A STUDY OF THE EFFECTS OF PROCESSING AND ALLOYING ON THE MICROSTRUCTURE AND DEFORMATION BEHAVIOR OF WROUGHT MAGNESIUM ALLOYS

By

Zhe Chen

A DISSERTATION

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Materials Science and Engineering

ABSTRACT

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Lightweight magnesium (Mg) alloys are being progressively incorporated into structural applications where weight reduction is an important design priority. Higher strength and better formability can be achieved by the development of new processing methods and new alloy compositions, which entail a comprehensive understanding of the processing-microstructure-property relationship and deformation mechanisms.

A systematic study on the processing-microstructure-property relationships was performed on a Mg-6Al-0.3Mn (wt%) (AM60) alloy. The effect of thermomechanical processing and subsequent heat treatment on the tensile, fatigue, and creep behavior was investigated. The specific processing conditions investigated were: (1) as-Thixomolded® (asmolded), (2) Thixomolded® then thermomechanically processed (TTMP), and (3) TTMP then annealed (annealed). Compared to the as-molded material, both the TTMP and annealed materials exhibited a significant increase in the tensile yield strength, the ultimate tensile strength, and the fatigue strength, but a decrease in the creep resistance. The altered microstructure was responsible for the change in the mechanical properties. In particular, the resulting mechanical properties were correlated with reduced porosity, texture modification, work hardening, and grain refinement effects introduced by the processing. A detailed analysis was performed on the small fatigue crack growth behavior. The effect of processing and mechanical loading parameters (including maximum applied stress and stress ratio) was evaluated. The applicability of a dislocation-based fracture mechanics relationship to the material was demonstrated by successfully modeling the effect of stress ratio on the crack growth rate using this relationship. Near surface pores were found to be the most preferential sites for fatigue crack initiation.

Dilute rare earth (RE) additions to wrought Mg alloys can lead to texture randomization and potentially improved formability. A novel testing method was employed to study the tensile deformation mechanisms in a conventional wrought Mg alloy Mg-3Al-1Zn (wt%) (AZ31) and a newly-developed RE-containing Mg alloy Mg-1Mn-1Nd (wt%) (MN11) for the temperature range of 50 °C to 250 °C. Twinning and dislocation slip activity were identified by a combination of *in-situ* tensile testing and electron backscatter diffraction (EBSD) analysis. Grain boundary sliding (GBS) activity was evaluated using atomic force microscopy (AFM). For the highlytextured rolled AZ31, prismatic slip was prevalent at all testing temperatures and exhibited increased activity with increasing temperature. The plastic strain ratio was found to decrease with increasing temperature, and was attributed to the increase in both second-order pyramidal < c+a > slip and GBS activity. For the weakly-textured extruded MN11, basal slip was prevalent at all testing temperatures and exhibited increased activity with increasing temperature. It is believed that the addition of RE provides effective strengthening to the basal slip system. The strengthening effect was reduced with increasing temperature due the increased diffusion rate of RE solutes, which led to a decrease in the critical resolved shear stress (CRSS) of basal slip.

Overall the work performed in this dissertation provides new insight into the processingmicrostructure-property relationships and deformation mechanisms in Mg alloys, which can serve as a guidance for alloy development and microstructural optimization. The information and data provided in this dissertation can also be incorporated into future modeling efforts for predicting the deformation pathway and mechanical properties of simulated microstructures. Copyright by Zhe Chen 2012

ACKNOWLEDGEMENTS

First and foremost I would like to thank my advisor Dr. Carl Boehlert. He offered me the opportunity to perform this work. He provided guidance to my study and he was willing to discuss research with me at any time. He also helped me obtain research experiences at Oak Ridge National Laboratory (ORNL). Moreover, in everyday life he showed us how to be a nice person. I will always remember the question he often asked us when we were traveling together, "What should you do if you see a person with a car broken down on the highway", for which the answer may vary for different person and different situation, but the key point is to be helpful to others as best as you can. He is a great advisor and friend, and I cannot thank him enough.

I would also like to thank my committee members, Dr. Thomas Bieler, Dr. Martin Crimp, and Dr. Xinran Xiao, for their guidance in the completion of my degree. Dr. Bieler and Dr. Crimp always led instructive and helpful discussion in our weekly group meeting. Their knowledge, passion, critical thinking, and commitment to the highest standards showed us how to be a great scientist.

Next, I would like to thank Dr. Amit Shyam at ORNL for his help with the fatigue crack growth study. I thank Dr. Jane Howe at ORNL for her help with the transmission electron microscopy work. I thank Dr. Larry Walker, Dr. Odis Cavin, Dr. Thomas Watkins at ORNL for their help with the microprobe and X-ray diffraction characterization. I thank Dr. Ivan Gutierrez-Urrutia at Max Planck Institute for Iron Research (Germany) and Mr. Dongdi Yin at Shanghai Jiaotong University (China) for their help in preparing samples for electron backscatter diffraction analysis. I thank Dr. Jy-An Wang and Dr. Fei Ren at ORNL for their help with my summer internship at ORNL. I thank Dr. Teresa Perez-Prado and Dr. Javier Ilorca at IMDEA Materials Institute (Spain), Dr. Jan Bohlen, Dr. Sangbong Yi, Dr. Dietmar Letzig at Magnesium Innoviation Centre MagIC (Germany), Dr. Jack Huang, Dr. Raymond Decker, Dr. Stephen LeBeau at NanoMag, LLC., (Ann Arbor, MI) for their discussion and contribution to several publications.

I would like to thank my parents for their financial and emotional support for me during my study in all these years. I would like to thank my fellow graduate students and friends Bite Zhou, Xingyi Yang, Bai Nie, Hongmei Li, Di Kang, Ajith Chakkedath, and all other friends at Michigan State University, for helping me with my study, and making my life here much more enjoyable than it would have been without them.

Last, I would also like to thank my girlfriend who never showed up in the first 26 years of my life, thus neither helped nor supported me in my daily life and study, so that I can become much more strong and independent than I would have been with her.

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LIST OF SYMBOLS AND ABBREVIATIONS

2a	Surface crack length
<i>a</i> 0	Critical crack length for small cracks
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
b	Burgers vector
BSE	Backscattered electron
Al	Aluminum
AFM	Atomic force microscopy
at%	Atomic percent
CRSS	Critical resolved shear stress
d	average grain size
D	Diffusion coefficient
da/dN	Fatigue crack growth rate
D_0	Frequency factor
Dgb	Grain boundary diffusion coefficient
$D_{\rm L}$	Lattice diffusion coefficient
DRX	Dynamic recrystallization
ΔK	Stress intensity factor range
ΔK_{th}	Fatigue crack growth threshold
$\Delta K_{\rm tran}$	Stress intensity factor corresponding to the transition crack length

$D_{\mathcal{V}}$	Lattice diffusion coefficient
EGBS	Grain boundary sliding strain
$\dot{arepsilon}_{ m S}$	Steady-state creep rate
E	Young's modulus
EBSD	Electron backscatter diffraction
ED	Extrusion direction
EDM	Electrodischarge machined
EDS	Energy dispersive X-ray spectroscopy
Ef	Elongation-to-failure
$\phi_{\rm c}$	Cyclic crack tip displacement
<i>ø</i> m	Monotonic crack tip displacement
FWHM	Full width at half maximum
G	Shear modulus
GBS	Grain boundary sliding
Н	Average grain boundary step height
НСР	Hexagonal close-packed
IPF	Inverse pole figure
k	Boltzman's constant
Κ	Stress intensity factor
K _C	Critical stress intensity factor, "non-plane-strain fracture toughness" in this study
V	

<i>K</i> _{max}	Maximum stress intensity factor
K _t	Stress concentration factor
LD	Longitudinal direction
LEFM	Linear elastic fracture mechanics
Mg	Magnesium
Mn	Manganese
ν	Poisson's ratio
n	Creep stress exponent
Ν	Number of cycles
ND	Normal direction
Nf	Cycles-to-failure
р	Grain size exponent
Q	Activation energy
r	Plastic strain ratio
r _{pz}	Scaled monotonic plastic zone size
R	Stress ratio
RD	Rolling direction
RE	Rare earth
RT	Room temperature
SE	Secondary electron
SEM	Scanning electron microscopy
SRX	Static recrystallization

Т	Temperature
T _m	Melting temperature
σ	Applied stress
$\sigma_{ m e}$	Fatigue strength (endurance limit)
$\sigma_{\rm max}$	Maximum applied stress
$\sigma_{ m ys}$	Yield strength
TD	Transverse direction
TEM	Transmission electron microscopy
TMP	Thermomechanical processing
wt%	Weight percent
UTS	Ultimate tensile strength
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Rationale

Magnesium (Mg) and its alloys are the lightest commonly used structural metals. Mg alloys exhibit a low density (~1.8g/cm³) and a high specific strength, which are important properties for applications that benefit from weight reduction. They also exhibit good castability, good machinability, and good damping behavior (Kainer and von Buch 2004). Compared with plastics, which generally exhibit a density of less than 1.5g/cm³ ('Plastic'), Mg alloys have much better electromagnetic interference (EMI) shielding ability, higher thermal conductivity, and better recyclability (Landkof 2006). All these properties make them attractive for a variety of applications, including military, portable electronic devices, power hand tools, automobiles, and aerospace components (Landkof 2006, Kainer and von Buch 2004, Friedrich and Mordike 2006, Luo 2004).

Currently, the large scale application of Mg alloys is mainly carried out in the automotive industry (Kainer and von Buch 2004). With the increases in fuel cost, public environmental awareness, and government legislation, the automobile manufacturers are forced to increase the fuel efficiency of the vehicles. According to the Corporate Average Fuel Economy (CAFE) standards, the target fuel economy for passenger cars with size similar to the Mercedes-Benz S-class is 21 miles per gallon (MPG) as expressed in the Environmental Protection Agency (EPA) window sticker value, and this target will be changed to 34 MPG by the year 2025 ('Corporate Average Fuel Economy'). Weight reduction is a straightforward yet important way to improve vehicle fuel economy. For example, a 10% weight reduction can lead to an approximately 7%

increase in fuel economy for a vehicle (Cheah 2010). Thus, Mg alloys are gaining increasing interest in automobile industries in order to reduce vehicle weight and subsequently increase fuel economy.

Mg alloys have been used by automobile manufacturers to make a number of vehicle components. For example, the 1996 Chevrolet Express Van has a 12.3 kg instrument panel beam manufactured using a Mg alloy, which lead to a 32% weight saving over a steel-based design (Taub *et al.* 2007). The 2006 Chevrolet Corvette Z06 has a Mg die-cast cradle, which weighs 10.5 kg, and it is 35% lighter than the Al cradle it replaced (Taub *et al.* 2007). Some other Mg products for automobiles are illustrated in Figure 1.1 and Figure 1.2 (Friedrich and Mordike 2006). The Magnesium Front End Research and Development (MFERD) project launched in 2007 aimed to develop a Mg front end body structure with 50% weight savings compared to the present steel technology (McCarty 2008). Under this project, the unibody front end design of the 2008 Cadillac CTS resulted in a Mg-intensive design that weighed 55.3 kg, which is a 44.3 kg (44.5%) weight reduction compared to the steel baseline design (Luo 2011).



Figure 1.1. The Mg tailgate inner part equipped with the Volkswagen 3L Lupo (Friedrich and Mordike 2006).



Figure 1.2. A 5.9 kg Mg wheel made of AZ91 alloy, used for Porsche 944 model sports car (Friedrich and Mordike 2006).

However, the application of Mg alloys in automobiles is still much lower than steel and Al alloys. This can be attributed to several factors. (1) The choice of Mg alloys available is limited, and the price is relatively high (Kainer and von Buch 2004). (2) The poor formability at room temperature (RT) limits the application of Mg alloys, especially for wrought Mg alloy products. Mg exhibits a hexagonal close-packed (HCP) crystal structure. The critical resolved shear stress (CRSS) for basal slip is significantly smaller than that of non-basal slip systems at RT (Hutchinson and Barnett 2010). Investigations of pure Mg reveal that basal slip and twinning are the dominate deformation mechanisms at RT, and non-basal slip becomes significant only at temperatures higher than 225 °C (Roberts 1960, Sebastian et al. 2006). Thus, the von Mises criterion of five independent slip systems for homogeneous plastic deformation cannot be satisfied at RT, and an increase in formability is expected only at temperatures higher than 225 $^{\circ}$ C (Sebastian et al. 2006). Moreover, the prevalence of twinning in Mg alloys during deformation is known to be a reason for premature fracture (Barnett 2007a, 2007b). Mg alloys tend to form a strong texture during deformation (Agnew et al. 2001), and this leads to anisotropic deformation behavior and mechanical properties (Agnew et al. 2006), which can be undesirable. (3) There is a lack of both fundamental knowledge and practical experience in the application of Mg alloys (Kainer and von Buch 2004). For example, the fatigue properties and durability of Mg alloys are important considering their importance for safety in applications such as steering wheels, instrument panel beams, and seat structures. However, there is only limited data available on the fatigue properties and durability of Mg alloys, especially for wrought Mg alloys (Nyberg et al. 2008). Therefore, the research and development of new alloys and new processing techniques, the fundamental understanding of processing-microstructure-property relationships of Mg and its

alloys, and the establishment of their property database are all very important for the application and development of Mg alloys.

To benefit the application of Mg alloys, the formability problem should be overcome. Recently, a Thixomolded[®] Thermomechanical Processing (TTMP) process was proposed by nanoMAG, LLC (Ann Arbor, Michigan) to produce high-strength wrought Mg alloys (Huang *et al.* 2010). This process takes advantage of the Thixomolded[®] Mg alloys' fine microstructure, which is able to suppress twinning and provide higher strength (Huang *et al.* 2010). Therefore, it is important to provide a detailed characterization of the microstructure and mechanical properties of the processed Mg alloys, which will improve the understanding of processingmicrostructure-property relationships. A better understanding of the processing-microstructureproperty relationship will add knowledge and provide guidance for the application and development of Mg alloys.

Moreover, fundamental studies on the deformation mechanism of Mg alloys are essential for alloy development. It was recently noted that wrought Mg alloys containing rare earth (RE) elements exhibit weak deformation textures, which endow them with a more isotropic mechanical behavior and better formability than conventional wrought Mg alloys (Bohlen *et al.* 2007). Therefore, an investigation and comparison of the deformation mechanisms of both the conventional and RE-containing Mg alloys is beneficial for Mg alloy development.

1.2 Work Performed

This section provides a brief introduction of the work performed in this dissertation. The work was divided into two parts, and three alloy systems were studied.

The first part was a detailed characterization of the microstructure and mechanical properties of a Mg-6Al-0.3Mn (wt%) (AM60) Mg alloy. The AM60 alloy was received from nanoMAG, LLC. The as-received materials were in a sheet geometry and were processed using three different conditions including: (1) As-Thixomolded® (as-molded), (2) Thixomolded® then thermomechanically processed (TTMP), and (3) Thixomolded, thermomechanically processed, then annealed (annealed). The microstructures were characterized using optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). X-ray diffraction (XRD) analysis was also performed for texture determination. Conventional mechanical testing was performed on the AM60 alloy, in a direction parallel with the sheet longitudinal direction, for each of the three processing conditions. Tensile tests were performed at RT and 150 °C using a strain rate of 10^{-3} s⁻¹. Fatigue tests were performed at RT and 150 °C, with a stress ratio R = 0.1, and a frequency of 5 Hz. The maximum applied stress (σ_{max}) ranged from 50 to 175 MPa. Fatigue crack growth experiments were performed at RT using specimens with an artificial notch machined prior to testing. The maximum applied stress ranged between 60% and 90% of the yield strength (σ_{vs}) of each material. The stress ratios applied were R = 0.1, 0.3, and 0.5, and the frequency was 5 Hz. The crack growth behavior was characterized in-situ with the aid of a long working-distance optical microscope. Conventional tensile-creep tests were performed at temperatures between 100 and 200 $^{\circ}$ C and maximum applied stresses between 20 and 75 MPa. In-situ tensile-creep tests were performed inside an SEM on the annealed material, with specimens machined both along the longitudinal and transverse directions. The experiments were performed at 150 $^{\circ}$ C and maximum applied stresses of 50 and 75 MPa.

The second part of this dissertation work was an *in-situ* analysis of the deformation mechanisms in two wrought Mg alloys: Mg-3Al-1Zn (wt%) (AZ31), which represents a conventional wrought Mg alloy and Mg-1Mn-1Nd (wt%) (MN11), which represents a REcontaining Mg alloy. The AZ31 alloy was purchased from Magnesium Elektron (Manchester, UK) in the form of rolled and annealed sheet. In-situ tensile experiments were performed at a constant strain rate of $\sim 10^{-3} \text{s}^{-1}$, both along the rolling direction (RD) and transverse direction (TD) of the sheet. The testing temperatures were 50 $^{\circ}$ C, 150 $^{\circ}$ C, and 250 $^{\circ}$ C. Electron backscatter diffraction (EBSD) mapping was performed on selected areas of the specimens' surfaces both before and after deformation, and the data provided information about the orientations of individual grains. SEM images were acquired at selected times within the region in which the EBSD maps were acquired. In this manner, quantitative information of the deformation modes as a function of the strain, temperature, and grain orientation was obtained. In addition, in-situ tensile creep experiments were performed at 150 $^{\circ}$ C and a maximum applied stress of 75 MPa, both along the RD and TD. Atomic force microscopy (AFM) was performed on the tested samples in order to evaluate the grain boundary sliding (GBS) activity. The MN11 alloy was gravity cast, then extruded at 300 °C to produce a round bar. In-situ SEM tensile experiments were performed at a constant strain rate of $\sim 10^{-3} \text{s}^{-1}$ along the extrusion direction (ED) of the material. The testing temperatures were 50 °C, 150 °C, and 250 °C. EBSD was performed on selected areas of the specimens' surface both before and after deformation. Similar to that for the AZ31 material mentioned above, quantitative information of the deformation modes as a function of the strain, temperature, and grain orientation was obtained based on the *in-situ* SEM images and EBSD analysis. For all the experiments mentioned above, the load, displacement, temperature, and time were recorded throughout the experiments.

1.3 Research Objectives

The overall goal of this dissertation was to gain a greater knowledge of the deformation behavior of structural Mg alloys under a variety of mechanical testing conditions in order to establish a fundamental understanding of the processing-microstructure-property relationships. As pointed out in section 1.1, this understanding would be advantageous for the implementation of Mg alloys in future structural applications. The following describes the specific aims and tasks in this dissertation work for each of the studied alloy systems.

AM60 Alloy:

Microstructure

- (1) To understand the effect of thermomechanical processing and subsequent heat treatment on the microstructural evolution.
- (2) To quantify the microstructure features, such as grain size and phase volume fraction, in order to obtain an understanding of the relationship between the microstructural features and the mechanical properties.
- (3) To determine the texture under different processing conditions, using XRD and EBSD, in order to understand the effect of processing on the texture development.

Tensile Behavior

- (1) To obtain RT and elevated temperature (150 °C) tensile properties as a function of the processing condition.
- (2) To choose the appropriate applied stress levels for the fatigue and creep experiments based on the tensile properties.

Fatigue Behavior

- (1) To obtain fatigue S-N data at RT and elevated temperature (150 °C) under a maximum applied stress range of 50-175 MPa and a stress ratio R = 0.1.
- (2) To characterize the RT fatigue crack growth behavior under different loading conditions. Evaluate the effect of alloy processing, maximum applied stress, and stress ratio on the fatigue crack growth behavior.
- (3) To fit the fatigue crack growth rate with both a linear elastic fracture mechanics (LEFM) based model and a dislocation-based fracture mechanics model. To evaluate the relative applicability of these models.

Creep Behavior

- (1) To evaluate the creep strain versus time behavior at 150 ℃ for an applied stress range of 20-75 MPa. To evaluate the dependency of the steady-state creep rate on the applied stress and temperature over the applied stress range of 20-75 MPa and the temperature range of 100-200 ℃.
- (2) To characterize the tensile-creep deformation evolution by performing *in-situ* creep experiments.
- (3) To identify the dominant secondary-creep mechanisms using several methods including the *in-situ* test data, post-test SEM characterization, and the calculated creep stress exponents and apparent activation energies.

AZ31 Alloy:

- (1) To perform EBSD both before and after deformation in order to obtain information on the texture evolution as well as the crystallographic orientation of individual grains in selected areas.
- (2) To determine the onset of different slip and twinning systems during deformation as a function of temperature.
- (3) To quantify the relative activities of the slip and twinning systems as a function of strain and temperature.
- (4) To obtain evidence useful for establishing the relative importance of GBS and nonbasal slip activity on the anisotropic behavior as a function of temperature.

MN11 Alloy:

- (1) To perform EBSD both before and after deformation in order to obtain information on the texture evolution as well as orientation of individual grains in selected areas.
- (2) To determine the onset of different slip and twinning systems during deformation as a function of temperature.
- (3) To quantify the relative activities of the slip and twinning systems as a function of strain and temperature.
- (4) To compare the deformation behavior of a Mg alloy which does not exhibit a strong wrought texture (MN11) with that for a conventional Mg alloy which exhibits a strong wrought texture (AZ31) in order to shed insight into the anisotropic behavior of Mg alloys as a function of both temperature and alloy content.

CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

2.1 Fundamentals of Mg and Mg Alloys

2.1.1 History

Mg constitutes 2% of the mass of the earth's crust and 0.13% of the mass of sea water. It is the third most abundant among the dissolved minerals in sea water (Kainer and von Buch 2004). The history of Mg dates back to 1755 when a Scottish chemist, Joseph Black, discovered it in magnesia – a name which originates from an area in ancient Greece. It was not until 1808 that this element was firstly isolated by the British chemist and scientist Sir Humphrey Davy (Friedrich and Mordike 2006).

Early applications of Mg and Mg alloys were mainly in the automobile and military fields, such as the Volkswagen Beetle, which was designed in 1938 and contained 20kg of Mg, and the B-36 bomber, which served the United States Air Force in the 1950s and contained 19,000 lbs of Mg parts (Friedrich and Mordike 2006). The production and application of Mg dropped significantly after World War II and increased slowly thereafter. The annual production of Mg in the 1990s was comparable to the peak value of that in the 1940s (Friedrich and Mordike 2006, Powell *et al.* 2010). However, with the increasing demand of light-weight applications for fuel efficiency, the production of Mg has almost doubled in the past ten years (Powell *et al.* 2010), indicating the potential and importance of Mg.

The production methods of Mg can be generally divided into two types: the electrochemical method and the thermal reduction method. In the electrochemical method, anhydrous magnesium chloride (MgCl₂), which can be produced either from ores or sea water
(Friedrich and Mordike 2006), is used in electrolysis to produce Mg based on the following chemical equation:

$$MgCl_{2(l)} \rightarrow Mg_{(l)} + Cl_{2(g)}$$

$$(2.1)$$

In the thermal reduction method, dolomite (MgCO₃ CaCO₃) or magnesite (MgCO₃), which are used as the raw materials, first undergo calcination (Emley 1966):

$$MgCO_3 CaCO_{3(s)} \rightarrow MgO CaO_{(s)} + 2CO_{2(g)}$$
(2.2)

$$MgCO_{3(s)} \rightarrow MgO_{(s)} + CO_{2(g)}$$
 (2.3)

Then, ferrosilicon (Si(Fe)) is used for reduction, and depending on the temperature and pressure, one of the following reactions is used to produce Mg (Friedrich and Mordike 2006):

$$2MgO_{(s)} + Si(Fe) \rightarrow 2Mg_{(g)} + SiO_{2(l)} + Fe$$
(2.4)

$$4MgO_{(s)} + Si(Fe) \rightarrow 2Mg_{(g)} + Mg_2SiO_{2(s)} + Fe$$
(2.5)

$$2MgOCaO_{(s)} + Si(Fe) \rightarrow 2Mg_{(g)} + Ca_2SiO_{4(l)} + Fe$$
(2.6)

The thermal reduction method is mainly used in China. It requires much less capital investment than the electrochemical methods. However, it needs much more energy consumption and labor, and it is less environmentally friendly (Friedrich and Mordike 2006).

2.1.2 Metallurgy of Mg and Mg Alloys

Mg is an alkaline earth metal. It has a HCP (A3 type) crystal structure which belongs to space group P6₃/mmc ('Crystal Lattice Structures: The HCP (A3) Crystal Structure'), and the lattice parameters at RT are a = 0.32089 nm and c = 0.52101 nm (Delfino *et al.* 1990). Thus, the *c/a* ratio at RT is 1.624, which is very close to the ideal value of 1.633 for the HCP structure.

Some physical and mechanical properties of pure Mg at RT are listed in the following (Buch 1999, Daniels 2006, Kainer and von Buch 2004): The density is 1.74 g/cm³. The Poisson's ratio is 0.35. The elastic modulus is 45 GPa, and the shear modulus is 17 GPa. The tensile yield strength is 21MPa for cast Mg, and is 121 MPa for worked Mg. The ultimate tensile strength (UTS) is 90 MPa for cast Mg, and is 207 MPa for worked Mg. The elongation-to-failure (ε_f) is 16%. The melting point is 650 °C, and the boiling point is 1090 °C. The recrystallization temperature ranges from 93 °C to 177 °C. Pure Mg is soft, so it has to be alloyed with other elements for practical structural application.

The codification of Mg alloys according to ASTM standards ('Standard Practice for Codificatoin of Certain Nonferrous Metals and Alloys, Cast and Wrought' 2006) is to list the abbreviation letters representing the alloying element, then list the rounded figures representing the weight percent of the corresponding element. An additional letter can be added after the rounded figures to represent the stage of development of the alloy. The letters representing different alloy elements are summarized in Table 2.1. It is noted that in this study, the Mg-1Mn-1Nd alloy is labeled as "MN11" which is not consistent with the standard code, because "N" refers to neodymium, not nickel, in this alloy.

Abbreviation Letter	Alloying Element	Abbreviation Letter	Alloying Element
А	Aluminum (Al)	Ν	Nickel (Ni)
В	Bismuth (Bi)	Р	Lead (Pb)
С	Copper (Cu)	Q	Silver (Ag)
D	Cadmium (Cd)	R	Chromium (Cr)
E	Rare Earths	S	Silicon (Si)
F	Iron (Fe)	Т	Tin (Sn)
G	Magnesium (Mg)	V	Gadolinium (Gd)
Н	Thorium (Th)	W	Yttrium (Y)
J	Strontium (Sr)	Х	Calcium (Ca)
Κ	Zirconium (Zr)	Y	Antimony (Sb)
L	Lithium (Li)	Z	Zinc (Zn)
М	Manganese (Mn)		

Table 2.1. Letters representing alloying elements in the ASTM specification ('Standard Practice for Codification of Certain Nonferrous Metals and Alloys, Cast and Wrought' 2006).

A few alloying elements related to the alloys investigated in this study will be introduced in the next section.

(1) Aluminum

Aluminum (Al) is a common alloying element in Mg alloys. The addition of Al can increase the castability of Mg alloys. It can also increase the strength and hardness by solid solution hardening and precipitation strengthening (Friedrich and Mordike 2006). The Mg-Al binary phase diagram is shown in Figure 2.1 (Massalski *et al.* 1986). The solubility of Al in the HCP α -phase Mg solid solution has a maximum value of 12.7 wt% at the eutectic temperature of 437 °C. However, it drops below 1 wt% at RT. The intermetallic γ -phase (also called the β phase) has a stoichiometric composition of Mg₁₇Al₁₂. Its RT elastic modulus is 80 GPa (Braszczynska-Malik 2011). It has a cubic crystal structure and a unit cell of α -Mn (A12) type, which belongs to space group I43m, with a lattice parameter of a = 1.0544 nm (Schobinger-Papamantellos and Fischer 1970). Figure 2.2 illustrates a Mg₁₇Al₁₂ unit cell, which contains 58 atoms occupying four symmetric sites in the A12 type structure. Mg occupies three out of the four symmetric sites, and Al occupies the remaining one, as illustrated in Figure 2.3. The symmetric positions of atoms in the A12 type unit cell are summarized in Table 2.2 ('Crystal Lattice Structures: The α Manganese (A12) Type', 'Wyckoff Positions of Group 217 (I-43m)'). The parameters used for the Mg₁₇Al₁₂ unit cell are shown in Table 2.3 (Schobinger-Papamantellos and Fischer 1970).

The Mg₁₇Al₁₂ γ -phase can exist both within grains and at the grain boundaries, and it can form during either solidification or subsequent heat treatment. During non-equilibrium solidification of Mg-Al alloys with Al concentrations smaller than that of the eutectic composition, a binary $\alpha + \gamma$ eutectic can form at the interdendritic regions. A fully-divorced eutectic is favored at higher cooling rates, and a partially divorced eutectic can be obtained at slower cooling rates (Dantzig and Rappaz 2009). A discontinuous precipitate of γ -phase with lamellar microstructure can also form at even slower cooling rates (Braszczynska-Malik 2011). During aging of supersaturated Mg-Al alloys, both continuous and discontinuous precipitation of $Mg_{17}Al_{12}$ can occur, and the former is favored at higher temperatures while the latter is favored at lower temperatures (Braszczynska-Malik 2011). A discontinuous precipitation (DP) indicates the growth of a lamellar structure of alternating plates of the α - and γ -phases, and it is favored at the grain boundaries. In continuous precipitation (CP), the γ -phase nucleates and grows inside the α -grain (Braszczynska-Malik 2011), on dislocations or twin boundaries (Kuffova 2011). For both precipitates, the predominant α - γ orientation relationship (OR) is the Burgers OR: $(0001)_{\alpha}$

 $(1 \uparrow 0)_{\gamma}$ and $[2 \uparrow \uparrow 0]_{\alpha} \parallel [111]_{\gamma}$, although some other ORs have also been reported (Zhang and Kelly 2003).



Figure 2.1. Mg-Al phase diagram (Massalski et al. 1986).



Figure 2.2. A unit cell of $Mg_{17}Al_{12}$. The Mg atoms are represented by the orange balls, and the Al atoms are represented by the blue balls. This drawing was generated using the VICS II software.



Figure 2.3. A $Mg_{17}Al_{12}$ unit cell in which the four symmetric sites in the A12 type structure are illustrated by balls of different colors. This drawing was generated using the VICS II software.

Position	Wyckoff	Site	Internal	Coordinates
	Notation	Symmetry	Parameters	(0, 0, 0) + (1/2, 1/2, 1/2,) +
1	(2a)	43m	None	(0, 0, 0)
2	(8c)	.3m	[<i>x</i> ₂]	$(x_2, x_2, x_2) (x_2, -x_2, -x_2) (-x_2, x_2, -x_2)$
				$(-x_2, -x_2, x_2)$
3	(24g)	m	$[x_3, z_3]$	$(x_3, x_3, z_3) (z_3, x_3, x_3) (x_3, z_3, x_3)$
				$(-x_3, x_3, -z_3) (-z_3, x_3, -x_3) (-x_3, z_3, -x_3)$
4	(24g)	m		$(x_3, -x_3, -z_3) (z_3, -x_3, -x_3) (x_3, -z_3, -x_3)$
				$(-x_3, -x_3, z_3) (-z_3, -x_3, x_3) (-x_3, -z_3, x_3)$
			$[x_4, z_4]$	$(x_4, x_4, z_4) (z_4, x_4, x_4) (x_4, z_4, x_4)$
				$(-x_4, x_4, -z_4) (-z_4, x_4, -x_4) (-x_4, z_4, -x_4)$
				$(x_4, -x_4, -z_4) (z_4, -x_4, -x_4) (x_4, -z_4, -x_4)$
				$(-x_4, -x_4, z_4) (-z_4, -x_4, x_4) (-x_4, -z_4, x_4)$

Table 2.2. Summary of the symmetric positions of atoms in the A12 type unit cell ('Crystal Lattice Structures: The α Manganese (A12) Type', 'Wyckoff Positions of Group 217 (I-43m)').

Table 2.3. Summary of the parameters used to describe atom positions in the $Mg_{17}Al_{12}$ unit cell (Schobinger-Papamantellos and Fischer 1970).

Atom	Symmetric Position and its Corresponding Wyckoff Notation	Parameters in Coordinates
Mg	1 (2a)	None
Mg	2 (8c)	$x_2 = 0.3240$
Mg	3 (24g)	$x_3 = 0.3582$ $z_3 = 0.0393$
Al	4 (24g)	$x_4 = 0.0954$ $z_4 = 0.2725$

(2) Zinc

Zinc (Zn) is an important alloying element usually used in conjunction with Al (Friedrich and Mordike 2006). Compared with binary Mg-Al alloys, there are no new phases in the Mg-Al-Zn alloy system when the Al-to-Zn ratio is over 3:1 (Celotto and Bastow 2001). Similar to the Al effect, the addition of Zn can also increase the castability and strength of Mg alloys (Kainer and von Buch 2004).

(3) Manganese

The Mg-Mn binary phase diagram has only been determined for the Mg-rich region so far, as seen in Figure 2.4 (Massalski *et al.* 1986). Mn can reduce the solubility of iron (Fe) in Mg alloys, which leads to an improvement in the corrosion resistance (Friedrich and Mordike 2006). Based on the phase diagram, the solubility of Mn in Mg is low, and with decreasing temperature, precipitation of α -Mn from supersaturated solid solutions is expected. Mn is also usually used together with other alloying elements such as Al, however, some binary alloys such M1A (Mg-1Mn wt%) are used for forging or extrusion (Friedrich and Mordike 2006).

(4) Rare Earth Elements

Mg alloys with high alloying contents of RE elements (such as yttrium, cerium, neodymium, terbium, erbium, dysprosium, gadolinium, etc.) are commonly used for high temperature applications. The solubility of RE elements in Mg is generally very limited (Kainer and von Buch 2004). Depending on the alloy system, RE elements can form a variety of precipitates with Mg during the solution heat treatment + aging process (Nie and Muddle 2000, Apps *et al.* 2003, He *et al.* 2006). These precipitates are thermally stable, thus significantly improve the high temperature strength and creep resistance of Mg alloys.

In addition, RE elements can serve as a texture modifier for Mg alloys. It has recently been reported that the addition of RE elements can lead to a more random texture in extruded and rolled Mg alloys (Ball and Prangnell 1994, Bohlen *et al.* 2007, Mackenzie and Pekguleryuz 2008). Thus, Mg alloys containing dilute additions of RE elements can possess mechanical properties suitable for wrought processing.



Figure 2.4. Mg-Mn binary phase diagram for the Mg-rich region (Massalski et al. 1986).

2.1.3 Deformation Mechanisms

Mg has an HCP crystal structure in which the (0001) basal plane is the close-packed plane and the <1120> directions (<*a*> directions) are the close-packed directions. The (0001) <1120> basal slip is an easily-activated slip mode that is widely reported in Mg (Bakarian and Mathewson 1943, Hauser *et al.* 1956, Roberts 1960). In addition, slip along the <*a*> directions can also take place on non-basal planes, including the {1010} (first-order prismatic) planes (Hauser *et al.* 1956, Reed-Hill and Robertson 1957, Ward Flynn *et al.* 1961) and {1011} (firstorder pyramidal) planes (Bakarian and Mathewson 1943, Reed-Hill and Robertson 1957). Moreover, slip along the <1123> directions, i.e., the <*c*+*a*> directions, on the {1122} (secondorder pyramidal) planes has also been reported (Obara *et al.* 1973, Agnew *et al.* 2001, Agnew and Duygulu 2005), and they are considered to be important in accommodating deformation along the *c*-axis.

Various types of twinning have also been reported, including {1012}, {1121}, {1011}, {3034}, {1013}, {1015}, and {1124} twins (Hauser *et al.* 1956, Couling and Roberts 1956, Yoshinaga and Horiuchi 1963). Twinning is a polar mechanism (Christian and Mahajan 1995). As a result, extension twinning such as the { $1012}<1012$ twin is active only when the deformation conditions are such that an extension along the c-axis takes place. In turn, contraction twinning, such as for the { $1011}<1012$ and { $1013}<3032$ twins, takes place when the deformation gives rise to compression along the c-axis. Twinning leads to reorientation of the lattice. For example, the { $1012}<1012$ extension twin leads to an ~86 ° reorientation of the lattice about the <1210 axis, and the { $1011}<1012$ contraction twin leads to an ~56 ° reorientation of the lattice about the <1210 axis (Al-Samman and Gottstein 2008). In general, twinning is favored at high strain rates and low temperatures (Christian and Mahajan 1995).

Figure 2.5 illustrates four slip systems and two twinning systems. These deformation modes are considered to be important in Mg, especially for modeling the deformation behavior (Agnew *et al.* 2001, Barnett 2003).



Figure 2.5. Illustration of four slip systems and two twinning systems in Mg: (a) $\{0001\}<1210>$ basal slip, (b) $\{1100\}<1120>$ first order prismatic slip, (c) $\{1101\}<1120>$ first order pyramidal <a> slip, (d) $\{1212\}<1213>$ second order pyramidal <c+a> slip, (e) $\{0112\}<0111>$ extension twin, and (f) $\{0111\}<0112>$ contraction twin.

The dominance of a specific slip or twinning system is highly dependent on the deformation temperature. Although widespread values of the CRSS have been reported for the various slip and twinning systems of Mg alloys (Barnett *et al.* 2006, Hutchinson and Barnett 2010), it is generally accepted that the CRSS follows the trend $CRSS_{basal} < CRSS_{\{10\Gamma2\}}$ twinning $< CRSS_{prismatic} \leq CRSS_{pyramidal}$ at RT. As a result, basal slip and extension twinning are the dominant deformation mechanisms in randomly-oriented polycrystals at low temperatures (including RT) and low strain rates (Hauser *et al.* 1956, Couling *et al.* 1959, Kelly and Hosford 1968).

