148 mm/min, 583 RPM. d) 248 mm/min, 583 RPM.

different depths of the plate. Attallah et al. [55] have shown 2XXX or 5XXX series aluminum plate with bands of intermetallic particles will result in a banded FSW
nugget with variations in grain size and particle distribution, while at the same processing conditions a homogeneous plate will produce a homogeneous nugget without optically visible onion rings.

In contrast to FSW in Al based alloys, onion ring banding reported in the AZ alloy series of Mg alloys is generally attributed to local compositional changes [9, 45] caused as the $\beta$ intermetallic phase is affected by the temperatures and shear deformation of material during FSW. Depending on the intermetallic volume fraction and heat input conditions either the precipitate becomes dissolved into the matrix causing local composition changes and different local etching behaviour, or at high RPM it forms liquid films which can lead to cracking or degradation of mechanical properties [45]. Since tapered pins produce greater heat input compared to cylindrical pins [63], formation of onion rings will be favoured by tapered tools, as can be noted by comparison of the microstructures produced by Borle et al. [45] and Yang et al. [9]. An alternate type of banding in AZ61 FSW was created with a cooled fixture by Lee et al. [64], who found alternating bands of fully recrystallised fine grains and partially recrystallised coarser grains in the nugget, presumably resulting from cyclic variations in shear strain such as those modelled by Xu et al. [47].

Texture change during FSW is dominated by shear deformation [57, 60], which at high magnitudes causes formation of the $B$ shear fiber in aluminum with close packed $\langle 110 \rangle$ directions along the shear direction and $\{112\}$ planes aligned with the shear plane and at lower magnitudes causes formation of the $C$ fiber [73]. Using this information, the shear plane, direction, and a rough estimate of strain magnitude may all then be extracted from texture data in aluminum. Investigations of aluminum texture banding in AA2195 have shown it to be consistent with deposition of material
layers oriented with $B$ fiber at one surface, $C$ fiber at the interior, and then the opposite direction of $B$ fiber at the final surface of the deposited layer [59].

For magnesium, shear strain causes formation of the $B$ shear fiber, with the (0001) basal plane aligned with the shear plane and some weak preferential alignment of the $\langle 1\bar{1}20 \rangle$ axis in the slip direction [73, 13]. The macroscopic shear strain during FSW involves rotation around the shoulder near the top of the weld and the pin lower in the weld [74], and so the three dimensional texture distribution in magnesium can be visualised as a surface created by revolving the AS interface line around the axis of the tool, with basal planes tangent to this surface [13]. It should be noted that in magnesium the positive and negative $B$ shear fiber are identical, and no other shear textures are formed at lower shear strain magnitudes [75]. Due to this limitation, shear textures in magnesium may only be used to determine the plane of shear, and neither the direction nor the magnitude of strain can be extracted. To date, no investigation of periodic texture changes along the welding direction has been reported for magnesium FSW.

When these shear strains are combined with the temperatures generated during FSW, dynamic recrystallisation (DRX) occurs in the weld nugget [67, 69, 42]. While Chang et al. [67] have reported that DRX will result in reduction in intensity of the texture as new grains with different orientations are nucleated, the intense textures formed during magnesium FSW [13] indicate that shear strain is by far the dominant influence on texture formation in a magnesium FSW. In essence, shear deformation applied with a constant vector is only capable of intensifying texture in magnesium, and any decrease in the texture intensity means that less shear strain was applied, nucleation of new grains occurred following shear, or other strain modes such as
The current work investigates the effect of FSW heat input on periodicity of texture and microstructural features within the weld nugget, with the aim of explaining why these features were found only at certain processing conditions, and why the occurrence of periodic texture is reported frequently in aluminum based alloys but not in magnesium to date. As part of this work, a new vector format of microtexture representation is used which takes advantage of the low symmetry of magnesium and the tendency to form strong shear textures to present material flow information. To the authors’ knowledge this is the first work to present a texture analysis of the periodic flow of material in magnesium FSW, the first report on change in texture periodicity with processing condition in any alloy, and also the first work to show that texture intensity in the nugget is influenced by compression of layers behind the tool.

4.2 Experimental Procedure

All welds were made in a bead-on-plate configuration on an as-extruded 6.3 mm thick AZ80 sheet (composition in wt% of 7.75 Al, 0.65 Zn, 0.14 Mn, 0.09 Si, 0.005 Fe, and balance Mg) transverse to the extrusion direction. FSW was performed using a cylindrical M4 right-hand threaded tool with shoulder diameter 10 mm, and a pin diameter 4 mm by 2.2 mm long, tilted 2.5° away from the welding direction with counter clockwise rotation. As a result, in no case did the welded area extend through more than half the thickness of the plate, allowing for investigation of the deformation directly beneath the weld. Four combinations of welding speed and RPM were used as described in Table 4.1 giving a range of heat inputs to the weld. The
highest heat input condition was selected per the work of Borle et al. [45] to produce sound welds with no visible onion rings, while other conditions were chosen for faster production. The ultimate tensile strength was found to be greatest in the weld with the lowest pitch, decreasing with the heat input as described in a previous work [8]. Heat input, which is strictly determined by the FSW processing parameters is not directly related to weld temperature over all possible conditions [128], however it is anticipated that for the range of processing parameters used here decreasing the weld pitch and increasing the heat input will each result in greater nugget temperature, (see [8] for supporting evidence).

<table>
<thead>
<tr>
<th>Heat input</th>
<th>Welding speed</th>
<th>RPM</th>
<th>Advance per Rotation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>88</td>
<td>820</td>
<td>0.107</td>
</tr>
<tr>
<td>2</td>
<td>88</td>
<td>583</td>
<td>0.151</td>
</tr>
<tr>
<td>3</td>
<td>148</td>
<td>583</td>
<td>0.254</td>
</tr>
<tr>
<td>4</td>
<td>248</td>
<td>583</td>
<td>0.425</td>
</tr>
</tbody>
</table>

Following sectioning, samples were optically polished; the optimum procedure was found to be diamond paste abrasives of sizes 9, 3 and 1 µm lubricated with a 3:1 ethanol:glycerol solution on napped cloths. A final polish with colloidal silica on napped cloth was followed by immersion etching in either 1 g tartaric acid and 20 mL H₂O for up to 20 s or in 2:100 nitric acid:ethanol for 10 s as indicated. Optical microscopy images were captured digitally. As noted in figure captions, GIMP [134] was used for combining images in conjunction with edge blending using the Enblend [135] software tool, while for some images depth of field was improved by using the ImageJ [127] Stack Focuser Tool [136]. Most samples for electron back-scattered
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diffraction (EBSD) were re-polished with colloidal silica followed by ion milling in a Hitachi IM4000 where the optimum procedure was found to be 3° inclination to surface, 4.5 kV acceleration and 1.5 kV discharge, 0.1 cm³ min⁻¹ argon gas and 25 RPM for 30 min.

EBSD scans were performed in an FEI nanoSEM at an accelerating voltage of 20 kV with a Bruker e-flash detector. Data analysis was done with the MTEX [23] software module for MATLAB [137]. For all grain size measurements the misorientation threshold was set at 10°, and grains consisting of a single point were removed (see figure captions for resolution). In addition to the standard IPF coloration, other EBSD data representation methods were developed for use in this paper and are detailed below.

To generate the misorientation coloration used for some EBSD maps, the angle between the [0001] axis of an indicated reference point and of all other points of the map was calculated. To generate the EBSD vector plots, data was segmented into rectangular regions from which a contoured (0001) pole figure was generated. Shear deformation in magnesium generates a single strong B fiber orientation which can be located by taking the maximum of the [0001] pole figure and using it to calculate a 3D vector which represents the shear plane normal. By repeating this procedure a collection of shear plane normal vectors can be plotted, colored to indicate the degrees between the 3D vector and the plane of the EBSD scan. To calculate the fiber % values in this paper, the percentage of orientations with [0001] axes within 30° of the greatest intensity location on the contoured (0001) pole figure was calculated.

It should be emphasised that given the intense texture formed in the nugget during welding, more scatter within the nugget represents non-shear deformation applied
after material movement around the pin or DRX. In contrast, changes in the plot of misorientation to the reference represent a change in the shear plane between that point and the reference, the direction of which may be determined from the vector plot. By comparing these two factors information about the deformation process can be extracted from the texture data.

In all cases, positive directions are defined as ND (normal direction, upwards through weld), WD (welding direction, in the direction of tool movement) and TD (transverse direction, towards the AS interface).

4.3 Results and Discussion

Samples sectioned on the ND-TD plane, (shown in previously published work [8]) presented no optically visible onion ring structures. Figure 4.1 shows optical micrographs of the first 20 mm for each weld heat input along the WD midplane from which all optical micrographs of this work were taken. At the start of each nugget where the tool was first inserted are darker zones of finer grain size than the surrounding material, which in some cases extended periodically in the WD for approximately 1 mm. Finer grain size is associated with lower temperature welding conditions [9], and so the extent of these fine grains indicates the distance for which the tool acted as a heat sink. This in turn implies that changes in tool thermal conductivity and heat capacity will affect the final grain size, which is supported by the work of Padmanaban et al. [103].

The white arrows at the top right of Figure 4.1 indicate the depth of the shoulder, nugget and swirl zones. Qualitatively, the microstructure has the most homogeneous
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appearance in the nugget flow region followed by the shoulder, and is least homoge-
neous in the swirl zone at the root of the weld. Within the shoulder layer, the
microstructure is most homogeneous near the surface and includes progressively in-
creasing fractions of second phase regions closer to the nugget zone. These changes in
homogeneity of microstructure imply that the shear strain was greatest at the top and
bottom of the nugget flow as well as immediately under the shoulder. This correlates
well with simulations of threaded tool FSW by Yu et al. [82], which show high strain
rate underneath the shoulder and lowest threads of the pin, which would result in
greater shear deformation in these locations.

The base material (shown as Figure 4.2a) contains second phase intermetallic
colonies and near-equiaxed untwinned grains. Beneath the tool insertion point at the
start of the weld there is a darker region (shown as Figure 4.2b) indicating the thermo-
mechanically affected zone (TMAZ) extends deeper here than in other locations due
to a brief dwell time before the tool began to travel across the sheet. Comparing
this area to the base material, large numbers of twins are present, and zones of fine
grains are clustered around the colonies of intermetallic. In contrast the swirl zone
at the root of the nugget (shown as Figure 4.2c) has no visible twins, and bands of
fine grains as well as colonies that have been elongated by the welded material flow
are present as dark coloured streaks.

Periodic features within the nugget were found at every condition, decreasing in
prominence as heat input increased as shown in Figure 4.3. Periodicity within the
highest heat input weld was only visible as surface relief rather than colour change
after etching, and one example of this flow path has been noted by a dashed line
added to Figure 4.3a. In this weld, periodic flow patterns were most visible at the
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Figure 4.2: Magnified views of microstructure, tartaric etch, images stack focused with *ImageJ*. a) Undeformed material far below the weld. b) Twinned material beneath the tool insertion area (see Figure 4.1 for location). c) Bands of fine grains in the weld root TMAZ (see Figure 4.1 for location).

top of the nugget adjacent to the shoulder flow, and not optically visible at all depths. In the case of the lowest heat input weld nugget shown in Figure 4.3b, the periodic patterns were clearly visible and extended the length of the weld, incorporating voids with the cyclic flow patterns. Flow lines extending from the root to slightly below the shoulder were also optically visible (see curve traced in Figure 4.1d), indicating the flow has periodic aspects even in the shoulder region at these conditions. A literature search did not reveal other reports of this phenomenon which is unsurprising given the atypically high welding speed used in this case.

Calculation of shear plane normal vectors from EBSD data shown adjacent to the weld microstructure gives insight into the flow of material through the weld depth, as shown in Figure 4.4a. Comparison between the vector plot at the left and the AS interface profile trace (copied to scale from the ND-TD plane) shows that the shear plane normal vectors run perpendicular to the overlaid profile, meaning that the shear plane corresponded with a revolved AS interface profile and confirms that the majority of the weld texture is imposed by shear deformation around the pin
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Figure 4.3: Magnified views of periodic areas of Figure 4.1. a) Highest heat input condition, nitric etch, image defocused to show relief with dashed line to indicate the flow path for one typical period of ~7 visible. Micron bar equal to 3x weld pitch. b) Lowest heat input condition, arrows indicate void locations, tartaric etch. Micron bar equal to 1x weld pitch.

axis (in agreement with multiple other publications c.f. [138]). Below the base of the welding tool passage the extent of the TMAZ was small, as material rapidly returned to the ND orientation typical of extruded base material.

A higher resolution EBSD area scan of the high heat input weld nugget region spanning three weld pitches was performed and is shown as Figure 4.4b. While Figure 4.3a shows periodic patterns in the nugget region, confirming that as expected periodic material flow occurred in this area, no evidence of periodic variations in texture or grain size was found. Energy-dispersive X-ray spectroscopy scans of this area did not show periodic variation in the levels of Al or Mg present. These results clearly indicate that periodic flow occurred in this location without resulting in detectable texture, grain size, or compositional periodicity.
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Figure 4.4: Highest heat input weld data. a) (From left) Micrograph (tartaric etch) extending from the weld surface into the base material. Multiple images aligned and blended. Shear plane normal vectors covering an equivalent area generated from EBSD data. EBSD scan resolution 0.83 $\mu m^2$ for vectors at left, 64 $\mu m^2$ for vectors at right. These two figures overlaid with AS interface profile of high heat input weld rotated from the ND-TD plane to scale. Graph of grain size and fiber texture intensity through weld depths, calculated from EBSD data with resolution 0.83 $\mu m^2$. b) Higher resolution data from area indicated by the box in Figure 4.4a. (From left) Optical image (tartaric etch, scale indicates 1 period, multiple images aligned and blended). EBSD scan (resolution 2.0 $\mu m^2$) coloured as degrees of [0001] misorientation to the reference point indicated with an ‘x’ (see Figure 4.5 for color map). Pole figures of indicated areas, with (0001) at left and (10\(\overline{1}0\)) at right. Numbers are max intensity of (0001) contoured data.

Since etching contrast normally arises from differences in chemical composition, grain size, texture, or dislocation density, it suggested that the periodic features visible in the nugget of the highest heat input weld are the result of periodic dislocation density changes along the WD. While work by Park et al. [110] has shown that dislocation density was fairly constant across the nugget in the TD, there has yet to be any work comparing dislocation density across one weld pitch.

