Hot Deformation Mechanisms in Mg - x%Al - 1%Zn - y%Mn Alloys

by

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Abstract

Magnesium sheet for automotive applications is very attractive due to its light weight. The poor formability of magnesium and its alloys at room temperature, however, has limited the applications of these alloys. For this reason, at present, magnesium must be formed at elevated temperatures. This study investigates the hot deformation and fracture characteristics of Mg-1wt% Zn alloys containing a range of Al and Mn levels. Hot-rolled specimens were tensile tested over a range of strain rates and temperatures. Strain rate versus flow stress diagrams plotted on log-log scales revealed a transition in deformation mechanisms as a change in slope (the 'stress exponent'). Specifically, non-uniform deformation (i.e. necking) is observed at high strain rates, while uniform deformation is observed at low rates. This transition is accompanied by a change in fracture mechanism from dimpled rupture at high strain rates to cavitation and cavity interlinkage at low strain rates. Specimens which had a stress exponent of ~ 2 and which failed through uniform deformation showing interlinked cavities have been associated with the grain boundary sliding (GBS) deformation mechanism. Specimens which had a stress exponent of ~5 and which failed through necking showing a dimpled fracture surface have been associated with the dislocation creep deformation mechanism. Increasing aluminum appears to somewhat favour the GBS regime as indicated by a slightly decreasing stress exponent. Manganese also appears to favor the GBS regime, since the onset of cavitation appears at higher strain rates compared to alloys without Mn.

Résumé

Le poids léger des tôles de magnésium les rendent très intéressantes pour l'industrie automobile. Par ailleurs, la faible formabilité du magnésium et de ses alliages à température ambiante réduit leurs applications. C'est pour cette raison qu'actuellement, le magnésium n'est mis en forme qu'à de hautes températures. Cette étude porte sur la déformation à chaud et les caractéristiques de ruptures des alliages de Mg-1%m Zn contenant différentes concentrations d'Al et de Mn. Les échantillons laminés à chaud ont été soumis à des tests de tractions en variant le taux de déformation ainsi que la température. Les graphiques des contraintes d'écoulement en fonction du taux de déformation, tracés sur une échelle logarithmique, révèlent une transition dans le mécanisme de déformation sous-forme de changement de pente ('indice de durcissment'). Plus précisément, des déformations non-uniformes (i.e. striction) sont obervées à de hauts taux de déformation tandis que des déformations uniformes sont observées à de bas taux de déformations. Cette transition est accompagnée d'un changement au niveau du mécanisme de rupture allant d'un faciès du type ductile pour de hauts taux de déformation à un faciès de cavités et de cavités interreliées pour des bas taux de déformations. Les échantillons qui ont eu un indice de durcissement d'environ 2 et qui ont rompu après une déformation uniforme démontrant des cavités interreliées ont été associés au mécanisme de déformation par glissement aux joints de grains. D'autre part, les échantillons qui ont eu un indice de durcissement d'environ 5 et qui ont rompu après une striction démontrant un faciès du type ductile ont été associés au mécanisme de déformation par mouvement de dislocations. Augmenter le pourcentage d'aluminium semble favoriser le glissement des joints de grains tel que démontré par une diminution légère de l'indice de durcissement. Le manganèse semble aussi favoriser le glissement des joints de grains, vu que l'apparition des cavités se produit à de plus hauts taux de déformations comparativement aux alliages sans Mn.

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CHAPTER 1. Introduction

Magnesium is the lightest structural metal. Consequently, magnesium has found a niche in the aerospace and transportation industries, which finds its strength-to-weight ratio advantageous. Desirable properties of magnesium alloys also include high damping capacity, good machinability and high recycling potential. There has been a relatively slow growth rate in magnesium utilization due in large part to the limited use of wrought alloys and the limited capacity of the magnesium producers. Applications of wrought magnesium products are very restricted due to its poor formability at room temperature. For this reason, magnesium alloy sheet with improved formability is being investigated.

One of the most popular alloying elements is aluminum, in part because it is also very light. Mg-Al alloys include small additions of zinc or manganese to improve strength and/or corrosion resistance. This Mg-Al system is an attractive choice as it offers the possibility of strengthening via grain refining and solid solution effects.

Much of the alloy development effort has gone into magnesium castings, for the reasons mentioned earlier. Therefore, the influence of alloying elements in wrought form is still an avenue to be investigated. The majority of the research into wrought alloys undertaken has been in the AZ31 type alloys (nominally Mg - 3wt%Al - 1wt%Zn). Due to the focus of research on AZ31, much is still to be understood on the effects and characteristics of aluminum at various levels in wrought form ranging from 1-9wt%Al. Significant alloying elements whose effects in wrought form still remain to be fully understood include zinc and manganese; Zinc being added mainly with aluminum to improve strength and manganese mainly to improve corrosion resistance.

Therefore, the aim of this work is to study the effects that aluminum and manganese have in 'AZ' series magnesium alloy sheet during elevated temperature uniaxial tensile deformation. In so doing, the influences of both temperature and strain rate on tensile behaviour will be investigated and the deformation mechanisms present under various testing conditions will be studied.

Chapter 2 is a literature review describing the basic properties of magnesium and the influences of its more common alloying elements. A description of hot deformation mechanisms will be presented, in which restoration processes and creep behaviour are discussed.

The materials and experimental procedures used are outlined in Chapter 3. The experimental results are presented in Chapter 4. A full discussion on the results presented is in Chapter 5. Finally, the conclusions drawn from this study are outlined in Chapter 6.

CHAPTER 2. Literature Review

2.1. Magnesium Characteristics

Important properties of pure magnesium are listed in **Table 2.1** [1,2]. Magnesium has a hexagonal closed-packed (HCP) crystal structure as seen in **Figure 2.1** [1,2,3]. The lattice parameters for magnesium at 25°C have been determined to be a = 0.32092nm and c = 0.52105nm which gives a c/a ratio of 1.6236 [2]. The a and c change upon thermal expansion and **Figure 2.2** illustrates this effect on both a and c [1]. The axial ratio for magnesium is very close to the ideal ratio for HCP which is 1.633 [4].

Grand Contraction Grand Grand Grand	en eral			
Symbol, Number	Mg, 12			
Chemical series	alkaline earth metals			
Atomic Mass	24.3050 g/m ol			
Crystal structure	hexagonal closed packed			
Physica	l Properties			
Density (20°C)	1.738 g/cm ³			
Density (T _m)	1.584 g/cm ³			
Melting point	650°C			
Boiling point	1090°C			
Mise	ellaneous			
Thermal conductivity	(@27°C) 156 W/mK			
Thermal expansion	(@25°C) 24.8 um/mK			
Young's modulus	45 GPa			
Shear modulus	17 GPa			
Bulk modulus	45 GPa			
Poisson ratio	0.29			

 Table 2.1. Basic Properties of Pure Magnesium [1,2]



Figure 2.1. HCP unit cell.



Figure 2.2. Thermal expansion of a and c lattice spacings [1].

2.2. <u>Alloying</u>

2.2.1. Alloy Identification

Magnesium is alloyed with other metals when it is used for engineering applications [1,5,6]. Each alloy is identified using an ASTM standard system. Key alloying elements have a letter associated with them, **Table 2.2**, and each alloy is normally identified with

letters representing the two main alloying elements. The two letters in the designation are then followed by the rounded weight percentages for each. For example, a typical wrought alloy, AZ31, has 3wt%Al and 1wt%Zn. Additional letters after this designation indicate factors such as the degree of purity. The ASTM also has a code for the temper designations of magnesium and its alloys. **Table 2.3** shows these code letter and description. The temper designation follows the alloy designation.

Abbreviation letter	Alloying element	Abbreviation letter	Alloying element
Α	aluminum	N	nickel
В	bismuth	Р	lead
С	copper	Q	silver
\mathbf{D}^{*}	cadmium	R	chromium
Е	rare earths	S	silicon
F	iron	Т	tin
H	thorium	W	yttrium
K	zirconium	Y	antim ony
L	lithium	Z	zinc
Μ	manganese		

 Table 2.2. Letter designation for Mg alloys [1,5,6]

 Table 2.3. Temper Designation for Magnesium Alloys [1,5,6]

- Code	Description
F	As-Fabricated
0	Annealed, recrystallized (wrought products)
H1	Strain hardened only
H2	Strain hardened and then partially annealed
H3	Strain hardened and then stabilized
T1	Cooled and naturally aged
T2	Annealed (cast products)
T3	Solution heat treated and then cold worked
T4	Solution heat treated
T5	Cooled and artificially aged
T6	Solution heat treated and artificially aged
T7	Solution heat treated and stabilized
T8	Solution heat treated, cold worked and artificially aged
Т9	Solution heat treated, artificially aged and cold worked
T10	Cooled, artificially aged and cold worked

2.2.2. Casting and Wrought Alloys

The worldwide consumption of magnesium in 1999 is illustrated in **Figure 2.3** adapted from Friedrich and Mordike [6]. Casting products have a much larger market share relative to wrought products. Magnesium alloy casting techniques include hot chamber die casting, rheocasting, thixocasting, pore free die casting and injection forming processes [6,7]. Wrought products include extrusion, forging and sheet.

The main casting alloys are the AZ series which contain 8-9wt%Al, small amounts of Zn between 1-3wt%Zn and ~0.5wt%Mn. These have good room temperature properties, but low heat and creep resistance and limited ductility [6]. Other casting alloys based on the AM (Al-Mn) and AS (Al - Si) series offer additional benefits. The AM series which contains between 2-6wt%Al and a small amount ~0.3wt%Mn has improved room temperature ductility due to a lower Al content and elimination of Zn but lowered room temperature flow strength and castability [5,7]. The AS series with aluminum between 2-4wt% and silicon at ~1wt%Si has significantly higher heat and creep resistance but, again, lower castability [5,7]. Some typical casting alloys are identified in **Table 2.4**; important mechanical properties are also highlighted [6].

Property	Unit	AZ91	- AM60	AM50	AM20	AS41	AS21==	AE 42
Ultimate Tensile Strength	MPa	250	240	230	210	240	220	230
Tensile Yield Strength (0.2% offset)	MPa	160	130	125	90	140	1 20	145
Compressive Yield Strength	MPa	148	nm	113	74	nm	106	103
Fracture Elongation (L ₂ = 50mm)	%	7	13	15	20	15	13	11
Elastic Modulus (Tension)	GPa	45	45	45	45	45	45	45

 Table 2.4. Mechanical Properties for Typical Casting Alloys [6]



Figure 2.3. Worldwide magnesium consumption in 1999 adapted from Friedrich and Mordike [6].

Figure 2.3 showed that wrought products account for a small portion of the entire magnesium market [6]. This is largely because magnesium has limited formability, as mentioned previously. The main alloying elements in wrought products are currently aluminum, manganese and zinc. Some typical wrought alloys can be seen in **Table 2.5**, along with some of their mechanical properties [6].

Alloy/Condition	Tensile Strength (MPa)	0.2% - Yield Strength (MPa)	0.2% - Compression Strength (MPa)	Fracture Elongation
Solid Profile				
AZ31B - F	262	193	103	14
AZ61A - F	317	228	131	17
AZ80 - T5	379	276	241	7
HM31A - T5	303	269	172	10
ZK60A - T5	365	303	248	11
Tubes/Hollow Profiles				
AZ31B - F	248	165	83	16
AZ61A - F	283	165	110	14
ZK60A - T5	345	276	200	11
Sheets/Bands				
AZ31B - H24	290	221	179	15
AZ31B - 0	255	152	110	21
HK 31A - H24	262	207	159	9
HK31A - 0	228	200	97	23
HM21A - T8	248	193	145	11

Table 2.5. Mechanical Properties of Some Wrought Alloys [5]

2.2.3. Aluminum Alloying

The most important alloy addition in both cast and wrought alloys is aluminum. Commercial alloys based on the Mg-Al system typically fall in the range of 3-9wt%Al and have good mechanical properties and excellent corrosion resistance [5,6,7]. Aluminum content is decreased for improved hot rolling and elevated temperature formability [6]. The addition of Al improves strength, hardness and castability [1,6,7]. Formation of the brittle Mg₁₇Al₁₂ intermetallic precipitate β phase above ~3wt%Al and gradually increasing in volume fraction lowers strength, and limits ductility and creep resistance.

