TENSILE PROPERTIES OF AS-CAST AA5182 ALUMINUM ALLOY CLOSE TO THE SOLIDUS TEMPERATURE

by

LEO JOHN COLLEY
B.Eng., University of Wales, Swansea, 2000

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF APPLIED SCIENCE

in

THE FACULTY OF GRADUATE STUDIES

(DEPARTMENT OF METALS AND MATERIALS ENGINEERING)

We accept this thesis as conforming to the required standard

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July 2003
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Department of **METALS AND MATERIALS ENGINEERING**

The University of British Columbia
Vancouver, Canada

Date **8/8/03**
Abstract

In order to address the demand for accurate property data in the partially solidified state, an experimental apparatus has been developed to enable mechanical property measurements of aluminum alloys at temperatures close to the solidus temperature and strain rates applicable to conventional casting processes.

The experimental apparatus has been used to measure the tensile properties of an industrially DC cast AA5182 aluminum alloy in the as-cast condition between 500°C and 580°C, under strain rate conditions of \(-10^4\) s\(^{-1}\) to \(-10^2\) s\(^{-1}\). A digital camera and zoom lens were used to acquire a series of high-resolution images of the specimen diameter during testing, from which the instantaneous dimetral strain was calculated. The commercial finite element software ABAQUS\textsuperscript{TM} was used to develop a 2-D axisymmetric steady-state thermal analysis of the tensile specimen in order to determine accurate deformation temperatures, and thereby the correct mechanical properties.

The fracture surfaces and microstructures of the tested specimens have been examined using optical microscopy techniques, SEM and EDX analysis to develop a relationship between the mechanical properties of the alloy and changes in microstructure and fracture behaviour. Literature values of the solidification characteristics of the alloy have also been used to relate the properties and microstructure with fraction liquid.

Mechanical property measurements indicate that AA5182 exhibits significant tensile strength up to a temperature of \(-570^\circ\mathrm{C}\) and fraction liquid of \(-0.05\), after which there is a sharp drop in strength. In comparison, ductility decreased steadily with temperature, and complete loss of ductility occurred between 560°C and 570°C dependant
on strain rate. Examination of the microstructure and fracture surfaces enabled these mechanical property characteristics to be linked with the presence of intergranular liquid films in the alloy. EDX analysis confirmed that the liquid films consist of melted intermetallic phases that are present in the intergranular region of as-cast alloys as a result of several eutectic reactions during the final stages of solidification.
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## List of Symbols

### Latin Symbols

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<thead>
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<th>Symbol</th>
<th>Description</th>
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<td>$A$</td>
<td>a set of differential equations representing a problem</td>
<td>-</td>
</tr>
<tr>
<td>$B$</td>
<td>a set of differential equations representing a boundary</td>
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</tr>
<tr>
<td>$C_p$</td>
<td>specific heat</td>
<td>J kg$^{-1}$</td>
</tr>
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<td>$D$</td>
<td>instantaneous diameter of the specimen</td>
<td>m</td>
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<td>$D_o$</td>
<td>original diameter of the specimen</td>
<td>m</td>
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<tr>
<td>$E$</td>
<td>Young’s modulus of elasticity</td>
<td>GPa</td>
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<td>$f_s$</td>
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<td>$f_l$</td>
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<td>$m$</td>
<td>strain hardening coefficient</td>
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<td>power supplied or energy per second</td>
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<td>$q$</td>
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### Greek Symbols

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<tr>
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<td>$\epsilon$</td>
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<td>$\epsilon_{\text{el}}$</td>
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<td>$\epsilon_p$</td>
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<td>$\phi$</td>
<td>total extended volume fraction</td>
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<td>m$^2$</td>
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<td>$\Omega$</td>
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<tr>
<td>$\rho$</td>
<td>density</td>
<td>kg m$^{-3}$</td>
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<tr>
<td>$\rho_{AI}$</td>
<td>resistivity</td>
<td>$\Omega$m</td>
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Acknowledgements

I would like to express my heartfelt gratitude to my supervisors; Dr. Mary Wells and Dr. Daan Maijer, for their guidance and support during the course of this research project. I would particularly like to thank Dr. Wells for her kind spirit and infectious enthusiasm, and Dr. Maijer for his invaluable help in overcoming a wide range of experimental and computational challenges. Special thanks also to Dr. Steven Cockcroft for his sage words regarding many of the issues addressed in this thesis.

I am grateful to Alcan Inc for their support of this academic research programme. The keen interest and commitment to the project shown by Mr. André Larouche, Mr. Yves Caron, Mr. Joseph Langlais, and Mr. Jean-Pierre Martin was greatly appreciated.

I would like to thank Mr. Peter Wilander and Mr. Hai Hao for their assistance while conducting the experimental measurements, and Ms. Belinda Howes for her sterling work preparing metallographic specimens. The experimental apparatus could not have been developed successfully without the help of Mr. Gary Lockhart, Mr. Binh Chau, Mr. Ross McLeod, Mr. Carl Ng and Mr. David Torok.

I would also like to thank the Nadeau and Wilson families for their continued support of the John S. Nadeau Memorial Scholarship.

Finally, I am thankful for the invaluable support from my family and friends, especially my parents, my girlfriend Lindsey and my colleagues at Koerners’ Pub.

This thesis is dedicated to the memory of my grandmother, Heidi Wainwright.
Chapter I – Introduction

1.1 The Emerging Market for Aluminum Sheet in the Automotive Industry

Currently, the major growth market for the aluminum industry is transportation. Annual growth of aluminum consumption for transportation applications averaged 8% throughout the 1990s, and in 1999 the transportation market became the largest consumer of aluminum in North America\(^1\). A key factor is the increasing use of aluminum in automobiles, where the potential benefits of replacing steel with aluminum are estimated at a 20-30% reduction in vehicle weight and fuel economy improvements of 12.5-20%\(^2\). Despite its high cost compared to steel, aluminum autobodies of the kind shown in Figure 1.1\(^3\) are being developed using high-strength AA5xxx and AA6xxx alloys. Current aluminum-bodied vehicles tend to be expensive, low-volume models, and the cost of the aluminum autobody must be reduced significantly before it will be competitive against steel in mass-produced vehicles. As a result, efforts are being carried out to reduce costs by optimizing production processes in the aluminum industry.

![Figure 1.1 - Ford P2000 body-in-white developed in association with Alcan](image-url)
1.2 The Direct Chill (DC) Casting Process

Direct Chill (DC) Casting is the dominant route for billet and sheet ingot production in the aluminum industry. The DC casting process, shown in Figure 1.2, consists of a water-cooled mould with a vertically adjustable bottom block. At the start of casting, the bottom block is partially inserted into the mould and liquid metal is poured into the cavity. The liquid contacts the mould, solidifies, and moves down as the bottom block is lowered. As solidified metal exits, more liquid is introduced and an ingot of similar cross-section to the mould is produced.

![Diagram of the direct chill (DC) casting process](image)

Figure 1.2 - The direct chill (DC) casting process

Direct chill casting typically consists of three stages, namely: Stage I (transient), Stage II (steady-state) and Stage III (end-phase). Stage I occurs at the beginning of casting when thermal fields change with time. Most of the ingot is cast during Stage II,
when the temperature profile remains constant relative to the mould. Final solidification occurs during Stage III. The process is semi-continuous; casting stops after the production of a suitable length, and the ingot is allowed to solidify and is removed. The operation is then repeated after the bottom block is repositioned.

![Diagram](image)

Figure 1.3 - Cooling mechanisms operating during DC casting\(^4\)

Figure 1.3 shows the cooling mechanisms during DC casting. Primary cooling occurs as heat is transferred from the metal to the mould, accounting for \(\sim 20\%\) of the heat extracted under steady state conditions and ensuring that a solid shell forms before the ingot leaves the mould. Secondary cooling takes place as chill water exits the mould through a series of holes at its base and contacts the ingot surface, extracting the remaining \(\sim 80\%\). During start-up, heat transfer from the ingot to the bottom block (base cooling) is also significant.
1.3 Defect Occurrence During The Start-Up Phase of the DC Casting Process

Important quality issues for DC cast ingots are dimensional non-uniformity and cracking. Throughout casting, the ingot is subjected to distortions arising from coupled thermal and mechanical effects. The most critical stage with respect to ingot quality is the transient start-up phase, when the ingot surface temperature and liquid metal pool profile change with time relative to the mould. High cooling rates during start-up generate large thermal stresses that cause the ingot to bow, as shown in Figure 1.4. This is known as “butt curl” and has several adverse effects on ingot quality and process control. Butt curl reduces the amount of thermal contact between the ingot and bottom block, and allows water incursion that further affects the cooling rate and deformation.

![Figure 1.4 - Quality issues arising during DC casting start-up](image)

Figure 1.4 – Quality issues arising during DC casting start-up
Excessive thermal stresses and strains during casting can cause hot tears and cold cracks. Hot tearing takes place in the mushy zone at high solid fractions when a tensile strain is imposed across partially solidified grains. Hot tears have been linked to high casting speeds, bottom block design, thermal gradients and variability in cooling conditions during the transient phase. In contrast, cold cracks form in the solid state.

Industrial procedures to reduce deformation and cracking include high levels of mould cleanliness and dimensional tolerance, and recommended grain refiners and alloying elements. Nevertheless, tight process control is necessary to produce sound ingots of uniform cross-section. This is not easy, as the influence of such parameters as pour temperature, withdrawal rate, water flow rate, ingot size, and bottom block design on distortion and cracking is complex. Furthermore, high strength alloys used in aluminum autobodies are difficult to cast and prone to hot tearing. Hence, it is critical to develop understanding of the influence of casting parameters on defect formation, as well as the mechanisms by which such defects as hot tears occur.

1.4 Thermo-Mechanical Modeling of the DC Casting Process

Relatively little fundamental work has been carried out to optimize the design of the DC casting process with respect to ingot quality. In recent years mathematical models based on fundamental principles have been developed to quantify the influence of casting parameters on the development of stress and strain in the ingot. Two tasks must be completed for this to be possible; firstly, prediction of the thermal behaviour in the ingot; secondly, calculation of the resulting thermal stresses and strains occurring in the ingot.
Mathematical models of DC casting have evolved steadily over the past 25 years. Most early models were 2-D thermal analyses of the steady-state regime. Coupled thermal-mechanical models of the start-up phase are much more challenging because the analysis must solve complex and inter-related thermal and mechanical fields. However, this approach eliminates the efforts required to optimize the process through trial-and-error and in recent years several models dealing with the transient state have been presented\textsuperscript{[11-15]}. 

Critical to accurate stress-strain predictions in the ingot are the appropriate mechanical properties in both the solid and partially solidified regimes. For example, Brody et al\textsuperscript{[16]} have indicated that accurate mechanical property values for the material at temperatures around the solidus are crucial for successful thermal-mechanical behaviour predictions. In addition, it has been found that model predictions are very sensitive to the mechanical properties in this temperature range\textsuperscript{[17]}, and so it is important for these values to be well known. However, the measurement of mechanical properties in the region of the solidus is not straightforward and a lack of understanding of the mechanical behaviour at these temperatures puts modelling efforts in jeopardy. The purpose of the research presented here is to make accurate measurements of the tensile properties of a DC cast AA5182 alloy in this critical temperature range (i.e. 500-580°C), so that a thermomechanical model to simulate the start up phase of the DC casting process can be developed.
References


Chapter II – Literature Review

Although recent years have seen significant progress in the field of numerical simulation of casting and solidification processes, one aspect that has not been well defined is how the material behaves mechanically, particularly in the partially solidified state. Understanding and quantification of this behavior is critical if defects such as hot tearing are to be minimized. The following review addresses some of the pertinent issues surrounding mechanical property measurements of partially solidified aluminum alloys, including microstructural development during solidification, the mechanical response of these alloys in compression, shear and tension, and the formation of hot tears.

2.1 Microstructural Development of AA5182 During Solidification

AA5182 is a high-strength, non-heat treatable aluminum alloy that is used in a number of applications, including inner body panels for automotive applications and beverage can ends. In terms of alloying, it is solid solution hardened by an addition of magnesium (~4.5wt%), augmented by a small addition of manganese (~0.35wt%)\(^1\).

The solidification characteristics of AA5182 have been studied by Arnberg\(^2\). According to this investigation, solidification under equilibrium conditions begins with the formation of primary \(\alpha\)-aluminum dendrites at 637°C. As the alloy cools and continues to solidify, a series of eutectic reactions take place. The first eutectic to form is \(\text{Al} + \text{Al}_6(\text{Fe},\text{Mn})\) at 623°C when the alloy has a fraction solid \((f_s)\) of 0.4, followed by the precipitation of \(\text{Mg}_2\text{Si}\) at 582°C and \(f_s=0.93\). A complex eutectic phase that consists of \(\text{Al} + \text{Mg}_2\text{Si} + \text{Al}_3\text{Fe} + \text{Al}_5\text{Mn}_5\), is formed at 560°C and \(f_s=0.98\), and complete
solidification occurs at 536°C. Figure 2.1 summarizes the reactions that occur during solidification of AA5182.

Figure 2.1 - Microstructural development during solidification of AA5182

2.2 Mechanical Properties

The formation of casting defects such as porosity and hot tearing are controlled in part by the feeding of liquid during solidification, and are therefore influenced by the mechanical properties of the partially solidified alloy. The formation of other casting defects, such as butt curl in the DC casting process, are dependent on the mechanical properties of the alloy at all temperatures from coherency, the point at which the material is first able to accumulate strain, down to room temperature. As a result, it is imperative that the mechanical properties of the alloy at all temperatures are known and understood in order to predict and control the formation of these defects.
Above the liquidus temperature, aluminum alloys behave as a viscous melt. As the melt cools below the liquidus, primary $\alpha$-aluminum dendrites begin to form and the viscosity increases. This mixture of liquid and free-floating solid exhibits shear-rate dependent behaviour typical of semi-solid slurries and pseudoplastic materials\textsuperscript{[3, 4]}. At a critical fraction solid, the first bridging between dendrites occurs and the alloy begins to resist deformation. This is known as the dendrite coherency point, at which a continuous solid skeleton is established and any further liquid feeding must take place through interdendritic openings. After coherency, the strength of the alloy increases as solidification progresses and solid interlinking becomes more prevalent\textsuperscript{[5]}.