The top pole figure shown in Figure 4.4b confirms that the top section of this EBSD scan captured the effect of the shoulder forces. The second pole figure down
shows a mix of shoulder and nugget texture, while the rest of the pole figures reflect
the shear deformation around the pin within the nugget. It can be observed that the
various locations of the (0001) peak are in good agreement with the gradual curvature
of the flow surface through this region seen in Figure 4.3a, although less so with the
revolved interface profile. This series of pole figures shows that the texture profile
does not follow the AS interface, but instead has an abrupt transition at the interface
between shoulder and nugget flows, indicating that the material flows were discrete,
as described by Chen et al. [51].

Although the texture within the nugget is dominated by shear deformation around
the pin it can be observed from Figure 4.4a that the fiber % drops to a value of 52%
in this area as compared to the shoulder or swirl areas. This is confirmed by the
higher resolution analysis of Figure 4.4b where the maximum intensity of the (0001)
pole figures is far greater at the base of the nugget than near the top. This decrease
in intensity of the nugget texture has been attributed to DRX in the nugget by
Chang et al. [67], and to texture banding by Chen et al. [139], neither of which is
supported by current results. It is evident from the microstructure of Figure 4.4b,
that the entire FSW is composed of recrystallised grains, which agrees with multiple
past published works (e.g. [67]). Since the fiber percentages shown in the graph of
Figure 4.4a decrease only in the nugget area as opposed to the shoulder or swirl zones
it is unlikely that DRX is correlated with decrease in nugget texture intensity.

The lack of periodic texture effects seen in Figure 4.4b likewise eliminates tex-
tural banding as the source of this effect. Our current hypothesis is that during
movement around the tool, nugget material developed the commonly reported high
intensity shear texture which was then altered during compaction into final position.
The compression strains applied during this process would have been almost directly aligned with the high intensity [0001] axis, and thus any texture change would have resulted in dispersion of the texture previously developed. Evidence for the existence of periodic compressive forces in the WD and in the ND can be found in the work by Yan et al. [56] who instrumented an FSW tool and found that during welding forces in the ND and WD were cyclic with a period equal to the tool advance per rotation. This finding is in good agreement with successive compaction of each layer of deposited material behind and under the tool as described by Gratecap et al. [54].

The current results suggest these compression forces in the nugget were significant enough to noticeably decrease the fiber % and the maximum intensity of the pole figures (as shown in Figure 4.4), but not sufficient to reorient the texture peak.

An EBSD scan of the lowest heat input weld is shown as Figure 4.5, covering the nugget and extending from the lower part of the shoulder region just into the base material near the base of the figure. In the TMAZ beneath the weld, grains become refined and the basal flow normals reorient towards the WD as was also observed in the higher heat input weld (see Figure 4.4a). Above this, a layer of material exists which corresponds with the swirl flow discussed by Chen et al. [51]. This layer is separated from the nugget flow by voids of various sizes, with evidence of periodic texture both above and below this layer.

The majority of the periodic flow is located above the layers of voids, indicating that it is associated with the nugget flow material rather than that swirled under the tool. Furthermore it implies that the two flows of material are separate and rejoin incompletely, which is in agreement with both the work of Chen et al. [51] and the sharp termination of periodic banding near the root of the weld shown by
various authors ([47, 62]). Between the ovals of out-of-plane texture that occur with a cycle equal to the weld pitch, the typical texture perpendicular to the AS interface re-establishes itself as can be seen from the left hand side of the vector plot in Figure 4.5. The presence of thin layers with the standard shear texture indicates the out-of-plane shear normal vectors shown in the nugget are not a necessary consequence of rotation around the pin or thread interactions, and that for one point in each cycle, flow here was similar to that seen around the shoulder. At the top of the figure, the shoulder region of the misorientation map shows subtle evidence of continuing periodicity corresponding to the periodic shoulder flow lines that can be observed in Figure 4.1d, while the vector representation of texture in this area conforms to the in-plane shear behaviour described previously.

Our current hypothesis of nugget compression dictating texture here is further supported by the periodic behaviour of the lowest heat input weld swirl zone (see Figure 4.5) under the voids. The sawtooth distribution of these voids aligned along the interface between material flows is best explained by flow from the nugget cyclically displacing the swirl flow and pushing the voids trapped between the two layers downwards towards the weld root causing periodic texture reorientation of the swirl zone material. The distribution of the voids is uneven, with larger voids trending towards the weld start side. If both nugget and swirl zone flows had been periodic it is unlikely that an interface would have formed between them during welding. Since the nugget flow was periodic, it follows that the swirl zone flow was continuous beneath the tool (i.e. not deposited in discrete layers), triggering the division of these flows during welding which becomes a mechanism for void formation.

At the right of Figure 4.5, a comparison of various sections of the periodic weld...
is provided with the grain sizes and fiber %. Similarly to the results seen in the highest heat input weld, the shoulder areas and the swirl zone showed high fiber % as compared to the nugget region, although the relatively high intensity in zones (c) and (d) indicates that the entire thickness of the nugget experienced sufficient shear strain to develop the $B$ fiber. It can also be seen that areas (a) and (d) have higher fiber % as compared to the adjacent areas of (b) and (e) which also show a different (and atypical for this area) orientation. This indicates that the material for areas of (b) and (e) was not simply rotated or sheared on a different vector, but that the deformation mechanism was notably different. All of these results support a mechanism of compaction behind the tool, where areas (a), (c) and (d) were sheared and reoriented while areas (b) and (e) were compressed and reoriented. Within the nugget, areas (a), (b) and (c) have nearly identical grain sizes but greatly different fiber %, indicating that compression of zone (b) was accommodated either by slip or that twinning was followed by recrystallisation. Comparing zones (d) to (e) it appears that for each cycle the material on the tool side (+WD) was more affected by compaction than that towards the weld start (-WD), also in agreement with the distribution of voids discussed previously.

The nugget textures can be compared between highest and lowest heat input conditions. The highest heat input weld showed increased scatter in the nugget, but the orientation of the main texture peak was generally constant through the weld pitch and in agreement with the expected shear plane profile. This suggests that while flow was periodic and compression occurred in the nugget the net effect on the texture of the deposited volume of material was essentially constant along the WD. In contrast, the texture in the lowest heat input weld was reoriented to
4.3. RESULTS AND DISCUSSION

Figure 4.5: Lowest heat input weld data covering the area shown in Figure 4.3b. Left; EBSD scan (resolution 3.2 $\mu m^2$) of the periodic weld features at the weld root coloured as degrees of [0001] misorientation to the reference point indicated with an ‘x’. Circles added to indicate void locations. AS interface profile rotated and overlaid to scale. Horizontal line indicates the depth of Figures 4.6 and 4.7. Center; shear plane normal vectors of half the EBSD map data showing the degrees out-of-plane. Right; Selected areas with values of grain size ($\mu m^2$)/Fiber (%).

Varying degrees for most areas of the nugget, while the scatter was also increased for a portion of these areas. This result is consistent with rotation of each layer of the nugget material during deposition, followed by compression of the outside of each layer facing the tool. While the rotation seen in the nugget flow is currently difficult to explain, the inhomogeneous distribution of compression effects in the lowest heat input nugget along the WD can be attributed to the greater weld pitch here. For the lowest heat input weld the deposited layer will be $\sim$4x as thick as the high heat input weld (see Table 1) which when combined with the lower nugget temperature resulted in a far more inhomogeneous strain distribution of the compressive forces applied by the tool.
Once the temperature in the weld has stabilized, the grain size becomes constant in the longitudinal direction. From Figure 4.4a the grain size in the ND of the highest heat input weld can be observed to increase from $14 \mu m^2$ near the surface to $35 \mu m^2$ at the bottom of the nugget, and then abruptly decrease to $21 \mu m^2$ in the swirl zone before increasing in the base metal ($\approx 66 \mu m^2$). For the lowest heat input weld, grain sizes are listed in Figure 4.5 and can be seen to decrease slightly from the lower shoulder region to the base of the swirl zone and remain constant in the TMAZ before increasing towards the base material. Comparing the nugget zone grain size in the ND-TD plane, it was found to decrease with heat input, from $31 \mu m^2$ to $12 \mu m^2$ to $4 \mu m^2$ (0.22 $\mu m^2$ resolution) for the welds shown in Figure 4.1 a) c) and d) respectively, all significantly finer than the base material.

Final grain size in the weld is determined by a combination of the strain rate, which will reduce the grain size, and the local temperature which will increase it. Simulation work by Yu et al. [82] shows temperature decreasing from the shoulder downwards to the root while strain rate is high both at the shoulder and near the root, with a decrease in the nugget area. Given the lower grain size in the highest heat input weld at the shoulder and in the swirl zone at the root of the nugget, this implies that the strain rate is a dominating factor for these conditions. In contrast, for the lowest heat input weld the grain size decreases vertically through the lower shoulder, nugget, and swirl zone monotonically (although the upper shoulder is omitted from this data set) which implies that at these conditions thermal input has a larger effect on final grain size.

Neither the highest nor the lowest heat input weld showed grain size changes along the WD, and so it can also be concluded that cyclic changes in the plastic
strain magnitude such as those found in simulation work by Xu et al. [47], were either not present or of insufficient magnitude to cause measurable grain size changes following recrystallisation.

To gain additional perspective on texture distribution, the lowest heat input weld was sectioned in the TD-WD plane. An EBSD scan shown as Figure 4.6 reveals that while the periodically deposited layers are still visible, flow is far less regular due to minor flow depth variations which can also be seen in Figure 4.3b. From the misorientation map, the deposited layers can be seen to vary slightly in thickness along this plane, and to have a slight texture change between the edges of the layer and the center with voids located between these layers along the weld center line which are a likely precursor to a larger volumetric defect.

![Figure 4.6: Lowest heat input weld data. RS towards left, AS towards right (resolution 7.1 \(\mu m\)^2). White areas along the centerline (indicated with a vertical line) represent voids. Left; EBSD scan data coloured as degrees of [0001] misorientation to the reference point indicated with an ‘x’. Right; shear plane normal vectors of the same data showing the degrees out-of-plane.](image)

The vector analysis at the right of Figure 4.6 shows that flow near the AS side of the nugget was very consistent, while that towards the RS side of the weld was less regular but more in-plane. Along the center line a large segment with the shear plane
normals in the ND is present. The material near the RS is swirl flow moving mostly in-plane, while around the center line the flow becomes material forced downwards from the nugget displacing the voids ahead of it, leading to a reorientation of the material to show shear plane normals now near the ND.

Large volumetric lack-of-fusion defects (also called wormhole defects) which form a continuous channel of missing material aligned in the WD at the root of the weld are not uncommon in FSW. Various authors have shown this defect to form where the nugget and swirl flows join [49, 65], and to be associated with insufficient heat input to join successively deposited layers of material together. While Doude et al. [140] have shown that in AA2219, this defect initiates at insufficient RPM and increases in cross section with further reduction in RPM, there is little information on how this defect first develops from a sound weld as the weld heat input drops.

The periodic voids found in the lowest heat input weld immediately above the swirl flow are consistent with both the expected position of a lack-of-fusion defect and the processing conditions where it would be expected to initiate. It is likely that these voids are a precursor to the formation of a continuous wormhole defect created as the flow of nugget material becomes increasingly irregular with decreasing heat input. Seidel et al. [141] have shown that increasing RPM is far more effective at increasing heat generation than decreasing welding speed, and so in cases where voids have been detected in a weld at low heat input conditions, increases to the tool RPM will be the most effective method of preventing further occurrences.

A scan to the side of Figure 4.6 spanning the AS interface (Figure 4.7a) shows that the nugget texture matched the conical shear deformation expected, and is approximately 50° out-of-plane. Periodic texture effects visible in the EBSD map agree
4.3. RESULTS AND DISCUSSION

with those of Figure 4.6, although these are not reflected in the vector map due to averaging of areas. A sharp change in orientation and an abrupt refinement in grain size were both found at this interface, which agrees with previous results [8]. Of additional interest here is the reorientation of vectors across the AS interface, indicating the grain refinement in the TMAZ corresponds with a texture change inclining the shear plane normals opposite to the WD. This reorientation occurs due to a combination of tool contact forces, as the material is first sheared away, and compression forces, as the material behind the tool is pushed into place. Similar indications of deformation at this interface can be seen in other work such as that by Fonda et al. [138] in AA2195 showing grains sheared towards the WD in this area, and the stop-motion work by Chen et al. [51] showing a deformed layer at this interface even before the weld nugget material is pushed into place.

Comparing the AS interface to Figure 4.7b which shows the RS interface, it is evident that the RS side TMAZ has undergone more grain refinement and is of greater width both in terms of grain refinement and of texture change. This observation is in agreement with the work of Chen et al. [51] which also shows a much wider TMAZ at the RS of the weld. Within the nugget area of Figure 4.7b near the RS interface, the texture is mostly smooth conical shear deformation with multiple patches showing vectors oriented towards the ND. This latter orientation is not present at the equivalent position near the AS interface. It is likely that these regions are due to base material being drawn into the weld without sufficient shear deformation to cause reorientation, a theory for which much evidence exists including the work by Fonda et al. [58], who showed material being drawn from the TMAZ into the nugget area near the root of the weld, and the work by Prangnell et al. [65] showing grains
drawn into the weld at the RS. This result also agrees with previously published work by the current authors [8] where an ND-TD EBSD scan in an equivalent location on a weld made at 148 mm/min and 583 RPM revealed a similar texture component to that reported here.

### 4.4 Conclusions

The current work shows that while FSW nugget textures in a magnesium alloy are dominated by radial shear strain, reduction in intensity of the nugget texture as compared to the shoulder area is attributable mainly to compaction during the final stages
of FSW material deposition. Evidence has been presented to show that whether this compaction affects the texture of the deposited layer homogeneously or not depends on the weld pitch. The reduced tendency of AZ80 to form periodic texture changes as compared to aluminum based alloys can be attributed to the convergence of the texture from the initial orientations into a single homogeneous $B$ shear fiber orientation, which is not seen in aluminum. At the conditions which give good mechanical performance (i.e. the highest heat input tested here (\$[8]\$)) the thickness of layers is insufficient to develop noticeable periodicity either of compaction or fiber orientation, resulting in welds that are homogeneous along the WD and explaining the general absence of longitudinal periodicity reports in this alloy. For the first time it has been demonstrated that periodic flow in the nugget will not necessarily equate to periodic texture, compositional, or grain size changes in the weld. It has thus been established that while the presence of an onion ring feature necessitates periodic flow, the absence of this feature does not require a different flow path. Indeed, this and other work strongly indicates that flow which forms the nugget is periodic in all cases. In addition this work uses wide EBSD scans to demonstrate that flow in most of a FSW is well aligned with a revolve of the AS interface profile, by means of a newly presented texture representation method ideally suited for materials with low symmetry where strong textures form.