Higher aluminum content in AZ91 improves castability and allows more complex parts to be cast [5]. Casting alloys with lower aluminum content are chosen more for ductility and impact energy absorption [5]. Aluminum is added in elemental form directly to the melt between 600-750°C [6]. The solubility in magnesium at room temperature is ~2.6wt%Al with a maximum solubility of ~12.7wt%Al at the eutectic temperature of 436°C [1,6]. The decreasing solubility from the eutectic temperature to room temperature makes some Mg-Al alloys amenable to heat treatments and aging [3,5,6,7].

Alloys greater than 6wt%Al can be heat treated to give an optimum balance between strength and ductility [2]. The disadvantages at 6wt%Al additions include: (1) the appearance of the Mg₁₇Al₁₂, (2) susceptibility of stress corrosion cracking (SCC) and (3) the poor hot rollability due to edge cracking [1,5,6]. Mg-Al alloys become more susceptible to SCC with increasing amounts of Al above 3wt%Al [1]. It is for these reasons that lower (<6wt%) Al content alloys have been singled out for use as wrought sheet. **Figure 2.4(a)** presents the variation of mechanical properties for the Mg-Al system. The material labelled 'soft' was the most ductile specimen of hot rolled and annealed material. The annealing condition resulting in the 'softest' material was not specified. The material labelled 'hard' is in the cold rolled state exhibiting the highest yield and ultimate tensile strength [2]. Extrusions and forgings can contain up to 10wt%Al and have a high tensile strength [6]. Hot rolling can be performed up to a maximum of 7wt%Al but an increase in Al causes processing problems. Cold rolling after hot rolling is only possible with lower aluminum levels. Aluminum is an important wrought alloy addition and several systems: the AZ, AE, AM and AS [6].



Figure 2.4. Variation of mechanical properties with composition. (a) with Al, (b) with Zn [2].

2.2.4. Zinc Alloying

Zinc improves the castability and is usually added in its elemental form between 600-750°C [6]. In excess of ~2wt%Zn, and in the presence of Al, a second phase of $Mg_{32}(AlZn)_{49}$ is formed rather than the $Mg_{17}Al_{12}$. In the absence of aluminum, zinc levels may be as high as 7wt%Zn [1,6]. Zinc also improves corrosion resistance as

measured by salt water tests but may increase susceptibility to SCC [1,6]. There may also be an increase in microporosity and hot cracking with a decrease in weldability [3,8].

Zinc is commonly alloyed with aluminum (AZ series) and zirconium (ZK series) [6]. Precipitation strengthened alloys, in particular, have good strength at room temperature [1,6]. Zinc improves strength especially greater than 3wt%Zn, however, also leads to lowered ductility [6]. Figure 2.4(b) presents the variation of mechanical properties for the Mg-Zn system. Again, the material labelled 'soft' was the most ductile specimen of hot rolled and annealed material and the material labelled 'hard' is in the cold rolled state exhibiting the highest yield and ultimate tensile strength [2].

2.2.5. Manganese Alloying

The solubility of manganese in magnesium is relatively limited (about 0.4wt% in the binary). The maximum amount in commercial alloys is about 2.5wt%Mn where MgMn2 is counted as an alloy for general use with medium strength values [1,6]. Manganese, however, is usually added at much lower levels, typically in the range of 0.2-0.4wt%Mn, in conjunction with other major alloying additions, notably in the AM and AZ systems [6,9]. Manganese is difficult to add to Mg due to its high melting point and is therefore usually added as a master alloy [6]. Mn precipitates as the pure element if aluminum is absent. If aluminum is present, a Mn-Al compound is formed if the Mn content is >0.3wt%Mn.

Manganese improves corrosion resistance of Mg alloys. It combines with iron impurities and precipitates out, collecting at the bottom of the melt [6,9]. Manganese also grain refines and improves weldability. Manganese has little effect on tensile strength but it does increase yield strength slightly [6,10]. Manganese is used if moderate strength is desired as it only slightly increases strength after rolled or extruded [6]. Some Mncontaining alloys have good creep resistance.

2.2.6. Lithium Alloying

Lithium is highly reactive but reduces the density of magnesium [6]. It also has a relatively high solubility of 5.5wt%Li at room temperature. Above this, a body centered cubic (BCC) phase of Mg is formed, which is much more amenable to room temperature deformation. However, this is accompanied by decreases in strength and corrosion resistance [2,6].

2.2.7. Rare Earths Alloying

Rare earths are usually added to magnesium as a Mischmetal, a combination of rare earth elements such as cerium, lanthanum, neodymium and praseodymium [1,6,11]. Rare earth elements improve castability, high temperature strength and creep resistance by precipitation hardening. Al₄RE, Al₁₁RE₃ and AlMnRE intermetallics reduce the volume fraction of the brittle Mg₁₇Al₁₂ [6,12-14]. ZE10 alloy shows medium strength with high elongation in the worked condition. It is therefore suited for plate and sheet [6].

2.2.8. Impurities

Copper, iron and nickel all decrease corrosion resistance and must be kept to a minimum. Although copper in amounts greater than 0.05wt% decreases corrosion resistance, it also improves high temperature strength [1,6]. Iron and nickel both have an upper limit of 0.005wt%.

2.3. <u>Aluminum-Zinc (AZ) Series</u>

Mg-Al-Zn is the most widely used alloy series used in industry [6,15]. The most important alloys include AZ31, AZ61 and AZ80. AZ31 shows medium strength but is formable and weldable, and has good corrosion resistance. AZ61 and AZ80 have greater

strength, are also weldable but only have good corrosion resistance in a high purity state [6].

The AZ series has limited high temperature creep resistance, and the ductility decreases with an increase in alloying levels [6,15,16]. Aluminum and zinc impart solid solution strengthening and the potential for age hardening [17-19]. The elastic modulus was found to increase then decrease with increasing amounts of Al or Zn from 0 - 8wt%Al in solid solution due to a combination of solute in solution, volume fraction of precipitates and porosity [20]. Zinc may cause microporosity and alloys with greater than 2wt%Zn are susceptible to hot cracking caused by more zinc in solution and an increase in the volume fraction of the Mg₁₇Al₁₂ and Mg₁₇(Al,Zn)₁₂ phase [21,22]. In AZ31 extruded billets, an optimum manganese range has been found to be 0.2-0.4wt%Mn. This avoids surface cracks attributed to the large intermetallics, but still takes advantage of the slight improvements in mechanical properties due to grain refining [23].

2.4. Deformation

The principal planes that contribute to the deformation of magnesium and its alloys can be seen in **Figure 2.5** [1,24,25]. Plastic deformation in magnesium occurs by dislocation glide and twinning and the mechanical properties are influenced by the interaction of these two mechanisms [4,6,26]. Restriction of dislocation glide through crystal defects such as solid solution, precipitates, dispersoids and grain boundaries strengthen the material. These strengthening mechanisms depend on the size, distribution and volume fraction of solid solution elements, precipitates and dispersoids.

2.4.1. Slip Systems

In magnesium at room temperature, glide of dislocations mainly occurs on the {0001} basal planes. Magnesium therefore lacks five independent slip systems which are required for homogeneous plastic deformation [6,27]. The lack of slip systems at low

temperatures causes magnesium to have a high strength, limited cold workability and directionality of properties, all of which contribute to early failure. As the basal slip system is insufficient for homogenous deformation, twinning is important at low temperatures [6]. Non-basal slip systems in magnesium include first order prismatic slip and second order pyramidal slip. The activities of these slip systems depends on test temperature, alloying additions and texture, but it is generally accepted that these systems are very difficult to activate at low temperatures.



Figure 2.5. HCP structure and the principal planes involved in magnesium deformation [1,24,25].

The critical resolved shear stress (CRSS) is the shear stress required to generate slip in a specific slip plane. The temperature dependence of CRSS in single crystals for important slip and twinning systems is indicated in **Figure 2.6** [28]. An important transition temperature occurs between 200°C and 250°C. It is in this range that the CRSS values for both first order prismatic and second order pyramidal systems decreases dramatically. This resulting increase in slip system activity is usually accepted as being the reason why formability of magnesium increases at higher temperatures.

Alloying additions may affect the balance of slip systems that contribute to deformation [6]. The basal CRSS increases as aluminum is added up to 8wt%. The CRSS for prismatic slip, however, decreases with increased aluminum content [2,19]. Enhanced prismatic slip caused by Al additions decreases prismatic CRSS from ~10MPa to ~2MPa and improves the room temperature ductility compared to pure magnesium [19]. The increased CRSS of the basal slip system controls the solid solution strengthening of Mg-Al solid solutions [19]. A study on Mg alloys with both Al and Zn additions concluded that prismatic CRSS decreases continuously with the addition of solute at low temperatures, however, at elevated temperatures, ~150°C, the *prismatic* CRSS increases at low (~0.05at%) solute concentrations followed by a decrease at higher (between 0.2 - 0.5at%) solute levels [17]. This has been attributed to an increase in the athermal component and a decrease in the thermally activated component of the flow stress with alloying [17].



Figure 2.6. Temperature dependence of the CRSS in single crystals for each slip system and twinning [28].

The exact effects of solid solution, precipitate and dispersoid strengthening is complicated due to several factors. These include the offsetting effects of the strengthening of the basal plane and softening of the prismatic plane and the grain refining aspect of Al which inevitably results in two strengthening mechanisms, the Hall-Petch effect and solid solution [19].

2.4.2. Twinning

In HCP material deformation begins by slip but a lack of slip systems causes twinning to become an important deformation mode [26]. Deformation twinning is a process in which a region of a crystal undergoes shear producing the original crystal structure in a new orientation. The structural rearrangement produces a mirror image reflected along a crystallographic plane. Twinning in HCP materials occurs by the glide of steps, twinning dislocations, over a twin boundary plane [4].

In magnesium, twinning at room temperature normally occurs on the pyramidal $\{10\overline{1}2\}$ and allows for c extension. **Figure 2.6** indicated that twinning CRSS does not vary significantly with temperature [28]. Twinning depends on factors including texture, the direction of the applied stresses, deformation conditions and grain size [6]. The $\{10\overline{1}2\}$ twinning in compression occurs with stresses parallel to the basal planes and, in tension, with stresses perpendicular to the basal planes [3,16]. Double twinning may occur under compression. This type of double twinning is initiated on the $\{10\overline{1}1\}$ planes followed by twinning on the $\{10\overline{1}2\}$ planes. In the initial stages of deformation, twinning reorients the basal planes into possibly more favourable slip positions, but the boundaries are also barriers, preventing dislocation motion [3,16].

Twinning is more prominent at lower temperatures due to the lack of slip systems. Other slip systems become active with increased temperatures and the prominence of twinning is reduced [28,29]. There is also a transition in AZ31 from twinning dominated deformation at large grain sizes to slip with small grain sizes, **Figure 2.7** [28,29]. An

AZ91 with a fine grain size of $\sim 1\mu$ m deforms without the occurrence of twinning [30]. In the absence of twinning, alternative deformation modes may be activated and texture development could be considerably different than in usual Mg alloy material [30].



Figure 2.7. The Hall-Petch effect in addition to the effect of grain size on deformation [28,29].

2.4.3. Texture

Preferred crystallographic orientation within a polycrystalline material is known as texture. The rolling texture is influenced by the balance of the deformation mechanisms that contribute to deformation. The basal texture developed in Mg during thermomechanical forming occurs when the basal planes align perpendicular to the applied stress. In rolling AZ31 for example, the basal planes rotate and align parallel to the rolling direction [2,3,6]. Texture affects the yield stress of magnesium alloys in several ways. It affects both the resolved shear stress on a slip plane and twinning. This leads to anisotropic mechanical behaviour in magnesium [31-36].