The temperature dependence of the CRSS of different slip and twinning systems in Mg has been investigated in many studies. It has been reported that the CRSS of basal slip in Mg decreases with increasing temperature below 300K, but becomes temperature independent above 300K (Akhtar and Teghtsoo 1972). The CRSS of {1012} extension twinning is also considered to be temperature insensitive at RT and above (Barnett 2003, Chapuis and Driver 2011). However, the CRSS of prismatic slip, pyramidal slip, and contraction twinning was reported to decrease with the increase of temperature (Reed-Hill and Robertson 1957, Obara et al. 1973, Yoshinaga and Horiuchi 1963). The CRSS of prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip systems are estimated to become lower than that of {1012} twinning, reaching that of basal slip as the temperature increases (Barnett 2003, Chapuis and Driver 2011). Therefore, the activity of these non-basal slip systems increases with temperature, facilitating intergranular compatibility (Ward Flynn et al. 1961, Vagarali and Langdon 1981, Agnew and Duygulu 2005, Meza-Garcia et al. 2007), thereby increases the likelihood for slip transfer across grain boundaries and decreases the likelihood for grain boundary crack formation. However, the temperature at which this transition occurs is still unclear.

Due to the limited number of active slip systems, the deformation of Mg alloys is also highly dependent on texture (Gehrmann *et al.* 2005, Al-Samman and Gottstein 2008). This effect is especially pronounced in wrought Mg alloys, which generally exhibit a marked initial texture. For example, rolled Mg alloy sheets usually have a strong basal-type texture where the c-axis is parallel to the normal direction (ND), thus extension twinning is the dominant deformation mode during the first stages of deformation when compressive loading is applied in a direction parallel to the rolling plane. The extension twinning leads to an ~86 ° reorientation of the lattice, so the c-axis of most grains becomes parallel to the applied stress. At this point, the Schmid factors of basal slip and extension twinning are both very low, thus non-basal slip (mainly <c+a> slip) is favored. Moreover, when rolled Mg sheets are strained in tension along the RD or TD, non-basal slip systems can also be activated (Agnew and Duygulu 2005, Keshavarz and Barnett 2006). These differences in the deformation modes as a function of the loading direction lead to an asymmetry in the yield strength (Ball and Prangnell 1994, Barnett *et al.* 2004, Lou *et al.* 2007).

Moreover, GBS may also contribute to the deformation of Mg alloys, although the significance of its contribution is still under debate. Some studies suggest that GBS occurs even at a temperature as low as 78K, and becomes prevalent at RT (Hauser *et al.* 1956). Many studies suggest that a combination of GBS and crystallographic slip contribute to the plastic deformation, and the contribution of GBS becomes more important at high temperatures (Koike *et al.* 2003, Del Valle *et al.* 2005, Del Valle and Ruano 2007, Hutchinson *et al.* 2009). However, some other studies, performed at temperatures ranging from RT to 250 °C, suggest that the contribution of GBS is negligible (Del Valle and Ruano 2009, Stanford *et al.* 2011).

Plastic anisotropy is an important feature of rolled sheet metal. The plastic anisotropy is

measured by the plastic strain ratio (*r*-value), which is defined as $r = \frac{\varepsilon_{\rm W}}{\varepsilon_{\rm t}}$, where $\varepsilon_{\rm W}$ is the in-plane

plastic strain, and e_t is the through-thickness plastic strain. It has been reported in several studies that highly-textured rolled AZ31 sheet shows a significant drop in the plastic strain ratio between RT and 250 °C (Agnew and Duygulu 2005, Hutchinson *et al.* 2009, Barnett *et al.* 2009, Stanford *et al.* 2011). An *r*-value larger than 4 is found at RT, while it decreases to ~2 at 150 °C, and ~1.4 at 250 °C. Generally, the decrease in the plastic strain ratio is explained by two different causes. Some authors suggest it to be a result of the increased activity of pyramidal $\langle c+a \rangle$ slip with increased temperature (Agnew and Duygulu 2005, Stanford *et al.* 2011). However, some other authors attribute this phenomenon to the activation of GBS (Barnett *et al.* 2009, Hutchinson *et al.* 2009). These two hypotheses were both supported with simulations based on viscoplastic selfconsistent (VPSC) modeling, and it was demonstrated in both cases that the reduced anisotropy at higher temperatures could be obtained by increasing the activation of their respective additional deformation modes. Thus, it is not possible to discriminate from the modeling perspective which deformation mode is responsible for the reduction in anisotropy.

2.1.4 Deformation and Recrystallization Texture

Wrought Mg alloys generally exhibit a strong texture. An ideal deformation texture for a rolled Mg sheet or an extruded Mg rod is such that the basal planes of all grains are parallel to the surface of the rolled sheet or extrusion direction, respectively (Roberts 1960). In practice, the textures of wrought Mg alloys deviate from these ideal textures, with the basal poles tilted toward either the RD/ED or TD, depending on the alloy composition, deformation condition, or

even the position within the sample at which the measurements are made (Roberts 1960). The direction of the (1010) poles is also preferentially orientated in some cases. For extruded rods, a (1010) pole parallel to the ED is considered to be typical for many HCP metals (Dillamore and Roberts 1965), including conventional wrought Mg alloys (Bohlen *et al.* 2010). For rolled Mg sheets, a uniform spread of the (1010) pole in the RT-TD plane was observed in a number of studies (Agnew and Duygulu 2005, Hutchinson *et al.* 2009), which is, in fact, expected for an HCP metal with c/a ratio close to the ideal value of 1.633 (Rollet and Wright 1998).

However, the texture of hot-worked Mg alloys could actually be a combination of deformation texture and recrystallization texture. As mentioned in the previous sections, Mg has poor RT formability and is usually worked at elevated temperature. Thus, deformation and recrystallization often occur simultaneously during the thermomechanical processing. Moreover, an annealing process is often applied after hot working to serve as a strain relief process which may enhance the ε_f of the alloy. Therefore, static recrystallization can also occur in the annealing process, further affecting the final texture obtained. In fact, it has been suggested by several studies (Ball and Prangnell 1994, Mackenzie *et al.* 2007, Bohlen *et al.* 2007, 2010, Stanford 2010) that the deformation texture of different Mg alloys are basically the same, while the recrystallization texture plays a dominant role in determining the final texture of the processed alloy. For many conventional wrought Mg alloys, the deformation texture is basically retained after recrystallization. However, for RE-containing wrought Mg alloys, a texture modification is expected.

For extruded Mg alloys, a recent study suggests that the extrusion texture changes with increasing concentrations of different RE elements including lanthanum (La), yttrium (Y), gadolinium (Gd), and cerium (Ce), following a common path: from "pure Mg texture", to

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"conventional texture", and finally to "RE texture" (Stanford 2010). In that study, for pure Mg or binary Mg alloys with dilute addition of RE elements (specifically, 0.02 at% for Y, 0.01 at% for La, 0.015 at% for Gd, and 0.016 at% for Ce), a pure Mg texture is obtained. This type of texture can be described by an inverse pole figure (IPF) in the extrusion direction (ED) as shown in Figure 2.6(a), where the maximum intensity lies on an arc that is offset from the [$\Gamma 2 \Gamma 0$]-[$01 \Gamma 0$] arc by 14°. With increasing amounts of RE element, the extrusion texture changes to that of the more conventional wrought texture, as illustrated in Figure 2.6(b). The maximum intensity lies on the [$\Gamma 2 \Gamma 0$]-[$01 \Gamma 0$] arc, and is more toward the [$01 \Gamma 0$] pole. At a sufficiently high RE concentration (specifically, 0.03 at% for La, 0.06 at% for Gd, and 0.04 at% for Ce), the RE texture is obtained, as illustrated in Figure 2.6(c). In this texture, the maximum intensity peak shifts to the [$\Gamma 2 \Gamma 1$] pole, which is usually referred to as the RE component.



Figure 2.6. IPFs in the extrusion direction of extruded (a) pure Mg, (b) Mg-0.02at% La, and (c) Mg-0.06at% Gd, representing the pure Mg texture, conventional texture, and RE texture, respectively (Stanford 2010).

In another work, Bohlen et al. (2010) studied the effect of RE elements on the texture development during extrusion of Mg-Mn alloys. In that study, extrusion was performed on Mg-Mn, Mg-Mn-Ce, Mg-Mn-Y, and Mg-Mn-Nd alloys, and either fully-recrystallized or partiallyrecrystallized microstructure were obtained. The grains were categorized into deformed unrecrystallized grains and recrystallized grains, and the recrystallized grains were further categorized into small recrystallized grains and large recrystallized grains, which corresponded to a different extent of grain growth. The contribution of each category to the texture was evaluated separately. It was shown that in all the alloys with and without RE elements, the deformed unrecrystallized grains contributed to a [1010] peak on the IPF in the ED. For the Mg-Mn alloy, which represents a conventional Mg alloy, the recrystallized grains contributed to an intensity uniformly distributed on the [1010]-[1120] arc. For the Mg-Mn-RE alloys, the recrystallized grains resulted in additional [2021] and [1121] components on the IPF in the ED. With increasing recrystallized grain size, the intensity shifted from the [2021] pole to the [1121] pole. Mackenzie et al. (2007) studied the texture evolution during hot extrusion and subsequent annealing of three Mg alloys, namely AZ31, ZC71, and WE43, and obtained similar observations. The extrusion texture of all three alloys exhibited a <1010> component on the IPF in the ED, which was attributed to the deformed unrecrystallized grains. After annealing (24h at 435 ℃ for AZ31 and ZC71, 24h at 525 ℃ for WE43), all microstructures were fully recrystallized. In the AZ31 and ZC71 alloys which represent conventional Mg alloys, the <2110> component became dominant on the IPF in the ED. In the WE43 alloy, which represents the RE containing Mg alloys, the maximum intensity was found between the [0001] pole and [1210] pole.

For rolled Mg alloys, the texture modification effect of RE elements was also reported. Mackenzie and Pekguleryuz (2008) studied the texture evolution during hot rolling and subsequent annealing in Mg-Zn and Mg-Zn-Ce alloys. Rolling was conducted at either 150 °C or 400 °C in order to reach different extents of recrystallization, and in both cases, a 400 °C annealing treatment followed in order to achieve a highly-recrystallized microstructure. For both alloys, after rolling at 150 °C without a subsequent annealing treatment, only a limited amount of recrystallization occurred. This led to the same type of texture in which the basal poles had a maximum intensity in the ND, splitting slightly toward the RD. After either rolling at 400 °C or annealing at 400 °C, both of which led to a highly recrystallized microstructure, the Mg-Zn alloy maintained the same texture as the 150 °C rolling texture, while the Mg-Zn-Ce alloy showed a relatively weak texture with the basal poles tilted ~45 ° from the ND toward the TD.

All the studies mentioned above show the importance of RE elements in texture modification of wrought Mg alloys. Basically, the deformation texture of both conventional Mg alloys and RE-containing Mg alloys are essentially the same, both having the basal poles nearly parallel to the ND. During recrystallization, conventional Mg alloys tend to retain the deformation texture, only with a possible rotation of the [1010] poles around the ND, while in RE-containing Mg alloys, the recrystallization texture has the basal poles tilted significantly away from the ND. It is generally considered that the RE elements have a strong grain boundary pinning effect and a strong interaction with dislocations, and can lead to preferential grain growth for certain orientations (Mackenzie and Pekguleryuz 2008, Stanford 2010, Bohlen *et al.* 2010). However, a detailed understanding of the mechanism that causes this has yet to be determined.

2.2 Fatigue Background

2.2.1 Fundamentals of Fatigue

Fatigue is the progressive damage of a material under a cyclic loading condition. A material should not fracture if a single tensile loading less than the UTS is applied. However, cyclic loads less than the UTS or the yield strength (YS) can lead to fatigue failure under certain conditions. Generally, the fatigue life can be divided into the crack initiation period (including crack nucleation and microcrack growth) and the crack growth period (which is related to macrocrack growth), as illustrated in Figure 2.7 (Schijve 2009).



Figure 2.7. Different periods of fatigue life and the dominant factors for each period (Schijve 2009).

Crack initiation is often considered to be a material surface phenomenon, because there is relatively low constraint on plastic deformation at free surfaces, and there is often stress concentration at material surface due to roughness, corrosion, or fretting, facilitating crack nucleation (Schijve 2009). However, internal flaws such as voids or inclusions can also provide a free surface or lead to stress concentration, and they can also act as crack initiation sites (Sanford 2002). It is widely accepted that for smooth specimens, cyclic slip occurs preferentially in surface grains that have orientations favorable for slip. Because cyclic slip is not fully

reversible, it leads to slip band extrusions out of the material or intrusions into the material, which can be considered as micro scale cracks. Upon further cyclic loading, slip and material decohesion at the crack tip cause the microcrack to grow gradually into a macro scale crack. Microcrack growth is dependent on the local stress state, grain orientation, and barriers, such as grain boundaries. Microcracks can become non-propagating in some conditions, for instance, when the local stress state changes during microcrack growth, or when the grain boundary barrier is too large to overcome (Schijve 2009). Macrocrack growth, on the other hand, is a continuous process along the crack front, and depends on the crack growth resistance as a bulk property of the material (Schijve 2009). The transition from the initiation period to the crack growth period cannot be quantitatively defined, rather, it is usually qualitatively defined as the point when microcrack growth is no longer affected by the material surface conditions (Schijve 2009).

As shown in Figure 2.7, the fatigue behavior at different stages of fatigue life is affected by different factors. The stress concentration factor (K_t) is the dominant parameter that affects crack initiation, the stress intensity factor (K) is an important parameter that affects crack growth, and the fracture toughness (K_{IC}) is the parameter to be considered when the material breaks in the last cycles of fatigue life. Basically, these factors are quantities obtained in analyzing the stress state within a material using linear elastic theory. When a notch, a sharp corner, or a hole exist in the material, there is an increase in the local stress field near these geometric discontinuities. The stress concentration factor, K_t , is defined as the ratio between the maximum local stress and the remote applied stress. For a crack with a sharp tip, linear elastic solutions predict that the stress increases to infinity at the crack tip, which is not the real case. However, the near field solution can be considered to be accurate. For a polar coordinate system (r, θ) with the origin at the crack tip, the stress field σ_{ij} near the crack tip has a similar form ('Stress Intensity Factor'):

$$\sigma_{ij}(r, \theta) = \frac{K}{\sqrt{2\pi r}} f_{ij}(\theta) + \text{higher order terms}$$
(2.7)

in which f_{ij} is a dimensionless quantity depending on the geometry and load. The factor *K* is defined as the stress intensity factor, and it provides an indication of the intensity of the stress field near the crack tip. A more general definition for the stress intensity factor of a mode I crack loaded in the y direction is that (Sanford 2002):

$$K_I = \lim_{\delta^+ \to 0} \sigma_y|_{\theta=0} \sqrt{2\pi\delta}$$
(2.8)

where δ represents the distance measured from the crack tip. The stress intensity factor is also used to evaluate the material's ability to resist brittle fracture. When fracture occurs, the critical value of the stress intensity factor is defined as the fracture toughness (*K*_{*IC*}, for mode I crack).

Fatigue crack growth is considered as a competition between intrinsic and extrinsic mechanisms (Ritchie 1999). The intrinsic mechanism, which is more important for ductile materials, refers to the microstructural damage ahead of the crack tip, such as slip or cleavage fracture, which promotes fatigue crack growth. The extrinsic mechanism, which is more important for brittle materials, refers to the shielding effect at the wake of the crack tip, such as crack deflection, grain or fiber bridging, and crack closure, which retards the fatigue crack growth (Ritchie 1999). Fatigue crack growth in ductile materials, such as metals, is generally explained as a progressive process of blunting and resharpening of the crack tip, leading to crack

extension (Ritchie 1999, Sanford 2002). This process on the micro scale involves dislocation motion and is therefore a plastic behavior. However, when the plastic zone ahead of the crack tip is very small, the stress intensity factor can still provide a meaningful indication of the severity of the stress field around the crack tip zone (Schijve 2009). In fact, the stress intensity factor, which is a parameter from linear elastic theory, has long been demonstrated to be an effective parameter for characterizing the fatigue crack growth behavior in metals.

Figure 2.8 shows an idealized three-stage fatigue crack growth curve. In the figure, da/dN represents the crack length extension per cyclic loading, i.e., the fatigue crack growth rate. ΔK represents the stress intensity factor range (the difference between the maximum and minimum value of stress intensity factor during one cycle of loading). For stage I, ΔK_{th} represents the threshold stress intensity factor range below which a macrocrack will stop growing. Stage III represents that when the maximum stress intensity factor (K_{max}) reaches a critical value K_{C} , complete failure will occur.

Extensive mathematical modeling has been performed on stage II fatigue crack growth, and such models are mainly based on phenomenological methods. One of the well-recognized empirical relationships is "Paris Law" (Paris and Erdogan 1963), suggesting a linear relationship between da/dN and ΔK in the log-log scale:

$$\frac{\mathrm{d}a}{\mathrm{d}N} = C(\Delta K)^m \tag{2.9}$$

Here, C and m are empirically determined constants for a given material in a given testing condition. The above relationship can only be applied to a fixed stress ratio (R value, which is defined as the ratio between the minimum stress and the maximum stress during one cycle of loading) and as such the constants C and m need to be determined as the R value is changed.

Other relationships have been proposed to incorporate the effect of stress ratio. A simple relationship was proposed by Walker (1970), in the following form:

$$\frac{\mathrm{d}a}{\mathrm{d}N} = C \left(K_{\mathrm{max}}\right)^p \left(\Delta K\right)^n \tag{2.10}$$

where the K_{max} can be related to *R* and ΔK by the relationship:

$$K_{\max} = \frac{\Delta K}{(1-R)} \tag{2.11}$$

Modifications to the Paris model, such as Equation (2.10), provide better fitting to the data. However, they also add more degrees of freedom to the fitted relationships.



Figure 2.8. An idealized fatigue crack growth curve.

2.2.2 Modeling for Small Fatigue Crack Growth

It has been shown that the empirical fatigue crack growth relationships based on LEFM, such as Paris Law, are generally inappropriate for small cracks. The small cracks can be defined as (1) microstructurally small (when their length is comparable to the characteristic microstructural dimensions, e.g., grain size), (2) mechanically small (when their length is comparable to the scale of local plasticity), or simply (3) physically small (e.g., smaller than 1mm) (Suresh and Ritchie 1984). The "small crack effect" was first discovered in aluminum (Al) alloys (Pearson 1975) and this effect refers to the phenomenon where small cracks can propagate faster than long cracks at equivalent LEFM driving force (ΔK). Thus, crack growth data obtained from long crack growth experiments may lead to a non-conservative lifetime prediction in the "defect-tolerant" engineering design approach.

Investigations by various researchers have suggested that other parameters might be applicable for characterizing the growth rate for small cracks. These include both the crack tip opening displacement (Tanaka *et al.* 1986) and plastic zone size (Edwards and Zhang 1994b, Shyam *et al.* 2004). Shyam *et al.* (2005) studied the small fatigue crack growth behavior in two cast Al alloys. They observed that small cracks propagate at ΔK values smaller than the long crack propagation threshold value, ΔK_{th} , and propagate faster than long cracks at an equivalent ΔK . Moreover, they found that increasing the maximum applied stress led to an increase in the small fatigue crack growth rate at an equivalent ΔK , which is a behavior not accounted for by LEFM relationships. It was then shown that the small fatigue crack growth rate can be linearly related to a new dislocation-based fracture mechanics parameter, which is defined as the product of the monotonic and cyclic crack tip displacement. The monotonic crack tip displacement (ϕ_{m}) is defined as:

$$\phi_{\rm m} = \frac{8\sigma_{\rm ys}(1-\nu^2)a}{\pi E} \ln(\sec(\frac{\pi\sigma_{\rm max}}{2\sigma_{\rm ys}})) \tag{2.12}$$

and the cyclic crack tip displacement (ϕ_c) is defined as:

$$\phi_{\rm c} = \frac{16\sigma_{\rm ys}(1-\nu^2)a}{\pi E} \ln(\sec(\frac{\pi\sigma_{\rm max}(1-R)}{4\sigma_{\rm ys}}))$$
(2.13)

where *v* is the Poisson's ratio, 2*a* is the surface crack length, and *E* is the Young's modulus (Shyam *et al.* 2005). In this new dislocation-based fracture mechanics parameter, defined as $\phi_{\rm m}\phi_{\rm c}$, the effects of mechanical loading conditions (such as $\sigma_{\rm max}$ and *R*) and material properties (such as *v* and $\sigma_{\rm ys}$) are both incorporated. It has been demonstrated that this new parameter can also be applied to other engineering alloys including titanium (Ti) alloys and nickel (Ni)-based superalloys (Shyam *et al.* 2007). However, the applicability of this parameter to Mg alloys is still not known.

2.3 Creep Background

Creep is defined as the time-dependent deformation of a material at a constant stress and temperature (Dieter 1986). For an engineering creep test, a constant load is usually applied rather than a constant stress (Dieter 1986). Figure 2.9 illustrates an idealized creep curve for a constant-load creep test, and the three stages can be observed on this curve. In reality, a creep curve with three well-defined stages can only be obtained under certain combinations of stress and temperature (Dieter 1986). The strain ε_0 corresponds to the instantaneous strain upon loading. The first stage of creep, known as the primary creep, has a decreasing creep rate with increasing strain. The second stage of creep, known as the secondary creep or steady-state creep,

has a nearly constant creep rate, which is due to the competition between the strain hardening and recovery processes. The average value of the creep rate at this stage, called the minimum creep rate, is an important design parameter. The third stage of creep, called tertiary creep, corresponds to a rapid increase of creep rate until final fracture of the material. The possible reasons for this increase in creep rates includes necking, void formation, coarsening of precipitates, recrystallization, and so on (Dieter 1986).

Based on stress and temperature, creep deformation is dominated by different mechanisms. Generally, the creep deformation mechanisms include dislocation glide, dislocation creep, diffusion creep, and GBS. Figure 2.10 shows a simplified deformation mechanism map (Ashby 1972) illustrating the dominant deformation mechanisms at certain combination of stress and temperature. The $T_{\rm m}$ refers to the melting temperature of the material. Several creep mechanisms can operate at the same time. If they operate in parallel, the fastest mechanism will have the largest contribution to the overall creep behavior. On the other hand, if different mechanisms operate in series, the slowest creep mechanism will dominate the creep behavior (Dieter 1986).



Time, t

Figure 2.9. An idealized creep curve for constant load creep test, showing the three stages.



Figure 2.10. A simplified deformation mechanism map (Ashby 1972).

In general, the steady-state creep rate ($\dot{\epsilon}_s$) at elevated-temperatures can be expressed in the form (Mukherje *et al.* 1969, Del Valle and Ruano 2007):

$$\dot{\varepsilon}_{\rm S} = A \, \frac{D \, G \, b}{k \, T} \left(\frac{b}{d}\right)^p \left(\frac{\sigma}{G}\right)^n \tag{2.14}$$

where *A* is a dimensionless constant, *G* is the shear modulus, *b* is the Burgers vector, *k* is the Boltzmann's constant, *T* is the absolute temperature, *d* is the average grain size, σ is the applied stress, *p* is the grain size exponent, and *n* is the stress exponent, *D* is the appropriate diffusion coefficient and $D = D_0 \exp(-\frac{Q}{RT})$, where D_0 is a frequency factor, *Q* is the activation energy for the deformation process, and *R* is the gas constant. For different creep mechanisms, the exponents (*p* and *n*) and activation energy (*Q*) exhibit different values. Note that there are some other forms of constitutive equations of steady-state creep rate similar to Equation (2.14), and the elastic modulus (*E*) is sometimes used instead of the shear modulus (*G*) (Ruano and Sherby 1988).

2.3.1 Dislocation Glide

The dislocation glide mechanism of creep usually occurs at a stress level $\sigma/G > 10^{-2}$, and this stress is relatively high compared to those usually considered in creep tests (Dieter 1986). In fact, this behavior can be viewed as thermally-activated deformation similar to high temperature tensile deformation, in which dislocation glide occurs by overcoming obstacles such as precipitates, solute atoms, or other dislocations (Dieter 1986).

2.3.2 Dislocation Creep

Dislocation creep occurs at $10^{-4} < \sigma/G < 10^{-2}$, and the mechanism is dislocation glide aided by thermally-assisted activities involving vacancy/interstitial diffusion (Dieter 1986). At elevated temperature, when the gliding of dislocations is impeded by an obstacle, it can be overcome by dislocation climb, which requires vacancy/interstitial diffusion (Dieter 1986). In this mechanism, the major strain is produced by the glide of dislocations, but the rate-controlling process is diffusion-controlled dislocation climb (Weertman 1968). A steady-state creep can be reached by the competition between dislocation glide, which is regarded as a work hardening process, and dislocation climb, which is regarded as a recovery process (Weertman 1968). In the dislocation creep regime, a grain size dependence is not expected, which means p = 0 in Equation (2.14) (Maruyama *et al.* 2002, Del Valle and Ruano 2007). In general, for intermediate to high stress levels and a temperature above ~0.5 $T_{\rm m}$, the steady-state creep rate for dislocation climb controlled creep can be described by the following power law relationship (Dieter 1986):

$$\dot{\varepsilon}_{\rm s} = \frac{A G b}{k T} \left(\frac{\sigma}{G}\right)^n D_0 \exp(-\frac{Q}{RT}) \tag{2.15}$$

Equation (2.15) can be re-arranged into a simpler form (Dieter 1986):

$$\dot{\varepsilon}_{\rm s} = B \ \sigma^n \exp(-\frac{Q}{kT}) \tag{2.16}$$

which is in the same form as the relationship proposed by Dorn (Dorn 1954).

For pure metals and alloys that have similar creep behavior as pure metals, the value of *n* is generally around 5, and *Q* approximately equals the lattice self-diffusion energy (Dieter 1986, Kassner 2009a). However, at low stress levels, below $\sigma/G = 5 \times 10^{-6}$ (Dieter 1986), a linear dependence of the steady-state creep rate on the applied stress, i.e., *n*~1, is observed, and this is

known as Harper-Dorn creep (Harper and Dorn 1957). The activation energy for Harper-Dorn creep is also about that of the lattice self-diffusion (Kassner 2009a). The detailed mechanism of Harper-Dorn creep is not fully understood, and it is possibly a mechanism related to dislocation climb (Dieter 1986, Kassner 2009a). At high stress levels, $\sigma/G > 10^{-3}$, the stress exponent increases rapidly with increasing stress, and this is referred to as the power-law-breakdown (PLB) (Dieter 1986). Within PLB, the activation energy generally decreases as n increases, and it was suggested that although creep is still dislocation climb controlled, the activation energy corresponds to that of dislocation pipe diffusion (Kassner 2009a).

2.3.3 Diffusion Creep

Diffusion creep refers to the creep deformation by vacancy or interstitial flow under the influence of applied stress. It is the dominant creep mechanism at high temperatures and low stresses ($\sigma/G < 10^{-4}$) (Dieter 1986). It includes Nabarro-Herring creep (Nabarro 1948, Herring 1950) and Coble creep (Coble 1963).

Nabarro and Herring proposed a lattice diffusion controlled model in which the creep deformation occurs by stress-directed diffusion of atoms within grains (Nabarro 1948, Herring 1950). When stress is applied to a polycrystal material, the chemical potential of atoms on the surface of grains will change. As a result, vacancies flow from the grain boundaries which are under tensile stress to the grain boundaries which are under compressive stress, i.e., from grain boundaries perpendicular to the tensile direction to those parallel to the tensile direction. Equivalently, an atomic flow is in the opposite direction, and the grains are elongated along the tensile direction (Dieter 1986, Perez-Prado and Kassner 2009). The steady-state creep rate can be expressed as (Dieter 1986):

$$\dot{\varepsilon}_{\rm s} = \frac{14\sigma b^3 D_{\nu}}{k T d^2} \tag{2.17}$$

where D_{v} is the lattice diffusion coefficient.

Coble (1963) suggested a boundary diffusion controlled creep model in which the diffusion of atoms is along grain boundaries instead of interior of grains, as in the condition of Nabarro-Herring creep. This mechanism applies to a lower temperature, when the contribution of grain boundary diffusion is larger than that of lattice diffusion. The steady-state creep rate of Coble creep is expressed as (Dieter 1986):

$$\dot{\varepsilon}_{\rm s} = \frac{50\sigma b^4 D_{\rm gb}}{k T d^3} \tag{2.18}$$

where $D_{\rm gb}$ is the grain boundary diffusion coefficient.

2.3.4 GBS

GBS refers to the relative movement of neighboring grains in a polycrystalline material, leading to a shear at the grain boundary (Dieter 1986). It is suggested that GBS must be accommodated by other deformation modes, such as diffusion creep or dislocation creep (Hertzberg 1989). This accommodation process is independent of the GBS process, and the slower one will be the rate controlling process for creep deformation (Ruano and Sherby 1988). When GBS is lattice diffusion controlled, the steady-state creep rate can be expressed as (Ruano and Sherby 1988):

$$\dot{\varepsilon}_{\rm s} = 6.4 \times 10^9 \frac{D_{\rm L}}{d^2} (\frac{\sigma}{E})^2 \tag{2.19}$$

When GBS is grain boundary diffusion controlled, the steady-state creep rate can be expressed as (Ruano and Sherby 1988):

$$\dot{\varepsilon}_{\rm S} = 5.6 \times 10^8 \frac{D_{\rm gb}}{d^3} \left(\frac{\sigma}{E}\right)^2 \tag{2.20}$$

An increase in the temperature and/or decrease in the strain rate will promote GBS (Dieter 1986). A decrease in the grain size will enhance the GBS mechanism relative to slip creep (Del Valle and Ruano 2007). The observation of GBS is usually achieved by scribing a line on the polished surface of a creep specimen, then observing the shear offset of the lines across grain boundaries after the creep deformation (Dieter 1986).

2.3.5 Creep Stress Exponents and Apparent Activation Energies

In Equation (2.14), n is called the creep stress exponent, which describes the variation of the steady-state creep rate with the change in applied stress. Q is called the apparent activation energy, which describes the variation of the steady-state creep rate with change in temperature. A more general definition of the stress exponent is that (Kassner 2009b):

$$n = \left(\frac{\partial \ln \dot{\varepsilon}_{\rm S}}{\partial \ln \sigma_{\rm S}}\right)_T \tag{2.21}$$

where σ_s is the stress corresponds to the steady-state creep rate $\dot{\varepsilon}_s$. A more general definition of the activation energy is that (Kassner 2009a):

$$Q = -k \left(\frac{\partial \ln \dot{\varepsilon}_{s}}{\partial (1/T)}\right) \sigma_{s}/G$$
(2.22)

for which it is required that the microstructure for specimens tested at different temperatures should be approximately the same. The experimentally determined value of n and Q are used to

predict the active secondary creep mechanism for single phase pure metals and some alloys. The n value can be calculated by fitting $\dot{\varepsilon}_s$ and σ in the log-log scale and measuring the slope of the fitted curve. The Q value is generally determined by assuming a simple Arrhenius-type relationship (Dieter 1986):

$$\dot{\varepsilon}_{\rm s} = A \, \exp(-\frac{Q}{RT}) \tag{2.23}$$

And Q is calculated from the slop of a linear curve fit of $\ln \dot{\varepsilon}_{s}$ versus $\frac{1}{T}$

In general, the value of n, p, and Q in Equation (2.14) for different creep mechanisms can be summarized as follows (Ruano and Sherby 1988):

For dislocation creep, p = 0, and $n = 1 \sim 8$ depending on the specific dislocation mechanism. Generally, *n* is around 5 for diffusion controlled dislocation climb. For Harper-Dorn creep, n = 1. The activation energy *Q* for dislocation creep generally equals to that of lattice self-diffusion.

For diffusion creep, n = 1. For Nabarro-Herring creep, p = 2 and $Q = Q_L$. For Coble creep, p = 3 and $Q = Q_{gb}$.

For GBS creep, n = 2. When GBS is controlled by lattice diffusion, p = 2, and $Q = Q_L$ (lattice self-diffusion energy). When GBS is controlled by grain boundary diffusion, p = 3, and $Q = Q_{gb}$ (grain boundary diffusion energy).

2.4 Thixomolding® of Mg Alloys

Semisolid metal processing is a relatively new technique for metal production, and it was firstly studied at MIT in the 1970's (Spencer 1971). Based on this original work, three commercialized routes, known as semisolid forging, semisolid billet casting, and Thixomolding®, have been developed in following years (Walukas *et al.* 1997). Among the three developments, Thixomolding® is the only one that has been commercialized for the production of Mg alloy parts. The baseline AM60 alloy studied in this dissertation work was produced by the Thixomolding® method.

Figure 2.11 illustrates a schematic of this technique. During processing, Mg alloy granules are fed through a volumetric feeder into a heated barrel, which contains a protection atmosphere of argon. Inside the barrel, the granules are heated to a temperature of 560-630 $^{\circ}$ C (Decker and LeBeau 2008) and brought into semi-solid state. A screw rotates and propels the material forward. The shear force provided by the screw breaks the dendrites in the root solid particles and rounds them. This creates a thixotropic slurry in which spheroidal solid particles are distributed in a liquid matrix. The slurry is accumulated through a non-return value, and finally injected into a preheated mold to produce a net or near-net shape part (Walukas *et al.* 1997).

Compared with traditional die-casting, Thixomolding® has many advantages, such as decreased turbulence and lower temperature in the molding process, which can lead to lower porosity and an associated increase in ε_f , as well as lower shrinkage and thus higher dimensional stability (Decker and LeBeau 2008, Walukas *et al.* 1997). It is also more environmental friendly, because the production process does not require a SF₆ cover gas. In comparison, the SF₆ used in the die casting process exhibits a strong greenhouse effect which is 23,900 times that of an

equivalent weight of CO_2 (Edgar 2006). Due to the relatively low processing temperature and high cooling rate, a fine grain size can also be obtained, which potentially increases both the strength and formability of the material.



Figure 2.11. Schematic of a Thixomolding® machine (Friedrich and Mordike 2006).

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Material

3.1.1. AM60 Alloy

Mg-6Al-0.3Mn (AM60) alloy was provided by nanoMAG, LLC (Ann Arbor, MI). Three different processing conditions were applied to the AM60 alloy: (1) as-Thixomolded® (as-molded), (2) Thixomolded® and then thermomechanically processed (TTMP), and (3) Thixomolded®, thermomechanically processed, and then annealed (annealed). The as-molded materials were Thixomolded® plates of 3.2mm thickness. Some of the as-molded materials were then thermomechanically processed (TMP) by one pass of warm rolling at ~573K (300 °C) with a reduction ratio of 0.5 (which corresponded to a final thickness of 1.6 mm), and this material is referred to as in the TTMP condition. Subsequently, some of the TTMP plates were annealed at ~573K (300 °C) for ~30 minutes, and this material is referred to as in the annealed condition. It is important to note that both the production and heat treatment of this alloy were conducted at nanoMAG, LLC (Ann Arbor, MI), and due to proprietary reasons, the exact processing temperature and time was not provided by the company. The measured bulk chemical composition of the as-molded AM60 alloy was Mg-6.29Al-0.28Mn-0.05Zn-0.001Fe-0.02Si-0.0009Ni-0.0007Be (wt.%).

3.1.2. AZ31 Alloy

Rolled and annealed Mg-3Al-1Zn (AZ31) sheet of 3mm thickness, was purchased from Magnesium Elektron (Manchester, UK). The bulk chemical composition of the as-rolled
material, measured using an Inductively Coupled Plasma Analyzer, was Mg-2.9Al-0.73Zn-0.2Mn(wt.%).

3.1.3. MN11 Alloy

The Mg-1Mn-1Nd (MN11) material was provided by Magnesium Innovation Center MagIC (Geesthacht, Germany). It was gravity cast to produce a billet of original diameter equal to 125 mm. The billet was machined to a diameter of 93mm in preparation for extrusion. The billet was homogenized at 350 $\$ for 15 hours prior to extrusion. Indirect extrusion was carried out at 300 $\$ to produce a round bar with a diameter of 17 mm, which corresponded to an extrusion ratio of 1:30. The extrusion was performed with a ram speed of 5.6 mm/s, which corresponded to a profile exit speed of 10 m/min. The total length of the extruded rod was 1.5 m. The as-extruded rod exhibited a weak texture. The bulk chemical composition of the as-extruded rod, measured using spark emission spectroscopy, was Mg-0.95Mn-0.94Nd-0.04Al-0.01Si-0.008Zn-0.007Ce (wt.%).

3.2 Microstructure Characterization

3.2.1 Metallography Preparation

Metallographic samples of AM60 alloy were prepared for imaging using optical microscopy and SEM. Three sections of the AM60 sheets, represented as face, longitudinal, and transverse, as illustrated in Figure 3.1, were characterized. The sections were cut from the sheets using a Buehler IsometTM low speed saw with a diamond wafering blade, and mounted in KonductometTM compression mounting compounds using a Buehler Pneumet®-I mounting press, forming 25.4mm-diameter mounts. The mounts were ground successively using silicon

carbide (SiC) paper of American National Standards Institute (ANSI) standard 60 grit, 240 grit, 400 grit, 600 grit, and then polished successively using diamond paste of 6 μ m, 3 μ m, 1 μ m, and 0.25 μ m grain size. Ethanol was used as a lubricant during the grinding and polishing process. Some of the polished samples were etched using Nital solution (5% volume percent nitric acid + 95% volume percent ethanol) or Acetic-Picral solution (4.2 g picric acid + 5 ml acetic acid + 10 ml H₂O + 70 ml ethanol) in order to reveal the microstructure.



Long-transverse/transverse direction

Figure 3.1. Schematic illustrating the sections of the AM60 sheets characterized.

3.2.2 Microscopy

Scanning electron microscopy was used for the microstructure analysis and the fracture surface analysis for mechanically tested specimens. The SEMs used in this dissertation work include a JEOL 6500 field emission SEM, and a Hitachi S4800 field emission SEM, both of which were located at Oak Ridge National Laboratory (Oak Ridge, TN), and a JEOL 7500F field emission SEM, a Carl Zeiss EVO LS25 Variable Pressure SEM, and a TESCAN Mira3 field emission SEM, and these latter three were located at Michigan State University (MSU, East Lansing, MI).

Secondary electron (SE) SEM images were taken to depict the surface topography of the samples, while backscattered electron (BSE) SEM images were taken to depict the difference in atomic number in the sample. The phase volume fraction in the AM60 alloy was measured using ImageJ software on several BSE SEM images taken for each section of the sheet. This software can be used to shade pixels that show a brightness value within a set range. On the BSE SEM images, different phases were represented by pixels with different brightness. The fraction of the pixels corresponding to one phase, among the total number of pixels, was used to represent the phase volume fraction. The pore volume fraction in the AM60 alloy was evaluated using the same method.

