Micromechanisms of failures in ultralight magnesium alloys

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MICROMECHANISMS OF FAILURES IN ULTRALIGHT MAGNESIUM ALLOYS

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Abstract

Failure is always a potential problem to engineers whenever materials are used. Much effort has been directed towards the development of satisfactory fracture criteria to enable structural engineers to design confidently against such possible failures. However, so far understanding of failures in many materials is quite poor, although considerable knowledge has been obtained on failure mechanisms of steels. The project, therefore, aimed to gain better understanding of deformation and fracture processes in those engineering materials whose fracture mechanisms are poorly understood. Two ultralight magnesium alloys AZ91D and AM50A were chosen for the study due to their importance to engineering applications.

In this project, a technique was developed to combine the capabilities of optical microscope or SEM and mechanical testing device. A microtensile tester was installed under the optical microscope or inside an SEM to make it possible to carry out in-situ observation of deformation and fracture processes when the tensile test is going on. The testing results for notched specimens of AZ91D magnesium alloy with different notch angles show that the smaller the notch angle, the higher the fracture stress level. This is quite unexpected, but the phenomenon observed can be explained in terms of sampling process. Small notch angle leads to localized plastic deformation zone and therefore makes it less likely to “sample” large defects in the magnesium alloy.

The in-situ observation revealed clearly the deformation and fracture process in as-cast AZ91D, die-cast AZ91D and AM50A. First, plastic deformation occurred at the notch root, as can be seen from the slip lines. In the 2nd stage, the plastic deformation and the progressively higher loading led to cracking of the brittle intermetallic particles $Mg_{17}Al_{12}$ (especially those located at the grain boundaries), because they were strong barriers to dislocation movement and thus caused build-up of local stress. In the final stage, when both applied stress increased and the crack length increased, brittle fracture propagation suddenly occurred when the Griffith equation was satisfied.

The dynamic stress strain relation of magnesium alloy AM50A has been obtained using the Hopkinson bar apparatus. The strain rate ranges between 600 /s and 1300 /s. The magnesium alloy AM50A displays about 50% higher tensile stress at the strain rate of about 1300/s than at static loading rate. Fast shearing is the fracture mode at the highest strain rates.

In-situ observation of failures in magnesium alloy after oxidation in Thermalgravimetric Analysis (TGA) was carried out. Thermalgravimetric measurements revealed 3 different stages of reaction. Firstly the formation of an initial protective oxide, followed by an incubation period and finally degrading to a non protective oxide. In-situ observation was also conducted using a heating stage to study the change in surface morphology. Microtensile specimens were prepared and heated to different temperature range causing oxidization. The temperature 420 °C is found to be critical for AZ91D magnesium alloys. For specimens heated to 450 °C, there is a great reduction in mechanical strength (about 50 %). Even though minor oxidation take place on the surface of the alloy before 420 °C but it does not affect the mechanical strength much. The voids left behind by the melted intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) when heated above 450 °C provide an easy path for the fracture process thus lowering the mechanical strength. The preferential sites for oxide growth are the intermetallics and the intersection of grain boundaries.

Neodymium-doped Yttrium Aluminum Garnet (Nd:YAG) laser is used on the welding of AZ91D magnesium alloys. Butt welds of AZ91D magnesium alloy were produced using a Nd:YAG laser and with Argon as protective gas. The microhardness of the weld has significantly higher compared with the substrate. The main weld defect was porosity in the fusion zone. Hot cracking was also observed. Optimum tensile properties were obtained for welds made with a welding speed of 10 mm/s, power of 250K and a spot size of 1 mm. From the in-situ microtensile test, the fracture point is at the substrate instead of it breaking at the laser welded boundaries or weld. The results

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showed that the laser weld is able to withstand tensile testing more than the substrate. The fractography showed that brittle was the mode of fracture. Both SEM and optical microscope in-situ tensile testing demonstrated that the weld was stronger than the substrate.

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Chapter 1 Introduction

1.1 Background

Failure is always a potential problem to engineers whenever materials are used. Numerous failures have occurred, for example, in bridges, ships, boilers and offshore oil-drilling platforms. Much effort has been directed towards the development of satisfactory fracture criteria to enable structural engineers to design confidently against such possible failures. However, so far understanding of failures in many materials is still quite poor, although considerable knowledge has been obtained on failure mechanisms of steels (Knott, 1981). The project, therefore, aimed to gain better understanding of deformation and fracture processes in magnesium alloys, whose fracture mechanisms are poorly understood.

