EFFECT OF HOMOGENIZATION ON HIGH TEMPERATURE DEFORMATION
BEHAVIOUR OF AA3XXX ALUMINUM ALLOYS

by

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Abstract

The effect of homogenization practice and alloy Mn content on the dispersoid density and high temperature constitutive behaviour of AA3xxx aluminum alloys has been studied. Experimental work was conducted on three as-cast alloys with different Mn compositions: AA3102 (0.26 wt% Mn), an intermediate Mn alloy (0.75 wt% Mn), and AA3003 (1.27 wt% Mn). Homogenization treatments were conducted at soak temperatures from 500 to 630°C and times of 1 to 24 hours.

The evolution of microstructure during homogenization (at 600°C over a soak time of 24 hours) was characterized using electrical conductivity measurements and optical microscopy. Electrical conductivity measurements were used to estimate the variation of Mn in solid solution during homogenization. Qualitative observations of constituent particle morphology and dispersoid precipitation/dissolution were made.

Compression tests were conducted using a Gleeble 3500 thermomechanical simulator at strain rates (0.1 to 10s⁻¹) and deformation temperatures (300 to 600°C) experienced during the industrial extrusion of AA3xxx alloys. Yield stress, flow stress, and work hardening results were obtained from the compression tests and a physically-based constitutive model was employed to describe the results. The model was used to quantify the effect of homogenization practice and alloy Mn content on constitutive behaviour. Homogenized microstructure observations were combined with model results to determine the effect of dispersoid density on constitutive behaviour.

Extrusion trials were performed on Rio Tinto Alcan’s state-of-the-art research extrusion press located at the Alcan Research and Development Center in Jonquiere, Quebec. AA3102 and AA3003 alloys were extruded in as-cast and homogenized
conditions at temperatures of 400 and 500°C. Extrusion pressure results were used to develop a relationship between extrusion pressure and flow stress.
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<td>$\mu\Omega\cdot\text{cm}$</td>
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Chapter I - Introduction

The aluminum industry is an important part of Canada’s economy with Canada being the second largest exporter of primary aluminum in 2005.\(^1\) In 2007, Canada’s largest aluminum company, Alcan, was taken over by Rio Tinto and Rio Tinto Alcan was formed creating one of the world’s largest producers of bauxite, alumina and aluminum.\(^2\) One key market to the aluminum industry is the value-added products group which supplies engineered products such as extrusion billets to customers in the manufacturing sector. Of the value-added products, 3xxx series aluminum extrusion billets are seen as an area for future growth.\(^3\)

AA3xxx alloys combine moderate strength, formability, brazeability, corrosion resistance, and good thermal conductivity and are widely used for sheet and extruded products.\(^4,5\) In commercial applications, AA3xxx alloys are predominantly used for thin walled components such as beer cans, building plates and heat transfer tubes.\(^6\) AA3102 and AA3003 are the most important of the extruded alloys with heat exchanger tubing being their main application.\(^4\) While AA3102 is the more extrudable of the two alloys, being used extensively in mini-microport and thin wall round tubing, AA3003 provides better strength.\(^4\)

The manufacturing of AA3xxx alloys used for extrusions typically includes: Direct Chill (DC) casting, followed by homogenization, extrusion, cold deformation and brazing as shown in Figure 1.1. At each stage of the processing route, the microstructure is modified and hence the thermomechanical history experienced by AA3xxx during homogenization, extrusion, cold deformation, and brazing impacts the microstructure and
mechanical properties of the end product. In this study two of the processing steps, homogenization and extrusion, will be examined.

1.1 Homogenization

As-cast billets are homogenized prior to extrusion to decrease extrusion pressures and improve the surface finish of the final product. By homogenizing AA3003, the extrudability is increased by at least 50% and a finer as-extruded grain size is produced. Figure 1.2 shows a schematic of the temperature profile experienced by an AA3xxx alloy during homogenization and extrusion.

Figure 1.1- Typical manufacturing process of AA3xxx extrusion alloys used to make heat exchangers.
During the homogenization heat treatment, billets are heated to a soak temperature (or holding temperature). Typical industrial heating rates range from 50 to 250°C/hr. The billet is held at the soak temperature for several hours and then cooled to room temperature. Prior to extrusion the billet is rapidly heated to the extrusion temperature typically using induction heating. The billet is extruded and is again cooled to room temperature.

![Figure 1.2- Schematic of typical thermal history experienced during homogenization/extrusion of AA3xxx alloys.](image)

**1.2 Extrusion**

Extrusion is a forming process in which pressure is applied to the back of the billet causing it to deform as it exits through a die. Extrusion typically occurs at temperatures between 400 and 600°C. Figure 1.3 shows the internal components of an extrusion press. A dummy block is attached to the ram sealing the billet in the container.
and preventing metal from traveling backward. The container liner keeps the billet in place during extrusion. As pressure is applied, the billet is forced through the die and emerges with the desired profile. A die backer holds the die in place. After exiting the die, the extruded product travels down a run out table and is cooled.

![Diagram of extrusion process](image)

**Figure 1.3- Internal components of a typical direct extrusion press.**

1.3 Optimizing the Manufacturing Process

The thermomechanical history experienced by AA3xxx alloys during homogenization and extrusion has a large impact on the final mechanical properties of the product. Currently, little information on the processing of AA3xxx can be found in the literature and knowledge is limited. In order to optimize the manufacturing process the influence that homogenization has on extrusion, for various alloy compositions, must be determined.
In 2006 the Materials Engineering department at the University of British Columbia began a project with the primary objective being the development of AA3xxx knowledge based models to aid industry in improving the properties and quality of these alloys. The current work is the initial part of this project and looks at the homogenization and extrusion processing steps.

To meet the proposed objective, this study will focus on developing a qualitative model to predict the microstructure at each stage of the extrusion process. The influence of the alloy content (Mn) and homogenization heat treatment on the microstructure evolution and high temperature constitutive behaviour of AA3xxx alloys was quantified using experimental and modelling techniques.
Chapter II - Literature Review

During complex manufacturing routes such as those used to manufacture heat exchanger tubes from AA3xxx series alloys, it is increasingly important to develop quantitative knowledge of the microstructure evolution at each stage of the process as well as the inter-dependencies between the processes. For example, a change in the homogenization parameters for an AA3xxx alloy (with the same chemistry) alters the microstructure which may in turn influence the flow stress and hence the extrusion pressure. To quantitatively determine the constitutive behaviour of AA3xxx alloys the influence of deformation temperature, strain rate and strain as well as starting microstructure must be quantified.

This chapter provides background information and details research relevant to the current research study. First, the as-cast structure is discussed and the microstructure evolution relevant for aluminum alloy AA3xxx during homogenization is reviewed. The influence of the microstructure on the constitutive behaviour of AA3xxx aluminum alloys is then discussed and flow stress models which have been developed to quantitatively predict the high temperature constitutive behaviour of the material are evaluated. The suitability of each model to this study is discussed.

2.1 As-Cast Microstructure

AA3xxx alloys have three main alloying elements Mn, Fe and Si. After solidification, most of the Mn is in solid solution while the majority of Fe and Si have formed intermetallic (constituent) particles during solidification. A micrograph of a
The as-cast microstructure is cellular to dendritic with the rod/plate-like constituent particles forming during solidification mainly as interdendritic eutectic networks.\textsuperscript{6,10,11} Constituent particles consist of $\text{Al}_6(\text{Mn,Fe})$ and $\alpha$-$\text{Al}(\text{Mn,Fe})\text{Si}$ phases with the former making up the majority.\textsuperscript{6,10} The formability of AA3xxx alloys is influenced by the constituent particles with the $\alpha$-$\text{Al}(\text{Mn,Fe})\text{Si}$ phase being the harder of the two phases.\textsuperscript{10}

The addition of alloying elements in AA3xxx alloys has a strong influence on precipitation both during solidification and homogenization.\textsuperscript{12} The solubility of Mn in solid solution is decreased with the addition of Fe and Si and the formation of particles is accelerated.\textsuperscript{9} Segregation occurs around the constituent particles with Fe and Si segregating toward particles and Mn segregating away from the particles.\textsuperscript{6}
Eddy current inspection is a non-destructive testing method frequently used to measure material conductivity; using this method, an alternating current (or exciting current) is passed through an electric coil that has been placed on the sample to be measured.\(^{13}\) The alternating current causes eddy currents (circulating flow of electrons) to flow in the sample due to electromagnetic induction. The eddy currents themselves induce magnetic fields opposing changes in the coil’s magnetic field; the greater the conductivity of a sample, the greater the magnitude of the Eddy currents and the induced magnetic fields. The magnetic fields produced by eddy currents can then be sensed either through the effects of the field on the electric coil or by using an independent sensor.

Electrical conductivity or inversely resistivity, measurements are a method commonly used to determine the average amount of Mn in solid solution (or conversely the amount of Mn that has precipitated). Matthiesen’s rule\(^{14}\) sums impurity contributions to the resistivity and is given as:

\[
\rho = \rho_T + \rho_D + \rho_V + \rho_{SS} \tag{2.1}
\]

Where, \(\rho, \rho_T, \rho_D, \rho_V\) and \(\rho_{SS}\) are the measured, thermal, dislocation, vacancy, and solid solution components of the resistivity, respectively. In the current study, resistivity measurements were made at room temperature on undeformed (with exception) samples that were quenched. Under these conditions the \(\rho_T, \rho_D,\) and \(\rho_V\) resistivity components are negligible, giving the equation:

\[
\rho \approx \rho_{SS} \tag{2.2}
\]
\[ \rho = \sum_{i=1}^{n} \lambda_i C_i \]  

(2.3)

Where, \( \lambda_i \) is the average increase in resistivity of an alloying element in/out of solid solution, \( C_i \) is the concentration of the alloying element, and \( n \) is the number of alloying additions. The maximum solubility and average resistivity increase for the elements of interest in this study are given in Table 2.1. The base resistivity for high-purity Al at 20°C is 2.65 \( \mu \Omega \cdot \text{cm} \).  

Table 2.1- Maximum solubility and average increase in resistivity of elements in aluminum.  

<table>
<thead>
<tr>
<th>Element</th>
<th>Maximum solubility in Al (%)</th>
<th>Average increase in resistivity per wt% (( \mu \Omega \cdot \text{cm} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>In solution</td>
</tr>
<tr>
<td>Fe</td>
<td>0.052</td>
<td>2.56</td>
</tr>
<tr>
<td>Mn</td>
<td>1.82</td>
<td>2.94</td>
</tr>
<tr>
<td>Si</td>
<td>1.65</td>
<td>1.02</td>
</tr>
</tbody>
</table>

In the work of Dons and coworkers, \(^6,7^\) resistivity was calculated using the following equation:

\[ \rho = 0.0267 + 0.033C_{Mn} + 0.032C_{Fe} + 0.0067C_{Si} \]  

(2.4)

Where \( \rho \) is the measured resistivity in \( \mu \Omega \cdot \text{m} \) and \( C \) is the concentration of the alloying element in solid solution (wt%). Dons and coworkers used Equation 2.4 to estimate the amount of Mn in solid solution for an AA3003 alloy assuming that a
maximum of 0.05% of the Fe and 0.5% of the Si were in solid solution. They suggested that using this method could result in an over estimation of 0.06 wt% Mn in solid solution. Equation 2.4 gives estimated Mn in solid solution values that are significantly lower than those obtained using Equation 2.3 and are in better agreement with experimental measurements.

In AA3xxx aluminum alloys, only very small concentrations of Fe and Si are in solid solution and it is reported that their concentrations vary little during heat treatment. Mn is the main contributor to the electrical conductivity (from here on referred to as conductivity) in these alloys and conductivity measurements are approximately inversely proportional to the average amount of Mn in solid solution. Thus, precipitation of Mn causes an increase in conductivity and dissolution of Mn causes a decrease in conductivity.

Although conductivity values are a convenient way of examining changes in the amount of Mn in solid solution, the measurements should be used as a relative comparison of values. Lok et al. observed that conductivity measurements resulted in significantly larger Mn solute values than those expected from thermodynamic calculations for AA3103; this discrepancy did not exist between solute measurements made using another method, thermoelectric power (TEP), and thermodynamic calculations. The most significant discrepancy was seen in the as-cast state where conductivity measurements of AA3103 over estimated Mn solute levels by almost 0.2 wt%. About 40 to 50% of the excess could be accounted for by the networks of large constituent particles. The cause of the remaining excess was unclear as vacancies, dislocations and grain-boundaries were considered to be unlikely possibilities. The study
found that corrections could be made to conductivity values if exact amounts of Mn in solid solution were needed for comparison with thermodynamic calculations. Corrections were made using coefficients determined from two binary Al-Mn curves: the expected linear dependence of electrical resistivity with increasing solute level and the Nordheim-Gorter plot (from TEP measurements).

### 2.2 Homogenization

Homogenization heat treatments of the as-cast material reduce the concentration of Mn in solid solution and are used to modify the size, number density and distribution of particles.\(^9\) During homogenization primary (or constituent) particles undergo a change in morphology and chemistry while secondary particles (dispersoids) precipitate initially and then subsequently dissolve. The homogenized microstructure can have a large effect on the recrystallization behaviour, texture, grain size and mechanical properties of the final product.\(^9,17\) Large particles (>1 or 2µm) promote recrystallization while high concentrations of finely dispersed particles that precipitate after solidification called dispersoids (<0.1µm) inhibit recrystallization.\(^6,10\) For example, it has been found that dispersoids present prior to extrusion can have a significant effect on recrystallization behaviour after hot deformation.\(^17\) In a study by Tangen et al.,\(^19\) a material with a high dispersoid density recrystallized into relatively inhomogeneous and coarse grain structures after extrusion with the effect being attributed to Zener drag.

Models have been developed to predict the evolution of microstructure during homogenization. One of the first microstructure models applied to AA3xxx was ALSTRUC, a computer model initially developed in 1993 by Tromborg et al.\(^20\) The
model first calculated the alloy solidification path and as-cast structure and then the microstructure changes during homogenization. Extensive diffusion calculations based on Fick’s laws and on empirical nucleation criteria were employed. The output of the model included: constituent particle and dispersoid size distributions, concentrations of alloying elements in solid solution, and the particle diameters/volume fractions of each phase as a function of time. The preliminary ALSTRUC model was capable of predicting alloying elements, within experimental accuracy, in solid solution in the as-cast state and during homogenization.