Transmission electron microscopy analysis was performed using either a Tecnai 20 TEM operated at 200 kV or a Hitachi HF-3300 TEM operated at 300 kV. Both TEMs were located at Oak Ridge National Laboratory. The TEM foils were prepared by polishing the samples to a thickness between 50 and 100 μ m, then thinning them using a Fischione model 1010 ion miller/polisher at 3 keV and 3 to 5 mA at a 15 °incident angle until perforation at RT.

Optical microscopy was performed using a Nikon Model Epiphot 200 optical microscope located at MSU.

3.2.3 Microprobe Analysis

Microprobe analysis was performed on mounted and polished AM60 samples using a JEOL JXA-8200 wavelength dispersive/energy dispersive (WD/ED) combined microanalyzer operated at 10 kV and an beam current of 46 nA. The weight percentages of Mg, Al, Mn, and Si

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in the individual phases were measured. The low accelerating voltage was chosen to minimize the overlap between the matrix and precipitate phases. In this technique, the minimum detectable level of Si was estimated to be 0.01wt%, while for Mg, Al, and Mn the minimum detectable level was estimated to be 0.1-0.2wt.%. This analysis were performed at Oak Ridge National Laboratory.

3.2.4 EBSD Analysis

EBSD was performed on the gage section of *in-situ* test specimens. The EBSD scans were performed in a JEOL 6500F field emission SEM at Max Planck Institute for Iron Research (Düsseldorf, Germany), and a Leo 1455VP SEM equipped with a EDAX-TSL (Mahwah, NJ) EBSD system located at General Motors Technical Center (Warren, MI), using a step size of 1µm. The sample preparation for EBSD included grinding successively with SiC paper of ANSI 400 grit, 600 grit, 800 grit, and 1200 grit size, mechanical polishing using a water-free colloidal silica solution, and final electrochemical polishing for 60 seconds at 20 V using the AC2TM commercial electrolyte.

3.2.5 XRD

X-ray diffraction (XRD) 2θ scans were performed using a rotating anode Powder-Texture-Stress (PTS) goniometer (Scintag, Inc. Sunnyvale, CA) with copper K_{α} radiation, for a 2θ range of 30-80°. XRD pole figures on a 5° by 5° grid were acquired using the same PTS goniometer with a copper K_{α} radiation over a tilt (chi) range from 0-75°. The XRD analysis was performed at Oak Ridge National Laboratory.

3.2.6 AFM

AFM was performed using a Dimension 3100 scanning probe microscope located at Michigan State University (East Lansing, MI). For each scan, the scanned area was 30 μ m x 30 μ m. The scan range in the height was limited to ~5 μ m. The scans were performed in tapping mode using a TESP sharp silicon probe (Bruker Nano Inc., Camarillo, CA) in order to minimize the damage to the sample surface. The AFM scan data was collected and processed using NanoScope® III (version 5.31R1) software (Digital Instruments, Tonawanda, NY).

3.3 Mechanical Testing

3.3.1 Sample Configuration

3.3.1.1 Conventional Tensile, Fatigue, and Creep Samples

Flat dogbone samples with a gage dimension of 25 mm length by 13 mm width were used for conventional mechanical testing including tensile, fatigue, and creep testing. The samples were electrodischarge machined (EDM) from the AM60 sheets, all with the tensile axis parallel to the rolling/longitudinal direction of the sheets, as illustrated in Figure 3.2. Figure 3.3 shows a photo of the sample, and Figure 3.4 shows the detailed dimension of the sample. The thickness of the as-machined samples was the same as the thickness of the as-processed sheets. However, before testing, all samples were polished using 600 grit SiC paper in order to remove the surface recast layers, and to reach a sample thickness that fits the testing fixtures. In general, the thickness of the polished samples was about 1-1.5mm.



Figure 3.2. Schematic illustrating the machining of the conventional mechanical testing samples.



Figure 3.3. A photo of the sample for conventional tensile and fatigue testing.



Figure 3.4. Schematic showing the dimension of the conventional mechanical testing samples.

3.3.1.2 Fatigue Crack Growth Samples

Flat dogbone samples with the same geometry as those used for conventional fatigue test were used for fatigue crack growth experiments. All samples were machined with the tensile axis parallel to the longitudinal direction of the sheets. In order to control the crack initiation site, a small notch was machined on the surface in the middle of the gage section of the sample, using a flat bottom drill. The notch was only partly through the thickness of the sample, with the depth of about 150-400 μ m. The length of the notch was typically 400-800 μ m. The longer notches were machined for the as-molded AM60 specimens with the largest pre-existing defects. A photo illustrating the gage section with the artificial notch of the fatigue crack growth sample is shown in Figure 3.5. Before testing, the sample surface was polished using SiC paper and diamond paste to a finish of 1 μ m.



Figure 3.5. A photo of the gage section of the fatigue crack growth test sample. The artificial notch is in the middle part of the gage section.

3.3.1.3 In-situ Test Samples

For the *in-situ* tensile and creep tests, dogbone samples with a gage dimension of 3 mm width by 10 mm length were used. The samples were EDM cut both along the longitudinal and transverse directions of the sheet material, and along the longitudinal/extrusion direction of the extruded bars, as illustrated in Figure 3.6. Figure 3.7 shows a photo of an *in-situ* test sample, and the detailed dimension of the samples are shown in Figure 3.8. Before testing, samples were polished using SiC paper and diamond paste to a finish of 1 μ m, which enabled them to be analyzed using SEM. For samples which were analyzed using EBSD, the preparation methods described in Section 3.2.4 were used.



Longitudinal/rolling direction

Longitudinal/extrusion direction

Figure 3.6. Schematic illustrating the cutting of the *in-situ* tensile and creep samples. Note that the samples cut from the extruded bar were typically around 1.5 mm thick.



Figure 3.7. A photo of an *in-situ* tensile and creep test sample.



Figure 3.8. Schematic showing the dimension of the *in-situ* tensile and creep test samples.

3.3.2 Conventional Mechanical Testing

3.3.2.1 Tensile Testing

Conventional uniaxial tensile tests were performed on AM60 alloy in an air environment at RT and 150 °C, using an MTS® servo-hydraulic testing machine equipped with a MTS® Flex Test SE controller. The test setup is shown in Figure 3.9. The samples were placed horizontally and fixed by screw-driven friction grips. The tests were performed using a displacement rate of 0.0254 mm/s, which corresponded to a strain rate of 10^{-3} /s. The strain was measured by an alumina-arm high-temperature extensometer with a 12 mm gage length, attached to the gage section of the sample. For elevated temperature tests, the gage section of the sample was heated by eight quartz lamps placed ~1 cm above and below the sample, while the grip section of the sample was cooled by the friction grips which had cooling water running through them. The temperature was monitored by four thermocouples attached to the gage section of the sample, and was kept within ±5 °C of the target temperature by adjusting the power supply to the quartz lamps. A 20-minute period was used to stabilize the thermal stress prior to applying load. The time, displacement, strain, load, stress, and temperature were recorded throughout the tests. A minimum of two tests were performed for each condition.



Figure 3.9. A photo showing the setup of the conventional tensile testing.

3.3.2.2 Fatigue Testing

Fatigue tests were performed on the AM60 at RT and 150 °C in an air environment using the same testing system as the one used in conventional tensile testing (Section 3.3.2.1). The tests were conducted by load control. The stress ratio was R = 0.1 and the frequency was 5 Hz. The samples were tested under a number of stress levels, with the maximum applied stress ranging from 50 to 175 MPa. The runout condition was considered to be 10⁶ cycles. The time, displacement, strain, load, stress, and temperature were recorded at desired intervals during the tests. A minimum of three tests were performed for each condition.

3.3.2.3 Creep Testing

Conventional tensile creep tests were performed using vertical load frames manufactured by Applied Test System, Incorporated (ATS, Butler, PA). A testing frame is shown in Figure 3.10. The test temperatures ranged between 100 and 200 $^{\circ}$ C, and the applied stresses ranged between 20 and 75 MPa. Constant load was applied by the weights through a 20:1 ratio lever arm. Because the cross section area did not change significantly in most of the time during the creep test, the stress was considered to be constant. The temperature was monitored by chromelalumel type K thermocouples, two of which were spot-welded to the gage section of the samples, and an additional one pressed lightly onto the gage section of the sample. The power supply to the furnace was adjusted to keep the temperature within $\pm 3 \,^{\circ}$ C of the target temperature. Before loading, a 60-minute period was given to stabilize the thermal stresses. The strain was measured by a linear variable differential transformer (LVDT) on an ATS high-temperature extension with a 25.4 mm gage length attached directly to the gage section of the specimen. After a steady-state strain rate was achieved, either the load or the temperature was increased or the test was stopped. Upon terminating the creep tests, the samples were maintained under load while the temperature was decreased in order to retain the deformed state of the sample. The time, load, and displacement were documented at desired intervals during the creep test.



Figure 3.10. A photo of an ATS creep test frame used in this work.

3.3.3 Fatigue Crack Growth Testing

Fatigue crack growth experiments were performed using an MTS servo-hydraulic testing system equipped with a 407 controller. The experimental setup is shown in Figure 3.11. The samples were cyclically loaded under load control, and the cracks emanating from the artificial notch were directly observed with the aid of a Questar® long working-distance microscope focused on the specimen surface. Depending on the growth rate of the crack, the tests were paused at desired intervals to capture images of the crack. During imaging, the samples were

loaded at the mean stress of the cyclic stress to enable a proper opening of the crack. In order to evaluate the effect of the mechanical loading parameters, different maximum applied stress and stress ratios were applied. The maximum applied stress ranged between 60% and 90% of the yield strength of the studied material. Different stress ratios, R = 0.1, 0.3, and 0.5, were applied to different specimens. All the fatigue crack growth tests were performed at RT and a frequency of 5 Hz. Most of the samples were tested to failure. The fracture surfaces of the tested samples were examined using a Carl Zeiss EVO LS25 Variable Pressure SEM or a TESCAN Mira field emission SEM. The fatigue crack growth tests were performed at Oak Ridge National Laboratory, while the SEM observation was performed at MSU.



Figure 3.11. Experimental setup of the fatigue crack growth test.

3.3.4 In-situ Mechanical Testing

3.3.4.1 In-situ Tensile Testing

In-situ tensile tests were performed on the AZ31 and MN11 alloys using a screw-driven tensile stage built by Ernest F. Fullam, Inc. (this company was recently bought by MTI Instruments, Inc., Albany, NY) placed inside a Carl Zeiss EVO MA15 SEM. A photo of the *in*situ tensile test stage is shown in Figure 3.12. The tests were performed at 50 $^{\circ}$ C, 150 $^{\circ}$ C, and 250 ℃. The temperature was monitored by a fine-gauge K-type thermocouple spot-welded to the gage section of each sample. The heating element was a 6-mm diameter tungsten-based heater located below the gage section of the sample, operated by a constant-voltage power supply. An open-bath, closed-loop chiller was used to circulate cooling water through copper tubes to prevent the tensile stage from overheating. The temperature was maintained within 5 $^{\circ}$ C of the targeted temperature. A 30 minute period was used to stabilize the thermal stress prior to testing. The tests were performed at a constant displacement rate of 0.004 mm/s, which corresponded to a strain rate of $\sim 10^{-3}$ /s. The surface deformation process was monitored throughout the test, and the loading was paused at several strain levels in order to take SEM images of the sample. The time, load, and crosshead displacement were recorded and used to estimate the stress-strain relationship. It should be noted that the crosshead displacement value included that of the sample as well as the gripping fixtures, thus did not represent the actual displacement of the reduced gage section of the sample. The experiments were usually terminated after ~25% total strain was achieved. The width and thickness of the tested samples were measured and compared to those values measured before testing, and were used to evaluate the plastic anisotropy.



Figure 3.12. A photo of the *in-situ* tensile test stage.

3.3.4.2 In-situ Creep Testing

In-situ creep experiments were performed on the annealed AM60 alloy and AZ31 alloy, at 150 °C, using the same equipment as described in Section 3.3.4.1. The tests were performed using load control. The load was applied at ~5 N/s until reaching the desired creep stress, and then was kept at a nearly constant value. The fluctuation of stress was within \pm 5 MPa of the targeted stress during the tests. SEM images were taken at periodic displacement values throughout the creep tests without interrupting the experiment. The pressure in the SEM chamber never exceeded 10⁻⁴ Pa during the experiment, therefore oxidation did not detrimentally affect the SEM imaging.

CHAPTER 4

RESULTS

This chapter presents the experimental results for each of the studied materials, namely AM60, AZ31, and MN11. It is divided into three sections, each of which presents experimental results for one of the studied alloys. For the AM60 alloy, microstructure analysis and different types of mechanical testing were performed in order to get an understanding of the processing-microstructure-property relationships. For the AZ31 and MN11 alloys, *in-situ* mechanical testing and EBSD analysis were performed in order to investigate the deformation mechanisms. AFM was also performed on AZ31 in order to evaluate the GBS activity.

4.1 AM60 Alloy

4.1.1 Microstructure Analysis

4.1.1.1 Microstructure

Figure 4.1 illustrates BSE SEM images of the face, longitudinal, and transverse sections of the as-molded, TTMP, and annealed materials. The longitudinal direction (LD), normal direction (ND), and transverse direction (TD) of the sheet are indicated by the arrows. The equilibrium phases in the AM60 alloy at RT include α -Mg solid solution, γ -Mg₁₇Al₁₂, and Mn₅Al₈ (Gertsman *et al.* 2005). As indicated by the arrows in Figure 4.1(a), the dark matrix phase was the Mg-rich α -phase, which has a hexagonal crystal structure. The brightest particles, which were located throughout the microstructure and ranged in size from 50 nm to 10 µm, were the Mn₅Al₈ phase, which has a rhombohedral crystal structure (Gertsman *et al.* 2005). The

phase with intermediate brightness, which is typically of irregular shape and located within the medium-contrast grain boundary network, was the Mg₁₇Al₁₂ γ -phase, which exhibits a cubic crystal structure. The medium-contrast network observed at the grain boundaries was likely to be an divorced eutectic (α -Mg + γ -Mg₁₇Al₁₂). Figure 4.2 shows a higher-magnification BSE SEM image of an annealed sample, illustrating the difference in contrast between the Mg₁₇Al₁₂ phase and the Mn₅Al₈ phase. Figure 4.3 shows a bright-field TEM image of the as-molded sample. Energy dispersive X-ray spectroscopy (EDS) was performed in four different regions. Region 1, located at the grain boundary, contained both Mg and Al, and is likely to be the Mg₁₇Al₁₂ γ -phase. Region 2 and 3 corresponded to a precipitate located in the grain interior. Apart from Mg, it contained both Al and Mn, so it was likely to be the Mn₅Al₈ phase. Region 4 was located within the grain, and it contained mostly Mg, therefore it corresponded to the Mg matrix.



(a) as-molded



Figure 4.1. BSE SEM images illustrating the microstructure of different sections the AM60 sheets processed under different conditions. (a) as-molded, (b) TTMP, and (c) annealed. Note that the rolling direction was parallel to LD.

Figure 4.1 (cont'd)



(c) annealed



Figure 4.2. BSE SEM image of the annealed material illustrating the difference between the $Mg_{17}Al_{12}\gamma$ -phase (gray) and the fine Mn_5Al_8 precipitates (bright).



Figure 4.3. A bright-field TEM image of the as-molded material (a) with EDS data (b)-(e) taken from the four labeled regions.

Table 4.1 lists the average volume percentage of the studied AM60 alloys, as measured from the BSE SEM images. For each processing condition, a minimum of ten images were measured for the phase volume percentage. The α -matrix encompassed ~90% of the microstructure by volume. The grain boundary phase encompassed ~7% to ~10% of the microstructure by volume. The "grain boundary phase" refers to the medium-contrast network observed at the grain boundaries. This might include the γ -phase as well as the α -phase in the eutectic microstructure, as no distinctive boundaries could be resolved between them based on the BSE SEM images used for this analysis. It is pertinent to note that after annealing, the volume fraction of the grain boundary phase decreased slightly from ~10% to ~7% and the volume fraction of the grain boundary phase diffuse into the matrix during the annealing treatment. The measured volume fraction of Mn₅Al₈ precipitates was ~1% in each material.

Table 4.1. Average phase volume percentage of the studied AM60 alloys (standard deviation in parentheses).

	Matrix (α)	Grain boundary phase	Mn ₅ Al ₈ particle
As-molded	88.7 (1.8)	10.0 (1.7)	1.2 (0.3)
TTMP	88.4 (1.4)	10.4 (1.3)	1.2 (0.2)
Annealed	92.1 (1.7)	6.8 (1.9)	1.1 (0.3)

The microstructure of the as-molded sample is shown in Figure 4.1(a). The average equiaxed grain diameter was ~10 μ m. However, there was a bimodal grain size distribution as some of the α solid particles remained solid in the thixomolder during the Thixomolding® operation, and these resulted in grains significantly larger than 10 μ m. Some of these large grains can be easily distinguished in Figure 4.1(a). Some pores can be observed in the as-molded material, as indicated in Figure 4.1(a). Moreover, as shown in Figure 4.4(a), these pores tended to distribute in two clusters along the transverse/longitudinal direction of the sheets, forming two porosity segregation bands located at $\sim \frac{1}{3}$ and $\sim \frac{2}{3}$ of the thickness. The pore volume fraction in the as-molded material, measured from Figure 4.4(a), was ~2%. In comparison, for the TTMP and annealed material, such high porosity was not observed, see Figure 4.4(b) and (c).

The microstructure of the TTMP sample is illustrated in Figure 4.1(b). The grains were severely deformed and elongated along the rolling/longitudinal direction, and contracted along the ND. The average grain size was similar to that of the as-molded material.

Figure 4.1(c) illustrate the microstructure of the annealed sample. As mentioned earlier, the annealing treatment appeared to have reduced the amount of the grain boundary phases in the material, as there was much less contrast apparent near the α -phase grain boundaries. Optical microscope images of etched samples revealed some other details of the microstructure that was not observed from the BSE SEM images. Figure 4.5 shows the optical microscope images of the AM60 alloys processed under different conditions. As shown in Figures 4.5(a) and 4.5(b), some large grains can be observed in the as-molded material and TTMP material. After annealing, some smaller, equiaxed grains were observed, and they occupied some regions that used to be occupied by a large grain, such as the one shown on the left-hand-side of Figure 4.5(c). Therefore, it is likely that static recrystallization (SRX) had occurred, leading to grain nucleation

and grain growth during the annealing treatment. However, the recrystallization was not complete. Figure 4.5(d) illustrates another large grain in an annealed sample, in which only a small part appeared to have recrystallized. The average grain size of the annealed material was $\sim 5 \mu m$, which is smaller than that of the as-molded material.



(a) as-molded, transverse section



(b) TTMP, longitudinal section

Figure 4.4. BSE SEM images of the AM60 alloy comparing the porosity in two processing conditions. (a) as-molded, transverse section, (b) TTMP, longitudinal section, and (c) annealed, longitudinal section.

Figure 4.4 (cont'd)



(c) annealed, longitudinal section



(b) TTMP, longitudinal

Figure 4.5. Optical microscope images of the AM60 alloys processed under different conditions. (a) as-molded, longitudinal section, (b) TTMP, longitudinal section, (c) annealed, face section, and (d) annealed, face section.



(d) annealed, face

4.1.1.2 Microprobe Analysis

Microprobe analysis was performed on mounted and polished AM60 samples of each processing condition, in order to evaluate the weight percentage of each of the different elements, including Mg, Al, Mn, and Si, in different phases. The microprobe analysis data is shown in Table 4.2. The matrix α -phase was enriched with Mg. The measurements of the grain boundary γ -phase showed a higher Mg/Al ratio than the normal ratio of Mg₁₇Al₁₂. However, a similar result was found in a work elsewhere (Wang *et al.* 2003) and this was suggested to be a result of the interaction volume encompassing both this grain-boundary phase and the matrix α -phase. The composition of the brightest phase was roughly consistent with Mn₅Al₈. It is noted that all the reported measurements for the Mn₅Al₈ phase were taken from the largest precipitates, which were on the order of 10 µm in diameter. However, previous research on an AM50 alloy showed that nanoparticles were also present with this composition (Gertsman *et al.* 2005). Thus, it is likely that the nanoparticles in the current work, such as the bright precipitates in Figure 4.2, were also Mn₅Al₈.

AM60 Alloy Condition	Phase	Mg	Al	Mn	Si
as-molded	Matrix (α)	94.1 ± 1.5	5.8 ± 1.5	0 ± 0	0 ± 0
		(94.7 ± 1.3)	(5.3 ± 1.3)	(0 ± 0)	(0 ± 0)
	$Mg_{17}Al_{12}(\gamma)$	75.4 ± 2.1	24.4 ± 2.1	0 ± 0	0.2 ± 0.3
		(78.4 ± 2.0)	(21.2 ± 2.0)	(0 ± 0)	(0.3 ± 0.3)
	Mn_5Al_8	2.7 ± 1.5	38.8 ± 1.1	57.8 ± 1.5	0.7 ± 0.1
		(5.3 ± 2.3)	(54.6 ± 1.6)	(39.2 ± 1.5)	(0.9 ± 0.1)
TTMP	Matrix (α)	94.4 ± 1.4	5.5 ± 1.4	0 ± 0	0 ± 0
		(95.0 ± 1.3)	(5.0 ± 1.3)	(0 ± 0)	(0 ± 0)
	$Mg_{17}Al_{12}(\gamma)$	73.6 ± 4.3	25.9 ± 4.8	$0.4\ \pm 0.9$	0.1 ± 0.1
		(75.7 ± 4.3)	(24.0 ± 4.5)	(0.2 ± 0.4)	(0.1 ± 0.1)
	Mn_5Al_8	1.2 ± 2.3	39.1 ± 1.3	59.7 ± 3.3	0.6 ± 0.1
		(1.6 ± 3.5)	(55.5 ± 1.1)	(42.1 ± 3.2)	(0.9 ± 0.1)
annealed	Matrix (α)	94.6 ± 1.6	5.4 ± 1.6	0 ± 0	0 ± 0
		(95.1 ± 1.5)	(4.9 ± 1.5)	(0 ± 0)	(0 ± 0)
	$Mg_{17}Al_{12}(\gamma)$	71.1 ± 9.1	$28.0~\pm 9.5$	$0.5\ \pm 0.9$	0.5 ± 0.6
		(73.3 ± 8.6)	(26.1 ± 9.0)	(0.2 ± 0.4)	(0.4 ± 0.5)
	Mn_5Al_8	4.0 ± 3.3	$38.5\ \pm 0.8$	56.7 ± 3.1	0.7 ± 0.1
		(6.2 ± 5.0)	(53.8 ± 1.9)	(39.0 ± 3.2)	(1.0 ± 0.1)

Table 4.2. Phase compositions in wt% (at.% is in parentheses) for the AM60 materials.

4.1.1.3 Texture

4.1.1.3.1 XRD Analysis

Figure 4.6 illustrates the XRD intensity versus 2θ plots for the face and longitudinal sections of the AM60 alloys processed under different conditions. Table 4.3 shows the powder diffraction data of the seven strongest peaks for Mg (PDF #35-0821).

As shown in Figure 4.6(c), for the as-molded material, the intensity versus 2 θ plots were similar between the face section and longitudinal section, both showing a strong peak for the (10 Γ 1) plane and less intense peaks for the (0002) and (10 Γ 0) planes. The relative intensity of different peaks was similar to the value listed in the powder diffraction file as shown in Table 4.3, suggesting that the as-molded material exhibited a relatively random texture.

The TTMP and annealed materials exhibited similar XRD intensity versus 20 plots. As shown in Figures 4.6(a) and 4.6(b), comparing with the as-molded material, both of the TTMP and the annealed materials exhibited a much stronger (0002) peak in the face section scan, but a much weaker (0002) peak in the longitudinal section scan. Moreover, Figure 4.7 shows the (0002) XRD pole figure of the face section for the TTMP and annealed materials, and this data verified the high intensity of the (0002) pole. Thus, the TTMP process resulted in a basal texture in which the basal planes were parallel with the sheet face, and this texture was retained after the annealing treatment.

A detailed examination of Figures 4.6(a) and 4.6(b) also shows that the TTMP material exhibited a larger full width at half maximum (FWHM) value than in the annealed material for the same peak. This is reasonable since there should be a larger residual strain in the TTMP material, resulting in deviation in the plane spacing. The texture exhibited by the TTMP and annealed materials might be responsible for the significant enhancement in the tensile and fatigue strengths as will be presented in sections 4.1.2 and 4.1.3.



Figure 4.6. XRD intensity versus 2θ plots for the AM60 alloys. (a) face section scans and (b) longitudinal section scans for the AM60 of different processing conditions. (c) face section scan and longitudinal scan for the as-molded material.

Figure 4.6 (cont'd)



Table 4.3. Powder diffraction data of Mg (Powder Diffraction File # 35-0821)* showing the intensity of different peaks.

#	d (Å)	I (f)	(h k l)	2-Theta
1	2.7782	25.0	(100)	32.193
2	2.6050	36.0	(0 0 2)	34.398
3	2.4520	100.0	(101)	36.619
4	1.9002	15.0	(102)	47.828
5	1.6047	12.0	(1 1 0)	57.374
6	1.4730	16.0	(103)	63.056
7	1.3664	13.0	(1 1 2)	68.631

Radiation = Cu $K_{\alpha 1}$

* Powder Diffraction File, The International Center for Diffraction Data, Swarthmore, PA.



Figure 4.7. (0002) XRD pole figures of the face section for the AM60 sheets: (a) TTMP, and (b) annealed. (Chi is tilt angle, and Phi is azimuthal angle.)

Figure 4.7 (cont'd)



(b)
4.1.1.3.2 EBSD Analysis

EBSD scans were performed on the face section of the AM60 sheets, with only the α -Mg phase analyzed. Figure 4.8 shows the EBSD IPF map and the corresponding pole figures of an as-molded sample. The black areas on the EBSD IPF map are regions that were not indexed, possibly corresponding to the γ -phase. As shown in the figure, the as-molded material showed a relatively random texture. The (0001) pole figure showed several uniformly distributed peaks with intensity values larger than 1.5 times random. The maximum intensity was about two times random.

Figure 4.9 shows an EBSD IPF map and the corresponding pole figure for an annealed sample. The (0001) pole exhibited relatively high intensities around the ND. The maximum intensity was about six times random. This suggests that the annealed material is moderately textured, with the basal planes aligned parallel to the sheet face. Within the circled region in Figure 4.9(a), two large grains exhibited some orientation gradients within the grains, corresponding to an ~5 °spread in orientation. These appear to be deformed grains that were not recrystallized during the annealing treatment. The color of these grains were generally red, indicating that the c-axis is nearly parallel to the ND.

Figure 4.10 shows an EBSD IPF map and the corresponding pole figure for another annealed sample. The (0001) texture is slightly weaker compared to that in Figure 4.9, with a maximum intensity ~2.6 times random. Within the circled area in Figure 4.10(a), there were grains that were likely to have been recrystallized from a single large grain. Some of these grains exhibited yellow or purple color on the EBSD IPF map, suggesting that they had their c-axis tilted away from the ND. Therefore, recrystallization was likely to have led to a decrease in the intensity of the texture.



Figure 4.8. EBSD data for the face section of an AM60 as-molded material: (a) EBSD IPF map for the α -Mg phase, boundaries with rotation angle larger than 15 °were shown, (b) (0001) and (1010) pole figure for the α -Mg phase. The color code of the EBSD map represents the orientation of the ND. The longitudinal direction of the sheet is horizontal.



Figure 4.9. EBSD data for the face section of an AM60 annealed material: (a) EBSD IPF map for the α -Mg phase, boundaries with rotation angle larger than 15 °were shown, (b) (0001) and (101 0) pole figure for the α -Mg phase. The color code of the EBSD map represents the orientation of the ND. The longitudinal/rolling direction of the sheet is horizontal. Within the circled region in figure (a), two relatively large grains exhibited a large orientation gradient rather than recrystallization.



Figure 4.10. EBSD data for the face section of an AM60 annealed material: (a) EBSD IPF map for the α -Mg phase, boundaries with rotation angle larger than 15 ° were shown, (b) (0001) and (10 Γ 0) pole figure for the α -Mg phase. The color code of the EBSD map represents the orientation of the ND. The longitudinal/rolling direction of the sheet is horizontal. In figure (a), static recrystallization is likely to have occurred in the circled region where used to be a large deformed grain. Note that this is a different sample from that in Figure 4.9.

4.1.2 Tensile Behavior Results

Tensile tests were performed at RT and 150 °C. Each of the samples were tested along the longitudinal/rolling direction of the sheet. Figure 4.11 shows representative engineering stress versus engineering strain curves for the AM60 samples tested at different temperatures. The average RT and 150 $^{\circ}$ C tensile properties, including the Young's modulus (E), YS, UTS, $\varepsilon_{\rm f}$, and strain-hardening exponent (*n*-value), were summarized in Table 4.3. At both RT and 150 $^{\circ}$ C, the TTMP material exhibited a significant increase in the YS and UTS compared to the asmolded material. At RT, the YS and UTS of the TTMP material were ~2.5 times and ~1.8 times that of the as-molded material, respectively. At 150 °C, the YS and UTS of the TTMP material were ~2.2 times and ~1.6 times that of the as-molded material, respectively. At both RT and 150 °C, the $\varepsilon_{\rm f}$ of the as-molded and TTMP materials was similar. The annealed material, while exhibiting YS and UTS values intermediate to those for the as-molded and TTMP materials, exhibited a significantly larger $\varepsilon_{\rm f}$ than the as-molded and TTMP materials at both temperatures, which was ~19% at RT, and >27% at 150 °C. The larger ε_f values exhibited by the annealed material could be rationalized if the annealing treatment resulted in a lower dislocation density compared to that for the TTMP material. This would also be consistent with the lower strength exhibited by the annealed material compared to the TTMP material. Compared to RT, each of the studied materials exhibited a lower YS and UTS, but a higher ε_f at 150 °C.

The strain-hardening exponents (*n*-values) were calculated based on the stress-strain relationship, and the values were summarized in Table 4.4. Drastically different strain hardening behavior was exhibited by the three conditions. The TTMP material exhibited the lowest strain hardening exponent and the greatest UTS value. This may be expected since this material was

the most severely worked without a subsequent anneal. The as-molded condition exhibited the largest strain hardening exponent and the lowest UTS value.

Figure 4.12 illustrates the fracture surfaces of representative RT tensile samples. Generally, all fracture surfaces exhibited quasi-cleavage fracture features. Cleavage was the dominate feature, however, ductile features, including tear ridges and shallow dimples, were also visible. Compared to the TTMP material, a larger volume fraction of regions containing ductile features were visible for the as-molded and annealed materials.



Figure 4.11. Representative engineering stress versus engineering strain curves for the AM60 alloys tested at RT and 150 C.

Condition	Temperature	<i>E</i> , GPa	YS, MPa	UTS, MPa	Ef, %	<i>n</i> -value
As-molded	RT	37.8	131	204	5.7	0.23
TTMP		41.2	331	380	3.4	0.06
Annealed		44.6	227	302	19.4	0.19
As-molded	150 ℃	37.0	119	190	6.6	0.21
TTMP		37.2	240	297	9.0	0.04
Annealed		44.0	188	250	>27.4	0.17

Table 4.4. Summary of the average RT and 150 $^{\circ}$ C tensile properties, including Young's Modulus (*E*), YS, UTS, ε_{f} , and strain-hardening exponent (*n*-value), for the AM60 alloys.



Figure 4.12. SE SEM images of RT tensile fracture surfaces of AM60 (a) as-molded, (b) TTMP, and (c) annealed samples.

Figure 4.12. (cont'd)



4.1.3 Fatigue Behavior Results

4.1.3.1 Fatigue Behavior

Figure 4.13 shows the fatigue maximum applied stress (σ_{max}) versus cycles-to-failure (N_f) plots (S-N behavior) for the studied AM60 alloys at RT and 150 °C. For each individual material, both the fatigue life and the fatigue strength did not show a significant variation between RT and 150 °C. At equivalent maximum stress levels, the annealed material exhibited the longest fatigue lives and the as-molded material exhibited the shortest fatigue lives. At both RT and 150 °C, the annealed material exhibited the highest fatigue strength, which was considered to be between 125-150 MPa. The TTMP material exhibited a slightly lower fatigue strength, which was between 100-125 MPa, and the as-molded material exhibited the lowest fatigue resistance, and the reason for this was considered to be related to its combination of tensile strength and ε_f .

Figure 4.14 shows σ_{max} /UTS versus N_f plots for the studied materials at RT and 150 °C. For a given σ_{max} /UTS value the annealed material exhibited the longest fatigue lives. The asmolded material showed some overlap in the fatigue lives with that of the TTMP material, although the fatigue lives of the as-molded material were generally slightly longer. With increasing temperature, each individual material showed a slight increase in the fatigue ratio, which is defined as the ratio between fatigue strength and UTS. At RT, the fatigue ratio was ~0.4 for the annealed material, and was ~0.3 for the as-molded and TTMP materials. At 150 °C, the fatigue ratio was ~0.5 for the annealed material, and was ~0.4 for the as-molded and TTMP materials.



(b)

Figure 4.13. Maximum applied stress (σ_{max}) versus cycles-to-failure (N_f) plots for the studied AM60 samples at (a) RT and (b) 150 °C. Arrows indicated runout samples which did not fail.



(b)

Figure 4.14. $\sigma_{\text{max}}/\text{UTS}$ versus N_f plots for the studied AM60 samples at (a) RT and (b) 150 °C. Arrows indicated runout samples which did not fail.

4.1.3.2 Fracture Analysis

After failure, the fracture surface of the AM60 fatigue-tested samples were observed using SEM. For most of the samples, a distinguishable crack initiation site and a well-defined fatigue crack propagation region were observed on the fracture surface. Multiple crack initiation sites were observed for some samples. Representative fracture surfaces of RT fatigue-tested AM60 samples are illustrated in Figures 4.15, 4.16, and 4.17.

For the as-molded material, the fatigue cracks initiated preferentially at pores near the sample surface. Figure 4.15 shows the fracture surface of an as-molded sample. The fatigue crack was likely to have initiated from a large pore as indicated by the arrow in Figure 4.15(a). Figure 4.15(b) is a higher-magnification image of this region, where rounded grains/particles were observed. Near-surface pores with similar features were observed in other studies and were identified as shrinkage pores (Rettberg *et al.* 2012).

For the TTMP and annealed materials, almost all samples had crack initiation sites located on the sample surface. Figure 4.16 shows the fracture surface of a TTMP sample. It appeared that the fatigue crack propagation region was a combination of a semi-elliptical region and a quarter-elliptical region, as illustrated by the dotted lines, originating from different crack initiation sites. The possible crack initiation sites were indicated by the arrows, and many of them appeared to be inclusions.

Figure 4.17 shows the fracture surface of an annealed sample. The fatigue crack was likely to have initiated from the surface, as shown in Figure 4.17(b). No inclusion was observed in this region, so the crack initiation might have been due to cyclic slip, leading to the formation of micro notches or cracks at the surface. A large pore was observed on the fracture surface, and it was flat, which is typical for the pores in the annealed material. This was different from the

pores observed in the as-molded samples. Moreover, the flat pores in the annealed material generally did not appear to be crack initiation sites.

Figure 4.18 to 4.20 show the fracture surfaces of some of the 150 °C fatigue-tested AM60 samples. In general, the features were similar to those for the RT fatigue-tested samples. However, for the as-molded material, a few samples showed surface crack initiation sites. Also, the crack initiation sites appeared to be rough, as shown in Figure 4.18(b), possibly due to the high temperature exposure, which accelerated oxidation. Figure 4.19 shows the fracture surface of a TTMP sample in which surface crack initiation was observed. The EDS data gathered at the initiation site indicated that unusually high levels of oxygen and silicon were present. Thus, this crack initiation site may have been at an inclusion.



Figure 4.15. (a) Low- and (b) high-magnification SE SEM images of the fracture surface of an as-molded AM60 sample fatigue tested at RT and a maximum applied stress of 125 MPa. It failed at 7,504 cycles. The arrow in (a) indicates a pore which served as a crack initiation site, and (b) shows a higher magnification image of this region.



Figure 4.16. (a) Low- and (b) high-magnification SE SEM images of the fracture surface of a TTMP AM60 sample fatigue tested at RT and a maximum applied stress of 150 MPa. It failed at 26,643 cycles. The arrows indicate potential crack initiation sites.



Figure 4.17. (a) Low-magnification SE SEM images of the fracture surface of an annealed AM60 sample fatigue tested at RT and a maximum applied stress of 150 MPa. It failed at 755,000 cycles. The dotted line indicates the boundary between the fatigue crack growth region and the fast fracture region. The arrow in (a) indicates a potential crack initiation site, and a higher magnification image of this region is shown in (b).



Figure 4.18. (a) Low-magnification SE SEM image of the fracture surface of an as-molded AM60 sample fatigue tested at 150 $^{\circ}$ C and a maximum applied stress of 75 MPa. It failed at 422,532 cycles. The arrow in (a) indicates the fatigue crack initiation site, and a higher magnification image of this region is shown in (b).



Figure 4.19. (a) Low- and (b) high-magnification SE SEM images of the fracture surface of a TTMP AM60 sample fatigue tested at $150 \,\text{C}$ and a maximum applied stress of 150 MPa. It failed at 32,747 cycles. The surface crack initiation site is believed to be near the top center of each image.



Figure 4.20. SE SEM images of the fracture surface of an annealed AM60 sample fatigue tested at $150 \,^{\circ}$ C and a maximum applied stress of 150 MPa. It failed at 328,786 cycles. The arrow indicates the crack initiation site at the edge of the sample.