4.5 Acknowledgements

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Chapter 5

Distribution of Residual Stresses in 2D Across Magnesium AZ80 Friction Stir Welds at Different Processing Conditions

J. Hiscocks, M. R. Daymond, B. J. Diak, and A. P. Gerlich

Abstract

While residual stress has a significant effect on deformation behaviour, much of the available research on this topic is in the form of line scans rather than area maps. To address this, low angle synchrotron transmission diffraction with fine measurement spacing has been used to scan friction stir welds made at three processing conditions. The resulting high-resolution spatial maps reveal several features that would not be detectable by line scans. The asymmetric material flow in a friction stir weld which has previously been shown to result in asymmetric temperature and texture distribution is shown here to also result in asymmetric residual stress and strain distributions. Comparison of residual stress between three friction stir welding conditions shows how the stresses at the interface can reverse from compressive to tensile with decrease in
5.1 Literature Review

5.1.1 Introduction

Friction stir welding is a solid state joining technique offering multiple advantages for use with materials that are challenging or impossible to weld successfully by fusion methods [6]. The tool used consists of a non-consumable rotating shoulder and narrower threaded pin. The tool progresses along the joint line in the welding direction (WD) while rotating, and the incoming material is heated by the tool action and sheared or extruded around the pin and under the shoulder.
5.1. LITERATURE REVIEW

The high Al containing Mg alloys AZ80-AZ91 are one example of materials unsuitable for fusion welding due to the brittle intermetallic formed along the grain boundaries [7], however friction stir welds of good quality have been successful [8, 9]. Of the two alloys, AZ91 is used in a cast form, while the more deformable AZ80 can be extruded. AZ80 offers a weight reduction of about 26% for parts of the same yield strength as AA6061 in exchange for a reduction in ductility [1, 2] and higher cost [3].

Residual stresses are the self-equilibrating stresses that exist internally to a material body in the absence of any applied stresses [91] and exist on a range of length scales. Type I macrostresses extend over the scale of many grains [92, p. 47], and combine with applied stresses [91, p. 8110], affecting the fracture resistance [91, p. 8151] of the part. Residual stresses of this type, particularly those in the transverse direction (TD) [94] also influence distortion of the weld [91, p. 8110, 8123].

Residual stresses from fusion welding are due to thermal effects, solidification, phase transformations, and material movement such as that between two butted plates [91, p. 8122] in addition to contributions from mechanical deformation [95]. For AZ magnesium alloys, neither solidification nor phase transformation effects are expected to occur during cooling of the nugget following friction stir welding. While the mechanical deformation in the nugget of a friction stir weld does impact the residual stress state, Woo et al. [95] confirmed that the deformation from the pin was a less significant factor in the final residual stress than the heat input from the shoulder. Similarly, Steuwer et al. [142] stated that the residual stress distribution was a function of the local thermal gradients and the material yield strength during friction stir welding. Friction stir welds made for research purposes are frequently done along the surface of a continuous plate to avoid material movement concerns,
as was done for the current work. This leaves thermal effects, mainly the inhibited thermal contraction of the friction stir welded nugget during cooling as the main source of residual stresses in a friction stir weld [93].

During friction stir welding the material around the tool is heated and plasticized, in some cases as high as the solidus temperature of the material [81]. At this high temperature, the yield strength of the material is low and thermal expansion effects are readily accommodated within the nugget. While the hot material of the recrystallised weld cools it undergoes thermal contraction, and at some point the yield strength of the material becomes high enough that plastic strain of the material from thermal contraction ceases. The residual elastic strains in the material from the differential thermal contraction that remain can be measured by various methods including diffraction, allowing for changes in processing to minimize them or optimize their distribution.

5.1.2 Thermal Distribution

The magnitude of residual stress developed has been reported to be related to the temperature differential during welding, the material thermal expansion coefficient, elastic modulus [143], and yield stress [90]. Prior to discussing the residual stress distribution, it is therefore necessary to have a good understanding of both texture (such as that presented in [8]) and temperature distribution within a friction stir weld.

Multiple thermal measurements in friction stir welds agree that temperature will be highest under the tool shoulder [79, 80, 81], and distributed asymmetrically [83, 80, 81] between the advancing side (AS) and retreating side (RS) of the friction stir
weld. Kandaswaamy [80] reported that in descending order, the temperatures were consistently shoulder AS, pin AS, shoulder RS, under pin centerline, and finally pin RS, unaffected by changes in the RPM, welding speed, or tool shoulder diameter. Fehrenbacher [81] attributed the higher temperature at the AS to higher shearing and velocity gradients there, as well as the direction of material flow, which results in the cooler new material entering at the front of the tool and flowing around the RS first.

Friction stir welds made at different processing conditions may be compared in terms of the heat input, a ranking which increases with increasing tool rotation rate and decreases with increasing welding speed [46, 6]. While the heat input should not be conflated with weld temperature and is not always proportional [81], within the range of processing parameters currently used it can be assumed that increases in heat input will result in increases in temperature. Comparing the relative impact of lower welding speed and higher rotational speed on the temperatures at the shoulder and the pin, Fehrenbacher [81] found that RPM generally had a more significant impact on temperature than welding speed [81, p.54-56], although in some cases the effects of welding speed could dominate over those of RPM [81, p. 82].

Asymmetry of the weld thermal profile has been found to increase with higher welding speeds [81], which the work of Huetsch et al. [83] attributed to the decreasing importance of rotational material flow and increasing importance of extrusion phenomena about the pin. Similarly, low RPM and high welding speeds were found to increase the disparity in temperature between the shoulder and the nugget [81].

Material thermal properties will also affect the temperature distribution during friction stir welding, which was found to become more homogeneous about the pin.
as the thermal diffusivity increased for a series of aluminum alloys [81]. For the current alloy series, thermal diffusivity decreases from $5.7 \times 10^{-5} \text{ m}^2/\text{s}$ for AZ31 [87] to $3.9 \times 10^{-5} \text{ m}^2/\text{s}$ for AZ80 [88] as aluminum content increases. According to the work of Fehrenbacher [81], this decrease in thermal diffusivity will lead to higher thermal gradients in the nugget area, which is predicted to result in higher residual stresses for AZ80 as compared to AZ31.

5.1.3 Residual Stress

For comparison with current results, a brief overview of transverse direction (TD) residual stress trends reported in literature for friction stir welds is provided. Residual stress in the normal direction (ND) for friction stir welds is not discussed as reports are quite limited and inconsistent.

Reported transverse residual stress results show significant variation, and in most cases peak stresses are aligned with the edge of the tool shoulder’s passage, where the highest temperatures are recorded during friction stir welding [84]. In some cases, the peak transverse residual stress is reported to be tensile (e.g. [78, 97, 99]), while in others this peak residual stress is reported to be compressive (e.g. [94, 100]). In the center of the weld the residual stress approaches zero.

Woo et al. [93] suggest that in aluminum the underlying mechanism for this central decrease is microstructural softening from precipitate dissolution and other sources, limiting the maximum residual stress the region can support. This softening along the weld centerline in aluminum may be seen by hardness testing (e.g. [96]). In contrast, for magnesium AZ31, Woo and Choo [96] found that hardness was essentially constant between the nugget and the base material, and instead attributed this decrease in
residual stress to a local reduction in the yield stress due to the texture change across the friction stir weld nugget. This theory was not supported by work of Webster et al. [101] which found that for a friction stir weld in AA7108-T79 the texture and residual stress were not closely coupled, and that the texture distribution did not align spatially with the residual stress distribution.

Due to the highly anisotropic nature of magnesium, it is certainly plausible that the texture influences the local yield strength and so the local maximum residual stress that is developed during cooling, however investigations of this theory have not been found in literature. Given the combination of strong plastic anisotropy of magnesium and the current experiment where a partial texture was measured for each point (see [8] for experimental details and limitations) simultaneously with the residual stress, the current work provides an ideal basis from which to address this question.

5.1.4 Precipitate Dissolution

While the focus of this work remains on residual stress analysis of the AZ80 matrix, it was determined that the current diffraction data could also be used to investigate the compositional change from dissolution of precipitates across a friction stir weld at high spatial resolution. Precipitation in AZ80 is limited to the Mg$_{17}$Al$_{12}$ intermetallic, for which there is no measurable growth at room temperature [36].

Under static conditions, time for solutionisation of this precipitate can be lengthy and a typical solutionising heat treatment for AZ80 is 24 h at 415 °C [66]. During friction stir welding dissolution of the precipitate is assisted by the shear deformation of the tool, which Chang et al. [67] report to be in the range of 5-50 s$^{-1}$ for AZ31,
causing dissolution of the precipitate to proceed rapidly. Yang et al. [9] report that for AZ80 friction stir welds made at 400-1200 RPM and 100 mm/min SEM measurements showed that the majority of the Mg$_{17}$Al$_{12}$ precipitate present in the base material was dissolved in the nugget. They reported the level of aluminum in solution was greater in the nugget than the base material, and TEM work confirmed that the Mg$_{17}$Al$_{12}$ precipitate was dissolved or broken up within the nugget [9]. Feng et al. [68] also reported that most of the Mg$_{17}$Al$_{12}$ phase was dissolved into the matrix during friction stir processing, with corresponding increases in the content of Al in solution. However, in this case fine Mg$_{17}$Al$_{12}$ particles were found to be distributed along the grain boundaries. In contrast, for thixomoulded AZ91, for four different processing conditions friction stir welding was found to completely dissolve the Mg$_{17}$Al$_{12}$ precipitate in the nugget based on optical microscopy and TEM of this region [69].

Consequently, it is expected that in the case of higher content AZ series alloys, precipitates will be present in the base material, partially or completely dissolved within the nugget, and of an intermediate state between these areas. In order to quantify the distribution in the varying regions of the nugget, a more convenient method than SEM or TEM of assessing the extent of precipitation spanning a large region would be valuable for both industrial and research applications.

5.1.5 Distribution of Residual Strain in a Friction Stir Weld

The thermal profile of the weld is known to be asymmetric, not constant through the depth, and affected by the material thermal diffusivity. Should the residual stress distribution be strongly correlated to thermal profile, it will also be dependent on
these factors. Based on the temperature distribution in friction stir welds previously described, we would estimate the magnitude of residual stresses on the ND-TD plane will be greatest at the AS interface, next highest at the RS interface near the top, followed by the region under the pin, and the lower RS interface. We would likewise expect residual stresses to become more asymmetric with decreasing heat input and decrease in overall magnitude with increasing heat input. Compared to AZ31, residual stress in AZ80 would be expected to be higher due to the lower thermal diffusivity and greater yield strength.

Validating these predictions can be challenging, as the vast majority of available results are in the form of line scans at the mid-thickness of a friction stir weld, and few works examine the change in residual stress values with friction stir welding processing parameter change. Additionally, reports on residual stress or strain in the AZ series of magnesium alloys are limited to the work in AZ31 by Woo et al. [96, 93, 97] with no apparent works for AZ80 or other high alloy-content materials in this series. Comparisons between literature results are complicated by the fact that the location of measurement with respect to the weld start is also expected have an influence on the transverse residual stress, and the magnitudes measured will decrease with the size of the sample being examined [89, 90]. Within these constraints, some published works supporting or contradicting the predicted distribution of residual stresses are described briefly.

Looking at the few available 2D residual stress or strain maps, the work of Prime et al. [90] based on contour data shows high WD residual stress at the AS and RS under the shoulder and along the AS interface, compressive residual stress lower in the nugget, and small residual stress low at the RS interface. These longitudinal
stress results are mostly consistent with the residual stress distribution predicted from the temperature distribution. While transverse and normal residual stress maps are available from some other sources, they either do not span the whole weld [94], have insufficient resolution to clarify the issue [102], or have methodological concerns [101].

The work of Lombard et al. [78] provides line scans of TD residual stress at a wide range of processing conditions. This work provides evidence that transverse residual stress is more impacted by changes in the welding speed than changes in RPM, and that as predicted higher heat input decreases residual stresses. However this work does not support the prediction that higher welding speeds will promote asymmetry, and instead shows decreasing difference between the AS and RS peak value as the welding speed increases [78].

The current work examines friction stir welds made at three processing conditions in extruded AZ80, with the goal of assessing possible correlations between texture, thermal gradients, and the residual stress distribution discussed above, in addition to providing basic residual stress measurements for AZ80 which are not currently available in the literature.

To this end, the most suitable form of data is a closely spaced 2D grid of points, mapping strain on the cross-section of a series of friction stir welds created at different processing conditions. To acquire the large number of measurements required, low-angle synchrotron x-ray transmission diffraction techniques were used. Compared to the $2\theta$ scanning method commonly used, low-angle transmission diffraction has several advantages including higher measurement speeds and fewer potential measurement errors, as there is no rotation of the sample required, or change in the length of the
beam path which may cause changes in background intensity. A typical $2\theta$ measurement made with neutrons in magnesium uses diffraction volumes of $2 \text{ mm}^3$ [97] and a measurement time on the order of 20 min per peak [115], while the current transmission experiments used diffraction volumes less than 0.02 mm$^3$ at 3 s per measurement with corresponding effects on the maximum possible resolution and equipment time requirements.

Low-angle synchrotron x-ray transmission diffraction techniques have been used for strain measurements by e.g. Daymond and Withers [144], Korsunsky et al. [145], and are discussed in detail by Hanan et al. [124] and Fitzpatrick et al. [92]. During low angle transmission diffraction, the direct beam passes through the sample and is intercepted by a beam stop before contacting the detector, and the complete forward diffracted Debye-Scherrer rings for multiple crystallographic planes are recorded simultaneously on one large area detector. If the distance between the diffracting volume and the detector is accurately known (generally calibrated using an oxide powder for each sample), and the incoming wavelength is known, the periodic spacing of that lattice plane can be calculated using Bragg’s law. Following each diffraction measurement, the sample is displaced in the ND-TD plane to form a 2D grid of measurement locations.

The three most significant microstructural factors in terms of influence on the Debye-Scherrer rings are chemical changes (which affect the ring diameter), texture changes (which affect the distribution of intensity around the ring circumference), and residual strains. While hydrostatic residual strains cause changes in the diameter of the diffracted rings, deviatoric strains cause eccentricity of the diffracted ring, and so the two can be independently determined if the diameter of the ring when under only
chemical effects ($d_0$) is known.

In addition, for each measurement the transmission approach gathers a complete diffracting ring, allowing for determination of the magnitude and orientation of strains relative to the sample axes for multiple crystallographic planes simultaneously, and providing redundancy against gaps in the ring due to intense texture.