In 1996, Suni et al.\textsuperscript{21} developed a physically based microstructure model to predict the evolution of dispersoids during the preheating stage of the homogenization of 3xxx aluminum alloys. The model used nucleation, growth, and coarsening input to calculate dispersoid evolution. The model was very simplistic, and ignored evolution of constituent particles during homogenization and thus the linkage between constituent particle and dispersoid evolution. The model was compared with test data and was found to provide mixed results according to the RMS error (in Mn wt%), and had limited use. The model was modified in 1998 by Suni and Shuey,\textsuperscript{22} coupling the evolution equations for dispersoid and constituent particles. The model did not attempt to model near boundary depletion of across cell diffusing species and as-cast microsegregation.

In 2001, details of an updated ALSTRUC version were published by Dons.\textsuperscript{23} The updated microstructure model is comprehensive and detail oriented, providing semi-quantitative estimates of changes in the homogenized microstructure during the later part of the heating stage and during holding. The ALSTRUC model has subroutines for AA3xxx, AA5xxx, AA6xxx and AlSi foundry alloys. Model output parameters have a
degree of fit with experimental data that is at best of the same order of magnitude as the experimental error. For AA3003, the program has been verified against the types of Fe-bearing particles precipitated and the density of dispersoids after soaking. The ALSTRUC model has been used in the VIR project\textsuperscript{24} through process model. Although the ALSTRUC model has proven to be useful, not all aspects of the model have been validated and discrepancies between the model and experimental measurements exist. Of the models developed to predict the evolution of microstructure for AA3xxx alloys during heat treatment, a model has never been fully validated (over the entire evolution process) against detailed experimental measurements.

\section*{2.2.1 Heating}

Typical industrial homogenization heating rates range from 50 to 250°C/hr.\textsuperscript{7} A time-temperature profile with corresponding measured conductivity values for an AA3003 alloy is shown in Figure 2.2.

During this study the microstructure was examined at various temperatures and times to better understand the microstructure evolution during both heating and holding. As the alloy is heated to 300°C the constituent particles experience almost no change in morphology or size.\textsuperscript{10} At 400°C the eutectic networks of constituent particles begin to break up. At approximately 400°C and above, some of the Al\textsubscript{6}(Mn,Fe) constituent particles transform to the $\alpha$-Al(Mn,Fe)Si phase and the composition of the particles changes.\textsuperscript{10} Concurrently, the supersaturated solid solution starts to decompose by precipitating Mn-rich dispersoids with a composition of Al\textsubscript{6}(Mn,Fe) or $\alpha$-Al(Mn,Fe)Si.
Initially the dispersoids contain very little Fe as only a small amount is available in the matrix. As temperature increases, long range diffusion becomes feasible and Fe is able to diffuse from the constituent particles to the dispersoids.\textsuperscript{9} As shown in Figure 2.2, dispersoid nucleation begins around 300°C; this is reflected in the large conductivity increase (shown in Figure 2.2).\textsuperscript{9} The rate of dispersoid nucleation is dependant on the composition of the alloy and is relatively independent of the heating rate within the industrial ranges typically used.\textsuperscript{7} Initially during heating, due to the low temperature, dispersoids can only nucleate in areas that are highly enriched with Mn making nucleation in Mn-depleted areas (near constituent particles and on grain boundaries) difficult.\textsuperscript{9} As the temperature increases dispersoids continue to nucleate and precipitation slowly extends to the Mn-depleted areas.\textsuperscript{9} Nucleation of dispersoids can occur on discontinuities such as dislocations and subgrain boundaries.\textsuperscript{9,11,12}
After nucleation the dispersoids begin to grow and the rate of Mn diffusion is rate controlling. The dispersoid number density increases with temperature and reaches a maximum at about 400 to 450°C. Even though the maximum number density has been reached, growth of the particles is still taking place causing the particle volume fraction to increase. The conductivity reaches a maximum at the same temperature as the maximum volume fraction of dispersoids at approximately 530°C. As the homogenization temperature continues to increase to the soak temperature, the solid solubility of Mn increases with temperature. At approximately 530°C, just after the maximum dispersoid volume fraction is reached, the temperature is sufficiently high enough to cause the dispersoids to begin to dissolve back into the matrix resulting in a decrease in conductivity. As the temperature increases beyond 530°C up to the desired soak temperature, coarsening and dissolution of the dispersoids continues and the conductivity decreases further. The morphology of dispersoids during heating is shown in Figure 2.3.

Re-dissolution of the dispersoids produces a final number density (upon the completion of heating) that is relatively independent of heating rate. The local solid solubility of Mn and Fe at the soak temperature and the amount of Si available determine the number density of dispersoids as the soak begins. Meanwhile, spheroidization of the constituent particles occurs as the homogenization temperature increases and by 560°C most of the constituent particles have broken up into smaller particles. Above 560°C the number density of the constituent particles decreases due to coarsening. During heating, growth of the constituent particles is negligible (due to the difficulty of long range diffusion,
particularly at lower temperatures) causing no significant change in area fraction; this indicates that the variation of Mn in solid solution is almost completely due to the precipitation/dissolution of dispersoids.$^{10}$

Figure 2.3- TEM images of AA3003 dispersoid morphology during heating. (a) 350°C, (b) 400°C, (c) 500°C, and (d) 580°C.$^9$
2.2.1.1 Spatial Distribution of Dispersoids

The segregation of Mn in the microstructure and the diffusion rate of Mn at a given temperature dictate the spatial distribution of dispersoids. Precipitate free zones (PFZ) form in Mn-depleted areas. A strong depletion of Mn in solid solution is found around the constituent particles. At low temperatures dispersoid precipitation around the constituent particles is difficult but as the temperature increases and more Mn precipitates from the matrix the area of the PFZ decreases. When the temperature is high enough that the dispersoids begin to dissolve, dissolution begins in areas that are the most Mn-depleted (i.e., near the constituent particles) and the PFZ area increases. Close to the soak temperature the diffusion rate of Mn has significantly increased permitting diffusion of Mn from the Mn-rich areas to the Fe-rich constituent particles.

2.2.2 Soaking

Initially during soaking the conductivity increases quickly with holding time as shown in Figure 2.4. The rate of the increase in conductivity then begins to slow down as the soak progresses and with long soak times the concentration of Mn in solid solution approaches the overall equilibrium concentration.

During soaking the constituent particles are spheroidized and the ratio of $\alpha$-Al(Mn,Fe)Si to $\alpha_6$(Mn,Fe) constituent particles increases with homogenization time. Coarsening and growth of the constituent particles occurs over the soak period (occurring slowly over the span of the soak period provided the soak temperature is high enough to permit long range diffusion) and is reflected in the size evolution of the particles; the
particles experience a decrease in number density and an increase in area during soaking.\textsuperscript{26}

\textbf{Figure 2.4- AA3003 conductivity evolution during homogenization at long soak times.}\textsuperscript{10}

The size evolution of the constituent particles is not only influenced by coarsening but also by growth with the main driving force for growth being the difference in composition between the constituent particles and the dispersoids.\textsuperscript{7} As the dispersoids dissolve and Mn is precipitated from the supersaturated matrix the solute is able to diffuse to the constituent particles due to the high soak temperature which makes long range diffusion possible. The evolution of the area fraction and number density of constituent particles during a 600°C soak is shown in Figure 2.5.
Initially during soaking the area fraction of constituent particles increases quickly, due to the dissolution of the Mn-rich dispersoids and to a lesser degree due to precipitation of Mn from the supersaturated matrix. The increase in area fraction then begins to slow as any remaining dispersoids have dissolved and precipitation of Mn from the matrix has decreased (due to a decrease in super saturation of Mn in the matrix). The growth of the constituent particles at the expense of the Mn-rich dispersoids and the Mn in solid solution causes an increase in the Mn/(Mn+Fe) ratio of the constituent particles. The increase in Mn/(Mn+Fe) ratio with time follows a curve of similar shape to the area fraction curve.

Although the final number density of dispersoids precipitated during heating is independent of heating rate it is dependent on the soak temperature. A higher soak temperature gives fewer dispersoids at the onset of soaking. The evolution of dispersoid size and number density during soaking are shown in Figure 2.6; where $D_{\text{circle}}$ is the
average equivalent diameter, $f_{\text{min}}$ and $f_{\text{max}}$ (which describe the aspect ratio of the dispersoids) are the minimum and maximum ferret diameters, respectively.

Initially during soaking a large number of fine dispersoids are present. At the beginning of the soak period the size of the dispersoids increases with soak time but as the soak progresses the size begins to decrease. Coarsening occurs not only between the dispersoids but also between the constituent particles and the dispersoids causing dissolution of the dispersoids especially near grain boundaries (an area depleted in Mn).

The combined effect of dissolution and coarsening causes a decrease in the number density of dispersoids with soak time and the dissolution process causes the volume fraction to decrease with soak time. The dissolution of dispersoids continues and after a soak time of 24 hours, at a temperature of 600°C, no dispersoids are found in the matrix.

![Figure 2.6](image.png)

Figure 2.6- Size and number density of dispersoids during soaking at 600°C for AA3003.9
2.3 Constitutive Behaviour of AA3xxx During the Extrusion Process

During the extrusion process the homogenized billet is rapidly re-heated to the deformation temperature and then extruded in the press (typically at 400 to 520°C and strain rates from 0.1 to 2/s). The grain structure becomes deformed and then may recrystallize after deformation. Although the extrusion process occurs in a matter of minutes the extrusion conditions may cause dynamic precipitation/dissolution of dispersoids. In a study by Parson and Ramanan, as-cast AA3003 was extruded at temperatures 500 and 530°C and in both cases it was found that Mn solid solution levels decreased due to the precipitation of Mn dispersoids during hot deformation. A commercially homogenized AA3003 alloy (extruded under the same conditions) was found to have relatively constant Mn solid solution levels from the homogenized billet through the extrusion process.

The amount of Mn in an alloy is a variable that can have an affect on flow stress. Solid solution hardening (reviewed by Haasen) occurs when foreign atoms are introduced into a crystal lattice causing interactions between the solute atoms and mobile dislocations. The interaction increases the yield stress and reduces the dynamic recovery rate of dislocations resulting in a higher dislocation density and higher work hardening rate. In work done by Ryen et al. the strength of various aluminum alloys was found to have a nearly linear dependency on the concentration of Mn in solid solution for tensile tests conducted at room temp.

In describing the constitutive behaviour of aluminum alloys, strain hardening effects as well as strain rate sensitivity and temperature effects must be considered. Strain hardening occurs during most working and forming operations performed at a low
homologous temperature and is a method commonly used to increase strength. As the material is deformed dislocations are generated and at the same time annihilation and dynamic recovery occurs, ie. when the net change in dislocation density is positive strengthening of the material occurs. Dislocation tangles, cells and subgrain walls can form decreasing the mean free slip distance.\textsuperscript{31} As the deformation temperature is increased strain hardening effects decrease until a temperature is reached above which no effective strain hardening occurs.\textsuperscript{32} For extrusion conditions strain hardening will be negligible except possibly at the lower range of temperatures but there will be a strong strain rate sensitivity as well as sensitivity to deformation temperature.

2.4 Constitutive Modelling of Material Behaviour During the Extrusion Process

A key parameter for modelling extrusion is a constitutive behaviour model and how it is influenced by macroscopic variables like temperature, strain rate and strain but also by microscopic variables such as dispersoid content and Mn in solid solution. A flow stress model that accurately describes the extrusion deformation conditions (high temperatures and a large range of strain rates and large levels of strain) is needed to predict the stress-strain relationship for each homogenization condition.

Modelling techniques applicable to the extrusion process range from simple power law equations, to rate based Zener-Hollomon equations, creep based equations, physically based equations (that incorporate knowledge of the microstructure), and to internal state variable equations. The following sections detail constitutive equations commonly used in modelling the deformation of aluminum alloys during extrusion.
2.4.1 Power Law Equation

Work hardening curves when plotted as a function of true stress and true strain for annealed, recrystallized aluminum alloys are approximately parabolic and can typically be characterized using a simple power law equation; however this equation is only applicable to lower temperatures (at or close to room temperature) and is less accurate than other models.

At elevated temperatures both deformation temperature and strain rate affect the flow stress characteristics of a material. The combined strain and strain rate effects on flow stress (at a constant temperature) can be described using Equation 2.5.\(^\text{31}\)

\[
\sigma = K\varepsilon^{n_H} \dot{\varepsilon}^{m_H}
\]  
(2.5)

Where \(\sigma\) is the true stress, \(K\) is a strength constant (dependant on strain, temperature and material), \(\varepsilon\) is the true strain, \(n_H\) is the work hardening exponent, \(\dot{\varepsilon}\) is the strain rate and \(m_H\) is the strain rate sensitivity of the flow stress. The strain rate sensitivity is dependent on temperature with higher temperatures giving higher values of strain rate sensitivity; this is attributed to the increased rate of thermally activated processes.\(^\text{31}\)

Constitutive behaviour can be characterized at both room and higher temperatures using this simple empirical power law equation. This equation takes into account strain hardening and strain rate sensitivity but breaks down at high temperatures. In addition, the model does not take into account microstructure.
2.4.2 Zener-Hollomon Equation

The Zener and Hollomon equation treats plastic strain as a rate process using the Arrhenius rate law given in Equation 2.6. Zener and Hollomon proposed that the strain rate, $\dot{\varepsilon}$ (s$^{-1}$), could be expressed as given in Equation 2.7.

$$rate \propto \exp\left(-\frac{Q}{RT}\right) \quad (2.6)$$

$$\dot{\varepsilon} = A_i \exp\left(-\frac{Q}{RT}\right) \quad (2.7)$$

Where, $A_i$ is the pre-exponential constant that is dependent on both stress and strain, $Q$ is the activation energy, $R$ is the gas constant, and $T$ is the deformation temperature. $A_i$ is dependent on both stress and strain. At a constant strain, $A_i$ is only a function of stress, $\sigma$, and Equation 2.7 can be expressed as shown in Equation 2.8 or simplified to Equation 2.9 where $Z$ is the Zener-Hollomon parameter and is given by Equation 2.10.

$$A_i(\sigma) = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \quad (2.8)$$

$$\sigma = f(Z) \quad (2.9)$$

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \quad (2.10)$$
Using this model and plotting strain rate (on a logarithmic scale) versus $1/T$ should give a straight line with a slope of $-Q/R$. High temperature deformation can be characterized using the Zener Hollomon equation. This empirical equation takes into account strain rate and temperature effects however the equation does not take microstructure or work hardening into account.