The driving need for weight reduction, particularly in portable microelectronics, telecommunication and automotive applications, has stimulated engineers to be more adventurous in their choice of materials. Magnesium, with one quarter the density of steel and only two thirds that of aluminium, and a strength to weight ratio that far exceeds either, fulfills the role admirably as an “ultralight” alloy, and possible applications of its composites can also be envisaged (Aghion and Arnon, 2007; Beals et al., 2007; Deng et al., 2005; El-Saeid Essa et al., 2003; Ganesh and Chawla, 2004; Serra et al., 1999; Sklenicka et al., 2000; Ye and Liu, 2004), *e.g.*, when wear resistance (Gao et al., 2007) is an important requirement. Figure 1.1 shows examples of new applications of magnesium alloys and other examples are shown later in Figure 2.3 (Albright and Ruden, 1994b). Recent applications of magnesium alloys also include biomedical usages such as biodegradable hard tissue implants (Xin et al, 2007).

In production, magnesium’s low heat content and low reactivity to steel provide fast cycle times with minimized die wear and a high tolerance capability due to low thermal distortions. This latter point often renders secondary machining unnecessary and thus

improves the precision processing economics. This coupled with the high fluidity of magnesium alloys allows complex and finely detailed component fabrication.

Figure 1.1 Examples of applications of magnesium alloys: top left - handphone chassis, top middle - thin wall chassis, top right - video camera case, bottom left - camera body, bottom right DVD chassis.

Due to the increasingly wider engineering applications of magnesium alloys, considerable effort has been made to understand various aspects of the materials, including study of their corrosion properties and corrosion prevention (Ambat et al., 2000a; Ambat et al., 2000b; Aung and Zhou, 2002; Cao et al., 2007; Chen et al., 2007; Cheng et al., 2007; Choi et al., 2007; Inoue et al., 2001; Lindstrom et al., 2004; Mathieu et al., 2002; Shipilov, 2002; Song et al., 2004a, Song et al., 2004b; Zhou et al., 2006), work on how to process the alloys to form products (Ca'ceres et al., 2001; Eliezer et al., 1998; Huang et al., 2000; Gariboldi and Conte, 2004; Kohzu et al., 2004; Matsumoto et al., 2007; Wei et al., 2003), experiments on their creep resistance (Kim and Kim, 2007; Regev et al., 1998; Regev et al., 2001; Yoo et al., 2003; Zhang, 2005), fatigue resistance (Bag et al., 2000; Cavaliere and De Marco, 2007; Gall et al., 2004; McDowell and Fan, 2004; Horstemeyer et al., 2002; Horstemeyer et al., 2004; Ishikawa et al., 1997; Potzies and Kainer, 2004; Sriwastan and Wei, 1997; Teng et al., 2002; Tokaji et al., 2004; Tsujikawa et al., 2004; Venkateswaran et al., 2004), tensile fracture behaviour (Bag and

Zhou, 2001; Barbagallo and Cerri, 2004; Biallas et al., 2004; Fan et al., 2006; Jager et al., 2004; Jiang et al., 2003; Lee, 2007; Li et al., 2002; Lu et al., 2000; Mabuchi et al., 2003; Park et al., 2003; Seshan et al., 2003; Wang et al., 2002; Yan et al., 2003), and impact properties (Vedani, 2000; Vedani and Mapelli, 2001). However, close examination of the literature shows that micromechanisms of fracture in magnesium alloys and their dynamic properties are still poorly understood.

In general, the mechanical properties of the alloys are well studied prior to the design and development of structural applications. However, deterioration of mechanical properties may occur during service as a result of long-term exposure to elevated temperatures. In automobiles, the operating temperatures of components are high and vary widely. For modern cars, the development of automotive technology has resulted in higher engine power outputs and consequently, components in the cars have to endure higher temperatures. For example, in the extremely hostile working conditions experienced by automobiles, the average working temperatures of brakes and clutches could go up to 500 °C (Luo, 2000). At these high service temperatures, the mechanical properties of magnesium alloys may deteriorate and cause premature failures; however limited studies were done on the effect of oxidation on the fracture mechanism of magnesium alloys.