## 5.2 Experimental Procedure

The current findings are the result of further analysis of previously gathered monochromatic transmission synchrotron diffraction experiment data, from which texture information has been published [8]. To summarise, samples were cut 30 mm from the start of AZ80 bead on plate friction stir welds made with the process parameters listed in Table 5.1. For these samples, experiments were conducted on the 1-ID beam line at the Advanced Photon Source, Argonne National Laboratory using diffraction volumes limited by aperture to 0.05 mm x 0.05 mm and by sample thickness to about 7.5 mm, with measurement spacing of 0.1 in the TD, and 0.1 or 0.2 mm in the ND and a wavelength of 0.14410 Å. About 6 hours were required to gather the 2100 measurements for each of HI#1 and HI#2, which includes both data gathering and stage movement, while for HI#3 over 4400 points were scanned. For all synchrotron data, multiple exposures were stacked to improve image statistics, and images were dark corrected to remove detector aberrations. The previous publication [8], provides further experimental details including the material chemistry, tooling dimensions, a schematic of the low angle transmission method used, and additional results for these samples such as optical micrographs, tensile testing results, hardness maps, and textures of the TD-ND plane.
Table 5.1: Friction stir welds ranked from greatest heat input to least, with corresponding processing parameters

<table>
<thead>
<tr>
<th>Heat input</th>
<th>Tool Rotation (RPM)</th>
<th>Welding speed (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HI#1</td>
<td>820</td>
<td>88</td>
</tr>
<tr>
<td>HI#2</td>
<td>583</td>
<td>88</td>
</tr>
<tr>
<td>HI#3</td>
<td>583</td>
<td>148</td>
</tr>
</tbody>
</table>

While multiple diffracted rings were recorded on the area detector for each measurement point, the previously published texture measurements [8] were only based on (0002) basal plane diffraction, as the low symmetry produces a single reflection per grain and therefore provides more detailed orientation information. In contrast, for the strain measurements of the current work {10\overline{1}0} prismatic plane diffraction was used, as the higher multiplicity provides a diffracted ring with fewer gaps caused by texture [117, p. 303]. For chemical analysis both (0002) and {10\overline{1}0} planes were used in concert.

One assumption frequently made during small angle transmission diffraction is that the diffracted plane normals are perpendicular to the transmitted beam. For the {10\overline{1}0} plane in the current samples, the scattering vector on which the measurements are made is actually $88.5^\circ$ to the incoming beam, yet we assume that we have measured properties in the ND-TD sample plane which simplifies calculations considerably. In the case of larger incident wavelengths this will no longer be a valid approximation, measurements will have a significant out-of-plane component, and significant modifications will need to be made to the analysis.

The analysis process used here was similar to the work of Korsunsky et al. [145], and each diffracted ring under consideration was first divided into 36 azimuthal arcs each covering $10^\circ$, which were then summed along the azimuth using Fit2D [129]. The
5.2. EXPERIMENTAL PROCEDURE

resulting 36 plots of intensity versus $d$ spacing were each fit to a constant background and Gaussian peak combination from which the parameters of maximum intensity, FWHM, and $d$ spacing at the Gaussian center were extracted, as shown graphically in Figure 5.1b using the software Fityk [146].

All low intensity data was filtered out (below 30 units for (0002) and below 20...
units for \{10\bar{1}0\}) as it is associated with poor sampling statistics, and more likely to produce erroneous values of peak position [117, p. 301]. Peak position measurements outside the range of 2.747-2.7696 Å for the \{10\bar{1}0\} plane, and outside the range of 2.577-2.604 Å for the (0002) plane were also removed. Each measurement location on the sample therefore resulted in a set of up to 36 \(d\) spacing values and azimuthal angles. FWHM values for each point were based on an intensity-weighted average of the (0002) peak width using only filtered data.

An ellipse and circle were then fit to each set of up to 36 peak position measurements using a least-squares algorithm as shown in Figure 5.1c. Due to the effects of beam deflection [144] or off-axis principal strain, fitting routines must allow a non-zero ellipse center. For the ellipse, the maximum and minimum radius and tilt angle of the principal axis were recorded, while for the circle fit the average circle radius was recorded.

A practical step by step guide to the analysis of 2D synchrotron diffraction data including samples of all software analysis macros has been published online [147]. More detailed information on the theoretical basis of diffraction measurements and the software set-up and analysis procedure is provided in that document.

5.2.1 Deviatoric Residual Strain Analysis

Once each measurement point has an ellipse and circle fitted, it is straightforward to calculate the deviatoric strains of the sample (in percent) using the equation \(100\times(r_{\text{ellipse}} - r_{\text{circle}})/r_{\text{circle}}\). This calculation is independent of chemical and hydrostatic strain changes across the sample [148] and is shown for one measurement point as Figure 5.1d.
5.2. EXPERIMENTAL PROCEDURE

5.2.2 Calculation of $d_0$, and Displacement Error

Both chemical changes and hydrostatic residual strains cause changes in diameter of the diffracted ring, and so separating them requires a calibration sample representative of the material chemistry from which type I macrostrains have been removed [92, p. 66]. Assuming that complete solutionisation of precipitates occurs in the friction stir welded nugget of the current AZ80 alloy, and using values from Hardie and Parkins [19], the (incorrect) assumption of no chemical change across the sample would result in a spurious residual strain of about 1%, completely obscuring the actual elastic residual strains.

The macrostrain free reference sample on which measurements of $d_0$, the strain free lattice parameter, are made may be in the form of a powder, small cubes of material, or a comb [21]. For cases such as welding of AZ80 where precipitate solutionisation is expected to occur throughout the weld and some of the surrounding area, accurately accounting for changes in $d_0$ in two dimensions is critical to good measurements, as described by several authors including Krawitz and Winholtz [148]. One advantage of a comb over the use of small cubes or powder is that the spatial relationships of the measured points are inherently maintained - handling hundreds of tiny cubes is a somewhat laborious process fraught with potential for error.

The calibration comb shown in Figure 5.2a was electrically discharge machined from a slice of the weld HI#3 and used to deconvolute strain and chemical information. Comb diffraction was done at the same beamline with a wavelength of 0.18972 Å and the diffraction volume limited by aperture to 0.1 x 0.1 mm, and by sample thickness to 0.7 mm.

Work by Hughes et al. [149] using an EDM comb with teeth 3 x 3 x 20 mm showed
that values of $d_0$ measured in alignment with the short direction of the comb tooth only matched those made in alignment with the long direction of the comb tooth at the comb tooth end. They concluded that macrostrains were maintained along the length of the comb tine, and that measurements of $d_0$ on a comb should be made aligned with the short tine direction for most accurate results.

To confirm this, the current dataset was analysed to see if the distance along the comb tine had an impact on the value of $d_0$ calculated. It was determined that the measurements aligned with the sample TD were nearly identical to those in the sample ND at the tip of each comb tine, but diverged significantly after about 0.6 mm from the tip, validating the results reported by Hughes et al. [149]. To address this concern, $d_0$ calculations were based on an average of only those six measurements from azimuthal angles of $0^\circ \pm 10^\circ$ and $180^\circ \pm 10^\circ$ and thus mainly aligned with the short dimension of the comb tine. Calculated values of $d_0$ for $\{10\overline{1}0\}$ diffraction were then interpolated across the weld, with the results shown as Figure 5.2b representing the change in unit cell parameter from solutionisation of the precipitates during welding.

By comparing the diameter of the diffracted rings from the comb to those of the
sample, chemical effects are compensated for, and so the hydrostatic strains may be determined, combined with the deviatoric residual strains, and the total residual strains of the sample calculated.

Comparing the results for average radius of the fitted circles between the comb and sample base material, a systematic sample shift exactly matching a displacement of half the sample thickness was found to have occurred. It was determined that during setup of the samples, the sample-detector distance was calibrated to the front of the sample face, while the centroid of the diffracting volume is actually equal to the sample mid-thickness. To correct for this, the diffraction results for each sample were scaled such that the mean value in the base metal for the samples was equal to the mean value in the base metal of the transverse comb (an addition of 0.004563 Å for HI#1, 0.004094 Å for HI#2, and 0.005591 Å for HI#3).

5.2.3 Chemical Analysis Procedure

Given experimental measurements of the Debye-Scherrer rings in a strain free sample, the calculated \( d \) spacing of the unit cell may be used in conjunction with literature values to calculate the average composition of the diffracting volume. This method has been commonly used to determine composition of binary alloys [116, pp. 388], over volumes which in the case of synchrotron diffraction could easily be less than 0.01 mm\(^3\). This allows for much finer sampling than typical chemical methods which require samples in the neighbourhood of several grams. What is shown here is a proof of concept for extension of the binary technique to ternary systems for non-cubic alloys, by making use of the asymmetry of the crystal system as an independent measurement.
5.2. EXPERIMENTAL PROCEDURE

This diffraction approach has two main limitations, namely that hydrostatic strains must be accounted for, and that the maximum number of elements that can be measured is limited by the crystallographic symmetry of the sample. Each independent axis of the unit cell provides one equation for the linear system. In a cubic sample, there is only one independent crystallographic axis as all sides of the unit cell are equal in length, and so the composition of only one element may be determined (i.e. binary alloys). In HCP alloys such as magnesium, the $c$ and $a$ axes are independent, and thus up to two elements can be determined, while for orthorhombic it should theoretically be possible to extract the variation in composition of three elements across the sample. Of course, the effect of any additional elements is unaccounted for and may prove a significant source of error depending on content and effect on unit cell dimensions. One additional caveat is that for each axis the percent change in length with atomic percent alloy addition must be different to result in a unique solution to the system of equations. While there are also potential complications such as those that stem from multiple phases (see [150] for a discussion of this in the cubic system), that is not an issue with the current alloy series.

Compared to the $d$ spacing method described above, electron based techniques such as EDS (Energy Dispersive X-ray Spectroscopy) have finer maximum resolution in exchange for a far smaller depth of penetration. For example, in magnesium EDS is limited to a depth of about 5 $\mu m$ at 20 keV [119] in comparison to several mm for the $d$ spacing method. EDS also has the advantage of simultaneously extracting and categorizing data from multiple elements in a qualitative fashion but for accurate quantitative results calibration targets are necessary. While requiring good surface preparation, EDS is also more easily available for research purposes as it is routinely
installed in most modern scanning electron microscopes.

Two advantages of the \( d \) spacing method as compared to destructive chemical analysis methods are the low volume of material required, and the ability to differentiate between elements precipitated and those dissolved in the matrix. When investigating the precipitation or dissolution of phases by diffraction, the \( d \) spacing method would be a valuable complement to the technique of checking for the presence of diffraction peaks. While the presence of diffraction peaks associated with a precipitating phase is a definitive indication of the presence of a coherent phase, for small quantities of precipitate the peaks will be low in intensity and therefore difficult to separate from background noise. If the two approaches are combined, the simultaneous decrease in solutionised phase measured by the \( d \) spacing technique in conjunction with an increase in the intensity of the diffracted precipitate peaks could be used to more precisely detect the onset of precipitation.

The results demonstrated here are based on measurements of two binary systems (Mg-Al and Mg-Zn). It is highly likely that that the ternary system axis lengths will not be a simple linear rule of mixtures and that calibration samples will be necessary before accurate measurements can be made. Effectively, a ternary ‘phase diagram’ of axis lengths for both crystallographic axes may be necessary to fully develop the technique. Hopefully this calibration process may be short-cut considerably by using atomistic level simulations to bridge the gaps between known values.

To determine the chemical composition of the diffracting volume, for each measurement point in the comb the average \( d \) spacings for the \{10\bar{1}0\} and (0002) diffracting planes were compared to those published by Hardie et al. [19] for pure magnesium. These measured spacings of the (0002) planes are doubled to determine the spacing
of the magnesium (0001) plane, which does not diffract. Given literature values for the rate of change in each plane spacing with alloying content [19], a system of two linear equations to be solved for the weight percent Al and Zn could be written (all units in Å).

\[
\begin{bmatrix}
-54.0 \times 10^{-4} & -80.6 \times 10^{-4} \\
-41.1 \times 10^{-4} & -48.1 \times 10^{-4}
\end{bmatrix}
\begin{bmatrix}
x_1 \\
x_2
\end{bmatrix}
= \begin{bmatrix}
m_1 - 5.2108 \\
m_2 - 3.2099
\end{bmatrix}
\] (5.1)

The leftmost term is the change in unit cell axis length per atomic percent addition (Al at left, Zn at right, (0001) axis on top, \{11\bar{2}0\} axis on bottom), \(x_1\) and \(x_2\) represent the atomic percent Al and Zn respectively, \(m_1\) and \(m_2\) are the diffraction measurements of length of the (0001) and \{11\bar{2}0\} plane \(d\) spacing respectively, and the values at right represent the axis lengths for pure magnesium ((0001) axis on top, \{11\bar{2}0\} axis on bottom).

5.2.4 Hydrostatic Residual Strain Analysis

Since the strain in the WD cannot be measured in the current experimental configuration, it is not possible to calculate the true hydrostatic strain. We can however calculate the in-plane hydrostatic strain across the weld by comparison of the sample measurements to those of the strain-relieved comb.

The experimentally measured comb values of \(d_0\) were manually aligned with those of each sample based on the nugget weld profiles, and for each measurement location in the sample a corresponding local value of \(d_0\) was interpolated from the comb. Strain was then calculated in percent using the equation \(100(\frac{d_{\text{sample}} - d_0}{d_0})\). Some potential sources of error from using a single comb made from HI\#3 for three welds
include minor misalignments of the weld profiles, changes in the shape of the weld profile with processing condition as shown in previous work [8, p. 192], and changes in the values of $d_0$ with welding parameter change. Alignment errors were calculated by overlaying all weld interfaces on the comb and measuring the maximum difference in hydrostatic strain between each point on the comb interface and each weld interface in an analogous location. This method gave a maximum hydrostatic strain error of 0.028 % in HI#1, 0.023 % in HI#2, and 0.013 % in HI#3 at one point on the weld interface, decreasing away from that location. Irrespective of these concerns this method should remain a significant improvement even over the use of multiple separate $d_0$ standards, as has been done elsewhere in the literature [148].

5.2.5 Residual Stress Analysis

It is worth emphasising that the stresses calculated in any small sample removed from a friction stir weld will be significantly relaxed as compared to the full welded sample [89, 90], for precisely the same reasons that the macrostresses in the reference comb are expected to be minimal. Consequently, stress distributions are of greater interest and repeatability than absolute stress values.

For the current stress analysis, we assume firstly that the sample is in a plane stress situation, and secondly that the AZ80 magnesium sample may be treated as elastically isotropic, an assumption supported by the work of various authors [26, 22, 24].