### 2.4.3 Sellars-Tegart Equation

The Sellars-Tegart\textsuperscript{33} or Garofalo’s equation can be used at high deformation temperatures to describe the steady state deformation behaviour of materials and is given in Equation 2.11.

\[
\dot{\varepsilon} = A_i \left[ \sinh \left( \frac{\sigma_{\text{flow}}}{\sigma_0} \right) \right]^{n_H} \exp \left( -\frac{Q}{RT} \right) \tag{2.11}
\]

Where, $A_i$, $\sigma_0$ and $n_H$ are material constants, $\sigma_{\text{flow}}$ is the steady state flow stress, $Q$ is the activation energy, $R$ is the gas constant, and $T$ is the deformation temperature. Parameters in this equation are independent of temperature. Temperature is expressed explicitly making the equation advantageous when compared with other models. Equation 2.11 can be rewritten as:

\[
\dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = A_i \left[ \sinh \left( \frac{\sigma_s}{\sigma_0} \right) \right]^{n_H} \tag{2.12}
\]
Where, $Z$ is the Zener-Hollomon parameter described in Equation 2.10. The parameters used in this model can be calculated by plotting $Z$ versus $\sigma_s$.

Sheppard and Jackson$^{34}$ used the Sellars-Tegart equation to model the flow stress behaviour of aluminum alloys. They conducted hot compression tests on a commercially homogenized 7075 alloy at temperatures from 260 to 420°C and strain rates from 0.008 to 80/s. The study concluded by stating that vastly different homogenization practices could be presumed to cause significant variations in flow stress. Although the Sellars-Tegart equation is useful it is only applicable to high deformation temperatures where the material does not experience any strain hardening.

### 2.4.4 Hardening-Sellars-Tegart Equation

The Sellars-Tegart equation can be modified to account for strain hardening giving the Hardening-Sellars-Tegart (HST) equation by integrating it with the simple hardening law given. Using this method, the steady state flow stress, $\sigma_s$ is replaced with $\sigma_H$ giving:

$$\sigma_H = (\sigma_0 + k_1 \sqrt{\delta(T)} \varepsilon) \cdot f(Z)$$  \hspace{1cm} (2.13)  

$$\delta = \frac{1}{1 + \exp(a_1 + a_2 T)}$$  \hspace{1cm} (2.14)  

$$f(Z) = \min \left[ 1, \arcsin \left( \frac{Z}{A_j} \right)^{ma} \right]$$  \hspace{1cm} (2.15)
Where, $\delta$ is the hardening parameter and $\varepsilon$ is the strain. $\sigma_0, A_i,$ and $m_H$ are material constants (with $m_H = 1/n_H$), and $k$ is a constant. $a_1, a_2,$ are fitting parameters. All other quantities have the same meanings as previously described. The parameter $\delta$, describes the work hardening of a material at the given temperature as compared to room temperature work hardening and its value ranges from 0 (at high temperatures) to 1 (at low temperatures); the parameter experiences a smooth transition between the two values at intermediate deformation temperatures. At high temperatures and low stress levels the HST equation becomes the power law equation. The HST equation can predict constitutive behaviour from room temperature up to the solidus temperature and is well adapted to strongly varying strain rate conditions. Parameters determined at low strain rates can be used to predict high strain rate flow stress values with moderate success.

Haaften et al.\textsuperscript{35} modelled the flow stress of AA3104 using the HST equation. Parameters for the equation were determined using hot compression tests carried out over a large range of temperatures (50 to 500°C) and using strain rates from $5 \times 10^{-3}$ to $1 \times 10^{-5}$. The study found the temperature independence of the parameters in the HST equation to be advantageous but found that the model had difficulty describing the intermediate temperature regime where the material experienced strain hardening and strain rate dependence. Although the HST model is comprehensive, extrusion temperatures are within the intermediate temperature regime and experience significantly higher strain rates than the conditions examined by Haaften et al. The HST equation does not incorporate knowledge of the material microstructure.
2.4.5 Physically-Based Flow Stress Model

Kocks and Chen\textsuperscript{36} developed a physically-based flow stress model that applies to the viscous dislocation motion regime. The regime is characterized by a class of materials and conditions (“Class A”) distinguished on the microscopic scale by the continuous motion of dislocations accompanied by diffusion of solute. The macroscopic strain rate can be related to dislocation motion as follows:

\[ \dot{\varepsilon} = b \rho_d v \]  \hspace{1cm} (2.16)

Where, \( \dot{\varepsilon} \) is the strain rate, \( b \) is the magnitude of the temperature dependent Burgers vector, \( \rho_d \) is the mobile dislocation density, and \( v \) is the dislocation velocity. The Forest theory of work hardening gives the following relationship:

\[ \sigma \propto \mu b \sqrt{\rho_d} \]  \hspace{1cm} (2.17)

\[ \rho_d \propto \left( \frac{\sigma}{\mu b} \right)^2 \]  \hspace{1cm} (2.18)

Where, \( \sigma \) is the applied stress, \( \mu \) is the temperature dependent shear modulus. The motion of dislocations in the viscous deformation regime requires a mass transfer of solute atoms by volume diffusion. The dislocation velocity is then linked to the diffusion of solute giving Equation 2.19. The diffusion coefficient is given by Equation 2.20.
\[ v \propto \left( \frac{D}{kT} \right)(\sigma b)b \] (2.19)

\[ D = D_0 \exp\left( -\frac{Q_D}{RT} \right) \] (2.20)

Where, \( D \) is the diffusion coefficient, \( k \) is the Boltzmann constant, \( T \) is the deformation temperature, \( \sigma b \) is the driving force per unit length of dislocation and \( b \) is the magnitude of the temperature dependent Burger’s vector (accounting for the unit advance distance), \( D_0 \) is the diffusion constant, \( Q_D \) is the activation energy for diffusion of the diffusing species, and \( R \) is the gas constant. Equation 2.19 may depend on detailed assumptions made about the solute dislocation interaction. Combining Equations 2.16 to 2.20 gives the kinetic law:

\[ \dot{\varepsilon} = A \left( \frac{\sigma}{\mu} \right)^n \frac{\mu b^3}{kT} \exp\left( -\frac{Q_D}{RT} \right) \] (2.21)

Where, \( A \) is a pre-exponential constant and \( n \) is the stress exponent (\( n=3 \) for solute drag); these two parameters take into account the variation in 2.18 and 2.19.

This model was applied to the hot deformation of AA5182 by Kocks and Chen.\(^{37}\) Compression tests were conducted at temperatures between 200 and 550°C with strain rates from \( 10^{-3} \) s\(^{-1} \) and 1 s\(^{-1} \). Temperature/strain-rate conditions producing work-hardening curves that were considered for all purposes flat were labelled as having “Class
A” behaviour and being in the regime where the solute drag mechanism dominates.

Rearranging the terms in Equation 2.21 and taking the logarithm gives Equation 2.22.

\[
\log \left( \frac{\sigma}{\mu} \right) = \frac{1}{n} \log \left[ \frac{\dot{\varepsilon}}{s} \frac{kT}{\mu b^3} \exp \frac{Q_D}{RT} \right] - \frac{1}{n} \log A
\]  

(2.22)

Plotting the \( \log(\sigma/\mu) \) versus the \( \log\left(\frac{\dot{\varepsilon}/s^{-1}}{\mu b^3}\right) \exp\left(\frac{Q_D}{RT}\right) \) at different strain rates and temperatures a straight line can be fit to the data points as shown in Figure 2.7.

![Figure 2.7- Yield stress data for AA5182.](image-url)
The slope of the curve is $1/n$, providing a means of determining $n$. The stress-strain response of the material at any position along the curve can be predicted within the temperature/strain-rate regime. The stress exponent found using the physically-based flow stress model describes the dominant deformation mechanism. In the work shown in Figure 2.7, the stress exponent is approximately 3 as solute drag is the dominant deformation mechanism experienced by the Al-Mg alloy, 5182, in the given conditions.

The application of a physically-based flow stress model provides a means of predicting the stress-strain response of aluminum alloys in the temperature/strain-rate regime experienced during extrusion. The model can be used to compare the stress-strain response of the alloys in this study as well the effect of homogenization and microstructure changes on flow stress.

2.4.6 Internal State Variable Flow Stress Model

Physically-based internal state variables can be used to describe the underlying microstructural phenomena that occur during thermomechanical processing. Typically three or fewer internal state variables mathematically characterize microstructure evolution using measurable microstructure parameters. The independent state variables are described by a simultaneous set of differential equations:

\[
\frac{dS_1}{dt} = g_1(S_1, S_2, \ldots, T, \dot{\varepsilon}) \quad (2.23)
\]

\[
\frac{dS_2}{dt} = g_2(S_1, S_2, \ldots, T, \dot{\varepsilon}) \quad (2.24)
\]
Where, \( S \) are the independent state variables, \( T \) is the instantaneous temperature, and \( \dot{\varepsilon} \) is the instantaneous strain rate. The evolution of microstructure during a thermomechanical process is characterized by solving the equations iteratively. A ‘response equation’ for a quantity dependent on microstructure (such as flow stress, \( \sigma \)) is then given by Equation 2.25.

\[
\sigma = g_3(S_1, S_2, \ldots)
\]  

(2.25)

Nes\(^39\) applied the internal state variable approach to develop a work hardening theory. The microstructure is assumed to be comprised of three state variables. The cell/subgrain size, \( \delta_C \), the dislocation density, \( \rho_i \), and the sub-boundary misorientation, \( \varphi \). Evolution laws are presented for these state variables and the flow stress response is written as:

\[
\tau = \tau_i + \tau_p + \alpha_1 G b \sqrt{\rho_i} + \alpha_2 G b (1/\delta_C + 1/D)
\]

(2.26)

Where, \( \tau_i \) is the stress contribution due to rate and temperature dependent interactions with short range obstacles, \( \tau_p \) is the stress contribution due to non-deformable particles, \( \alpha_1 \) and \( \alpha_2 \) are constants, \( G \) is the shear modulus, \( b \) is the magnitude of the Burger’s vector, \( D \) is the grain size, and the sub-boundary misorientation is not included.

The approach described by Nes\(^39\) was applied over deformation conditions ranging from ambient temperature to high temperature creep by Nes and Marthinsen\(^40\).
The internal state variable approach to flow stress modelling links real non-isothermal process histories to the fundamental isothermal histories of microstructure evolution. Careful consideration must be taken when choosing the internal state variables of significance to the problem, balancing model accuracy and complexity. Coupling the thermomechanical history and microstructure evolution during industrial manufacturing processes that involve multiple processing steps are an important application of this approach to constitutive modelling. Although this type of modelling has many advantages, the multitude of adjustable parameters (including experimental, material constant and physical constant parameters) makes this approach challenging. The complexity of internal state variable models and the extensive numerical computation required is unsuitable in applications where a more simplistic physically-based model can accurately describe constitutive behaviour.

2.4.7 Summary of Constituent Models

Empirically based constitutive models such as the power law, Zener-Hollomon, Sellar-Tegart, and HST equations do not incorporate knowledge of the material microstructure and are overly simplistic. Empirical models require a large amount of experimental data to accurately model constitutive behaviour and are very specific to that application, limiting predictive power. Physically-based flow stress models, such as the Kocks and Chen model, link microstructure to constitutive behaviour. The Kocks and Chen model should be capable of accurately modelling flow stress under extrusion deformation conditions and can easily be applied to different alloy compositions; relatively few parameters are needed (compared with HST and internal state variable
models) and the experimental parameters are macroscopic and easily obtained. Internal state variable models while very useful in linking multiple processing steps are overly complex for the application of this study.
Chapter III – Scope and Objectives

3.1 Scope

In this study, experimental work is conducted on 3 AA3xxx alloys (compositions detailed in Section 4.1). The as-cast state and 5 homogenization treatments (described in Section 4.2) are examined. Deformation conditions range from 300 to 600°C at strain rates of 0.1 to 10/s.

3.2 Objectives

The industrial manufacturing process for AA3xxx extruded products involves a complicated thermal mechanical history occurring over multiple processing steps. Optimizing the process to improve the properties of the end product and reduce production costs requires an increased understanding of the effect of homogenization practice and alloy chemistry on extrusion.

The objectives of this research project are as follows:

1. To develop an improved understanding of microstructure evolution during homogenization using simple characterization techniques.

2. To quantify the effect of homogenization treatment and alloy Mn content on subsequent deformation using flow stress measurements.

3. To develop a constitutive model that predicts flow stress values at temperatures and strain rates in the industrial extrusion range.

4. To validate the correlation between flow stress and extrusion pressure using extrusion trial data.
Chapter IV – Experimental Methodology

Experimental work consisted of a multi-faceted approach to measuring the microstructure changes (via resistivity and selected microscopy) that occur during homogenization of AA3xxx aluminum alloys as well as the effect of chemistry and subsequent deformation on the constitutive behaviour of the material. Once developed the model was qualitatively validated by performing a series of extrusion trials using the instrumented press available at ARDC.

4.1 Initial Material

The experimental work was conducted on three aluminum alloys of industrial interest: AA3003, AA3102, and an intermediate Mn composition alloy. The alloys were provided in the as-cast state by the Alcan Research and Development Center (ARDC) of Rio Tinto Alcan located in Jonquiere Quebec. As-cast billets homogenized at soak temperatures 500, 550 and 630°C for 8 hours were also provided by the ARDC for each of the three alloys. The chemical compositions of the alloys are given in Table 4.1.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Alloy</th>
<th>Mn</th>
<th>Fe</th>
<th>Si</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Mn</td>
<td>AA3102</td>
<td>0.26</td>
<td>0.50</td>
<td>0.10</td>
<td>0.02</td>
<td>bal.</td>
</tr>
<tr>
<td>Med Mn</td>
<td>Med Mn</td>
<td>0.75</td>
<td>0.52</td>
<td>0.10</td>
<td>0.02</td>
<td>bal.</td>
</tr>
<tr>
<td>High Mn</td>
<td>AA3003</td>
<td>1.27</td>
<td>0.54</td>
<td>0.10</td>
<td>0.02</td>
<td>bal.</td>
</tr>
</tbody>
</table>

Table 4.1 – Chemical composition of alloys used in experiments (wt %).
4.1.1 Characterization

Initial Characterization of the as-cast AA3003 material was conducted along the radius of the billet. Samples were taken from the billet in three sections: 1-center, 2-middle, and 3-outer as shown in Figure 4.1.