The polished face section (subsurface) of the samples near the fractured region of some fatigue tested AM60 samples were also observed using SEM in order to study the fatigue damage and in particular the secondary cracking behavior. As shown in Figure 4.21, small cracks were observed near the fracture surface. Some cracks were observed in the grain boundary region, and it appeared that some of them eventually grew into the Mg matrix, as indicated by the black arrows. No phase instability was observed during the RT or elevated-temperature fatigue exposures. Figure 4.22 shows the subsurface microstructure near the fracture surface in a fatigue tested TTMP AM60 sample. A crack appeared to have initiated from an inclusion, and grew in a transgranular fashion perpendicular to the loading direction. Figure 4.23 shows the subsurface microstructure in a fatigue-tested annealed AM60 sample. A cracked area originating from inclusion particles was found at the edge of this sample. EDS data of the cracked area indicated a high level of oxygen.



Figure 4.21. BSE SEM images of fatigue deformed AM60 samples near the fracture surface: (a) as-molded, RT fatigue deformed, with a maximum applied stress of 125 MPa and 7,504 cycles to failure, and (b) TTMP, 150 $^{\circ}$ C fatigue deformed, with a maximum applied stress of 150 MPa and 30,182 cycles to failure. The loading direction is vertical in these images and the arrows point to secondary cracks.



Figure 4.22. BSE SEM image of a fatigue deformed TTMP AM60 sample showing the subsurface microstructure of the face section near fracture surface. This sample was fatigue tested at RT, with a maximum applied stress of 150 MPa and 24,369 cycles to failure. The loading direction is vertical in this image.



Figure 4.23. BSE SEM image of a fatigue deformed annealed AM60 sample showing the subsurface microstructure of the face section. This sample was fatigue tested at $150 \,$ °C, with a maximum applied stress of 175 MPa and 24,062 cycles to failure. The loading direction is vertical in this image.

4.1.4 Fatigue Crack Growth Behavior Results

During the fatigue crack growth experiments on AM60, the crack growth behavior was observed using a long working-distance microscope. Optical images of the cracks emanating from the artificial notches were taken at different intervals during the test. Figure 4.24 shows representative surface images of fatigue cracks that had propagated from the machined artificial notches. For some of the as-molded specimens, such as the one shown in Figure 4.24(a), cracks also initiated at other locations apart from the machined notch. These cracks then linked to the main crack and increased the tortuosity of the crack. However, for the TTMP material, the cracks emanating from the notch generally grew straight and perpendicular to the loading direction, see Figure 4.24(b). At longer crack lengths, the butterfly-shaped plastic zone could be observed at the crack tip. Figure 4.24(c) illustrates a crack in an annealed AM60 specimen. Similar to the cracks in the TTMP specimens, the cracks in the annealed specimens were pure mode I cracks (i.e. perpendicular to the loading direction).



Figure 4.24. Representative images of cracks emanated from the machined notch in an (a) asmolded specimen, tested at $\sigma_{\text{max}} = 105$ MPa, R = 0.1, at 27,000 cycles, (b) TTMP specimen, tested at $\sigma_{\text{max}} = 200$ MPa, R = 0.1, at 10,000 cycles, and (c) annealed specimen, tested at $\sigma_{\text{max}} = 140$ MPa, R = 0.1, at 36,000 cycles.

4.1.4.1 Fatigue Crack Growth Rate Characterization

Optical images of the specimen surfaces, taken at different fatigue cycles, were used to calculate the fatigue crack growth rate. The surface crack length, projected on the plane perpendicular to the loading direction, was measured using calibrated optical images. The crack growth rate was determined using the following methodology: If a crack length a_1 was determined on an image taken at N_1 cycles, and a crack length a_2 was determined on an image

taken at N₂ cycles, then the value $\frac{\Delta a}{\Delta N} = \frac{a_2 - a_1}{N_2 - N_1}$ was used to represent the crack growth rate,

da/dN, for a crack with a length of $a = \frac{a_1 + a_2}{2}$. The ΔK was calculated using the Newman-Raju solutions (Newman and Raju 1981) for a small surface crack in a finite plate. The surface crack was assumed to be of semi-circular shape, and the ΔK at the interior front of the crack was used for analysis. Details for the expression of the Newmann-Raju solution and the calculation of ΔK in this study are provided in Appendix A.

The fatigue crack propagation curves, depicted as da/dN versus ΔK , are shown in Figure 4.25. The curves were fitted to the Paris law relationship (Paris and Erdogan 1963) as expressed in Equation (2.9). The fitting parameters are summarized in Table 4.5. A majority of the Paris exponents were between 2 and 4, and in particular, all the Paris exponents for an *R* value of 0.1 were above 3.

As shown in Figure 4.25, the crack growth rates showed a relatively anomalous behavior during the early stage of fatigue crack growth. Both temporary retardation and acceleration of crack growth rates were observed, possibly due to the influence of the local microstructure or notch tip plasticity on the small fatigue crack. The fatigue crack growth threshold (ΔK_{th}) values were estimated by extrapolating the Paris law fatigue crack growth curve fits to a crack growth rate of 10⁻⁹ m/cycle. The corresponding ΔK_{th} values are also summarized in Table 4.5.

An alternative method to present the fatigue crack growth rate, suggested by Shyam *et al.* (2005, 2007), is to plot da/dN versus the product of $\phi_{\rm m}$ and $\phi_{\rm c}$, which are defined in Equations (2.12) and (2.13). Their results suggested that da/dN was almost linearly related to $\phi_{\rm m}\phi_{\rm c}$ (Shyam *et al.* 2005, 2007). In the current work, the da/dN versus $\phi_{\rm m}\phi_{\rm c}$ plot is shown in Figure 4.26, and the curves were fitted to the following relationship:

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \kappa \left(\phi_{\mathrm{m}}\phi_{\mathrm{c}}\right)^{q} \tag{4.1}$$

The fitted values of κ and q were summarized in Table 4.6. The fitted values of q were generally around 1.



Figure 4.25. da/dN versus ΔK plot for the studied AM60 alloys tested at RT and 5 Hz.

Condition	$\sigma_{ m max}$	R	С	т	ΔK_{th} , MPa $\sqrt{\text{m}}$
as-mold	80	0.1	3.80×10^{-10}	3.65	2.2
as-mold	105	0.1	2.80×10^{-12}	8.50	2.3
TTMP	200	0.1	7.86×10^{-10}	3.02	1.1
TTMP	265	0.1	4.72×10^{-11}	4.62	2.0
TTMP	300	0.1	1.54×10^{-10}	4.02	3.7
TTMP	265	0.3	1.03×10^{-9}	3.11	0.9
TTMP	265	0.5	1.96×10^{-9}	2.93	1.9
annealed	140	0.1	2.31×10^{-10}	3.54	1.7
annealed	180	0.1	2.54×10^{-10}	3.84	2.4
annealed	200	0.1	6.55×10^{-10}	3.20	1.8
annealed	140	0.3	4.57×10^{-9}	2.41	0.8
annealed	180	0.3	4.26×10^{-10}	3.77	1.5
annealed	140	0.5	6.06×10^{-9}	2.30	0.6
annealed	180	0.5	8.66×10 ⁻⁹	2.05	1.0

Table 4.5. Summary of the fitting parameters for the fatigue crack growth curves presented in Figure 4.25, and the ΔK_{th} values determined from these curves. The curves were fitted using Equation (2.9).



Figure 4.26. Fatigue crack growth rate (da/dN) versus $\phi_m \phi_c$ plot for the studied AM60 alloys.

Table 4.6. Summary of the fitting parameters for the fatigue crack growth curves presented in Figure 4.26. The curves were fitted using Equation (4.1).

Condition	$\sigma_{ m max}$	R	К	<i>q</i>
as-mold	80	0.1	5.21×10 ⁵	1.19
as-mold	105	0.1	1.00×10^{19}	2.39
TTMP	200	0.1	5.38×10^{2}	0.87
TTMP	265	0.1	3.00×10^{7}	1.32
TTMP	300	0.1	2.19×10 ⁴	1.04
TTMP	265	0.3	4.74×10^2	0.88
TTMP	265	0.5	1.79×10^2	0.84
annealed	140	0.1	3.40×10 ⁴	1.07
annealed	180	0.1	2.13×10 ⁴	1.05
annealed	200	0.1	3.06×10^2	0.89
annealed	140	0.3	6.91×10^{1}	0.79
annealed	180	0.3	3.11×10 ⁴	1.06
annealed	140	0.5	1.04×10^{1}	0.72
annealed	180	0.5	2.95×10 ⁻¹	0.59

In order to evaluate the effect of mechanical loading parameters and the effect of processing on the fatigue crack growth behavior, the fatigue crack growth data were subdivided into groups based on different criteria and comparisons were made between data within each group.

4.1.4.1.1 Effect of Maximum Applied Stress

In order to evaluate the effect of maximum applied stress on the fatigue crack growth rate, fatigue crack growth curves of a given material tested at a given stress ratio under different maximum applied stresses were compared. Figure 4.27(a) shows crack growth curves for the TTMP materials tested at R = 0.1, under three levels of maximum stress: 200 MPa, 265 MPa, and 300 MPa, which correspond to 60%, 80%, and 90% of the σ_{ys} . Figure 4.27(b) shows crack growth curves for the annealed materials tested at R = 0.3, under two levels of maximum stress: 140 MPa and 180 MPa, which correspond to 60% and 80% of the σ_{ys} . Similar comparison of the fatigue crack growth curves for the rest of the specimens are shown in Figure B.1 in Appendix B. In each individual plot, different curves showed similar crack growth rates at equivalent ΔK values. Therefore, it appeared that increasing the maximum stress alone did not lead to a discernible change in the average crack growth rate.



Figure 4.27. Fatigue crack growth plots for the (a) TTMP AM60 materials tested at R = 0.1, and three different maximum stress levels (200 MPa, 265 MPa, and 300 MPa, which corresponded to 60%, 80%, and 90% of the σ_{ys}), and (b) annealed AM60 materials tested at R = 0.3, and two different maximum stress levels (140 MPa and 180 MPa, which corresponded to 60% and 80% of the σ_{ys}).

4.1.4.1.2 Effect of Material Processing

In order to evaluate the effect of material processing on the fatigue crack growth behavior, the fatigue crack growth curves of each of the processed materials, tested at a given stress ratio under different maximum applied stresses, were compared. Figure 4.28(a) shows the corresponding fatigue crack growth curves for each of the material, tested at R = 0.1 and different maximum applied stress levels. Figure 4.28(b) shows fatigue crack growth curves for the TTMP and annealed materials tested at R = 0.3 and different maximum applied stress levels. Figure 4.28(c) shows fatigue crack growth curves for the TTMP and annealed materials tested at R = 0.3 and different maximum applied stress levels. Figure 4.28(c) shows fatigue crack growth curves for the TTMP and annealed materials tested at R = 0.5 and different maximum applied stress levels. Since it was shown in Section 4.1.4.1.1 that changing the maximum stress alone did not lead to a discernible change in the fatigue crack growth rate, any noticeable difference in the da/dN versus ΔK behavior in Figure 4.28 could be attributed to the effect of processing. However, this was not the case as in Figure 4.28, under a given stress ratio, different materials exhibited similar crack growth rates at equivalent ΔK values. Therefore, it appeared that the processing condition did not have a significant effect on the fatigue crack growth rate of the AM60 alloys.



Figure 4.28. Fatigue crack growth plots for the (a) as-molded, TTMP, and annealed AM60 materials tested at R = 0.1, and different maximum stress levels, and (b) TTMP and annealed AM60 materials tested at R = 0.3, and different maximum stress levels, and (c) TTMP and annealed AM60 materials tested at R = 0.5, and different maximum stress levels.

Figure 4.28 (cont'd)



4.1.4.1.3. Effect of Stress Ratio

In order to evaluate the effect of stress ratio on the fatigue crack growth behavior, the fatigue crack growth curves of a given material tested at different stress ratios under a given maximum applied stress level were compared. Figure 4.29 shows the fatigue crack growth curves for the annealed materials tested at $\sigma_{max} = 180$ MPa, and three different stress ratios (R = 0.1, 0.3, and 0.5). In Figures 4.29(a), (b), and (c), the fatigue crack growth rates are plotted against ΔK , K_{max} , and $\phi_m \phi_c$, respectively. Similar comparisons of the fatigue crack growth rates at equivalent that with increasing stress ratio, the specimens exhibited higher crack growth rates at equivalent ΔK values, as shown in Figure 4.29(a). Conversely, it was determined that with increasing stress ratio lower crack growth rates at equivalent K_{max} values, as shown in Figure 4.29(b). Therefore, the fatigue crack growth rate was affected by the stress ratio. However, a similar crack growth rate was observed at equivalent $\phi_m \phi_c$ values for a given material tested at different stress ratios, as is shown in Figure 4.29(c).



Figure 4.29. Fatigue crack growth plots for the annealed AM60 materials tested at $\sigma_{\text{max}} = 180$ MPa, and three different stress ratios (R = 0.1, 0.3, and 0.5), showing the crack growth rate versus (a) ΔK , (b) K_{max} , and (c) $\phi_{\text{m}}\phi_{\text{c}}$.

Figure 4.29 (cont'd)


4.1.4.2 Fracture Analysis

In general, the primary crack that led to failure of the fatigue crack growth sample initiated from the machined artificial notch due to stress concentration. However, for some of the as-molded and TTMP samples, failure occurred due to the growth of cracks that initiated at locations other than the artificial notch.

Figures 4.30, 4.31, and 4.32 illustrate representative fracture surfaces of the AM60 fatigue crack growth samples in which the primary crack initiated from the artificial notch. Features corresponding to different stages of fatigue crack growth, including the crack initiation region, the crack propagation region, and the fast fracture region, are identified in the low magnification images, as shown in Figures 4.30(a), 4.31(a), and 4.32(a). For the as-molded sample shown in Figure 4.30(a), although the primary crack initiated from the artificial notch, additional cracks initiated at other locations were also observed. Figure 4.30(c) illustrates a large pore observed in one of the additional crack initiation regions.

The fatigue crack generally exhibited a semi-circular or semi-elliptical shape. As shown in Figures 4.30(b), 4.31(b), and 4.32(b), in the regions near the notch, flat cleavage planes were commonly observed. Fine striation-like patterns were also occasionally observed on the cleavage planes, as indicated by the arrow in Figure 4.30(b). River patterns dividing multiple cleavage planes were also commonly observed. With the increase of the distance from the notch, less cleavage plane patterns were observed, and the fracture surface became rougher, indicating a tortuous fatigue crack growth path, see Figures 4.30(c), 4.31(c), and 4.32(c). Some randomly orientated serrated surfaces were also observed, as indicated by the arrow in Figure 4.30(c). Based on extensive observations, a "transition crack length" was determined for most of the specimens, as summarized in Table 4.7. The cleavage planes, with striation-like patterns and

river patterns on them, were mainly observed in regions corresponding to a crack length smaller than the transition crack length. In the regions corresponding to a crack length larger than the transition crack length, cleavage patterns were hardly observed, while the tortuous crack growth path pattern dominated.

In the fast fracture region, dimple-like features were observed, as shown in Figures 4.30(d), 4.31(d), and 4.32(d), suggesting ductile overload occurred. The features exhibited by the fast fracture/overload regions were comparable to those of the tensile test fracture surfaces as shown in Figure 4.12.



Figure 4.30. SE SEM images of the fracture surface of as-molded AM60 fatigue crack growth samples: (a) the overall fracture surface, (b) the crack propagation region near the notch, (c) the crack propagation region near a large pore, and (d) an area in the fast fracture region. (a), (c) and (d) are from a sample tested at $\sigma_{\text{max}} = 105$ MPa, R = 0.1, and failed at 28,977 cycles. (b) is from a sample tested at $\sigma_{\text{max}} = 80$ MPa, R = 0.1, and failed after 61,876 cycles.



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Figure 4.31. SE SEM images of the fracture surface of a TTMP AM60 fatigue crack growth sample, tested at $\sigma_{\text{max}} = 265$ MPa, R = 0.3, and failed at 6,769 cycles: (a) the overall fracture surface, (b) an area in the crack propagation region near the machined notch, (c) an area in the crack propagation region far from the machined notch, and (d) and area in the fast fracture region.





Figure 4.32. SE SEM images of the fracture surface of annealed AM60 fatigue crack growth samples: (a) the overall fracture surface, (b) an area in the crack propagation region near the machined notch, (c) an area in the crack propagation region far from the machined notch, and (d) an area in the fast fracture region. (a), (c), and (d) are from a specimen tested at $\sigma_{\text{max}} = 140$ MPa, R = 0.1, and failed after 47,000 cycles. (b) is from a specimen tested at $\sigma_{\text{max}} = 180$ MPa, R = 0.3, and failed at 22,985 cycles.



Table 4.7. Summary of the approximate transition crack length determined for some of the AM60 fatigue crack growth specimens. The cleavage features on the fracture surface were mainly observed in regions corresponding to a crack length smaller than this transition crack length.

Condition	$\sigma_{ m max}$, MPa	R	Transition crack length, μm
as-mold	80	0.1	990
as-mold	105	0.1	520
TTMP	265	0.3	140
TTMP	265	0.5	220
annealed	140	0.1	410
annealed	200	0.1	260
annealed	140	0.3	580
annealed	180	0.3	430
annealed	140	0.5	740
annealed	180	0.5	600

For some as-molded and TTMP specimens, the cracks that lead to fracture did not initiate at the machined notch. In such cases, the cracks that initiated at the notch only exhibited limited growth. Figure 4.33 shows the fracture surface for such a case exhibited by an as-molded specimen. A cluster of pores that served as the crack initiation site can be observed on the rough fracture surface. The crack propagation region was not readily apparent. Figure 4.34 shows the fracture surface of a TTMP specimen that did not fail from the crack that initiated from the machined notch. A crack propagation region of quarter-ellipsoidal shape was observed. A higher magnification image shows that the crack might have initiated at a defect near the surface at the corner of the cross section of the specimen as shown in Figure 4.34(b). Thus, whenever failure proceeded from a crack which did not initiate from the machined notch, it was associated with an extrinsic defect in the specimen. Because such cracks were not able to be monitored during the fatigue crack growth experiment, they were not used for the fatigue crack growth rate analysis.

The total number of specimens tested and the number of specimens in which the primary crack initiated at the machined notch, for each of the three processing conditions, are summarized in Table 4.8. For the specimens in which crack initiation was observed at the machined notch, a plot of the applied stress range versus the number of cycles at which the crack initiation was observed is provided in Figure 4.35.



Figure 4.33. SE SEM images of the fracture surface of an as-molded AM60 fatigue crack growth sample tested at $\sigma_{\text{max}} = 105$ MPa, R = 0.1, and failed at 13,612 cycles: (a) the overall fracture surface, and (b) an area with a large pore. This specimen failed at a crack that did not initiate from the notch.



Figure 4.34. SE SEM images of the fracture surface of a TTMP AM60 fatigue crack growth sample tested at $\sigma_{\text{max}} = 200$ MPa, R = 0.1, and failed at 8,755 cycles: (a) the overall fracture surface, and (b) the crack initiation region. This specimen failed at a crack that did not initiate from the notch. The defect at the corner of the cross section is indicated by the arrows.

Condition	Number of specimens tested	Number of specimens in which the primary crack initiated at the notch
As-molded	6	2
TTMP	10	4
Annealed	7	7

Table 4.8. Summary of number of specimens tested and number of specimens with crack initiated at the notch.



Figure 4.35. Applied stress range versus the number of cycles at which crack initiation was observed at the notch for the studied AM60 fatigue crack growth specimens.

4.1.5 Creep Behavior Results

4.1.5.1 Creep Properties

Figure 4.36 shows the creep strain versus time curves for the studied AM60 alloys tested at 150 $^{\circ}$ under three different applied stress levels. Figure 4.37 shows the creep rate versus creep strain curves for the AM60 materials tested at 150 $^{\circ}$ under three different applied stress levels. The creep rate in Figure 4.37 was calculated using the creep strain and time data in Figure 4.36 from every 30 data points. The MATLAB code used for this calculation can be found in Appendix C. It is noted that a more robust method was used to determine the minimum creep rate, but this method served to depict the general trend of creep rate versus creep strain.

In most cases, the samples did not rupture before the tests were interrupted. The samples that ruptured are indicated by the "×" marked on the plots. Tertiary creep was only evident for a few samples, such as the as-molded sample tested at 50 MPa, and the as-molded and annealed samples tested at 75 MPa.

Under all three stress levels examined, the as-molded material exhibited the greatest creep resistance. The annealed material exhibited a significantly higher creep rate than the as-molded and TTMP materials under applied stress level of 20 MPa. At 50MPa and 75 MPa, the annealed material showed similar creep rates as the TTMP material, and they were both much higher than that of the as-molded material.

The minimum creep rate versus stress plots for the studied AM60 alloys are shown in Figure 4.38. The creep stress exponents (n) were calculated based on these plots, and the n values at 150 °C were 4.9, 5.8, and 4.5 for the as-molded, TTMP, and annealed materials, respectively. Figure 4.39 shows the temperature dependence of the minimum creep rate under 20 MPa applied stress, and the apparent activation energy, Q, was calculated based on this plot.

The plot showed a two-stage linear relationship. Between 100-150 °C, the Q for the as-molded, TTMP, and annealed materials were 48 kJ/mol, 64 kJ/mol and 67 kJ/mol, respectively. Between 150-200 °C, the Q for the as-molded, TTMP, and annealed materials were 128 kJ/mol, 174 kJ/mol, and 126 kJ/mol, respectively.

Figure 4.40 shows SEM images of the polished subsurface of some creep tested AM60 samples. Voids and cracks are observed at some grain boundaries. The as-molded materials exhibited a high porosity in the undeformed microstructure, so it was difficult to determine whether the cracks/voids shown in Figure 4.40(a) existed in the material before deformation or formed during creep deformation. However, the cracks in Figure 4.40(a) do seem to be larger than the pre-existing pores in the as-molded material. In addition, although not shown here, volume fraction measurements comparing the polished longitudinal section of the creepdeformed gage section and the undeformed grip section of the same sample suggested that there was an approximately 20% higher pore/voids volume percentage in the creep-deformed gage section. Therefore, voids and cracks were likely to be formed at the grain boundaries during creep deformation in the as-molded material. The TTMP and annealed materials exhibited a lower porosity in the undeformed state compared with the as-molded material. Figures 4.40(b)and (c) clearly show voids in the creep deformed TTMP and annealed materials. Therefore, it appeared that the grain boundaries served as crack nucleation sites during the creep deformation for these processed materials.



Figure 4.36. Creep strain versus time plot of the AM60 materials tested at 150 °C and three stress levels: (a) $\sigma = 20$ MPa, (b) $\sigma = 50$ MPa, and (c) $\sigma = 75$ MPa.

Figure 4.36 (cont'd)





Figure 4.37. Creep strain rate versus creep strain plot for the AM60 materials creep tested at $150 \text{ }^{\circ}\text{C}$ and three applied stress levels: (a) 20 MPa, (b) 50 MPa, and (c) 75 MPa.

Figure 4.37 (cont'd)





Figure 4.38. Minimum creep rate versus stress plot at $T = 150 \,^{\circ}$ C for the studied AM60 alloys. The creep exponent (*n*) values were determined from the plot.



Figure 4.39. Minimum creep rate versus reciprocal of temperature plot for the studied AM60 alloys at $\sigma = 20$ MPa. The apparent creep activation energy values were determined from the plot.



Figure 4.40. BSE SEM images of the subsurface for creep tested AM60 samples: (a) as-molded sample, tested at 20-50 MPa, 150 \degree , total strain was 2.7%, (b) TTMP sample, tested at 75 MPa, 150 \degree , total strain was 4.8%, (c) annealed sample, tested at 20 MPa, 100-200 \degree , total strain was 10.5%.



4.1.5.2 In-situ Creep Behavior

Figure 4.41 shows SE SEM images of an annealed AM60 sample taken during an *in-situ* creep test at 50 MPa and 150 °C. The crosshead displacement recorded during the test is indicated on the images. With increasing creep deformation, void formation was observed at grain boundaries, and grain boundary cracking became evident. Figure 4.42 shows SE SEM images of the surface of an annealed AM60 sample after *in-situ* creep testing at 150 °C and 75 MPa. Slip traces can be observed in some of the grains, as indicated by the white arrows in Figure 4.42(b). The black arrows in Figure 4.42(b) indicate grain boundary locations where surface fiducial scratches are jogged. Thus, it appeared that the grain boundary cracking was associated with, and may have been accommodating, the GBS.



Figure 4.41. SE SEM images of an annealed sample taken during an *in-situ* creep test at 50 MPa and 150 $^{\circ}$ C. The displacement were indicated in the images. A significant amount of grain boundary cracking was evident.











Figure 4.42 (a) Low-magnification and (b) high-magnification SE SEM images of the surface of an annealed AM60 sample after *in-situ* creep testing at 150 °C and 75 MPa. The final displacement was 3.13 mm. The fiducial scratches indicate offsets at α -phase grain boundaries, as indicated by the black arrows in (b). Some slip traces were observed, as indicated by the white arrows in (b).

4.2 AZ31 Alloy

In-situ tests were performed on the AZ31 alloy with the tensile axis parallel to the RD or the TD of the as-received sheet. The tests were performed at 50 °C, 150 °C, and 250 °C. A total of seven experiments were performed and at least two tests were performed at each temperature. EBSD scans were performed on selected areas of the samples both before and after the *in-situ* test, and SEM images of these areas were also taken at desired intervals during the *in-situ* tensile test. The active deformation modes, including slip and twinning, were determined based on the *in-situ* SEM images and the EBSD information. AFM scans were performed on randomly selected areas within the deformed gage section of some of the tensile tested AZ31 samples in order to evaluate the GBS activity. Two samples, one along the RD and the other one along the TD, were creep-tested at 150 °C and 75 MPa, with SEM images acquired *in-situ* without interrupting the experiment.

4.2.1 In-situ Tensile Test

The *in-situ* tensile stress versus displacement curves corresponding to the seven tests performed at the different test temperatures are plotted in Figure 4.43. It should be noted that the displacement values recorded included both the deformation of the gage section and non-gage section. Little difference was observed between the RD and TD tests. Samples were not taken to failure, in order to limit the deterioration of the sample surface so that EBSD orientation mapping could be performed after the test without further preparation. However, each experiment was performed to a displacement value greater than 0.2 mm (which corresponded to a strain of ~20%). The yield stress was approximately 110 MPa, 60 MPa, and 40 MPa at 50 $^{\circ}$ C,

150 °C, and 250 °C, respectively. These values are comparable to those found in the literature for a textured AZ31 (Khan *et al.* 2011).

The width and thickness of the samples tested along the RD were measured both before and after the tensile test. These values were used to determine the plastic strain ratio (*r*-value). Figure 4.44 shows the calculated *r*-value for samples tested at the three different temperatures in the current study, as well as the *r*-values measured in similar studies (Stanford *et al.* 2011, Agnew and Duygulu 2005, Barnett *et al.* 2009). Comparable results were obtained in these studies.



Figure 4.43. Stress versus displacement plots for AZ31 samples tensile tested at 50 °C, 150 °C, and 250 °C. The load drops indicate the stress relaxation that occurred when the experiment was paused to acquire SEM images.



Figure 4.44. The plastic strain ratio (*r*-value) measured for the current study as compared with literature values (Stanford *et al.* 2011, Agnew and Duygulu 2005, Barnett *et al.* 2009).

The active slip and twinning systems were identified using the EBSD data and the SEM images acquired during the *in-situ* tensile experiment. The slip and twinning modes considered in the deformation analysis include the following:

- 1. {0001}<2110>: basal <*a*> slip
- 2. $\{0110\} < 2110 >:$ first-order prismatic <a> slip
- 3. $\{1122\} < \Gamma \Gamma 23 >:$ second-order pyramidal < c+a > slip
- 4. {1012}<1011>: extension twin
- 5. $\{10\Gamma1\} < 10\Gamma2 >:$ contraction twin

Figure 4.45 illustrates an example of the slip trace methodology used for the active slip system identification. Figure 4.45(a)-(d) shows SEM images of the same region in an AZ31 sample taken at different strain levels during the *in-situ* test. The strain value was estimated by measuring the change in distance between identifiable features on the *in-situ* SEM images, therefore it represented the local strain for the area where the analysis was performed. Slip traces in the grain indicated by the black arrow was observed from ~5% strain, and the trace directions are illustrated by the blue lines. Figure 4.45(e) shows the EBSD IPF map for this region, taken before the tensile deformation. From the EBSD information, slip traces for all possible slip systems can be calculated. Two of the calculated slip traces represented by the red line and black line, which were closest to the observed slip trace direction, are shown in Figure 4.45(e). It can be found that with increasing deformation, the slip traces observed on the SEM images changed their direction, i.e., rotated clockwise. Therefore, the slip traces calculated based on pre-deformation EBSD scan with directions rotated counter-clockwise to the observed slip traces appeared to be reasonable choices for possible active slip system. Moreover, in this example, multiple possible slip systems showed similar calculated slip trace directions. The red line in Figure 4.45(e) indicates the calculated slip trace direction for basal slip system (0001)[121 0], which had a Schmid factor of 0.47. The black line in Figure 4.45(e) indicates the calculated slip trace direction for pyramidal $\langle c+a \rangle$ slip sytem (1122)[TT23], which had a Schmid factor of 0.31. Thus, the basal slip system, which had a higher Schmid factor, was determined to be the active slip system in this example. A similar method was used to identify the active twinning systems. The surface trace direction for all the possible slip and twinning systems were calculated using a MATLAB code that used the Euler angles of the grains as an input parameter. Details on the calculation and the codes can be found in APPENDIX D.



Figure 4.45. Examples of slip trace analysis performed on the grain indicated by the black arrow in (b). (a)-(d) are SE SEM images taken from an AZ31 sample tested along RD at 150 °C, and different tensile strain levels: (a) 0%, (b) 5%, (c) 11%, and (d) 21%. (e) is the EBSD IPF map for this region, taken before the tensile deformation. The color code corresponds to the ND IPF. The slip trace directions observed at different strain levels was illustrated by the green lines of different patterns. The red line in(e) indicated the calculated slip trace direction for basal slip system (0001)[Γ 210], which had a Schmid factor of 0.47. The black line in (e) indicated the calculated slip trace direction for pyramidal $\langle c+a \rangle$ slip system (1122)[Γ 123], which had a Schmid factor of 0.31. The basal slip system was chosen to be the active slip system.




4.2.1.1 50 °C Tensile Deformation

Figure 4.46 shows the EBSD IPF maps and the corresponding pole figures, both before and after the 50 $^{\circ}$ C tensile test, for the same area in an AZ31 sample. As shown in Figure 4.46(a), the EBSD IPF map had most of the grains in red, which is typical for the studied AZ31 sample in the undeformed state. This suggests that the undeformed sample exhibited a basal texture in which the basal planes were mostly aligned parallel to the sheet surface. The pole figures show that basal poles were mainly aligned in the ND, with a maximum intensity of ~20 times random, and the (1010) prismatic poles spread uniformly on the RD-TD plane.

The basal texture was maintained after deformation, see Figure 4.46(b). A six-fold symmetry was observed on the (1010) pole figure, and the alignment of (1010) prismatic poles with the RD (tensile axis) revealed the occurrence of prismatic slip. Additionally, the (0001) poles were slightly inclined towards the TD. An new texture component was also observed in the TD on the (0001) pole figure, indicating that some of the basal poles were aligned parallel to the TD of the sample, which is in agreement with the fact the some grains with bright blue color were observed on the EBSD IPF map after deformation. This revealed the activation of extension twinning.



Figure 4.46. EBSD IPF maps and the corresponding pole figures for the same area in an AZ31 sample tested at 50 °C along the RD: (a) before testing and (b) after ~15% tensile strain. The color code of the EBSD maps corresponds to the ND IPF.

During the *in-situ* experiment, twinning was observed to be operative at the early stages of deformation (~2-3% strain). Slip traces were also observed after the sample reached ~4% strain, but not in large quantities. Furthermore, some cracks became apparent at the grain boundaries as the deformation increased. Figure 4.47(a)-(d) shows SEM images of the same region taken at different strain levels during the *in-situ* experiment. Deformation twins were found in grains labeled as 1, 2, and 3 at \sim 3% strain, as shown in Figure 4.47(a). With increasing deformation, the twin area increased, and some additional twinning was observed in grains 1 and 4 at ~10% strain, see Figure 4.47(c). With further deformation, these twins ultimately consumed a large part of the parent grain, or even the entire parent grain. Figure 4.47(e) and (f) show the EBSD IPF map of this region before and after the tensile test. The reorientation of the grains due to twinning is indicated by the unit cell drawn on the map. The orientation relationships between the reoriented grain and the parent grain were all around 89° about the <1120> direction, as summarized in Table 4.9. This suggests the formation of $\{102\} < 101 >$ extension twin, which theoretically reorients the lattice 86.3° about the $\langle 1120 \rangle$ direction. Extension twins are not inherently favored considering the texture and loading direction of the sample. The Schmid factors for twinning were small, see Table 4.9, suggesting that twinning is easily activated at low temperature compared with the other deformation modes.



Figure 4.47. SE SEM images for an AZ31 sample tested at $50 \,^{\circ}$ C along RD and different displacement values: (a) 1.02 mm (~3% strain), (b) 1.57 mm (~6% strain), (c) 2.60 mm (~10% strain), and (d) 3.69 mm (~15% strain). (e) EBSD IPF map before test, and (f) EBSD IPF map after test. The color code corresponds to the ND IPF. Twinning was observed in the grains labeled 1-4.

Figure 4.47 (cont'd)



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Figure 4.47 (cont'd)



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Grain	Orientation relationship between	Twin type	Schmid Factor
	parent grain and reoriented grain		for twinning
1	89.7 °@<1120>	(1102)[1101] extension twin	0.05
2	88.0 °@<1120>	(1102)[1101] extension twin	0.05
3	89.7 °@<1210>	(T012)[10T1] extension twin	0.08
4	89.6 °@<2110>	(0112)[0111] extension twin	0.05

Table 4.9. Twinning modes observed for area in Figure 4.47 at 50 $^{\circ}$ C.

In order to better understand the deformation mechanisms controlling plasticity under the current test conditions, the active deformation mechanisms in 25 grains were investigated by combined SEM and EBSD observations. Thirteen grains were observed to reorient due to deformation twinning, and the data suggest that all underwent extension twinning. No contraction twins were observed. The extension twinning formed at early stages of deformation (mostly less than 3% strain) and low Schmid factors. Slip was observed in 12 grains. Figure 4.48 summarizes the Schmid factor distribution and strain levels at which the identified deformation modes were observed. Among the 12 slip traces analyzed, one was basal slip, ten were prismatic $\langle a \rangle$ slip, and one was pyramidal $\langle c+a \rangle$ slip. Figure 4.49 shows the IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at 50 °C. For most of the grains in which prismatic slip was observed, the tensile axis lay near the arc of the unit triangle. While for the grains in which extension twinning was activated, the tensile axis lay in the middle part of the unit triangle.



Figure 4.48. The Schmid factor distribution and strain levels at which twinning and slip traces were observed for each of the deformation modes for AZ31 samples tensile tested at 50 $^{\circ}$ C.



Figure 4.49. IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at 50 C.

4.2.1.2 150 °C Tensile Deformation

At 150 °C, tensile tests were performed on two AZ31 samples along the RD, and one sample along the TD. Figure 4.50 shows the EBSD IPF maps and the corresponding pole figures, before and after the test, for the same area in an AZ31 sample deformed along RD. The grains in the EBSD IPF map after deformation did not exhibit a significant change in color compared to that before deformation, which is different from the observation for samples tested at 50 $^{\circ}$ C, as shown in Figure 4.46. This suggests that deformation, such as twinning, which leads to a severe change in grain orientation might not have been activated in the samples tested at 150 °C. This is also consistent with the observation that the texture component <0001>||TD was not observed in the pole figure for sample deformed at 150 °C. However, the deformed sample showed a six-fold symmetry in the (1010) pole figure with an alignment of the (1010) poles in the RD (tensile axis), indicating the activation of prismatic slip, which is similar to the observation for the 50 $^{\circ}$ C tested sample. Figure 4.51 shows the pole figures of an AZ31 sample tested at $150 \,^{\circ}$ C along the TD. Similar to the sample tested along the RD, a six-fold symmetry was observed in the (1010) pole figure for the sample tested along the TD, and the (1010) poles were also aligned with the tensile axis, which is the TD for this sample.

Figure 4.52 illustrates the SEM images of the same area of a sample tested along RD in the undeformed state and different strain levels. Slip traces were first observed between $\sim 2\%$ and 5% strain. An increase in the density of slip traces can be clearly observed with increasing strain in Figures 4.52(a)-(d). The appearance of some grain boundary ledges could be also distinguished after $\sim 2\%$ strain. As the deformation proceeded, some grain boundary cracks became apparent. These cracks tended to form on grain boundaries oriented perpendicular to the tensile axis.



Figure 4.50. EBSD IPF maps and the corresponding pole figures for the same area in an AZ31 sample tested at 150 $^{\circ}$ C along the RD: (a) before testing and (b) after ~21% tensile strain. The color code of the EBSD map corresponds to the ND IPF.



Figure 4.51. Pole figures for the same area in an AZ31 sample tested at 150 $^{\circ}$ C along the TD: (a) before testing and (b) after ~12% tensile strain.



Figure 4.52. SE SEM images for an AZ31 sample tensile tested at $150 \,^{\circ}$ C along the RD. The approximate strain values were: (a) 0%, (b) 2%, (c) 5%, and (d) 21%.



In the three samples tested at 150 °C, the active slip systems were determined for 41 different slip traces. Figure 4.53 summarizes the Schmid factor distribution and stress levels at which slip traces were observed for each slip system in the samples tested at 150 °C. Among the 41 slip traces analyzed, 17 were basal slip, 18 were prismatic slip, and six were pyramidal $\langle c+a \rangle$ slip. No twinning was observed. Basal slip and pyramidal $\langle c+a \rangle$ slip occurred over a wide range of Schmid factors. With increasing strain, more basal slip and pyramidal $\langle c+a \rangle$ slip with smaller Schmid factors were activated. Prismatic $\langle a \rangle$ slip was activated only when a high Schmid factor (>0.4) was present. Figure 4.54 shows the IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at 150 °C. Similar to the observation for the 50 °C tested samples, most of the grains in which prismatic slip was identified had the tensile axis located near the arc of the unit triangle. For the grains in which basal and pyramidal $\langle c+a \rangle$ slip was activated, the tensile axis orientation was distributed over a large part of the unit triangle.