The transition between the mechanical response in the partially solid state and conventional solid constitutive behaviour is not well understood and has been observed to differ for several aluminum alloys\textsuperscript{[6]}. As shown in Figure 2.2, it appears that there are two types of behaviour with respect to changes in strength with reduced temperature, $T_R$. Some alloys develop significant tensile strength at a critical temperature in the solidification range (i.e. $0 < T_R < 1$), whereas other alloys develop significant strength only at the end of solidification. The exact physical significance of this transition is not well understood and difficult to examine, although some researchers have attempted to link the development of strength with the rate of change of fraction liquid with temperature, and the formation of eutectic phases at the end of solidification\textsuperscript{[6-8]}.

\textsuperscript{1} Reduced temperature, $T_R$, is calculated using the equation; $T_R = \frac{(T-TE)(T_L-TE)}{(T_L-TE)}$

where $T$ is the test temperature, $T_E$ is the eutectic temperature and $T_L$ is the liquidus temperature.
Below the solidus, plastic deformation of polycrystalline aluminum alloys is controlled primarily by the movement and mutual interaction of dislocations. As the temperature decreases, the ability of dislocations to cross-slip and climb is reduced, resulting in higher yield stresses. At cold-working temperatures, work hardening occurs as the dislocation density increases during plastic deformation. At higher temperatures, dynamic recovery counteracts work hardening, the dislocation density remains constant, and a steady state flow stress is obtained\textsuperscript{[9]}. Strain rate sensitivity also occurs, as the dislocation density is greater under higher deformation rates. At elevated temperatures creep can also have a significant effect on high temperature mechanical properties.

The thermomechanical history experienced during DC casting varies throughout the ingot. Cooling rates at the ingot surface are relatively fast, whereas at the centre cooling takes place much more slowly. As a result, the strain rates experienced at the surface are in the order of 1s\textsuperscript{-1}, whereas at the centre of the ingot the strain rates experienced are typically in the region of 10\textsuperscript{-5}s\textsuperscript{-1}\textsuperscript{[10]}.
2.2.1 Solid-State Mechanical Properties

Most of the high temperature solid-state mechanical properties reported in the literature for aluminum alloys are for wrought materials\(^1, 11, 12\) whose microstructure is quite different to that of as cast materials. However, a comparison of the constitutive behaviour of as-cast AA5182 to wrought material showed that the strength difference between the two is relatively insignificant\(^{10}\). Experimental results for as-cast AA5182 showed that work hardening occurred at temperatures up to 350°C. Above this temperature there was very little evidence of work hardening and steady-state flow stresses were reached at much lower strains, shown in Figure 2.3. A strong strain rate dependency was also measured at temperatures greater than 250°C, shown in Figure 2.4.

![Figure 2.3 - Steady state and work hardening conditions during compression tests for AA5182 alloy at a strain rate of 1.0s\(^{-1}\)\(^{10}\)](image)

Figure 2.3 - Steady state and work hardening conditions during compression tests for AA5182 alloy at a strain rate of 1.0s\(^{-1}\)\(^{10}\)
2.2.2 Mechanical Properties in the Region of the Solidus Temperature

Although the mechanical properties close to the solidus are important for modelling thermomechanical stresses and hot tearing during casting, they are usually poorly known. Within the general framework of research into the mechanical properties of alloys in the partially solidified state, tests have been carried out under different deformation modes including compression, shear and tension. However, there is no comprehensive study that includes a wide range of aluminum alloys and deformation modes, and individual investigations have usually been limited to a few alloys and only one deformation mode.
2.2.2.1 Compression Tests

Measurement of the mechanical properties of partially solidified alloys under compression has been carried out by a number of researchers\textsuperscript{[13-16]}. However, most of this work has been directed towards thixoforming and rheocasting processes, in which the alloys have a semi-solid (globular) rather than as-cast (dendritic) microstructure. In the globular state, the solid phase morphology is spherical and non-dendritic, and is significantly different to the highly branched dendrites typical of as-cast microstructures. This difference is shown in Figure 2.5\textsuperscript{[14]}.

![Figure 2.5 - Comparison of as-cast (left) and non-dendritic (right) microstructures in an aluminum alloy\textsuperscript{[14]}](image)

Due to these microstructural differences, mechanical property measurements carried out on non-dendritic microstructures may not be comparable to the properties of dendritic morphologies. Suery and Flemings performed compression tests on dendritic, fine dendritic and non-dendritic morphologies of Sn-15wt%Pb, and the different mechanical responses are shown in Figure 2.6\textsuperscript{[17]}.
Although compression testing of partially solidified dendritic alloys may provide useful information regarding their deformation and flow behaviour, it is known that hot tearing during DC casting occurs as a result of the development of tensile strains in the partially solidified state. Compression testing does not allow the study of this phenomenon, and as a result material property data relevant to the prediction of thermomechanical behaviour during DC casting cannot be obtained in this way.
2.2.2.2 Shear Tests

Mechanical property measurements have been carried out on a number of partially solidified aluminum alloys in shear. Experiments have usually been carried out using either a Couette viscometer (vane testing)\cite{2, 5, 18}, or a shear cell\cite{19, 20}.

Viscometry tests have typically been carried out at low solid fractions, and have been used extensively to measure the dendrite coherency point, at which the alloy is first able to resist plastic deformation, and the development of strength immediately after coherency. The influence of grain refinement on the coherency point and development of strength in a Al-4wt\%Cu alloy is shown in Figure 2.7\cite{5}. The literature shows that there is a strong relationship between the kinetics of dendritic growth and the temperature or fraction solid at which the dendrite coherency point is measured by this method\cite{5, 18}.

![Graph showing the influence of Ti additions on the development of strength in partially solidified Al-4wt\%Cu alloy.](image)

**Figure 2.7** - Results of viscometry tests carried out on an Al-4wt\%Cu alloy, indicating the influence of Ti additions on the development of strength in the partially solidified state\cite{5}
Mechanical property measurements at higher solid fractions can be made using shear cells. Results show that the increase in strength after coherency can be divided into two parts, as shown schematically in Figure 2.8\textsuperscript{[19, 20]}. Shear strength increases slowly after coherency until a second critical point is reached, called the maximum packing fraction. After this point, it is proposed that dendrites interlock and provide rigidity to the partially solidified alloy, thus accounting for the rapid increase in strength as solidification continues.

![Figure 2.8 - Schematic of development of shear strength in partially solidified alloys, showing the influence of dendrite coherency and maximum packing fraction\textsuperscript{[19]}](image)

The shear properties of partially solidified alloys have typically been investigated at $f_s < 0.6$, and this deformation mode has not been used to study the increase in strength at higher solid fractions. Hot tearing is known to form at high solid fractions ($f_s > 0.9$) as a result of tensile strains, and therefore it is unlikely that shear testing will provide suitable data with which to predict the formation of hot tears.
2.2.2.3 Tension Tests

Only a limited number of investigations have been carried out on partially solidified alloys in tension. In one of the first studies of its kind, Williams and Singer tested a number of binary Al-Sn alloys in the partially solidified state\textsuperscript{[21]}. Unfortunately the cast material was extruded into rods prior to machining into test specimens, thereby eliminating the as-cast structure. A sharp drop in ultimate tensile strength was observed at 228°C, corresponding to the eutectic temperature of the Al-Sn system. Above the eutectic point, strength decreased steadily to zero. For an Al-8.4wt\%Sn alloy, shown in Figure 2.9, significant tensile strength was measured up to 600°C.

![Figure 2.9 - Tensile Strength-Temperature for an Al-8.4wt\%Sn alloy\textsuperscript{[21]}](image)

Ackermann et al. used a novel apparatus to perform in-situ strength measurements on Al-Mg alloys\textsuperscript{[22]}. They measured the force required to separate two cylinders after a
layer of solidified material had formed across their surfaces. Strain rate was calculated indirectly, based on the assumption that the vertical length of solidified material deforms uniformly at the same rate as the separation rate. Strain-rate sensitivity was observed in the solidification interval; however, the results have limited use because of the presence of significant thermal gradients during testing.

Nakagawa et al. melted specimens of an Al-Mg-Si alloy inside a mullite sleeve using an induction coil and tension tested them during controlled solidification using a 2kN load cell\cite{23}. The alloy fractured in a brittle manner at all test temperatures and significant tensile strengths were only measured at solid fractions higher than 0.91. The use of a sleeve around the specimen prevented accurate strain measurement.

Chu and Granger tested specimens of AA3004, AA2024 and AA7075 that were heated to temperatures above their eutectic points by a radiant furnace\cite{6}. The specimens were machined from grain-refined directionally solidified castings so that the tensile axis was normal to the dendrite growth direction. After reaching the target temperature a tensile load was applied at a predetermined rate until failure. No details regarding strain measurement were given. As shown in Figure 2.2, two types of behaviour were observed with respect to the variation in strength. AA2024 and A7075 exhibited similar behaviour to that reported previously by Williams and Singer; a sharp drop in tensile strength at the eutectic temperature. In comparison, AA3004 exhibited significant strength to a reduced temperature of 0.6, at which point a sharp decrease occurred. AA3004 exhibited strain rate sensitivity and grain boundary strengthening up to the drop in strength, as shown in Figures 2.10 and 2.11. Strain rate and level of grain refinement did not appear to influence the temperature at which the decrease in tensile strength occurred.
Figure 2.10 - Influence of dendritic grain size on the tensile strength of a commercial AA3004 alloy\cite{6}

Figure 2.11 - Influence of applied strain rate on the tensile strength of a commercial AA3004 alloy\cite{6}
Magnin et al. used several experimental techniques to study the tensile properties of an Al-4.5wt%Cu alloy from room temperature up to the coherency temperature\(^{[24]}\). Specimens tested in the partially solid state were cast in an insulated mould between two jaws, one of which could be moved to apply a strain once the test temperature was reached. Experimental results were not presented, although a constitutive equation based on an elasto-viscoplastic power law of the form shown in Equation 2.1 was derived, and the authors proposed that it gave a continuous description of the behaviour of the alloy across the entire temperature range.

\[
\sigma = K \cdot f_1(\varepsilon_p^*) \cdot f_2(\dot{\varepsilon}_p)
\]

\[
\sigma = E \varepsilon_{el}
\]

In Equation 2.1, \(\sigma\) is the flow stress, \(\varepsilon_p^*\) is the plastic strain, \(\varepsilon_p\) is the equivalent plastic strain rate, \(\varepsilon_{el}\) is the elastic strain, \(E\) is the Young's Modulus, and \(K\) is a coefficient of the constitutive equation.

Spittle et al. used a Gleeble 1500 Thermomechanical Simulator connected to a low-force mechanical testing machine to examine the tensile properties of a number of alloys in the partially solidified state\(^{[8]}\). The authors eliminated large temperature gradients from the specimen in order to perform lengthwise strain measurement. An investigation into the mechanical properties of an AA2024 alloy in the as-cast and wrought states showed that the tensile strength and ductility of the alloy differ, as shown in Figure 2.12. It was suggested that differences in the amount of liquid in the as-cast material compared with the wrought material for a given temperature accounts for this
behaviour. Mechanical properties of the as-cast AA2024 were in reasonable agreement with those measured previously by Chu and Granger.

\[ \text{TEMPERATURE, } ^{\circ}\text{C} \]

\[ 460 \quad 480 \quad 500 \quad 520 \quad 540 \quad 560 \quad 580 \quad 600 \quad 620 \quad 640 \]

\[ 460 \quad 480 \quad 500 \quad 520 \quad 540 \quad 560 \quad 580 \quad 600 \quad 620 \quad 640 \]

Figure 2.12: a) Influence of temperature on the tensile strength of as-cast and wrought and recrystallized AA2024,

b) \( f_L \) vs. temperature for AA2024 under equilibrium and scheil conditions\[^{[8]}\]

Van Haaften et al. conducted tension tests on partially solidified as-cast AA3104 and AA5182 using a Gleeble 3500 Thermomechanical Simulator with low-force jaws\[^{[25]}\]. Specimens were machined from a DC cast ingot so that the tensile direction was parallel to the casting direction. Resistance heating was used to rapidly heat the specimens to the target temperature. Tests were carried out under load-control, except at the highest temperatures when stroke control was used because the measured force was too small for accurate control. Strain measurements were made using a dilatometer. Strain rate sensitivity was observed at all temperatures up to 550°C, above which the tensile strength
became very small. The authors developed a modified creep law to provide a continuous 
description of the constitutive behaviour of the alloy into the partially solidified region.

Recently, Dahle et al. compared the tensile and shear properties of partially 
solidified Al-Cu alloys\cite{26}. They indicated that coherency occurs at different points for 
each mode, and confirmed that shear strength develops after dendrite coherency. Tensile 
strength was first measured at the maximum packing fraction, the same point at which the 
rate of strength development increases in shear\cite{19}. At high $f_s$, strength measurements 
converge. Consequently, the authors suggest that deformation mechanisms in shear and 
tension become similar at high solid fractions. A summary of strength development in 
shear and tension and proposed strengthening mechanisms is given in Figure 2.13.

1. Change in Bonded Area of Primary Dendrites 
2. Change in Bonded Area Associated with Eutectic Formation 
3. Final Eutectic Solidification

Figure 2.13 - Comparison of the development of shear strength and tensile strength in the partially solidified state\cite{26}
In order to determine how critical the mechanical properties in the region of the solidus temperature are with respect to predictive modelling of casting processes, Giron et al. studied the development of stress and deformation during DC casting of a commercial Al-Cu-Mg-Zn alloy with two different mechanical property datasets. One dataset was based on a hyperbolic sine law that had been extrapolated from the solid state into the solidification interval, while the second dataset consisted of experimentally measured values from an investigation based on Chu and Granger’s previous work. The computational domain was developed using the commercial finite element software ABAQUS™, and the model results, shown in Figure 2.14, show that the development of strain during solidification and cooling are highly sensitive to the specified mechanical properties at these temperatures.

![Figure 2.14 - Effective plastic strains for “App Case” (hyperbolic sine equation) and “Base Case” (measured data) at a location close to the ingot rolling face](image)

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2.3 Hot Tearing in Aluminum Alloys

Hot tearing is a common problem encountered during the casting of aluminum alloys. While it is known that hot tearing is an intergranular fracture as illustrated in Figure 2.15, resulting from excessive tensile strains imposed on the partially solidified alloy, the mechanisms by which hot tears form are not well understood, and identification and quantification of the factors that influence the occurrence and severity of hot tearing is not straightforward. Consequently, there has been a consistent and continued effort to develop an understanding of the hot tearing phenomenon.