Being high in specific strength and stiffness and light in density, magnesium alloys are potential candidates to replace steel and aluminum alloys in many structural and mechanical applications. Therefore, developing techniques for joining these magnesium alloys has become crucial. Although some researches on joining of magnesium alloys have been carried out prior to 1950 (Albright and Ruden, 1994b), the newly improved magnesium alloys and the special structural design entail further study to improve our knowledge on joining of magnesium. In the past few years, some researchers have sought to get useful information about welding of magnesium alloys using various welding methods, such as laser welding (Wang et al, 2007), TIG welding and electron beam welding. Although their experimental results add to our knowledge on magnesium

welding, in-depth investigation of the joining technology is still limited with magnesium joining technology is still in its infancy. The weldability of magnesium alloys is reported to be better with Nd:YAG laser due to its shorter wavelength, which in turn reduced the threshold irradiance required for keyhole mode welding and produced a more stable weld pool (Leong et al., 1998; Sanders et al., 1999; Watkins, 2003). Compared with the knowledge of welding steels, the knowledge on magnesium welding (especially laser welding) needs to be improved.

1.2 Research Methodology

The earliest theory on fracture was perhaps that proposed 2000 years ago by the ancient Chinese (Needham et al., 1962). It was recorded in an old book “Mohist”. The Mohists seemed to be maintaining that the reason why a material fractured under tension was that it was formed of elements unequally strong, or unequally cohesive. They had a good understanding of materials failures, considering they lived 2000 years ago. Unfortunately, the ancient Chinese did not have the right research tools to study fracture further.

When the optical microscope was invented at the end of the sixteenth century or at the beginning of the seventeenth century (Bradbury, 1968), fracture surfaces began to be examined. The invention of the optical microscope provided a most advanced tool at that time to study failures of materials. Thereafter, fracture began to be explained in terms of microstructures. In the past century, the invention of scanning electron microscope (SEM) and mechanical testing machines have provided much better tools for understanding the relationship between mechanical properties and microstructures of materials.

In this project, a technique was developed to combine the capabilities of optical microscope or SEM with a mechanical testing device. A microtensile tester was installed under an optical microscope or inside an SEM to make it possible to carry out

in-situ observation of deformation and fracture processes of ultra-light magnesium alloys when the tensile test is going on. Similar technique was also applied to study the fracture process of laser welded butt joints of magnesium alloys.

The in-situ experimental technique was later extended to the study of oxidation by using an optical microscope incorporated with a heating stage to study oxidation process of magnesium alloys at elevated temperatures. In addition to in-situ experimental techniques, other special tools such as instrumented impact tester and Hopkinson split bar were used to study dynamic properties of the magnesium alloys.

1.3 Objectives and Scope

The overall objective of this project was to understand micromechanisms of failures in ultralight magnesium alloys. The scope of research includes three parts:

- (1) Study of micromechanisms of fracture in magnesium alloy ingots and die castings;
- (2) Study of oxidation processes at high temperatures and effect of the oxidation on fracture properties; and
- (3) Study of laser welding and how the laser processing techniques affect the microstructures and properties of the magnesium alloys.

The method of approach to understanding fracture micromechanisms was to carry out in-situ observation of deformation and fracture processes during mechanical testing. To make in-situ observation possible, a microtensile tester was installed either under an optical microscope or inside an SEM to carry out “observation” and “mechanical testing” simultaneously. Two important ultralight magnesium alloys AZ91D and AM50A were selected for the study. One of the alloys, AM50A was also used for study of dynamic properties because this alloy is potential material for instrument panel of cars but its impact properties are not well understood.

In order to satisfy the needs of today, the corrosion resistance and high temperature properties of the widely used magnesium alloy such as AZ91D has to be promoted. Thermalgravimetric analyses (TGA) were carried out where as-cast magnesium alloy AZ91D was exposed to air in the temperature range from 400 °C to 500 °C for time intervals up to 1 hour. Results were also obtained for in-situ observation of failures in magnesium alloy after exposing to high temperature oxidation and tensile testings.