The assumption of plane stress is a less than ideal one, due to the external dimensions of the samples being approximately 19 mm in TD by 6 mm in ND by 7.5 mm in WD. In other words, the sample has a thickness greater than its height. Further, literature values such as those of Woo et al. [97] indicate that stress in the WD of an
AZ31 magnesium friction stir weld is close in magnitude to that in the other sample directions, and therefore far from negligible. However assuming plane strain (not shown here), which is the opposite bounding case, results in a near-identical spatial distribution of stress. Comparing identical locations, switching between the plane stress and plane strain assumption causes changes in stress magnitude of about 10-20 MPa. Since these stress magnitudes are not expected to be representative of those in a full uncut weld, this shows that selecting between the plane stress and plane strain assumption has little impact on the main features of the results reported here.

For analysis purposes, we designate the major axis of the strain ellipse fitted to each measurement point as $1$ and the minor axis as $2$ (see Figure 5.1c). The axis perpendicular to these (out of plane, and not measured) is $3$. Assuming complete elastic isotropy of the material, the strain axes are aligned with the stress axes, and so the stress ellipse will be identical in shape to the strain ellipse shown in Figure 5.1c, but scaled in magnitude. Although strain axes $1$ and $2$ are not aligned with the sample axes, the in-plane angle of rotation is known from the strain ellipse fitting operations described earlier.

For each principal axis $i=1,2,3$ we can calculate the stress using the equation (notation adapted from [21, eq 5.2]):

$$
\sigma_i = \frac{E}{(1 + \nu)}[\varepsilon_i + \frac{\nu}{(1 - 2\nu)}(\varepsilon_1 + \varepsilon_2 + \varepsilon_3)]
$$

(5.2)

Under the plane stress assumption we define $\sigma_3 = 0$, at which point the left side of the equation is zero, $\varepsilon_1$ and $\varepsilon_2$ are measured experimentally, and $\varepsilon_3$ can be calculated. At this point $\sigma_1$ and $\sigma_2$ are solved for using analogous equations and then resolved along the sample axes using the known tilt angle. For the bulk elastic modulus value
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(E), the mean of six experimental measurements on full-size tensile samples cut from a different lot of extruded AZ80 sheet was used with two samples cut parallel to the extrusion direction, two perpendicular, and two at 45°. For the Poisson’s ratio $\nu$, the estimation of the mean polycrystalline value from Tromans [22, p. 478] was used.

5.2.6 FWHM

The broadening of a diffracted peak measured from the FWHM as shown in Figure 5.1b is a function of numerous microstructural factors working on a length scale lower than the diffracting volume (for a comprehensive list see Ungár [151]). The most significant contributors are expected to be dislocations, microstrains due to intergranular effects, and chemical heterogeneities. In friction stir welds, works on this topic are limited to that of Woo et al. [152] who made linear measurements across an AA6061-T6 friction stir weld, and the work of Steuwer et al. [153] who measured a grid of points spanning an AA7010 friction stir weld. The present work was the only one found comparing FWHM between friction stir welds made at a range of processing conditions.

Woo et al. [152] analysed FWHM data for multiple diffracting planes, and concluded that the dislocation density in the nugget decreased by over 70 % due to recrystallisation as compared to the base material. While another potential contributor to FWHM changes is microstrains, these are expected to be a secondary factor to dislocation density in any case where plastic deformation has occurred after recrystallisation. In recrystallised material where plastic strain has not occurred, microstrains are less significant in cases of high texture intensity and low elastic and thermal anisotropy. Given the high texture intensity in magnesium, and the low elastic and
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thermal anisotropy, microstrains are expected to contribute only in a limited fashion. The relative impact of chemical heterogeneity on the FWHM is difficult to assess, but potentially significant in the current case. To isolate this effect, comparison to a sample that was solution heat treated prior to friction stir welding at the same conditions would be required.

5.3 Experimental Results and Discussion

5.3.1 Chemistry

Histograms of the aluminum and zinc concentrations for the strain-relieved comb were calculated in weight percent and plotted as Figure 5.3. While the mean value of aluminum content for the comb calculated (6.33 wt% Al over 589 points) from the $d$ spacing is close to the bulk material composition of 7.75 wt% Al, the calculated values for zinc were less realistic, and as low as -8.74 wt% Zn, a physical impossibility. The overall comb mean value of 2.5 wt% Zn calculated over 589 points is somewhat higher than the bulk material composition of 0.65 wt% Zn. The unrealistic values for zinc are likely due to the lack of ternary calibration samples for this work, rather than other sources of error such as interference from additional elements.

The mean concentration of aluminum calculated for the weld nugget region (6.83 wt%) was greater the bulk material aluminum content of 6.33 wt% Al, which was nearly identical to the calculated base metal aluminum content (6.26 wt% Al). The histogram of aluminum concentration in the weld region shows lower spread of results than the base material indicating the material was more homogeneous in this area. These results are in agreement with the expected dissolution of the Mg$_{17}$Al$_{12}$ precipitate in the friction stir weld nugget discussed in Section 5.1.4.
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Figure 5.3: Histogram of the alloy content in the comb for Al and Zn. Mean of all comb calculations (vertical red line) and the measured value for the base material (vertical blue line) indicated for each element and labelled on the plot. (left) Al (right) Zn.

The preliminary assessment conducted here indicates that this experimental method is a useful method of locating precipitate dissolution across a weld with good spatial resolution and fine sampling volume. However, for reasonable levels of accuracy, literature values for $d$ spacing change with single element additions are likely to be insufficient. Calibration samples are most likely required, particularly when working with elements present in lower concentration.

5.3.2 In Plane Hydrostatic Residual Strain

The in-plane residual hydrostatic strain was calculated and plotted as Figure 5.5 for three friction stir welding conditions. Most clearly seen in HI#1 and HI#2, the level of hydrostatic strain in the center of the weld nugget and the base metal are similar. At the root of HI#1 there is a large concentration of compressive residual strain, and a narrow band of compressive strain is visible at the root of HI#3 as well.
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Figure 5.4: Histogram of the weight percent aluminum content in two regions of the comb. Mean of all comb calculations (vertical red line) and the histogram mean (vertical blue line) labelled for each plot. (left) 288 points from the base material spanning a difference of 9.55 wt% Al (right) 97 points from the friction stir weld nugget spanning a difference of 3.81 wt% Al.

The difference in processing conditions between HI#1 and HI#2 is solely the rate of tool rotation, indicating that changes in RPM without changes in welding speed can produce notable changes in residual strain distribution. Corresponding to the decrease in temperature of the friction stir welding conditions from HI#1 to HI#3, strains along the AS interface change from slightly compressive (HI#1) to slightly tensile (HI#2), and then completely tensile (HI#3), becoming increasingly asymmetric. In general, there are no major strain changes across the RS interface, and the values in that location for all three processing conditions were similar. Examining the overall strain, decreasing heat input caused the average hydrostatic strain for the samples to become increasingly tensile.

Looking at the distribution of in-plane hydrostatic strains in the base material of HI#3, it is evident that there was a minor bias between the left and right sides of
5.3. EXPERIMENTAL RESULTS AND DISCUSSION

Figure 5.5: In-plane hydrostatic strains in %. Dashed black line indicates weld, with AS to the left, RS to the right. Negative values indicate compression, and positive values indicate tension while the white contour indicates zero strain. Axis tics mark 1 mm spacing. (top) HI#1 88 mm min\(^{-1}\) 820 RPM, (mid) HI#2 88 mm min\(^{-1}\) 583 RPM, (bottom) HI#3 148 mm min\(^{-1}\) 583 RPM.

The sample which is not visible in either of the other samples. Possible sources of this effect include alignment error of the sample plane relative to the beam, or unequal sample thickness.

5.3.3 Deviatoric Residual Strains

A good understanding of deviatoric strains is more critical for applications as compared to hydrostatic, as deviatoric strains have greater influence on the onset of
plastic flow [21, p. 206]. For deviatoric strain calculations the use of \( d_0 \) values is not required [21, p. 206], removing multiple potential sources of error. Comparing the deviatoric residual strains shown in Figure 5.6 to the hydrostatic residual strains shown in Figure 5.5, we can see that in the base material of HI#3 the deviatoric strains are equal between the left and right side of the sample. This shows that as expected, the source of error that caused this apparent asymmetry effect has been eliminated from the calculation of deviatoric strains.

![Figure 5.6: In-plane deviatoric strains in %](image)

Figure 5.6: In-plane deviatoric strains in %. Dashed black line indicates weld interfaces, with AS to the left, RS to the right. Negative values indicate compression, and positive values indicate tension while the white contour indicates zero strain. Axis tics mark 1 mm spacing. (top) HI#1 88 mm min\(^{-1}\) 820 RPM, (mid) HI#2 88 mm min\(^{-1}\) 583 RPM, (bottom) HI#3 148 mm min\(^{-1}\) 583 RPM.

All deviatoric strain plots are symmetric, irrespective of heat input. The TD deviatoric strain plots at the left side of Figure 5.6 show that the top of each nugget is in deviatoric compression, with the compressive region extending down both the AS and RS interface to approximately equal extent with increasing depth and magnitude.
as the heat input decreases. The rest of the weld and base material is in deviatoric tension to a depth greater than that of the weld root, with maximum values near the mid-depth of the nugget.

In HI#3 near the nugget RS, zones of high transverse compressive and tensile deviatoric strain are in close proximity, as indicated by an arrow in Figure 5.6. Such high contrasting strain regions would be nearly impossible to locate using traditional $2\theta$ diffraction techniques due to the larger diffracting volumes, and may indicate the onset of a defect.

For the ND deviatoric strain plots, the strain distribution is the exact inverse of the TD results, with tensile values at the top of the nugget, extending down the weld interfaces. This is a consequence of mapping in-plane deviatoric strains, as the fitted ellipse from which deviatoric strain is calculated will always have the major and minor axes perpendicular, and so regions of high ND deviatoric strain will be matched with regions of low TD residual strain.

### 5.3.4 Deviatoric Residual Strains as a Vector

The current experimental data provides the capability to plot vectors representing the maximum tensile in-plane deviatoric strain magnitude and direction relative to the sample axes. Under the assumption that the residual deviatoric strains are dominated by thermal contraction effects, this allows us to correlate the vector orientation to the direction of thermal contraction and the vector magnitude to the thermal gradient following friction stir welding.

At the top of the weld, tensile strain was high and oriented largely in the ND, indicating that heat transfer in this region was mainly vertical, and relatively rapid.
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Figure 5.7: In-plane deviatoric strains in samples processed at various welding conditions. Vector shows major deviatoric strain direction with length scaled to magnitude. $180^\circ$ rotations equivalent. Dotted red line indicates weld, measurements are 0.1 mm apart in the TD, and 0.2 mm apart in the ND for Heat input 1-2, 0.1 in ND for Heat input #3. (top) Heat input #1 88 mm min$^{-1}$ 820 RPM, (mid) Heat input #2 88 mm min$^{-1}$ 583 RPM, (bottom) Heat input # 3 148 mm min$^{-1}$ 583 RPM.

This is in agreement with the high temperatures expected at the top of the friction stir weld [84]. Within the nugget center, deviatoric strain is much smaller, consistent with a lower thermal gradient allowing more time for strain relaxation, and many of these vectors are horizontal, indicating that the thermal gradient was in the TD. Vectors along the interfaces tend to be smaller than those at the top of the nugget.
5.3. EXPERIMENTAL RESULTS AND DISCUSSION

but larger than those in the center of the nugget, indicating an intermediate rate of cooling in these locations. Interface vectors are largely oriented at about 45° from the ND-TD axes indicating contraction was approximately equal in both the ND and TD.

All of the friction stir welds show evidence of limited asymmetry in this plot, for example in HI#1 compare the AS interface near the root, where heat transfer is largely horizontal to the RS where there is a greater ND component.

Within the base material, deviatoric residual strains are inhomogeneous. While the HAZ is not visible optically for these samples [8], it may be seen in Figure 5.7 as the region of deviatoric residual strains homogeneously oriented outside the weld interfaces, as is particularly clear for HI#3. The HAZ extends approximately 0.5 mm vertically below the weld root at the centreline, and as far as 2.7 mm in the TD at plate mid thickness, with vectors oriented to indicate that most of the thermal contraction in the HAZ was in the TD.

In precipitation-hardenable aluminum alloys, the HAZ is of critical importance to deformation behaviour as the added heat input from friction stir welds frequently renders this microstructural region over-aged and prone to failure [112]. For AZ alloys, fracture will generally occur at the AS interface due to the textures here [8, 72], and so the HAZ is of far less general importance.

5.3.5 FWHM Changes Across the Nugget

Mentioned previously, dislocation density, microstrains, and compositional heterogeneity are expected to be the main contributors to the FWHM plots shown as Figure 5.8. The recrystallised nugget would have lower dislocation density, higher texture
intensity, and a more homogeneous composition, and so in all three cases, the peak broadening effects would be expected to be smaller in the nugget than in the base material, matching the current results.

The band of high FWHM along the AS (and to a lesser extent along the RS) of HI#1 is not well explained by changes in dislocation density across the sample. Two possible explanations for this feature are local plastic deformation from thermal contraction, or locally high microstrains due to the abrupt change in texture along this interface.

In the base material under the nugget root of all FWHM maps there is a region

Figure 5.8: FWHM of (0002) in Å as an indication of intergranular strain. Dashed black line indicates weld, with AS to the left, RS to the right. Axis tics mark 1 mm spacing. (top) HI#1 88 mm min$^{-1}$ 820 RPM, (mid) HI#2 88 mm min$^{-1}$ 583 RPM, (bottom) HI#3 148 mm min$^{-1}$ 583 RPM.
of high FWHM which may be associated with the friction stir welding tool forces. Given the starting texture of the base material, the (0002) axis is largely aligned with the ND, and poorly oriented for basal slip.

The current high-resolution maps make a valuable starting point for TEM work in friction stir welding, directing researchers to areas of interest based on a preliminary assessment of the dislocation density. For example, TEM work on the AS interface of HI#1 would explain the source of peak broadening and is likely of greater interest than work on HI#3 in the same location. Conversely if a researcher wished to examine an area of low dislocation density, the nugget root is likely to be of greater interest than other locations of the nugget.

If the FWHM is indeed closely associated with dislocation density, there are indications that greater deformation occurred in a band across the nugget than at the top or root of the nugget. This is consistent with reports that the flow in this region is periodic rather than continuous (e.g.[52]), and that the material in this location is compacted behind the tool as a periodic layer [16].

5.3.6 Residual Stress Data

For comparison purposes with available literature results, the total residual stresses comprised of the sum of the in-plane hydrostatic and deviatoric stresses are shown as Figure 5.9. Unsurprisingly many of the features in the hydrostatic and deviatoric results are visible here as well.