Figure 2.15 - SEM micrograph of the fracture surface of a hot tear in a DC cast AA5182 billet[^28]

2.3.1 Experimental Investigations

One of the first investigations into hot tearing was carried out by Pellini, who cast an Al-Cu alloy in a restrained bar apparatus[^29]. The apparatus allowed accurate
measurement of temperature and control of contraction in the solidifying alloy. Hot tears were detected by means of an X-ray. It was concluded that hot tearing represents fracture through interdendritic liquid at temperatures immediately prior to complete solidification.

Clyne and Davies performed tests on Al-Mg, Al-Si and Al-Zn alloys using a “dog-bone” (restrained bar) apparatus, and presented a general relationship between the initial composition of the alloy and the occurrence of hot tearing, characterized by the ‘lambda curve’, shown in Figure 2.16\cite{30}. They showed evidence of the presence of liquid at the point of fracture, with chemical analysis indicating that higher-than-expected levels of low melting point segregates were present at the fracture surfaces.

![Figure 2.16 - Lambda curve illustrating the variation of hot tear susceptibility with wt% Cu in the Al-Cu binary system\cite{31}](image)

Figure 2.16 - Lambda curve illustrating the variation of hot tear susceptibility with wt% Cu in the Al-Cu binary system\cite{31}
The 'lambda curve' relationship was extended to Al-Cu alloys by Spittle and Cushway[32], who also determined that the number of interdendritic liquid films in the strained section of the casting is a critical hot tearing parameter. Strains can be accommodated to greater levels, avoiding hot tearing, if a larger number of liquid films exist in the restrained section. Thus, the influence of the developing microstructure on hot tearing susceptibility is controlled by factors such as the amount of superheat, grain refinement and initial alloy composition.

From these early studies, it is clear that hot tearing is a specific phenomenon that affects alloys in the partially solidified state, and is distinct from cracks that form in the fully solid state.

More recently, Van Haaften et al. reheated specimens of an as-cast AA5182 alloy and performed tensile tests inside a scanning electron microscope to observe hot tear formation[33]. Crack initiation was found to take place at any weak point in the mush, such as a pore or area of liquid, while crack propagation was predominantly intergranular. The authors also reported the presence of 'spikes' on the fracture surface, and suggested that fracture of solid bridges between grains can take place, resulting in localized areas of extreme ductility. These spikes have also been noted in a number of other aluminum alloys[18, 30, 32, 34], and have since been reported in the magnesium alloy AZ91[35].

As a means of visualizing in-situ hot tear formation, Farup et al. initiated hot tearing in the organic model alloy of succinonitrile (SCN) and acetone[34]. Hot tear nucleation was observed either directly in the liquid, or at micropores and air bubbles. Once nucleated, the hot tear propagated by movement of liquid in the direction of the
applied strain. Spike formation on the fracture surfaces was observed, and two types of spike were identified; one that shows evidence of plastic deformation and another that has a more liquid appearance. The study concluded that some spikes are evidence of the ductile fracture of solid bridges, while others are the solidified remains of a liquid film.

2.3.2 Hot Tear Criteria

Several researchers have developed mathematical criteria in attempts to quantitatively define the occurrence of hot tearing. Clyne and Davies developed one of the earliest criteria by considering the time a solidifying alloy spends in a ‘vulnerable’ region of the solidification interval as a proportion of the time for complete solidification. The vulnerable region was defined as the final stages of solidification, 0.9\(<f_s<0.99\), in which liquid exists as thin films between solid grains. Although this criterion is a useful indicator of hot tearing susceptibility, it is an empirical ‘rule-of-thumb’, and does not address the fundamental physical principles that control hot tear formation.

Feurer considered the nucleation of a hot tear based on the opening of an intergranular region due to solidification shrinkage and the inability of the liquid to feed into this opening. Although this criterion is based on fundamental principles, it only considers solidification shrinkage and does not take into account interdendritic openings arising from thermal contraction in the solid or uniaxial tensile deformation.

Rappaz et al. extended Feurer’s approach to account for feeding associated with tensile deformation of the solidified material. The criterion is strain-rate based, and computes the pressure change in the interdendritic liquid as a result of solidification
shrinkage and an externally applied tensile deformation. This change in pressure is
compared to a critical pressure change required for cavitation in the liquid. As such, the
criterion is based on the nucleation of a pore, rather than the propagation of a hot tear.

Lahaie and Bouchard have developed a model in which they have assumed the
stress/strain relationship of the alloy is the major cause of hot tearing\[38\]. The model has
been developed to examine deformation stresses within the mushy zone and incorporates
a stress-based hot tear criterion. The model calculates deformation stresses due to
viscous flow of liquid and liquid dilation as a function of strain, strain rate and fraction
solid, and compares these with a critical stress at which hot tearing will occur, based on
the stress required to separate two plates bonded by capillary forces in a liquid.

The criteria reviewed above can be incorporated into thermal-mechanical models
of casting processes in order to predict the occurrence of hot tears during casting. As part
of the development of these models, the mechanical response of the material must be
known for the temperature range and deformation conditions relevant to the process, and
therefore the mechanical properties of alloys at temperatures close to the melting point
and in the solidification interval are of particular interest.
2.4 Summary

Some of the key material property data required to accurately model the development of stresses and deformations during casting is the mechanical response of the alloy being cast from the point at which it first develops some mechanical strength (i.e. the coherency temperature) down to room temperature.

Although the behaviour in the solid state is reasonably well known, the literature review shows that there is uncertainty regarding the mechanical properties of the material at temperatures in the region of the solidus temperature, where the presence of liquid influences the mechanical response of the alloy. The issue has been compounded by the fact that only a limited number of investigations have been carried out on the tensile properties of aluminum alloys at these temperatures, while it is unclear whether other experimental approaches (compression testing, use of shear cells) provide the appropriate data. Nevertheless, it has been shown that the constitutive behaviour in the region of the solidus temperature has a significant influence over model predictions of stress and deformation development during casting.

Furthermore, a critical quality issue for many casting processes is the phenomenon of hot tearing, a low ductility failure of the alloy in the partially solidified state when it is very weak. Hence, complete thermomechanical modelling of casting processes must include a criterion that will predict the occurrence of hot tearing during casting.
References


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27. A. Giron, M.G. Chu, and H. Yu, "Effect of Mushy Zone Mechanical Properties on the Calculated Stresses and Deformations During the Casting of an Aluminum Alloy Ingot", Light Metals 2000, TMS.


Chapter III – Scope and Objectives

The direct chill (DC) casting process is the dominant route for billet and sheet ingot production in the aluminum industry. Important quality issues for DC cast ingots are dimensional non-uniformity and cracking arising from thermally induced stresses and strains. These issues are particularly apparent during the production of high strength aluminum alloys such as AA5182, and successful casting requires an improved understanding of the mechanical behaviour of these alloys at all temperatures from coherency down to room temperature in order to predict and control the formation of defects. One area that requires specific attention is the tensile response of the partially solidified alloy, as these properties are highly important with regard to the formation of hot tears. The literature review presented in the previous chapter indicates that there is a need to quantify the mechanical behaviour of aluminum alloys at temperatures near and above the solidus. In fact, only a few studies have been done to measure the tensile behaviour of the material in the semi-solid region. Part of the reason for this lack in experimental data is that the measurement of the tensile properties of partially solidified aluminum alloys is quite difficult as temperature gradients in the sample may exist and it is difficult to measure the strain that develops in the material during testing.
3.1 Objectives

In order to address the demand for accurate materials property data in the partially solidified state, the objectives of this research project will be the following;

- To develop a mechanical testing apparatus that enables the measurement of the tensile response of aluminum alloys at temperatures in the region of the solidus and strain rates applicable to conventional casting processes.

- To use this mechanical testing apparatus to determine the tensile properties of an industrial AA5182 aluminum alloy in the as-cast condition from 500°C to the coherency temperature, under similar strain rate conditions as it is exposed to during the DC casting process.

- To model the thermal profile and temperature gradients in the specimen at test temperatures, in order to determine accurate deformation temperatures and thereby obtain the correct mechanical property data.

- To examine the fracture surfaces and grain structure of tested specimens in order to gain understanding of the structure-property relationships of the alloy around the solidus temperature.
3.2 Methodology

The methodology used to carry out this research involved both experimental measurements on industrial as-cast aluminum alloy samples using an apparatus designed and built at the University of British Columbia, as well as mathematical modelling of the temperature profile in the tensile specimen at test temperatures to determine the exact temperature at which the experiments were carried out.

After the identification of a suitable testing method, based on the literature review presented in the previous chapter, a low-force Instron mechanical testing machine was modified to enable its connection to a Gleeble 1500 thermomechanical simulator. Preliminary testing was carried out to measure and minimize any temperature gradients generated along the length of the specimen during testing. A non-contact strain measurement system was developed, incorporating a high-resolution digital camera to obtain accurate measurements of the change in specimen diameter during testing. Using this apparatus, tensile tests were carried out on specimens machined from an industrially DC cast ingot in the as-cast condition at strain rates close to those experienced during casting ($10^{-4}s^{-1}$ to $10^{-2}s^{-1}$) and temperatures in the region of the solidus (500°C to 580°C).

The commercial Finite Element (FE) software package ABAQUS™ was used to model the temperature gradients and thermal profile in the specimens at temperature, in order to determine the exact temperature of deformation for each test and thereby obtain accurate material property data. An examination of specimen microstructures and fracture surfaces was conducted with optical and scanning electron microscopy to develop an improved understanding of the structure-property relationships of the alloy at temperatures in the region of the solidus.
Chapter IV – Experimental

4.1 Start Material

The experimental work has been carried out on a commercially significant DC cast aluminum alloy AA5182, supplied in the as-cast state by the Arvida Research and Development Centre (ARDC) of Alcan International. This alloy was chosen primarily because of its long freezing range and well-documented hot tearing susceptibility. Furthermore, AA5182 is an important commercial alloy, and a significant amount of information on Al-Mg alloys is present in literature. In addition, the results from this investigation will be used as part of a related project underway at the University of British Columbia to develop a fully coupled thermal-mechanical finite element model of an AA5182 ingot during the start-up phase of the DC casting process in ABAQUS™. The chemical composition of the alloy used in this study is shown in Table 4.1.

<table>
<thead>
<tr>
<th>Al (%)</th>
<th>Mg (%)</th>
<th>Mn (%)</th>
<th>Si (%)</th>
<th>Cr (%)</th>
<th>Fe (%)</th>
<th>Cu (%)</th>
<th>Ti (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal.</td>
<td>4.66</td>
<td>0.31</td>
<td>0.06</td>
<td>0.002</td>
<td>0.21</td>
<td>0.057</td>
<td>0.023</td>
</tr>
</tbody>
</table>

It has been shown previously that the grain size of the DC cast ingot varies significantly from the surface to the interior\textsuperscript{[1]}. There is a chill zone consisting of very fine equiaxed grains at the surface of the ingot. Inside the ingot, columnar dendrites with typical grain diameters of 1mm are present close to the surface, and then the microstructure becomes equiaxed at the centre. Photographs showing typical chill and columnar zone microstructures are shown in Figure 4.1.
a) Sub-surface showing chill zone and columnar zone\textsuperscript{[1]}

b) 5cm from ingot surface, transverse to the direction of columnar dendrites\textsuperscript{[1]},

c) 5cm from ingot surface, parallel to the direction of the columnar dendrites.
Figure 4.1 (on previous page) - Starting microstructure of DC cast AA5182

a) Sub-surface showing chill zone and columnar zone\(^{[1]}\),

b) 5cm from ingot surface, transverse to the direction of columnar dendrites\(^{[1]}\),

c) 5cm from ingot surface, parallel to the direction of the columnar dendrites.

For the purposes of this investigation, experiments have been carried out on specimens with a columnar microstructure, close to the surface of the ingot. Cylindrical tensile specimens were machined from DC cast ingots normal to the casting direction and parallel to the surface of the ingot. The specimen orientation relative to the DC cast ingot is illustrated in Figure 4.2. This orientation was chosen so that cracking in the specimen takes place in the same direction as hot tearing occurs in the ingot during DC casting.

![Figure 4.2 - Specimen orientation within ingot](image)
A schematic of a typical tensile specimen is shown in Figure 4.3, and these were machined to have a central reduced section of 8mm diameter and 100mm length. The specimen ends had an increased diameter and were threaded such that they could be mounted into the tensile testing apparatus.

Figure 4.3 - Schematic of specimen geometry (N.B. Not To Scale)

a) axial direction and b) cross-section
4.2 Technical Challenges

The accurate measurement of the mechanical properties of partially solidified alloys in the as-cast state poses several significant technical challenges. In terms of the structure of the material being tested, an as-cast microstructure consisting of cored dendrites with segregated liquid or eutectic phases between the dendrite arms must be present during testing. Two methods can be used to achieve this end: cooling from the fully liquid state prior to testing, or reheating a previously cast material from room temperature quickly enough to avoid significant homogenization. It was shown in the literature review presented earlier that the mechanical properties of alloys in the partially solidified state can vary significantly with structure, and as a result care must be taken to achieve the relevant microstructure when employing either approach.

If a partially solidified as-cast microstructure is developed from the liquid state, a container is required to hold the melt. Attention must be paid when selecting a material for the container to avoid any chemical reaction with the specimen. Furthermore, the container must not impart any friction that would influence load measurements during testing. Heat transfer processes must be carefully controlled in order to develop the required microstructure, ensure low temperature gradients along the length of the specimen, and produce a sound casting. With respect to the project undertaken in this study, a columnar microstructure representative of a DC cast ingot would have to develop normal to the tensile direction.

Partially solidified as-cast microstructures can also be generated through reheating a previously cast alloy. In this case, the main challenge is to maintain the as-cast microstructure during testing. Solid-state diffusion rates are high at temperatures
close to the solidus, and homogenization processes may dissolve non-equilibrium phases, leading to changes in fraction liquid as the alloy moves towards the equilibrium state. Hence, testing must be complete before the structure deviates significantly from the as-cast state.

The partially solidified state is associated with low strength and ductility. As a result, sensitive methods of measuring small changes in load and specimen dimensions must be found. The challenge presented by accurate measurement of mechanical properties in the partially solidified state is further complicated by the inability to use a contact strain measurement device due to the low resistance to plastic deformation. Additionally, imposed loads arising from thermal expansion and contraction of the specimen during heating and cooling must be avoided, as these will affect any load and strain measurements made during the test.