Barnett *et al.* found a strong dependence of texture and deformation mode on flow stress at low strains and temperatures [32]. Twinning readily occurs with compressive stresses

acting parallel to the basal plane, therefore, wrought products show lower longitudinal proof stress in compression than in tension [3]. Yield stress in the transverse direction is also higher than that observed in the rolling direction due to the spread of basal planes in typical textures [34]. Anisotropy of mechanical properties (yield and ultimate tensile strengths) is reduced by heat treatments for 30 minutes and increasing temperatures from 150°C to 300°C. This reduction in anisotropy is associated to recovery processes in the stressed regions and an easier activity of dislocation glide and especially twinning [34]. Equal channel angular extrusion (ECAE) may develop a different texture from the basal texture observed in extruded AZ31 [37,38]. The ECAE samples show increased ductility attributable to the difference in texture, **Figure 2.8** [37].

Both normally and asymmetrically hot rolled AZ31 magnesium alloy sheets showed a basal texture intensity gradient decreasing from the upper surface to the lower surface [35,39]. Annealing at 300°C for 1 hour resulted in a reduction in the intensity of the basal texture through the thickness possibly through discontinuous recrystallization and grain refinement [35,39]. Large strain hot rolling of extruded AZ61 sheet showed grain refinement which appeared to correlate to a significant decrease in initial basal texture after the first pass, but in subsequent passes the basal texture intensity increased [31].



Figure 2.8. The effect of ECAE processing to normally extruded material on flow curves. The difference has been attributed to texture [37].

2.4.4. Deformation Heterogeneities

Deformation, transition and shear bands are illustrated in Figure 2.9 [26]. During plastic deformation individual grains accumulate dislocations (Figure 2.9(a)) eventually forming dislocation boundaries (Figure 2.9(b)). Regions of different orientations (Figure 2.9(c)) are formed within individual grains. These are termed deformation bands and deform on different slip systems producing different textures. The narrow regions between deformation bands are termed transition bands. Figure 2.9(d) shows a typical shear band inclined to the rolling direction passing through several grains. They are the result of plastic instability.



Figure 2.9. Schematic illustrating various features in the microstructure when a metal is deforming by slip. The features are shown at an increasing scale [26].

2.5. Recovery and Recrystallization

2.5.1. Background

Both recovery and recrystallization are thermally activated processes by which solid state diffusion removes or rearranges defects and dislocations to a lower energy state [26]. Recovery is the annihilation and rearrangement of dislocations but only involves partial restoration of properties as the dislocation structure is not completely removed. Recrystallization is a restoration process whereby new dislocation-free grains form within the deformed structure and grow to consume the old grains. These restoration processes are schematically illustrated in **Figure 2.10** adapted from Humphreys [26]. During hot deformation, recovery and recrystallization are termed 'dynamic'. This distinguishes them from the 'static' processes which may occur in the hot microstructure after deformation, or during post deformation annealing [26].



Figure 2.10. Schematic diagram of the restoration processes; (a) Deformed material, (b) Recovered, (d) Partially recrystallized and (d) Fully recrystallized.

Grain refinement through recrystallization is another important strengthening mechanism well established through the Hall-Petch equation, **Equation 2.1** [6].

$$\sigma = \sigma_{\rm o} + {\rm kd}^{-1/2}$$
 2.1

Generally, yield stress (σ) increases as grain size (d) decreases where σ_0 is a constant and k is the stress intensity factor for plastic yielding depending on temperature, texture and composition [4,6,26,27]. This assumes that the deformation mechanisms are not altered (e.g. **Figure 2.7**). Additionally, ductility is increased with decreased grain size as seen in **Figure 2.11** for pure Mg [3,6].



Figure 2.11. The effect of grain size on the ductility [3,6].

2.5.2. Dynamic Recrystallization

During the initial stages of deformation, dislocations begin to accumulate. An increased rate of dynamic recovery results and a dynamic equilibrium/steady state flow stress is attained where dislocation generation is equal to dislocation annihilation [26]. If dynamic recovery is limited, dynamic recrystallization may commence. Dynamic recrystallization occurs when new grains originate at old grain boundaries, twins and/or

deformation bands [26]. Nucleation may occur at twins depending on temperature and strain rate [26].

Continued deformation and the growth of new grains reduce the driving force for growth, and the recrystallized grains eventually stop growing [26]. A schematic diagram adapted from Humphreys of the discontinuous dynamic recrystallization phenomenon occurring at grain boundaries is illustrated in **Figure 2.12** [26]. The evolution of new grains at grain boundaries is termed a 'necklaced' structure. In discontinuous dynamic recrystallization, there is a clear distinction between nucleation and growth stages.



Figure 2.12. The development of the 'necklaced' microstructure during discontinuous dynamic recrystallization adapted from Humphreys [26].

In continuous dynamic recrystallization, there is no distinction between nucleation and growth of recrystallized grains [26]. Evidence of continuous dynamic recrystallization process in magnesium alloys was reported by Ion *et al.* [40]. The 'rotation' recrystallization mechanism involves the progressive rotation of subgrains adjacent to pre-existing grain boundaries due to the lack of slip systems. Dynamic recovery causes a rearrangement of dislocations and there is subgrain formation. These subgrains eventually evolve into new grains [26,40]. **Figure 2.13** is a schematic diagram of this type of rotational continuous dynamic recrystallization [40]. In this progressive process, there is no clear distinction between nucleation and growth stages. Non-basal slip is easier at higher temperatures causing deformation to become more homogeneous so

deformation is not concentrated in boundary regions and dynamic recrystallization becomes discontinuous [26,41].

Dynamic recrystallization in AZ31 appears to initiate at ~250°C with larger recrystallized grains as the temperature rises and strain rate declines from 1.0 to 0.01s⁻¹ [10,42-49]. No significant work hardening was observed in AZ31 material above 250°C where the flow stress was nearly constant indicating a dynamic balance between hardening and recrystallization processes [36]. Kaibyshev and Sitdikov, however, also found dynamic recrystallization in room temperature deformation under heavy loading for pure magnesium [50,51]. In AZ61 hot compression deformation, dynamic recrystallization occurred partially at ~300°C with a combination of primary grains and recrystallized grains [52]. Dynamic recrystallization in AZ61 at higher temperatures ~400°C went to completion where primary grains were completely replaced by recrystallized grains [52]. The recrystallized grains are much finer at the lower temperatures than the higher temperatures [52]. It appears aluminum delays the onset of dynamic recrystallization as it occurs at ever increasing temperatures with increasing Al content.



Figure 2.13. Schematic of proposed mechanism for rotational continuous dynamic recrystallization. (a) Shearing near grain boundaries. (b) Dynamic recovery. (c) Formation of new subgrains or grains [40].

2.6. Hot Rolling

Magnesium alloys are typically rolled within the temperature range $300 - 480^{\circ}$ C [6,31]. **Table 2.6** presents a typical rolling schedule for AZ31 [2]. In the current processing route, direct chilled slabs are preheated to temperatures up to 500° C, and the high aluminum alloys such as AZ61 require 'homogenization' to dissolve the Mg₁₇Al₁₂ second phase [2,6].

Initial breakdown of the large grain cast structure is quite efficient and results in a relatively fine microstructure of 20µm or less [6]. During breakdown of the cast slab, however, the low volumetric heat content of Mg causes the slab temperature to drop relatively quickly and may lead to cracking [3]. Intermediate reheat steps are therefore required. Heated rolls may reduce the number of rolling passes [6]. Treatments required following rolling include pickling and side shearing to remove edge cracks [3]. Cold rolling is restricted to 5% reduction per pass to obtain the required mechanical properties, dimensions and surface quality [6].

% Reduction Initial Temp. % Reduction Remarks (0C) per heat per pass Breakdown to 425-450 90-95 10-20 Usually heated rolls 0.25" Hot rolls with lubricant or Sheetrolling 350-440 25-50 5-20 cool rolls without lubricant 250 5 Cold rolls 15-25 Finish rolling

 Table 2.6. Typical Rolling Schedule for AZ31 [3]

The final microstructure can be controlled through thermo-mechanical manipulation of the recovery and recrystallization processes which are sensitive to alloy composition, deformation parameters, reheating cycles, final cooling and additional heat treatments [6]. Various aspects of rolling are yet to be fully understood including texture evolution and dynamic recrystallization.

2.7.1. Background

Superplasticity is the ability of polycrystalline materials to exhibit large elongations in excess of 200% [26,27,53]. In contrast, a normally ductile metal will fail by necking at less than 50% elongation. A fine uniform equiaxed microstructure ($<10\mu$ m), a relatively high deformation temperature ($>0.5T_m$) and low strain rates ($\sim10^{-5} - 10^{-1}s^{-1}$) are three of the requirements for superplasticity to occur [26,53,54]. Commercially, however, it is hard to exploit superplastic forming (SPF) due to its slow strain rates and it has been limited to specialized applications. Higher strain rate superplasticity $>10^{-2}s^{-1}$ has been achieved in Al alloys [54]. High strain rate superplasticity ($>10^{-2}s^{-1}$) has been attributed to a very small grain size [54-56].

Superplasticity exploits the recrystallization and grain growth phenomena observed during thermo-mechanical deformation in that a very small grain size and the inhibition of dynamic grain growth are key requirements for SPF processes [26]. A superplastic alloy during elevated temperature deformation is characterized by a low flow stress, a high strain rate sensitivity, suppression of significant cavity interlinkages and a high resistance to non-uniform deformation, i.e. necking [26,57].

2.7.2. Deformation Mechanisms

Superplasticity occurs in the strain rate and temperature range usually associated with creep. Creep is a plastic deformation process that occurs in solids at high temperatures, $0.5T_m$, and superplasticity can be considered a creep process [53]. Plastic flow during creep is associated with three mechanisms: diffusional creep, grain boundary sliding (GBS) and dislocation creep through glide and climb [26,53,57]. The two main competing mechanisms during high temperature deformation (e.g. superplasticity) involve GBS and dislocation creep [55-70]. Superplasticity is expected when GBS dominates deformation and normal ductility when dislocation creep dominates [53].
These two mechanisms are considered independent therefore the fastest one is the rate controlling one, **Figure 2.14**, adapted from Nieh *et al.* [53]. Although independent, in some cases, one mechanism may be necessary to permit accommodation of another.

Stresses concentrate at grain boundary areas during GBS leading to the nucleation of voids and cavities. This leads to fracture, and is, therefore, a limiting factor to the degree of superplasticity [26,55]. To avoid extensive cavitation and premature fracture, an accommodation process is necessary to accompany GBS. For example, dislocation slip may be an accommodation process for GBS [53]. Additional accommodation processes include grain boundary migration, recrystallization or diffusional flow [53].

The onset of dynamic recrystallization may lead to changes in deformation mechanisms [26]. If dynamic recrystallization leads to very small grains, deformation may occur preferentially by GBS [26,71]. This tends to occur where deformation by dislocation glide and climb is particularly difficult such as in Mg which has limited slip systems [26]. Fine grained materials are favoured to deform through GBS [26,53-70]. The formation of filaments on the fracture surfaces are a sign of GBS and could be due to the high grain boundary diffusion rate of magnesium at elevated temperatures [55]. These fibers extend from the fracture surfaces along the tensile direction and bridge surface cavities in this tensile direction [71-73].



Figure 2.14. Schematic of high temperature deformation behaviour of fine grained materials [53].

Accommodation of GBS by diffusional processes is explained through a gradual grain rearrangement by diffusional flow [53]. GBS is accommodated during temporary grain shape changes and grain motion occurs by grain boundary diffusion [53]. GBS accommodated by dislocation slip involves the sequential steps of glide and climb. Grains slide until they reach an unfavourably oriented grain or obstacle at which point dislocations relieve the stress concentration produced from the strained grains [Luke]. Solute drag dislocation creep is the label given the alloys where the dislocation glide mechanism (n = 3) is the rate controlling step in the GBS accommodation process due to solute atoms impeding dislocation motion [53].