We see again that the residual stress distribution does not match the predicted local thermal gradients, as we would expect a stress concentration at the top of the RS interface, which is not observed.
5.3. EXPERIMENTAL RESULTS AND DISCUSSION

Figure 5.9: In-plane total stresses in MPa based on plane stress assumption. Dashed black line indicates weld, with AS to the left, RS to the right. Negative values indicate compression, and positive values indicate tension while the white contour indicates zero strain. Axis tics mark 1 mm spacing. (top) HI#1 88 mm min$^{-1}$ 820 RPM, (mid) HI#2 88 mm min$^{-1}$ 583 RPM, (bottom) HI#3 148 mm min$^{-1}$ 583 RPM.

In agreement with the work of Lombard et al. [78], higher heat input was found to decrease the magnitude of residual stresses. However, in contradiction to that same work it was found that higher welding speeds promoted asymmetry (compare HI#1 and HI#2), and reduced heat input from reduced tool RPM likewise increased asymmetry (compare HI#2 and HI#3). These changes in residual stress with processing condition are in agreement with the changes of the thermal profile, and the discrepancy is probably attributable to the tighter measurement spacing of the current work.

It is also shown that the stresses along the advancing side interface can change from tensile to compressive with processing condition (compare HI#1 to HI#2 to HI#3). This result is particularly useful, as it explains why reports of the transverse stress distribution have tensile peaks in some cases (e.g. [78, 97, 99]) and compressive
peaks in others (e.g. [94, 100]).

Since applied stresses are cumulative with residual stresses, we expect samples with a higher TD tensile residual stress to yield at lower stresses when loaded in the TD. Similarly, except for one sample which fractured in the grips, all of the tensile samples cut from welds of HI#1,2&3 failed at the AS interface, and so we expect higher residual stresses at the AS to correlate with lower ultimate tensile strength. Comparing the average tensile results for these samples [8] to the current calculations for residual stress, there is an excellent correlation of both our predictions with the experimental results. While increasing residual stresses with lower heat inputs is a highly plausible explanation for the reduction in mechanical properties with lower heat input observed here, a larger number of measurements would be required to draw definitive conclusions.

Figure 5.10: (orange data) Maximum residual stress at the AS interface versus tensile yield strength (blue data) Maximum residual stress versus ultimate tensile strength for samples welded at (circle) HI#1 88 mm min\(^{-1}\) 820 RPM, (x mark) HI#2 88 mm min\(^{-1}\) 583 RPM, (triangle) HI#3 148 mm min\(^{-1}\) 583 RPM.
5.3. EXPERIMENTAL RESULTS AND DISCUSSION

As a final note, it is worth comparing the residual stresses to the partial texture information gathered during the same synchrotron diffraction experiment, published previously as [8]. Examination of the HAZ vector measurements of Figure 5.7 shows that thermal contraction around the weld was largely in the TD. Based on measurements of these welds [8], textures located in the nugget root, slightly towards the AS from the centerline are resistant to deformation in the TD. Conversely in the upper part of the nugget towards the RS, and along both interfaces (with a greater extent on the AS) textures will be weak to deformation in the TD. If texture and residual stress are correlated, we would expect to see high residual stress near the nugget root, and low residual stress at the AS interface, which is the opposite of the results observed here.

Based on the current results, it is probable the areas of high residual stress are independent of texture effects, supporting the conclusions of Webster et al. [101], over those of Woo and Choo [96]. It is therefore likely that residual stresses are dominated by the thermal history to the point where texture has little effect. Given the higher residual stresses at cooler welding conditions it is likely residual stresses are proportional to the thermal gradients of the weld, rather than the peak temperatures of the friction stir weld.

The residual stress magnitudes reported by Woo et al. [97] in AZ31 are completely different from the current results, showing transverse residual stresses of up to 180 MPa in tension and -20 MPa in compression, as compared to the current peak residual stress values of about 40 MPa in tension or compression. This disparity is expected to be due to sample sizes used, as those used for the current measurements were relatively small while those of Woo et al. [97] were made halfway along a continuous
300 mm friction stir weld.

5.4 Conclusions

The previous practice of measuring the composition of binary alloys by diffraction has been extended to additional elements, and has many potential applications particularly in metals of lower crystallographic symmetry, where the technique could theoretically be extended to quaternary alloys. Not previously evident in literature, this modified technique offers an avenue for improvement in the detection of the onset of precipitate formation or dissolution, but requires calibration with samples of known composition for absolute value measurements.

The current high-resolution stress and strain maps have shown several features that would not be detectable by line scans, including concentrations in compressive stress in the weld root of higher heat input welds. Comparison of residual stress between three friction stir welding conditions has shown how the stresses at the interface can reverse from compressive to tensile with decrease in the heat input, explaining a significant disparity in literature results.

Independent examination of the hydrostatic and deviatoric components of residual strain has shown that hydrostatic strains are asymmetric and show high strain concentrations at the AS interface but not the RS, while deviatoric strains are mostly symmetric and show increasing magnitude at both AS and RS interface as temperature decreases.

The current work has confirmed that the spatial distribution of residual strains does not well correspond to the thermal gradient or nugget texture, and that both
asymmetry and peak residual strain magnitude increase as heat input decreases, either from increasing welding speed or decreasing tool rotation rate. Residual stress increases in the welds were found to be consistent with lower yield and ultimate tensile strength values, indicating that residual stress has a direct and measurable impact on mechanical performance. While avoiding the formation of liquid films in the nugget [45], selecting hotter manufacturing conditions to minimize residual stresses is expected to improve the mechanical performance of AZ80 friction stir welds.

5.5 Acknowledgements and Funding

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Chapter 6

Strain localisation and failure of dissimilar magnesium AZ series friction stir welds under transverse load

J. Hiscocks, B. J. Diak, A. P. Gerlich and M. R. Daymond

Abstract
Dissimilar butted combinations of magnesium alloys AZ31, AZ61, and AZ80 were friction stir welded and tensile tested under transverse tensile load until failure. 2D mapping of strain across the deforming weld nugget and interfaces was carried out to correlate the deformation path to the local microstructures. Mechanical performance was found to be highly dependent on the spatial distribution of alloys within the nugget, and thus highly sensitive to changes in the tooling geometry. This finding indicates that process control requirements for repeatable, high-quality dissimilar friction stir welds will be far more rigorous and challenging than those of similar welds of the same alloys. In addition, proximity to the point of tool insertion was found to decrease tensile performance, likely due to changes in tooling temperature.
6.1. Introduction

Joining of dissimilar magnesium alloys broadens the potential applications by tailoring properties locally to the anticipated loading while maintaining the characteristic light weight advantage. The AZ series magnesium alloys are not considered fusion weldable \cite{7} due to the formation of a brittle intermetallic phase, and so solid-state techniques such as friction stir welding are preferable.

While AZ series alloys are the most widely commercially available magnesium structural alloys, dissimilar friction stir welding research in this series is limited. Extrapolation from the wider body of information available for dissimilar aluminum friction stir welds is of limited use as many of the Al alloys are age hardenable, affecting the deformation and failure in ways that are not transferable to the magnesium AZ series, where age hardening has limited impact and much slower kinetics (\cite{36, 40}).

Within the AZ series magnesium alloys, AZ31, AZ61, and AZ80 are all extrudable, resulting in a fine wrought structure and better mechanical properties than casting. With increasing weight percent of aluminum content in the alloy, there is an increase in mechanical strength at the cost of ductility reduction. One aim of the current work is to investigate how the processing conditions affect achievable mechanical properties, with the aim of eventually improving them.

Of primary importance to the final mechanical properties is the selection of the alloys to be joined, as the base material properties determine the maximum achievable performance of the friction stir weld. Given a wrought starting microstructure, the
properties of a dissimilar weld will be at best intermediate to the two base materials, or at worst, inferior to both. One example of this is the work of Liu et al. [14] who found their dissimilar AZ80-AZ31 friction stir welds had a UTS below that typical for either of the base materials used [2].

Although various authors have measured a lag before steady-state temperatures are reached around the friction stir welding tool [82, 80, 81, 86], there is little information on the effect of this initial thermal transient on mechanical properties. Work by Bitondo et al. [154] reported that this transient had little effect on the mechanical properties in an AA2198 friction stir weld, however no experimental work on this phenomenon in magnesium alloys has been found.

Tool wear has a significant effect on the final material properties of a friction stir weld due to the erosion of both pin length and features which promote stirring [104]. These features include the threads of the pin which force downward motion of the softened material [58] and improve material flow around the pin [103]. The resulting lack of downward force contributes to the formation of defects at the weld root [104] which include wormhole defects. However, there are few reports of this mechanism at intermediate stages to the formation of large defects, raising questions as to the effect of wear on the microstructure and mechanical properties prior to the formation of wormholes.

The purpose of this work is to investigate the effect of base material choices, distance from the start of the weld, and wear effects with the goal of providing a basic set of ‘best practices’ for production and development of dissimilar magnesium friction stir welds with good mechanical performance, particularly for the AZ series alloys.
6.2. EXPERIMENTAL PROCEDURE

Table 6.1: Principal alloying elements (balance Mg) in wt% for the extruded sheet materials of the current study, as tested with an inductively coupled plasma-atomic emission spectrometer. Content of other tested elements (Zr, Be, Cu, Fe, Ni, Pb, Si) was below 0.02 wt%.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Aluminum</th>
<th>Zinc</th>
<th>Manganese</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ31</td>
<td>2.67</td>
<td>0.77</td>
<td>0.32</td>
</tr>
<tr>
<td>AZ61</td>
<td>7.12</td>
<td>0.42</td>
<td>0.35</td>
</tr>
<tr>
<td>AZ80</td>
<td>8.77</td>
<td>0.44</td>
<td>0.19</td>
</tr>
</tbody>
</table>

6.2 Experimental Procedure

6.2.1 Materials and Tooling

The base material consisted of extruded sheets 4.9 mm thick by 15.2 cm wide. The alloys AZ31, AZ61, and AZ80 had measured chemical compositions as shown in Table 6.1. Various alloy sheet combinations were split along the centerline and butted parallel to the extrusion direction for welding. Friction stir welding conditions used a rotation rate of 900 RPM, tool tilt angle of 2°, and either 63 or 90 mm/min welding speed. Tool geometry was as shown as Figure 6.1, and to assess the impact of tooling wear on defect formation and mechanical properties of the friction stir weld, a version of Tool B worn down by friction stir welding of metal-matrix composites was used for some welds.

To assess the relationship between strain localisation and the microstructure, DIC (digital image correlation) was used to calculate strain across the sample based on images captured during tensile testing. Tensile samples were waterjet cut transverse to the extrusion direction, centered on the weld, to the dimensions of sheet-type specimens (most samples) or subsize specimens (one sample as noted) [155].

When cut, samples became curved from the partial relief of transverse residual
6.2. EXPERIMENTAL PROCEDURE

Figure 6.1: Triflat tools used in this study, with truncated conical pins, threaded outer surface, and three 0.5 mm flats. All dimensions in mm. Tool A (shown) resulted in a lack of penetration defect. Tool B, with no shoulder recess and pin length increased to 5 mm produced higher quality welds. Tool C was machined to the same design as B.

strains as detailed in [91, p. 8110, 8123]. While the axis of this bend was perpendicular to the plane of DIC examination, it caused non-linearities in the initial loading curves, and so all strain values were offset to ensure stress-strain curves begin at zero. It is expected that this initial sample curvature will prevent accurate determination of yield points, and so strain localisation of 2 % locally as measured from DIC is used instead.

To accommodate simultaneous imaging of the weld microstructure and strain mapping, samples were first mechanically polished in the gauge length to a minimum finish of 1 µm before immersion etching in 1 g tartaric acid dissolved in 20 mL water for approximately 10 s to reveal the weld cross section. Speckles were applied in a random pattern over the gauge length for DIC strain tracking purposes, with the optimum distribution found to result from matte black spray paint with a fan-type nozzle.

Initial DIC measurements used a Xenoplan 2.8/50-0902 lens to capture images during tensile testing, resulting in a spatial strain measurement resolution of approximately 0.9 mm². Later measurements used a telecentric lens with a 17 mm field of view, resulting in a final spatial strain measurement resolution of approximately 0.24
6.2. EXPERIMENTAL PROCEDURE

Strain analysis was performed using Matlab R2016a and the DIC code by Jones [156]. Comparison of DIC and extensometer measurements on multiple samples gave excellent agreement, and so for samples where no extensometer data was available strain and strain rates were measured from DIC data. Strain rates reported are averaged between the plastic 0.2 % offset yield and failure.

For comparison purposes, two base metal samples of each composition were waterjet cut from the extruded sheet transverse to the extrusion direction in the ASTM sheet-type dimensions, and tested to failure with the results shown in Table 6.2.

Table 6.2: Base material transverse tensile properties. Values shown are the mean of two samples.

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
<th>Elastic Modulus (GPa)</th>
<th>Strain rate (s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ31</td>
<td>113</td>
<td>244</td>
<td>16.2</td>
<td>39</td>
<td>4.0 ×10⁻⁴</td>
</tr>
<tr>
<td>AZ61</td>
<td>203</td>
<td>309</td>
<td>17.8</td>
<td>43</td>
<td>4.8 ×10⁻⁴</td>
</tr>
<tr>
<td>AZ80</td>
<td>198</td>
<td>340</td>
<td>15.4</td>
<td>42</td>
<td>4.1 ×10⁻⁴</td>
</tr>
</tbody>
</table>

As a convention, welds are shown with the advancing side (AS) to the left, and described with the AS side material listed first, followed by the retreating side (RS) material. An S- suffix indicates the sample number, and Table 6.3 lists the mechanical properties of all friction stir welds discussed in addition to the tool used to make these welds. Weld centerlines are marked on all micrographs.
Table 6.3: Dissimilar friction stir welded combinations. Strain localisation is based on a level of 2% in or near the weld. Joint efficiency is based on UTS comparison to the weaker of the two dissimilar composing alloys, note that all welded samples failed at the UTS. Distance to start measured from the farthest shoulder contact to the closest point on the tensile sample.