The usage of Neodymium-doped Yttrium Aluminum Garnet (Nd:YAG) laser has been selected in the welding of AZ91D magnesium alloys. Study of laser welding was to address engineering problems in the structural applications of magnesium alloys. As magnesium alloys are increasingly widely used, their applications are not limited to castings and welding techniques are needed to join different structural components.

1.4 Layout of Report

Chapter 2 reviews basic information on magnesium alloys and fundamentals of fracture. Chapter 3 describes materials used and the experimental procedure in detail. Chapter 4 summarizes the results of in-situ observation of fracture processes for AZ91D ingot and its die castings with a fracture model proposed. Chapter 5 examines the fracture model further using a different magnesium alloy, AM50A and presents additional results on dynamic properties of the alloy. In Chapter 6, the in-situ observation technique was extended to study oxidation of AZ91D and fracture of the materials after oxidation damage. Chapter 7 deals with the very important issues on engineering applications of magnesium alloys in welding. The main conclusions of this work are presented in Chapter 8, together with suggestions for further research.

Chapter 2 Literature Review

2.1 History of Magnesium

Magnesium is the sixth most abundant metal element on the crust of the earth at about 2.7% of its composition (Stanner, 1976). An almost limitless supply of magnesium is contained in sea water (1.27g/l Mg^{2+}), mineral rocks and natural brines with a high concentration of magnesium chloride and as by-products of the potash industry (Kaye and Street, 1982).

Magnesium has its name originated from the magnesia ancient city in Asia Minor. British chemist Humphry Davy is said to have produced an amalgam of magnesium in 1808 by electrolysing moist magnesium sulphate, using mercury as a cathode. The first metallic magnesium, however, was produced in 1828 by the French scientist A. A. B. Bussy. His work involved the reduction of molten magnesium chloride by metallic potassium. In 1833, the English scientist Michael Faraday was the first to produce magnesium by the electrolysis of molten magnesium chloride. His experiments were repeated by the German chemist Robert Bunsen.

In the early 1900s, magnesium was usually used for the purpose of photography as flares in photographic flashbulbs. This is due to the fact that the element burns in brilliantly bright flames. However, magnesium and its alloys are far more capable of being just a flare. Magnesium is easily alloyed and its alloys have remarkable properties. The magnesium industry grew rapidly during the period of the two world wars in response to the need for lightweight military aircraft engines, airframes, wheels and other parts (Mezoff, 1988).

It was during World War I that magnesium was first used as a structural material. Charles Ball who founded the British magnesium industry claimed that a German 77 mm shell was one of magnesium's first structural applications (Kaye and Street, 1982).

Before World War II, it is reported that Germany was a forerunner of the Mg-Al-Zn alloys. Development of appropriate metallurgy, processing and structural applications began first in Germany. Particularly during the 1930s, an impressive number of structural applications were present. For example, pressure die casting for cars included oil pump housings, engine cooling fans, blower impellers and gearboxes; for aircraft included tail wheels, brake shoes, brake levers and pistons (Mezoff, 1988).

During World War II, magnesium began to be used as a structural metal in a large scale. It was used for aircraft landing gears and engine components, wheels for artillery, gun mounts of many types and German jeep parts. World War II also introduced magnesium to US industry, primarily as airplane parts.

However when the war ended, the aircraft production halted. After World War II, the nonmilitary market began using significant amounts of magnesium alloys in power tools, luggage, lawn mower decks, sporting equipment, camera cases, conveyors and a variety of automotive components such as brackets, covers, housings and wheels (Mezoff, 1988).

When the Korean War began in 1950, US's attention was again diverted to military applications. Magnesium is used for many new types of aircraft and ordinance applications. After the Korean War, producers of competing structural materials, such as secondary aluminum from which aluminium die castings and commercial extrusion billets were made, being in a highly competitive situation, were forced to reduce profits in order to retain share. This resulted in that structural magnesium became uncompetitive and lost such hard-won applications as most of the automotive applications.

Figure 2.1 (a) shows the world consumption of six non-ferrous metals since World War II noting the increasing trend for magnesium development. Figure 2.1(b) shows total US magnesium consumption for structural and non-structural uses (Mezoff, 1988). It is seen that during World War II plenty of magnesium was used in structural applications, surpassing the consumption for aluminium. Structural volume fell back in the aftermath of the war, then grew during the Korean War period and kept around 20,000 ton per year level

ever since. Namely, the growth of the total consumption of magnesium was very fast since the end of World War II until the 1970s.