![Figure 4.1- Initial characterization of billet.](image)

Optical microscopy was performed on the three samples to examine and compare the grain size at the 1-center, 2-middle, and 3-outer positions as shown in Figure 4.2. Minimal grain size variation can be seen along the radius of the billet. It was noted that an area of inverse segregation, approximately 500 µm in thickness, existed along the outer edge of the billet.
Figure 4.2 a) 1-center section of AA3003 billet radius (500x magnification).

Figure 4.2 b) 2-middle section of AA3003 billet radius (500x magnification).

Figure 4.2 c) 3-outer section of AA3003 billet radius (500x magnification).
4.1.2 Sample Preparation

Two types of samples were taken from the as-cast and homogenized billets: samples to be used for resistivity measurements and cylindrical compression samples. In both cases, the samples were taken from the section of the as-cast and homogenized billets that had been initially characterized as having minimal grain size variation; samples were not taken from the area of inverse segregation or within a 5 mm radius of the center of the billet, as casting defects in this area would be more probable. Resistivity and compression samples were taken along the axial direction such that the deformation during compression was parallel to the axial direction of the billet. Figure 4.3 shows the sample orientations and metallographic faces of interest in relation to the billet direction. Resistivity and compression sample dimensions are provided in Figure 4.4. The length of the compression samples was limited by the amount of space between the jaws of the compression test equipment.

Figure 4.3- Sample orientation and metallographic faces of interest.
4.2 Homogenization

Homogenization heat treatments were performed on the resistivity and compression samples taken from the as-cast billets. The homogenization treatments were
initially performed using a box furnace (refer to Appendix A for more information). The box furnace was later replaced with a recirculating air furnace.

The Carbolite™ (HRF) 750°C recirculating air furnace was purchased due to its ease of control and ability to heat large sections of material homogeneously. Using this furnace, modifications to the furnace setup were not needed to provide homogeneous heating of the samples. Thermocouples that had been welded onto a steel plate were placed inside the furnace to record temperatures in three locations along the length of the furnace. Samples to be heat treated were packed tightly together on the steel plate in between thermocouples one and two (numbering from front of furnace to back). Thermocouple data was recorded using instruNet™ software and an instruNet Model 100 DAQ. The actual furnace temperature (measured by the thermocouples) had a maximum deviation of ± 5°C from the controller set point temperature during the hold (soak) cycle.

The samples in the recirculating air furnace were easy to remove when quenching. The short quench time, as well as the furnace’s high heating rate capabilities, meant that the furnace temperature could be controlled very accurately even after opening the furnace door for a short period of time; this allowed multiple samples to be heat treated and quenched at differing times without affecting the homogenization of samples that remained within the furnace. A photo of the recirculating air furnace is given in Figure 4.5. All homogenization heat treatments were performed with a heating rate of 150°C/h, and a heating rate of 50°C/h for the last hour to the homogenization soak temperature. The heating rates were chosen by Rio Tinto Alcan to be consistent with the homogenization heating process used in industry. The 50°C/h rate was used to model a
more gradual shift from the heating of the material to the soak temperature, as experienced during industrial practices.

![Recirculating air furnace](image)

**Figure 4.5- Recirculating air furnace.**

After soaking, a water quench was performed on the billets/samples. The quench allowed the homogenized materials to be used in compression tests without the microstructural changes that would have occurred if the material had been cooled slowly. Quenching of the resistivity samples after homogenization allowed electrical conductivity measurements and metallography to be used to examine the microstructure prior to deformation. Figure 4.6 shows the various homogenizations profiles examined in this study.
4.3 Electrical Conductivity Measurements

Electrical conductivity measurements of the resistivity and compression samples were made using a Sigmatest® 2.069 (by Foerster Instruments Inc.™) with an 8 mm probe. Standards and the air point were used to calibrate the unit each time before use. Measurements were made using a frequency of 60 KHz. The data was output in units of MS/m. Electrical conductivity measurements performed during extrusion trials at the ARDC were also made using a Sigmatest® 2.069. The electrical conductivity of the billets prior to extrusion and the extruded strip were measured using 60 KHz and 480 KHz (due to the strip thickness), respectively. Output data had units of %IACS.
(International Annealed Copper Standard) where the conductivity of annealed, unalloyed copper is arbitrarily rated at 100% and the conductivities of other alloys are expressed as a percentage of this standard. The units were converted using Equation 4.1. Where, $\sigma$ is the electrical conductivity in MS/m and $x$ is the electrical conductivity in %IACS.

Electrical conductivity was converted to resistivity using Equation 4.2, where $\rho$ is the resistivity of the sample.

\[
\sigma = \frac{x(100)}{172.41} \text{[MS/m]} \tag{4.1}
\]

\[
\rho = \frac{1}{10^3 \cdot \sigma} \text{[nΩ·m]} \tag{4.2}
\]

4.3.1 Resistivity Samples

After homogenization electrical conductivity measurements were made on the resistivity samples. For each sample, two electrical conductivity measurements were made on both the top and bottom surfaces (refer to Figure 4.4); the measurements were made close to the center of the sample.

4.3.2 Compression Samples

Electrical conductivity measurements were made for the homogenized compression samples before deformation and after deformation. One measurement was
taken on each end of the cylindrical specimen. After deformation, the ends of the compression samples had nickel paste lubricant on them and the surfaces themselves were not as smooth as prior to deformation. The contaminants and roughness of the surface potentially could have had an effect on the electrical conductivity measurements so these two effects had to be removed. The nickel paste was removed and the surface smoothed by grinding the compression sample ends with 60 grit paper and then 600 grit paper. The electrical conductivity measurements were then made on the deformed samples.

4.3.3 Extruded Material

Prior to extrusion, electrical conductivity measurements were made of the homogenized billets at the ARDC. The measurements were taken at three points on the end of each cylindrical billet (giving a total of six measurements for each billet). After extrusion, measurements were made on the extruded strip. Three measurements were made on each side of the strip, two at the end points and one in the center.

4.4 Metallography

Metallography was performed by the metallographic technicians at the ARDC. A rectangular piece was cut from the center of each resistivity sample. Samples were hot mounted using Buehler PhenoCure™. Samples were ground and polished using a Struers automatic preparation system. Water was used as a lubricant during grinding and
extender oil was used as a lubricant during polishing. The grinding and polishing schedules are given in Tables 4.2 and 4.3, respectively.

Etching of the samples was done using a mixture of distilled water and hydrofluoric acid (0.5% volumetric HF). The samples were immersed for 5s and 60s, and swirled around. Room temperature water was used for rinsing. Optical microscopy was then performed using equipment at the ARDC.

Table 4.2- Grinding schedule.

<table>
<thead>
<tr>
<th>Type of Paper</th>
<th>Grit (µm)</th>
<th>Speed (rpm)</th>
<th>Total Force on 6 samples (N)</th>
<th>Time (min)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>120</td>
<td>150</td>
<td>150</td>
<td>2</td>
<td>When finished: rinse with water, compressed air</td>
</tr>
<tr>
<td>SiC</td>
<td>240</td>
<td>150</td>
<td>150</td>
<td>1</td>
<td>When finished: rinse with water, compressed air</td>
</tr>
<tr>
<td>SiC</td>
<td>320</td>
<td>150</td>
<td>150</td>
<td>1</td>
<td>When finished: rinse with water, compressed air</td>
</tr>
<tr>
<td>SiC</td>
<td>400</td>
<td>150</td>
<td>150</td>
<td>1</td>
<td>Listen carefully if sounds bad take it off, paper may be worn. When finished: rinse with water, compressed air</td>
</tr>
</tbody>
</table>

Table 4.3- Polishing schedule.

<table>
<thead>
<tr>
<th>Type of Cloth</th>
<th>Size (µm)</th>
<th>Speed (rpm)</th>
<th>Total Force on 6 samples (N)</th>
<th>Time (min)</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Texmet</td>
<td>6</td>
<td>150</td>
<td>125</td>
<td>8</td>
<td>When finished: rinse with water, scrub with cotton swab, compressed air.</td>
</tr>
<tr>
<td>Microcloth</td>
<td>1</td>
<td>150</td>
<td>125</td>
<td>5</td>
<td>When finished: spray 10% FL-70 detergent (90% water), rinse with water, scrub with cotton swab, compressed air. Microscope: check for particles, esp. corners of sample</td>
</tr>
<tr>
<td>Chemotextile</td>
<td>0.05</td>
<td>150</td>
<td>125</td>
<td>4</td>
<td>IMMEDIATELY clean with water and rub with cotton, do not use detergent may leave residue</td>
</tr>
</tbody>
</table>
4.5 Compression Testing

Compression tests were performed at strain rates of 0.1, 1, and 10/s at deformation temperatures between 300 to 600°C using the Gleeble® 3500 thermal-mechanical testing machine shown in Figure 4.7, available at UBC. Prior to testing, the diameter and length of each compression sample was measured to ensure that it conformed to the expected dimensions (within ±0.05mm). A thermocouple (K type) was spot welded to each sample approximately mid-point along the length. Compression samples were lubricated using nickel paste which was applied liberally. Low force jaws were used in the compression test apparatus (Appendix A details problems encountered with the compression test setup and the changes that were made). The low force setup upon completion of a compression test is shown in Figure 4.8.

![Gleeble thermal-mechanical tester used for compression tests.](image)

For the higher temperature, low strain rate tests a 1,000 lb (4.4 kN) load cell was used. For larger flow stress tests a 5,000 lb (22.2 kN) load cell was used to remain in the
proper load cell range. Strain measurements were made using a c-gauge that was located at the mid-point of the sample length. The ram was moved to a position in which it was exerting a small force on the sample (< 200 N) to hold the sample in place as the weight of the C-gauge would otherwise move the sample; when this position was reached the ram was set to what would be the zero position for the subsequent test. The rough vacuum was used to evacuate the experimental chamber to a pressure of $2.7 \times 10^{-1}$ torr.

Figure 4.8- Low force jaw setup. 1) Jaws, 2) thermocouple, 3) compression sample, 4) small load cell.

Compression samples were heated at a rate of $5^\circ$C/s to the required deformation temperature. The sample was then held at temperature for three seconds before deformation at the required strain rate. Compression tests performed using a strain rate of $10/s$ required that the servo response be set to fast. Cooling of the sample was not controlled, it occurred partially in the Gleeble chamber and partially under atmospheric conditions. Curves which exhibited noise in the data had the sampling rates decreased in the Gleeble program for that condition. Gleeble output data included: time, diameter,
force, strain, stress, stroke, and temperature. Stress-strain curves were constructed using the data; the yield stress and steady state flow stress values were found for each test (method described in Appendix B).

4.6 Extrusion Trials

Extrusion trials were performed at the ARDC. The trials were done on two separate days with the 400°C deformation temperature completed on the first day and the 550°C completed on the second.

4.6.1 Extrusion Press

Rio Tinto Alcan’s state-of-the-art research extrusion press was used for the extrusion trials. Photos of the press and the water quench are given in Figure 4.9.
Figure 4.9- a) Extrusion press used in trials. 1) Extruded strip exits here, 2) billet position for extrusion, 3) mechanical billet loading system, 4) ram.

The homogenized billets were placed in an induction heater for a few minutes (the time between billets) and heated to the selected deformation temperature. The billet was then moved mechanically into position in the press. After the billet was in position the ram would begin the push. The extruded strip would exit the die and travel through a water quench of turbulent water. The end of the extruded strip would then be pulled lightly by a technician to ensure that the strip did not back up in the press. The technician would pull the strip down the length of the run out table.
4.6.2 Extrusion Parameters

Prior to the billets of interest, 4 warm up billets (preheat billets) were used to warm up the extrusion press. The die used in the test was preheated to a temperature of 450°C and 480°C for the 400°C and 550°C extrusion trials respectively. For both trials the extruded strip had an I-beam profile with an extrusion ratio of 130. The extrusion ratio (ER) was calculated as follows:
\[ ER = \frac{A_b}{A_s} \]  

(4.3)

Where:

\[ A_b = \pi (50 \text{mm})^2 \]  

(4.4)

\[ A_s = 2A_f + A_w \approx 60.5 \text{mm}^2 \]  

(4.5)

Where \( A_b \) is the circular area of the billet, \( A_s \) is the area of the extruded strip profile, \( A_f \) is the area of the flange, and \( A_w \) is the area of the web.

The billet temperatures (heated prior to being extruded using the induction heater) corresponded to the required extrusion trial deformation temperature; for the first trial this temperature was 400°C and for the second trial it was 550°C. The exit speed of the extruded strip was 1.8 m/s for both tests as recommended by Rio Tinto Alcan. The time each billet took to be extruded (from induction heater to water quench) differed slightly and can be determined from the acquired data.

### 4.6.3 Data Acquisition

Data acquired from the extrusion tests included: ram position, ram velocity, exit temperature (laser measurement), exit temp (probe measurement), ram pressure, billet temperature (at the middle of the billet as it was loaded into the press), liner thermocouples (twelve in total), container thermocouples (five in total), one die thermocouple (this thermocouple did not work properly for the 550°C trials). Data was acquired on a PC in an excel file format.
Chapter V – Results

The experiments conducted during this research consisted of varying homogenization treatments for a series of AA3xxx aluminum alloys with different Mn levels (low, medium and high). The deformation behavior of both as-cast and homogenized alloys was measured using a Gleeble 3500. Results from these experimental measurements will be presented in this chapter.

The microstructure evolution that occurred during homogenization was assessed both qualitatively through optical metallography as well as quantitatively through electrical conductivity (conductivity) measurements at selected times during the homogenization process. Deformation behavior of the various alloys/homogenization treatments were measured through compression tests using a Gleeble 3500 under various temperatures and strain rate conditions while conductivity measurements provided information on the relationship between deformation conditions and microstructure. Results from the extrusion trials were used to assess the influence of alloy composition, amount of Mn in solid solution, homogenization treatment and extrusion temperature on the required extrusion pressure.