Figure 4.53. The Schmid factor distributions and strain levels at which the slip trace was observed for each of the slip systems for AZ31 samples tensile tested at $150 \,^{\circ}\text{C}$.



Figure 4.54. IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at 150 $^{\circ}$ C.

4.2.1.3 250 °C Tensile Deformation

Figure 4.55 shows the EBSD IPF maps and the corresponding pole figures before and after the test for the same area of an AZ31 sample tested along the RD at 250 °C. This figure shows that the deformation texture that developed at 250 °C is different from that formed at lower temperatures (50-150 °C). Basically, no texture variation with respect to the initial texture was developed at 250 °C, indicating that plastic strain was controlled by different deformation modes than those controlling plasticity at lower temperatures.

Figure 4.56 shows SEM images of an AZ31 sample tested along the RD at 250 $^{\circ}$ C. As shown in the images, the slip traces were more obvious than in the samples tested at 50 $^{\circ}$ C and 150 $^{\circ}$ C. As the deformation proceeded, some grain boundary relief was observed. Grain boundaries became wavy as soon as the test started.



Figure 4.55. EBSD IPF maps and the corresponding pole figures for the same area in an AZ31 sample tested at 250 $^{\circ}$ C along the RD: (a) before testing and (b) after ~15% tensile strain. The color code of the EBSD maps corresponds to the ND IPF.



Figure 4.56. SE SEM images of an AZ31 sample tensile tested at 250 $^{\circ}$ C along the RD. The approximate strain values were: (a) 1%, and (b) 15%. The tensile direction was horizontal in the images.

The slip systems were determined for 47 slip traces in the two samples tested at 250 °C. Out of the 47 slip traces, ten were basal slip, 36 were prismatic $\langle a \rangle$ slip, and one was pyramidal $\langle c+a \rangle$ slip. Thus, it appears that prismatic $\langle a \rangle$ slip became dominant and more easily activated at higher temperatures compared to basal slip. This would be expected based on the literature (Kelly and Hosford 1968, Agnew and Duygulu 2005, Agnew *et al.* 2003, Barnett 2003, Lou *et al.* 2007, Christian and Mahajan 1995), since the CRSS for prismatic and pyramidal slip systems decreases as the temperature increases while basal and twining are relatively independent of temperature. Figure 4.57 depicts both the distribution of the Schmid factors for each of the slip traces observed and the strain levels at which the slip traces were observed. Most of the active slip systems exhibited a relatively high Schmid factor (>0.30). Basal slip occurred evenly over a wide strain level, while prismatic slip was prevalent at higher strain levels.

Figure 4.58 shows the IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at $250 \,$ °C. Most of the grains in which prismatic slip was identified had the tensile axis located near the arc of the unit triangle. For the grains in which basal slip was identified, the tensile axis tended to lie in the middle part of the unit triangle.



Figure 4.57. The Schmid factor distributions and strain levels at which the slip trace was observed for each of the slip systems for AZ31 samples tensile tested at $250 \,^{\circ}$ C.



Figure 4.58. IPF of the tensile direction for grains in which different deformation modes were observed in AZ31 samples tensile tested at 250 $^{\circ}$ C.

4.2.2 In-situ Creep Test

In-situ creep tests were performed on the AZ31 alloy along RD and TD, at 75 MPa and 150 °C. The creep displacement versus time curves, compared with those of the AM60 alloy, are shown in Figure 4.59. The common primary and secondary stages of creep were exhibited. There was little difference between the different orientations in terms of the creep strains and strain rates. Figure 4.60 shows SEM images of an AZ31 sample taken at different strain levels during the creep test. Grain boundaries served as crack nucleation sites during creep. No slip traces were observed. However, it is noted that during the 150 °C *in-situ* tensile test, four basal slip traces and six prismatic slip traces, with Schmid factors between 0.35 and 0.5, were observed at a strain less than ~3%, which corresponded to a stress less than ~78 MPa.



Figure 4.59. Creep displacement versus time plots for AZ31 samples tested along RD and TD at 150 $^{\circ}$ C and 75 MPa. Also shown are curves for AM60 annealed samples tested under identical conditions.



Figure 4.60. SE SEM images taken for the same area during a creep test at $150 \,^{\circ}$ C and 75 MPa for a sample tested along TD. The creep time and approximate strain are (a) 0.1 h, 1%, (b) 1.1 h, 2%, (c) 12 h, 5%, and (d) 45.6 h, 14%.



4.2.3 AFM Analysis

AFM was performed on the tensile deformed AZ31 samples in order to evaluate the GBS activity. The scanned area was randomly selected from the deformed gage section of the samples. The grain boundary step height was measured by performing "section analysis", in which the sample surface height along a line across the grain boundary was resolved, and used to calculate the height of grain boundary steps representing the extent of GBS activity. All the grain boundaries in each scanned area were analyzed in order to get statistically significant information.

Figure 4.61 shows an example of grain boundary step height measurement for an AZ31 sample tested at 50 °C. The two-dimensional (2D) and three-dimensional (3D) images of the sample surface are illustrated in Figures 4.61(a) and (b), and the surface height along the line drawn in Figure 4.61(a) is shown in Figure 4.61(c). The red arrows in Figure 4.61(a) indicate the two edges of grain boundaries on both sides of the sheared region at the grain boundary. A drop in the surface height was observed in Figure 4.61(c) between the two arrows, and the grain boundary step height was determined to be ~530 nm.

Figure 4.62 shows an example of a grain boundary that did not exhibit sliding/shearing. Although a drop of surface height was observed between the two red arrows as indicated in Figure 4.62(c), the grain boundary was located between the two red arrows, as illustrated in Figure 4.62(a). Therefore, it appeared that deformation had occurred near the grain boundary region, but the grain boundary itself did not exhibit sliding.



Figure 4.61. (a) 2D- and (b) 3D- AFM images of an AZ31 sample tensile tested at 50 °C, and (c) plot illustrating the surface height along the line drawn in (a). The grain boundary step height was determined to be \sim 530 nm.



Figure 4.62. (a) 2D- and (b) 3D- AFM images of an AZ31 sample tensile tested at 50 $^{\circ}$ C, and (c) plot illustrating the surface height along the line drawn in (a). The grain boundary did not exhibit shearing.

Figures 4.63, 4.64, and 4.65 illustrate representative AFM images for the scanned areas in samples tested at 50 °C, 150 °C, and 250 °C, respectively. At 50 °C, only part of the grain boundaries analyzed exhibited sliding. As shown in Figure 4.63(a), a relatively large amount of sliding (~480 nm step height) was identified at grain boundary 1, a relatively small amount of sliding (~20 nm step height) was identified at grain boundary 2, and no sliding could be resolved at grain boundary 3. At 150 °C and 250 °C, all grain boundaries analyzed exhibited sliding, and the amount of sliding appeared to increase with increasing temperature. Moreover, the grain boundaries were wavy in the 250 °C tested samples. Cracks were observed at grain boundaries, or even in the grain interior near boundaries, as indicated by the black arrow in Figure 4.65(a).



Figure 4.63. (a) 2D- and (b) 3D- AFM images of an AZ31 sample tensile tested at 50 $^{\circ}$ C to a tensile strain of ~15%. Grain boundary 1 exhibited an ~480 nm step height. Grain boundary 2 exhibited an ~20 nm step height. No sliding could be resolved at grain boundary 3.



Figure 4.64. (a) 2D- and (b) 3D- AFM images of an AZ31 sample tensile tested at 150 % to a tensile strain of ~21%.



Figure 4.65. (a) 2D- and (b) 3D- AFM images of an AZ31 sample tensile tested at $250 \,^{\circ}$ C to a tensile strain of ~15%. The black arrow in (a) indicates a crack in the interior of a grain.

A summary of the AFM analysis performed on the AZ31 alloy is shown in Table 4.10. The strain in the length and width direction was estimated by measuring the change in distance between identifiable features on the *in-situ* SEM images before and after deformation. The strain in the thickness direction was calculated by assuming a constant volume of the sample during deformation. The GBS strain (the strain accommodated by GBS) was estimated using the ratio between the average grain boundary step height and the average grain diameter, which is 13 μ m for the studied AZ31 alloy. The contribution of GBS to the strain in the length or width direction. For example, the average grain boundary step height for the 50 °C tested samples was 50 nm. Therefore, the GBS strain was estimated to be 50 nm divided by 13 μ m, which is 0.0038 (or 0.38%). From *in-situ* SEM images, the strain in the length direction was estimated to be 0.38% divided by 15%, which is 0.025 (or 2.5%).

As shown in Table 4.10, at 50 °C, only ~40% of the grain boundaries analyzed exhibited sliding, while at 150 °C and 250 °C, all grain boundaries analyzed exhibited sliding. With increasing temperature, the average grain boundary step height increased from ~50 nm to ~322 nm. Considering that the tensile strain was similar between the different samples analyzed, it appeared that GBS activity was enhanced with increasing temperature. Moreover, from 50 °C to 250 °C, the contribution of GBS to the strain in the length direction increased by a factor of ~7, from 2.5% to 17%. The contribution of GBS to the strain in the thickness direction increased by a factor of ~4, from 8.8% to 39%. Therefore, it appeared that at 250 °C, GBS had a significant contribution to the reduction of the thickness of the sample.

	50 °C	150 °C	250 °C
Number of grain boundaries analyzed	55	33	26
Number of grain boundaries exhibited sliding	22	33	26
Average grain boundary step height, nm (STDEV)	50 (108)	223 (156)	322 (232)
GBS strain	0.38%	1.7%	2.5%
Strain in length direction	15%	21%	15%
Strain in width direction	-9.1%	-11%	-7.1%
Strain in thickness direction	-4.3%	-7.1%	-6.4%
Contribution of GBS to strain in length direction	2.5%	8.1%	17%
Contribution of GBS to strain in thickness direction	8.8%	24%	39%
Plastic strain ratio (r-value)	4.1	2.3	1.6

Table 4.10. Summary of AFM analysis on GBS activity in the AZ31 samples.

4.3 MN11 Alloy

Similar to the experiments performed on AZ31 alloy, *in-situ* tensile tests were performed along the extrusion direction (ED) of the MN11 alloy at 50 °C, 150 °C, and 250 °C. EBSD scans were performed on selected areas in the gage section of the samples both before and after the *in-situ* tests, and SEM images of these areas were taken at desired intervals during the test. The active deformation modes including slip and twinning were determined using the same method as that used for the AZ31 alloy.

The stress versus displacement curves are plotted in Figure 4.66. Samples were not taken to failure, in order to limit the deterioration of the sample surface so that EBSD could be performed after the test without further preparation. However, each experiment was performed to a displacement value greater than 0.2 mm. The yield stress was approximately 100 MPa, 80 MPa, and 70 MPa at 50 °C, 150 °C, and 250 °C, respectively. These values are comparable to those found in the literature for extruded MN11 (Bohlen *et al.* 2010). The plastic strain ratio was determined to be close to unity for the MN11 samples tested at all three temperatures, indicating this alloy exhibited isotropic behavior unlike that for the AZ31 alloy, which exhibited a temperature dependent *r*-value, as previously described in section 4.2.1.



Figure 4.66. Stress versus displacement plots for MN11 samples tensile tested at 50 °C, 150 °C, and 250 °C. The load drops indicate the stress relaxation that occurred when the experiment was paused to acquire SEM images.

Figure 4.67 shows the EBSD IPF map and the corresponding pole figure of an MN11 sample without deformation. The as-extruded rod exhibited a weak texture, as reported previously (Bohlen *et al.* 2010), and similar weak textures have been observed in other Mg-RE alloys (Stanford 2010). The average equiaxed grain diameter was approximately 5 μ m. As shown in Figure 4.67(b), the (0001) poles were tilted to the left and right toward the ED, with a maximum intensity of ~2.8 times random.

Figure 4.68 shows the EBSD IPF map and the corresponding pole figure for another MN11 sample. A banded feature was distinctive on the EBSD IPF map. Some grains with relatively larger size formed a band in the upper-middle part of the map, dividing bands of grains with relatively smaller grain size. Similar banded feature was also observed in other Mg-RE alloys (Stanford 2010). It was suggested the smaller grains, which were recrystallized grains during hot extrusion, had their c-axis tilted away from the TD and toward the ED (Stanford 2010). Moreover, grains within the same band exhibited a common, similar orientation, i.e., with the c-axis either tilted to the left or to the right toward the extrusion direction (Stanford 2010). As shown in Figure 4.68(b), the (0001) poles were mostly tilted to the right toward the ED, which is different from that in Figure 4.67(b). This suggests that a different local grain orientation distribution might be obtained for different areas analyzed.



Figure 4.67. (a) EBSD IPF map and (b) the corresponding pole figure of an MN11 sample without deformation. The color code of the EBSD map corresponds to the ND IPF.


Figure 4.68. (a) EBSD IPF map and (b) the corresponding pole figure of an MN11 sample without deformation. The color code of the EBSD map corresponds to the ND IPF. The EBSD scanned area is smaller than that in Figure 4.67. Note that this is a different sample from that in Figure 4.67.

4.3.1.1 50 °C Tensile Deformation

After tensile deformation at 50 $^{\circ}$ C, the weak texture in the MN11 alloy was retained, and no distinctive difference was observed on the pole figure compared to that before deformation.

During the *in-situ* experiment at 50 °C, twinning was observed at the early stages of deformation (95-112 MPa / 0-2% strain). However, the amount of twinning was not large enough to introduce significant changes in the pole figures after deformation. Slip traces were observed after the sample reached ~4% strain. Figure 4.69 shows SEM images of the same region in an MN11 sample, taken at different strain levels during the test. Bright particles were aligned as stringers in the extrusion direction, indicating that the cast billet was inhomogeneous with respect to the alloying elements. EDS analysis showed that these second-phase particles were enriched in Nd content. Due to the low volume fraction of these second-phase particles, their effect on the deformation mechanisms was considered to be negligible.

Deformation twins were observed in several grains, such as the ones labeled as A, B, C, D, E, and F in Figure 4.69. For grains A, B, D, and F, twinning was first observed at ~2% strain. As the deformation proceeded, the twin area fraction increased, and twinning ultimately consumed a larger part of the parent grain. Figure 4.69 also shows the EBSD IPF map of this region both before (Figure 4.69(f)) and after (Figure 4.69(g)) the tensile test. The reorientation of the grains due to twinning is indicated by the unit cell drawn on the map. For grains C and E, the twinned region is not distinctive on the SEM image, but can be identified on the IPF map. The orientation relationship between the reoriented grain and the parent grain, as summarized in Table 4.11, ranged between 84.5-89.8 ° about the <1120> direction. This suggests the formation of the {102}<1101> extension twin, which ideally reorients the lattice 86.3 ° about the <1120> direction. No contraction twins were identified within the region of observation.



Figure 4.69. SE SEM images of an MN11 sample tensile tested at 50 $^{\circ}$ C along the extrusion direction, taken at different displacement values: (a) 0.15 mm (~0% strain), (b) 0.33 mm (~2% strain), (c) 0.69 mm (~4% strain), (d) 1.70 mm (~10% strain), and (e) 2.72 mm (~16% strain). (f) EBSD IPF map before test, and (g) EBSD IPF map after test. The color code corresponds to the ND IPF. Twinning was observed in the grains labeled as A-F in the figures after strains of greater than 2%.



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Table 4.11. Twinning modes observed for area in Figure 4.69 at 50 °C.

Grain	Orientation relationship between parent grain and reoriented grain	Twin type	Schmid Factor for twinning
А	88.2 °@<2110>	(10f2)[f011] extension twin	0.15
В	85.0 °@<1210>	(1012)[1011] extension twin	0.26
С	88.9 °@<1210>	(10f2)[f011] extension twin	0.30
D	87.9 °@<2110>	(0112)[0111] extension twin	0.35
E	84.5 °@<1210>	(0112)[0111] extension twin	0.07
F	89.8 °@<1210>	(1012)[101] extension twin	0.23

The active deformation modes were determined for 29 slip traces and 13 twins in different grains for the MN11 samples tested at 50 °C. Figure 4.70 summarizes the Schmid factor distribution for the identified slip and twinning activities. Among the 29 slip traces analyzed, 21 were basal $\langle a \rangle$ slip, two were first order prismatic $\langle a \rangle$ slip, and six were second order pyramidal $\langle c+a \rangle$ slip. Basal slip and extension twinning were the dominant deformation modes. Extension twinning occurred over a wide Schmid factor range, even when the Schmid factor was around 0.15. Figure 4.71 shows the IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at 50 °C. For most of the grains in which extension twinning or basal slip was activated, the tensile axis lay in the middle part of the unit triangle. For most of the grains in which pyramidal $\langle c+a \rangle$ slip was observed, the tensile axis orientation was distributed over a large part of the unit triangle.



Figure 4.70. The Schmid factor distribution of the observed deformation modes for MN11 samples tensile tested at 50 $^{\circ}$ C.



Figure 4.71. IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at 50 $^{\circ}$ C.

4.3.1.2 150 °C Tensile Deformation

Similar to the tensile test at 50 °C, the weak texture of the MN11 sample was retained after tensile test at 150 °C, and no distinctive difference could be observed on the pole figure compared to that before deformation.

As was found at 50 °C, extension twinning was observed at 150 °C during the early stages of deformation (81-94 MPa / 2-3% strain). The relative amount of twinning was significantly lower than that observed at 50 °C. No contraction twins were found. Figure 4.72 shows the SEM images of the same area of an MN11 sample at different strain levels during the tensile test. Slip traces were first observed between ~3-6% strain. An increase in the density of slip traces can be clearly observed with increasing strain in Figures 4.72(a)-(d).

In Figure 4.72, grain A represents an example in which the slip trace became wavy as the deformation increased. This suggests the activation of dislocation cross-slip. In some other grains, wavy slip traces were also observed. However, the slip trace direction was generally not clear enough due to the severe deformation of the sample, so the corresponding slip systems were not identified. In a few grains, slip traces for two slip systems with different Burgers vectors were observed. For instance, grain B contained two sets of slip traces corresponding to different prismatic slip systems: $(10\Gamma0)[1210]$ and $(\Gamma100)[1120]$. Both of them had a Schmid factor larger than 0.4.



Figure 4.72. SE SEM images for an MN11 sample tensile tested at 150 $^{\circ}$ C along the extrusion direction, at approximate strain values of: (a) 0%, (b) 6%, (c) 19%, and (d) 23%. Cross-slip was observed in grain A. In grain B, two sets of slip traces were observed, and the Schmid factor and predicted trace direction for the most likely slip systems calculated from the EBSD data are labeled in (d). The tensile direction was horizontal in the images.



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In the samples tested at 150 °C, the active deformation modes were determined for 46 different slip traces and three twins. Figure 4.73 summarizes the Schmid factor distribution and strain levels at which slip traces were observed for each slip system in these samples. Among the 46 slip traces analyzed, 29 were basal $\langle a \rangle$ slip, 14 were prismatic $\langle a \rangle$ slip, and three were second order pyramidal $\langle c+a \rangle$ slip. Most slip occurred with Schmid factors greater than 0.4.

Figure 4.74 shows the IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at $150 \,^{\circ}$ C. Most of the grains in which basal slip was activated had the tensile axis orientation distributed in the middle part of the unit triangle, and most of the grains in which prismatic slip was identified had the tensile axis orientation distributed near the arc of the IPF, which is similar to observations made for the AZ31 alloy.



Figure 4.73. The Schmid factor distributions and strain levels at which twinning and slip traces were observed for each of the deformation modes for MN11 samples tensile tested at $150 \,^{\circ}$ C.



Figure 4.74. IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at $150 \,^{\circ}$ C.

4.3.1.3 250 °C Tensile Deformation

Similar to the tensile tests at 50 $^{\circ}$ C and 150 $^{\circ}$ C, the weak texture of the MN11 sample was retained after tensile testing at 250 $^{\circ}$ C, and no distinctive differences could be observed on the pole figure compared to that before deformation.

In the samples tested at 250 °C, the slip traces and grain boundaries were more obvious than in the samples tested at lower temperatures. Figure 4.75 shows SEM images of the same area of an MN11 sample tested at 250 °C at different strain levels. Some grain boundary ledges could be distinguished at about 3% strain, as shown in Figure 4.75(b), while slip traces were first observed at ~3-6% strain. Some slip traces were wavy-type, such as in grain A in Figure 4.75(d).

The slip systems were determined for 119 slip traces in the samples tested at 250 °C: 105 were basal $\langle a \rangle$ slip, four was prismatic slip, and ten were second order pyramidal $\langle c+a \rangle$ slip. This indicates that basal $\langle a \rangle$ slip was the dominant deformation mode at high temperatures. Figure 4.76 depicts the distribution of the Schmid factors for each of the slip traces observed and the corresponding strain levels. More active slip systems with relatively lower Schmid factors were observed in the samples tested at 250 °C compared to the specimens tested at 150 °C.

Figure 4.77 shows the IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at 250 °C. Most of the grains exhibited basal slip, and the tensile direction were mainly distributed in the middle part of the unit triangle. For most of the grains in which pyramidal $\langle c+a \rangle$ slip was observed, the tensile axis orientation was distributed over a large part of the unit triangle.



Figure 4.75. SE SEM images for an MN11 sample tensile tested at 250 $^{\circ}$ C along the extrusion direction, at approximate strain values of: (a) 0%, (b) 3%, (c) 6%, and (d) 17%. Grain boundary ledges and slip traces are indicated by the arrows. Wavy slip traces are indicated in grain A.



(d)



Figure 4.76. The Schmid factor distributions and strain levels at which the slip traces were observed for each of the deformation modes for MN11 samples tensile tested at 250 $^{\circ}$ C.



Figure 4.77. IPF of the tensile direction for grains in which different deformation modes were observed in MN11 samples tensile tested at $250 \,^{\circ}$ C.

CHAPTER 5

DISCUSSION

This chapter provides a detailed analysis and discussion of the results presented in Chapter 4. First, the effect of thermomechanical processing and subsequent heat treatment on the microstructure and mechanical properties (including tensile, fatigue, and creep properties) of the AM60 alloy is discussed. An emphasis is put on the fatigue crack growth behavior. The effect of maximum stress, processing, and stress ratio on the fatigue crack growth behavior of the AM60 alloy is evaluated, and the applicability of different fracture mechanics models to the AM60 alloy is discussed. Next, the deformation mechanisms of AZ31 alloy at different temperatures is discussed. An explanation to the different plastic anisotropy observed at different temperatures is explained based on the observed slip activity and GBS activity. Finally, the deformation mechanism of MN11 at different temperatures is discussed, and compared to that of AZ31.

5.1 AM60 Alloy

5.1.1 Microstructure Discussion

The studied AM60 alloy has two major alloying elements, Al (6.29 wt%) and Mn (0.28 wt%). The RT solubility of Mn in Mg solid solution is very low (less than 0.1 wt%) (see Figure 2.4), and together the Mn and Al form Mn₅Al₈ precipitates in this alloy. As shown in the Mg-Al binary phase diagram (Figure 2.1), the RT solubility of Al in Mg solid solution is less than 1 wt%. The microstructure of Mg-Al alloys generally consists of the α -Mg solid solution and the intermetallic γ -phase (or β -phase) Mg₁₇Al₁₂. During non-equilibrium solidification of common

Mg-Al alloys, an α + γ eutectic microstructure is commonly formed at the interdendritic regions, and the morphology is dependent on the alloy composition as well as the solidification condition (Chadha *et al.* 2007, Braszczynska-Malik 2011). Generally, with the Al concentration decreasing from ~33 wt% to less than 10 wt% in a Mg-Al alloy, the morphology of the eutectic changes from lamellar to partially divorced, and finally to a fully-divorced microstructure (Chadha *et al.* 2007). For the AM60 alloy in the current study, a divorced eutectic morphology was observed at the grain boundary regions, and no lamellar eutectic morphology was observed (see Figures 4.1 and 4.2). This is typical for Mg-Al alloys solidified under a fast cooling rate (Gertsman *et al.* 2005, Dantzig and Rappaz 2009).

As noted in Table 4.1, the volume fraction of the grain boundary phases was slightly smaller in the annealed AM60 material compared with the as-molded and TTMP materials. It is likely that dissolution of this phase occurred during the 300 °C annealing treatment and the elements redistributed into the matrix α -phase solid solution. The dissolution of the grain boundary γ -phase was observed previously during hot working of AZ31 alloys (Mwembela *et al.* 1997, Myshlyaev *et al.* 2002). As seen in the Mg-Al binary phase diagram (Figure 2.1), the solubility of Al in Mg solid solution is around 6 wt% at 300 °C. Moreover, Ren *et al.* (2012) determined the phase equilibriums in the Mg-Al binary system at 250-400 °C, and found that at 300 °C the equilibrium composition of the α -Mg phase contains 6.6 at% Al. This is higher than the concentration of Al elements in the AM60 alloy. Therefore, it is expected that all the γ -phase would be dissolved if an equilibrium state is reached at 300 °C. However, a complete dissolution of the γ -phase might require a significantly long time.

A more likely explanation is that the measured grain boundary phases are likely to be a mixture of the γ -phase and a supersaturated α -Mg solid solution with a high Al concentration

based on their similar contrast using BSE imaging. Based on the phase diagram, the α -phase component in the $\alpha + \gamma$ eutectic is expected to have a high Al concentration. In addition, a concentration gradient can be formed during non-equilibrium solidification, leading to higher Al concentration in the grain boundary regions. For example, a network of the Mg matrix with high Al concentration, referred to as solidification cells, is commonly observed in the grain boundary regions in high pressure die cast Mg-Al alloys such as AM50 (Gertsman et al. 2005). Both of the above-mentioned reasons could explain the higher Al concentration in the grain boundary regions for the AM60 alloy in the current study. In Figure 5.1(a), the bright phase with irregular shape is likely to be the Mg₁₇Al₁₂ γ -phase in the divorced eutectic microstructure. The medium contrast component, forming a network surrounding the α -grains as well as the γ -phases, could be the supersaturated α -Mg solid solution with a high Al concentration. It is possible that, during the annealing process, the high Al contents in the supersaturated α -Mg solid solution could diffuse into the interior of the α -matrix. Therefore, as shown in Figure 5.1(b), the medium contrast component at the grain boundaries was reduced in volume fraction. It is important to note that the exact procedure for the annealing treatment was not provided by the manufacturer. However, it appeared that the annealed material did not have an equilibrium microstructure.

The microprobe data for the phase composition analysis was shown in Table 4.2. Taking into account the standard deviation, the difference in the composition determined for the matrix phase in the as-molded, TTMP, and annealed materials was considered to be insignificant. The measurement of the γ -phase showed a higher Mg/Al ratio than the nominal ratio of 17/12, and this is likely to be related to the interaction volume encompassing both the grain boundary γ -phase and the matrix α -phase.



Figure 5.1. BSE SEM images of undeformed AM60 alloys: (a) as-molded, and (b) annealed. The medium-contrast grain boundary network was much less apparent in the annealed material.

The 300 \C rolling treatment was performed on the as-molded AM60 alloy with the aim of refining the grain size and eutectic phases (Huang *et al.* 2010). However, it is clearly shown in Figure 4.4 that the rolling treatment decreased the porosity of the material, as no porosity segregation band was observed in the TTMP and annealed materials. This was expected to be beneficial to the mechanical properties of this alloy.

Grain refinement achieved by dynamic recrystallization (DRX) during hot working has been widely reported for Mg alloys (Ion *et al.* 1982, Mackenzie *et al.* 2007, Myshlyaev *et al.* 2002). During the processing, small recrystallized grains form at the boundaries of large unrecrystallized grains, and they can often form a banded structure, which can have orientations favorable for deformation. As shown in Figures 4.1 (d)-(f) and Figure 4.5(b), severe deformation revealed by the elongated grains was observed in the TTMP material. However, the formation of dynamically recrystallized grains was not readily discernable. It is speculated that nuclei for new grains might have formed in the TTMP material, but there was no extensive grain growth.

Berman *et al.* (2011) studied the microstructure evolution of an Thixomolded® AZ61 alloy during TTMP treatment similar to the one performed in this study. They found both recrystallized α -grains and refined γ -particles in the TTMP sample. However, the recrystallized α -grains and refined γ -particles did not exhibit unique morphologies or contrast, and were therefore differentiated based on the EDS analysis (Berman *et al.* 2011). Huang *et al.* (2010) suggested that the γ -particles in the Mg alloy can be subdivided, dissolved, and reprecipitated during the rolling process, and the redistributed fine γ -particles at the grain boundaries can retard grain growth.

Figure 5.2 shows a TEM image of the TTMP AM60 material in this study. Although no EDS analysis was taken for this region, by comparing the microstructure with that found in the

literature (Berman *et al.* 2011), it is possible that the small grains indicated by arrows in Figure 5.2 could be either recrystallized α -grains or refined γ -particles. The size of this grains are on the order of 100nm, suggesting that there was not extensive grain growth.

As shown in Figure 4.5, the microstructure for the annealed material is significantly different from that for the TTMP material. Smaller, equiaxed grains were observed in the annealed AM60 alloy, and some of them occupied regions that previously contained large grains before annealing. Therefore, static recrystallization and grain growth might have occurred in the annealed material, and the newly formed equiaxed grains might have grown from the nuclei formed during the rolling treatment.



Figure 5.2. A bright-field TEM image of a TTMP AM60 sample.

The as-molded AM60 alloy exhibited a random texture. As shown in Figure 4.6(c), the relative intensity for different peaks in the XRD intensity versus 20 plots are similar between the face section and longitudinal section. In addition, the relative intensity for different peaks corresponded well to the powder diffraction data listed in Table 4.3, which represents the intensity for peaks in a sample with random texture. Moreover, as shown in Figure 4.8(b), the EBSD pole figure did not exhibit strong texture components. A random texture is typical for both as-Thixomolded® Mg alloys and as-cast Mg alloys (Berman *et al.* 2011, Agnew *et al.* 2001, Perez-Prado *et al.* 2004).

The TTMP AM60 material exhibited a basal texture. As shown in Figure 4.6(a), the XRD intensity versus 20 plot for the face section showed a (0002) peak with an intensity much stronger than that for all the other peaks, while the intensity for this peak was only ~36% of the strongest peak in the powder diffraction data (Table 4.3). This suggested that the basal planes were preferentially aligned parallel to the RD-TD plane of the TTMP sample. Due to the severe deformation of the TTMP material, EBSD was not successful.

The annealed materials exhibited similar XRD intensity versus 20 plots as the TTMP material, see Figures 4.6(a) and (b), suggesting that the basal texture was retained after annealing. This is in agreement with observations that the recrystallization texture for conventional Mg alloys is similar to the deformation texture (Mackenzie *et al.* 2007, Bohlen *et al.* 2010). However, comparing the EBSD IPF maps and the pole figures of the annealed AM60 alloy (Figures 4.9 and 4.10) with those of the rolled + annealed AZ31 alloy (Figure 4.46), it can be seen that the texture of the annealed AM60 alloy was much weaker, with a moderate intensity of only ~3-6 times of random. Similarly, a weak basal texture was also observed in a

Thixomolded® AZ61 alloy processed using a similar TTMP and annealing treatment (Berman *et al.* 2011).

A more random texture has been suggested to be beneficial for formability (Bohlen *et al.* 2007). In addition, as reported in sections 4.1.2 and 4.1.3, the tensile strength and fatigue strength of the TTMP and annealed AM60 materials were both greater than those for the as-molded material. Therefore, the thermomechanical processing and subsequent annealing appear to be effective treatments for achieving both enhanced tensile and fatigue strength and formability of the as-molded AM60 alloy.

5.1.2 Tensile Behavior Discussion

The thermomechanical processing and subsequent heat treatment significantly altered the tensile properties of the AM60 alloy. As shown in Table 4.4, the TTMP material exhibited significantly greater tensile strength compared to the as-molded material without an expense of ε_{f} . The annealed material, which exhibited a tensile strength intermediate to that of the as-molded and TTMP materials, showed significantly larger ε_{f} .

The fact that the TTMP material exhibited the highest tensile strength may be explained by the texture and strain hardening. At low temperatures, basal slip is the most easily activated slip system in Mg. However, in the TTMP material, the basal planes tended to be aligned nearly parallel to the sheet surface, which resulted in a very low Schmid factor for basal slip during tensile deformation along the RD. This means most of the grains were in a "hard orientation". The strain hardening was introduced by the rolling process, which led to severe deformation of the material, thereby leaving a high dislocation density. The significant strain hardening exhibited by the TTMP material was consistent with its much smaller strain hardening exponent compared to the as-molded and annealed AM60 materials, see Table 4.4.

The annealed material exhibited a higher tensile strength compared to the as-molded material. This is likely due to the texture and grain refinement. The basal texture of the TTMP material was retained after annealing, so most of the grains were still in a hard orientation under a tensile stress along the RD. The reduced grain size of the annealed material may be explained by static recrystallization without extensive grain growth. Such strengthening by grain size refinement has generally been explained by the Hall-Petch relationship (Hall 1951, Petch 1953). In addition, the greater pore volume fraction in the as-molded material is expected to have prevented this microstructure from reaching its potential strength due to porosity induced fracture.

Although the studied AM60 alloy exhibits lower tensile strengths compared with most other commonly used structural alloys such as steel, Ti alloy, Al alloys, and Ni alloys, their specific strength is relatively high due to their low density. At RT, the specific UTS values for the TTMP AM60 alloy was ~204 kN m/kg. For comparison, the specific UTS was ~90-280 kN m/kg for an AISI 4340 steel (Lee and Su 1999), ~90-150 kN m/kg for an Al alloy 7075 (Kumar *et al.* 2012), ~205-265 kN m/kg for an Ti-6Al-4V alloy (Venkatesh *et al.* 2009), and ~95-145 kN m/kg for an Ni-Cr alloy 625 (Mathew *et al.* 1999). This suggests that the studied AM60 alloy is comparable with other structural alloys on a density-normalized basis. Moreover, the lower density of Mg alloys means that for structures such as beams which are used under bending conditions, sections of larger thickness can be made without increasing the overall weight. As the strength of a structure under bending is proportional to the square of the section thickness, Mg alloys are considered to be favorable for applications which require superior

bending or buckling strength (Easton *et al.* 2006). This is important for applications in the automotive, sports and recreation, biomedical, electronic, and other industries. The thermomechanical processing and subsequent heat treatment processing might be a viable route to process other stronger Mg-based alloys that have potential for implementation in structural applications.

5.1.3 Fatigue Behavior Discussion

As illustrated in Figure 4.13, the increased temperature did not significantly affect N_f , and each processing treatment performed on the as-molded AM60 alloy resulted in greater fatigue strength. Previous studies on cast AM60 alloys showed that the RT fatigue strength was generally between 50 MPa and 70 MPa (Mayer et al. 2003, Khan et al. 2008, Kulyasova et al. 2009). The current study showed that the fatigue strength of the as-molded AM60 alloy was similar to that of cast AM60 alloys. In addition, through thermomechanical processing and subsequent heat treatment of the Thixomolded® AM60 alloy, the fatigue strength can be almost doubled. This improvement is of similar magnitude as that involved in tensile strengthening. As shown in Figure 4.14, the fatigue ratio (the ratio between fatigue strength and UTS) was greater than 0.4 for the annealed AM60 alloy, and was ~0.3 for the as-molded and TTMP materials. This is comparable to Al alloys, which exhibit a fatigue ratio of around 0.3-0.4, and slightly lower than that of steel and Ti alloys, which exhibit fatigue ratios of around 0.5 (Hertzberg 1989). Thus, the fatigue ratio of lightweight Mg alloys is within the range exhibited by other structural metals with greater densities. This would make Mg alloys quite attractive on a density normalized basis.

A number of studies have been performed to investigate the effect of microstructure, processing, and alloy composition on the fatigue behavior of Mg alloys. Kulyasova et al. (2009) suggested that the increase in the fatigue strength of an AM60 alloy was related to the decrease in grain size introduced by equal-channel angular pressing (ECAP), and the effect of grain size can be well described by a Hall-Petch type relationship. Khan et al. (2006) found that small additions of Mn up to 0.4 wt% in Mg-Al alloys led to significant grain refinement with a corresponding increase in tensile strengths and fatigue lives. However, Mn concentrations higher than 0.58 wt% led to a decrease in the fatigue lives, possibly due to the intermetallic precipitates assisting in crack initiation and propagation. Mayer et al. (2003) studied the influence of porosity on the fatigue strength of several high-pressure die cast Mg alloys and found that more than 98% of the failed samples suffered from crack initiation at pores. Uematsu et al. (2009) studied the effect of friction stir processing (FSP) on the fatigue behavior of a cast AZ91 alloy in which no porosity was observed. They found that cracks initiated at the intermetallic phase in the as-cast alloy, whereas in the FSP processed alloy, cracks initiated due to cyclic slip. The increased crack initiation resistance was attributed to grain refinement and the breaking up of coarse precipitates due to FSP (Uematsu et al. 2009). The small crack growth resistance was also enhanced, and this was attributed to grain refinement and the increase in hardness due to FSP (Uematsu et al. 2009). Thus, the above mentioned studies highlight that grain refinement is beneficial for tensile and fatigue strength, which is consistent with the results of the current work.

Fracture surface analysis in the current study showed that, for the as-molded AM60 alloy, fatigue cracks initiated preferentially at large pores (mostly had a diameter larger than 100 μ m) near the sample surface. This means that the as-molded microstructure might not have been low

in fatigue resistance intrinsically. As shown in Figure 4.4(a), the as-molded material showed a relatively high porosity (~2% pore volume fraction). After the rolling process, the porosity was significantly reduced, and pores were not apparent in Figure 4.4(b). As a result, the TTMP and annealed materials mainly exhibited crack initiation at surface locations, especially at precipitated particles, or where microcracks formed probably caused by cyclic slip. This suggests that porosity is a very important microstructure feature that affects crack initiation, which is also documented in literatures elsewhere (Horstemeyer *et al.* 2002, Mayer *et al.* 2003, Gall *et al.* 2004).