Temperature control is a critical factor in performing mechanical property measurements close to the solidus temperature. Significant structural changes occur over relatively small temperature ranges, and therefore it is important that the test temperature is held constant throughout the duration of the test. This has implications with regard to testing methods that utilize the electro-resistance heating technique, such as temperature control using a Gleeble Thermomechanical Simulator. The control of temperature in the Gleeble is carried out using feedback control through one of the thermocouples attached to the specimen, usually at the central point axially. If there is a significant change in specimen cross-sectional area away from the control thermocouple, the current density in this area will change as deformation proceeds, leading to variations in the local temperature. To avoid any variation in the test temperature resulting from dimensional
changes, any necking that occurs in the specimen must be located very close to the control thermocouple. It follows that the evolution of heat in the specimen and heat transfer from the specimen to surrounding areas must be controlled such that the peak temperature occurs at the control thermocouple.

A further consideration regarding temperature control is the influence that temperature measurement devices may have on mechanical property measurements. Thermocouples that are embedded into the specimen cannot be used as they may induce defects in the specimen and affect the measured properties. An alternative means of attachment must be able to withstand the high temperature and presence of liquid during testing, while still providing accurate measurements.

Finally, it is necessary to find a means of accurately determining the fraction liquid during testing. Only when this has been achieved will it be possible to relate any changes in mechanical behaviour to the amount of liquid in the alloy.

4.3 Experimental Apparatus

For the purposes of this investigation, it was decided to adopt an experimental approach involving a tensile mode of deformation, and development of the partially liquid as-cast microstructure by heating previously cast material, following the approach made previously by Spittle et al[2].

A custom built apparatus was developed for the experimental measurements. The experimental set-up consisted of a modified Instron mechanical testing machine with a 4.5kN (1000lb) load cell to enable accurate measurement of the low loads associated with the partially solidified state, connected to a Gleeble Thermomechanical Simulator that
allowed rapid heating of the alloy via electro-resistance heating. An overview of the apparatus is shown in Figure 4.4.

Figure 4.4 - Overview of the experimental apparatus

Electrical current for resistance heating was transferred from the jaws of the Gleeble by heavy welding cables that were connected to the frame of the Instron by two water-cooled copper blocks attached to non-conducting material. The current was then passed to the load train by a series of thinner, multi-strand copper cables. These cables
connect the water-cooled copper blocks to a second set of copper blocks attached to the pull rod of the load train. In this way, a direct connection between the welding cables to the load train was avoided, preventing them from influencing load measurements during testing. The electrical current transfer apparatus is shown in Figure 4.5.

![Image of a modified Instron testing machine showing the modified setup.](image)

**Figure 4.5 – Overview of the modified Instron testing machine**

The tensile specimen was held in place on the load train by two stainless steel grips positioned adjacent to the copper blocks. The purpose of the grips was to reduce the rate of heat transfer from the tensile specimen to the water-cooled copper blocks and thereby lessen the temperature gradient along the length of the specimen during testing. The grips also delivered the electrical current to the specimen and provided the mechanical linkage between the specimen and the load train.
Figure 4.6 – Schematic of Instron load train and cage assembly

The movement of a stainless steel cage attached to the crosshead enabled deformation of the specimen. The cage surrounded the load train as far as the bottom steel grip, and a 12.5mm diameter circular hole in the bottom plate allowed the specimen to pass through. Deformation occurred when the crosshead was moved down, forcing the bottom plate of the cage against the lower stainless steel grip. The cage can be seen in
Figure 4.5 and a schematic of the load train and cage assembly is shown in Figure 4.6. A circular alumina disk with 30mm diameter and 2.5mm thickness was used to insulate the bottom of the cage from the lower stainless steel grip. A 12.5mm circular hole was cut through the centre of the disk to allow the specimen to pass through. Electrical insulation was also required to prevent current from traveling up into the load cell and frame of the Instron. This was achieved by incorporating an insulating flange consisting of two 36mm diameter, 6mm thick stainless steel plates separated by a non-conducting 46mm diameter, 6mm thickness asbestos disk into the pull rod. The stainless steel plates were joined through the disk by four stainless steel bolts that were lined with alumina sleeves.

Type K thermocouples were spot welded to the surface of the tensile specimen to enable accurate measurements of the temperature distribution along its length. Omega CC High Temperature Cement, a zircon-based two-part ceramic cement was applied to the thermocouples using a cocktail stick to anchor them to the specimen and prevent their detachment when testing at partially solidified temperatures. The thermocouple spot-welded to the axial center of the specimen acted as a feedback thermocouple to control temperature during testing.

4.3.1 Preliminary Testing and Development of the Experimental Test Procedure

A number of preliminary tests were carried out to establish electrical and thermal control prior to the commencement of the experimental investigation. Initially, it was necessary to confirm that the modified section of the electrical circuit was stable. It was also necessary to ensure that temperature in the specimen was steady and controllable.
during heating and holding at test temperatures. Finally, attempts were made to minimize
the thermal gradient along the length of the tensile specimen during testing.

During initial testing to establish the stability of the electrical circuit, arcing
between the load-train mounted copper contact blocks and the pull rods was encountered.
This was also observed to a lesser extent between the pull rod and the stainless steel grips
at the specimen ends. This problem was solved by applying a Never-Seez copper-based
anti-seize lubricating agent by Bostik with a paintbrush at the interfaces between the
copper contact blocks and pull rod after every six tests, and by positioning the copper
contact block so that it was in firm contact with the adjacent stainless steel grip.
Dismantling the apparatus and cleaning all electrical contact surfaces with acetone after
every six tests minimized arcing between the pull rod and stainless steel grips. During
this stage of preliminary testing, it was also found that deformation of the threaded
specimen ends, resulting from thermal expansion of the aluminum alloy inside the
stainless steel grips during testing, made specimen removal difficult. The application of a
suspension consisting of equal parts molybdenum powder and ethanol to the specimen
threads prior to each test reduced the occurrence and severity of this problem.

Preliminary testing carried out using a 10mm diameter specimen indicated that
the temperature in the specimen was not stable during heating and holding at temperature,
and that thermal cycling was taking place, as shown in Figure 4.7. In comparison, the
thermal history for a specimen with a diameter of 8mm shows that temperature control in
this case is much more stable. Hence a diameter of 8mm was used for the test specimens.
Figure 4.7 – Temperature-time data for preliminary thermal control tests at 200°C

a) 10mm diameter specimen

b) 8mm diameter specimen

a) 10mm diameter specimen

b) 8mm diameter specimen
After confirming that the thermal behaviour in the specimen during heating and holding at temperature was stable, work was carried out to optimize the thermal profile along the length of the specimen. The primary concern was to ensure that the peak temperature occurred close to the control thermocouple at the centreline of the specimen axially, and secondary to this was the minimization of the thermal gradients along the length of the specimen. Initial testing indicated that the thermal profile tended to be asymmetric in nature and the peak temperature did not occur at the centre. As indicated previously, this would be unacceptable during mechanical testing because significant dimensional changes in the specimen away from the control thermocouple would affect the temperature of the test being carried out. Hence, a number of investigations were carried out to determine the influence of the experimental apparatus and specimen geometry on the level of asymmetry in the thermal profile. Ideally, the thermal profile would be completely symmetrical, with the peak occurring at the centre where the control thermocouple was located.

The depth of threading of the specimen ends into the stainless steel grips was found to be critical with respect to obtaining a peak temperature at the centreline of the specimen length, as shown in Figure 4.8. This is due to the inverse relationship between the cross-sectional area of the electrical circuit and the power generated by the resistance heating technique. The deeper the specimen is threaded, the less power supplied and heat generated at the interface between the specimen and the grip. As a result, care was taken to ensure that the specimen was threaded to the same depth at each end into the two stainless steel grips. Furthermore, it was found that the load-train mounted copper blocks must be in firm contact with the stainless steel grips for sufficient control and
repeatability of thermal test conditions. This was achieved by threading the pull rod and copper contact blocks, and ensuring that the surfaces of the copper contact blocks and stainless steel grips were flush with each other and held in place with a locking nut.

![Diagram showing thermal profile](image)

Figure 4.8 – Effect of specimen threading depth on specimen thermal profile

The development of a uniform temperature (i.e. within 1-2°C) along the entire length of the specimen would enable the use of a lengthwise strain measurement system, and for this reason, an effort was made to reduce the thermal gradients along the length of the specimen. To lower the rate of heat transfer to the copper blocks the stainless steel grips were lengthened from 25mm to 35mm, and the area of the grips were reduced from 490mm² to 370mm². As can be seen in Figure 4.9 this had a noticeable improvement on the homogeneity of the thermal profile at the centre of the specimen, extending it out to a length of 5-8mm on either side of the peak temperature. With this in mind, the stainless steel grips were lengthened and the interfacial area between the steel and copper was
reduced, in order to lower the rate of heat transfer from the specimen ends to the copper blocks and thereby reduce the temperature gradients in the specimen.

Figure 4.9 – Effect of redesigned stainless steel grip on specimen thermal profile

a) Effect along entire specimen length

b) Effect along the central 40mm of specimen length
4.4 Strain Measurement

Strain measurement was carried out using a non-contact strain measurement system developed specifically for this research program. The strain measurement set-up consisted of a Sony DFW-SX900 high-resolution (1280x1024 pixels) digital video camera, Navitar 7000 zoom lens and a data acquisition system to collect images of the specimen cross-section during testing. These images were used to measure the instantaneous diameter of the specimen during testing, and strain was calculated from the change in diameter using the equation for true strain shown in Equation 4.1, assuming that the cross-section of the specimen remains circular at the point of measurement throughout the tension test.

\[
\varepsilon = -2\ln\left(\frac{D}{D_0}\right)
\]  

(4.1)

Where \( \varepsilon \) is the strain, \( D_0 \) is the original diameter of the specimen, and \( D \) is the instantaneous diameter. Strain calculations are described in Appendix A1.1.

The minimum strain increment measurable by this method can be calculated by considering the optimum case, where the diameter of the specimen almost completely fills the image created. This calculation is shown in Appendix A1.2, and results in a minimum measurable strain increment of 0.001566. More likely is a case where the specimen diameter covers only 75% of the width of the image. The minimum measurable strain increment in this case is 0.002076. It should be noted that the minimum measurable strain increment becomes larger as the test progresses and the specimen becomes thinner.
This method of strain measurement is advantageous over other non-contact strain measurement systems in that it is possible to perform several measurements on a single image to locate the minimum diameter. Laser dilatometry measures the diameter at a single point and it is not certain that the measurement occurs at the minimum diameter.

4.4.1 Image Acquisition and Analysis

Analysis of the images acquired from the digital camera was carried out using Image Tool for Windows, developed by the University of Texas Health Science Centre in San Antonio (UTHSCSA). Instantaneous specimen diameter was determined by measuring the number of pixels across the specimen width and converting this value to a distance. As shown in Figure 4.11, several measurements were taken for each image in order to determine the minimum diameter of the specimen, and the minimum value was used to calculate the instantaneous strain.

Figure 4.11 – Image of tensile specimen indicating diameter measurements
In order to calibrate the strain measurement system, the diameter at the centre of each specimen was measured using a Mitutoyo digimatic caliper prior to testing. When the specimen was at the test temperature, an image was acquired at the centre of the specimen prior to loading using the digital camera system. The relationship between distance and the number of pixels was derived from this image, with an adjustment for the thermal expansion of the specimen according to Equation 4.2.

\[ \alpha \Delta T = -2.3493 \times 10^{-2} + 1.98 \times 10^{-5} T + 3.625 \times 10^{-8} T^2 \]  

(4.2)

Where \( \alpha \) is the coefficient of thermal contraction, \( \Delta T \) is the change in temperature between 577°C and the temperature of interest, and \( T \) is the temperature of interest. This equation was calculated previously by Wiskel for thermal contraction during cooling\[3\].

4.5 Tension Testing Procedure

The experimental program was carried out using the Gleeble-Instron apparatus described above. The apparatus was assembled as shown in Figures 4.4 to 4.6, with the control thermocouple positioned at the axial centerline of the tensile specimen. Care was taken to ensure that the specimen was evenly threaded into the two stainless steel grips, and also that no loading of the specimen occurred prior to testing. The camera was mounted on a tripod and positioned so that an image containing both edges of the specimen would be acquired. A backlight positioned behind the apparatus ensured there was sufficient edge resolution. Immediately prior to the start of the test, the cooling water system was switched on to remove heat from the copper components.
A thermal history of the form shown in Figure 4.12 was imposed on the specimen, and four thermocouples were used to acquire temperature data along the length of the specimen throughout each test. The tensile specimens were heated at 1°Cs\(^{-1}\) to a test temperature in the range 500-580°C, then held at temperature for one minute to ensure a stable temperature profile prior to deformation at a constant crosshead speed until failure. During holding, the position of the digital camera was checked to ensure it was targeted at the location where the specimen was expected to neck (i.e. at the peak temperature), and then the data acquisition system was activated to collect load data and images.

After the hold period, downward movement of the crosshead was initialized, imposing deformation on the specimen through the cage assembly. Images and load data were acquired until specimen failure occurred, at which point the electrical circuit was broken, causing the Gleeble to switch off automatically and stop heating.

Specimen load was measured using a D tensile load cell calibrated to 8 load ranges on a MTS 442 Controller with external voltage divider for 0 to 100lb (445N). The output signal from the load cell amplifier was digitized using the same sampling interval as the acquisition of camera images. Most tests were carried out with a sampling rate of 1Hz, however data from quicker tests, such as those performed under high deformation rates and high temperatures, were acquired at sampling rates in the range 1.75-1.9Hz. The limiting factor preventing higher sampling rates was the speed of the acquisition system, which used a LabVIEW 6i program running on a Dell notebook with a 733MHz Pentium III processor and 256Mb RAM, a RATOC CBFW1U IEEE1394 Cardbus PC card to acquire digital images from the camera system, and an Omega DAQ PC card to acquire voltage data simultaneously.
A total of 31 tension tests were run and the experimental test matrix is shown Table 4.2. The temperature gradient along the specimen during testing, strain rate, stress and strain were evaluated from the measured data and used in the data analysis, and are shown in Appendix B1.