5.1 Electrical Conductivity

Conductivity measurements were made at select times during a homogenization heat treatment. The conductivity of compression samples before deformation and again after deformation was measured to determine if Mn had precipitated from solid solution. The conductivity of the homogenized extrusion billets and extruded material were
measured to determine if precipitation had occurred and as a comparison with compression sample conductivity measurements. The main result of these measurements was an observed increase in conductivity for the Low and High Mn alloys in the as-cast state as well as a decrease in conductivity for the High Mn alloy 8 hr, 500°C homogenization (see Appendix C for a summary of all measurements); this will be discussed in section 6.1.2.

5.1.1 Heat Treatments

The conductivity was measured throughout a homogenization treatment for each of the three alloys to examine the change in conductivity with time. As discussed in Chapter III, the conductivity has an inverse relationship with the amount of Mn in solid solution. The results for the Low, Med, and High Mn alloys are given in Figures 5.1 to 5.3, respectively. A typical change in conductivity during homogenization is shown in Figure 5.2. Initially during heating the conductivity is constant. When a homogenization temperature of 300°C is reached the conductivity begins to increase. The conductivity increases quickly with temperature up to a peak at around 520°C and then begins to decrease. The conductivity then starts to increase upon (or close to) the completion of heating. During the soak period the conductivity initially increases quickly but this increase tapers off with soak time. The trend of the conductivity evolution for the High Mn and Med Mn alloys are similar; the Low Mn alloy conductivity curve has a noticeably different shape as the conductivity decreases with soak time. Starting values for the conductivity curves are all different which is consistent with different starting levels of solute due to different levels of Mn in solid solution.
Figure 5.1- Measured conductivity during homogenization of the Low Mn alloy.

Figure 5.2- Measured conductivity during homogenization of the Med Mn alloy.

Figure 5.3- Measured conductivity during homogenization of the High Mn alloy.
5.2 Homogenized Microstructure

At each point that the conductivity was measured during homogenization, samples were examined with optical metallography; this was done for each of the three experimental alloys. The micrographs provide information on an individual alloy’s microstructure changes during a 600°C, 24 hour homogenization as well as a means of comparing the microstructure of the alloys used in this study at specific points during the heat treatment. The progression of the homogenization microstructure for the High Mn alloy is shown in Figure 5.4 to 5.9. Additional metallography was done for the Low Mn and Med Mn alloys and will be discussed in Chapter VI.

The as-cast microstructure is shown in Figure 5.4. Micrographs at 384°C and 528°C during the heating ramp are shown in Figures 5.5 and 5.6, respectively. Micrographs of 1 hour, 8 hour, and 24 hour soak times are shown in Figures 5.7 to 5.9. The as-cast and 384°C micrographs (Figures 5.4 and 5.5) exhibit a very fine series of constituent particles in the interdendritic regions and no dispersoid precipitation can be seen. At 528°C (Figure 5.6) the constituent particles have spheriodized and a large numbers of dispersoids have precipitated. After heating and a soak time of 1 hour (Figure 5.7) the constituent particles have coarsened and the number density of dispersoids has decreased, particularly in areas close to grain boundaries. The 8 hour soak microstructure image shows further coarsening of the constituent particles and a significant decrease in the number density of dispersoids (Figures 5.8). A soak time of 24 hours (Figure 5.9) shows significant coarsening of the constituent particles with no dispersoids remaining.
Figure 5.4- High Mn alloy, as-cast.

Figure 5.5- High Mn alloy quenched at: 384°C, 2.5 hrs (ramp).

Figure 5.6- High Mn alloy quenched at: 528°C, 3.4 hrs (ramp).
Figure 5.7- High Mn alloy quenched at: 596°C, 1 hr (soak).

Figure 5.8- High Mn alloy quenched at: 601°C, 8 hr (soak).

Figure 5.9- High Mn alloy quenched at: 602°C, 24 hr (soak).
5.3 Compression Test Results

In total, over 140 compression tests were conducted using the Gleeble thermal-mechanical tester (as described in Section 4.5) including initial tests performed using a larger sample size and repeated tests (conducted to ensure repeatability). In the following section examples will be given to illustrate trends in the compression test results. A complete table (excluding the larger sample and repeated tests) of the yield stress and flow stress results, for each of the three alloys, is found in Appendix C.

Flow stress curves for the high Mn alloy with a single homogenization condition (8 hr, 500°C soak) are shown in Figures 5.10 and 5.11. Figure 5.10 shows the effect of deformation temperature on flow stress for a single strain rate (1/s); an increase in deformation temperature causes a decrease in the flow stress of the material. Figure 5.11 shows the effect of strain rate on flow stress for a single deformation temperature (500°C); an increase in strain rate causes an increase in the flow stress of the material.

The effect of homogenization treatment on flow stress (for the High Mn alloy with a deformation temperature of 500°C and a strain rate of 1/s) is shown in Figure 5.12. From the figure, it can be seen that homogenization treatments decrease flow stresses substantially. The 500°C (8 hr) homogenization treatment is seen to have a significantly higher flow stress than the other homogenization treatments, with a flow stress closer to that of the as-cast state. The 600°C (24 hr) has the lowest flow stress for these deformation conditions. At large strains the 550°C (8 hr), 600°C (24 hr), and 630°C (8 hr) flow stress curves appear to converge while the 500°C (8 hr) remains relatively unchanged.
Figure 5.10- Effect of deformation temperature on flow stress. High Mn alloy with a 500°C, 8 hr homogenization soak (strain rate 1/s).

Figure 5.11- Effect of strain rate on flow stress. High Mn alloy with a 500°C, 8 hr homogenization (deformation temperature 500°C).
Figure 5.12- Comparison of homogenization treatment flow stress results for the High Mn alloy with a deformation temperature of 500°C and a strain rate of 1/s.

Steady state flow stress values provide a means of easily comparing the different heat treatments and alloys. The methods used to calculate the steady state flow stress values (hereafter shortened to flow stress) and the yield stress values are given in Appendix B.

A comparison of the flow stress values for deformation temperatures 400°C, 500°C, and 600°C are given in Figure 5.13 (for the High Mn alloy with a deformation temperature of 500°C and a strain rate of 1/s). Some results are unavailable (and are marked with an ‘*’) as deformation conditions were selected for the flow stress model (to be discussed in Chapter VI) to decrease the size of the experimental matrix. In Figure 5.13, flow stress values decrease with an increase in deformation temperature. It can be seen that the 500°C (8 hr) homogenization has the largest flow stress value of the
homogenized materials for all three deformation temperatures. For the three deformation conditions flow stress appears to initially decrease with increasing homogenization temperature but subsequently increases or levels off.

Figure 5.13- Comparison of homogenization flow stress results for the High Mn alloy with deformation temperatures 400, 500, and 600°C and a strain rate of 1/s.

Unavailable data is represented by ‘*’.

A comparison of flow stress values for the three alloys (for a deformation temperature of 500°C and a strain rate of 1/s) is shown in Figure 5.14. For a given deformation temperature and strain rate an increase in alloy Mn content increases the flow stress.
Figure 5.14: Comparison of as-cast alloy flow stress results for a deformation temperature of 500°C and a strain rate of 1/s.

5.4 Summary of Compression Test Results

The compression test results have shown that increasing deformation temperature causes a decrease in flow stress values and increasing strain rate causes an increase in flow stress values; the relationship between deformation temperature, strain rate, and flow stress is as would be expected, with the results quantifying the magnitude of these effects. Further, increasing the Mn alloy content increases the flow stress. An increase in homogenization temperature and/or time can decrease flow stress values however this is not always the case; it is apparent that there exists a complex relationship between the evolution of homogenization microstructure and the corresponding effect on flow stress values.
5.5 Extrusion Trial Results

A large amount of data (as described in Section 4.6.3) was collected from the extrusion trials. One parameter that can influence extrusion is the material flow stress behavior. By altering the homogenization treatment and alloy composition the flow stress behavior can be altered and this effect can be assessed qualitatively by examining the trends in the measured extrusion pressures during the trials.

Extrusion pressures from a trial performed at 400°C (for the High Mn alloy) are given in Figure 5.15. Extrusion pressures from a trial performed at 550°C (for the High Mn alloy) are given in Figure 5.16. All trials were performed at a constant ram speed (extrusion parameters are detailed in Section 4.6.2).

Relative values of the extrusion pressures in Figure 5.15 can be compared with relative values of the flow stress curves (over the range of 0.4 to 0.6 strain) in Figure 5.12. In both figures the largest extrusion pressure/flow stress values are seen for the as-cast state. Extrusion pressure/flow stress values then decrease for the 500°C, 8 hr homogenization condition. The smallest extrusion pressure/flow stress values are seen for the 550°C, 8 hr and 600°C, 24 hr homogenizations.

Relative extrusion pressures seen in the 550°C extrusion trial (given in Figure 5.16) exhibit different trends to those seen in the 400°C trial; this suggests that the extrusion pressures for the various homogenization treatments are dependant on deformation temperature and that the effect of deformation temperature must be taken into account when optimizing the extrusion process.
Figure 5.15- High Mn alloy extrusion pressures (400°C deformation temperature).

Figure 5.16- High Mn alloy extrusion pressures (550°C deformation temperature).
Extrusion pressures (at the industry standard 800 mm ram position) for the 400°C and 550°C trials are given in Figures 5.17 and 5.18, respectively. Note that the scale used in the Figures differs in order to visually show the differences in the homogenization extrusion pressures for each trial.

The 400°C trial extrusion pressures exhibit a similar trend between the Low Mn and High Mn alloys for the as-cast state. The Low Mn alloy extrusion pressures show a slight decrease from the 500°C, 8hr to the 550°C 8hr homogenization while the 600°C, 24 homogenization shows an increase in extrusion pressure. The High Mn alloy does not show this same trend, the extrusion pressures decrease from the 500°C, 8hr to the 550°C 8hr homogenization; The 550°C 8hr and 600°C, 24 hr homogenization conditions have similar extrusion pressures.

![Figure 5.17- Extrusion pressures (at a ram position of 800 mm) for a deformation temperature of 400°C.](image)
The 550°C trial extrusion pressures for the Low Mn alloy show little difference between the as-cast state and the homogenized materials, suggesting that at this deformation temperature homogenization has little effect on the extrusion pressure. The 550°C trial extrusion pressures for the High Mn alloy follow similar trends to those seen in the High Mn alloy, 400°C extrusion trials; the extrusion pressures decrease from the 500°C, 8hr to the 550°C, 8 hr homogenization, however, for this extrusion trial the 600°C, 24 hr homogenization has the lowest extrusion pressure.

![Figure 5.18](image)

**Figure 5.18- Extrusion pressures (at a ram position of 800 mm) for a deformation temperature of 550°C.**

In both trials the Low Mn alloy has significantly lower extrusion pressures than the High Mn alloy. A complete table of the extrusion trial data (at a ram position of 800 mm) can be found in Appendix C.
Chapter VI – Discussion

This chapter presents a discussion of the effect of homogenization and alloy chemistry on the flow stress of AA3xxx alloys and its impact on extrusion pressure. The increase in Mn solubility with temperature, the evolution of homogenized microstructure and the possibility of dynamic precipitation/dissolution of dispersoids during extrusion are discussed. A comparison of flow stress results is conducted using the physically-based constitutive model against the extrusion pressures measured during the industrial plant trial; the applicability and validation of the model are evaluated.

6.1 Evolution of Microstructure

6.1.1 Mn Solid Solubility

The solid solubility of Mn increases with temperature and is dependent on the nominal Mn content of the alloy. The relationship between Mn solubility and temperature under equilibrium conditions was determined using Thermo-Calc™ and is shown by the curves in Figure 6.1; the estimated Mn in solid solution after a 24 hr soak at 600°C was calculated using conductivity measurements and is shown as data points for the Low, Med, and High Mn alloys.

The Low Mn alloy solid solubility is significantly less than that of the Med and High Mn alloys and the curve differs in shape above 600°C. At 400°C all three alloys have solid solubilities of less than 0.1 wt% Mn. As the temperature increases to 500°C, the solid solubility of each of the three alloys has increased by more than 60%. After a 24 hr soak at 600°C the estimated Mn in solid solution (data points in Figure 6.1) is near
equilibrium for the Med and High Mn alloys while the Low Mn alloy is further from equilibrium.

![Figure 6.1- Temperature vs. Mn solid solubility under equilibrium conditions (Thermo-Calc), and after a 24 hour, 600°C soak (EC measurements).](image)

### 6.1.2 Homogenization

Distinct microstructures were created for each of the three alloys by changing the homogenization soak time. The evolution of microstructure with soak time (at 600°C) is shown for the Low, Med, and High Mn alloys in Figures 6.2 to 6.4, respectively; the heating rate is typically 150°C as described in Section 4.2. Homogenization at high temperatures (such as 600°C) and over long periods of time allows both short range and long range diffusion to occur, causing the constituent particles to grow and coarsen with time and the dispersoids to coarsen and dissolve.
Figure 6.2- Low Mn conductivity curve for 600°C homogenization. Microstructure at: (1) 1, (2) 8, and (3) 24 hr soak times.
Figure 6.3- Med Mn conductivity curve for a 600°C homogenization. Microstructure at: (1) 1, (2) 8, and (3) 24 hr soak times.
Figure 6.4- High Mn conductivity curve for a 600°C homogenization. Microstructure at: (1) 1, (2) 8, and (3) 24 hr soak times.
The Mn content of an alloy impacts constituent particle morphology and the amount of dispersoids observed at a given soak time. Understanding the effect of soak time and Mn alloy content (at a given soak temperature, in this case 600°C) allows the microstructure to be optimized for a particular application. Short homogenization times such as 1 hour (refer to Figures 6.2.1, 6.3.1, and 6.4.1), give the largest number density of constituent particles during the soak. The constituent particles are small in size as minimal coarsening has occurred. Short homogenization times give the largest number density of dispersoids during the soak (provided the Mn content of the alloy is sufficiently high enough to precipitate dispersoids). An intermediate soak time such as 8 hrs (refer to Figures 6.2.2, 6.3.2, and 6.4.2), gives fewer but larger constituent particles than a short soak time. The number density of dispersoids has decreased with time as some of the Mn has re-dissolved back into solution at the soak temperature (this is only applicable to the High Mn alloy). Long homogenization times such as 24 hours (refer to Figures 6.2.3, 6.3.3, and 6.4.3), provide the largest size and smallest number density of constitutive particles. No dispersoids can be seen after a 24 hour soak in any of the alloys studied.