At 150 °C, a few as-molded samples exhibited surface crack initiation sites. In addition, the EDS data gathered at the crack initiation site shown in Figure 4.19 indicated high levels of oxygen. Therefore, environmental effects, such as exposures at elevated temperatures, might also significantly influence the fatigue crack initiation, as has been documented for titanium alloys (Chen and Boehlert 2008).

The fatigue crack growth behavior will be further discussed in detail in the following section (Section 5.1.4).

5.1.4 Fatigue Crack Growth Behavior Discussion

In the following discussion, the fatigue crack growth curves of different specimens shown in Figure 4.25 will be selected for comparison in order to evaluate the effects of processing and mechanical loading parameters on the fatigue crack growth behavior of AM60.

5.1.4.1 Effect of Maximum Stress

As shown in Figure 4.27 and Figure B.1 in Appendix B, for specimens with the same processing condition tested at the same stress ratio, increasing the maximum stress alone did not lead to a discernible change in the average crack growth rate.

The effect of maximum stress on fatigue crack growth rate can be accounted for using LEFM relationships if similitude and small scale yielding conditions apply. At a fixed stress ratio, K_{max} and ΔK are linearly related by Equation (2.11), so both Equation (2.9) and Equation (2.10) would predict a crack growth rate that is only dependent on ΔK . In other words, a change in the maximum stress would not lead to a change in the crack growth rate at the same value of ΔK . However, it was shown that for small crack growth experiments on Al alloys, increasing the maximum stress led to an increase in the crack growth rate at an equivalent ΔK value (Shyam *et al.* 2005). This result was related to the breakdown of the small scale yielding assumption as the crack size became comparable to the plastic zone size at the crack tip.

In the current study, changing the maximum stress did not result in a discernible change in the crack growth rate. Generally, a faster growth rate may be expected for a small crack below a critical size compared to a long crack at equivalent ΔK . The critical crack size was suggested by several criteria. Taylor and Knott (1981) suggested the critical crack size to be ten times that of the grain size. Lankford (1985) suggested the critical crack size was that which resulted in a plastic zone size equal to the grain size. Kitagawa and Takahashi (1976) showed that LEFM broke down for cracks smaller than a critical size a_0 . The value of a_0 can be estimated using the following equation:

$$a_0 = \frac{1}{\pi} \left(\frac{\Delta K_0}{\sigma_{\rm e}}\right)^2 \tag{5.1}$$

where ΔK_0 is the fatigue crack growth threshold for long cracks, and σ_e is the fatigue strength. Assuming ΔK_0 to be equal to the ΔK_{th} shown in Table 4.5 and σ_e to be 60 MPa, 110 MPa, and 135 MPa for the as-molded, TTMP, and annealed materials tested at R = 0.1, respectively, the critical crack size a_0 was calculated using Equation (5.1), as summarized in Table 5.1. It can be seen that the calculated a_0 values were between 430-470 µm for the as-molded material, between 32-360 µm for the TTMP material, and between 51-100 µm for the annealed material. In the current study, the notch size was ~800 µm for the as-molded samples and ~400 µm for the TTMP and annealed samples. Thus, the crack sizes were larger than the critical value above which LEFM relationships are applicable in most testing conditions. This is consistent with the experimental results suggesting that the LEFM relationships were applicable to the Mg alloy with the experimental variables used in the current study.

Table 5.1. Summary of the estimated ΔK_{th} and critical crack length (*a*₀) for the AM60 fatigue crack growth samples tested at *R* = 0.1.

Condition	$\sigma_{ m max}$, MPa	R	$\Delta K_{\text{th}}, \text{MPa}\sqrt{\text{m}}$	<i>a</i> ₀ , μm
as-mold	80	0.1	2.2	430
as-mold	105	0.1	2.3	470
TTMP	200	0.1	1.1	32
TTMP	265	0.1	2.0	110
TTMP	300	0.1	3.7	360
annealed	140	0.1	1.7	51
annealed	180	0.1	2.4	100
annealed	200	0.1	1.8	58

It is important to note that ASTM E647 ('Standard Test Method for Measurement of Fatigue Crack Growth Rates' 2008) suggested that crack growth rates between 10^{-9} and 10^{-10} m/cycle should be used to estimate a ΔK value corresponding to a crack growth rate of 10^{-10} m/cycle, and this ΔK value should be used to represent ΔK_{th} . In the current study, because the measured crack growth rates were all greater than 10^{-9} m/cycle, the ΔK corresponding to a crack growth rate of 10^{-9} m/cycle was used to represent ΔK_{th} . Therefore, the ΔK_{th} values shown in Table 5.1 might be larger than the values determined by following ASTM E647 strictly. If smaller ΔK_{th} values were determined, critical crack length (a_0) values smaller than those determined in Table 5.1 are also expected. However, they should still be smaller than the artificial notch sizes, which is not contradictory to the applicability of LEFM relationships to the current testing conditions.

Although the LEFM applies to the AM60 alloy, it was not applicable for the previouslystudied Al alloys for similar σ_{max}/σ_{ys} ratios (Shyam *et al.* 2004, 2005, 2007, Shyam and Lara-Curzio 2010). One possible reason for this is the low ε_f of the AM60 alloy compared with the previously-studied Al alloys. The higher Paris law exponent exhibited by the AM60 alloy compared to the previously-studied Al alloys appears to be consistent with the lower ε_f observed in the AM60 alloy. Shyam and Lara-Curzio (2010) proposed the following relationship:

$$m = 4 - 2\theta \tag{5.2}$$

where *m* is the Paris law exponent, and θ is an experimental constant. A larger θ value, and therefore a smaller *m* value, can be found in a material with a higher ε_{f} . As shown in Table 4.5,

in the current study, the Paris law exponent of the AM60 alloy was ~4. This implies a small θ value that corresponds to less plastic deformation exhibited at the crack tip. On the other hand, the Paris law exponents of ~2 have been observed for Al alloys (Shyam *et al.* 2007, Shyam and Lara-Curzio 2010). This corresponds to a larger θ value, i.e., more extensive plastic deformation exhibited at the crack tip. Thus, it is reasonable to believe that the LEFM assumptions are likely to be appropriate for the AM60 alloy, and the application of the LEFM relationships can reasonably model the crack growth rates.

5.1.4.2 Effect of Processing

Figure 4.28 compares the fatigue crack growth rate of the AM60 alloys tested at given stress ratios and different maximum stress levels. As shown in Section 5.1.4.1, changing the maximum stress did not lead to a discernible change in the crack growth rate for samples of the same processing condition tested at the same stress ratio. Thus, any noticeable difference in the da/dN versus ΔK behavior in Figure 4.28 could be attributed to the effect of processing. However, this was not the case, as in each plot in Figure 4.28 each processed material exhibited a similar crack growth rate at equivalent ΔK values. Thus, it can be concluded that the processing condition did not have a significant effect on the fatigue crack growth rate of the AM60 alloy.

Moreover, it is important to note that although the annealed material exhibited the highest fatigue strength, the fatigue crack growth rate of the annealed material was not lower than that of the TTMP material at equivalent ΔK . Thus, the improvement in the fatigue strength of the annealed material compared to the other conditions was not likely to be a result of an increase in the fatigue crack growth resistance. It is more likely that the increase in fatigue strength of the

annealed material was due to its higher fatigue crack initiation resistance, which in turn is related to the microstructure and size distribution of the defects in the alloys.

5.1.4.3 Effect of Stress Ratio

Based on the relationship shown in Equation (2.11), Equation (2.10) can be re-written as:

$$\frac{da}{dN} = C \frac{(\Delta K)^{p+n}}{(1-R)^p} = C (K_{\max})^{p+n} (1-R)^n$$
(5.3)

Thus, it is expected that increasing the *R* value would lead to an increase of the crack growth rate at an equivalent ΔK . Increasing the *R* value would also lead to a decrease of the crack growth rate at the equivalent K_{max} . Figure 4.29 shows the fatigue crack growth curves for the annealed material tested at a maximum stress of 180 MPa and three different stress ratios (R = 0.1, 0.3, and 0.5). As predicted by Equation (5.3), with the increase of stress ratio, the crack growth curve shifted to higher growth rates in the d*a*/d*N* versus ΔK plot (see Figure 4.29(a)), and shifted to lower growth rates in the d*a*/d*N* versus K_{max} plot (see Figure 4.29(b)). These demonstrate the limitation of the Paris-Law-type fatigue crack growth relationships. That is, for different stress ratios, the fitting parameters for the fatigue crack growth curve need to be re-determined.

Shyam *et al.* (2005) found that in some Al alloys, the crack growth rate is nearly linearly related to the product of the monotonic crack tip displacement (ϕ_m) and cyclic crack tip displacement (ϕ_c). They proposed the following relationship:

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \kappa \,\phi_{\mathrm{m}} \,\phi_{\mathrm{c}} \tag{5.4}$$

The definition of $\phi_{\rm m}$ and $\phi_{\rm c}$ can be found in Equations (2.12) and (2.13). As shown in Figure 4.29(c), for the annealed AM60 alloys tested at maximum stress of 180 MPa, different samples exhibited similar crack growth rates at equivalent $\phi_{\rm m}\phi_{\rm c}$ values, and the curves corresponding to different specimens fell into a narrow band. Shyam *et al.* (2007) have shown that the relationship described in Equation (5.4) applies well to a number of alloys, including Al alloys, Ni-based alloys, and Ti alloys. The current work suggests that the fatigue crack growth rate in the AM60 alloys can also be characterized using this relationship, which indicated the general applicability of this model. Therefore, a single relationship might be established to characterize the fatigue crack growth rate of materials over a wide range of experimental variables, including processing condition, maximum stress level, and stress ratio.

5.1.4.4 Fractography

As shown in Figures 4.30, 4.31, and 4.32, flat cleavage planes with striation-like patterns and rough crack growth regions with randomly oriented serrated surfaces were commonly observed on the fracture surface. Similar features on the fracture surface were also observed in a high cycle fatigue study of a cast AM60 alloy (Horstemeyer *et al.* 2002). The flat cleavage planes with striation-like patterns were correlated with transgranular crack growth through the dendritic cells, and the rough regions with serrated surfaces were correlated with intergranular crack growth through the β -Mg₁₇Al₁₂ particle-laden interdendritic arms (Horstemeyer *et al.* 2002). A transition from the former crack growth mechanism to the latter with the increase of the crack growth driving force, i.e., the stress intensity factor, was observed, accompanied by a change in the brightness of the fracture surfaces. This was attributed to whether the driving force was large enough to prematurely fracture/debond the β -particles (Horstemeyer *et al.* 2002).

As described in Section 4.1.4.2, the current study showed a transition in the predominant fatigue crack growth mechanism from transgranular crack growth to intergranular crack growth with the increase of the crack length. This observation could be rationalized as follows: The region near the notch corresponded to a relatively small crack size, and thus a small driving force. Moreover, the plastic zone size could possibly be smaller than the grain/dendrite arm size, so only individual grains deformed around the crack tip. As a result, the cracks tended to propagate through the grains and avoid grain boundaries. When cracks propagated to a region far from the notch, the driving force became larger due to a larger crack size, and the plastic zone became large enough to encompass many grain/dendrite arms. Thus, grain boundary cracking could be accomplished with the higher stress intensity factor associated with the longer crack.

The approximate value for a transition crack length (the cleavage features were mostly observed in fatigue crack propagation regions corresponding to a crack length smaller than this transition crack length), summarized in Table 4.7, was determined based on extensive fracture surface observations. The ΔK value corresponding to the transition crack length, ΔK_{tran} , was determined for each of the specimens, as shown in Table 5.2. These ΔK_{tran} values ranged between 2.3 to 3.4 MPa \sqrt{m} , which is comparable to the ΔK value that corresponded to the transition in the fatigue crack propagation mechanism in research elsewhere (Horstemeyer *et al.* 2002). According to Shyam *et al.* (2004), the scaled monotonic plastic zone size (r_{pz}) at the crack tip can be calculated by the following equation:
$$r_{\rm pz} = \phi \, a \, (\sec(\frac{\pi \sigma_{\rm max}}{2\sigma_{\rm vs}}) - 1) \tag{5.5}$$

where ϕ is a scaling factor whose applicability has been justified by experimental measurements (Edwards and Zhang 1994a, 1994b). The value of ϕ is set to be 0.05, which is obtained by visually determining the approximate plastic zone size from images of the cracks at high growth rates. The calculated r_{pz} values for each sample were summarized in Table 5.2, and it can be seen that these values were typically of an order of magnitude larger than the grain size. This is reasonable based on the previous explanation of the transition in the preferred fatigue crack growth mechanisms.

It should also be noted that, no distinctive macroscale boundaries between regions with the two different features can be observed from the low-magnification images of the fracture surface. This is different from an earlier study (Horstemeyer *et al.* 2002). In fact, a large number of the studied regions possessed a mixture of the transgranular crack growth and intergranular crack growth features, such as the region shown in Figure 4.31(b). Gall *et al.* (2004, 2005) suggested that, in general, small cracks propagated preferentially through the relatively soft Mg dendritic cells. However, microporosity in interdendritic regions or slip incompatibility between the α -Mg cells could locally amplify the crack tip driving force, making interdendritic regions the preferential fatigue crack growth path (Gall *et al.* 2004, 2005). Therefore, the mixture of the transgranular and intergranular crack growth path observed in the current study was possibly caused by the inhomogeneity of the microstructure in some regions, due to defects such as a high porosity, which might lead to a difference in the driving force as well as the local microstructural strength along the crack front. It is also important to note that, due to the mixed transgranular and intergranular crack growth features in the fatigue crack growth region, the transition crack length shown in Table 5.2 was actually an upper bound value determined for the crack length at which the crack growth mechanism changed. If these values were reduced, r_{pz} values closer to the average grain sizes would be expected.

Table 5.2. Summary of the approximate transition crack length and the corresponding stress intensity factor range value (ΔK_{tran}) and scaled monotonic plastic zone size (r_{pz}).

Condition	$\sigma_{ m max}$, MPa	R	Transition crack length, µm	ΔK_{tran} , MPa $\sqrt{\text{m}}$	r _{pz} , μm
as-mold	80	0.1	990	2.9	37
as-mold	105	0.1	520	2.6	59
TTMP	265	0.3	140	2.6	15
TTMP	265	0.5	220	2.3	25
annealed	140	0.1	410	3.0	16
annealed	200	0.1	260	3.4	57
annealed	140	0.3	580	2.9	22
annealed	180	0.3	430	3.1	46
annealed	140	0.5	740	2.3	28
annealed	180	0.5	600	2.7	64

Figure 4.35 summarized the stress ranges and the number of cycles in which crack initiation was observed at the artificial notch for all the specimens tested. For the as-molded material, the crack initiation resistance in a notched sample appeared to be lower compared to the other two processing conditions. Although the annealed material exhibited the highest fatigue strength, the crack initiation resistance in a notched sample of the annealed material did not appear to be higher than that of the TTMP material. Moreover, even though a notch was machined on the surface of every specimen before testing, cracks did not always initiate from the notch. As shown in Table 4.8, for all the seven annealed specimens tested, cracks initiated from the notch. For the as-molded and TTMP materials, more than half of the specimens exhibited crack initiation for the primary crack at locations other than the machined notch. This suggested that some internal defects in these materials may have acted as higher stress concentration sites.

As shown in Figure 4.33, a high level of porosity was observed in the as-molded material, and the pores served as crack initiation sites and/or assisted the fatigue crack growth process. Moreover, in Figure 4.25, the as-molded specimen tested at $\sigma_{max} = 105$ MPa and R = 0.1 exhibited a fatigue crack growth curve with a large slope corresponding to a Paris law exponent around 8, which was much larger than that for other specimens. This might be related to the high level of porosity of the as-molded material and merging of cracks that led to artificial acceleration in the crack emanating from the notch. In Figure 4.34, the defect near the specimen surface of the TTMP material might have been introduced during the rolling process. Thus, a higher defect density in the as-molded and TTMP materials may be the cause for their lower fatigue strength compared to the annealed material.

Representative images for the fracture surface of samples that have the primary crack initiated from the artificial notch are illustrated in Figures 4.30, 4.31, and 4.32. The boundaries

between the fatigue crack propagation region and the fast fracture region can be identified in the images. Therefore, it is possible to determine the crack length of the samples just before fracture, and calculate the critical stress intensity factor (K_C) values corresponding to fracture. These values can be considered as a "non-plane-strain fracture toughness" for the specific testing conditions in the current study, because the samples do not meet the requirement for determining a plane-strain fracture toughness (i.e., K_{IC}) as suggested by ASTM E399 ('Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials' 2000). Table 5.3 shows a summary of these "non-plane-strain fracture toughness" (i.e., $K_{\rm C}$) values calculated using Equation (A.1). It can be seen that, compared to the as-molded material, both the TTMP and the annealed materials exhibited a higher toughness when fracture occurred in a non-plane-strain condition. It is important to note that in the current study, the stress/strain state in the samples cannot be simplified as a pure plane-stress or plane-strain condition. Therefore, although the $K_{\rm C}$ values may be used to infer the K_{IC} of the materials, the actual K_{IC} values might differ considerably from the $K_{\rm C}$ values determined in the current study.

Condition	$\sigma_{ m max}$, MPa	R	$K_{\rm C}$, MPa $\sqrt{\rm m}$
as-mold	80	0.1	8.9
as-mold	105	0.1	9.7
TTMP	200	0.1	12.1
TTMP	265	0.1	14.9
TTMP	265	0.3	16.6
TTMP	265	0.5	18.5
annealed	180	0.1	15.5
annealed	180	0.3	14.7
annealed	180	0.5	13.6

Table 5.3. Summary of the "non-plane-strain fracture toughness" (K_C) for the AM60 fatigue crack growth samples.

5.1.5 Creep Behavior Discussion

5.1.5.1 Creep Stress Exponent and Activation Energy

As shown in Figure 4.38, the measured creep stress exponents at 150 °C were 4.9, 5.8, and 4.5 for the as-molded, TTMP, and annealed AM60 alloy, respectively. This implies that dislocation climb is the dominant creep mechanism controlling the secondary creep rate. Similar creep stress exponents have been reported in a number of studies, and these have been correlated with a dislocation creep mechanism. Mabuchi *et al.* (1996) reported n = 5 for a Mg-6Si alloy tested at strain rates of $2 \times 10^{-5} - 2 \times 10^{-2} \text{s}^{-1}$ and temperature range of 400-500 °C. Regev *et al.* (1997) reported that for a die-cast AZ91 alloy tested under applied stress range of 30-100 MPa, the value of *n* was 6.9 at 150 °C, and was 5.4 at 180 °C. Agnew *et al.* (2000) reported that for a die cast AM60 alloy creep tested at 150 °C, n = 4 was found for an applied stress range of 20-60 MPa. A much higher stress exponent, n = 10, was found for an applied stress range of 60-100 MPa, which indicated the power law breakdown (Agnew *et al.* 2000). Maruyama *et al.* (2002) showed that for Mg-Al alloys creep tested at 323 °C, the stress exponent changed from 4 to 7 when the stress increased from 5 MPa to 50 MPa, which corresponded to a transition in the creep mechanism from an alloy-type (controlled by the glide of dislocations dragging a solute atmosphere) to a metal-type (controlled by the recovery of dislocation substructure). Boehlert (2007) reported n = 4.2-7.7 for three Mg-Zn-Zr alloys creep tested at 150 °C under applied stress range of 30-50 MPa.

However, it has been reported in a number of studies that with decreasing applied stress, decreased stress exponents were observed. Vagarali and Langdon (1981) showed that for a polycrystal Mg creep tested between 323 °C and 473 °C, n = 6 was found at applied stress larger than 2.5 MPa, and n = 1 was found at applied stress smaller than 2.5 MPa. Mabuchi *et al.* (1996) reported that for a Mg-4Si-4Zn alloy tested at a temperature range of 400-500 °C, n = 4 was found for strain rates larger than 2×10^{-4} s⁻¹, and n = 2 was found for strain rates smaller than 2×10^{-4} s⁻¹. Luo *et al.* (2002) reported that for a Mg-Al-Ca alloy creep tested at 175 °C, n = 8.5was found for a stress range of 70-97 MPa, and n = 1.5 was found for a stress range of 42-70 MPa. Boehlert (2007) reported that for a Mg-5.4Zn-0.6Zr alloy creep tested at 150 °C, n = 6.8was found for a stress range of 40-83 MPa, and n = 2.2 was found for a stress range of 20-40 MPa. The decreased stress exponents were generally considered as an indication for a transition in the dominant creep mechanism. The creep stress exponents around 2 were commonly related to a GBS creep mechanism (Mabuchi et al. 1996, Luo et al. 2002, Del Valle et al. 2005, Del Valle and Ruano 2007, Boehlert 2007). The stress exponent n = 1 in the research performed by Vagarali and Langdon (1981) was attributed to a Nabarro-Herring creep mechanism.

As shown in Figure 4.39, the temperature dependence of the minimum creep rate under an applied stress of 20 MPa showed a two-stage behavior. For the temperature range of 150200 °C, the activation energies were 128 kJ/mol, 174 kJ/mol, and 126 kJ/mol for the as-molded, TTMP, and annealed AM60 alloys, respectively. For the temperature range of 100-150 °C, the activation energies were 48 kJ/mol, 64 kJ/mol and 67 kJ/mol for the as-molded, TTMP, and annealed materials, respectively. The measured activation energies in the high temperature regime (150-200 °C) were comparable to the lattice self-diffusion activation energy in Mg, which is 135 kJ/mol (Shewmon and Rhines 1954), and the chemical inter-diffusion energy of Al in Mg, which is 143 kJ/mol (Dargusch and Dunlop 1998). The measured activation energy for grain boundary diffusion in Mg, which is 92 kJ/mol (Frost and Ashby 1982), and the activation energy for discontinuous grain boundary precipitation of Mg₁₇Al₁₂ during aging in Mg-Al alloys, which is 30 kJ/mol (Uchida and Shinya 1995).

Different activation energies can be related to different creep mechanisms. Vagarali and Langdon (1981) reported that the activation energy for a polycrystal Mg creep tested at a temperature higher than 323-473 °C under an applied stress larger than 2.5 MPa was ~220 kJ/mol. Extensive non-basal slip was observed, and the creep mechanism was suggested to be cross-slip controlled dislocation creep (Vagarali and Langdon 1981). Mabuchi *et al.* (1996) reported that for a Mg-4Si-4Zn alloy tested at a temperature range of 400-500 °C, Q = 150 kJ/mol was found for strain rates larger than 2×10^{-4} s⁻¹, which was related to dislocation creep mechanism, and Q = 109 kJ/mol was found for strain rates smaller than 2×10^{-4} s⁻¹, which was related to grain boundary diffusion controlled GBS mechanism. Dargusch and Dunlop (1998) reported activation energies between 30 and 45 kJ/mol for creep of AZ91, AS21, and AE42 alloys creep tested at 150 °C under applied stress range of 20-80 MPa, and related them to GBS mechanism

promoted by discontinuous precipitation of Mg₁₇Al₁₂. Luo *et al.* (2002) reported that for a Mg-Al-Ca alloy creep tested at 83 MPa, Q = 120 kJ/mol was found for a temperature range of 150-200 °C, and Q = 40 kJ/mol was found for a stress range of 100-150 °C. The higher activation energy was related to the dislocation creep mechanism, and the lower activation energy was related to the GBS type creep mechanism (Luo *et al.* 2002). Boehlert (2007) reported that for a Mg-5.4Zn-0.6Zr alloy creep tested at 30 MPa, Q = 131 kJ/mol was found for a temperature range of 100-150 °C, which was related to dislocation creep mechanism, and Q = 66 kJ/mol was found for a temperature range of 150-200 °C, which was related to a GBS creep mechanism.

The above results suggest that for the AM60 alloys creep tested at 20 MPa in the current study, both the dislocation creep mechanism, which corresponds to the higher activation energy, and the GBS creep mechanism, which corresponds to the lower activation energy, might be operating in parallel and competing with each other. Temperature may have an influence on this competition. In the temperature range of $100-150 \,\text{C}$, GBS creep is the dominant creep mechanism; at temperatures range of $150-200 \,\text{C}$, dislocation creep is the dominant creep mechanism.

The activation of GBS was also revealed by the observations during *in-situ* creep experiments. As shown in Figure 4.41, void formation and grain boundary cracking was evident during the creep test. The jog of the surface fiducial scratch in Figure 4.42(b) clearly revealed the relative displacement of grains along the grain boundaries. Although not many, some slip traces were also observed in some of the grains in Figure 4.42(b). Thus, both grain boundary cracking and dislocation slip may have been accommodating GBS.

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5.1.5.2 The Role of γ-phase in Creep

Mg-Al alloys often exhibit limited creep resistance, and various explanations have been provided in different studies. In general, the limited creep resistance was attributed to the poor thermal stability of the γ -Mg₁₇Al₁₂ phase (Friedrich and Mordike 2006). In addition, some studies showed that the dynamic precipitation of γ -Mg₁₇Al₁₂ in Mg-Al alloys creep tested at \sim 200 °C can induce a dilation of the Mg lattice, potentially enhancing the creep rate under tensile loading compared to compressive loading (Agnew et al. 2000, Payzant et al. 2001). Dargusch and Dunlop (1998) suggested that for die cast Mg-Al alloys creep tested at 150 °C under applied stress range of 20-80 MPa, grain boundary sliding and migration aided by decomposition of the supersaturated α -Mg and discontinuous precipitation of γ -Mg₁₇Al₁₂ was the dominant creep mechanism. However, other studies suggested that the γ -Mg₁₇Al₁₂ precipitates did not have a detrimental effect for creep behavior of the Mg-Al alloys at temperatures around 150 °C. Fukuchi and Watanabe (1975) suggested that a noticeable softening of the γ -phase could only be found at temperatures of 260 °C and above. It was also shown that for die cast Mg-Al alloys creep tested at 150 °C and 50MPa, increasing Al concentrations led to an increase in the creep resistance (Dargusch *et al.* 2006). Therefore, the role of the γ -phase in creep behavior of Mg-Al alloys is still under debate. However, it appears that if the γ -phase were detrimental to the creep property, it is likely due to the dynamic precipitation process, rather than the γ -phase itself.

Dargusch and Dunlop (1998) studied the creep behavior of AZ91, AS21, and AE42 Mg alloys tested at 150 °C. Significant discontinuous precipitation of the γ -Mg₁₇Al₁₂ phase due to the decomposition of the supersaturated α -Mg was observed in AZ91, which is a Mg alloy with

relatively high Al concentration. Much less precipitation was observed in the more creep resistant AS21 and AE42 alloys. However, the similar activation energies suggested that the creep deformations for all three alloys were dominated by grain boundary sliding and migration mechanism promoted by the discontinuous precipitation of the γ -Mg₁₇Al₁₂ (Dargusch and Dunlop 1998). Figure 5.3 shows a BSE SEM image of an as-molded AM60 sample in the current study, which has been subjected to a temperature-jump test at 100-200 °C, under applied stress of 20 MPa, for over 830 hours. Compared to the as-molded microstructure before creep testing, which is represented by Figure 5.1(a), the supersaturated α -Mg solid solution at the grain boundary regions was reduced after creep testing, see Figure 5.3. There appeared to be some precipitates, possibly of globular or plate shape, but this is much less obvious compared to the observation elsewhere (Dargusch and Dunlop 1998) for an AZ91 alloy which has a higher Al concentration. Moreover, this decomposition and precipitation process is slow, and may take place over many hundreds of hours for the current testing condition (Dargusch and Dunlop 1998), but creep tests at higher stress levels in the current study generally lasted for only several tens of hours. Therefore, grain boundary sliding and migration aided by discontinuous precipitation of the γ -Mg₁₇Al₁₂ from supersaturated α -Mg matrix is a possible creep mechanism for the AM60 alloys in the current study, especially when the creep rate is relatively low, i.e., at lower temperature or stress levels. At higher temperature and stress levels, other competing creep mechanisms such as dislocation creep might have an increasing contribution to the creep behavior.



Figure 5.3. BSE SEM image of an as-molded AM60 sample which had been subjected to a temperature-jump creep test. The applied stress was 20 MPa, the temperature range was 100-200 $^{\circ}$ C, and the total time of creep exposure was over 830 hours.

5.1.5.3 Effect of Processing

As suggested by Figures 4.36, 4.37, and 4.38, at $150 \,\text{C}$, the as-molded AM60 alloy exhibited the lowest creep rates under all three applied stress levels (20 MPa, 50 MPa, and 75 MPa). The annealed material exhibited creep rates about an order of magnitude higher than those of the as-molded materials at all three applied stress levels. A possible reason for this might be the significant contribution of GBS to the creep rate, because the annealed material had a much smaller grain size compared to the as-molded material, and GBS can be enhanced with reduced grain size.

It is interesting to note that, compared to the as-molded and annealed materials, the creep rate of the TTMP AM60 alloy appeared to be more sensitive to applied stress and temperatures. As shown in Figures 4.38 and 4.39, at low temperature and low applied stress, the TTMP material exhibited creep rates similar to those of the as-molded material; at high temperature and high applied stress, the TTMP material exhibited creep rates similar to that of the annealed material. It is also interesting to note that, although the TTMP and annealed materials exhibited a moderate basal texture in which most of the grains were in a "hard orientation" for basal slip, they did not exhibit a higher creep resistance compared to the as-molded material, which exhibited a random texture. As discussed in Section 5.1.1, there appeared to be small recrystallized grains or nuclei in the TTMP material, therefore GBS might also have been enhanced. It appears that, under the testing conditions in the current study, the effect of texture on creep rate was overcome by the effect of grain size enhancing the creep rate for the AM60 alloys. Especially, as temperature increases, the CRSS of non-basal slip becomes closer to that of basal slip. If non-basal slip is enhanced, the effect of texture is expected to be reduced. Extensive cross-slip with increasing temperature was observed in a previous study on hightemperature creep of polycrystal Mg, and this was correlated with an increased activation energy larger than that of the lattice self-diffusion energy (Vagarali and Langdon 1981).

In summary, it appears that additional creep data at other temperatures and stress levels are needed to fully understand the creep mechanisms and the transition between the dominant creep mechanisms in the AM60 alloys. However, it is obvious that the thermomechanical processing and subsequent heat treatment, although significantly enhancing the tensile strength and fatigue strength, have reduced the creep resistance of the Thixomolded® AM60 alloy. This is a strong consideration for the implementation of TMP Thixomolded® Mg alloys in creep-driven applications, such as automotive engine applications, which are subjected to elevated temperatures.

5.2 AZ31 Alloy

In this section, the operative deformation mechanisms in the AZ31 sheet alloy in the temperature range 50-250 °C will be discussed based on the *in-situ* experiments and AFM analysis results. It is noted that the slip traces observed in the *in-situ* experiments were a result of dislocation motion. There are several reasons for no slip traces being observed for certain grains, such as the slip of dislocations being very homogeneous, the Burgers vectors being parallel to the sample surface, or the extent of slip being too small to develop slip traces. For the AZ31 samples investigated, more than half of the grains observed did not exhibit a slip trace. In most cases, only one set of slip traces was observed for one grain. In few cases, traces of two slip systems were identified in one grain. Although it is possible that multiple slip systems could have been activated in a single grain, traces for three or more slip systems were never identified in one grain.

5.2.1 Twinning and Dislocation Slip

The EBSD pole figures shown in Figures 4.46 and 4.50 are consistent with previous studies and reveal that when the AZ31 sheet alloy is deformed along an in-plane direction at 50 °C and 150 °C, a primary deformation mode is prismatic slip, which leads to the alignment of (1010) poles with the tensile axis. However, when deformation takes place at higher temperature (250 °C), the initial basal texture remains and the alignment of (1010) poles parallel to the tensile axis is not visible in the pole figures (see Figure 4.55). Thus, the dominant deformation modes at this temperature cannot be easily inferred from examination of the pole figures in this case.

The alignment of (1010) poles with the tensile axis, indicating the prevalence of prismatic slip, can be explained by Figure 5.4, which is similar to the analysis performed in a research elsewhere (Kelly and Hosford 1968). Figure 5.4 illustrates two representative grain orientations in the basal-textured AZ31 sheet, in which the c-axis is perpendicular to the tensile direction, and parallel to the ND of the sheet. The lattice rotation caused by prismatic slip tends to reduce λ , the angle between the tensile direction and the slip direction, and to increase ϕ , the angle between the tensile direction and the slip plane. In Figure 5.4(a), ϕ is initially 60° and λ is 30°. Slip on plane 1 tends to increase ϕ and harden this slip system. However, this will rotate plane 1' to an orientation favorable for slip, leading to a rotation in the opposite direction. Therefore, Figure 5.4(a) represents a stable orientation for tensile deformation.

In the case of Figure 5.4(b), ϕ is initially 30 ° and λ is 60 °. Slip on plane 2 tends to increase ϕ , making this slip system more favorable for slip. In addition, the lattice rotation will make plane 2' less favorable for slip. Therefore, slip on plane 2 will proceed, softening this slip system until ϕ reaches 45 °. Then slip will continue on plane 2, hardening this slip system until ϕ

reaches 60°, which is the condition in Figure 5.4(a). Similarly, other orientations with tensile direction perpendicular to the c-axis will also tend to rotate to the stable orientation in Figure 5.4(a). This leads to the texture in the AZ31 alloy after tensile deformation, which showed a six-fold symmetry on the (1010) pole figure, as shown in Figures 4.46 and 4.50.



Figure 5.4. Schematics illustrating $\{1010\} < 1210$ > prismatic slip in Mg under tensile loading perpendicular to the c-axis.

Both the EBSD measurements and the *in-situ* analysis revealed that extension twinning occurred during tensile deformation along RD at 50 $^{\circ}$ C in selected grains in which the basal plane was tilted away from the rolling plane. Extension twinning occurred in grains with low Schmid factors and it was observed at early stages of tensile deformation (~2-3% strain), indicating that this deformation mode is easily activated in Mg. Note that this deformation mode was not expected to occur in many grains as it can only be operative when the resultant stress on a specific grain causes an extension along the c-axis. However, extension twinning under tension along RD in AZ31 sheet has been observed occasionally in research elsewhere (Khan *et al.* 2011).

Recent literature has summarized the CRSS values in both single crystals and polycrystals of pure Mg and Mg alloys at RT (Raeisinia *et al.* 2011, Hutchinson and Barnett 2010), and a large variation can be found in these reported values. From the review by Hutchinson and Barnett (2010), one would expect very low CRSS values for basal slip, on the order of ~1MPa, for single crystal Mg (Bakarian and Mathewson 1943, Conrad and Robertson 1957). However, for the harder prismatic $\langle a \rangle$ slip in single crystal Mg, the values for CRSS ranged between 39-50 MPa (Reed-Hill and Robertson 1957, Ward Flynn *et al.* 1961). Due to the hardening process that takes place in polycrystals, the values expected for basal slip increase to 10-50 MPa, and the corresponding CRSS values for prism $\langle a \rangle$ slip increase to between 55-110 MPa (Agnew *et al.* 2003, 2006, Barnett *et al.* 2006, Muránsky *et al.* 2008). Thus, there is a significant discrepancy in the literature between the CRSS values between single crystals and polycrystals at RT. The hardening process in polycrystals could be treated as an additional term of similar magnitude to the CRSS values of the different slip systems, leading to the reduction of the ratio between the CRSS of non-basal and basal slip system in polycrystals compared to single

crystals. Raeisinia *et al.* (2011) suggest that by incorporating the effect of grain size and solute concentration on individual deformation modes into their polycrystal modeling, the difference between the CRSS values determined from single crystal experiments and poly crystal modeling can be reconciled.

The temperature dependence of the CRSS values of the various slip and twinning systems active in Mg alloys has been estimated by crystal plasticity models in polycrystals and also via experiments in single crystals. The results obtained from polycrystalline modeling are the following. Barnett (2003) used a full constraint Taylor model to estimate the evolution of the CRSS for prismatic $\langle a \rangle$ and second-order pyramidal $\langle c+a \rangle$ slip in an extruded AZ31 alloy at temperatures ranging from 150 $^{\circ}$ C to 450 $^{\circ}$ C. He assumed temperature independent values of the CRSS for basal slip and extension twinning and equal to 5 MPa and 32 MPa, respectively. Figure 5.5(a) illustrates the output of this model and predicts that the CRSS of prismatic slip is higher than that of pyramidal $\langle c+a \rangle$ slip within the temperature range 50-500 °C. The CRSS of prismatic $\langle a \rangle$ and pyramidal $\langle c+a \rangle$ slip are predicted to be smaller than that of twinning at temperatures higher than 233 °C and 200 °C, respectively. The CRSS of non-basal systems has been reported to be higher than that of basal slip until temperatures close 460 $^{\circ}$ (Barnett 2003). The results obtained from single crystal experiments are the following. Chapuis and Driver (2011) measured the CRSS of basal, prismatic, and second-order pyramidal $\langle c+a \rangle$ slip, as well as of various twinning systems, at temperatures ranging from 300-450°C, see Figure 5.5(b). In particular, they reported that the CRSS values of basal slip and extension twinning are basically temperature independent in the temperature range from 25-450°C, and their values are ~3 MPa and ~7-10 MPa, respectively. They also found that the CRSS of pyramidal $\langle c+a \rangle$ slip was higher than that of prismatic slip within the temperature range investigated and that neither of them became comparable to that of extension twinning until temperatures as high as 450°C. Thus, there is also a significant discrepancy in the literature between the CRSS values between single crystals and polycrystals at elevated temperature. Furthermore, the relative activity of the different deformation mechanisms in the temperatures ranges of interest is not well understood.



Figure 5.5. (a) Variation of the CRSS of different slip and twinning systems with temperature in an extruded AZ31 alloy according to the equations provided in reference (Barnett 2003). (b) Variation of the CRSS of different slip and twinning systems with temperature in pure Mg single crystals deformed in plane strain (Chapuis and Driver 2011).

The *in-situ* experiments in the current study showed that, the relative contributions of the different slip systems at 50 °C were 52% extension twin, 4% basal slip, 40% prismatic slip, and 4% pyramidal $\langle c+a \rangle$ slip. The relative contributions of the different slip systems at 150 °C were 41% basal slip, 44% prismatic slip, and 15% pyramidal $\langle c+a \rangle$ slip. The relative contributions of the different slip systems at 250 °C were 21% basal slip, 77% prismatic slip, and 2% pyramidal $\langle c+a \rangle$ slip.