A superplastic material will exhibit a high strain rate sensitivity, m, as defined in **Equation 2.3**:

$$\sigma = K \dot{\varepsilon}^{m}$$
 2.3

where σ is flow stress, $-\dot{\epsilon}$ is strain rate and K is a constant [26]. Superplastic material will have an m value in the range of 0.4 – 0.7, and is required for superplastic behaviour to occur [26]. A rearrangement of the above equation results in **Equation 2.4**:

$$\dot{\varepsilon} = K\sigma^n$$
 2.4

where n is the stress exponent equal to 1/m [53]. Each creep mechanism can basically be described by **Equation 2.4** where n can be used to distinguish the deformation mechanism [53,58-64]. A summary of the deformation mechanisms and their associated stress exponents, n, can be seen in **Table 2.7** [53]. The stress exponent, n, is the slope in each of the regions and distinguishes the deformation mechanism seen in **Figure 2.14** [53]. Parameters that enhance GBS and inhibit dislocation slip are the keys to optimizing superplasticity [53].

Deform Mechai	ation usm s	Stress Exponent n
Diffusion	al creep	1
Grain Bound	ary Sliding	
(GB	S)	
	Glide	
Dislocation	controlled	3
Creep	Climb	
	controlled	4 - 5

Table 2.7. The Stress Exponents, n, of the Active Deformation Mechanisms [53]

2.7.3. Superplastic Applications in Magnesium

Grain refinement is regarded as essential if magnesium alloys are to exhibit adequate ductility [54-70]. Generally, grain sizes must be below ~10 μ m to see evidence of superplasticity; however, grain growth has been observed during superplastic deformation of the AZ series alloys [54-56]. Despite this grain growth, superplastic characteristics have been improved in rolled AZ31 and AZ91 through dynamic recrystallization and grain refinement [54,55]. Hot rolled AZ61 material with an average grain size of 25 μ m also exhibited GBS [63]. In AZ91 grain refined through ECAE, superplasticity was observed at high strain rates (1x10⁻¹s⁻¹) and low temperatures (200°C), which has been attributed to a very small grain size (<1 μ m) [54]. Some commercial alloys are presented in **Table 2.8** along with their superplastic properties [53].

Alloy/Condition	Elongation (%)	Temperature (°C)	Strain rate (s ⁻¹)
AZ61A/cold rolled and			
annealed	213	-	3.3X10 ⁴
ZK60A/warm rolled	289	290	1.7X10 ⁻⁴
AZ31B/annealed	319	350	1.7X10 ⁻⁴
ZW3/aged	195	300	1.7X10 ⁴
Mg-12Al/hot rolled	162	360	8.5X10 ⁻⁴

Table 2.8. Superplastic Properties of Some Commercial Mg Alloys [53]

Two deformation regimes were found to be active in AZ31 at intermediate temperatures between $300^{\circ}C - 375^{\circ}C$ and a strain rate range from $3x10^{-5} - 5x10^{-2}s^{-1}$ [61]. Dynamic grain growth took place during the tests resulting in grain sizes ranging from 17 - 40µm [61]. At the higher strain rates, there was a stress exponent of ~6 and, at the lower strain rates, a stress exponent between 1.7 and 2.5 was observed [61]. The former is indicative of dislocation climb and the latter is indicative of GBS grain boundary diffusion [61]. In this low strain rate regime, GBS is initially the dominant mechanism. However, once dynamic grain growth occurs, dislocation slip becomes more important [61]. Again, this emphasizes the balance between the competing mechanisms.

2.8. <u>Summary</u>

Magnesium alloys show great potential as structural materials for the transportation industries due to their low density. Magnesium wrought alloys have improved mechanical properties compared to as-cast components but, due to their hexagonal structure and limited activity of slip systems at room temperature, have poor formability and exhibit anisotropic mechanical properties. In order to increase the use of magnesium alloys in wrought form, it is desirable to increase its plastic formability at lower temperatures.

It is important to understand the development of the microstructure during rolling, extrusion and forging. Investigation of the effects of alloying on hot working is also an important step in understanding microstructural development. The aims of this current study are to understand the role that both aluminum and manganese have in the deformation of sheet material. In so doing, mechanical properties will be investigated and topics such as superplasticity and deformation will be investigated.

CHAPTER 3. Materials and Methodology

3.1. <u>Materials and Casting Alloys</u>

Melts of 30kg each were prepared in a resistance furnace using a mild steel crucible under gas protection (CO2 + 1%SF6). Commercial wrought magnesium alloy AZ31 ingots from Timminco were melted to 730°C to prepare six compositions of the AZ type alloys with high and low Mn content. The composition of the AZ31 starting material is seen in **Table 3.1**. Degassing was performed with hexachloroethane (C₂Cl₆) tablet followed by purging with argon gas for 10 to 15 minutes. The melt was poured into a standard water cooled copper mould (**Figure 3.1**) to produce plate cast specimens (**Figure 3.2**).

Table 3.1. Chemical compositions of the commercial AZ31 starting material in wt%

AZ31	Al	Zn	Mn	Si	Cu	Fe	Ni
Spectrom etry Analysis	2.75	0.90	0.25	0.005	<0.005	0.0045	0.0007



Figure 3.1. Water cooled plate mould.



Figure 3.2. Plate casts of the six alloys, (a) standard Mn level alloys and (b) low Mn level alloys.

Six AZ series alloys with three different Al levels and two different levels of Mn were cast. The alloys were produced with the aim being three levels of Al contents (wt%) of 3%, 4% and 6%; one series with a standard level of Mn of approximately 0.25% and the other with a low level of approximately 0.05%Mn. Pure Al was added to AZ31 alloy to produce AZ41 and AZ61 compositions with standard 0.25wt%Mn content. AZ31 alloy was diluted with pure Mg ingots to achieve the 0.05wt% Mn content and additions of pure Al and Zn were made to produce the AZ31, AZ41 and AZ61 compositions with low (0.05wt%) Mn content.

The chemical compositions of the six alloys cast, listed in **Table 3.2**, were determined through spark emission spectrometry. Alloys with a standard level of Mn are labeled with a Mn proceeding AZ31 and this extra 'Mn' designation is absent from those with the low Mn levels. This form of identification is continued throughout the remainder of this thesis.

Alloy	Al	Zn	Mn	Si	Cu	Fe	Ni
AZ31Mn	2.70	0.93	0.250	0.005	0.006	0.0050	0.0007
AZ41Mn	3.70	0.94	0.240	0.006	0.005	0.0040	0.0006
AZ61Mn	5.70	0.95	0.240	0.008	0.006	0.0050	0.0005
- AZ31	2.80	1.00	0.062	0.005	0.005	0.0040	0.0006
AZ41	3.80	1.00	0.060	0.006	0.005	0.0040	0.0007
AZ61	5.85	1.08	0.058	0.007	0.005	0.0048	0.0006

Table 3.2.	Chemical	compositions	of the	experimental	allovs i	in wt%
Laure J.Z.	Chemicai	compositions	or une	capermicinai	anoysi	111 VV L /C

3.2. Hot Rolling

All cast alloys were rolled with the same rolling parameters. This was conducted at CANMET using a STANAT rolling mill. The rolling schedule consisted of seven passes of rolling, with each pass applying a 25% reduction in thickness. The initial thickness of the plate was ~12.7mm and after a surface cleaning procedure it was ~10mm. The final thickness ranged between 1.85mm - 1.97mm. The rolling speed was approximately 50rpm and the rolls with a 76.2mm radius were heated to approximately 150°C. All plate specimens were initially solution treated for 1 hour at 450°C prior to rolling. Between each pass, the specimens were immersed in a heated sand bath for reheating up to 400°C.

3.3. <u>Hot Tensile Testing</u>

The sheet material was machined to the dimensional specifications shown in **Figure 3.3** for an elevated temperature tensile experiment. The directions to be used throughout this thesis are defined as well. Wire EDM machining was used since the 0.5mm radius in the specimen where the gauge and grip sections meet is too tight for conventional machining equipment.



Figure 3.3. Schematic of a typical hot tensile specimen with the directions defined. Dimensions are in mm.

Elevated temperature tensile testing was performed using a model 5568 screw driven Instron with an Instron 3119-008 furnace, which is a resistance furnace with an attached temperature regulating fan. Data were acquired using Bluehill mechanical testing software; data acquisition rates varied depending on the strain rate of the specific test.

The tensile testing set-up can be seen in **Figure 3.4** along with the associated components for a tensile test through the viewing port. Two thermocouples were present during the test to ensure temperature uniformity; one was touching the specimen and lower anvil and the other was in the ambient air within the furnace. The upper anvil moves up during the test while the lower one remains stationary. The test stops automatically once rupture of the specimen occurred.

Three temperatures, 300° C, 400° C and 450° C, and four strain rates, $0.1s^{-1}$, $0.01s^{-1}$, $0.001s^{-1}$ and $0.0003s^{-1}$ were used in tensile testing. Experiments were repeated at 400° C and 450° C to obtain satisfactory elongation data. A typical deformation cycle can be seen in **Figure 3.5**.



Figure 3.4. Photographs showing the elevated tensile test set-up. (a) The set-up in its entirety and (b) the viewing window.



Figure 3.5. A schematic of a typical deformation cycle seen by the specimens.

All the specimens underwent the same procedure for deformation. The specimen was placed into the grips, the temperature was reached within 5 minutes and allowed to soak for 2 minutes prior to deformation. The specimen was then fractured, extracted and quenched in water after rupturing.

3.4. Materials Characterization

3.4.1. Optical

Macroscopic photographs of the fractured specimens were taken for characterization purposes. Microstructural examination was performed on the transverse section in the fractured region of the tensile bars. Specimens were cold mounted using Technovik 4004 mounting material. Following cold mounting, the specimens were ground on 240 grit SiC abrasive paper and polished successively at 9 μ m and 3 μ m for 6 minutes each and finally using a 0.04 μ m colloidal silica suspended in distilled water for 2 minutes. The specimens were etched in a fresh solution of 10ml acetic acid, 4.2g picric acid, 10ml H₂O and 70ml ethanol from 2-5 seconds. Following etching, the sample was immediately washed with denatured alcohol and then distilled water. The microstructures were examined using an Olympus optical microscope with Clemex image analysis software.

3.4.2. Scanning Electron Microscope (SEM)

The fracture surfaces were examined using a Carl Zeiss EVO 50 model SEM. Secondary electron images of the fractured surfaces were taken. Surfaces were prepared by cutting the fractured specimen down to a length adequate for SEM analysis. An ethanol solution was used to clean the samples and remove any debris or lubricant present. The fracture surfaces of AZ31Mn under all deformation conditions were analyzed. The highest and

lowest temperatures and fastest and slowest strain rates for AZ31, AZ61Mn and AZ61 fracture surfaces were also examined.

A working distance of 20.0mm was employed with an accelerating voltage of 15kV. Some of the samples suffered from surface charging and, in those cases, the accelerating voltage was reduced to 10kV to obtain a clearer picture.

CHAPTER 4. Results

4.1. Rolled Sheet

4.1.1. Sheet Appearance

Macroscopic inspection of the rolled sheet reveals edge cracking in the 6wt% Al alloys, especially in the low Mn alloy, **Figure 4.1**. **Figure 4.2** contains individual pictures of these 6wt%Al alloys highlighting the effect Mn has on the rolled sheet. In the Mn alloys, the edge cracking is less severe. There is no difference in the surface appearance of the alloys.



Figure 4.1. Photographs showing the rolled sheet material, (a) AZ series with a standard level of Mn and (b) AZ series with a low level of Mn.



Figure 4.2. Edge cracking in both 6wt% Al alloys; (a) 0.25wt% Mn and (b) 0.05wt% Mn. Cracks are especially evident in the low Mn alloys.

4.1.2. Cast Plate

Microstructures of both the as-cast and solution treated (450° C, 60 minutes) material are shown in **Figure 4.3** to examine the effects of composition and solution treatment on microstructure. There is a progressive increase in the volume fraction of second phases with an increase in aluminum content in the as-cast specimens, **Figure 4.3(a, b, c & d)**. **Figure 4.3(a)**, AZ31Mn, is visually estimated to contain <5% of the second phase in the microstructure. The two 'AZ61' alloys, **Figure 4.3(c & d)**, show the visually estimated volume fraction to be >25%. Heat treatment seems to have virtually eliminated the second phase in the samples. However, in the 6wt% alloys, 'coring' is clearly observed.