4.6 Analysis of Data

Stress and strain values were obtained from the data acquisition system using an Excel spreadsheet that converted load and specimen diameter data from the data acquisition system into true stress-true strain values. A full description of this procedure is given in Appendix A1.1.
Table 4.2 - Experimental matrix for tension tests

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Cross-Head Speed (mms⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td></td>
</tr>
<tr>
<td>520</td>
<td></td>
</tr>
<tr>
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<td>0.00847</td>
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<tr>
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</tr>
<tr>
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<td>565</td>
<td>0.08467</td>
</tr>
<tr>
<td>570</td>
<td></td>
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<tr>
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<td>0.21167</td>
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<td></td>
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<td>540</td>
<td></td>
</tr>
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<td>560</td>
<td>2.11667</td>
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<td>580</td>
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</tbody>
</table>
References


Chapter V – Mathematical Model

The tests performed in the experimental section of this project have been carried out to obtain accurate mechanical properties in the region of the solidus temperature. However, the presence of a thermal gradient along the length of the tensile specimen during testing made it difficult to obtain an exact measurement of the deformation temperature. Hence, in order to obtain an estimation of the deformation temperature, as well as an improved understanding of the temperature profile, the steady-state thermal conditions in the specimen were simulated using the commercial Finite Element (FE) software package, ABAQUS™. ABAQUS is a comprehensive, general-purpose finite element analysis package that is able to simulate the thermal state existing in a material for a given set of conditions.

In addition to providing a more accurate estimation of the deformation temperature during testing, the model developed in ABAQUS has also been used to improve the design of the experimental apparatus by providing a means with which to investigate patterns of heat transfer during testing. The model also provides a basis for further investigations into transient thermal analyses of Gleeble specimens during resistance heating, and it would be possible to apply mechanical boundary conditions to the model as a means to simulate the material constitutive behaviour.

5.1 Finite Element Formulation

Referring to Figure 5.1, the generalized concept of finite element analysis is to obtain an approximate solution, $\phi$, for a problem, represented by $A(\phi) = 0$, in the domain $\Omega$, with boundary conditions represented by $B(\phi) = 0$ on boundary $\Gamma$, using a series of
differential equations developed for sub-domain regions $\Omega_e$, known as elements. Using this approach, it is possible to solve a large number of complex engineering problems dealing with a wide range of phenomena, including heat transfer. Usually the desired solution in heat transfer investigations is the temperature distribution throughout the domain, resulting from prescribed boundary conditions such as fixed heat fluxes or temperatures. The reader is referred to the book written by Zienkiewicz and Taylor for a complete explanation of the issues related to finite element analysis techniques\textsuperscript{[1]}, as a complete discussion of this solution technique is beyond the scope of this thesis.

5.2 Thermal Model

A 2-D axisymmetric heat conduction model was developed to describe heat transfer in the tensile specimen, represented in Figure 5.2 by a cylinder. Heat transfer in the specimen was assumed to occur in the radial or $r$-direction to the outer surface as well as the axial or $z$-direction towards the ends of the specimen. Heat transfer in the $\theta$-direction was assumed to be equal to zero. Hence, this model is applicable in cases where there is little or no circumferential variation in heat transfer.

\textbf{Figure 5.1 – Schematic of problem domains as they apply to the FE method\textsuperscript{[1]}}
In view of the above assumptions, the flow of heat in the specimen can be described as follows.

\[ \frac{1}{r} \frac{\partial}{\partial r} \left( kr \frac{\partial T}{\partial r} \right) + \frac{\partial}{\partial z} \left( k \frac{\partial T}{\partial z} \right) + \dot{Q} = \rho C_p \frac{\partial T}{\partial t} \]  

(5.1)

In Equation 5.1, the thermal conductivity, \( k \) (Wm\(^{-1}\)K\(^{-1}\)), is temperature-dependent and has been assumed to be isotropic, and \( \rho \) (kgm\(^{-3}\)) is the density and \( C_p \) (kJkg\(^{-1}\)C\(^{-1}\)) is the specific heat at constant pressure. For steady state analysis only the thermal conductivity term is important, as \( \frac{\partial T}{\partial t} \) is zero under these conditions. As such, Equation 5.1 can be rewritten as follows.
\[
\frac{1}{r} \frac{\partial}{\partial r}(kr \frac{\partial T}{\partial r}) + \frac{\partial}{\partial z}(k \frac{\partial T}{\partial z}) + Q = 0 \tag{5.2}
\]

The internal heat generation occurring as a result of the resistance heating technique employed by the Gleeble is accounted for by the volumetric heat flux per, \( Q \) (Wm\(^{-3}\)).

The model requires specification of the specimen geometry, thermal boundary conditions and the thermophysical properties of the material.

5.2.1 Geometry

The model developed in ABAQUS to simulate the temperature profile of the tensile specimen was a 2-D axisymmetric model and the mesh can be seen in Figure 5.3. It has been assumed that the temperature is uniform around the circumference of the specimen at any given radius, \( r \), and time. The entire length of the tensile specimen was modeled, as the temperature profile was not symmetric about the horizontal centerline of the specimen. In order to reduce the complexity of the model, other components surrounding the free span of the specimen, such as the stainless steel grips and copper contact blocks, were not included and were represented by appropriate boundary conditions. The domain had a total height of 109mm with the gauge length occupying the central 100mm. The finite element mesh consisted of 259 2-D 4-noded isoparametric axisymmetric elements, with a total of 372 nodes.
Model predicted temperature gradients in the radial direction were found to be very small, and therefore a sensitivity analysis of the resolution of the mesh in the radial direction was not carried out. However, a sensitivity analysis was carried out to determine the influence of mesh resolution in the longitudinal, z-direction, on the model predicted temperatures. This analysis revealed that the model predicted temperatures were not sensitive to any decrease in the longitudinal resolution below 1.25mm. Therefore, a longitudinal resolution of 1.25mm was chosen for this investigation. This corresponded to 79 elements along the length of the specimen.

Figure 5.3 – 2-D Axisymmetric Finite Element Mesh of Tensile Specimen
5.2.2 Material Properties

Wiskel\cite{2} compiled the thermo-physical properties for AA5182 from available literature. The thermal conductivity data used for the steady-state thermal analysis presented in this work is shown in Table 5.1.

Table 5.1 – Thermal conductivity data for AA5182 used in the thermal model\cite{2}

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>k (Wm(^{-1})K(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>122.32</td>
</tr>
<tr>
<td>100</td>
<td>125.43</td>
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<td>500</td>
<td>134.78</td>
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<td>536</td>
<td>150.35</td>
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<tr>
<td>550</td>
<td>152.39</td>
</tr>
<tr>
<td>577</td>
<td>154.93</td>
</tr>
<tr>
<td>600</td>
<td>130.80</td>
</tr>
</tbody>
</table>

5.2.3 Simulation of Internal Heat Generation

The method by which the resistance heating technique is simulated must also be considered. Because of the focus on steady-state thermal behaviour, we are only interested in the resistance heating being applied during the hold. As a result, it was possible to treat the resistance heating effect as a fixed volumetric heat flux using an internal heat generation subroutine in ABAQUS. An analytical solution based on the equation for electrical power, shown in Equation 5.3, has been used to estimate this heat flux for each experiment.
\[ P = I^2 R \]  \hspace{1cm} (5.3)

In Equation 5.3, electrical power, \( P \), is given as a function of current, \( I \), and resistance, \( R \). It is known that the Gleeble operates with a mean electric current, \( I \), in the region of 2000A, which was assumed as a constant for the purposes of this investigation\[3\]. The resistance, \( R \), can be calculated from Equation 5.4.

\[ R = \rho_A L / A \]  \hspace{1cm} (5.4)

In Equation 5.4, the electric resistance in the specimen is calculated as a function of the resistivity of the material, \( \rho_A \) (\( \mu \Omega \)cm), as well as the length, \( L \) (m), and cross-sectional area, \( A \) (m\(^2\)), of the specimen. Resistivity is a material property and is dependent on temperature, as shown in Figure 5.4\[4\]. According to Figure 5.4, resistivity for aluminum alloys have not been determined at temperatures much above 100K. However, it is apparent for Figure 5.4 that the resistivity of aluminum alloys approaches that of pure aluminum at elevated temperatures. Consequently, the resistivity values used in this investigation are those of pure aluminum.
Figure 5.4 – Electrical resistivity of pure aluminum and aluminum alloys as a function of temperature\(^4\)

Equation 5.3 can be solved to give a constant value of power generated within the tensile specimen during the pre-testing hold stage at the test temperature for given temperature, power angle and current. This value can then be converted to a volumetric heat flux using Equation 5.5, where \(Q\) is the heat flux, \(P\) is the power or energy supplied.
per second, and \( V \) is the volume of the gauge length of the specimen, which is assumed to be a constant value of \( 5.55 \times 10^{-6} \text{m}^3 \). The calculated heat flux is then applied to the domain \( \Omega_1 \), shown in Figure 5.3.

\[
\dot{Q} = \frac{P}{V} \quad (5.5)
\]

A typical value for the calculated volumetric heat flux, \( \dot{Q} \), used in the model is \( 41.94 \text{MWm}^{-3} \) for a temperature of \( 500^\circ \text{C} \). This volumetric heat flux is applied to the model domain to simulate the heat generated in the specimen. Heat transfer coefficients at the ends of the specimens were adjusted until satisfactory agreement was reached between the measured and predicted temperatures.

5.2.4 Boundary Conditions

Four thermal boundary conditions were applied to the domain of the 2-D thermal model. These include a free convection boundary condition, a fixed heat flux boundary condition at each end of the specimen, and a fixed volumetric heat flux boundary condition to account for internal heat generation due to the resistance heating process.

A free convection boundary condition of the form shown in Equation 5.6 was applied to surface \( \Gamma_3 \), the surface of the tensile specimen.

\[
q = h(T_w - T_{amb}) \quad (5.6)
\]
In Equation 5.6, the ambient temperature, $T_{amb}$, was assumed to be 25°C, and the convection heat transfer coefficient, $h$, was assumed to be a constant value of 10 Wm$^{-2}$°C$^{-1}$. This value of the convection heat transfer coefficient was chosen after consideration of literature data available for a range of natural convection conditions, and was further modified to incorporate the effect of radiative heat transfer from the specimen surface $^{[5]}$.

A second heat transfer process that must be accounted for is the transfer of heat from the free span of the tensile specimen into the grip assembly. Fourier’s law of heat conduction can be used to describe the heat flow from the specimen into the grip apparatus, and is shown in Equation 5.7.

$$q = -k\frac{\partial T}{\partial z}$$

(5.7)

In Equation 5.7, $q$ (Wm$^{-2}$) is the heat flux and $k$ (Wm$^{-2}$°C$^{-1}$) is the thermal conductivity. In order to account for heat transfer from the specimen into the grip assemblies at each end of the specimen, fixed heat flux boundary conditions have been applied to surfaces $\Gamma_1$ and $\Gamma_2$ at each end of the specimen, as shown in Figure 5.2. Typical values for these heat fluxes were approximately $-300$ kWm$^{-2}$.

5.3 Application of the Model

The model has been used to generate temperature profiles along the surface of the gauge length of the specimen that match the measured experimental data taken from the thermocouples attached to the specimen during testing. From the thermal profiles predicted by the model it is possible to determine the position and value of the maximum
temperature occurring along the length of the specimen for each test, thereby obtaining a closer approximation of the deformation temperature than the reading from the control thermocouple.

The model was developed using ABAQUS version 6.3, and was run on an SGI Origin™ 200 machine equipped with four 175MHz MIPS® 64-bit R10000 microprocessors. The total CPU time of the model was approximately 4 minutes.

The values of the fixed heat flux boundary conditions at the specimen ends were adjusted until model predictions matched the measured temperatures generated by thermocouples positioned along the length of the specimen during testing. Initially, the model was used to simulate a test condition of 500°C (i.e., the control T/C was programmed to hold at 500°C prior to testing). Simulations of higher test temperatures were developed by adjusting the BC’s in the model generated for 500°C, to account for the higher volumetric heat flux due to the higher test temperatures and increased heat flux at the ends of the specimen. Model predicted temperatures for control thermocouple temperatures of 500°C, 540°C and 580°C are shown in Figure 5.5. It is clear that the model predicts the thermal distribution in the specimen fairly accurately, particularly in the region of interest along the central section of the gauge length (i.e. distance ~ 0.05m). The boundary conditions used in the model for each case can be seen in Table 5.2.
Figure 5.5 – Measured temperatures and model predicted temperatures for experimental tests carried out at control thermocouple temperatures of:

a) 500°C, b) 540°C and c) 580°C.
Table 5.2 – Boundary condition data for model predicted temperatures shown in Figure 5.5, including volumetric heat flux for resistance heating and fixed heat fluxes at specimen ends.

<table>
<thead>
<tr>
<th>Control Thermocouple Temperature (°C)</th>
<th>Volumetric Heat Flux from analytical solution (MWm⁻³)</th>
<th>Heat flux at top end (kWm⁻²)</th>
<th>Heat flux at-bottom end (kWm⁻²)</th>
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<tr>
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<td>-423</td>
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<tr>
<td>580</td>
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<td>-377</td>
<td>-465</td>
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</table>

It should be noted that the resistivity used to derive the internal heat generation heat flux has been calculated assuming that the entire specimen has a uniform temperature, determined by the temperature of the control thermocouple. In reality, there exists a significant temperature gradient along the length of the specimen. Lower local temperatures away from the control thermocouple close to the specimen ends result in lower local resistivities, and therefore internal heat generation close to the specimen ends would be smaller than the values used in the model. To compensate for the excess heat generated due to this unrealistic boundary condition, it is likely that the fixed heat flux boundary conditions at the specimen ends are unrealistically high also. Nevertheless, the model is able to predict the thermal profile along the length of the specimen fairly accurately, particularly along the central section of the gauge length, which is the region of interest.
References


Chapter VI – Results and Discussion

Measurements of the tensile properties of an as-cast AA5182 aluminum alloy between 500°C and 580°C and at strain rates applicable to the DC casting process (i.e., $10^{-4}$ s$^{-1}$ to $10^{-2}$ s$^{-1}$) have been performed according to the experimental matrix described previously. For each test, the applied strain rate has been calculated from experimental data and the thermal model has been used to estimate the exact deformation temperature. A post-testing microstructural investigation has been carried out on several specimens using optical and scanning electron microscopy techniques to develop an understanding of the change in mechanical properties and fracture behaviour with temperature and microstructure. The fraction liquid in each specimen has also been estimated in an attempt to develop a relationship between the tensile properties, fracture behaviour, and the presence of liquid in the alloy.