Extension twinning was observed at 50 °C, and many of them exhibited low Schmid factors, indicating that this deformation mode is relatively easy to activate. However, at 150 °C, no extension twinning was observed. In addition, slightly more pyramidal $\langle c+a \rangle$ slip compared was observed compared to that at 50 °C, and they exhibited a relatively large range of Schmid factors. Because extension twinning and pyramidal $\langle c+a \rangle$ slip are the two competing mechanisms to provide deformation in the c-axis direction, it is expected that the CRSS of pyramidal $\langle c+a \rangle$ slip might have become similar to that of extension twinning at a temperature around 150 °C.

From 150 $\$ to 250 $\$, a transition in the dominant deformation mode occurs from basal + prismatic to mainly prismatic slip. Basal slip takes place at 150 $\$ even in grains in which the Schmid factor is very low (as low as 0.05) (see Figure 4.53). This is consistent with the fact that the CRSS for basal slip is very small. Conversely, 16 out of the 18 prismatic slip traces occurred in grains in which the corresponding Schmid factor was greater than 0.45. This suggests that the CRSS for prismatic slip might be still significantly higher than that of basal slip at 150 $\$. The increase in the contribution of prismatic slip with temperature, from 44% at 150 $\$ to 77% at 250 $\$, suggests that the CRSS for prismatic slip might be on the order of that of basal slip at 250 $\$. For all three testing temperatures, prismatic slip accounted for more than 40% of the

observed deformation activity. From Figures 4.49, 4.54, and 4.58, it can be seen that for the grains in which prismatic slip was activated, the tensile direction was near the arc of the unit triangle on the IPF. These are the preferred grain orientations in the AZ31 sheet, and exhibited relatively high Schmid factor for prismatic slip. Therefore, although prismatic slip has a CRSS value larger than that of basal slip, they are relatively easy to be activated due to the preferred grain orientation.

Regarding the temperature dependence of the relative CRSS values of different slip systems, the current observations seem to be in agreement with the transition temperature predicted by crystal plasticity models (Barnett 2003), see Figure 5.5(a). In fact, the CRSS values provided by single crystal experiments (Figure 5.5(b)) might not directly reflect the applied shear stress that is necessary to activate certain deformation systems in polycrystalline materials since most hardening contributions are not proportional to the CRSSs but are additive to them, making the relative CRSS values of different slip systems in polycrystals different from those in singles crystal (Hutchinson and Barnett 2010).

It is noteworthy that the activity of pyramidal $\langle c+a \rangle$ slip, which was 4% at 50 °C, 15% at 150 °C, and 2% at 250 °C, remained low in the temperature range analyzed in the current study.

5.2.2 GBS

The role of GBS during deformation of Mg alloys is controversial. Some studies have suggested that this mechanism contributes to deformation even at RT in fine grained Mg alloys (Koike *et al.* 2003), while others have reported to have found no evidence of GBS at low temperatures in Mg alloys with a wide range of grain sizes (Del Valle and Ruano 2009). An increasing contribution of GBS at high temperatures, even in materials with conventional grain

sizes, has been inferred by several authors from *ex-situ* experimental data (Del Valle *et al.* 2005, Del Valle and Ruano 2007, Hutchinson *et al.* 2009). However, some other studies (Stanford *et al.* 2011) have refuted this idea.

The current *in-situ* observations revealed a large amount of grain boundary cracking and jogging of fiducial lines, both potential signs of GBS. Moreover, by performing AFM analysis, the height of the steps formed at the grain boundaries due to GBS was directly measured. As suggested by Figures 4.63, 4.64, and 4.65, GBS occurred at all three temperatures at which the *in-situ* tensile tests were performed. As summarized in Table 4.10, for samples with similar tensile strain level ranging from 15% to 21%, an increase in the average grain boundary step height was observed with increasing temperature. Moreover, the percentage of grain boundaries that exhibited sliding also increased with temperature. These results suggested that there was an increased GBS activity with increasing temperature.

The GBS strain (the strain accommodated by GBS) was estimated using the equation:

$$\mathcal{E}_{\text{GBS}} = \frac{H}{d} \tag{5.6}$$

where *H* is the average grain boundary step height, and *d* is the average grain size, which is 13 μ m for the studied AZ31 alloy. This is the same method as the one used in a studies elsewhere (Barnett *et al.* 2009, Stanford *et al.* 2011). However, a significant increase in the strain accommodated by GBS was observed with increasing temperature, which is different from the study by Stanford *et al.* (2011).

It should be noted that, the measured average grain boundary step height, i.e., H in Equation (5.6), is the average value of the GBS resolved in the sample normal direction, and it is a rough assumption that the average value of the GBS resolved in the transverse and longitudinal directions of the samples is the same as this value H. A more precise measurement of the GBS

resolved in the transverse and longitudinal directions of the samples might be achieved by making grids on the sample surface using focused ion beam miller, and measuring the deformed grids after testing, such as in the study performed by Stanford *et al.* (2011).

5.2.3 Anisotropy

Figure 4.44 compares the *r*-values of rolled AZ31 as a function of temperature, including the results of the current study. It can be seen that the results in the current study are in agreement with previous data (Stanford et al. 2011, Agnew and Duygulu 2005, Barnett et al. 2009). There was a considerable drop in the r-value with temperature. This is additionally reflected in the more isotropic texture found after deformation at 250 $^{\circ}$ C. The decrease in anisotropy with increasing temperature has been attributed to an increase of the contribution of GBS (Hutchinson et al. 2009, Barnett et al. 2009) or to the enhanced operation of pyramidal $\langle c+a \rangle$ slip (Agnew and Duygulu 2005, Stanford *et al.* 2011) with increasing temperature. Agnew and Duygulu (2005) showed that increasing the relative activity of pyramidal $\langle c+a \rangle$ from ~0% to ~15%, with a commensurate decrease in prismatic slip activity, can lead to a reduction of the r-value by a factor of two. The observations made by Stanford et al. (2011) suggested that the reduction in the r-value was likely caused by an increase in the pyramidal $\langle c+a \rangle$ slip activity as they did not observe any significant contribution of GBS. However, increased GBS with increasing temperatures was observed in other studies (Hutchinson et al. 2009, Barnett *et al.* 2009), which was suggested to be the reason for the decreased *r*-value.

The current study showed that from 50 °C to 150 °C, the relative activity of pyramidal $\langle c+a \rangle$ slip increased from 4% to 15%. This is in agreement with the hypothesis that the decreased *r*-value was caused by increasing activity of pyramidal $\langle c+a \rangle$ slip. However, at

250 °C, the observed relative activity of pyramidal $\langle c+a \rangle$ slip decreased to 2%. This is contradictory to the further decrease in the *r*-value. On the one hand, there could be some variation for the amount of activity observed for this deformation mode. On the other hand, GBS could also contribute to the decreased *r*-value. Because the deformation accommodated by GBS is not dependent on crystallographic texture, pure GBS should lead to an *r*-value of ~1. As shown in Table 4.10, with increasing temperature, the strain accommodated by GBS increased. Especially, a significant amount of strain in the thickness direction was contributed by GBS. This is important for the decrease in *r*-value, because according to the definition of plastic strain

ratio, $r = \frac{\mathcal{E}_{W}}{\mathcal{E}_{t}}$, if the strain in the thickness direction is difficult to achieve, a large *r*-value is

expected. The increasing contribution of GBS to strain may also be revealed by comparing the Schmid factors for the active deformation activities at different temperatures, shown in Figures 4.48, 4.53, and 4.57. At 50 °C and 150 °C, a large number of twinning or slip activities with relatively low Schmid factors were observed, possibly due to the compatibility requirement for deformation. At 250 °C, the observed slip activities generally exhibited high Schmid factors. This could be rationalized if the increased GBS has accommodated for a large amount of deformation which is difficult to be accommodated for by slip.

In summary, increasing activity of pyramidal $\langle c+a \rangle$ slip might be a reason for the decreased *r*-value at higher temperatures. However, it was obvious in the current study that GBS activity was enhanced at higher temperatures. Moreover, the enhanced GBS activity might also have a significant contribution to the decreased plastic anisotropy.

5.2.4 *In-situ* Creep

As expected, the creep rate for the AZ31 alloy exhibited little difference between the transverse and longitudinal orientations. However, it is clear that rolled and annealed AZ31 is more creep resistant than the annealed AM60 sheet in the current study, see Figure 4.59. Similar to that for AM60, grain boundaries served as crack nucleation sites in creep, see Figure 4.60. Grain boundary cracking may have been accommodating GBS. Further evidence that GBS may be an active deformation mechanism during creep is the fact that significantly fewer slip traces were detected in comparison with the tensile tests performed at the same temperature. Low strain rates favor the activation of GBS and this mechanism seems to be dominant in the creep tests, unlike that observed for the higher strain rate tensile tests.

Extensive studies of GBS in the field of superplasticity have shown that GBS requires an accommodation mechanism (Gifkins 1982). Without additional accommodation, the microstructure would rapidly develop voids as the grains slide past one another, and this would lead to early failure. There are two well described accommodation mechanisms for GBS: diffusion (Ashby and Verrall 1973) and slip (Mukherje 1971, Ball 1997). GBS accommodated by diffusion is typically observed at high temperature, such as those encountered during creep. In this model, the cavity that would be created by the relative movement of one grain past another is filled by diffusing atoms. This mechanism allows for a shape change of the grains during the deformation. The other accommodation mechanism for GBS is dislocation slip. When two grains slide past each other, stresses accumulate at the grain boundary, which are released by the intragranular movement of dislocations. Of the two accommodation mechanisms, the former is more likely for the conditions evaluated in the *in-situ* creep experiment, as grain boundary cracking, rather than slip, was observed.

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5.3 MN11 Alloy

For the MN11 alloy in the current study, the relative contributions of the different deformation modes at 50 °C were 31% extension twin, 50% basal slip, 5% prismatic slip, and 14% pyramidal $\langle c+a \rangle$ slip. The relative contributions of the different deformation modes at 150 °C were 6% extension twin, 59% basal slip, 29% prismatic slip, and 6% pyramidal $\langle c+a \rangle$ slip. The relative contributions of the different slip systems at 250 °C were 87% basal slip, 2% prismatic slip, and 11% pyramidal $\langle c+a \rangle$ slip. Thus, it appeared that the activity of basal and non-basal slip is more balanced at lower temperatures compared with higher temperatures, at which basal slip was more dominant.

As shown in Figures 4.71, 4.74, and 4.77, the tensile direction of most of the grains in which basal slip or extension twinning was identified lay in the middle part of the IPF for all three different temperatures at which experiments were performed. Most of the grains in which prismatic slip was identified had the tensile direction distributed near the arc of the IPF, which is similar to observations made for the AZ31 alloy in the current study. There was a larger variation in the tensile direction for grains in which pyramidal $\langle c+a \rangle$ slip was found. Thus, the higher percentage of prismatic slip observed at 150 °C might be related to the fact that more grains in which prismatic slip was favorable were taken into account in the analysis. This is supported by Figure 5.6, which shows the discrete IPF and IPF of the tensile direction for regions in which the *in-situ* SEM images were taken for the deformation mode analysis. For the 150 °C tested sample, the tensile direction was distributed more uniformly on the IPF compared to samples tested at the other two temperatures. In other words, the region analyzed at 150 °C had a different local orientation distribution compared to those regions analyzed at the other two

temperatures. Thus a direct comparison of the percentage of the different modes operating at the different temperatures may not be completely fair.

Therefore, a smaller area was cropped from the original region within which the deformation mode analysis was performed. This smaller area showed a more similar local orientation distribution as the regions analyzed under the other two temperatures, see Figure 5.7(a). Figure 5.7(b) shows the IPF of the tensile direction for grains in which different deformations activities were observed at 150 $^{\circ}$ C, with only grains within this cropped area being considered. This results in the percentage of basal slip increasing from 50% to 75% to 88% with increasing temperature from 50 °C to 150 °C to 250 °C, respectively. Extension twinning decreases from 31% to 3% to 0% with increasing temperature from 50 % to 150 % to 250 %, respectively. The prismatic slip and pyramidal $\langle c+a \rangle$ slip system percentages exhibit a slight decrease with increasing temperature. Upon grouping the extension twinning and basal slip systems, the percentage of these two "soft" modes remains basically the same, with a slight increase with increasing temperature. Essentially, the strain accommodated by twins is replaced by basal slip at the higher temperatures. Taking this analysis into account, Figure 5.8 shows the percentages of each deformation mode with respect to temperature, and this plot illustrates that basal slip replaces extension twinning at elevated temperatures. Overall, the results suggest that the activation of basal slip compared to non-basal slip and twinning is easier at higher temperatures, and the relative activity of extension twinning is reduced significantly with increasing temperature.



Figure 5.6. Discrete IPFs and IPFs of the tensile direction for regions in which the *in-situ* SEM images were taken for deformation mode analysis. (a), (b) and (c) are for MN11 samples tested at 50 \degree , 150 \degree , and 250 \degree , respectively.



Figure 5.7. (a) Discrete IPF and IPF of the tensile direction for an area cropped from the regions in which the *in-situ* SEM images were taken for deformation mode analysis for the 150 $^{\circ}$ C tested MN11 sample. (b) IPF of the tensile direction (extrusion direction) for grains in which different deformation modes were observed, with only grains within the cropped area being considered.



Figure 5.8. Plot depicting the percentages of each deformation mode in MN11 samples with respect to temperature after taking into account the differences in the grain orientations in the regions analyzed for the three different deformation temperatures. That is, the data points in Figure 5.6(b) were used instead of data points in Figure 4.74.

At 50 $^{\circ}$ C, the CRSS of basal slip and extension twinning for MN11 seems to be quite close, since both are active to similar extents. Extension twinning was activated even with relatively low Schmid factors. However, at higher temperatures, deformation in MN11 takes place mainly by basal slip. Conversely, the relative activity of non-basal slip remained relative constant (i.e. between ~10-20% for all the temperatures analyzed when taking into account the grain orientation analysis mentioned in the above paragraph) and prismatic slip traces occurred in grains in which the corresponding Schmid factor was high. Although CRSS values for the deformation modes in the polycrystalline microstructure were not able to be examined, the present study reveals that in the MN11 alloy the CRSS of basal slip appears to decrease with temperature. This result is different from that obtained in conventional Mg alloys, such as AZ31, where the CRSS values are expected to remain constant. It is possible that as temperature increases, RE-atom diffusion takes place more readily and thus dislocation movement along basal planes becomes increasingly easier. If the temperature is high enough, it is likely that the CRSS of basal slip in the MN11 alloy might tend to that of pure Mg and more conventional Mg alloys not containing RE elements. Extension twinning was prevalent at 50 °C, but its intensity decreased at 150 $^{\circ}$ C and it was not observed at 250 $^{\circ}$ C. This suggests that the CRSS of both basal and non-basal slip become lower than that of extension twinning at a temperature around 150 $^{\circ}$ C. This transition temperature appears to be slightly higher than that observed in the AZ31 alloy in the current study, and slightly lower than that predicted by Figure 5.5(a).

At 50 °C, the YS in MN11 (YS~100 MPa) was lower than that for the rolled and annealed AZ31 sheet tensile tested along RD (YS~150 MPa), where prismatic slip dominates. This agrees with the expected higher CRSS for prismatic slip compared to basal slip at lower temperatures. It is noteworthy however, that at elevated temperatures the strength of the MN11 alloy is greater

than that for AZ31. For example, at $150 \,^{\circ}$, the YS and UTS in MN11 was approximately 80 MPa and 150 MPa, respectively, while the YS and UTS in AZ31 was approximately 90 MPa and 120 MPa, respectively. At 250 $^{\circ}$, the YS and UTS in MN11 was approximately 70 MPa and 125 MPa, respectively, while the YS and UTS in AZ31 was approximately 50 MPa and 60 MPa, respectively. Thus, the RE addition, which is expected to be responsible for this elevated-temperature strengthening through both solid-solution strengthening and precipitation strengthening (Rokhlin 2003), is more dominant than the texture effects at elevated temperatures.

CHAPTER 6

SUMMARY AND CONCLUSIONS

6.1 Summary

The work in this dissertation constituted a study of several of the aspects of the physical metallurgy of a few wrought Mg alloys. A systematic study on the processing-microstructure-property relationships was performed on an AM60 alloy produced using a novel process. A study of the deformation mechanisms was performed on a conventional wrought Mg alloy AZ31 and a newly-developed RE-containing wrought Mg alloy MN11. Overall, the work performed in this dissertation has provided new insight that was previously nonexistent into the processing-microstructure-property relationships and deformation mechanisms of these newly-developed wrought Mg alloys.

The effect of processing on the microstructure and mechanical properties of a Thixomolded® AM60 alloy was investigated. Microstructural analysis was performed using various characterization techniques including electron microscopy, optical microscopy, XRD, microprobe, and EBSD. Tensile experiments were performed both at RT and 150 °C. Fatigue experiments were performed at RT and 150 °C, at a stress ratio R = 0.1 and maximum applied stress levels of 50-175 MPa. Creep experiments were performed at temperature ranges of 100-200 °C and applied stress levels of 20-75 MPa. Fatigue crack growth experiments were performed at RT and stress ratios of R = 0.1, 0.3, and 0.5, at maximum applied stress levels of 60%-90% of the yield strength.

In-situ tensile experiments were performed in an SEM on an AZ31 and a MN11 Mg alloy at 50 $^{\circ}$ C, 150 $^{\circ}$ C, and 250 $^{\circ}$ C in order to analyze the local deformation mechanisms. EBSD was

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performed before and after the deformation. AFM was performed on deformed samples in order to evaluate the GBS activity. The relative activity of different slip and twinning systems as a function of temperature and grain orientation was determined.

6.2 Conclusions

6.2.1 Microstructure and Texture of AM60 Alloy

(1) The as-molded AM60 alloy exhibited an equiaxed microstructure with a bimodaldistributed α -Mg grain size. A fully divorced eutectic α -Mg + γ -Mg₁₇Al₁₂ was found at the grain boundary region. A high porosity level (~2% volume fraction) was found in the as-molded material. A random texture was identified for the as-molded material.

(2) The TMP treatment performed on the as-molded AM60 alloy resulted in severe deformation to the microstructure. Grains were severely deformed and elongated in the RD. Limited dynamic recrystallization was likely to have occurred in the TTMP material, whereas no significant grain growth was identified. The porosity level was significantly reduced compared to the as-molded material. The TTMP material exhibited a basal texture in which the basal plane was parallel with the plate face.

(3) The annealed AM60 alloy exhibited an equiaxed microstructure with a significantly reduced grain size. This was attributed to static recrystallization introduced by the annealing treatment. The basal texture in the TTMP material was retained after annealing treatment.

(4) During annealing treatment, the higher Al content in the supersaturated α -Mg solid solution near grain boundary regions was dissolved into the α -Mg matrix, leading to an apparent reduction of the grain boundary phase.

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6.6.2 Tensile Properties of AM60 Alloy

(1) Compared to the as-molded AM60 alloy, the TTMP material exhibited a significant increase in the RT tensile yield strength and the ultimate tensile strength without a debit of elongation-to-failure. The increase in the tensile strength was attributed to the basal texture and strain hardening effect.

(2) The annealed AM60 exhibited an intermediate RT tensile yield strength and ultimate tensile strength between the as-molded material and the TTMP material, but it exhibited the highest elongation-to-failure. The increased strength compared to the as-molded material was attributed to the basal texture and reduced grain size.

(3) At 150 °C, each material exhibited lower strength but higher elongation-to-failure compared to RT.

6.2.3 Fatigue Properties of AM60 Alloy

(1) The TTMP and annealed AM60 alloys exhibited greater fatigue strength both at RT and $150 \,^{\circ}$ compared with the as-molded material. For each of the as-molded, TTMP, and annealed AM60 alloys, no significant decrease in fatigue lives occurred due to the increase in temperature. The fatigue ratio was greater than 0.4 for the annealed material, and was ~0.3 for the as-molded and TTMP materials. This work has shown that through thermomechanical treatment of the Thixomolded® AM60, the fatigue strength can be almost doubled.

(2) Fatigue crack growth experiments indicated that, under the testing conditions used in the current study, the crack growth rate of the AM60 alloys could generally be characterized using LEFM parameters. This is partially attributed to the crack sizes being larger than the
critical size below which LEFM breaks down, in addition to the limited plastic deformation at the crack tip in the AM60 alloy, which makes the LEFM assumptions more accurate to apply.

(3) Changing the maximum stress did not lead to a discernible change in the crack growth rate at equivalent ΔK values. Increasing the stress ratio led to an increase in the crack growth rate at equivalent ΔK values, and a decrease in the crack growth rate at equivalent K_{max} values. The effect of stress ratio on the crack growth rate was modeled by a dislocation-based fracture mechanics relationship, indicating the general application of this relationship.

(4) The thermomechanical processing and subsequent heat treatment, which introduced different microstructures and thus strength values to the AM60 alloy, did not lead to a significant change in the fatigue crack growth rate at an equivalent ΔK value. This implies that crack nucleation controls the fatigue life of the material.

(5) For the as-molded material, fatigue cracks initiated preferentially at large pores near the sample surface. The TTMP and annealed materials mainly exhibited surface crack initiation. A difference in the defect density in the different processing conditions played an important role in determining the fatigue crack initiation and growth behavior.

(6) The fatigue crack propagation mechanism was a mixture of transgranular and intergranular failure at the crack tip. With increasing crack length, a gradual transition from the former mechanism to the latter mechanism was observed, and it was correlated with the increase in LEFM driving force and the scaled plastic zone size.

6.2.4 Creep Properties of AM60 Alloy

(1) At $150 \,^{\circ}$ under applied stress range of 20-75 MPa, the dominant creep mechanism for the AM60 alloys was dislocation creep. At applied stress of 20 MPa, GBS creep was the

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dominant creep mechanism at lower temperatures (100-150 $^{\circ}$ C), and dislocation creep was the dominant creep mechanism at higher temperatures (150-200 $^{\circ}$ C). Dislocation creep and GBS creep were two competing mechanisms operating in parallel, and this competition was influenced by temperatures as well as applied stresses.

(2) Among the AM60 alloys in three different processing conditions, the as-molded material exhibited the highest creep resistance, and the annealed material exhibited the lowest creep resistance. The creep experiments indicated cracking preferentially occurred at grain boundaries and GBS. Thus, grain size was expected to be an important microstructural parameter for the creep properties, and this partially explained the lowest creep resistance of the annealed material.

6.2.5 Deformation Mechanism of AZ31

(1) For the highly-textured AZ31 alloy tensile tested in the RD, extension twinning and prismatic slip were the dominant deformation modes at 50 °C. At 150 °C, extension twinning was not observed. The CRSS of second-order pyramidal $\langle c+a \rangle$ slip might have become similar to that of extension twinning at temperatures around 50 °C.

(2) From 150 °C to 250 °C, a transition in the dominant deformation mode occurred from basal slip + prismatic slip to mainly prismatic slip. The CRSS of prismatic slip might be on the order of that of basal slip at 250 °C.

(3) At all three temperatures, the relative activity of second-order pyramidal $\langle c+a \rangle$ slip remained lower than 15%.

(4) With increasing temperature, there was a significant increase in GBS activity.

(5) A significant drop in the plastic strain ratio was exhibited with increasing test temperature. This was partially attributed to the increase in second-order pyramidal $\langle c+a \rangle$ slip activity, and partially attributed to the significant increase in GBS activity.

(6) At elevated temperatures, the slip activity had more contribution to the tensile deformation compared to the lower stress and lower strain rate creep deformation, which was dominated more by GBS activity.

6.2.6 Deformation Mechanism of MN11

(1) For the weakly-textured MN11 alloy tensile tested in the extrusion direction, extension twinning was prevalent at 50 °C. Much less extension twinning was observed at 150 °C, and no extension twinning was observed at 250 °C. Thus, the CRSS of both basal and non-basal slip systems might have become lower than that of extension twinning at a temperature around 150 °C.

(2) Taking into account the different grain orientations involved in the analysis for the samples deformed at 150 °C, the prismatic and pyramidal $\langle c+a \rangle$ slip system percentages exhibited a slight decrease with increasing temperature. The percentages of the "soft" deformation modes (basal slip + extension twinning) remained basically the same, with a slight increase with increasing temperature.

(3) For the MN11 alloy, the CRSS of basal slip is believed to decrease with increasing temperature, which is different from conventional Mg alloys, such as AZ31. A possible explanation is the faster diffusion of rare-earth atoms with increasing temperatures.

(4) Compared to the AZ31 alloy, the MN11 alloy exhibited lower tensile strength at lower temperatures, but exhibited higher tensile strength at higher temperatures. It is believed

that the RE addition provides significant strengthening to the basal slip system. Although this strengthening effect decreases with increasing temperature, it is more effective compared to the strengthening effect of texture in the AZ31 at elevated temperatures.

6.3 Recommendations for Future Work

(1) By comparing the morphology of the samples in TEM analysis with that in the literature (Berman *et al.* 2011), it is suggested that dynamic recrystallization might have occurred in the TTMP AM60 alloy. This needs to be further confirmed by a detailed TEM analysis using selected area diffraction and EDS was well as EBSD.

(2) Fatigue crack growth experiments on the AM60 alloy for cracks smaller than those utilized in the current study need to be performed. Such experiments will assist in estimating the critical crack length above which LEFM can be applied to the AM60 alloy. The fatigue crack growth data will also provide more information for predicting the fatigue lives of the AM60 alloy in structural applications. For cracks smaller than the ones in the current study, i.e., in the range of 10-100 μ m, precise observations may be difficult to achieve through optical microscopy. An *in-situ* fatigue crack growth experiment can be performed, using an experimental setup similar to that of the *in-situ* tensile and creep experiments performed in the current study. SEM could provide precise measurement of the surface crack length. In addition, better observation for crack initiation behavior can be achieved. Moreover, EBSD analysis can be combined with the fatigue crack growth study, so that the effect of microstructure, such as crystallographic orientations and grain boundary characteristics, on the fatigue crack initiation and growth behavior can be studied.

(3) Creep experiments for the AM60 alloy need to be performed at more temperatures and applied stress levels in order to fully understand the temperature and stress dependence of the dominant creep mechanisms and the transition between these creep mechanisms.

(4) Similar to the experiments performed to the AZ31 and MN11 alloys, *in-situ* tensile experiments combined with EBSD analysis should be performed on the AM60 alloys. Because the as-molded AM60 alloy exhibited a random texture, the annealed AM60 alloy exhibited a basal texture, but the intensity was much weaker compared to the AZ31 alloy. It would be beneficial to compare the deformation mechanisms of the AM60 alloys with that of the AZ31 and MN11 alloys. Such experiments will help establish a more complete understanding of the effect of processing and alloying on the plastic deformation mechanism in Mg alloys.

(5) Modeling work, using the deformation behavior of the AZ31 and MN11 alloys analyzed in the *in-situ* experiments as an input, can be performed to estimate the CRSS values of the different deformation modes as a function of temperature in the polycrystalline microstructures.

APPENDICES

APPENDIX A

For the fatigue crack growth study, the stress intensity factor for the fatigue crack was calculated using the Newman-Raju equation (Newman and Raju 1981), which is an empirical solution of the stress intensity factor for a surface crack in a finite plate, as illustrated in Figure A.1. The length, width, and thickness of the finite plate was 2h, 2b, and t, respectively. The depth and length of the semi-elliptical surface crack was a and 2c, respectively. ϕ is the angle related to the location for which the stress intensity factor was to be calculated. The original equation applied for a plate under a combination of remote tension and bending load. However, in the current study, only a remote uniform tension stress of σ_t along the Y direction was applied. Under this loading condition, the solution for the mode I stress intensity factor, K_I, should be written as:

$$K_I = \sigma_t \sqrt{\pi \frac{a}{Q}} F(\frac{a}{t}, \frac{a}{c}, \frac{c}{b}, \phi)$$
(A.1)

The expression of Q is:

$$Q = 1 + 1.464 \left(\frac{a}{c}\right)^{1.65} \tag{A.2}$$

F is a function of *a*, *b*, *c*, *t*, and ϕ :

$$F = [M_1 + M_2(\frac{a}{t})^2 + M_3(\frac{a}{t})^4] f_{\phi} g f_{W}$$
(A.3)

And,

$$M_1 = 1.13 - 0.09(\frac{a}{c}) \tag{A.4}$$

$$M_2 = -0.54 + \frac{0.89}{0.2 + (a/c)} \tag{A.5}$$

$$M_3 = 0.5 - \frac{1.0}{0.65 + (a/c)} + 14(1.0 - \frac{a}{c})^{24}$$
(A.6)

$$f_{\phi} = \left[\left(\frac{a}{c}\right)^2 \cos^2 \phi + \sin^2 \phi \right]^{1/4}$$
(A.7)

$$g = 1 + [0.1 + 0.35(\frac{a}{t})^2](1 - \sin\phi)^2$$
(A.8)

$$f_{\rm W} = \left[\sec(\frac{\pi c}{2b}\sqrt{\frac{a}{t}})\right]^{1/2} \tag{A.9}$$

In the current study, the stress intensity factor at the interior front of the crack was used for analysis, and this corresponded to $\phi = 90^{\circ}$. It is also assumed that the crack is of a semicircular shape, i.e., a = c. Substituting the maximum applied stress σ_{max} for σ_{t} in Equation (A.1), the maximum stress intensity factor, K_{max}, can be calculated as:

$$K_{\max} = \sigma_{\max} \sqrt{\pi \frac{a}{2.464}} \left[1.04 + 0.2(\frac{a}{t})^2 - 0.1(\frac{a}{t})^4 \right] \left[\sec(\frac{\pi a}{2b} \sqrt{\frac{a}{t}}) \right]^{1/2}$$
(A.10)

Which is a function of the applied stress σ_{max} and geometric parameters including *a* (or equivalently, *c*), *t*, and *b*.

Substituting the applied stress range $\Delta \sigma$ for σ_{max} in Equation (A.10), the stress intensity factor range, ΔK , can be calculated.



Figure A.1. Schematic of a surface crack in a finite plate.

APPENDIX B

This section contains comparison of the fatigue crack growth curves for different AM60 specimens tested under various conditions. It is complementary to the comparisons made in Sections 4.1.4.1.1 and 4.1.4.1.3.



Figure B.1. Fatigue crack growth plots for the (a) as-molded AM60 materials tested at R = 0.1, and three different maximum stress levels (200 MPa, 265 MPa, and 300 MPa, which corresponded to 60%, 80%, and 90% of the σ_{ys}), (b) annealed AM60 materials tested at R = 0.1, and three different maximum stress levels (140 MPa, 180 MPa, and 200 MPa, which corresponded to 60%, 80%, and 90% of the σ_{ys}), an d(c) annealed AM60 materials tested at R = 0.5, and two different maximum stress levels (140 MPa and 180 MPa, which corresponded to 60% and 80% of the σ_{ys}).

Figure B.1 (cont'd)





Figure B.2. Fatigue crack growth plots for the TTMP AM60 materials tested at $\sigma_{\text{max}} = 265$ MPa, and three different stress ratios (R = 0.1, 0.3, and 0.5), showing the crack growth rate versus (a) ΔK , (b) K_{max} , and (c) $\phi_{\text{m}}\phi_{\text{c}}$.

Figure B.2 (cont'd)





Figure B.3. Fatigue crack growth plots for the annealed AM60 materials tested at $\sigma_{\text{max}} = 140$ MPa, and three different stress ratios (R = 0.1, 0.3, and 0.5), showing the crack growth rate versus (a) ΔK , (b) K_{max} , and (c) $\phi_{\text{m}}\phi_{\text{c}}$.

Figure B.3 (cont'd)



APPENDIX C

This section documented the MATLAB code used to determine the approximate creep rate at different creep strain levels. The purpose of this code is to get a quick and rough estimation of the relationship between the creep rate and creep strain which can be used to generate Figure 4.37. The minimum creep rate values used in other plots such as Figures 4.38 and 4.39 were not determined using this code.

The code is written as the following:

```
% Zhe Chen, 2012-2-22
% input format: four columns
% creep_time(hour) creep_strain(%) creep_time(s) time(s)
```

clear; clc;

```
a=importdata('09-40.xls'); % import data from excel file
```

```
Time=a.data.Sheet1(:,1); % copy creep time
d=a.data.Sheet1(:,2); % copy creep strain
Strain=d;
c=size(Time); % size of the matrix
N=c(1);
```

```
for i=11:N-10; % Strain is taken as an average of 21 left & right nearby values
   Strain(i)=0;
   for j=0:20;
      Strain(i)=Strain(i)+d(i-10+j);
   end
   Strain(i)=Strain(i)/21;
end
% Strain[]&Time[] valid index: 11 ~ N-10
```

```
for i=11:30:N-42; % creep rate = delta Strain/ delta Time
j=(i-11)/30+1;
creep_time(j)=Time(i);
creep_time_s(j)=creep_time(j)*3600;
```

```
creep_strain(j)=Strain(i);
  t1=Time(i);
  t2=Time(i+30); % every 30 data points are used, calculated is an average/approximate value
  d1=Strain(i);
  d2=Strain(i+30);
  creep_rate(j)=(d2-d1)/(t2-t1);
  if creep_rate(j)<0 % ensure positive creep_rate</pre>
     creep_rate(j)=creep_rate(j-1);
  end
  creep_rate_s(j)=creep_rate(j)/360000;
end
% plot(creep_time,creep_strain,'red') % this can plot creep time vs. strain
% hold;
plot(creep_time,creep_rate)
```

hold off;

result = [creep_time.', creep_strain.', creep_rate.', creep_time_s.', creep_rate_s.'];

% output to a txt file. Format is five columns:

% Time(hr), Strain(%), Rate(%/hr), Time(s), Rate(1/s)

save data out.txt result -ASCII

APPENDIX D

This section documented the MATLAB code used for trace analysis in order to determine the active slip or twinning system. The input parameter is the Euler angle (ϕ_1 , Φ , ϕ_2), which describes how the EBSD Lab coordinate system can be rotated into the crystal coordinate system. The x-axis of the EBSD Lab coordinate system is pointing downward, therefore an additional 90 ° rotation is needed to rotate this system into the more commonly used coordinate system with x-axis pointing toward the right direction. Moreover, when *in-situ* tensile test were performed, the sample might have been placed in a slightly different orientation compared to that when EBSD scans were performed, so an additional rotation might be needed between these two orientations. The Global coordinate system was chosen such that the x-axis was parallel with the horizontal direction in the *in-situ* SEM images. Therefore, the stress state can be assumed as a uniaxial tensile stress along the x direction. With these information, the slip plane trace on the sample surface (i.e., the x-y plane in the Global coordinate system), as well as the Schmid factor for a certain slip system can be calculated. The twinning plane trace and Schmid factor for a certain twinning system can also be calculated in similar manner.

The code for slip trace calculation is written as the following:

- % At MSU and IMDEA, EBSD Lab system is X pointing down.
- % All the rotations are summarized here:
- % 1) The Global system is the one in SEM images, x axis point to right.

[%] Code modified from and Hongmei's and Prof. Bieler's version % Zhe Chen 2011-05-06 % This code plots slip trace projected to the X-Y plane of Global Coordinate Systems. Euler angle is ZXZ convention.

[%] Note: Here I chose a Global Coordinate as the common one, i.e., X is right, Y is up,

[%] So I add more transformation, i.e., apart from the traditional definition, I further modify g.

[%] Samples may have been placed slight differently when taking *in-situ* SEM images and taking EBSD.

[%] So, rotate Global system until its x axis points to the right direction in the EBSD map.