The grain size results using the linear intercept method are shown in **Table 4.1** for the cast and heat treated material at 450° C for various times. There is no grain refinement with increased aluminum and no grain growth with increasing treating time. The 60 minutes heat treatment is highlighted because it is the condition seen prior to hot rolling, **Figure 4.3(e, f, g & h)**. Grain sizing in several of the 'AZ61' alloys was hindered by the presence of casting features obscuring the grain boundaries, **Figure 4.3(g & h)**.



Figure 4.3. Optical micrographs of AZ31Mn, AZ41Mn, AZ61Mn and AZ61 comparing the as-cast (a, b, c & d) and heat-treated (d, e, f & g) plate samples. There is dissolution of the second phases during heat treatment.

Minutes	AZ31Mn	AZ31	AZ41Mn	AZ41	AZ61Mn	AZ61
Cast	72.3	102.7	98.6	102.9	n/m	n/m
15	63.3	134.6	105.4	87.4	n/m	n/m
60	75.1	81.7	93.8	142.5	n/m	n/m
120	67.9	135.5	94.6	137.9	n/m	n/m
300	74.7	108.2	98.7	163.5	122.38	n/m
600	78.5	119.0	83.1	142.6	131.3	167.5

Table 4.1. Grain Size (Microns) Results for the Cast Plate and Heat Treated at $450^{\circ}\mathrm{C}$

4.1.3. Rolled Microstructure

Table 4.2 show the grain sizes for the rolled sheet and heat treated at 450°C for various times. The as-rolled grain sizes for the six alloys are highlighted. The as-rolled sheet microstructures are presented in **Figure 4.4** in the plane normal to the sheet surface. The microstructures for the six alloys reveal fine grains encompassing >95% of the microstructure. A few large grains in the order of ~100µm are also visible in **Figure 4.4(a, b & c)**. Etching reveals the incomplete breakdown of the cast structure in the 'AZ61' alloys, **Figure 4.4(c & f)**, as shown by the dark regions (arrowed in red) which are probably segregated regions. These were elongated in the rolling direction. **Figure 4.5** for the transverse section of the sheet reveals similar results to the normal direction. Large ~100µm grains are dispersed through the microstructure surrounded by a matrix of ~5µm fine grains, **Figure 4.5(a & b)**. Etching, again, reveals remnants of the cast structure (dark regions) for 'AZ61' alloys, **Figure 4.5(c & f)**, aligned with the rolling direction. Shear banding is also evident in several of the microstructures (arrowed in blue).

Table 4.2. Grain Size Results for the Rolled Sneet and Heat Treated at 450 C	Table 4.2. Grain	Size Results for	• the Rolled Sheet and	Heat Treated at 450°C
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Minutes	AZ31Mn	AZ31	AZ41Mn	AZ41	AZ61Mn	AZ61
Rolled	4.0	5.2	5.1	5,4	5.4	6.3
15	9.0	8.8	6.0	9.1	9.8	12.2
60	10.0	12.9	9.8	12.0	11.5	18.4
120	8.8	18.4	11.9	18.6	18.1	11.3



Figure 4.4. The as-rolled optical microstructures in the normal direction for the six alloys investigated taken at 200X magnification. (a, b, c) Mn alloys and (d, e, f) Low Mn alloys.



Figure 4.5. The as-rolled optical microstructures in the transverse section for the six alloys investigated taken at 200X magnification. (a, b, c) Mn alloys and (d, e, f) Low Mn alloys.

4.1.4. Pre-Tensile Testing Microstructure

Heat treatments on the as-rolled samples were carried out to determine the microstructure of the alloys immediately prior to elevated temperature tension testing. A heat treatment at 450° C for five minutes in the furnace was performed to simulate the five minute heating period prior to deformation. Figure 4.6 presents the hot-rolled and heat treated microstructures of the six alloys in the transverse section. The volume fraction of the second phase is optically negligible for the six alloys. The grain sizes at various annealing times are listed in Table 4.2. The 15 minutes heating time is highlighted as it is the grain size measured nearest to the time of the heating cycle in the furnace prior to deformation, and seems to lead to grain size increase for all alloys. The etching effects reveal the remnants of the cast microstructure are not as prominent in the 'AZ61' alloys after heat treatment, Figure 4.6(c & f). Shear banding is again evident in the microstructures (arrowed in blue).

4.2. Mechanical Testing

4.2.1. Flow Curves

Figure 4.7 is a typical stress-strain plot of the sheet samples pulled in tension. **Figure 4.7** is the 'AZ31' alloys at the three temperatures and the strain rates of $0.1s^{-1}$ and $0.0003s^{-1}$. The effect of temperature and strain rate yields three distinct flow curve types for all alloys. At low temperatures and high strain rates, the material exhibits very high rates of work hardening at low strains followed by very small uniform strain regions, i.e. stress peak and low ductility. At high temperatures and low strain rates, the material exhibits a low flow stress, gradual work hardening, a very long uniform strain region and a very short post uniform region.



Figure 4.6. The optical microstructures in the transverse section for the six alloys heat treated at 450° C for 5 minutes taken at 200X magnification. (a, b, c) Mn alloys and (d, e, f) Low Mn alloys.



Figure 4.7. The true stress versus true strain curves for 'AZ31' alloys tested under various conditions. For the low Mn alloy, only the two extreme conditions of 300°C, 0.1s⁻¹ and 450°C, 0.0003s⁻¹ are presented.

4.2.2. Effect of Temperature

Only one specimen per alloy and tensile test condition was used to generate the flow curves and the results are separated and graphed in groups, **Figures 4.8 – 4.11**, for comparison purposes: (1) temperature, (2) strain rate and (3) aluminum and manganese. These graphs are typical examples of flow behaviour for all alloys. The first comparison highlights the effects of temperature on the flow behaviour. **Figure 4.8** shows the effect of temperature in the 'AZ31' alloys at a strain rate of $0.1s^{-1}$. At the low temperature of 300° C, there is a stress peak at ~100MPa. The strain after the ultimate tensile strength (UTS) is relatively short and rupture occurs at ~0.5 strain. At the high temperature of 450° C, there is a very broad stress peak at ~30MPa with gradual work hardening and a long post UTS region. Rupture occurs at ~0.7 strain.



Figure 4.8. The true stress versus true strain curves 'AZ31' tested at 0.1s⁻¹ and the three temperatures indicated.

4.2.3. Effect of Strain Rate

The second set of results, **Figure 4.9**, highlights the general strain rate effect at 450° C, using as an example the 'AZ41' alloys. The $0.1s^{-1}$ high strain rate curve shows little work hardening, a stress peak at ~35MPa and rupture at ~0.7 strain. The $0.0003s^{-1}$ low strain rate curve shows yielding at ~5MPa, gradual work hardening to a stress peak of ~10MPa with a very long uniform strain region and very short post uniform strain region. Other differences going from a high strain rate to a low strain rate are the smoothness of the curves at a strain rate of $0.1s^{-1}$ and the oscillating nature of the curves at a strain rate of $0.0003s^{-1}$.



Figure 4.9. The true stress versus true strain curves for 'AZ41' tested at 450°C and the four strain rates indicated.

4.2.4. Effects of Aluminum and Manganese

The effects of Al and Mn on the tensile deformation behaviour of the alloys at 450° C and $0.1s^{-1}$ and $0.0003s^{-1}$, respectively, are shown in **Figures 4.10 - 4.11**. **Figure 4.10** reveals an increase in strength with an increase in aluminum content from ~33MPa in the 3wt%Al alloy to ~37MPa in the 6wt%Al alloys. All six alloys, especially the 4wt% and 6wt%Al, have a sharp peak initially in their flow curves with a similar 'yield point'. **Figure 4.11** shows no apparent effect of Al on the flow curves at the low strain rate. Manganese, however, shows a distinct separation in the curves for the two Mn levels at the low strain rate. The low Mn alloys all show a high rate of work hardening and their yield stresses are reached more rapidly than the standard Mn alloys.

At the high strain rates and low temperatures, **Figures 4.8 & 4.9**, there is generally an increase in flow strengths between of 1 - 5MPa with Mn additions. At the low strain rates and high temperatures, **Figures 4.9**, the Mn alloys generally show increased strain at failure from 1.2 strain in the low Mn alloy to 1.5 strain in the standard Mn alloy. The effects of both aluminum and manganese content on yield stress are summarized in **Figure 4.12 – 4.13**. Both figures show the effect of aluminum content on yield stress for all six alloys at each temperature: **Figure 4.12** at a strain rate of $0.1s^{-1}$ shows a slight increase with Al and **Figure 4.13** at a strain rate of $0.0003s^{-1}$ shows an increase for the low Mn alloys.



Figure 4.10. The true stress versus true strain curves for the six alloys tested at 450° C and $0.1s^{-1}$.



Figure 4.11. The true stress versus true strain curves for the six alloys tested at 450°C and 0.0003s⁻¹.



Figure 4.12. The effect of aluminum on yield stress deformed at $0.1s^{-1}$ and the three temperatures indicated.



Figure 4.13. The effect of aluminum on yield stress deformed at 0.0003s⁻¹ and the three temperatures indicated.

4.2.5. Elongation Data

The elongation data were determined from four tensile tests to obtain statistically improved data. The elongation at 450°C for each strain rate as a function of aluminum content is shown in **Figure 4.14**. Ductility increases with decreasing strain rate. Ductility also increases from 3wt% to 4wt%Al for both low and standard Mn alloys. Above 4wt%Al, the alloys with Mn level off or show a slight decrease in ductility going from 4wt% to 6wt%Al. The low Mn alloys show a downward trend in ductility above 4wt%Al.

Ductility data are plotted as a function of strain rate, **Figures 4.15**, for each of the six alloys at each strain rate. The trends indicate that three of the alloys AZ41, AZ41Mn and AZ61Mn generally show better elongation at each strain rate as compared to the other three alloys, AZ31, AZ31Mn and AZ61.



Figure 4.14. The effect of aluminum on percent elongation for the six alloys tested at 450°C for each strain rate.



Figure 4.15. The effect of strain rate on percent elongation for the six alloys tested at 450°C.

4.3. Deformation Mechanisms

4.3.1. Macroscopic Photographs

Figure 4.16 presents photographs of all six alloys pulled to fracture at 400°C with strain rates of 0.1, 0.01, 0.001, and 0.0003s⁻¹. Two different types of failure occur: (1) localized deformation where the specimen has necked to a point followed by rupture and (2) diffused necking with a large post-uniform strain region which can otherwise be described as uniform deformation of the specimen where the gauge length has deformed uniformly and ruptured. The 'AZ31' alloys both show good examples of the difference between necking and uniform deformation. **Figure 4.16(a & d)** have localized necking in their macroscopic samples at the two high strain rates and diffused necking deformation at the two low strain rates.

4.3.2. Strain Rate – Stress Behaviour

The effect of strain rate on yield stress is plotted logarithmically for each temperature in **Figures 4.17** - **4.18**. A change in the slope of the curves indicates a change in deformation mechanism. The 400° C and 450° C curves all have combinations where a slope (n) of ~5 is apparent at high strain rates and a slope (n) of ~2 at low strain rates. A summary of the slopes is listed in **Table 4.3** for the six alloys at the three temperatures tested.

At 300°C, the materials show no change in behaviour (i.e. one slope of 5) for both the normal and low Mn levels. There is generally no difference between the curves of the different Al levels. One exception may be alloy AZ31Mn at 300°C and the low strain rate of 0.0003s⁻¹, **Figure 4.17**. This deformation curve shows a yield stress somewhat lower than would be extrapolated from the higher strain rate results.



Figure 4.16. Macroscopic photographs of the tensile specimens for each alloy pulled at 400°C for each strain rate. Specimens left to right on each photograph; 0.1s⁻¹, 0.01s⁻¹, 0.001s⁻¹ and 0.0003s⁻¹. (a, b, c) Mn alloys and (d, e, f) Low Mn alloys.

Results



Figure 4.17. Logarithmic plots of strain rate versus yield stress showing the effect of aluminum at the three temperatures tested for the standard Mn level alloys.



Figure 4.18. Logarithmic plots of strain rate versus yield stress showing the effect Mn has on 'AZ61' alloys at the three temperatures tested.