6.1 True-Stress True Strain Measurements of As-Cast AA5182

Experimental measurements indicate that the measured flow stress is sensitive to deformation temperature and strain rate. True stress-true strain measurements of as-cast AA5182 between 503°C and 582°C at a strain rate of $\sim 2.0 \times 10^{-3}$ s$^{-1}$ are shown in Figure 6.1. Under a constant strain rate, the measured steady-state flow stress decreases with increasing temperature. As temperature increases, dislocations become more mobile, their ability to cross-slip and climb is enhanced, and the flow stress required for plastic deformation decreases. Also, the strain to failure decreases to very small levels (i.e. $\varepsilon_f < 0.01$) as temperature increases.
Figure 6.1 – True-stress true-strain results for as-cast AA5182 under a constant applied strain rate of \(~2.0 \times 10^{-3} \text{s}^{-1}\) at temperatures:

a) 503°C-571°C and b) 571°C-582°C
6.2 Determination of Strain Rate and Deformation Temperature

The mechanical testing machine used to perform the tension tests applies strain under stroke-control (i.e. movement of the crosshead at a constant speed). As it is not controlled directly, the strain rate experienced by the specimen will increase during the test after necking begins, and the material around a region of locally reduced specimen diameter may also become hotter due to an increase in the local current density. Figure 6.2 shows the variation in strain with time during a tension test performed at 500°C with a crosshead speed of 0.08467 mms\(^{-1}\). In Figure 6.2, strain was calculated based on measurements of the instantaneous diameter from images acquired by digital the camera system. Strain rate is relatively constant early in the test (up to ~60sec), and increases later due to localized deformation arising from combined effects of plastic instability and thermal gradients in the specimen.

![Figure 6.2 - Strain-time results for as-cast AA5182 tension tested at 500°C at a crosshead speed of 0.08467mms\(^{-1}\)](image)

Figure 6.2 – Strain-time results for as-cast AA5182 tension tested at 500°C at a crosshead speed of 0.08467mms\(^{-1}\)
The applied strain rate reported for each test is shown in Table 6.1, and was derived from the change in strain with time during the first stage of the tension test. Strain rates have not been reported for tests in which a very small strain to failure was measured. The instantaneous strain in the specimen was calculated using the dimetral strain equation (Equation 4.1), and the instantaneous diameter of the specimen during deformation measured from images collected by the digital camera system described earlier. Variation in the measured strain rate between specimens tested under the same crosshead speed was observed. This arose due to variation in the temperature profile along the length of each specimen, and the resultant variation in effective gauge length from specimen to specimen.

The mathematical model described in the previous chapter has been used to estimate the deformation temperature of each specimen during testing. The estimated deformation temperature was taken to be the maximum predicted temperature along the axial length of the specimen. Model predicted deformation temperatures are shown in Table 6.1 alongside the calculated strain rates. The error for all reported temperature values in this study is +/-3°C.
Table 6.1 - Model Predicted Deformation Temperatures and Initial Strain Rates

<table>
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<tr>
<th>Specimen ID</th>
<th>Test Temperature (°C)</th>
<th>Model Predicted Deformation Temperature (°C)</th>
<th>Crosshead Speed (mm s(^{-1}))</th>
<th>Calculated Initial Strain Rate (s(^{-1}))</th>
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</table>
6.3 Tensile Properties of As-Cast AA5182 at Temperatures Close to the Solidus

Tensile strength results for the entire test matrix are presented in Figure 6.3. The results are also tabularized in Appendix B1. The range of crosshead speeds was equivalent to a strain rate range from \( \sim 2.0 \times 10^{-4}\text{s}^{-1} \) to \( \sim 7.5 \times 10^{-2}\text{s}^{-1} \). The maximum stress has been defined as the steady state flow stress, or, in the absence of plastic deformation, the maximum stress prior to fracture. Measurements carried out at the highest crosshead speed above 540°C may be inaccurate, as deformation and fracture took place too quickly for the sampling frequency of the data acquisition system to resolve the loading history sufficiently. A sharp decrease to low strength occurs between 570°C and 575°C at all other strain rates, and strain rate sensitivity is observed at all temperatures up to the transition. At temperatures above 570°C no sensitivity to strain rate was observed.

Figure 6.3 – Variation in maximum tensile stress with temperature between 500°C and 580°C under applied strain rates between \( \sim 2.0 \times 10^{-4}\text{s}^{-1} \) and \( \sim 7.5 \times 10^{-2}\text{s}^{-1} \)
Ductility measurements for the entire test matrix are shown in Figure 6.4. The ductility has been quantified by measuring the percentage reduction in area (%RA) between the original specimen cross-sectional area and the area of the fracture surface. The ductility of the alloy decreases to an insignificant value (i.e. %RA<0.01) as temperature increases, and the temperature at which this occurs is sensitive to the applied strain rate. Loss of ductility occurs at lower temperatures when the alloy is subjected to higher strain rates. Specimens tested at ~560°C had very low ductility when tested at a strain rate of ~10^{-2}s^{-1}, whereas specimens tested at similar temperatures and lower strain rates were more ductile. All specimens tested above 570°C showed no significant ductility.

![Figure 6.4 - Variation in ductility (%RA) with temperature between 500°C and 580°C under applied strain rates between ~2.0x10^{-4}s^{-1} and ~7.5x10^{-2}s^{-1}](image-url)
6.3.1 Temperature Effects

Referring to Figures 6.1, 6.3, and 6.4, it is clear that there are two transitions in the mechanical behaviour of the alloy between 565°C and 575°C. Under test conditions of 566°C and \( \sim 2 \times 10^3 \text{s}^{-1} \), plastic deformation occurs at a steady-state flow stress of 13.1MPa to a strain of 0.25, at which point failure occurs. At 571°C and \( \sim 2 \times 10^3 \text{s}^{-1} \), a maximum tensile stress of 10.8MPa is measured, however, no plastic deformation occurs and failure takes place immediately upon reaching the maximum stress. This change in mechanical response suggests that the alloy loses all significant ductility between 566°C and 571°C. The test conducted at 575°C and \( \sim 2 \times 10^3 \text{s}^{-1} \) indicated that the alloy remains brittle, and the maximum stress had reduced to a very low level, suggesting that the alloy loses all significant mechanical strength between 571°C and 575°C.

6.3.2 Strain Rate Effects

Figure 6.5a) shows the mechanical response of the as-cast AA5182 alloy under a range of applied strain rates from \( \sim 2.0 \times 10^4 \text{s}^{-1} \) to \( \sim 4.0 \times 10^7 \text{s}^{-1} \) at approximately 500°C. As expected, there is an increase in the measured steady-state flow stress as the applied strain rate increases. Strain rate sensitivity is observed at all temperatures up to 570°C.

The influence of strain rate above 570°C is different to that observed at lower temperatures. Figure 6.5b) shows the mechanical response of the as-cast AA5182 alloy under a range of applied strain rates from \( \sim 2.0 \times 10^3 \text{s}^{-1} \) to \( \sim 4.0 \times 10^2 \text{s}^{-1} \) at approximately 580°C. No clear relationship can be seen between the maximum stress and the applied strain rate. In contrast, the failure strain appears to increase with increasing strain rate, although the maximum strain measured at all strain rates is very small.
Figure 6.5 – True stress-true strain results for as-cast AA5182 at a range of applied strain rates: a) at ~500°C and b) at ~580°C
6.3.3 Repeatability

The repeatability of experiments carried out using the Gleeble-Instron apparatus was investigated as part of this study. This was done to ascertain the level of confidence with which the measured properties can be treated. Three specimens were tested under the same experimental conditions. Each specimen was heated at a rate of 1°C/s to 560°C, held for one minute, and tensile tested at a strain rate of ~5.0x10^{-3}s^{-1}. The true-stress true-strain and strain-time results are shown in Figure 6.6 and 6.7 respectively.

The measured steady-state flow stresses in each test are very close, however a significant variation in the strain to failure was found. Two specimens failed after deforming to strains of 0.18 and 0.21, while the third failed much earlier, at a strain of 0.07. It is likely that a casting defect such as porosity was present in the third specimen, or that damage to the specimen prior to testing had occurred, that caused it to fail at such low strain. Also shown are strain-time data for the repeatability tests in Figure 6.7. The strain rate contains almost no variance at all. It was concluded that in terms of the flow stress data, the experimental method is repeatable given that specimen conditions are constant however the strain to failure is much more variable, especially when tested at temperatures close to the solidus. This is likely due to the variability of the local microstructure in the vicinity of the deformation.
Figure 6.6 – True stress-true strain results of the repeatability analysis

\[ T_{def} = 560^\circ C, \text{ strain rate}=5 \times 10^{-3} \text{ s}^{-1} \]

Figure 6.7 – Strain rate measurements for repeatability tests
6.4 Metallographic Examination of Specimens Tested at ~5.0x10^{-3}s^{-1}

An examination of the microstructure of the tested specimens was carried out to determine what microstructural changes occur in the alloy as temperature increases, and what effect these changes have on the measured mechanical properties presented above.

Specimens tested at an applied strain rate of ~5.0x10^{-3}s^{-1} were mounted axially in resin according to Figure 6.8. The mounted specimens were then milled back by 4mm so that the interior of the specimen was exposed. The exposed surface of the specimen was polished and etched in a solution of Barker’s reagent in order to make the grain structure of the specimen visible. In this way, a cross-sectional ‘slice’ of the fracture surface, as well as the microstructure immediately below the area of fracture, was obtained. In all micrographs shown, the specimen has been positioned such that the tensile direction of loading occurred in the horizontal direction across the image.

Figure 6.8 – Preparation of metallographic specimens from tensile test pieces
An etched cross-section of the fracture surface of the specimen tested at 522°C is shown in Figure 6.9. Second phase particles are elongated in the direction of the applied tensile load as a result of large amounts of deformation during testing.

Figure 6.9 – Micrograph showing a cross-section of the ductile fracture surface formed during tension testing at 522°C under an applied strain rate of \( \sim 5.0 \times 10^{-3} \text{s}^{-1} \)

Intergranular openings were observed some distance away from the fracture surface in all specimens tested at 541°C and above. A typical intergranular opening in a specimen tested at 541°C is shown in Figure 6.10. Loading took place in the horizontal direction across the width of the image, and consequently the interdendritic openings are either perpendicular to, or 45° from the load direction. Bridging between intergranular openings is also visible.
Figure 6.10 – Micrograph showing intergranular openings occurring as a result of tensile loads in a specimen tested at 541°C under a strain rate of $5.0 \times 10^{-3} \text{s}^{-1}$

Although intergranular openings were found within the specimen tested at 541°C, the area close to the fracture surface exhibited many similar characteristics to the ductile fracture surface shown in Figure 6.9. In contrast, the fracture surfaces of all specimens tested above 541°C were located along grain boundaries. In some cases, cracks propagated along several grain boundaries prior to complete fracture and as a result these specimens contained several large cracks away from the fracture surface. The surfaces of a large crack in a specimen tested at 569°C can be seen in Figure 6.11. The intergranular nature of the fracture can be clearly seen, and in comparison with fracture occurring at 522°C (referring to Figure 6.9), no evidence of deformation is observed.
Figure 6.11 – Micrograph showing brittle, intergranular cracking in a specimen tested at 569°C under an applied strain rate of \( \sim 5.0 \times 10^{-3} \text{s}^{-1} \)

Figure 6.12 shows an area of the fracture surface of a specimen tested at 577°C. Intergranular fracture can be seen along several grain boundaries. A deformed grain is visible, and appears to have been bent outwards from the fracture surface. This indicates that sufficient coherency existed in the alloy for deformation of the grain to take place.

The microstructure of the specimen tested at 582°C is quite different to that observed in specimens tested at lower temperatures. The cross-section of the fracture surface, shown in Figure 6.13, indicates that a large number of voids have formed behind the fracture surface of the specimen. In contrast with intergranular openings observed at lower temperatures, these voids do not appear to be located solely at grain boundaries, and many are aligned perpendicular to the loading direction.
Figure 6.12 - Micrograph showing intergranular fracture surfaces, sub-surface cracks and voids in a specimen tested at 577°C and a strain rate of \(-5.0 \times 10^{-3}\) s\(^{-1}\).

Figure 6.13 - Micrograph showing extensive intergranular openings close to the fracture surface of a specimen tested at 582°C and a strain rate of \(5 \times 10^{-3}\) s\(^{-1}\).
The microstructural investigation indicates that as-cast AA5182 is ductile between 500°C and 540°C. Throughout this temperature range, the amount of deformation observed in each specimen decreases as temperature increases, in agreement with the measured decrease in ductility. At 541°C, there is little evidence of deformation, and the measured ductility is low. The microstructural examination showed that intergranular openings were present in the specimen below the fracture surface. All specimens tested above 541°C were found to contain increasing numbers of intergranular openings. Over the same temperature range, the measured ductility decreased from already low levels to an insignificant amount. As the solidus temperature reported in the literature is 536°C\(^{[1]}\), it is possible that softening or liquidation of eutectic phases present in intergranular regions of the alloy first occurred in the specimen tested at 541°C, resulting in loss of ductility and the opening of the intergranular areas. As temperature increases, the amount of liquid or softened eutectic would be expected to increase, resulting in further reduction in ductility.

The sharp decrease in strength between 570°C and 577°C was not accounted for by a visible change in the microstructure. However, the microstructure observed changed significantly above 577°C. Many more voids were observed in the specimen tested at 582°C, and these may have formed as a result of the movement of liquid through interdendritic regions during loading. This would account for the orientation of voids perpendicular to the loading direction, because liquid would move to compensate for any straining. In comparison, intergranular openings at lower temperatures tended to be oriented 45° to the loading direction. This could be explained by grain boundary sliding activated by small amounts of liquid at grain boundaries. Examination of the fracture
surfaces is required to explain the changes in mechanical response and fracture behaviour with respect to the presence of liquid within the alloy at these temperatures.

6.5 Fracture Surface Investigation of Specimens Tested at \( \sim 5.0 \times 10^{-3} \text{s}^{-1} \)

The fracture surfaces of specimens tested at a strain rate of \( \sim 5.0 \times 10^{-3} \text{s}^{-1} \) have been examined using a scanning electron microscope in an attempt to relate the mechanical response and fracture behaviour of as-cast AA5182 to the deformation temperature and any presence of liquid in the alloy.