<table>
<thead>
<tr>
<th>Sample (RS-AS-#)</th>
<th>RPM</th>
<th>Welding speed (mm/min)</th>
<th>Strain localisation (MPa)</th>
<th>UTS (MPa)</th>
<th>Joint efficiency (%)</th>
<th>Tool and wear state</th>
<th>Distance to start (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ80-AZ31-S1</td>
<td>900</td>
<td>89</td>
<td>110</td>
<td>198</td>
<td>81</td>
<td>New Tool A</td>
<td>9.6</td>
</tr>
<tr>
<td>AZ80-AZ31-S2</td>
<td>900</td>
<td>89</td>
<td>106</td>
<td>203</td>
<td>83</td>
<td>New Tool A</td>
<td>18.0</td>
</tr>
<tr>
<td>AZ80-AZ31-S3</td>
<td>900</td>
<td>63</td>
<td>90</td>
<td>205</td>
<td>84</td>
<td>New Tool B</td>
<td>0.8</td>
</tr>
<tr>
<td>AZ80-AZ31-S4</td>
<td>900</td>
<td>63</td>
<td>99</td>
<td>230</td>
<td>94</td>
<td>New Tool B</td>
<td>3.3</td>
</tr>
<tr>
<td>AZ80-AZ31-S5</td>
<td>900</td>
<td>63</td>
<td>109</td>
<td>247</td>
<td>101</td>
<td>New Tool B</td>
<td>5.8</td>
</tr>
<tr>
<td>AZ80-AZ31-S6</td>
<td>900</td>
<td>63</td>
<td>75</td>
<td>216</td>
<td>89</td>
<td>New Tool C</td>
<td>11.3</td>
</tr>
<tr>
<td>AZ80-AZ31-S7</td>
<td>900</td>
<td>90</td>
<td>79</td>
<td>228</td>
<td>93</td>
<td>New Tool C</td>
<td>7.0</td>
</tr>
<tr>
<td>AZ80-AZ31-S8</td>
<td>900</td>
<td>90</td>
<td>96</td>
<td>178</td>
<td>73</td>
<td>Mod worn B</td>
<td>6.4</td>
</tr>
<tr>
<td>AZ80-AZ31-S9</td>
<td>900</td>
<td>90</td>
<td>98</td>
<td>107</td>
<td>44</td>
<td>High worn B</td>
<td>14.2</td>
</tr>
<tr>
<td>AZ31-AZ31-S10</td>
<td>900</td>
<td>90</td>
<td>56</td>
<td>157</td>
<td>64</td>
<td>High worn B</td>
<td>6.0</td>
</tr>
<tr>
<td>AZ80-AZ61-S11</td>
<td>900</td>
<td>89</td>
<td>133</td>
<td>224</td>
<td>72</td>
<td>New Tool A</td>
<td>5.3</td>
</tr>
<tr>
<td>AZ80-AZ61-S12</td>
<td>900</td>
<td>89</td>
<td>142</td>
<td>221</td>
<td>71</td>
<td>New Tool A</td>
<td>16.7</td>
</tr>
<tr>
<td>AZ80-AZ61-S13</td>
<td>900</td>
<td>89</td>
<td>136</td>
<td>220</td>
<td>71</td>
<td>New Tool A</td>
<td>18.7</td>
</tr>
<tr>
<td>AZ80-AZ61-S14</td>
<td>900</td>
<td>90</td>
<td>149</td>
<td>237</td>
<td>77</td>
<td>Mod worn B</td>
<td>7.9</td>
</tr>
<tr>
<td>AZ61-AZ80-S15</td>
<td>900</td>
<td>90</td>
<td>108</td>
<td>160</td>
<td>52</td>
<td>Mod worn B</td>
<td>7.9</td>
</tr>
<tr>
<td>AZ80-AZ80-S16</td>
<td>900</td>
<td>89</td>
<td>155</td>
<td>226</td>
<td>66</td>
<td>New Tool A</td>
<td>15.1</td>
</tr>
</tbody>
</table>
6.3 Results and Discussion

6.3.1 Alloying Content Combinations

Figure 6.2: Transverse tensile curves. (left) Three friction stir welded alloy combinations, compared to the base materials (two tests per base material). These welds were made using Tool A at 900 RPM and 89 mm/min welding speed and tested at strain rates from \(2.6 \times 10^{-4}\) s\(^{-1}\). (right) Test results for welded samples only, data for AZ80-AZ61-S11 shifted as indicated by the red arrow.

Initially, a series of welds was made to compare the effect of material selection. These friction stir welds were made using Tool A (see Figure 6.1) at 900 RPM and 89 mm/min welding speed and tested at strain rates from \(2.6 \times 10^{-4}\) s\(^{-1}\) with the tensile results shown as Figure 6.2. As seen for other dissimilar magnesium friction stir welds (e.g. [107]), in all cases significant loss of ductility and strength as compared to the parent material properties listed in Table 6.2 has occurred. Comparing the AZ80-AZ31-S2 friction stir weld to AZ80-AZ61-S12 to AZ80-AZ80-S16, the stress at which strain localisation occurred increased (from 106 MPa to 142 MPa to 155 MPa) as did the UTS (from 203 MPa to 221 MPa to 226 MPa) while the total elongation at failure decreased (from 3.7 % to 2.7 % to 1.6 %), as expected from the base materials.
used. Comparing the two AZ80-AZ61 curves shown in Figure 6.2 to each other, after AZ80-AZ61-S11 has been shifted to the right by 0.4 % strain to compensate for the curve in the elastic region the mechanical behaviour was similar to that of AZ80-AZ61-S12. The curvature in the loading region of the AZ80-AZ61-S11 tensile curve is the result of sample curvature from springback upon being cut from the welded sheet.

Figure 6.3: (top) Micrographs of selected tensile samples from Figure 6.2 taken prior to testing. 2 mm scale par shown. (bottom) DIC strain maps based on these samples, colorised from 0-10 % equivalent strain, listed with applied tensile stress. 10 mm scale bar shown. (a) AZ80-AZ31-S2, (b) AZ80-AZ61-S12, (c) AZ80-AZ80-S16.

The micrograph of AZ80-AZ31-S2 in Figure 6.3a shows the horizontal bands of intercalated AZ31 material drawn through the nugget by the thread action have reached as far as the AS interface, indicating the span of the welded nugget and the vertical orientation of the AS interface. In contrast, for AZ80-AZ61-S12 shown in Figure 6.3b, most bands of AZ61 material have only reached part way across the nugget, without formation of clear intermixed layers, most likely due to the higher flow stress of AZ61 at elevated temperatures as compared to AZ31 [157]. In the case
of the similar AZ80-AZ80-S16 joint shown in Figure 6.3c, the internal structure of the nugget is not visible, however the boundary of the fine-grained weld stir zone can be clearly seen to be vertical at the AS and RS up to the shoulder affected region near the top of the nugget.

From the DIC images of Figure 6.3a, it can be seen that strain in the AZ80-AZ31-S2 joint initially localised exclusively along the RS interface, before becoming distributed throughout the more ductile base material on the RS. When an applied stress of 145 MPa is reached, strain localisation at the AS interface has became noticeable, at all times remaining lower in magnitude than that at the RS interface. Strain in the nugget center was consistently much lower than at either of the interfaces, while the strain in the more ductile AZ31 base metal was unsurprisingly larger than in the AZ80 base material. For the AZ80-AZ61-S12 joint, the DIC maps of Figure 6.3b show that strain also occurred preferentially at the RS during the initial stages of deformation, before increasing along the AS interface. For this weld, strain within the AZ80 and AZ61 base materials was more symmetrical on both sides of the weld due to the lower difference in Al content and ductility. For the AZ80-AZ80-S16 joint, behaviour was similar to the AZ80-AZ61 friction stir welds, but more localised around the interfaces.

To determine the sensitivity of these tensile tests to the strain rate, additional lower speed tests were done in AZ80-AZ31 and AZ80-AZ61 at strain rates of 3.7 and $4.3 \times 10^{-5}$ s$^{-1}$ respectively. Comparing the slower AZ80-AZ31-S1 test to the faster AZ80-AZ31-S2 test (see Table 6.3), and the slower AZ80-AZ61-S13 test to the faster AZ80-AZ61-S11 and AZ80-AZ61-S12 tests, changes in strain rate had less effect than the scatter between the similar AZ80-AZ61-S11 and AZ80-AZ61-S12 samples. This
shows that under the current sample production and analysis processes, changes in strain rate within the range of $3 \times 10^{-5}$ to $3 \times 10^{-4} \, \text{s}^{-1}$ will not have significant effects on the strain localisation or UTS.

While there is little work on this topic, Chowdhury et al. [158] tensile tested AZ31 friction stir welds at strain rates from $1 \times 10^{-2} \, \text{s}^{-1}$ to $1 \times 10^{-5} \, \text{s}^{-1}$, which showed inconsistent effects of strain rate on yield strength. For welds with higher yield strength, increasing the strain rate from $1 \times 10^{-5} \, \text{s}^{-1}$ to $1 \times 10^{-4} \, \text{s}^{-1}$ caused slight increases in the yield strength. It should however be noted that measurement procedures differed significantly, as that work measured overall sample yield instead of localised strain.

All of the samples made with Tool A and tested to failure fractured along the original material seam due to a lack of penetration defect. This resulted in each friction stir weld opening up at the root when loaded, effectively creating a notch at the centerline of the nugget root, and reducing the achievable elongation and UTS. Although the AZ80-AZ80-S16 nugget root shown as Figure 6.3c appears to contact the base of the plate, this weld too failed due to a lack of penetration defect, indicating that more complete contact than that shown here is required for a high-quality friction stir weld. Following higher magnification microstructural characterisation, the joint line remnant was indeed visible at the root of the weld, as were some small voids in the upper region of the nugget. Consequently, the friction stir welding tool was changed to Tool B, which prevented these defects from occurring in subsequent welds.
6.3.2 Effect of Distance from the Weld Start

An additional parameter requiring experimental control was noticed when three samples cut from an AZ80-AZ31 friction stir weld made at 900 RPM 63 mm/min and tested to failure at plastic strain rates ranging from 6.0 to \(6.2 \times 10^{-5}\) s\(^{-1}\) were found to have widely different tensile performance. As shown in Figure 6.4, with increasing distance from the weld start, the stress at which strain localisation occurred increased from 90 MPa to 99 MPa to 109 MPa, while UTS increased from 205 MPa to 230 MPa to 247 MPa, a behaviour likely due to an initial thermal transient in the friction stir weld. Fehrenbacher [81, Fig. 35] and Kandaswaamy [80, Fig. 49] report a decrease of tool temperature after the start of welding, while Darras [86, Fig. 5-15] reports an increase. This contradiction is likely due to changes in the initial dwell time, and possibly the tooling material [103], making the key point that changes in the mechanical properties may occur before steady-state is achieved.

Dependence of mechanical behaviour on distance from the tool insertion point is further evidence that for high-quality process control in friction stir welds, real-time monitoring of the tool is a necessity (see [81]). Simulations by Yu et al. [82] estimated that for parameters of 600 RPM and 66 mm/min, a travel distance of 8 cm is required to achieve steady state thermal conditions in AZ31. For the current work a shorter distance to steady state would be expected given the higher rotation rates used, and so all welds discussed below were cut no closer than 6 cm from the start of the friction stir weld.

Atypically for these welds, the micrograph of the AZ80-AZ31-S3 joint (shown inset in Figure 6.4), has a basin-shaped profile. The significant quantity of AZ31 (darker material) along the AS interface and distributed through the nugget indicates greater
intermixing of alloys occurred close to the weld start than for AZ80-AZ31-S5, cut adjacent but further along the weld. In contrast, the microstructural appearance and fracture behaviour of AZ80-AZ31-S4 (not shown) and AZ80-AZ31-S5 were virtually indistinguishable, despite a noticeable difference in strain localisation and UTS. It is likely that this difference in tensile behaviour is due to some factor not identified in the current work, such as residual strain.

6.3.3 Effect of Welding Speed

To verify that optimum processing parameters found in previous work with AZ80 bead on plate welds [8] were applicable to the current tooling and dissimilar materials, joint AZ80-AZ31-S6 was made at 63 mm/min and compared to AZ80-AZ31-S7 made at 90 mm/min. The tensile performance of both welds was nearly identical at lower
strains, with strain localisation occurring at 75 MPa and 79 MPa for the 63 and 90 mm/min conditions respectively. However, the 63 mm/min friction stir weld had inferior performance, with a UTS of 216 MPa and strain to failure of 4.8 % over the 50 mm gauge length as compared to the 90 mm/min sample, which had a UTS of 229 MPa and strain to failure of 6.1 % over the 50 mm gauge length.

Comparing the strain maps for the two welds, at a stress of 145 MPa both show strain starting to localise at the RS side of the top of the weld, where fracture will later initiate. The strain map of AZ80-AZ31-S6 shows an additional band of high
strain concentration aligned with the band of AZ31 traversing the top of the nugget, which is particularly visible at later stages of deformation. At a stress of 183 MPa, the path that fracture will follow in AZ80-AZ31-S6 is clearly visible as a ‘S’ shape band of high strain at the RS of the weld. In contrast, for the AZ80-AZ31-S7, the strain is more homogeneously distributed in the RS material, and no contiguous bands of high strain are present on the AS at all.

Fracture in the AZ80-AZ31-S7 joint initiated at the top of the weld on the RS, and proceeded directly towards the RS interface root at 45° to the loading axis as is typical for ductile shear failure. For AZ80-AZ31-S6, the initial fracture path was identical, but partway through the material thickness, the fracture doubled back into the surrounding base material on the RS side before proceeding jaggedly through to the base surface.

Comparing the distribution of AZ31 for these two welds provides a microstructural basis for the different strain localisation behaviour. For the AZ80-AZ31-S6 joint, the interface between the two materials is generally angled from the band of AZ31 crossing the nugget near the AS shoulder, downwards towards the root of the RS. This will promote shear deformation of the more ductile AZ31 material, leading to the band of high strain seen for AZ80-AZ31-S6 at 183 MPa, and earlier failure. In contrast, the additional band of AZ31 flow in AZ80-AZ31-S7 (see arrows in Figure 6.5) effectively made the interface more vertical, extending strain localisation at the RS prior to failure. From this, it becomes evident that for dissimilar welds minor changes to the flow of material and to the spatial distribution of materials in the nugget will result in major changes in strain localisation and mechanical performance.

In contrast to previous work by the current authors [8] which showed that in...
similar AZ80 friction stir welds more heat input improved the mechanical properties, provided defects were not present, the current results show that for dissimilar AZ80-AZ31 welds, higher heat input is not necessarily better. It is evident that the spatial distribution of AZ80 and AZ31 within the stir zone dominates the deformation behaviour instead of texture or residual strain, resulting in different overall trends.

6.3.4 Effect of Tooling Wear on Defect Formation

The effect of tool wear is examined as Figure 6.6a-c. From the least worn of these three conditions to the most (AZ80-AZ31-S7 versus AZ80-AZ31-S8 versus AZ80-AZ31-S9), the UTS decreased from 229 MPa to 191 MPa to 124 MPa, and the strain at UTS decreased from 6.17 % to 2.79 % to 0.83 %.