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Figure 4.17 illustrates the effect aluminum has on the standard Mn alloys. At the high strain rates where the slope is 5, there is no effect of Al. In the low slope regime, however, increasing Al seems to further decrease the slopes slightly. **Figure 4.18** presents the 'AZ61' alloys and highlights the effect Mn has on the transition of the deformation mechanisms. The higher Mn alloys tend to exhibit mechanism transitions at higher strain rates.

A There	450°C		40	0°C	300°C	
Alloy	Necking	Cavitation	Necking	Cavitation	Necking	Cavitation
AZ31Mn	7.76	2.22	4.59	1.63	6.73	2.78
AZ41Mn	6.57	1.88	5.49	2.50	5.43	n⁄a
AZ61Mn	5.24	1.95	3.83	1.37	4.79	n/a
AZ31	5.16	1.72	5.16	1.88	6.25	n⁄a
AZ41	4.32	2.01	4.79	2.33	5.81	n/a
AZ61	4.32	1.80	4.67	2.53	4.81	n/a
Average	5.56	1.93	4.75	2.04	5.64	-

Table 4.3. Tables of Slopes for Strain Rate versus Yield Stress for the ThreeTemperatures Tested

4.3.3. Fracture Surfaces

Figures 4.19 - 4.21 show the fracture surfaces of specimens after rupture. Figure 4.19 shows the fracture surfaces of the AZ31Mn alloy deformed at 450° C (a - d) and 400° C (e - h) at each strain rate. Dimpled fracture surfaces are seen at $0.1s^{-1}$ and both temperatures, Figure 4.19(a & e). At $0.0003s^{-1}$, non-dimpled fracture surfaces are seen in Figure 4.19(d & h). These are described as intergranular fracture with cavity interlinkages. There is a transition from a dimpled fracture surface to an intergranular fracture surface as strain rate decreases. At 450° C and $0.01s^{-1}$, Figure 4.19(b), there is a

combination of both dimpled areas and intergranular fractured areas. This same transition surface in specimens deformed at 400° C occurs at the lower strain rate of $0.001s^{-1}$, Figure 4.19(g).

Figure 4.20 presents the 450°C fracture surfaces of AZ61Mn at the two strain rates, (**a**) 0.1s⁻¹ and (**b**) 0.0003s⁻¹, and by comparing this to **Figure 4.19**, an effect of Al can be observed. For AZ61Mn deformed at 450°C and 0.1s⁻¹, **Figure 4.20(a)**, shows the dimpled fracture surface, which appears to be very similar to the dimpled fracture of AZ31Mn. At 450°C and 0.0003s⁻¹, **Figure 4.20(b)**, intergranular fractured grains and cavity interlinkages can be seen. The only difference between this and AZ31Mn is the appearance of fibers or filaments in the intergranular areas, which are circled in red.

Figure 4.21 highlights the effect Mn has on the fracture surfaces at 300°C and 0.0003s⁻¹. The 'AZ31' alloys are presented where (a) is AZ31Mn and (b) is AZ31. AZ31Mn, **Figure 4.21(a)**, shows a combination surface with some dimpled areas and within these dimpled areas, evidence of intergranular fracture. AZ31, **Figure 4.21(b)**, shows a dimpled fracture surface only.



Figure 4.19. SEM fracture surfaces of alloy AZ31Mn for the four strain rates tested. (a, b, c, d) @ 450°C and (e, f, g, h) @ 400°C.



Figure 4.20. SEM fracture surfaces of AZ61Mn samples pulled at 450°C showing the strong effect of strain rate.



Figure 4.21. SEM fracture surfaces of (a) AZ31Mn and (b) AZ31 pulled at 300°C and 0.0003s⁻¹ showing the effect of Mn.

4.3.4. Post-Deformation Microstructures

An analysis of the fracture tensile bars in the unstrained grip regions and the fracture tips was performed. All alloys were examined after deformation to fracture at 450°C and 400°C, for all strain rates.

Figure 4.22 shows the optical images for AZ31Mn deformed at 450°C at each strain rate. Figure 4.22($\mathbf{a} - \mathbf{d}$) shows the grip region (left column) and Figure 4.22 ($\mathbf{e} - \mathbf{h}$) shows the fracture section (right column). The first row is at a strain rate of $0.1s^{-1}$ ($\mathbf{a} \ll \mathbf{e}$) and decreases in strain rate until the last row which has a strain rate of $0.0003s^{-1}$ ($\mathbf{d} \ll \mathbf{h}$). The highest strain rate, $0.1s^{-1}$, takes approximately 5 seconds to fracture; the lowest strain rate, $0.0003s^{-1}$, fractures after about 75 minutes.

The high and low strain rates show little difference in grain sizes in the grip regions, **Figure 4.22(a & d)**. They both have a grain size of ~10µm. The fracture areas, **Figure 4.22(e – h)**, show that with a decrease in strain rate, cavitation increases. At $0.1s^{-1}$, **Figure 4.22(e)**, there is no evidence of cavities; At $0.0003s^{-1}$, **Figure 4.22(h)**, there is a significant amount of cavitation. The grain sizes at $0.1s^{-1}$ and $0.01s^{-1}$, **Figure 4.22(e & f)**, show a refined grain structure compared to their grip sections, with an approximate grain size of ~5µm. The grain sizes at $0.001s^{-1}$ and $0.0003s^{-1}$, **Figure 4.22(g & h)**, show a similar grain size when compared to their grip sections, with an approximate grain size of ~10µm.

Figures 4.23 – 4.26 present the grip (**a**) and fracture (**b**) regions of a few of the alloys under selected deformation conditions. **Figure 4.23** presents AZ31Mn at 400°C and 0.01s⁻¹, **Figure 4.24** shows AZ41Mn at 450°C and 0.01s⁻¹, **Figure 4.25** presents AZ31 at 450°C and 0.0003s⁻¹ and **Figure 4.26** presents AZ41 at 450°C and 0.1s⁻¹.

The grip regions, (a), for these alloys show a grain size ~10 μ m. The exception is AZ31, **Figure 4.25(a)**, with grain sizes ranging between ~10 μ m and 40 μ m in the grip region. The fracture areas show different features. AZ31Mn at 400°C and 0.01s⁻¹, **Figure 4.24(b)**, shows a refining effect with grain sizes of <5 μ m with little cavitation. AZ41Mn at 450°C and 0.01s⁻¹, **Figure 4.24(b)**, also shows fine grains of ~5 μ m with increased cavitation.



Figure 4.22. AZ31Mn optical microscope images in the thickness section of the pulled tensile bars at 450° C and each strain rate. Tension axis is horizontal. (a, b, c, d) Grip region. (e, f, g, h) Fracture area.



Figure 4.23. Figure 4.23. AZ31Mn optical microscope images in the thickness section of the pulled tensile bars at 400°C and 0.01s⁻¹ strain rate. Tension axis is horizontal. (a) Grip region. (b) Fracture area.



Figure 4.24. AZ41Mn optical microscope images in the thickness section of the pulled tensile bars at 450°C and 0.01s⁻¹ strain rate. Tension axis is horizontal. (a) Grip region. (b) Fracture area.

AZ31 at 450°C and 0.0003s⁻¹, **Figure 4.25(b)**, shows how the effect the absence of Mn affects microstructure. The fracture area shows grain growth with a grain size of ~50 μ m and there is evidence of cavitation. AZ41 at 450°C and 0.1s⁻¹, **Figure 4.26(b)**, reveals a bimodal microstructure where a matrix of small grains (~15 μ m) surrounds dispersed large grains (>50 μ m).


Figure 4.25. AZ31 optical microscope images in the thickness section of the pulled tensile bars at 450oC and 0.0003s-1 strain rate. Tension axis is horizontal. (a) In the unstrained grip region of the tensile bar. (b) At the fracture point of the tensile bar in the gauge length region.



Figure 4.26. AZ41 optical microscope images in the thickness section of the pulled tensile bars at 450° C and $0.1s^{-1}$ strain rate. Tension axis is horizontal. (a) In the unstrained grip region of the tensile bar. (b) At the fracture point of the tensile bar in the gauge region.

CHAPTER 5. Discussion

5.1. As-Cast and Rolled Sheet Structures

It was expected that with increased aluminum content there would be a grain refining effect in the as-cast structure due to the pinning of grain boundaries [19,26]. **Table 4.1** revealed that this is not the case as aluminum does not have a refining effect. Manganese, however, appears to have a slight grain size effect as the standard Mn alloys have a finer grain size in both 'AZ31' and 'AZ41' alloys. Grain boundaries in the 'AZ61' alloys were obscured by the cast structure, and grain sizes could not be measured.

Reheating dissolved almost all of the second phase particles in these three alloys, Figure 4.3(e - h), and homogenized the cast microstructure in AZ31Mn and AZ41Mn. The dissolution of the dendritic cast structure in the 6wt%Al alloys is incomplete, Figure 4.3(g & h) showing a cored structure. These casting structure 'remnants', which could be due to different segregation levels to grain boundaries, obscure the grain boundaries in the 6wt%Al specimens. There was no significant grain growth with increased annealing time. This was expected since the initial grain sizes are relatively large in the cast microstructure which reduces the driving force for grain growth in larger grains than in smaller ones [26].

The as-rolled microstructures of the six alloys in the normal direction, **Figure 4.4**, and transverse direction, **Figure 4.5**, are similar. The exceptions were the 'AZ61' alloys. Remnants of the cast inhomogeneities appeared as etching effects pancaked in the transverse section aligned in the rolling direction.

There was a matrix of fine grains surrounding a dispersion of a few large grains observed in both **Figures 4.4** and **4.5**. This was also seen in the heat treated samples **Figure 4.6**. Shear banding was present in the transverse sections indicating an accommodation of the imposed deformation through shearing. This shear banding effect has been reported by several researchers in both AZ31 and AZ61 alloys [74-76]. The combination of dispersed large grains and shear banding indicates dynamic recrystallization is occurring. It is assumed the material first dynamically recrystallizes through 'necklacing' which then forms an accumulation of recrystallized grains resulting in shearing. These shear bands undergo grain refinement around larger grains. 'Necklacing' could be occurring on a smaller scale than the shear bands shown which would lead to texture softening and then shear banding. The size of these larger grains decreases as small grains rotate away in the grain boundary region through rotation dynamic recrystallization [40]. Alternatively, at high strain rates seen during rolling, localized shear banding could promote recrystallization.

The rolled plates in **Figure 4.1** and **4.2** indicated that all the alloys containing 6wt%Al exhibited edge cracking but the severity and degree of edge cracking is more prominent in the low Mn alloys. In an attempt to explain this difference, the as-cast plates were analyzed, **Figure 4.3**. In the as-cast condition, there is an increase in the amount of second phases with increasing aluminum content, from 3wt% to 6wt%Al. However, on reheating to 450°C, almost all of the second phase is dissolved. There is a cored structure that remains even after reheating the AZ61 alloy, which may be associated with the edge cracking. However, in this respect, there appears to be little difference between the reheated AZ61Mn and AZ61 alloys.

Nevertheless, both the inhomogeneity and grain boundary characteristics may contribute to edge cracking in the 6wt% Al alloys. It has been well established that higher aluminum content alloys are harder to roll and require a thorough homogenization treatment [3,6]. Edge cracking was even reported in a pre-extruded AZ61 alloy before rolling to help breakdown the cast structure [31]. Nothing was apparent in the microstructure to explain the beneficial effect of Mn in reducing edge cracking. Murai *et al.* found extrusion surface cracks in AZ31 were present when Mn was absent and

eliminated with the addition between 0.2wt% - 0.4wt%Mn [23]. They attributed this manganese effect to increased fracture strength at elevated temperatures [23].

Note that the stage during rolling at which edge cracks developed was not noted in this work. If it occurred in the first pass, then the as-cast reheated structure is the cause. However, if it occurred several passes into the rolling, then the cause is more to do with the deformed structure (e.g. development of shear bands, as indicated above).