6.5.1 Low Magnification (x30-x50)

Examination of the specimen fracture surfaces has been carried out under low magnifications to confirm the nature of fracture taking place between 500°C and 580°C.

Figure 6.14 is an image of the fracture surface of the specimen tested at 501°C, showing typical ductile fracture characteristics, such as pitting and evidence of void formation and coalescence. Also visible is the melted region of the fracture surface that forms on the fracture surfaces of resistance heated tensile specimens as a result of electrical arcing at the moment of complete fracture. Ductile fracture characteristics are evident on the fracture surfaces of specimens tested below 541°C. The fracture surfaces change above 541°C, and features indicative of ductile fracture are replaced by a smoother, intergranular fracture surface with visible grain boundaries. Figure 6.15 and 6.16 show the fracture surfaces of specimens tested at 541°C and 569°C. The fracture surface of the specimen tested at 582°C is shown in Figure 6.17. Grain boundaries and dendritic features are visible on the intergranular fracture surface.
Figure 6.14 – SEM micrograph showing ductile fracture characteristics on the fracture surface of a specimen tested at 501°C and a strain rate of $\sim 5.0 \times 10^{-3} \text{s}^{-1}$

Figure 6.15 – SEM micrograph showing ductile fracture characteristics on the fracture surface of a specimen tested at 541°C and a strain rate of $\sim 5.0 \times 10^{-3} \text{s}^{-1}$
Figure 6.16 – SEM micrograph showing intergranular fracture characteristics on the fracture surface of a specimen tested at 569°C at a strain rate of $\sim5.0 \times 10^{-3}\text{s}^{-1}$

Figure 6.17 – SEM micrograph showing intergranular fracture characteristics on the fracture surface of a specimen tested at 582°C and a strain rate of $\sim5.0 \times 10^{-3}\text{s}^{-1}$
6.5.2 High Magnification (x400-x800)

A fracture surface investigation has been carried out at higher magnification to determine whether there is any evidence of grain boundary sliding in the specimens tested at 541°C, where both ductile fracture and intergranular openings have been observed in the metallographic examination presented previously in Figure 6.15.

Sharp secondary cracks are visible in specimens tested at 501°C and 522°C, shown in Figure 6.18. These secondary cracks are characteristic of high temperature ductile fracture in aluminum alloys and typically have a clean surface. However, in the specimen tested at 541°C the fracture surface and secondary cracks observed are covered with a fine 'feathered' phase, shown in Figure 6.19. This suggests that a large amount of highly localized deformation has occurred at the grain boundary region prior to fracture.

Figure 6.18 – SEM micrograph showing a secondary crack on the fracture surface of a specimen tested at 501°C and a strain rate of ~5.0x10⁻³ s⁻¹
Figure 6.19 – SEM micrograph showing whiskers on a secondary crack on the fracture surface of a specimen tested at 541°C and a strain rate of ~5.0x10⁻³ s⁻¹

The whiskers are not as widespread on the fracture surface of the specimen tested at 569°C, shown in Figure 6.20. Instead, the fracture surface is smoother, and is decorated with second phase particles. The fracture surface of the specimen tested at 577°C is also smooth, and is decorated with second phase particles, as shown in Figure 6.21. As the figures show, the fracture surface of the specimen tested at the higher temperature appears to be smoother and less decorated with second phase particles. At 582°C, a very smooth fracture surface with no second phase particles was observed. The dendritic microstructure of the grains had been exposed and bridging between dendrites was visible, as shown in Figure 6.22.
Figure 6.20 – SEM micrograph showing whiskers on a secondary crack on the
fracture surface of a specimen tested at 569°C and a strain rate of $-5.0 \times 10^{-3}\text{s}^{-1}$

Figure 6.21 – SEM micrograph showing grain boundary and precipitated particles
on the fracture surface of a specimen tested at 577°C and $-5.0 \times 10^{-3}\text{s}^{-1}$
An examination of the fracture surfaces of specimens tested at 501°C, 522°C and 541°C indicates that fine whiskers appear increasingly on the fracture surface as temperature increases. These whiskers may be evidence of large amounts of plastic deformation occurring in localized areas at grain boundaries prior to fracture. The presence of whiskers on intergranular fracture surfaces has been recorded previously in a number of studies investigating superplasticity in wrought aluminum alloys at temperatures and strain rates similar to those investigated here\textsuperscript{[2-4]}. One mechanism that has been proposed to explain the formation of whiskers during deformation is that of viscous flow at grain boundaries as a result of the presence of a soft or partially liquid phase in the intergranular region. Although this may not adequately explain the phenomenon of superplasticity in wrought aluminum alloys, a similar mechanism may operate during the deformation and fracture of as-cast AA5182 presented in this study.

Figure 6.22 - SEM micrograph showing evidence of significant amounts of liquid on the fracture surface of a specimen tested at 582°C and a strain rate of $\sim 5.0 \times 10^{-3}$ s$^{-1}$
At 541°C, testing was carried out close to the solidus temperature of the alloy, and softening and liquidation of eutectic phases at the grain boundaries is expected to occur in this temperature region.

At higher temperatures whiskers are less apparent, and the fracture surfaces, which have a smoother appearance, are decorated with second phase particles. The decrease in whisker occurrence corresponds to the temperature at which the alloy becomes extremely brittle. These changes in fracture characteristics and measured properties suggest that the soft or partially molten eutectic phase in the intergranular regions, responsible for the localization of plastic deformation at grain boundaries, has melted completely. As a result, the alloy would contain significant amounts of liquid in the form of films at the grain boundaries, leaving it prone to brittle intergranular fracture and exposing any second phase particles that have not melted. As the test temperature increased from 569°C to 577°C, it was observed that the number and size of the second phase particles appeared to decrease and the smoothness of the fracture surface increased, suggesting that the second phase particles are melting across this temperature range.

The dendritic structure of the alloy can be seen on the fracture surface of the specimen tested at 582°C, with few second phase particles observed. Clearly, a significant amount of melting has occurred in the alloy, and a liquid phase is present in the interdendritic regions at this temperature.

With reference to the microstructural development of AA5182 in Figure 2.1\([1]\), a number of eutectic reactions cause the precipitation of several intermetallic phases during solidification. The series of eutectic reactions have been summarized in Equations 6.1 to 6.5 with corresponding fraction solidified ($f_s$) and temperature data as follows;
\[
\text{Liquid} \rightarrow \alpha(Al) \quad f_s=0 \quad @632^\circ C \quad (6.1)
\]
\[
\text{Liquid} \rightarrow \alpha(Al) + Al_6(Fe,Mn) \quad f_s=0.4 \quad @623^\circ C \quad (6.2)
\]
\[
\text{Liquid} \rightarrow \alpha(Al) + Al_6(Fe,Mn) + Mg_2Si \quad f_s=0.93 \quad @582^\circ C \quad (6.3)
\]
\[
\text{Liquid} \rightarrow \alpha(Al) + Al_6(Fe,Mn) + Mg_2Si + Al_7Mg_5 + Al_3Fe \quad f_s=0.98 \quad @560^\circ C \quad (6.4)
\]
\[
\text{End of Solidification} \quad f_s=1 \quad @536^\circ C \quad (6.5)
\]

In the tests conducted in this study, the maximum test temperature was estimated to be 586°C. With respect to the solidification sequence given in Equations 6.1 to 6.5, it is likely that one or more of the Mg$_2$Si, Al$_8$Mg$_5$ and Al$_3$Fe phases will melt, or partially melt, at the higher test temperatures. These phases all form close to the end of solidification ($f_s>0.93$), and therefore would be located in the intergranular and interdendritic regions. Chemical analysis of the second phase particles observed on the fracture surfaces is required to identify which eutectic phases remain as temperature and fraction liquid increase, and which phases have become part of the intergranular liquid. In this way, a link can be constructed between temperature, the solidification characteristics of the alloy, the microstructure present during testing, and the measured tensile response of the alloy.

6.5.3 Energy Dispersive X-ray (EDX) Analysis
An investigation was carried out using Energy-Dispersive X-ray Spectroscopy (EDX) to determine the likely chemical composition of the second phase particles that appear on the fracture surface of the alloy at various test temperatures. EDX can help characterize specimens by providing qualitative data on their elemental composition, and in some cases credible quantitative data may be obtained. Obtaining quantitative data from fracture surfaces, however, can be extremely difficult, so this analysis has been carried out to obtain a qualitative understanding of the elements contained within each type of precipitate in order to aid their identification with respect to the eutectic phases and intermetallic compounds that form during solidification. It is expected that this investigation will provide the final information required for a full description of the relationship between the deformation temperature, solidification characteristics of the alloy, microstructure and tensile properties.

Initially, EDX was performed across the entire specimen fracture surface to measure the chemistry of the overall alloy. The results, shown in Figure 6.23, indicate that only aluminum, magnesium and silicon are present in sufficient quantities on the fracture surface to register on the analysis (i.e. >0.20wt%). EDX analysis measured 94.27wt% aluminum, 5.43wt% magnesium and 0.30wt% silicon. The silicon content measured by EDX was higher than expected, and iron and manganese were not detected, suggesting that they were present at levels less than 0.20wt%. However, the results are in fairly good agreement with the alloy chemistry of AA5182 given previously in Table 4.1.
Figure 6.23 – EDX results of the fracture surface of a specimen tested at 541°C

The fracture surface of the specimen tested at 541°C was analyzed to determine the chemistry of the whiskers present at the fracture surface and secondary cracks. The results are shown in Figure 6.24, and indicate that the areas examined consisted predominantly of aluminum and magnesium. It is likely that the bulk alloy composition was measured because the EDX technique was unable to resolve the whiskers due to their small size. Alternatively, the whiskers may be composed of an intermetallic phase containing large amounts of aluminum and magnesium.
The second phase particles on the fracture surfaces of specimens tested between 569°C and 577°C have been analyzed. Three different particles were found; acicular particles on the fracture surface, globular particles embedded in the fracture surface, and clusters of faceted particles. Examples of the particles observed are shown in Figure 6.25, and the results of the EDX investigation are shown in Figures 6.26, 6.27 and 6.28. The EDX results in Figure 6.26 show that the acicular particles consist predominantly of aluminum and iron. Analysis of the embedded globular particles, shown in Figure 6.27, indicated that these are highly enriched in magnesium and silicon. The results of EDX carried out on clusters of faceted particles are shown in Figure 6.28. These particles were enriched in many alloying elements, including iron, silicon and manganese.
Figure 6.25 – Precipitates on the fracture surface of a specimen tested at 570°C and a strain rate of $-5.0 \times 10^{-3} \text{s}^{-1}$.

<table>
<thead>
<tr>
<th>Element</th>
<th>Concentration</th>
</tr>
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<tbody>
<tr>
<td>Magnesium</td>
<td>4.12 wt%</td>
</tr>
<tr>
<td>Aluminum</td>
<td>72.84 wt%</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.64 wt%</td>
</tr>
<tr>
<td>Manganese</td>
<td>3.21 wt%</td>
</tr>
<tr>
<td>Iron</td>
<td>19.20 wt%</td>
</tr>
</tbody>
</table>

Figure 6.26 – EDX of acicular particles on the fracture surface of a specimen tested at 570°C.
Figure 6.27 – EDX of globular, embedded particles on a specimen tested at 570°C

<table>
<thead>
<tr>
<th>Concentration</th>
<th></th>
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<tr>
<td>Magnesium</td>
<td>45.15 wt%</td>
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<tr>
<td>Aluminum</td>
<td>19.02 wt%</td>
</tr>
<tr>
<td>Silicon</td>
<td>35.82 wt%</td>
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</table>

Figure 6.28 – EDX of clusters of faceted particles on a specimen tested at 570°C

<table>
<thead>
<tr>
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<tr>
<td>Aluminum</td>
<td>72.91 wt%</td>
</tr>
<tr>
<td>Silicon</td>
<td>4.95 wt%</td>
</tr>
<tr>
<td>Manganese</td>
<td>4.22 wt%</td>
</tr>
<tr>
<td>Iron</td>
<td>14.86 wt%</td>
</tr>
</tbody>
</table>
No second phase particles were observed on the surface of the specimen tested at 582°C, and a representative micrograph of the fracture surface is shown in Figure 6.29. EDX was carried out on the overall fracture surface, and also on the interdendritic bridges that were found across the fracture surface. The overall fracture surface indicated that the composition of the alloy was 95.79% aluminum, 3.92% magnesium and 0.29wt% silicon, as shown in Figure 6.30. This is in close agreement with the EDX results from the overall fracture surface of the specimen tested at 501°C shown in Figure 6.23. EDX carried out on the dendrite arms visible on the fracture surface returned similar results. In contrast, the interdendritic bridges were examined and found to contain a range of alloying elements, as shown in Figures 6.31 and 6.32.

![Figure 6.31](image1.png)

![Figure 6.32](image2.png)

**Figure 6.29** – SEM micrograph showing bridging between dendrites on the fracture surface of a specimen tested at 582°C and a strain rate of $\sim 5.0 \times 10^3 \text{s}^{-1}$
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<tr>
<td>Iron</td>
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</table>

Figure 6.30 – EDS results of the fracture surface of a specimen tested at 582°C

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<td>Silicon</td>
<td>7.12 wt%</td>
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</table>

Figure 6.31 – EDS of interdendritic bridges on a specimen tested at 582°C
Table: Concentration

<table>
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<td>Aluminum</td>
<td>88.35 wt%</td>
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<td>Silicon</td>
<td>1.38 wt%</td>
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<td>Iron</td>
<td>5.16 wt%</td>
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</table>

Figure 6.32 – EDX of interdendritic bridges on a specimen tested at 582°C

With respect to the solidification characteristics of AA5182 available in the literature, indicated in Figure 2.1 and Equations 6.1 to 6.5, some of the second phase particles found on the fracture surfaces of the specimens can be identified from the results of the EDX investigation. The globular and acicular particles are likely to be Mg₂Si and Al₃Fe respectively, both of which form as part of a series of eutectic reactions during solidification. The clusters of faceted particles could not be identified conclusively. The whiskers on the fracture surface of specimens tested at lower temperatures could not be identified conclusively either, although it is possible that this is Al₃Mg₅, which forms as part of a complex eutectic reaction immediately prior to the end of solidification. This intermetallic would form at intergranular regions when the alloy has a fraction solid greater than 0.98, and might therefore be the first phase to melt during heating. The
softening and melting of the Al$_8$Mg$_3$ intermetallic in the intergranular region would lead to the localization of strain at grain boundaries and subsequent embrittlement of the alloy. As the test temperature increases from 540°C to 580°C, the fracture surface becomes smoother and the precipitates become smaller and less numerous, suggesting that they are progressively melting. At a test temperature of 582°C, the metallographic, fracture surface, and EDX investigations suggest that almost all second phase particles have melted, and that sufficient liquid is present to cover any remaining solid particles in the interdendritic regions.