The other method of optimizing the microstructure is through chemistry modification (i.e., Mn alloy content). At a given soak temperature a decrease in Mn alloy content causes a decrease in the number density of both the constituent particles and dispersoids (provided enough Mn was present for dispersoids to have precipitated). The amount of Mn in solid solution slowly decreases with soak time as shown by the slow increase in conductivity; however, this is not the case for the Low Mn alloy. The Low Mn alloy experiences a decrease in conductivity with soak time which would suggest that the
amount of Mn in solid solution is increasing. An increase in Mn supersaturation would presumably move the system further from equilibrium which seems unlikely. At the current time the cause of the conductivity decrease with soak time for the Low Mn alloy cannot be explained and future work will need to be done to determine the cause of this phenomenon. At the onset of soaking the three alloys exhibit different conductivities. The initial soak conductivity decreases in the following order: Low Mn, Med Mn, High Mn; thus, an increase in Mn alloy content results in an increased concentration of Mn in solid solution. The increase in conductivity from the 1 hour to the 24 hour soak time is approximately the same (1 MS/m) for the Med and High Mn alloys suggesting that the same amount of Mn has precipitated for both alloys over the same soak period.

The amount of Mn in solid solution, size and number density of constituent particles, and number density of dispersoids can be optimized by controlling the alloy Mn content and soak time.

**6.1.3 Deformation**

A comparison of conductivity measurements made before and after deformation of the compression samples showed negligible changes in most cases. Conductivity was measured twice for each condition and the average was taken. Typical variations in EC_{before} measurements were ± 0.6 MS/m while variations in EC_{after} were ± 0.7 MS/m. Compression test data showing a 5% or greater change in conductivity after deformation are given in Table 6.1. Extrusion trial conductivity measurements made before (billet) and after (extruded strip) deformation, showing a 5% or greater change in conductivity
after deformation are given in Table 6.2. Complete conductivity data for both the compression tests and extrusion trials can be found in Appendix C.

### Table 6.1- Compression sample conductivities before and after hot deformation.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Homogenization Soak Time (h)</th>
<th>Homogenization Temp (°C)</th>
<th>Deformation Temp (°C)</th>
<th>Strain Rate (s(^{-1}))</th>
<th>EC before (MS/m)</th>
<th>EC after (MS/m)</th>
<th>Change (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>High Mn</td>
<td>As-cast</td>
<td>N/A</td>
<td>600</td>
<td>0.1</td>
<td>16.1</td>
<td>16.9</td>
<td>5</td>
</tr>
<tr>
<td>High Mn</td>
<td>8</td>
<td>500</td>
<td>600</td>
<td>10</td>
<td>22.9</td>
<td>21.6</td>
<td>-6</td>
</tr>
</tbody>
</table>

### Table 6.2- Extrusion trial conductivities before and after deformation.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Extradion Trial Parameters</th>
<th>Extrusion Trial Data</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Homogenization Soak Time (h)</td>
<td>Homogenization Temp (°C)</td>
</tr>
<tr>
<td>Low Mn</td>
<td>As-cast</td>
<td>N/A</td>
</tr>
<tr>
<td>Low Mn</td>
<td>As-cast</td>
<td>N/A</td>
</tr>
<tr>
<td>High Mn</td>
<td>As-cast</td>
<td>N/A</td>
</tr>
<tr>
<td>High Mn</td>
<td>8</td>
<td>550</td>
</tr>
<tr>
<td>High Mn</td>
<td>As-cast</td>
<td>N/A</td>
</tr>
<tr>
<td>High Mn</td>
<td>8</td>
<td>500</td>
</tr>
</tbody>
</table>

In both the compression tests (at 600°C) and extrusion trials (at 400 and 550°C) the High Mn alloy in the as-cast state showed a significant increase in conductivity. A significant decrease in conductivity was seen for both the compression tests (at 600°C) and extrusion trials (at 550°C) with an 8 hour, 500°C homogenization. In each of these cases the extrusion trials showed a greater change in conductivity for a given condition. The compression sample conductivities appear to be useful in predicting the largest
variations in extrusion trial conductivity but appear to be incapable of showing more subtle changes.

Positive increases in conductivity suggest the precipitation of Mn from the supersaturated matrix while decreases in conductivity suggest the dissolution of precipitates (an increase in Mn supersaturation); however, changes in microstructure such as recrystallization may have occurred, influencing the conductivity measurements. At the present time the cause of the conductivity changes and the discrepancy between the compression sample/extrusion trial measurements is unclear. Future work such as an optical analysis of the deformed microstructure is suggested.

6.2 Constitutive Model

The physically-based constitutive model developed by Kocks and Chen\textsuperscript{30} (discussed in Section 2.4.4) was applied to the compression test results (detailed in Chapter V) using the following equation:

\[
\dot{\varepsilon} = A \left( \frac{\sigma}{\mu} \right)^n \frac{1}{kT} \frac{4b^3}{\mu} \exp \left( -\frac{Q_D}{RT} \right)
\]

(6.1)

Where, \(\sigma\) is the stress, \(\mu\) is the temperature dependent shear modulus, \(n\) is the stress exponent, \(\dot{\varepsilon}\) is the strain rate, \(k\) is the Boltzmann constant, \(T\) is the deformation temperature, \(b\) is the temperature dependent magnitude of the Burgers vector, \(Q_D\) is the activation energy for diffusion of the diffusing species, \(R\) is the gas constant, and \(A\) is a
pre-exponential constant. The x-axis component of Equation 6.1 will be described as ‘x’ and is given as:

$$\log\left(\dot{\varepsilon} \frac{kT}{\mu b^3} \exp \frac{Q_D}{RT}\right) = x$$  \hspace{1cm} (6.2)

Model parameters used in this study are given in Table 6.3. The activation energy for diffusion of Mn, $Q_D$ was taken as 211.4 kJ/mol. The temperature dependence of the shear modulus and Burger’s vector were taken into account (refer to Appendix B for calculations).

**Table 6.3- Parameters used in AA3xxx constitutive model (for all Mn values and microstructures).**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\mu_0$</td>
<td>25400</td>
<td>MPa</td>
</tr>
<tr>
<td>$T_M$</td>
<td>933</td>
<td>K</td>
</tr>
<tr>
<td>$T_M \frac{d\mu}{\mu_0 dT}$</td>
<td>-0.5</td>
<td></td>
</tr>
<tr>
<td>$a$</td>
<td>$4.05 \times 10^{-10}$</td>
<td>m</td>
</tr>
<tr>
<td>$k$</td>
<td>$1.38065 \times 10^{-23}$</td>
<td>Pa·m$^3$·K$^{-1}$</td>
</tr>
<tr>
<td>$Q_D$</td>
<td>211400</td>
<td>J·mol$^{-1}$</td>
</tr>
<tr>
<td>$R$</td>
<td>8.314</td>
<td>J·K$^{-1}$·mol$^{-1}$</td>
</tr>
</tbody>
</table>

Yield stress, flow stress, and work hardening results for the High Mn alloy were plotted using the constitutive model and are given in Figures 6.4, 6.5 and 6.6, respectively [where Figure a) is the 500°C, 8 hour homogenization and b) is the 630°C
homogenization]. Work hardening values for a material were determined by subtracting the yield stress from the steady state flow stress. Compression test results are shown in the graphs as symbols. The model fitting parameters ($A$ and $n$) were determined by fitting a straight line to the semi-log plot. The fitting parameters were then applied to the model to produce the model prediction curve. The model predictions exhibit a good fit to the compression test data for the yield stress, flow stress, and work hardening results. As seen from the figures, both homogenizations exhibit unique curves and can be described by the stress exponent, $n$, and pre-exponential constant, $A$.

High temperature/low strain rate conditions give low ‘$x$’ values while low temperature/high strain rate conditions give high ‘$x$’ values. Select compression tests were performed at 300°C with strain rates of 1 and 10/s (only for the 8 hour 630°C homogenization); in Figures 6.5 b), 6.6 b), and 6.7 b), the tests deviate from the stress values predicted by the model. The model breaks down at ‘$x$’ values between approximately 16 and 17 (for a strain rate of 1/s this corresponds to temperatures between 314 and 348°C); thus the model is valid for our temperature range of 400 to 600°C and can be extended to temperatures as low as approximately 350°C (for a strain rate of 1/s). Model breakdown occurs due to a change in the dominant deformation mechanism.

By visually comparing Figures 6.5 and 6.6, it can be seen that the flow stress results have a better fit. Although the yield stress results provide a good correlation to the constitutive model, the compression testing equipment inherently gives more accurate flow stress results.
Figure 6.5- Yield stress data plotted using constitutive model for the High Mn alloy with a) 500°C, 8 hr and b) 630°C, 8 hr homogenizations.

Figure 6.6- Steady state flow stress data plotted using constitutive model for the High Mn alloy with a) 500°C, 8 hr and b) 630°C, 8 hr homogenizations.
As seen in Figure 6.7, the materials used in this study experienced little work hardening particularly at ‘x’ values less than 12 (i.e., high temperatures/low strain rates). For the High Mn alloy with a 500°C, 8 hr homogenization [Figure 6.7 a]), compression tests at ‘x’ values of approximately 10 and 11.6 did not experience any work hardening and thus are not shown.

The flow stress describes the resistance of the material to plastic deformation at a given temperature and strain rate. To a first approximation, one would expect a direct relationship between flow and extrusion pressure (under the same deformation conditions). Validation of the relationship between flow stress and extrusion pressure will be discussed in Section 6.4.

**Figure 6.7-** Work hardening data plotted using constitutive model for the High Mn alloy with a) 500°C, 8 hr and b) 630°C, 8 hr homogenizations.
6.3 Effect of Homogenization on Flow Stress

The constitutive model can be used to compare homogenizations over the range of temperatures and strain rates experienced during extrusion. A comparison of the High Mn alloy homogenization treatments is shown in Figure 6.8 (comparisons for the Low Mn and Med Mn alloys are given in Appendix D). The as-cast material exhibits the highest flow stress values of the various conditions except at low values of ‘x’ where the 500°C, 8 hr homogenization is higher. The 500°C, 8 hr homogenization has significantly higher flow stress values than the other homogenized materials except at high ‘x’ values.

Figure 6.8- Constitutive model High Mn alloy homogenization treatments.
The 550°C, 8 hr; 600°C, 1 hr; and 630°C, 8 hr homogenizations have similar flow stress values. The 600°C, 24 hr homogenization has the lowest flow stress values of all the conditions. Assuming that extrusion pressure increases with flow stress, the model can be used to compare relative extrusion pressures for the various homogenization treatments. For example, at a ‘x’ value of 15.5 (for a strain rate of 1/s this corresponds to a temperature of approximately 367°C) the 550°C, 8 hr and 600°C, 24 hr homogenizations have the same stress value; under these conditions cost savings could be achieved (time and energy) by extruding the 550°C, 8 hr homogenization without increasing extrusion pressure.

Stress exponent, \( n \), and pre-exponential constant, \( A \), values further quantify the effect of homogenization on flow stress. An increase in flow stress (for a given slope) is reflected by an increase in the y-intercept \( (A^{-1/n}) \) of the constitutive model. A decrease in slope (or conversely an increase in stress exponent) for a given y-intercept, reflects a decrease in a material’s dependency on temperature and strain rate. Stress exponent values, y-intercept values and qualitative observations of dispersoid quantities are given for the Low, Med, and High Mn alloys in Tables 6.4 to 6.6, respectively. Stress exponents given in the tables are based on three or more compression tests.

Dispersoids were not observed in the Low Mn alloy and the constitutive model gave stress exponents of approximately 7.0 for each condition. The Med Mn alloy gave a stress exponent of approximately 8.0 in cases where no or very few dispersoids were observed (except for the for the 600°C, 24 hr soak; a condition with little Mn estimated to be in solid solution); although no dispersoids were seen in the 600°C, 8 hr
homogenization, dispersoids may be present in the 500°C, 8 hr homogenization due to the lower soak temperature.

Table 6.4- Low Mn, stress exponent values.

<table>
<thead>
<tr>
<th>Homogenization Time (h)</th>
<th>Temp (°C)</th>
<th>$n_{flow}$</th>
<th>$A$ (s$^{-1}$)</th>
<th>Qualitative Observations of Dispersoids</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>7.0</td>
<td>$9.10 \times 10^{32}$</td>
<td>none</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>7.2</td>
<td>$6.28 \times 10^{33}$</td>
<td>very low/none</td>
</tr>
<tr>
<td>24</td>
<td>600</td>
<td>7.0</td>
<td>$3.15 \times 10^{33}$</td>
<td>none</td>
</tr>
</tbody>
</table>

Table 6.5- Med Mn, stress exponent values.

<table>
<thead>
<tr>
<th>Homogenization Time (h)</th>
<th>Temp (°C)</th>
<th>$n_{flow}$</th>
<th>$A$ (s$^{-1}$)</th>
<th>Qualitative Observations of Dispersoids</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>8.0</td>
<td>$8.84 \times 10^{34}$</td>
<td>none</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>9.6</td>
<td>$1.28 \times 10^{40}$</td>
<td>-</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>8.0</td>
<td>$6.07 \times 10^{35}$</td>
<td>low</td>
</tr>
<tr>
<td>24</td>
<td>600</td>
<td>7.3</td>
<td>$1.38 \times 10^{34}$</td>
<td>none</td>
</tr>
</tbody>
</table>

Table 6.6- High Mn, stress exponent values.