The Mn alloys appeared to have a slightly finer recrystallized grain size than the low Mn alloys in the transverse section of both the rolled and heat treated samples, **Figures 4.5** and **4.6** and **Table 4.2**. Grain refinement by manganese at 0.2wt%Mn was also reported in a study of AZ31 extrusions [23]. In wrought *Al-Mg* alloys, manganese retards recrystallization during deformation although it is not explained how [77]. Conversely, a similar parallel might occur in Mg-Al alloys with Mn additions. This would explain the smaller dynamically recrystallized grain size in the Mn alloys and, consequently, less severe edge cracking; finer grains leading to increased ductility. Also, the grain sizes in the Mn alloys show less grain growth with heat treating time, **Table 4.2**. Mn appears to be inhibiting grain growth. Solutes and particles are known to pin grain boundaries although aluminum additions were expected to have a similar effect and this cannot be seen [26].

Another way to consider the edge cracking is to examine the hot tensile data at the highest strain rate. Obviously this is problematic, since the highest strain rate was only 0.1 s⁻¹, and tensile straining is not the same as rolling (which is a combination of shear and compression). In **Figure 4.14**, the ductility of AZ61Mn is actually much higher than AZ31Mn, but these have a much higher ductility than without Mn, at least reinforcing the observed effect on edge cracking. **Figure 4.14** will be discussed in more detail below, but it seems that tensile testing, at the strain rates used here, will not be sufficient to indicate susceptibility to cracks during hot rolling.

5.2. <u>Tensile Testing</u>

5.2.1. General Observations of Flow Curves

Temperature and strain rate dependence on the flow stress was presented in **Figure 4.7**. As expected, the flow stress increased with decreasing temperature and increasing strain rate. As temperature was decreased and strain rate was increased, a high rate of work hardening was developed. A distinct stress peak was observed which indicated the presence of dynamic recrystallization. Grains were work hardened during deformation and once a critical strain was reached dynamic recrystallization was initiated. Following this stress peak, there was dynamic flow softening due to the presence of new unstrained grains [26]. The microstructural evolution has not been checked and it is assumed that softening took place, however, the instability point is a mechanical instability and some hardening may still be taking place.

The change in flow curve behaviour during elevated temperature tensile testing in this study has been reported by several researchers [41-52]. A peak in the tensile stress-strain curve was observed for the temperature range from 300° C – 350° C where there was grain growth of the recrystallized grains [51]. For the range from 400° C – 500° C, the tension peak broadened considerably and, as temperature increased, there was acceleration in the recrystallization process [51].

Yield point phenomena has not been observed (and therefore discussed) in the magnesium literature. In steels, yield point is connected with moving dislocations from the influence of 'Cottrell atmospheres' of interstitial elements. Perhaps the yield points observed in this work have a similar mechanism, but not necessarily involving interstitial elements.

5.2.2. Effect of Temperature

Isolating the effect of temperature on the flow curves, **Figure 4.8**, it was shown that at higher temperatures, the work hardening rate was relatively 'gradual'. Although there is a stress peak indicating dynamic recrystallization, it is much broader than the sharper stress peaks observed at the low temperatures. It is acknowledged that dynamic recrystallization in magnesium occurs in a temperature range from $0.5 - 0.98T_m$ and does occur in these alloys [50-51]. The intermediate temperatures show initial strain hardening over very small strains, followed by strain softening over much larger strains up to fracture.

The trends for the effect of temperature were clearly evident in **Figure 4.8** showing the 'AZ31'alloys tested at $0.1s^{-1}$ and the three temperatures. A stress peak was seen at 300°C and the material failed at a relatively low strain. At 400°C, there was limited work hardening before the peak strain was attained followed by gradual softening until fracture at a higher strain. At 450°C, the stress peak was difficult to distinguish and there does not appear to be much softening occurring in the flow curves before final fracture at a similar strain to 400°C. There was a transition from a clear recrystallization peak at the low 300°C temperature and a very broad, flat, peak at the high 450°C temperature. As temperature was increased, there was limited work hardening and the rate of softening decreased. Although softening occurs due to the dynamic recrystallization process, Bussiba *et al.* attribute the decrease in softening rate to an increase in dislocation activity contributing to the deformation mechanism and some strain hardening.

5.2.3. Effect of Strain Rate

The strain rate dependence on flow stress was shown in **Figure 4.9**. The oscillating nature of the flow curves at $0.0003s^{-1}$ has been attributed to the on/off cycling of the temperature fan regulator attached to the testing furnace. Further investigation is needed, however, to confirm that this is the actual cause of this effect and not a material response. A comparison with other magnesium alloys is required to determine this effect.

As strain rate was decreased, there was a drop in flow stress and an increase in ductility. The stress peak was reached at a higher strain and the softening rates were lower. The material showed a sensitivity to strain rate generally associated with superplastic material and related to flow stress through **Equation 2.3**, (section 2.7.2) [26,53]. Except for the tests performed at 300°C, the other material characteristics and deformation parameters fitted into the three key requirements for superplastic deformation. A fine uniform equiaxed microstructure (<10µm), a relatively high deformation temperature (>0.5T_m) and low strain rates (~10⁻⁵ – 10⁻¹s⁻¹) are the three requirements for superplasticity to occur [26,53,54]. Superplasticity has been reported in AZ alloys ranging from 3wt% to 9wt%Al, with grain sizes <25µm, temperatures >250°C and strain rates <10⁻¹s⁻¹ [54-70].

5.3. Deformation Mechanisms

5.3.1. Macroscopic Photographs

There are many indications of a transition from a high strain rate, low temperature deformation mechanism to a low strain rate, high temperature mechanism. For example, **Figure 4.16** revealed a transition from necking fracture at the high strain rates to uniform elongation rupture at the slow strain rates in all material at 400°C. As well, the flow curves of **Figure 4.9**, for example shows a transition from a high rate of post UTS flow softening to a low rate at a strain rate of 0.001s⁻¹. Superplasticity has been attributed to the suppression of flow instability (necking, in the case of tensile tests) [27]. A metal softening inhomogenously will cause the formation of a neck; this softening effect is opposed by strain or strain rate hardening [27]. In general, in the creep regime, GBS is regarded as being a superplastic mechanism, whereas dislocation creep tends to lead to 'non-superplastic' elongations to fracture.

5.3.2. Strain Rate – Stress Behaviour

The stress exponent, n, in Equation 2.4 has been used to distinguish between the creep mechanisms active during hot deformation [53]. The slopes in Figures 4.17 and 4.18 are the stress exponents and are an established method in determining creep mechanisms in magnesium alloys [53,58-64]. The fractured tensile bars show that necking was associated to samples with a slope (n) of ~5. The uniformly elongated (i.e. no necking but significant cavitation) specimens were associated with a slope (n) of ~2, as was summarized in Table 4.3. This is consistent with similar work by Kulas *et al.* in an Al alloy [73].

The 300°C curves all have a slope (n) of ~5, regardless of strain rate, which is indicative of dislocation creep [53,58]. This is in accordance to the results obtained by del Valle *et al.* for AZ31 who found an approximate slope (stress exponent) of ~6 for the dislocation *climb* creep mechanism [61]. The stress exponent (n) ranges between n = 5-7 for the dislocation climb controlled creep mechanism [58]. An exception may be AZ31Mn at 0.0003s⁻¹ which was beginning to show a change in slope. The slope is calculated to be 2.78, **Table 4.3**. This slope value is indicative of dislocation *glide* controlled creep otherwise known as solute drag creep [53,60]. It is one type of dislocation creep and has been reported to be active in coarse grained (130µm) AZ31 deformed at 375°C and $3x10^{-5}s^{-1}$ [60]. Solute drag creep has been also been reported in an AA5083 aluminum alloy deformed between 425°C - 500°C and $3x10^{-5} - 3x10^{-2}s^{-1}$ [71,72].

The 400°C and 450°C curves show a transition of slopes at certain strain rates. At high strain rates there is a tendency towards a slope (n) of ~5 which is indicative of dislocation climb controlled creep. At the low strain rates, the stress exponent (n) is ~2 which is indicative of the GBS creep mechanism [53,58]. The transition to GBS occurs at higher strain rates with increasing temperature; at 400°C, the change of slope in the curves takes place at a strain rate of $0.001s^{-1}$, whereas at 450°C it appears to be at $0.01s^{-1}$. This change with increasing temperatures has also been reported by several researchers [59,64].

The deformation curves suggest that necking failure is one type of deformation mechanism, dislocation creep, and uniform elongation is associated with GBS. It must be

noted, however, Kim *et al.* were able to distinguish three slope changes in an AZ31 alloy deformed between 350° C - 400° C and $2x10^{-4} - 1x10^{-1}$ s⁻¹, distinguishing between GBS and dislocation creep controlled by either solute drag or climb controlled [64]. There is insufficient data at this point to clearly determine the exact types of dislocation creep and the effect of the solute elements.

5.3.3. Fracture Surfaces

The fracture surfaces for AZ31Mn, **Figure 4.19**, showed a transition in characteristics as strain rate is increased. The fracture surfaces changed from a dimpled structure at high strain rates to an intergranular fracture with interlinked cavities at low strain rates. This transition in fracture surface correlates with the deformation curves presented. Dimples have been associated with the dislocation creep mechanism and cavities with the GBS mechanism. The dimpled surfaces show a close resemblance to the fracture surfaces reported by Marya *et al.* for AZ31 deformed at room temperature [78]. An acknowledged failure mode of GBS is through nucleation and growth of cavities formed during deformation [53,71]. The dimples and interlinked cavities can be seen in the fracture surfaces presented in **Figure 4.19**. A transition fracture surface also appears, **Figure 4.19(b & g)**, where characteristics of dimples and intergranular fracture are both evident. It appears the two competing deformation mechanisms are active in these specimens.

5.3.4. Post-Deformation Microstructures

Figure 4.22 showed a slightly coarser microstructure at the slower strain rates of the grip regions. **Table 4.2** highlighted this observation. Two features were noticed when comparing grip regions with fracture tips. Firstly, at the high strain rates, $0.1s^{-1}$ and $0.01s^{-1}$, no cavities/voids are seen in the microstructure and there is a grain refining effect as the grains in the fracture area are finer than in the unstrained grip section. It may be that dynamic recrystallization occurred with no significant time for grain growth.

Cavities are associated with GBS [53]. At the two low strain rates, there has been grain growth in the dynamically recrystallized grains, however, they show a grain size similar to that observed in their grips sections. Dynamic recrystallization has been reported by several researchers at these high temperatures [41-52]. There appears to be a steady state grain size for the material exhibiting GBS. For a steady state to occur there must be a dynamic balance between the nucleation of new grains and the migration of boundaries [26]. A steady state, or near steady state was observed in the flow curves at high temperatures and low strain rates, **Figure 4.7**.

5.3.5. Microstructure Characteristics

Observations of the final microstructure are important in the understanding of the deformation mechanisms, but it should be remembered that these might represent the microstructure of a component that has been formed 'superplastically'. Therefore, it is important to know how elevated temperature deformation at creep rates will affect the final microstructure.

Figure 4.23 demonstrates the effect that temperature has on the dynamically recrystallized grain size. The fracture tip shows a much finer grain size than that observed at 450°C. This sample shows a stress peak and little ductility, a steady state grain size was not attained. The driving force for dynamic recrystallization is higher at lower temperatures [26]. There was insufficient time for grain growth to occur before the material failed through the dislocation creep mechanism.

5.4. Effects of Aluminum and Manganese

5.4.1. Flow Curves

An investigation of the solid solution strengthening effect found that increasing aluminum in solid solution increases the flow strength [17,19]. Note that these data refer to room temperature properties. However, according to the elevated temperature work performed here (**Figures 4.10** and **4.13**), there is also some indication that Al is increasing the flow stress at high strain rates (**Figure 5.1**, which is an enlargement of **Figure 4.10**). However, the effect is small and only persists to small strains. At about 0.3 strain, the flow curves converge. In fact, it appears that there are two populations of alloys in this figure: (i) the 'AZ61' alloys and (ii) the others. The 'AZ61' alloys exhibit a yield point which is at least as high as the UTS, whereas in the other alloys, the yield point is always less than the UTS.



Figure 5.1. Enlargement of Figure 4.10.