Fracture behaviour in these welds was notably different, with AZ80-AZ31-S7 failing on the RS at 45° to the applied load, while AZ80-AZ31-S8 failed through the center of the nugget, generally aligned with both the material interface and a small void (see Figure 6.6b). It is expected that insufficient bonding along the interfaces of the nugget was the source of premature failure in AZ80-AZ31-S8, and the strain localisation behaviour of these two welds differed mainly in how homogeneously the strain was distributed at the AS interface.

In the AZ80-AZ31-S9 joint made with an extremely worn tool (Figure 6.6c), failure initiated at the large wormhole defect visible at the AS root. This defect type is associated with decreasing welding pressures [159] caused by the lack of tool threads to apply consolidation force to the material behind the tool.
6.3. RESULTS AND DISCUSSION

Figure 6.6: Friction stir welds made at 900 RPM and 89 or 90 mm/min using Tool B at various degrees of wear. (top) micrographs of the friction stir welds (bottom) maps of the equivalent strain at the indicated stresses, see Figure 6.5 for scale. (a) AZ80-AZ31-S7 made with a new tool, 183 MPa (b) AZ80-AZ31-S8 made with a worn tool (note defect marked with circle), 183 MPa (c) AZ80-AZ31-S9 made with an extremely worn tool (this sample subsized), 123 MPa (d) AZ31-AZ31-S10 made with the same extremely worn tool as (c), 108 MPa.

6.3.5 Effect of Material Orientation and Alloy on Defect Formation

With increases in the alloying content of AZ series alloys, the ductility decreases and the processing window for defect free welds becomes more limited [12]. Demonstrating this effect, the same worn tool and processing parameters which resulted in the large wormhole defect of AZ80-AZ31-S9 (Figure 6.6c) were used to make the similar AZ31-AZ31-S10 friction stir weld (Figure 6.6d). This resulted in a much smaller defect, now located on the weld centerline instead of at the AS interface. The interfaces of this
weld are far more sloped than those of a weld made in stiffer material (e.g. Figure 6.3c), showing that flow around the tool is heavily affected by the alloy.

The mechanical performance of the similar AZ31-AZ31-S10 weld is unsurprisingly superior to AZ80-AZ31-S9 in terms of UTS and elongation (157 versus 107 MPa), despite the earlier strain localisation (56 versus 98 MPa). The strain distribution in AZ31-AZ31-S10 is quite different to that shown in any of the dissimilar friction stir welds, and shows bands of high strain located on both of the interfaces (see Figure 6.6d). Typically for a similar friction stir weld of reasonable quality, failure in the AZ31-AZ31-S10 joint occurred along the AS interface.

Similarly, for friction stir welds made with a worn tool, placing the more ductile material on the RS (AZ80-AZ61-S14) results in a smaller defect than the reverse situation (AZ61-AZ80-S15). Liu et al. [14] attributed similar results in dissimilar AZ31-AZ80 friction stir welds to the greater difficulty of moving material from the RS to the AS as opposed to the opposite direction, which is somewhat relieved by selecting the more deformable material for this task. This behaviour also explains the tendency of wormhole defects to form at the AS in situations when flow from the RS to the AS is more limited, such as in cases with worn tools.

6.4 Summary and Conclusions

During tensile testing of high quality dissimilar AZ-series friction stir welds, high localised strains occur first under the RS shoulder, before spreading though the RS base material and the RS side of the nugget. At about this stage, strain increases at the AS interface and that side of the nugget while continuing to intensify in other locations. Near failure, strain is highest on the RS side spanning the interface, high in
the AS side of the nugget, and very low in the center of the nugget (see Figure 6.5a). Failure will occur in the softer material on the RS oriented at 45° to the applied load.

In contrast, for a good quality similar friction stir weld, strain first localises along the top portion of the AS interface and intensifies. Later, strain localisation at the top of the RS interface becomes apparent, remaining at all times lower than at the AS interface. As stress increases, strain begins to spread towards the nugget center (see 6.6d). Failure will occur by delamination of the AS interface.

For a similar friction stir weld the greatest transverse strain is limited to two narrow bands aligned with the interfaces, while for dissimilar friction stir welds strain is far more distributed in the base metal at the RS. In both cases deformation in the nugget center is minimal, as expected due to texture here which is poorly oriented for basal slip [8].

As expected, friction stir welds involving AZ80 were found to be more prone to the formation of defects than those using more ductile material such as AZ31, and placing the more ductile material on the RS was advantageous. In one sample, joint efficiency of 101% was achieved, but properties are significantly affected by proximity to the friction stir weld start and tool wear. Tool wear was found to be detrimental even prior to the formation of large, easily detectable defects, and was associated with the formation of void-type defects such as wormholes.

Conditions of 900 RPM and 90 mm/min were found to be superior to parameters of 900 RPM and 63 mm/min for joining these alloys, and strain mapping of the deformation behaviour showed tensile behaviour to be well correlated with the spatial distribution of material in the nugget. It is the conclusion of the authors that in these dissimilar friction stir welds the distribution of nugget material has far greater
impact on the mechanical performance than finer microstructural features such as grain size and texture. It is therefore anticipated that minor changes in the flow and resulting final distribution of material will result in major changes to the mechanical performance of the friction stir weld. In other words, mechanical performance of a dissimilar friction stir weld will be extremely sensitive to minor alterations of the tooling and material layout: far more so than a similar friction stir weld.

As a final note, sensitivity to initial thermal transients is of great interest to practical manufacturing applications of friction stir welding, and has been shown to extend for at least 3 cm from the weld start in the current case but potentially much farther. Work with an instrumented thermocouple and additional tensile testing would be valuable to investigate this effect.

6.5 Acknowledgements and Funding

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Chapter 7

Summary and Future Work

7.1 Summary

For the initial stage of this work, a series of similar friction stir welds was made at varying tool rotation rates and progression speeds on the surface of a continuous extruded sheet. These bead-on-plate welds were necessary to select suitable initial parameters for later use with more complex dissimilar configurations, given the limited body of literature specific to AZ80.

It was determined that within the processing window examined, higher heat input led to better transverse tensile properties, while the lowest heat input condition led to the periodic formation of wormholes along the welding direction [8]. However the microstructural basis for the improvement in tensile properties with greater heat input was unknown, and so optical, EBSD, and low-angle transmission synchrotron diffraction work was done. The synchrotron diffraction work proved particularly fruitful, as information about the texture, residual strain, and dislocation density spanning the entire friction stir weld could be extracted from a single experiment.

The synchrotron texture data was the basis for the first publication of this project.
7.1. SUMMARY

([8], included as Chapter 3). While there are many works which discuss the microstructural reasons for failure initiation at friction stir weld interfaces (e.g. [109, 110]), this publication is one of the few to discuss why the AS interface is favoured over the RS interface for fracture under transverse loading. The broader span of the synchrotron texture maps in contrast to EBSD work also allowed better comparison between friction stir welded textures and measurements of through-thickness contraction made using profilometry (see [8, Figure 6 and 11]). These results are highly applicable to computer models of the deformation of friction stir welds under tension.

From the texture analysis of [8], the extent and distribution of zones favourable for slip was found to be nearly constant between processing conditions, and so did not explain the tensile performance changes with processing condition. However, when this same data was later analysed for residual strain, the results were found to correlate well with the mechanical performance (see [17, Figure 10]), providing an explanation for the trends in tensile behaviour observed. As these measurements were made using a transmission diffraction technique, separate high resolution maps of deviatoric and hydrostatic strain across the weld could be made, showing the symmetry of deviatoric residual strains, the asymmetry of hydrostatic residual strains, and the changes in both with processing condition. This means that the higher heat input friction stir welding conditions will not only have better mechanical properties but also be more resistant to other concerns, such as warping and possibly stress corrosion cracking.

These transmission diffraction experiments provided evidence against correlation of texture and residual strain previously suggested in literature, and showed poor correlation of residual strain and expected thermal gradients. Attempts to compensate
for chemical changes by using a strain-relieved calibration comb supported work by Hughes et al. [149] confirming that residual strains along the length of a calibration comb impact measurements. Analysis of these measurements also led to a method extending existing diffraction techniques for analysing composition in binary alloys to ternary or quaternary alloys in lower symmetry materials.

EBSD analysis was then used to support the partial synchrotron textures measured and to examine the texture change across interfaces at higher resolution. It was found that the strong tendency to form these shear textures could be used to map flow in the weld nugget (see [8, Figure 9]). In addition, this dataset showed that multiple welds had zones of low texture intensity, one of which is shown in Figure 8 (line 6, near the top) of [8]. Given the tendency of magnesium to form high intensity textures, the presence of these low intensity zones in the nugget, bracketed by the expected high texture intensity zones did not conform to the classical textures expected in a magnesium friction stir weld prompting further investigation, later published as [16].

From the periodic distribution of voids at the coldest heat input condition it was evident that periodic flow had occurred based only on optical examination (see [16, Figure 3b]). However, in no case was the traditional ‘onion ring’ structure characteristic of periodicity in friction stir welds visible in the transverse section of any weld. These periodic features, commonly observed in friction stir welded aluminum alloys are rarely apparent in magnesium welds. This meant that either the flow had become continuous in the nugget with the increase in heat input, or the periodicity was simply difficult to perceive. As neither situation was addressed in the literature, the search for answers to these and other questions led to the next manuscript of the
7.1. SUMMARY

This publication, included as Chapter 4, showed that flow remained periodic in all cases and that the apparent lack of onion rings was mainly attributable to the differences in shear texture formation between magnesium and aluminum. This publication also provided explanation for the regions of low texture intensity in the nugget, which were found to result from compaction of material behind the tool. This work showed for the first time that periodicity of nugget flow will not necessarily result in periodicity of grain size, precipitate distribution, or orientation of texture. However, should the material form shear textures, the flow periodicity can still be observed from changes to texture intensity that result from compaction. As part of this work, a useful vectorising technique well suited to graphical presentation of EBSD data in lower-symmetry alloys was developed.

Based on these test results, butt welds of both similar and dissimilar types were made at higher heat input processing conditions. While preliminary efforts to correlate the texture and strain localisation were made in [8], profilometry is exclusively a post mortem technique, and so work was begun with digital image correlation systems. This allowed monitoring of the development of strain localisation during the entire course of a tensile test, which formed the basis of [18].

Several lessons learned during this project are captured in [18], the only (forthcoming) publication by the author on the topic of dissimilar friction stir welds. While initially conceived as a collection of more practical experimental concerns, including tooling wear which became a major concern at later stages of the project, this publication became an investigation of strain localisation and failure development.

Comparing the behaviour of various alloy combinations and processing conditions,
it quickly became apparent that the strain localisation and fracture behaviour was completely different between good quality similar and dissimilar friction stir welds. While this appears to be a trivial result, for similar magnesium friction stir welds the factors of primary importance to the deformation behaviour are the texture and residual strain, both of which are functions of the heat input and thus relatively easy to control. However, for dissimilar friction stir welds, the impact of heat input is a secondary factor, and the weld quality is dominated by the final material distribution in the friction stir weld nugget. In effect, process control changes from an easily altered input parameter to a problem of transient periodic material flow, a far more challenging proposition. This leads to the conclusion that for dissimilar friction stir welds, fine changes to the welding geometry including tool wear have the potential to cause major changes in the final material distribution and thus the mechanical performance within the current processing window, far more so than equivalent changes in a friction stir weld of similar materials. Of course, heat input and flow are interrelated by thermal effects on the material flow stress, as can be seen by the effect of an initial thermal transient on the mechanical properties and microstructure (Chapter 6, Figure 4).

As an additional contribution, the three technical manuals Getting started with MTEX for EBSD analysis [160] (currently with over 800 downloads), Getting Started with Low-Angle Transmission Synchrotron Diffraction [147], and Getting Started with Image Processing for Technical Applications [161] have been produced, and made freely available online. Several analysis techniques and software scripts specialised for investigations in lower symmetry alloys and developed during this project are included. It is the hope of the author that these manuals will facilitate the analysis of
this type of data by future researchers, and open the doorway to further advances in experimental methods, applications of these techniques, and discovery of interesting phenomena.

At the conclusion of this work, many of the major publication results can be summarised as investigations into the strain localisation of friction stir welds in AZ series alloys. Covering bead-on-plate and butt configurations, joining of similar and dissimilar materials, and a range of processing conditions, this work has been wide ranging and generated multiple new results of note within this topic area.

### 7.2 Proposals for Future Synchrotron Work

Several proposed improvements to the experimental methodology for future work in low angle transmission diffraction are listed below.

When manufacturing a sample from which to determine values of $d_0$, the unstrained lattice parameter, the specimen must be strain-relieved by material removal. Commonly done by rendering the sample into either a series of cubes or a comb, the comb has the advantage of retaining spatial alignment of material at the cost of retaining some minor strains on the long axis of the comb tine. Retaining the spatial alignment is valuable in cases like weld samples, where $d_0$ is expected to be highly inhomogeneous. To combine the advantages of both comb and cube, it is proposed to embed a slice of the weld in a soft resin (to allow strain relief), and then slice the metal into cubes using electrical discharge machining while using the resin to retain the spatial relationship.

During ellipse-fitting of low-angle transmission synchrotron data, it would be an enhancement to have software that took into account the relative intensities of the
points involved as a weighting factor, a topic discussed by He [117, p. 302].

For future applications of low angle transmission diffraction to magnesium friction stir welds, it would be worth combining residual stress analysis with precise thermal measurements during welding (such as those of Fehrenbacher [81]) to more precisely analyse correlations between the two.

By performing bead-on-plate welds on a solutionized plate, the analysis of Woo et al. [152] could be expanded to far higher resolution, using FWHM measurements to create full maps of dislocation density across an entire friction stir weld. AZ80 is well suited for this, as the effect of chemical inhomogeneity could then be completely neglected due to the slow precipitation kinetics involved. This would be of interest not only on the ND-TD plane, but if done on the ND-WD plane could be used to spot periodic dislocation density changes along the WD of the weld, given a sufficiently large weld pitch.

The chemical analysis techniques proposed in Chapter 5 are limited by the sparse input data available. Given improved experimental measurements, which would ideally be supported with atomistic level modelling, significant improvements to the accuracy of the resulting analysis are anticipated. This would allow for high accuracy, high penetration, high resolution, compositional measurements.

Finally as a general and more complex proposal, it would likely be possible to perform low angle synchrotron transmission experiments on a single sample in the ND, TD and WD directions for a 3D grid of points, and solve for the local strain ellipsoids computationally. This would allow for determination of the true 3D principal strain axes and values, and has not been done to date. It should be noted that the problem may well need to be overdetermined by means of full sampling along all three axes,
and will require significant computational work, as none of the diffracting volumes will be identical. For that work, methods for precise alignment of scans (down to the $\mu m$) will be required in three dimensions.
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