<table>
<thead>
<tr>
<th>Homogenization Time (h)</th>
<th>Temp (°C)</th>
<th>$n_{flow}$</th>
<th>$A$ (s$^{-1}$)</th>
<th>Qualitative Observations of Dispersoids</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>8.1</td>
<td>$4.53 \times 10^{34}$</td>
<td>none</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>10.5</td>
<td>$4.87 \times 10^{41}$</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>550</td>
<td>9.7</td>
<td>$1.04 \times 10^{40}$</td>
<td>-</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>8.8</td>
<td>$3.67 \times 10^{37}$</td>
<td>high</td>
</tr>
<tr>
<td>24</td>
<td>600</td>
<td>8.1</td>
<td>$1.06 \times 10^{36}$</td>
<td>none</td>
</tr>
<tr>
<td>8</td>
<td>630</td>
<td>8.0</td>
<td>$3.41 \times 10^{35}$</td>
<td>-</td>
</tr>
</tbody>
</table>
The High Mn alloy did not exhibit any dispersoids in the as-cast and 600°C, 24 hr homogenization conditions and stress exponents were approximately 8.0. The 600°C, 1 hr homogenization showed a high quantity of dispersoids and an increased stress exponent. It can be expected that the 500 and 550°C, 8 hr homogenizations would have a high density of dispersoids as dispersoids can seen in the High Mn 600°C, 8 hr homogenization.

In the model of Kocks and Chen, a stress exponent of 3 was predicted to describe a material that experiences solute controlled plasticity. In practice they determined a value of approximately 3.5 for a non-dilute Al-Mg alloy. In the current study, results suggest that the base stress exponent (no/few dispersoids) changes from 7.0 in dilute alloys (Low Mn) to 8.0 in non-dilute alloys (Med and High Mn) and is independent of the Mn in solid solution for the given alloy. For both the Med and High Mn alloys the presence of dispersoids causes a significant change in the stress exponent with the largest change seen in the High Mn alloy. The presence of dispersoids causes an increase in the stress exponent of that material which may be related to the intervention of alternative deformation mechanisms such as climb of dislocations around dispersoids. The discrepancy between the stress exponents predicted by this study and those predicted by Kocks and Chen will be discussed later in this section.

Med and High Mn as-cast and homogenization conditions with few or no dispersoids resulting in stress exponents of 8.0 are shown in Figure 6.9. The as-cast High Mn alloy has the largest amount of Mn in solid solution and has the highest flow stress values.
Figure 6.9- Constitutive model comparison of High Mn and Med Mn alloys with few or no dispersoids (n=8.0).

The as-cast Med Mn alloy has a large amount of Mn in solid solution gives high flow stress values. The homogenized materials give lower flow stress values. Figure 6.9 shows that a decrease in the amount of Mn in solid solution decreases flow stress values. In the case of the Med Mn 600°C, 1 hr and High Mn 600°C, 24 hr homogenizations either could be substituted to obtain nearly the same flow stress results. Equation 6.1 can be re-written in terms of $\sigma$:

$$\frac{\sigma}{\mu} = \delta^{\frac{1}{n}} \left( \frac{kT}{\mu b^3} \right)^{\frac{1}{n}} \left[ \exp \left( \frac{O_D}{RT} \right) \right]^\frac{1}{n} A^{-\frac{1}{n}}$$  

(6.3)
From Equation 6.3, the y-axis intercept of the flow stress model is given as $A^{1/n}$, providing a means of comparing relative flow stress values for the materials used in this study. The relationship between $A^{1/n}$ and the estimated Mn in solid solution before deformation is given in Figure 6.10 for the High Mn alloy.

![Graph showing the relationship between $A^{1/n}$ and estimated Mn in solid solution](image)

**Figure 6.10- Med and High Mn alloys with few or no dispersoids (n=8.0).**

Where few or no dispersoids are observed (i.e., $n=8.0$) a linear relationship exists between $A^{1/n}$ and the amount of Mn in solid solution and is given by:

$$A^{1/n} = 3.02C_{Mn} + 2.06 \quad (6.4)$$

Where, $C_{Mn}$ is the estimated concentration of Mn in solid solution (wt%) before deformation. Homogenization conditions where dispersoids were observed/expected (i.e.,...
8.8 \leq n \leq 10.5) have different $n$ values and thus cannot be directly compared to the solid solution curves. Experimental measurements of dispersoid number densities and sizes would be needed to further describe the effect of dispersoids on flow stress. As discussed in Chapter II, Li and Arnberg\textsuperscript{9} reported dispersoid diameters of 80 nm to 115 nm during a 7 hour soak period (for AA3003); during soaking spheriodization of the dispersoids was observed.

The physical basis of the flow stress model requires stress exponent values of or close to 3. Stress exponent values that are significantly larger (ie. 7 to 10.5) do not fit with the microstructure basis on which the model was developed, making the model an empirical equation. The stress exponents determined in this study do not satisfy the constraints of the microstructure component of the physically-based flow stress model, suggesting an alternative deformation mechanism(s) to that experienced by the 5182 alloy in the Kocks and Chen study. However, the model does provide a good mathematical representation of the experimental data. The physical basis of the model is an area that requires further analysis. Possibly, in this case it is important to consider other climb based mechanisms, for example climb around the constitutive particles.

### 6.4 Relationship Between Flow Stress and Extrusion Force

To validate the existence of a direct relationship between material flow stress and extrusion force a comparison was made. The mean strain rate experienced during the extrusion trials was calculated using Equation 6.5.
\[ \dot{\varepsilon} = \frac{6v_{\text{ram}} \ln R}{D_C} \approx 4 \text{s}^{-1} \]  

(6.5)

Where, \( v_{\text{ram}} \) is the ram velocity (14 mm/s), \( R \) is the extrusion ratio (130), and \( D_C \) is the billet diameter (100 mm).

Low and High Mn flow stress values were calculated (for a deformation temperature of 400°C and a strain rate of 4/s) using the model and plotted against extrusion force for the 400°C trials. The same was done for Low and High Mn flow stress calculations (for a deformation temperature of 550°C and a strain rate of 4/s) and the 550°C extrusion trials. Flow stress vs. extrusion force is shown in Figure 6.11.

**Figure 6.11** - Low and High Mn alloys. Calculated flow stress vs. extrusion force for 400 and 550°C extrusion trials (strain rate of 4/s).
Flow stress results are plotted as symbols while a trend line has been fit to the data. The trend line is given by:

\[ F_E = m_E \sigma_{\text{flow}} + b_E \]

(6.6)

Where, \( F_E \) is the extrusion force, \( m_E \) is the slope (0.047 MN·MPa\(^{-1}\)), \( \sigma_{\text{flow}} \) is the measured flow stress for the given strain rate, and \( b_E \) is the y-intercept (1.8 MN). The curve does not pass through the origin as would be expected under ideal circumstances and is likely due to complexities of the extrusion process such as overcoming friction and redundant work.

As seen in Figure 6.11, the calculated flow stress data correlates directly to the measured extrusion force during the industrial trials. The relationship between flow stress and extrusion pressure validates the previous assumption that the effect of homogenization on extrusion forces/pressures can be quantified by the effect homogenization has on flow stress values.
Chapter VII – Summary and Conclusions

7.1 Conclusions

Understanding the effect of homogenization practice and alloy chemistry on microstructure and high temperature constitutive behaviour is critical to optimizing the extrusion process. Three alloys of industrial interest: AA3102, an experimental Med Mn alloy, and AA3003 were investigated. An important part of the study was gaining an improved understanding AA3xxx microstructure evolution during homogenization. Homogenization treatments at soak temperatures of 500 to 630°C and soak times of 1 to 24 hours were performed at UBC and the ARDC. Constituent particle morphology and dispersoid precipitation/dissolution were examined using optical microscopy (at the ARDC) and electrical conductivity measurements (at UBC). Optical microscopy provided qualitative information on particle number densities, sizes, and spatial distributions. Electrical conductivity measurements were used to estimate the amount of Mn in solid solution throughout homogenization.

Quantification of the effect of homogenization treatment and alloy Mn content on flow stress required experimental measurements of the materials stress-strain response at deformation temperatures (300 to 600°C) and strain rates (0.1 to 10s\(^{-1}\)) experienced during extrusion; experiments were performed using the Gleeble 3500 thermomechanical simulator located at UBC. Results quantified the relationship between deformation temperature, strain rate, and flow stress. It was found that an increase in alloy Mn content increased the flow stress (at a given temperature/strain rate). A complex relationship between homogenized microstructure and flow stress was observed.
Flow stress results were applied to a physically-based constitutive model. The flow stress data exhibited a good correlation with the constitutive model over the temperature range of interest in this study (400 to 600°C). Breakdown of the model was observed at low temperatures/high strain rates. For a strain rate of 1/s, breakdown occurred between 315 and 350°C. The stress exponent ($n$) and pre-exponential diffusion constant values as a function of $n (A^{-1/n})$ were obtained from the constitutive model and used to further quantify the effect of homogenization on flow stress. In the as-cast state and homogenizations where few or no dispersoids were observed, stress exponents of 7.0 (for the Low Mn alloy) and 8.0 (for the Med and High Mn alloys) were observed. The stress exponent values observed in this study did not fit the microstructure basis of the flow stress model, making the model empirically based.

The expected correlation between flow stress and extrusion pressure was validated with extrusion trial results. Extrusion trials (at extrusion temperatures of 400 and 550°C) were performed using Rio Tinto Alcan’s state-of-the-art research extrusion press at the ARDC.

The main conclusions of this study are as follows:

1. An increase in homogenization soak time causes an increase in constituent particle size and a decrease in number density; this observation was independent of alloy Mn content. A decrease in alloy Mn content caused a decrease in dispersoid number density (at a given soak time) as did an increase in soak time.
2. Yield stress, flow stress, and work hardening results can be accurately described using the physically-based constitutive model at temperatures and strain rates in the industrial extrusion range (400 to 600°C). The effect of homogenization and alloy Mn content on constitutive behaviour was quantified.

3. The starting microstructure of the material prior to hot deformation has a significant impact on the measured flow stress. Microstructures with a large density of dispersoids increase stress exponents and flow stress values, consistent with climb controlled plasticity. In the absence of large dispersoid densities, flow stress values increase linearly with estimated Mn in solid solution; this relationship was seen in the High Mn and Med Mn alloys and appears to be dependent on alloy Mn composition.

4. The effect of homogenization on extrusion pressure can be quantified using flow stress results allowing the constitutive model to be used in optimizing the extrusion process.

7.2 Future Work

Further work can be carried out to determine the cause of the conductivity decrease with soak time observed for the Low Mn alloy. This would require further characterization of the microstructure possibly using TEP measurements and thermodynamic calculations.
Preliminary characterization of the extruded microstructure was performed using conductivity measurements which may have been influenced by the deformed microstructure making it unclear if dispersoid precipitation/dissolution was occurring. Optical microscopy and possibly SEM/TEM work would provide further insight into the deformed microstructure and the possibility of recrystallization.

The effect of dispersoids on flow stress could be studied further by quantifying the effect of dispersoid distribution, number density, and size. Experimental techniques could include TEM and image analysis. The microstructure basis of the physically-based flow stress model could be regained through modification of the model; this would require an in depth study of the deformation mechanisms experienced by AA3xxx during extrusion conditions.

The results of this study could be used in the development of a through process model describing the industrial manufacturing process for extruded AA3xxx alloys.
References


33. Sellars, C.M., and McG. W.J. Tegart, “In theoretical study of relation between resistance and structure in high temperature deformation, new equation is proposed to express relation between deformation rate, stress, and temperature,” Memoires Scientifiques de la Revue de Metallurgie, 1966, 63, pp. 731-746.


APPENDICES

APPENDIX A

A.1 Experimental Difficulties

A.1.1 Homogenization Using Box Furnace

The box furnace was used in conjunction with an Omega™ 2012 controller. Thermal gradients within the box furnace were found to be quite large so a small area of controlled atmosphere was created to provide homogeneous heating of the samples. To do this, a steel tube was placed in the furnace and butted up against the back wall. The diameter of the tube was large enough to allow the furnace control thermocouple inside. A steel plate and thermocouples (the same ones used in the air recirculation furnace) were placed inside the steel tube. A ceramic end cap was fitted to the end of the tube to provide a seal. The box furnace modifications are shown in Figure A.1.

Figure A.1- Box furnace setup. 1) Thermocouples, 2) steel pipe, 3) compression samples, 4) ceramic end cap.
Thermocouple data was recorded using instruNet™ software and an instruNet Model 100 DAQ. After comparing the controller and thermocouple data it was found that the actual temperature in the furnace was 20°C less than the programmed temperature; to correct this, an offset of 20°C was applied to the programmed temperature. Quenching could not be performed quickly as the end cap had to be removed from the steel tube; this problem along with the relatively slow heating rate of the furnace meant that it took a significant amount of time for the furnace to get back up to the specified temperature. The difficulties with the box furnace limited its capabilities so that it could only be used to perform a single heat treatment/quench at a time.

Before the purchase of the recirculating air furnace, the box furnace was used to heat treat 19 (out of 22) of the High Mn resistivity samples and two compression samples (both samples were High Mn alloys heat treated at 600°C with a 1 hr soak).

A.1.2 Gleeble High Force Jaws and Compression Sample Lubrication

High force jaws were used initially in the compression test setup and graphite paper approximately 0.28 mm in thickness was used as the lubricant. Flow stress data that was collected using this setup suggested that work hardening was occurring as the flow stress was seen to steadily increase after the yield point. This was seen in all tests conducted using this setup and was concerning as 3xxx series alloys are non-heat-treatable. Examination of the samples showed a rough contact surface and significant barreling of the compression sample; this suggested that the sample was not being properly lubricated during compression and that the increase in flow stress could be due to frictional effects.
Additional tests were done and it was found that the compression samples were cutting through the graphite disk meaning that some parts of the sample were not lubricated. Recommendations from Gleeble led to the use of a nickel paste (Thred Gard, high temperature 1427°C) as the lubricant which was applied liberally and was able to completely lubricate the contact surface. Changing the setup from using high force jaws to using low force jaws decreased the amount of friction inherent in the mechanical setup. By substantially decreasing the frictional effects the material flow stresses no longer showed ‘work hardening,’ giving a nearly constant slope in the plastic deformation region. The contact surface was noticeably smoother.
APPENDIX B

B.1 Temperature Dependent Parameters

The temperature dependent parameters used in this study were calculated using the methods detailed in this section.