At high temperatures and low strain rates (Figures 4.11 and 4.13), however, no systematic effect or trends with increasing aluminum is immediately apparent. On closer inspection, there appear to be two different effects depending on temperature (Figure 4.13). At the lowest temperature, the low and standard Mn alloys behave similarly, in the

sense that there is a peak at about 4wt% Al: at the intermediate temperature, for the low Mn alloys, increasing Al increases yield strength, but this is the opposite for high Mn alloys: at the highest temperature, there is virtually no effect of Al in either alloy, but the behaviour tends to be more similar to the intermediate temperature than the low. Note that at the lowest temperature, the results indicated dislocation creep as a flow mechanism, whereas at the higher temperatures, GBS dominates (at this strain rate).

As mentioned above, increasing Al increases the solid solution strengthening in Mg at room temperature. Regarding the 300°C tests, when dislocation creep dominates, the initial increase from 3 to 4wt%Al, may therefore be due to solid solution strengthening. The peak may be related to precipitation of Al, which would then reduce the solid solution strengthening effect. The problem with this is that generally precipitates are more effective at strengthening at elevated temperatures than solutes, so perhaps this behaviour is entirely related to precipitates, with the peak value associated with an optimum size and number of precipitates; beyond this Al level, coarser precipitates are formed, and lead to the decrease. Unfortunately, no precipitate studies were carried out in this work, and there seems to be little information about the effect of precipitates on elevated temperature flow behaviour in the literature. For GBS, it is more difficult to explain the diverging behaviours of the Mn and low Mn alloys. To help understand this behaviour, an understanding of the effect of Mn is required.

In this work, the effect of Mn depends on the level of Al to some extent. For example, in **Figure 4.12**, the *highest* strain rate data, which is largely dominated by dislocation creep, although there is a very small effect of Mn, at low Al levels, the strength of the standard Mn is higher, whereas at high Al levels, the effect of Mn is reversed. This is a very slight effect, but it is present at all test temperatures. Perhaps at the low Al levels, Mn contributes to solid solution strengthening, but at the high Al levels, it may be expected that Mn precipitates out with Al, as mentioned in the literature review; this would then reduce the solid solution strengthening effect of Al and lower the strength. A 10 - 25MPa increase in flow strength with Mn was also reported by Mwembela *et al.* in an AZ31 alloy for similar deformation conditions but in torsion [10]. They found that the

microstructures were similar but the AZ31 precipitate particles in the low Mn alloy were smaller and more closely spaced along the grain boundaries. If these precipitates were influencing flow, it might be expected that the finer, and more closely spaced particles would increase strength. However, the lower Mn is weaker. Thus, this result may be taken to reinforce the idea of Mn as a solid solution strengthener.

In **Figure 4.13**, the data at the *lowest* strain rate, which is largely dominated by GBS, especially at 400°C and 450°C, if precipitation does occur at high Al and standard Mn levels, then it appears that precipitates are associated with a decrease in the yield strength, i.e. the low Mn alloys are always stronger. Thus Al and possibly Mn in solid solution are somehow increasing the resistance to GBS, possibly by segregating to grain boundaries and 'locking' them by slowing down accommodation mechanisms such as dislocation motion. Again, the influence of precipitates in directly influencing deformation appears to be minimal. The behaviour of the alloys at 300°C and the lowest strain rate was found to fit dislocation creep rather than GBS, but it seems to have a considerable GBS character, in the sense that the low Mn yield strengths are consistently higher than the standard Mn counterparts, for the whole Al range. Thus, this condition represents a hybrid between dislocation creep and GBS and it is therefore difficult to clearly rationalize the behaviour observed.

At high temperatures and low strain rates, **Figures 4.11**, there appears to be two distinct populations of alloys, delineated by Al; the low Mn alloys show relatively high rates of work hardening at strain up to about 0.2, in fact all the low Mn alloys have almost unnaturally identical flow curves with no effect of Al at this stage. Above this strain, the curves appear to separate somewhat, but there seems to be no systematic effect of Al. The standard Mn alloys show a much lower, almost linear, rate of work hardening, up to about 0.5 strain. Again, the similarity of curve shape for all three Al levels is striking.

This may again be an indication of an effect of Mn in solid solution. The solid solubility of Mn in Mg is considerably lower than for Al (~0.4wt%Mn vs. ~2.6wt% at room

temperature) [6]. Thus, it may be that the segregation of Mn to Mg grain boundaries is much more severe than for Al. Therefore, any addition of Mn will strongly affect the grain boundary characteristics, and apparently overwhelms the effect of Al at the levels used in this work. However, it is not clear why Mn affects the work hardening rates in the observed manner.

5.4.2. Elongation Data

Figure 4.14 shows that ductility increases from 3 to 4wt%Al for both low and standard Mn alloys. Above 4wt%Al, however, two differing trends appear. The alloys with Mn level off or show a slight decrease in ductility up to 6wt%Al. The low Mn alloys show a downward trend in ductility above 4wt%Al. At 0.25wt%Mn level in this study, two factors may be contributing to promote ductility. Firstly, the presence of the Mg-Al second phase at the grain boundaries especially in the 6wt%Al. Secondly, the presence of Mn which may be pinning grain boundaries as the Mn level is insufficient for Al-Mn second phase particles to form. Murai *et al.* found that in the range of 0wt%-0.2wt%Mn a lamellar structure of Mg-Al-(Zn) second phase is present at the grain boundaries [23].

The low strain rate results in **Figure 4.15** show a separation in the curves at the low strain rates. The high aluminum in the presence of Mn appears to improve ductility whereas the low aluminum with low Mn exhibits lower elongation values. The combination of Al and Mn promotes superplasticity at these slow strain rates. Kim *et al.* also reported grain growth in an AZ31 alloy during testing which was not seen in AZ61 [64]. It has been well established that ductility increases as grain size decreases in magnesium [6]. As discussed earlier for **Table 4.2** (section 5.1), Mn alloys appear to have finer recrystallized grains and less grain growth upon heat treatment. This could mean that the low Mn alloys exhibit grain growth which lowers ductility.

5.4.3. Macroscopic Photographs

No significant differences are visible with increased aluminum levels. In the absence of manganese, however, necking is promoted and occurs at lower strain rates. Aluminum appears to have little effect on the strain softening/hardening, **Figures 4.10** and **4.11**, where the steady states flow stresses with changing aluminum contents basically overlap. Manganese alloys, however, show decreased rates of work hardening at the low strain rates, **Figure 4.11**, whereby dynamic recrystallization and softening occurs at a later strain in the flow curves when compared to the low Mn alloys. Suppression of necking during deformation causes uniform elongation.

5.4.4. Strain Rate vs. Yield Strength

Aluminum promotes GBS, **Figure 4.17**. At the low strain rates and high temperatures, increased amounts of aluminum decreases the yield stresses, although, from the limited data, aluminum has no effect on the transition point from dislocation creep to GBS. However, additional tensile tests are required to determine if this is indeed the case. **Figure 4.18** highlights the effect manganese has on the deformation mechanisms. It shifts the transition point from dislocation creep to GBS to higher strain rates, this being especially evident in the 450°C curves. Both aluminum and manganese therefore appear to promote GBS. Aluminum could be acting at the grain boundaries due to undissolved second phases or presence of the cast structure. Manganese could be inhibiting grain growth causing preferential GBS creep.

5.4.5. Fracture Surfaces

A comparison between **Figure 4.19** and **4.20** can be made to analyze the effect both Al and strain rate have on the fracture surfaces. No significant difference exists between the fracture surfaces for AZ31Mn and AZ61Mn deformed at 450°C and 0.1s⁻¹. At 450°C and 0.0003s⁻¹, however, it is observed that the intergranular fractured grains in AZ61Mn have a topographical 'texture' on their surfaces that is absent in the AZ31Mn alloy. These

resemble fibers or filaments, which may be indicative of the GBS mechanism as reported by Kulas *et al.* [71]. The addition of aluminum appears to be promoting GBS in these alloys as the fibers are prominent on the fracture surfaces in the AZ61Mn extending from the grain boundaries. This result may be taken to reinforce the idea of Al is acting at the grain boundaries. Also, the material at the lower strain rate is probably failing with a certain amount of porosity which would lead to the specimen deforming at a higher rate when the strain localized.

The AZ31Mn presented in **Figure 4.21** shows a transition fracture surface. It does not consist entirely of dimples and has some intergranular fracture within the scattered dimples. Manganese appears to promote a change in deformation mechanism as the AZ31 alloy only shows the dimpled structure associated with dislocation creep.

5.4.6. Effect of Tensile Testing on Final Microstructures

The AZ41Mn alloy, **Figure 4.24**, showed the effect aluminum has on the deformation mechanisms. Fine dynamically recrystallized grains indicative of dislocation creep are present, however, cavitation is also evident in this microstructure which is indicative of GBS. If compared to AZ31Mn under the same deformation conditions, **Figure 4.22(f)**, no cavitation is seen. Aluminum therefore seems to promote the GBS regime.

The grain growth that was seen in AZ31 at 450° C and 0.0003s⁻¹, Figure 4.25, demonstrates that the absence of manganese promotes grain growth through the lack of a pinning effect on grain boundaries (section 5.1). In addition, cavitation is less extensive as compared to AZ31Mn, which indicates GBS is less favored in this alloy. The grain growth seen during deformation may contribute to a change in deformation mechanism. The grain size is known to effect deformation mechanisms [53-62]. Tan *et al.* also attributed grain growth during deformation to failure due to a change in deformation mechanism from GBS to dislocation creep [55,56].

The bimodal structure seen in **Figure 4.26** for AZ41 at 450°C and 0.1s⁻¹ highlights the abnormal grain growth that appears to accompany the absence of manganese. Abnormal grain growth is a discontinuous process where a few grains grow and consume the matrix of smaller grains, leading to a bimodal grain size distribution [26]. The selective dissolution of second phase particles are a main factor leading to abnormal grain growth, if these are grain boundary pinning [26]. A very large grain will always grow more slowly than the average grain and eventually become a normal size [26]. In this study, it appears the lack of manganese in the grain boundary areas causes a lack of pinning and abnormal grain growth occurs. Increasing the time of deformation, however, caused a uniform structure.

In summary, the influence of Al and Mn seems to depend on the deformation mechanism. If GBS is occurring, Mn seems to directly, and significantly, affect flow, whereas Al is dominant in dislocation slip. These differences may be due to different grain boundary segregation characteristics of these two elements.

CHAPTER 6. Conclusions

1. Aluminum content has a significant effect on hot rollabillity of AZ type alloys. Increasing the Al level to 6wt% promotes edge cracking during hot rolling, especially in the absence of Mn. Manganese reduced the severity of edge cracking and has been attributed to the increased strength at elevated temperatures. Increasing the heat treating time and/or temperature before rolling will homogenize the 6wt% cast microstructure and help avoid edge cracking.

2. Shear banding was present in the rolled microstructures for all the alloys. The grains were refined during rolling through recrystallization. The Mn alloys had a slightly finer recrystallized grain size and may be due to grain growth inhibition and the pinning effect of manganese.

3. The material shows a temperature and strain rate dependence in the flow curves. Flow stress decreased and ductility increased with increasing temperature and decreasing strain rate. Increasing aluminum increased flow stress at high strain rates but shows no effect at low strain rates. Manganese increased flow stress slightly at high strain rates but decreased the yield stress at low strain rates. The material exhibited superplasticity at high temperatures and low strain rates. Manganese appears to improve the elevated temperature ductility of AZ type alloys.

4. All materials show a transition from GBS at high temperatures and low strain rates to dislocation creep at low temperatures and high strain rates. This transition is characterized by:

- i) a transition in stress exponent from $n \sim 2$ to $n \sim 5$.
- ii) a change in fracture behavior from uniform deformation with cavitation controlled fracture, to localized necking.

iii) a change in stress strain behavior from a lower flow stress with gradual work hardening and a very small post uniform region to very high rates of work hardening at low strains, a very small uniform strain region and low ductility.

5. Both aluminum and manganese appear to enhance GBS in AZ type alloys. It needs to be determined whether this is attributed to grain size differences or enhanced sliding.

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