% 2) Rotate EBSD map 90 degrees clockwise, then this is the EBSD Lab system.% 3) Rotate using three Euler angles, then this is the Crystal coordinate system.

clc;

clear;

phi_sys = -90; % rotate EBSD Lab system 90 degree, so its x-axis points right phi_error = -1; % angle error caused by placing the sample when doing experiment. i.e., rotate EBSD map into *In-situ* SEM image orientation.

euler=[155.352 56.129 211.21]; % Euler angle given by EBSD map. In MSU EB CamScan, EBSD Lab system X is down, Y is right. SIGMA=[1,0,0;0,0,0;0,0,0]; % stress state in Global System, be careful of its direction. Here tensile axis is horizontal. c_a=1.62; % c/a for Mg

% define slip systems, format: [slip PLANE, slip DIRECTION] % basal <*a*>-glide ssa(:,:,1) = [0 0 0 1; -2 1 1 0]; ssa(:,:,2) = [0 0 0 1; -1 2 -1 0]; ssa(:,:,3) = [0 0 0 1; -1 -1 2 0];

% prism <*a*>-glide ssa(:,:,4) = [0 1 -1 0; 2 -1 -1 0]; ssa(:,:,5) = [1 0 -1 0; 1 -2 1 0]; ssa(:,:,6) = [-1 1 0 0; 1 1 -2 0];

% 2nd order pyramidal $\langle c+a \rangle$ -glide

 $ssa(:,:,7) = [1 \ 1 \ -2 \ 2; \ -1 \ -1 \ 2 \ 3];$ $ssa(:,:,8) = [-1 \ 2 \ -1 \ 2; \ 1 \ -2 \ 1 \ 3];$ $ssa(:,:,9) = [-2 \ 1 \ 1 \ 2; \ 2 \ -1 \ -1 \ 3];$ $ssa(:,:,10) = [-1 \ -1 \ 2 \ 2; \ 1 \ 1 \ -2 \ 3];$ $ssa(:,:,11) = [1 \ -2 \ 1 \ 2; \ -1 \ 2 \ -1 \ 3];$ $ssa(:,:,12) = [2 \ -1 \ -1 \ 2; \ -2 \ 1 \ 1 \ 3];$

phi1=euler(1,1); PHI=euler(1,2); phi2=euler(1,3); g_sys = [cosd(phi_sys) cosd(phi_sys-90) 0; cosd(phi_sys+90) cosd(phi_sys) 0; 0 0 1]; g_error = [cosd(phi_error) cosd(phi_error-90) 0;

```
cosd(phi_error+90) cosd(phi_error) 0;
  001];
g phi1 = [
  cosd(phi1) cosd(phi1-90) 0;
  \cos(phi1+90)\cos(phi1)0;
  001];
g PHI = [
  100;
  0 cosd(PHI) cosd(PHI-90);
  0 cosd(PHI+90) cosd(PHI)];
g_phi2 = [
  cosd(phi2) cosd(phi2-90) 0;
  \cos(phi2+90)\cos(phi2)0;
  001];
g = g_{phi2*g_PHI*g_phi1*g_sys*g_error}; % g is the 'transformation matrix' defined in
continuum mechanics: x=gX, X is Global coordinate
```

for i = 1:1:12 % Change n & m to unit vector

 $\mathbf{n} = [ssa(1,1,i) (ssa(1,2,i)*2+ssa(1,1,i))/sqrt(3) ssa(1,4,i)/c_a]; \% Plane normal /c_a, into a Cartesian coordinate$

 $\mathbf{m} = [\operatorname{ssa}(2,1,i)*3/2 \ (\operatorname{ssa}(2,1,i)+\operatorname{ssa}(2,2,i)*2)*\operatorname{sqrt}(3)/2 \ \operatorname{ssa}(2,4,i)*c_a]; \% \text{ Slip direction } *c_a, \text{ into a Cartesian coordinate}$

ss(1,:,i) = n/norm(n); % slip plane, normalized
ss(2,:,i) = m/norm(m); % slip direction, normalized
end

```
ena
```

```
for j = 1:1:12
N = (g.'*ss(1,:,j).').'; % slip plane (i.e., plane normal) expressed in Global system
M = (g.'*ss(2,:,j).').'; % slip direction expressed in Global system
burgers_Z(j) = M*[0 0 1].'; % Z-component of burger's vector of slip system j (Percentage of
this vector in Z direction)
schmid_factor(j,1) = N*SIGMA*M.'; % Schimid factor for slip system j
abs_schmid_factor(j,1) = abs(schmid_factor(j,1)); % used to evaluate rank of SF
abs_schmid_factor(j,2) = j;
sliptrace(:,:,j) = cross(N, [0 0 1]);
sliptrace(:,:,j) = sliptrace(:,:,j)/norm(sliptrace(:,:,j));
trace_X(:,:,j) = [0, sliptrace(1,1,j)]; % (trace_X(1,2,;), trace_Y(1,2,:)) are coordinate of end of
lines starting from origin representing slip traces
trace_Y(:,:,j) = [0, sliptrace(1,2,j)];
```

```
end
```

Sorted_SF = sortrows (abs_schmid_factor, -1); % sort Schmid Factor

for k = 1:1:12 % Plot traces
 plot(trace_X(:,:,k), trace_Y(:,:,k));

axis([-1.25, 1.25, -1.25, 1.25]); axis square; text(trace_X(1,2,k)*(2+rem(k,7))/7, trace_Y(1,2,k)*(2+rem(k,7))/7, num2str(k)); % here 7 is just a randomly selected number with good effect to separate the labels hold on; end hold off; BIBLIOGRAPHY

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- Agnew, S. R., Brown, D. W. and Tome, C. N. (2006) 'Validating a Polycrystal Model for the Elastoplastic Response of Magnesium Alloy AZ31 Using in situ Neutron Diffraction', *Acta Materialia*, 54(18), 4841-4852.
- Agnew, S. R. and Duygulu, O. (2005) 'Plastic Anisotropy and the Role of Non-basal Slip in Magnesium Alloy AZ31B', *International Journal of Plasticity*, 21(6), 1161-1193.
- Agnew, S. R., Liu, K. C., Kenik, E. A. and Viswanathan, S. (2000) *Tensile and Compressive Creep Behavior of Die Cast Magnesium Alloy AM60B*, translated by Kaplan, H. I., Hryn, J. and Clow, B., TMS: Warrendale, PA, 285-290.
- Agnew, S. R., Tome, C. N., Brown, D. W., Holden, T. M. and Vogel, S. C. (2003) 'Study of Slip Mechanisms in a Magnesium Alloy by Neutron Diffraction and Modeling', *Scripta Materialia*, 48(8), 1003-1008.
- Agnew, S. R., Yoo, M. H. and Tome, C. N. (2001) 'Application of Texture Simulation to Understanding Mechanical Behavior of Mg and Solid Solution Alloys Containing Li or Y', Acta Materialia, 49(20), 4277-4289.
- Akhtar, A. and Teghtsoo, E. (1972) 'Substitutional Solution Hardening of Magnesium Single-Crystals', *Philosophical Magazine*, 25(4), 897-&.
- Al-Samman, T. and Gottstein, G. (2008) 'Room Temperature Formability of a Magnesium AZ31 alloy: Examining the Role of Texture on the Deformation Mechanisms', *Materials Science and Engineering: A*, 488(1-2), 406-414.
- Apps, P. J., Karimzadeh, H., King, J. F. and Lorimer, G. W. (2003) 'Precipitation Reactions in Magnesium-Rare Earth Alloys Containing Yttrium, Gadolinium or Dysprosium', 48(8), 1023-1028.
- Ashby, M. F. (1972) 'First Report on Deformation-Mechanism Maps', Acta Metallurgica, 20(7), 887-&.
- Ashby, M. F. and Verrall, R. A. (1973) 'Diffusion-Accommodated Flow and Superplasticity', *Acta Metallurgica*, 21(2), 149-163.
- Bakarian, P. W. and Mathewson, C. H. (1943) 'Slip and Twinning in Magnesium Single Crystals at Elevated Temperatures', *Transactions of the American Institute of Mining and Metallurgical Engineers*, 152, 226-253.
- Ball, A. (1997) 'Superplasticity in the Aluminium-Zinc Eutectoid an Early Model Revisited', *Materials Science and Engineering: A*, 234, 365-369.

- Ball, E. A. and Prangnell, P. B. (1994) 'Tensile-Compressive Yield Asymmetries in High-Strength Wrought Magnesium Alloys', *Scripta Metallurgica et Materialia*, 31(2), 111-116.
- Barnett, M. R. (2003) 'A Taylor Model Based Description of the Proof Stress of Magnesium AZ31 during Hot Working', *Metallurgical and Materials Transactions A*, 34A(9), 1799-1806.
- Barnett, M. R. (2007a) 'Twinning and the Ductility of Magnesium Alloys Part I: "Tension" Twins', *Materials Science and Engineering: A*, 464(1-2), 1-7.
- Barnett, M. R. (2007b) 'Twinning and the Ductility of Magnesium Alloys Part II. "Contraction" Twins', *Materials Science and Engineering: A*, 464(1-2), 8-16.
- Barnett, M. R., Ghaderi, A., Sabirov, I. and Hutchinson, B. (2009) 'Role of Grain Boundary Sliding in the Anisotropy of Magnesium Alloys', *Scripta Materialia*, 61(3), 277-280.
- Barnett, M. R., Keshavarz, Z., Beer, A. G. and Atwell, D. (2004) 'Influence of Grain Size on the Compressive Deformation of Wrought Mg-3Al-1Zn', *Acta Materialia*, 52(17), 5093-5103.
- Barnett, M. R., Keshavarz, Z. and Ma, X. (2006) 'A Semianalytical Sachs Model for the Flow Stress of a Magnesium Alloy', *Metallurgical and Materials Transactions A*, 37A(7), 2283-2293.
- Berman, T. D., Donlon, W., Decker, R., Huang, J., Pollock, T. M. and Jones, J. W. (2011) 'Microstructure Evolution in AZ61L During TTMP and Subsequent Annealing Treatments', in Sillekens, W. H., Agnew, S. R., Neelameggham, N. R. and Mathaudhu, S. N., eds., *Magnesium Technology 2011*, San Diego, Warrendale, PA: TMS, 599-603.
- Boehlert, C. J. (2007) 'The Tensile and Creep Behavior of Mg-Zn Alloys with and without Y and Zr as Ternary Elements', *Journal of Materials Science*, 42(10), 3675-3684.
- Bohlen, J., Nurnberg, M. R., Senn, J. W., Letzig, D. and Agnew, S. R. (2007) 'The Texture and Anisotropy of Magnesium-Zinc-Rare Earth Alloy Sheets', *Acta Materialia*, 55(6), 2101-2112.
- Bohlen, J., Yi, S. B., Letzig, D. and Kainer, K. U. (2010) 'Effect of Rare Earth Elements on the Microstructure and Texture Development in Magnesium-Manganese Alloys During Extrusion', *Materials Science and Engineering: A*, 527(26), 7092-7098.
- Braszczynska-Malik, K. N. (2011) 'Precipitates of γ-Mg17Al12 Phase in AZ91 Alloy' in Czerwinski, F., ed. *Magnesium Alloys Design, Processing and Properties*, Croatia: InTech, 95-112.
- Buch, A. (1999) *Pure Metals Properties: A Scientific-Technical Handbook*, Materials Park, Ohio: ASM International.

- Celotto, S. and Bastow, T. J. (2001) 'Study of precipitation in aged binary Mg-Al and ternary Mg-Al-Zn alloys using Al-27 NMR spectroscopy', *Acta Materialia*, 49(1), 41-51.
- Chadha, G., Allison, J. E. and Jones, J. W. (2007) 'The Role of Microstructure on Ductility of Die-Cast AM50 and AM60 Magnesium Alloys', *Metallurgical and Materials Transactions A*, 38A(2), 286-297.
- Chapuis, A. and Driver, J. H. (2011) 'Temperature Dependency of Slip and Twinning in Plane Strain Compressed Magnesium Single Crystals', *Acta Materialia*, 59(5), 1986-1994.
- Cheah, L. W. (2010) Cars on a Diet: The Material and Energy Impacts of Passenger Vehicle Weight Reduction in the U.S, unpublished thesis (0823342), Massachusetts Institute of Technology.
- Chen, W. and Boehlert, C. J. (2008) 'The Elevated-Temperature Fatigue Behavior of Boron-Modified Ti-6Al-4V(wt.%) Castings', *Materials Science and Engineering: A*, 494(1-2), 132-138.
- Christian, J. W. and Mahajan, S. (1995) 'Deformation Twinning', *Progress in Materials Science*, 39(1-2), 1-157.
- Coble, R. L. (1963) 'A Model for Boundary Diffusion Controlled Creep in Polycrystalline Materials', *Journal of Applied Physics*, 34(6), 1679-&.
- Conrad, H. and Robertson, W. D. (1957) 'Effect of Temperature on the Flow Stress and Strain-Hardening Coefficient of Magnesium Single Crystals', *Transactions of the American Institute of Mining and Metallurgical Engineers*, 209, 503-512.
- 'Corporate Average Fuel Economy', [online], available: http://en.wikipedia.org/w/index.php?title=Corporate_Average_Fuel_Economy&oldid=47 5430976 [accessed Feb 6, 2012].
- Couling, S. L., Pashak, J. F. and Sturkey, L. (1959) 'Unique Deformation and Aging Characteristics of Certain Magnesium-based Alloys', *Transactions of American Society for Metals*, 51, 94-105.
- Couling, S. L. and Roberts, C. S. (1956) 'New Twinning Systems in Magnesium', Acta Crystallographica, 9(11), 972-973.
- 'Crystal Lattice Structures: The aManganese (A12) Type', [online], available: http://cstwww.nrl.navy.mil/lattice/struk/a12.html [accessed Oct 21, 2004].
- 'Crystal Lattice Structures: The HCP (A3) Crystal Structure', [online], available: http://cstwww.nrl.navy.mil/lattice/struk/a3.html [accessed Feb 17, 2007].

Daniels, C. A. (2006) Metals: Structure and Properties, Washington D.C.: Abyss Books.

Dantzig, H. and Rappaz, M. (2009) Solidification, CRC Press.

- Dargusch, M. S. and Dunlop, G. L. (1998) 'Elevated Temperature Creep and Microstructure of Die Cast Mg-Al Alloys' in Mordike, B. L. and Kainer, K. U., eds., *Magnesium Alloys and Their Applications*, Frankfurt, Germany: Werkstoff-Informationsgesellschaft, 277-282.
- Dargusch, M. S., Pettersen, K., Nogita, K., Nave, M. D. and Dunlop, G. L. (2006) 'The Effect of Aluminium Content on the Mechanical Properties and Microstructure of Die Cast Binary Magnesium-Aluminium Alloys', *Materials Transactions*, 47(4), 977-982.
- Decker, R. F. and LeBeau, S. E. (2008) 'Thixomolding', Advanced Materials & Processes, 166(4), 28-29.
- Del Valle, J. A., Perez-Prado, M. T. and Ruano, O. A. (2005) 'Deformation Mechanisms Responsible for the High Ductility in a Mg AZ31 Alloy Analyzed by Electron Backscattered Diffraction', *Metallurgical and Materials Transactions A*, 36A(6), 1427-1438.
- Del Valle, J. A. and Ruano, O. A. (2007) 'Separate Contributions of Texture and Grain Size on the Creep Mechanisms in a Fine-Grained Magnesium Alloy', *Acta Materialia*, 55(2), 455-466.
- Del Valle, J. A. and Ruano, O. A. (2009) 'Effect of Annealing Treatments on the Anisotropy of a Magnesium Alloy Sheet Processed by Severe Rolling', *Materials Letters*, 63(17), 1551-1554.
- Delfino, S., Saccone, A. and Ferro, R. (1990) 'Phase-Relationships in the Neodymium-Magnesium Alloy System', *Metallurgical Transactions A-Physical Metallurgy and Materials Science*, 21(8), 2109-2114.
- Dieter, G. E. (1986) Mechanical Metallurgy, 3rd ed., New York: McGraw-Hill.
- Dillamore, I. L. and Roberts, W. T. (1965) 'Preferred Orientation in Wrought and Annealed Metals', *International Materials Reviews*, 10, 271-380.
- Dorn, J. E. (1954) 'Some Fundamental Experiments on High Temperature Creep', *Journal of the Mechanics and Physics of Solids*, 3(2), 85-116.
- Easton, M., Song, W. Q. and Abbott, T. (2006) 'A Comparison of the Deformation of Magnesium Alloys with Aluminium and Steel in Tension, Bending and Buckling', *Materials & Design*, 27(10), 935-946.

- Edgar, R. L. (2006) 'Global Overview on Demand and Applications for Magnesium Alloys' in Kainer, K. U., ed. *Magnesium Alloys and their Applications*, Wiley-VCH Verlag GmbH & Co. KGaA, 1-8.
- Edwards, L. and Zhang, Y. H. (1994a) 'Investigation of Small Fatigue Cracks .1. Plastic-Deformation Associated with Small Fatigue Cracks', *Acta Metallurgica et Materialia*, 42(4), 1413-1421.
- Edwards, L. and Zhang, Y. H. (1994b) 'Investigation of Small Fatigue Cracks .2. A Plasticity Based Model of Small Fatigue-Crack Growth', *Acta Metallurgica et Materialia*, 42(4), 1423-1431.
- Emley, E. F. (1966) Principles of magnesium technology, Oxford; New York: Pergamon Press.
- Friedrich, H. E. and Mordike, B. L. (2006) Magnesium Technology: Metallurgy, Design Data, Applications, Springer.
- Frost, H. J. and Ashby, M. F. (1982) Deformation-Mechanism Maps, Oxford: Pergamon Press.
- Fukuchi, M. and Watanabe, K. (1975) 'Temperature and Composition Dependence of Hardness, Resistivity and Thermoelectric Power of the γ-Phase in the Al-Mg System', *Journal of the Japan Institute of Metals*, 39(5), 493-498.
- Gall, K., Biallas, G., Maier, H. J., Gullett, P., Horstemeyer, M. F., McDowell, D. L. and Fan, J. H. (2004) 'In-situ Observations of High Cycle Fatigue Mechanisms in Cast AM60B Magnesium in Vacuum and Water Vapor Environments', *International Journal of Fatigue*, 26(1), 59-70.
- Gall, K., Biallas, G., Maier, H. J., Horstemeyer, M. E. and McDowell, D. L. (2005) 'Environmentally influenced microstructurally small fatigue crack growth in cast magnesium', *Materials Science and Engineering: A*, 396(1-2), 143-154.
- Gehrmann, R., Frommert, M. M. and Gottstein, G. (2005) 'Texture Effects on Plastic Deformation of Magnesium', *Materials Science and Engineering: A*, 395(1-2), 338-349.
- Gertsman, V. Y., Li, J., Xu, S., Thomson, J. P. and Sahoo, M. (2005) 'Microstructure and Second-Phase Particles in Low- and High-Pressure Die-Cast Magnesium Alloy AM50', *Metallurgical and Materials Transactions A*, 36A(8), 1989-1997.
- Gifkins, R. C. (1982) Paton, N. E. and Hamilton, C. H., eds., *Superplasticity in Crystalline Solids*, Littleton, CO: Metallurgical Society of AIME.
- Hall, E. O. (1951) 'The Deformation and Ageing of Mild Steel: III Discussion of Results', *Proceedings of the Physical Society*, 64B(9), 747-753.

- Harper, J. and Dorn, J. E. (1957) 'Viscous Creep of Aluminum near Its Melting Temperature', *Acta Metallurgica*, 5(11), 654-665.
- Hauser, F. E., Landon, P. R. and Dorn, J. E. (1956) 'Deformation and Fracture Mechanisms of Polycrystalline Magnesium at Low Temperatures', *Transactions of American Society for Metals*, 48, 986-1001.
- He, S. M., Zeng, X. Q., Peng, L. M., Gao, X., Nie, J. F. and Ding, W. J. (2006) 'Precipitation in a Mg-10Gd-3Y-0.4Zr (wt.%) Alloy During Isothermal Ageing at 250 Degrees C', *Journal* of Alloys and Compounds, 421(1-2), 309-313.
- Herring, C. (1950) 'Diffusional Viscosity of a Polycrystalline Solid', *Journal of Applied Physics*, 21(5), 437-445.
- Hertzberg, R. W. (1989) *Deformation and Fracture Mechanics of Engineering Materials*, 3rd ed., New York: John Wiley & Sons.
- Horstemeyer, M. F., Yang, N., Gall, K., McDowell, D., Fan, J. and Gullett, P. (2002) 'High cycle fatigue mechanics in a cast AM60B magnesium alloy', *Fatigue & Fracture of Engineering Materials & Structures*, 25(11), 1045-1056.
- Huang, J., Arbel, T., Ligeski, L., McCaffrey, J., Kulkarni, S., Jones, J., Pollock, T., Decker, R. and LeBeau, S. (2010) 'On Mechanical Properties and Microstructure of TTMP Wrought Mg Alloys', in S. Agnew, N. Neelameggham, E. Nyberg and Sillekens, W., eds., *Magnesium Technology 2010*, Seattle, TMS, 489-494.
- Hutchinson, B., Barnett, M. R., Ghaderi, A., Cizek, P. and Sabirov, I. (2009) 'Deformation Modes and Anisotropy in Magnesium Alloy AZ31', *International Journal of Materials Research*, 100(4), 556-563.
- Hutchinson, W. B. and Barnett, M. R. (2010) 'Effective Values of Critical Resolved Shear Stress for Slip in Polycrystalline Magnesium and Other HCP Metals', *Scripta Materialia*, 63(7), 737-740.
- Ion, S. E., Humphreys, F. J. and White, S. H. (1982) 'Dynamic Recrystallization and the Development of Microstructure during the High-Temperature Deformation of Magnesium', Acta Metallurgica, 30(10), 1909-1919.
- Kainer, K. U. and von Buch, F. (2004) 'The Current State of Technology and Potential for further Development of Magnesium Applications' in Kainer, K. U., ed. *Magnesium - Alloys and Technology*, Wiley-VCH Verlag GmbH & Co. KGaA, 1-22.
- Kassner, M. E. (2009a) 'Five-Power-Law Creep' in Kassner, M. E., ed. Fundamentals of Creep in Metals and Alloys, Elsevier, 9-93.

- Kassner, M. E. (2009b) 'Introductoin' in Kassner, M. E., ed. Fundamentals of Creep in Metals and Alloys, Elsevier, 1-8.
- Kelly, E. W. and Hosford, W. F. (1968) 'Deformation Characteristics of Textured Magnesium', *Transactions of the Metallurgical Society of Aime*, 242(4), 654-&.
- Keshavarz, Z. and Barnett, M. R. (2006) 'EBSD Analysis of Deformation Modes in Mg-3Al-1Zn', *Scripta Materialia*, 55(10), 915-918.
- Khan, A. S., Pandey, A., Gnaupel-Herold, T. and Mishra, R. K. (2011) 'Mechanical Response and Texture Evolution of AZ31 Alloy at Large Strains for Different Strain Rates and Temperatures', *International Journal of Plasticity*, 27(5), 688-706.
- Khan, S. A., Miyashita, Y., Mutoh, Y. and Bin Sajuri, Z. (2006) 'Influence of Mn Content on Mechanical Properties and Fatigue Behavior of Extruded Mg Alloys', *Materials Science and Engineering: A*, 420(1-2), 315-321.
- Khan, S. A., Miyashita, Y., Mutoh, Y. and Koike, T. (2008) 'Fatigue Behavior of Anodized AM60 Magnesium Alloy under Humid Environment', *Materials Science and Engineering: A*, 498(1-2), 377-383.
- Kitagawa, H. and Takahashi, S. (1976) *Applicability of Fracture Mechanics to Very Small Cracks or the Cracks in the Early Stage*, translated by ASM, Metals Park, Ohio, 627-631.
- Koike, J., Ohyama, R., Kobayashi, T., Suzuki, M. and Maruyama, K. (2003) 'Grain-Boundary Sliding in AZ31 Magnesium Alloys at Room Temperature to 523 K', *Materials Transactions*, 44(4), 445-451.
- Kuffova, M. (2011) 'Fatigeu Endurance of Magnesium Alloys' in Czerwinski, F., ed. *Magnesium Alloys - Design, Processing and Properties*, Croatia: InTech, 129-162.
- Kulyasova, O., Islamgaliev, R., Mingler, B. and Zehetbauer, M. (2009) 'Microstructure and Fatigue Properties of the Ultrafine-Grained AM60 Magnesium Alloy Processed by Equal-Channel Angular Pressing', *Materials Science and Engineering: A*, 503(1-2), 176-180.
- Kumar, S. R., Gudimetla, K., Venkatachalam, P., Ravisankar, B. and Jayasankar, K. (2012) 'Microstructural and Mechanical Properties of Al 7075 Alloy Processed by Equal Channel Angular Pressing', *Materials Science and Engineering: A*, 533, 50-54.
- Landkof, B. (2006) 'Magnesium Applications in Aerospace and Electronic Industries' in Kainer, K. U., ed. *Magnesium Alloys and their Applications*, Wiley-VCH Verlag GmbH & Co. KGaA, 168-172.
- Lankford, J. (1985) 'The Influence of Microstructure on the Growth of Small Fatigue Cracks', *Fatigue & Fracture of Engineering Materials & Structures*, 8(2), 161-175.

- Lee, W. S. and Su, T. T. (1999) 'Mechanical Properties and Microstructural Features of AISI 4340 High-Strength Alloy Steel under Quenched and Tempered Conditions', *Journal of Materials Processing Technology*, 87(1-3), 198-206.
- Lou, X. Y., Li, M., Boger, R. K., Agnew, S. R. and Wagoner, R. H. (2007) 'Hardening Evolution of AZ31B Mg Sheet', *International Journal of Plasticity*, 23(1), 44-86.
- Luo, A. A. (2004) 'Recent Magnesium Alloy Development for Elevated Temperature Applications', *International Materials Reviews*, 49(1), 13-30.
- Luo, A. A. (2011) *Magnesium Front End Research and Development (MFERD)*, 2011 DOE Merit Review Presentation.
- Luo, A. A., Balogh, M. P. and Powell, B. R. (2002) 'Creep and Microstructure of Magnesium-Aluminum-Calcium Based Alloys', *Metallurgical and Materials Transactions A*, 33(3), 567-574.
- Mabuchi, M., Kubota, K. and Higashi, K. (1996) 'Elevated Temperature Mechanical Properties of Magnesium Alloys Containing Mg2Si', *Materials Science and Technology*, 12(1), 35-39.
- Mackenzie, L. W. F., Davis, B., Humphreys, F. J. and Lorimer, G. W. (2007) 'The Deformation, Recrystallisation and Texture of Three Magnesium Alloy Extrusions', *Materials Science* and Technology, 23(10), 1173-1180.
- Mackenzie, L. W. F. and Pekguleryuz, M. O. (2008) 'The Recrystallization and Texture of Magnesium-Zinc-Cerium Alloys', *Scripta Materialia*, 59(6), 665-668.
- Maruyama, K., Suzuki, M. and Sato, H. (2002) 'Creep Strength of Magnesium-Based Alloys', *Metallurgical and Materials Transactions A*, 33(3), 875-882.
- Massalski, T. B., Murray, J. L., Bennett, L. H. and Baker, H. (1986) *Binary Alloy Phase Diagrams*, Metals Park, Ohio: ASM International.
- Mathew, M. D., Murty, K. L., Rao, K. B. S. and Mannan, S. L. (1999) 'Ball Indentation Studies on the Effect of Aging on Mechanical Behavior of Alloy 625', *Materials Science and Engineering: A*, 264(1-2), 159-166.
- Mayer, H., Papakyriacou, M., Zettl, B. and Stanzl-Tschegg, S. E. (2003) 'Influence of Porosity on the Fatigue Limit of Die Cast Magnesium and Aluminium Alloys', *International Journal of Fatigue*, 25(3), 245-256.
- McCarty, E. (2008) *Magnesium Front End Research and Development (AMD604)*, 2008 DOE Peer Review Presentation.

- Meza-Garcia, E., Dobron, P., Bohlen, J., Letzig, D., Chmelik, F., Lukac, P. and Kainer, K. U. (2007) 'Deformation Mechanisms in an AZ31 Cast Magnesium Alloy as Investigated by the Acoustic Emission Technique', *Materials Science and Engineering: A*, 462(1-2), 297-301.
- Mukherje, A. K. (1971) 'Rate Controlling Mechanism in Superplasticity', *Materials Science and Engineering*, 8(2), 83-89.
- Mukherje, A. K., Bird, J. E. and Dorn, J. E. (1969) 'Experimental Correlations for High-Temperature Creep', Asm Transactions Quarterly, 62(1), 155-&.
- Mur ánsky, O., Carr, D. G., Barnett, M. R., Oliver, E. C. and Šittner, P. (2008) 'Investigation of Deformation Mechanisms Involved in the Plasticity of AZ31 Mg Alloy: In situ Neutron Diffraction and EPSC Modelling', *Materials Science and Engineering: A*, 496(1–2), 14-24.
- Mwembela, A., Konopleva, E. B. and McQueen, H. J. (1997) 'Microstructural Development in Mg Alloy AZ31 during Hot Working', *Scripta Materialia*, 37(11), 1789-1795.
- Myshlyaev, M. M., McQueen, H. J., Mwembela, A. and Konopleva, E. (2002) 'Twinning, Dynamic Recovery and Recrystallization in Hot Worked Mg-Al-Zn Alloy', *Materials Science and Engineering: A*, 337(1-2), 121-133.
- Nabarro, F. R. N. (1948) Report of a Conference on the Strength of Solids, Physical Society, London.
- Newman, J. C. and Raju, I. S. (1981) 'An Empirical Stress-Intensity Factor Equation for the Surface Crack', *Engineering Fracture Mechanics*, 15(1-2), 185-192.
- Nie, J. F. and Muddle, B. C. (2000) 'Characterisation of Strengthening Precipitate Phases in a Mg-Y-Nd Alloy', 48(8), 1691-1703.
- Nyberg, E. A., Luo, A. A., Sadayappan, K. and Shi, W. F. (2008) 'Magnesium for Future Autos', *Advanced Materials & Processes*, 166(10), 35-37.
- Obara, T., Yoshinga, H. and Morozumi, S. (1973) '[11-22](-1-123) Slip System in Magnesium', *Acta Metallurgica*, 21(7), 845-853.
- Paris, P. and Erdogan, F. (1963) 'A Critical Analysis of Crack Propagation Laws', *Journal of Basic Engineering*, 85(4), 528-534.
- Payzant, E. A., Agnew, S. R., Han, Q. and Viswanathan, S. (2001) Mg17Al12 Phase Precipitation Kinetics in Die Casting Alloys AZ91D and AM60B, translated by Hryn, J., TMS: Warrendale, PA, 183-187.

- Pearson, S. (1975) 'Initiation of Fatigue Cracks in Commercial Aluminum Alloys and the Subsequent Propagation of Very Short Cracks', *Engineering Fracture Mechanics*, 7(2), 235-247.
- Perez-Prado, M. T., del Valle, J. A., Contreras, J. M. and Ruano, O. A. (2004) 'Microstructural Evolution during Large Strain Hot Rolling of an AM60 Mg Alloy', *Scripta Materialia*, 50(5), 661-665.
- Perez-Prado, M. T. and Kassner, M. E. (2009) 'Diffusional Creep' in Kassner, M. E., ed. *Fundamentals of Creep in Metals and Alloys*, Elsevier, 95-101.
- Petch, N. J. (1953) 'The Cleavage Strength of Polycrystals', *The Journal of the Iron and Steel Institute*, 174, 25-28.
- 'Plastic', [online], available: http://en.wikipedia.org/wiki/Plastic [accessed Feb 25, 2012].
- Powder Diffraction File # 35-0821, International Center for Diffraction Data, Swarthmore, PA
- Powell, B. R., Krajewski, P. E. and Luo, A. A. (2010) 'Magnesium Alloys for Lightweight Powertrains and Automotive Structures', *Materials, Design and Manufacturing for Lightweight Vehicles*, 114-173.
- Raeisinia, B., Agnew, S. and Akhtar, A. (2011) 'Incorporation of Solid Solution Alloying Effects into Polycrystal Modeling of Mg Alloys', *Metallurgical and Materials Transactions A*, 42(5), 1418-1430.
- Reed-Hill, R. E. and Robertson, W. D. (1957) 'Deformation of Magnesium Single Crystals by Nonbasal Slip', *Transactions of the American Institute of Mining and Metallurgical Engineers*, 209, 496-502.
- Regev, M., Aghion, E. and Rosen, A. (1997) 'Creep Studies of AZ91D Pressure Die Casting', *Materials Science and Engineering: A*, 234, 123-126.
- Ren, Y. P., Qin, G. W., Li, S., Guo, Y., Shu, X. L., Dong, L. B., Liu, H. H. and Zhang, B. (2012) 'Re-determination of g/(g+a-Mg) Phase Boundary and Experimental Evidence of R Intermetallic Compound Existing at Lower Temperatures in the Mg-Al Binary System', *Journal of Alloys and Compounds*, 540, 210-214.
- Rettberg, L. H., Jordon, J. B., Horstemeyer, M. F. and Jones, J. W. (2012) 'Low-Cycle Fatigue Behavior of Die-Cast Mg Alloys AZ91 and AM60', *Metallurgical and Materials Transactions A*, 43A(7), 2260-2274.
- Ritchie, R. O. (1999) 'Mechanisms of Fatigue-Crack Propagation in Ductile and Brittle Solids', *International Journal of Fracture*, 100(1), 55-83.

Roberts, C. S. (1960) Magnesium and Its Alloys, New York: Wiley.

- Rokhlin, L. L. (2003) Magnesium Alloys Containing Rare Earth Metals: Structure and Properties, Advances in Metallic Alloys, London: Taylor & Francis.
- Rollet, A. D. and Wright, S. I. (1998) 'Typical Textures in Metals' in Kocks, U. F., Tome, C. N. and Wenk, H. R., eds., *Texture and Anisotropy*, Cambridge: Cambridge University Press, 179-239.
- Ruano, O. A. and Sherby, O. D. (1988) 'On Constitutive-Equations for Various Diffusion-Controlled Creep Mechanisms', *Revue De Physique Appliquee*, 23(4), 625-637.
- Sanford, R. J. (2002) Principles of Fracture Mechanics, Prentice Hall.
- Schijve, J. (2009) Fatigue of Structures and Materials, New York: Springer.
- Schobinger-Papamantellos, P. and Fischer, P. (1970) 'Neutron Diffraction of Atom Distribution in Mg17al12', *Naturwissenschaften*, 57(3), 128-&.
- Sebastian, W., Droder, K. and Schumann, S. (2006) 'Properties and Processing of Magnesium Wrought Products for Automobile Applications' in Kainer, K. U., ed. *Magnesium Alloys* and their Applications, Wiley-VCH Verlag GmbH & Co. KGaA, 602-608.
- Shewmon, P. G. and Rhines, F. N. (1954) 'Rate of Self-Diffusion in Polycrystalline Magnesium', *Transactions of the American Institute of Mining and Metallurgical Engineers*, 200(9), 1021-1025.
- Shyam, A., Allison, J. E. and Jones, J. W. (2005) 'A Small Fatigue Crack Growth Relationship and Its Application to Cast Aluminum', *Acta Materialia*, 53(5), 1499-1509.
- Shyam, A., Allison, J. E., Szczepaiiski, C. J., Pollock, T. M. and Jones, J. W. (2007) 'Small Fatigue Crack Growth in Metallic Materials: A model and Its Application to Engineering Alloys', *Acta Materialia*, 55(19), 6606-6616.
- Shyam, A. and Lara-Curzio, E. (2010) 'A Model for the Formation of Fatigue Striations and Its Relationship with Small Fatigue Crack Growth in an Aluminum Alloy', *International Journal of Fatigue*, 32(11), 1843-1852.
- Shyam, A., Picard, Y. N., Jones, J. W., Allison, J. E. and Yalisove, S. M. (2004) 'Small Fatigue Crack Propagation from Micronotches in the Cast Aluminum Alloy W319', *Scripta Materialia*, 50(8), 1109-1114.
- Spencer, D. B. (1971) unpublished thesis Massachusetts Institute of Technology.
- 'Standard Practice for Codificatoin of Certain Nonferrous Metals and Alloys, Cast and Wrought', (2006)

'Standard Test Method for Measurement of Fatigue Crack Growth Rates', (2008)

'Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials', (2000)

- Stanford, N. (2010) 'Micro-Alloying Mg with Y, Ce, Gd and La for Texture Modification-A Comparative Study', *Materials Science and Engineering: A*, 527(10-11), 2669-2677.
- Stanford, N., Sotoudeh, K. and Bate, P. S. (2011) 'Deformation Mechanisms and Plastic Anisotropy in Magnesium Alloy AZ31', *Acta Materialia*, 59(12), 4866-4874.
- 'Stress Intensity Factor', [online], available: http://en.wikipedia.org/wiki/Stress_intensity_factor [accessed Mar 2, 2012].
- Suresh, S. and Ritchie, R. O. (1984) 'Propagation of Short Fatigue Cracks', *International Metals Reviews*, 29(6), 445-486.
- Tanaka, K., Akiniwa, Y., Nakai, Y. and Wei, R. P. (1986) 'Modeling of Small Fatigue Crack-Growth Interacting with Grain-Boundary', *Engineering Fracture Mechanics*, 24(6), 803-819.
- Taub, A. I., Krajewski, P. E., Luo, A. A. and Owens, J. N. (2007) 'The Evolution of Technology for Materials Processing Over the Last 50 Years: The automotive Example', *JOM Journal* of the Minerals, Metals and Materials Society, 59(2), 48-57.
- Taylor, D. and Knott, J. F. (1981) 'Fatigue Crack-Propagation Behavior of Short Cracks the Effect of Microstructure', *Fatigue of Engineering Materials and Structures*, 4(2), 147-155.
- Uchida, H. and Shinya, T. (1995) 'Estimation of Creep Deformation Behavior in Mg-Al Alloys by Using θ Projection Method ', *Journal of Japan Institute of Light Metals*, 45(10), 572.
- Uematsu, Y., Tokaji, K., Fujiwara, K., Tozaki, Y. and Shibata, H. (2009) 'Fatigue behaviour of cast magnesium alloy AZ91 microstructurally modified by friction stir processing', *Fatigue & Fracture of Engineering Materials & Structures*, 32(7), 541-551.
- Vagarali, S. S. and Langdon, T. G. (1981) 'Deformation Mechanisms in HCP Metals at Elevated-Temperatures .1. Creep-Behavior of Magnesium', *Acta Metallurgica*, 29(12), 1969-1982.
- Venkatesh, B. D., Chen, D. L. and Bhole, S. D. (2009) 'Effect of Heat Treatment on Mechanical Properties of Ti-6Al-4V ELI Alloy', *Materials Science and Engineering: A*, 506(1-2), 117-124.
- Walker, K. (1970) 'The Effect of Stress Ratio During Crack Propagation and Fatigue for 2024-T3 and 7075-T6 Aluminum' in *Effects of Environment and Complex Load History on Fatigue Life, ASTM STP 462*, American Society for Testing and Materials, 1-14.

- Walukas, D. M., Decker, R. F., Vining, R. E. and Carnahan, R. D. (1997) *Thixomolding of Magnesium*, Ann Arbor, MI, USA: Thixomat, Inc.
- Wang, R. M., Eliezer, A. and Gutman, E. M. (2003) 'An Investigation on the Microstructure of an AM50 Magnesium Alloy', *Materials Science and Engineering: A*, 355(1-2), 201-207.
- Ward Flynn, P., Mote, J. and Dorn, J. E. (1961) 'On the Thermally Activated Mechanism of Prismatic Slip in Magnesium Single Crystals', *Transactions of the Metallurgical Society* of Aime, 221(6), 1148-1154.
- Weertman, J. (1968) 'Dislocation Climb Theory of Steady-State Creep', Asm Transactions Quarterly, 61(4), 681-&.
- 'Wyckoff Positions of Group 217 (I-43m)', [online], available: http://www.cryst.ehu.es/cgibin/cryst/programs/nph-wp-list?gnum=217 [accessed Feb 25, 2012].
- Yoshinaga, H. and Horiuchi, R. (1963) 'Deformation Mechanisms in Magnesium Single Crystals Compressed in the Direction Parallel to Hexagonal Axis', *Transactions of the Japan Institute of Metals*, 4(1), 1-8.
- Zhang, M. X. and Kelly, M. X. (2003) 'Crystallographyof Mg17Al12 precipitates in AZ91D alloy', *Scripta Materialia*, 48(5), 647-652.