B.1.1 Temperature Dependence of the Shear and Young’s Moduli

The temperature dependence of the shear modulus was calculated using equation B.1. Quantities used in the calculation were obtained from Frost and Ashby. The temperature dependent Young’s modulus can be calculated by multiplying the temperature dependant shear modulus by 2.6 as shown in Equation B.2.

\[ \mu = \mu_0 \left( 1 + \frac{T - 300}{T_m} \frac{T_M}{\mu_0} \frac{d\mu}{dT} \right) \]  \hspace{1cm} \text{(B.1)}

\[ E = 2.6 \mu \]  \hspace{1cm} \text{(B.2)}

Where, \( \mu \) is the temperature dependent shear modulus, \( \mu_0 \) is the shear modulus at 300K (25.40 GPa), \( T \) is the temperature, \( T_m \) is the melting temperature (933 K), \( \frac{T_M}{\mu_0} \frac{d\mu}{dT} \) is the temperature dependence of the modulus (-0.5), and \( E \) is the Young’s modulus. The temperature dependent shear modulus and Young’s modulus values used in this study are given in Table B.1.
Table B.1- Temperature dependent shear and Young’s modulus values.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\mu$ (GPa)</th>
<th>$E$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>21.68</td>
<td>56.37</td>
</tr>
<tr>
<td>400</td>
<td>20.32</td>
<td>52.83</td>
</tr>
<tr>
<td>500</td>
<td>18.96</td>
<td>49.29</td>
</tr>
<tr>
<td>600</td>
<td>17.60</td>
<td>45.76</td>
</tr>
</tbody>
</table>

B.1.2 Temperature Dependence of the Burger’s Vector

Coefficient of thermal expansion (CTE) data for well-annealed 99.99% aluminum was obtained from Hatch.\textsuperscript{14} CTE values at the temperatures of interest were interpolated from the data and are given in Table B.2.

Table B.2- Interpolated coefficients of thermal expansion.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>$\alpha \times 10^{-6} \text{ K}^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>25.9</td>
</tr>
<tr>
<td>400</td>
<td>27.9</td>
</tr>
<tr>
<td>500</td>
<td>30.7</td>
</tr>
<tr>
<td>600</td>
<td>34.3</td>
</tr>
</tbody>
</table>

The temperature dependence of the lattice parameter and Burger’s vector were calculated using the following equations:

$$a = a_0 + \alpha T a_0$$ \hspace{1cm} (B.3)

$$b = \frac{a}{\sqrt{2}}$$ \hspace{1cm} (B.4)
Where, \( a \) is the lattice parameter at the given temperature, \( a_0 \) is the lattice parameter at 25°C (4.0496 x 10^{-10} \text{ m}), a is the CTE at the given temperature, \( T \) is the temperature, \( b \) is the Burger’s vector at the given temperature. The temperature dependent Burger’s vector values used in this study are given in Table B.3.

### Table B.3- Temperature dependent lattice parameters and Burger’s vectors.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>( a \times 10^{-10} \text{ m} )</th>
<th>( b \times 10^{-10} \text{ m} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>4.11</td>
<td>2.91</td>
</tr>
<tr>
<td>400</td>
<td>4.13</td>
<td>2.92</td>
</tr>
<tr>
<td>500</td>
<td>4.15</td>
<td>2.93</td>
</tr>
<tr>
<td>600</td>
<td>4.17</td>
<td>2.95</td>
</tr>
</tbody>
</table>

### B.2 Determination of Yield Stress and Steady State Flow Stress

Stress-strain data was collected from the compression tests. Yield stress and steady state flow stress values were determined from the stress-strain curves by the methods discussed in this section.

Yield stress values were determined using the temperature dependent Young’s modulus and the 0.2% offset method. The 0.2% offset method was applied to the temperature dependent Young’s modulus. The yield stress was found at the point of intersection of the flow stress and Young’s modulus curves. The identification of the yield stress (\( \sigma_y \)) for the High Mn alloy with a 600°C, 24 hour soak homogenization, 400°C deformation temperature, and a strain rate of 0.1/s is shown in Figure B.1.
Figure B.1- Identification of yield stress. High Mn alloy with a 600°C, 24 hr homogenization, 400°C deformation temperature, and 0.1/s strain rate.

The steady state flow stress values (σ_{flow}) provided in the tables are the average flow stress calculated over the range: 0.4 to 0.6 strain; this range of strains was chosen as the flow stress is most stable in this region of the test. Figure B.2 shows the range in which the average stress value was calculated for the High Mn alloy with a 600°C, 24 hour soak homogenization, 400°C deformation temperature, and a strain rate of 0.1/s.
Figure B.2- Identification of steady state flow stress. High Mn alloy with a 600°C, 24 hr homogenization, 400°C deformation temperature, and 0.1/s strain rate.
C.1 Summary of Compression Test and Extrusion Trial Results

Compression test results have been summarized in Tables C.1 to C.6. Low Mn results are given in Tables C.1 and C.2. Med Mn results are given in Tables C.3 and C.4. High Mn results are given in Tables C.5 and C.6. The tables provide electrical conductivity (or EC) measured before and after deformation, the difference, and the percent change (rounded to the nearest percent). Flow and yield stress results are provided in each table. Compression tests in which EC data is unavailable were completed prior to purchasing the Sigmatest EC meter with the 8 mm probe.

Extrusion trial results are summarized in Table C.7. The table lists the alloys extruded and the corresponding homogenization treatments. Results include: extrusion trial deformation temperature, break-through pressure, ram velocity, 3T (laser temperature measurement of the extruded material at the die exit), ram pressure, and liner temperature. Conductivity measurements made before deformation (billet), after deformation (extruded strip), the change, and percent change (rounded to the nearest percent) are included.
<table>
<thead>
<tr>
<th>Homogenization Soak Time (h)</th>
<th>Deformation Temp (°C)</th>
<th>Strain Rate (s⁻¹)</th>
<th>EC_{before} (MS/m)</th>
<th>EC_{after} (MS/m)</th>
<th>ΔEC_{after - EC_{before}} (MS/m)</th>
<th>Change (%)</th>
<th>σ_{flow} (MPa)</th>
<th>σ_y (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>400</td>
<td>0.1</td>
<td>26.5</td>
<td>26.6</td>
<td>0.1</td>
<td>0</td>
<td>38</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>400</td>
<td>10</td>
<td>26.5</td>
<td>26.6</td>
<td>0.1</td>
<td>0</td>
<td>69</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>500</td>
<td>0.1</td>
<td>26.4</td>
<td>26.6</td>
<td>0.2</td>
<td>1</td>
<td>26</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>600</td>
<td>0.1</td>
<td>26.5</td>
<td>27.0</td>
<td>0.5</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>400</td>
<td>0.1</td>
<td>29.3</td>
<td>29.7</td>
<td>0.4</td>
<td>1</td>
<td>30</td>
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<td>8</td>
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<td>10</td>
<td>29.3</td>
<td>29.6</td>
<td>0.3</td>
<td>1</td>
<td>53</td>
</tr>
<tr>
<td>8</td>
<td>550</td>
<td>400</td>
<td>10</td>
<td>29.6</td>
<td>30.0</td>
<td>0.4</td>
<td>1</td>
<td>52</td>
</tr>
</tbody>
</table>
Table C.2- Low Mn compression sample results (continued).

| Homogenization Soak Time (h) | Deformation Temp (°C) | Strain Rate (s⁻¹) | Compression Sample EC before (MS/m) | Experimental Data | Stress | | |
|-----------------------------|-----------------------|-------------------|-------------------------------------|-------------------|--------|---------|
|                             |                       |                   | EC before (MS/m) | EC after (MS/m) | EC after - EC before (MS/m) | Change (%) | σ_{flow} (MPa) | σ_y (MPa) |
| 1                           | 600                   | 400               | 0.1                  | 28.0             | 28.4             | 0.4       | 1          | 32        | 17.5 |
| 1                           | 600                   | 400               | 10                   | 28.1             | 28.5             | 0.4       | 1          | 58        | 22   |
| 1                           | 600                   | 500               | 1                    | 28.2             | 28.3             | 0.1       | 0          | 23        | 14   |
| 1                           | 600                   | 600               | 0.1                  | 28.3             | 28.5             | 0.2       | 1          | 8         | 7    |
| 24                          | 600                   | 400               | 0.1                  | 27.4             | 27.9             | 0.5       | 2          | 31        | 15.5 |
| 24                          | 600                   | 400               | 10                   | 27.2             | 27.7             | 0.5       | 2          | 55        | 23.5 |
| 24                          | 600                   | 500               | 1                    | 27.2             | 27.6             | 0.4       | 1          | 21        | 11   |
| 24                          | 600                   | 600               | 0.1                  | 27.2             | 27.7             | 0.5       | 2          | 8         | 6.5  |
| 8                           | 630                   | 400               | 10                   | 27.0             | 27.3             | 0.3       | 1          | 58        | 14   |
Table C.3- Med Mn compression sample results.

<table>
<thead>
<tr>
<th>Homogenization &amp; Deformation</th>
<th>Experimental Data</th>
<th>Stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soak Temp Time (h)</td>
<td>Temp (°C)</td>
<td>Deformation Temp (°C)</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>400</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>400</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>500</td>
</tr>
<tr>
<td>As-cast</td>
<td>N/A</td>
<td>600</td>
</tr>
<tr>
<td>8</td>
<td>500</td>
<td>400</td>
</tr>
<tr>
<td>8</td>
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<td>8</td>
<td>550</td>
<td>400</td>
</tr>
</tbody>
</table>
Table C.4 - Med Mn compression sample results (continued).

<table>
<thead>
<tr>
<th>Homogenization Soak Time (h)</th>
<th>Deformation Temp (°C)</th>
<th>Deformation Strain Rate (s⁻¹)</th>
<th>Experimental Data</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>EC before (MS/m)</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>400</td>
<td>22.90</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>400</td>
<td>23.00</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>500</td>
<td>22.90</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
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<td>23.90</td>
</tr>
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<td>600</td>
<td>500</td>
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</tr>
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<td>600</td>
<td>600</td>
<td>23.90</td>
</tr>
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<td>630</td>
<td>400</td>
<td>23.00</td>
</tr>
<tr>
<td>8</td>
<td>630</td>
<td>400</td>
<td>23.00</td>
</tr>
</tbody>
</table>
Table C.5. High Mn compression sample results.

<table>
<thead>
<tr>
<th>Experimental Parameters</th>
<th>Homogenization Temp (°C)</th>
<th>Deformation Temp (°C)</th>
<th>Temp Soak Time (h)</th>
<th>Compressive Sample EC before (MS/m)</th>
<th>Compression Sample EC after (MS/m)</th>
<th>EC after - EC before</th>
<th>Change (%)</th>
<th>Stress σ_flow (MPa)</th>
<th>Stress σ_y (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>400</td>
<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>N/A</td>
<td>400</td>
<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>As-cast</td>
<td>500</td>
<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>N/A</td>
<td>600</td>
<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>As-cast</td>
<td>800</td>
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<td>1</td>
<td>22.7</td>
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<td>0</td>
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<tr>
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<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>As-cast</td>
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<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>N/A</td>
<td>1000</td>
<td>16.0</td>
<td>1</td>
<td>22.7</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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</tr>
</tbody>
</table>

Note: The table contains data for different conditions, including homogenization temperature, deformation temperature, temp soak time, EC before, EC after, change in EC, and stress parameters. The data is presented in a tabular format with rows for each condition and columns for the parameters mentioned.
Table C.6- High Mn compression sample results (continued).

<table>
<thead>
<tr>
<th>Homogenization Soak Time (h)</th>
<th>Deformation Temperature (°C)</th>
<th>Deformation Temperature (°C)</th>
<th>Deformation Strain Rate (s⁻¹)</th>
<th>Compression Sample EC Change (%)</th>
<th>Stress σ_flow (MPa)</th>
<th>Stress σ_y (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>600</td>
<td>400</td>
<td>1</td>
<td>EC_{before} 20.0 20.2 0.2 1</td>
<td>52</td>
<td>29</td>
</tr>
<tr>
<td>1</td>
<td>600</td>
<td>400</td>
<td>10</td>
<td>EC_{after} 20.1</td>
<td>64</td>
<td>40</td>
</tr>
<tr>
<td>24</td>
<td>600</td>
<td>400</td>
<td>0.1</td>
<td>EC_{after - EC before} 0.4 2</td>
<td>13</td>
<td>11</td>
</tr>
<tr>
<td>24</td>
<td>600</td>
<td>400</td>
<td>1</td>
<td></td>
<td>17</td>
<td>13</td>
</tr>
<tr>
<td>8</td>
<td>630</td>
<td>300</td>
<td>1</td>
<td></td>
<td>33</td>
<td>21.5</td>
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<td>8</td>
<td>630</td>
<td>400</td>
<td>1</td>
<td></td>
<td>27</td>
<td>17</td>
</tr>
<tr>
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<td>630</td>
<td>500</td>
<td>1</td>
<td></td>
<td>11</td>
<td>9</td>
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<td>600</td>
<td>0.1</td>
<td></td>
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<td>20.5</td>
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<td>600</td>
<td>1</td>
<td></td>
<td>11</td>
<td>10</td>
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</table>
Table C.7- Extrusion trial results.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Homogenization</th>
<th>Extrusion</th>
<th>800mm Ram Position</th>
<th>Extrusion Trial EC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Time (h)</td>
<td>Temp (°C)</td>
<td>Temp (°C)</td>
<td>Break-through</td>
</tr>
<tr>
<td>Low Mn</td>
<td>As-cast N/A</td>
<td>400</td>
<td>400</td>
<td>14.2</td>
</tr>
<tr>
<td>Low Mn</td>
<td>8 500</td>
<td>400</td>
<td>400</td>
<td>12.6</td>
</tr>
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<td>400</td>
<td>400</td>
<td>13.0</td>
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<td>550</td>
<td>8.0</td>
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<tr>
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APPENDIX D

D.1 Constitutive Model Comparison of Low and Med Mn Alloys

The physically-based constitutive model was applied to Low Mn and Med Mn flow stress results as shown in Figures D.1 and D.2, respectively. The constitutive model quantifies the effect of homogenization treatment and alloy Mn content on flow stress.

![Figure D.1](image)

Figure D.1- Constitutive model comparison of Low Mn alloy homogenization treatments.
Figure D.2- Constitutive model comparison of Med Mn alloy homogenization treatments.