6.6 Relationship between Temperature, Fraction Liquid and Tensile Properties

In order to develop a relationship between the temperature, fraction liquid, microstructure and tensile properties of the as-cast AA5182 alloy between 500°C and 580°C, literature values of the evolution of fraction solid with temperature during solidification have been used$^{[1]}$. Strength measurements have been plotted against the estimated fraction liquid in Figure 6.33, showing that the transition from significant tensile strength to very low strength occurs over a small range of fraction liquid close to 0.05 irrespective of strain rate. These results are in agreement with the tensile strengthening mechanism for aluminum alloys proposed by Dahle et al.$^{[5]}$, who have suggested that the transition in strength occurs as a result of the increase in bonded area between grains due to eutectic formation close to the end of solidification. The tensile strengthening mechanism proposed by Dahle et al. is shown in Figure 6.34, and a comparison between Figures 6.33, Figure 6.34 and EDX results presented above clearly indicates the similarities between the measured properties and the proposed mechanism.
Figure 6.33 – Variation in maximum tensile stress with fraction solid between 500°C and 580°C under applied strain rates between \(-2.0 \times 10^4\) s\(^{-1}\) and \(-7.5 \times 10^{-2}\) s\(^{-1}\)

Figure 6.34 - Comparison of the Development of Shear and Tensile Strength\(^5\)

1. Change in Bonded Area of Primary Dendrites
2. Change in Bonded Area Associated with Eutectic Formation
3. Final Eutectic Solidification
The relationship between fraction liquid and temperature used in this study has been derived from solidification experiments, while the experimental program carried out has been conducted by heating the as-cast alloy until the material has partially liquefied. Solidification and melting characteristics have been found to differ significantly for aluminum alloys\[6\]. Specifically the position of the solidus temperature may occur at a significantly higher temperature when melting solid, in comparison with the temperature at which liquid freezing ends. Furthermore, data comparing the evolution of fraction liquid with temperature during melting and the evolution of fraction solid during solidification is not available for AA5182, and it is unclear whether the melting sequence matches the reverse of the phase changes that occur during solidification as shown in Equations 6.1 to 6.5. Consequently, there may be some error in the fraction liquid estimated to be present in the specimens that were tension tested. Nevertheless, the data presented in this study agrees closely with the strengthening mechanism proposed by Dahle et al. in Figure 6.34.

The change in ductility with estimated fraction liquid has been plotted in Figure 6.35. Ductility decreases more rapidly than tensile strength with an increase in fraction liquid. Furthermore, the decrease in ductility as fraction solid increases is sensitive to the applied strain rate. Higher applied strain rates cause the alloy to become brittle at lower fractions liquid. Under an applied strain rate of $\sim 2.0 \times 10^{-4} \text{s}^{-1}$, loss of ductility occurred at a fraction liquid between 0.07 and 0.08, whereas no ductility was measured at a fraction liquid of 0.045 under an applied strain rate of $\sim 4.0 \times 10^{-2} \text{s}^{-1}$.
6.7 Summary

In summary, research has been carried out to relate changes in tensile properties of as-cast AA5182 close to the solidus temperature with the changing microstructure in the alloy. Mechanical property measurements indicate that the tensile strength decreases sharply to a very small level between 570°C and 577°C. Measurements also show that the alloy becomes increasingly less ductile with temperature. Minimal ductility is first measured between 560°C and 570°C and is dependent on strain rate.

A metallographic investigation was conducted, showing that ductile fracture and large amounts of plastic deformation occurred during testing at 501°C. As temperature increased, plastic deformation became less apparent and at 569°C, fracture occurred in an intergranular manner with very little deformation. Intergranular openings were observed.
in all specimens tested from 541°C upwards. As temperature increased the number and size of the intergranular openings increased. At 582°C, large numbers of intergranular and interdendritic voids had formed during testing.

The transition from ductile to intergranular fracture was confirmed by a SEM investigation of the fracture surfaces. Whiskers were also observed on specimens tested at 541°C and 569°C. These have been seen previously in superplasticity studies on wrought aluminum alloys, and suggest that large amounts of highly localized deformation occurred at grain boundaries during testing. At higher temperatures, the intergranular fracture surfaces were smooth and decorated with second phase particles, and at 582°C, the dendritic structure of the alloy was visible with a few, small second phase particles.

EDX analysis was carried out on the whiskers, second phase particles and interdendritic material observed between 541°C and 582°C. Several of the second phase particles were identifiable after cross-referencing the EDX results with the solidification sequence of AA5182. Although no second phase particles were observed on the specimen tested at 582°C, the interdendritic material had high levels of Mg, Fe and Si. Changes in the tensile properties of the alloy have been linked to the microstructure, and fraction liquid-temperature data has been used to relate the variation in tensile properties to the development of a liquid phase in the alloy as temperature increases. The tensile strength of the alloy decreases sharply at a critical fraction liquid of ~0.06, in agreement with the mechanism proposed by Dahle et al. to describe the development of strength during solidification. In contrast, loss of ductility occurs at smaller liquid fractions, and appears to be strain rate dependent due to the localization of strain at grain boundaries when the first liquid is formed.
References


Chapter VII – Conclusions

An experimental apparatus has been developed to enable measurement of the tensile properties of aluminum alloys at temperatures close to the solidus. Measurements such as these are required to obtain an improved understanding of the mechanical response of alloys close to the solidus, so that the formation of defects during casting processes can be predicted and controlled more accurately. The experimental method involves the connection of a modified Instron mechanical testing machine to a Gleeble thermo-mechanical simulator. A high-resolution digital camera and zoom lens was used to obtain a series of images of the tensile specimen during deformation, from which the instantaneous dimetral strain was measured.

The tensile properties of a DC cast AA5182 aluminum alloy in the as-cast condition have been measured between 500°C and 580°C, at strain rates from $-10^{-4}$ s$^{-1}$ to $-10^{-2}$ s$^{-1}$. Due to the presence of thermal gradients within the specimen during testing, the thermal profile in the specimen during testing has been modeled in ABAQUS™ to obtain an improved estimate of the deformation temperature and thereby obtain the correct mechanical property data.

The fracture surfaces and microstructure of the tested specimens have been examined in an attempt to gather sufficient information to develop a relationship between the variations in measured tensile properties with changes in the alloy microstructure and fracture behaviour. This has involved the use of optical microscopy, SEM and EDX analysis. Literature values of the change in fraction liquid with temperature have been used to relate the tensile properties and microstructural changes to the presence of liquid
in the alloy. A number of conclusions regarding the variation in tensile properties, fracture behaviour and microstructure of AA5182 close to the solidus can be drawn as a result of this study.

- The alloy exhibits significant tensile strength from 500°C to ~570°C, which corresponds to a fraction liquid of ~0.05. The alloy is strongly sensitive to strain rate across this temperature range.

- At a temperature of ~570°C and fraction liquid of ~0.05, a sharp decrease in strength to ~1-2MPa was observed at each strain rate. The microstructural and fracture surface examinations suggest that the loss in tensile strength is associated with the presence of a critical fraction liquid in the alloy, which causes complete loss of tensile coherency.

- The ductility of the alloy decreased steadily with increasing temperature, and a transition from ductile to intergranular fracture was observed between the reported solidus temperature of 536°C and 560°C-570°C, at which point ductility became negligible. Intergranular openings containing fine whiskers suggest that grain boundary sliding occurs within this temperature range as a result of the presence of small amounts of intergranular liquid. At higher temperatures, smooth intergranular openings and fracture surfaces predominated, indicative of higher liquid contents and less intergranular bridging.

- EDX analysis has enabled the identification of some of the intermetallic phases present at grain boundaries in the as-cast alloy due to eutectic reactions taking place in the final stages of solidification. It has been shown that these intermetallic phases melt within the range of test temperatures in this study.
• The structure-property relationships detailed in the points above are in agreement with the mechanism proposed by Dahle et al. that attributes the development of tensile strength in partially solidified alloys to an increase in intergranular bonding during the solidification of eutectic phases.

7.1 Scope for Future Work

Further work can be carried out to optimize the experimental method. This may include modifying the design of the stainless steel grips and cooling water apparatus to improve control of heat transfer and reduce thermal gradients in the tensile specimen. Additionally, the data acquisition system can be improved. This may be achieved by using a more powerful computer to acquire data at faster rates, and a higher resolution camera could be used to reduce the minimum measurable strain increment.

Measurements of other alloys and as-cast structures can be made using the experimental apparatus that has been developed as part of this project. This would enable the determination of the influence of alloy chemistry, solidification characteristics and microstructure on the properties close to the solidus and development of strength in the partially solidified state.

The thermal model can be also developed and applied to transient analyses in order to include heating and variations in temperature during testing as the specimen deforms. Furthermore, the model can be modified into a coupled thermal-mechanical analysis, to model the mechanical response of the alloy and develop a hot tearing criterion based on mechanical property measurements for use in models of casting processes.
APPENDIX A

A1 - Method Used to Convert Load Displacement Data to Stress-Strain Data

It is desirable to present the load and displacement data acquired through tensile testing in terms of true stress and true strain. To this end, load-displacement data recorded was manipulated mathematically to convert them into stress-strain data.

A1.1 – Determination of True Strain

True strain has been calculated based on the variation in specimen diameter during deformation. The dimetral strain equation is given below.

\[ \varepsilon = -2 \ln \left( \frac{D}{D_0} \right) \]  

(A1.1)

where:

\( \varepsilon \) is the strain

\( D_0 \) is the original diameter of the specimen

\( D \) is the instantaneous diameter.

The original diameter of the specimens was measured at room temperature. The diameter of the specimens at the start of deformation was then calculated taking into account the variation due to thermal expansion when heated from room temperature to the deformation temperature. These dimensional changes were determined from the following relation:

\[ D_0 = D_{RT} + \partial D_{thermal} \]  

(A1.2)

where:
$D_0$ is the diameter at the start of deformation

$D_{RT}$ is the measured diameter at the room temperature

$\delta D_{thermal}$ is the change in diameter due to thermal expansion between room temperature and the deformation temperature

If the volume of the specimen is assumed to remain constant, then:

$$V_0 = V$$  \hspace{1cm} (A1.3)

where:

$V_0$ and $V$ are the volume of the specimen prior to, and after, deformation

Therefore;

$$A_0 L_0 = A L$$  \hspace{1cm} (A1.4)

where the cross-sectional area is determined from the following formulas:

$$A = \pi \cdot r^2 = \pi \cdot \frac{D^2}{4}$$  \hspace{1cm} (A1.5)

and

$$A = \pi \cdot \frac{D^2}{4} = \frac{A_0 L_0}{L} = \frac{V_0}{L}$$  \hspace{1cm} (A1.6)

where:

$L_0$ is the length at the deformation temperature and $A$ is the instantaneous cross-sectional area.

Therefore, from our assumption of constant volume and uniform deformation and rearranging Equation A1.4, we get:

$$\frac{L}{L_0} = \frac{A_0}{A}$$  \hspace{1cm} (A1.7)
\[
\frac{A_0}{A} = \left( \frac{D_0}{D} \right)^2
\]  \hspace{1cm} \text{(A1.8)}

Now from Equation A1.8 we can determine the strains as follows:

\[
\varepsilon = 2 \ln \left( \frac{D}{D_0} \right)
\]  \hspace{1cm} \text{(A1.9)}

This equation gives a negative value of strain when the specimen is tested in tension and therefore we consider the absolute value.

**A1.2 – Determination of True Stress**

The engineering stress-strain curve does not represent a true indication of the deformation characteristics of a material because it is based on the original cross-sectional area of the specimen, and this changes continuously during deformation. True stress is calculated using the instantaneous cross-sectional area as follows:

\[
\sigma = \frac{F}{A}
\]  \hspace{1cm} \text{(A1.10)}

\[
\sigma = 9.81 \frac{\text{LoadCell}}{A}
\]  \hspace{1cm} \text{(A1.11)}

where:

A is the instantaneous cross-sectional area calculated in Equation A1.5 above, and 9.81ms\(^{-1}\) is the acceleration due to gravity, used to convert the applied force into unit load with units of MPa.

All stress and strain data presented have been based on the true stress and true strain equations given in Equations A1.9 and A1.11.
Strain measurement was carried out using a Sony DFW-SX900 digital camera with a resolution of 1280x1024 pixels to collect high-resolution images of the specimen during deformation and thereby obtain measurements of the instantaneous diameter, $D_0$, for use in Equation A1.9. The increment in strain that this system can measure is directly related to the resolution of the camera and the number of pixels covering the diameter of the specimen.

**A2.1 – Calculation of the Minimum Measurable Strain Increment**

The minimum strain increment that this system can measure can be calculated by considering the ‘best case scenario’, where the diameter of the specimen covers almost the total width of the viewfinder. In this case, one pixel at the extreme left and right of the viewfinder would be required for edge detection, therefore the maximum number of pixels that can be used to measure the initial diameter of the specimen prior to deformation would be 1278. Therefore, the minimum strain increment can be calculated by considering a reduction in specimen diameter sufficient to reduce the pixel-count by one. Therefore, using Equation A1.9:

$$\varepsilon_{\text{min,inc.}} = 2 \ln \left( \frac{1277}{1278} \right)$$

(A2.1)

$$\varepsilon_{\text{min,inc.}} = 0.001566$$

(A2.2)

Note that the value of strain given in Equation A2.2 is an absolute value.
A more typical scenario would be the case where the specimen covers 75% of the width of the viewfinder. In this case, the initial pixel-count prior to deformation would be 960. Thus, a typical strain rate increment would be:

$$\varepsilon_{typ.} = 2 \ln \left( \frac{959}{960} \right)$$

(A2.3)

$$\varepsilon_{typ.} = 0.002084$$

(A2.4)
B1 – Summary of Test Data

Table B1.1 – Thermocouple (TC) Measurements During Tensile Testing

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Test Temperature (°C)</th>
<th>TC 1 (+44mm)</th>
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Table B1.2 - Summary of Tensile Test Results

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