Figure 2.1 (a) World nonferrous metal consumption. (b) US primary magnesium consumption (Mezoff, 1988).

Today, structural magnesium industry is developing itself as both the scientific and engineering bases for growth of structural applications. Some examples are Dow, Amax, and Thixomat in the US and Institute of Magnesium Technology in Canada, Norsk Hydro in Norway, CRC for Alloy & Solidification Technology (CAST) in Australia and Magnesium Electron in the UK, etc. Currently several manufacturers in Taiwan and Japan are doing magnesium die casting. In Japan, there is already a target area of new research into very thin wall, very highly finished magnesium material but the progress will have to depend very much on the cost structure of the final products (CSIRO media release, 1997).

In China and Germany, magnesium die castings have been used in some automobile industries. In Israel, magnesium alloys are produced from the rich natural resources of the Dead Sea water, based on the electrolytic decomposition of carnallite ($\text{MgCl}_2 \cdot \text{KCl} \cdot 6\text{H}_2\text{O}$).

2.2 Magnesium Alloys

Magnesium is the lightest of all structural metals. As such, it forms the basis for commercial alloys that have found successful use in a wide variety of applications.

Owing to its strong reactivity, it does not occur in the native state, but rather it is found in a wide variety of compounds in seawater, brines and rocks. The most common ores are the carbonates, dolomite ($\text{MgCO}_3 \cdot \text{CaCO}_3$) and magnesite (MgCO_3). The halide mineral carnallite ($\text{MgCl}_2 \cdot \text{KCl} \cdot 6\text{H}_2\text{O}$) is found to form salt deposits in natural brines and evaporates such as in the Great Salt Lake in Utah. Lime is added to from the roasted seashells and treated chemically to precipitate magnesium hydroxide. The hydroxide is filtered and treated with hydrochloric acid, resulting in magnesium chloride, which is dried and placed in an electrolytic cell. Magnesium results are then formed into ingots. The major source of magnesium is extracted from sea water, which contains about 0.13% Mg in unlimited supply (Shigley, 1951).

2.2.1 Basic Properties of Magnesium Alloys

Magnesium's atomic number is 12, atomic weight is 24.312 and melting point is 615 °C. It has a HCP (Hexagonal Close-Packed) crystal structure with $a=0.321$ nm, $c=0.520$ nm, c/a 1.62 (Boyer and Gall, 1985).

Generally, the ductility of HCP metal is not as good as compared to other metals, because it has less slip systems than FCC and BCC metals, but it is still good compared to ceramics. At temperatures below 498 K, plastic deformation is limited to basal $\{0001\} \langle 1120 \rangle$ slip

system and to pyramidal $\{1012\} \langle 1011 \rangle$ twinning system. Thus, the magnesium crystal has only 3 geometrical and 2 independent slip systems (Pettersen and Fairchild).

For comparison, aluminum crystal has twelve $\{111\} \langle 110 \rangle$ geometrical and five dependent slip system. The specific c/a ratio of the magnesium is such that it can be mechanically twined only in compression. Thus, in tension test of a polycrystalline sample, twinning cannot be used to activate new slip system, and hence Mg-based alloys have relative low ductility in tension. At temperatures above about 498 K, slip also occurs on the $\{1011\}$ pyramidal and $\{1010\}$ prismatic planes in the $\langle 1120 \rangle$ directions and twinning becomes less important. The most common magnesium alloy, AZ91D, starts to creep at 100 °C and has a maximum service temperature of 125 °C (Pettersen and Fairchild).

Figure 2.2 In a hexagonal crystal, the axes labeled a form two sides of a regular hexagon in the horizontal plane. The height c is independent of a , the $c:a$ ratio which describes the atomic spacing of the crystal. The unlabelled arrows show the independent directions of the slip system. For clarity the layer of atoms at $c/2$ has been omitted. This layer is identical to the top and bottom layers that are shown.

Magnesium is a good structural material because it not only has low density, high strength to weight ratio, but also has good availability, recyclability and castability. The density of

magnesium alloy is only 1.738 g/cm^3 , which is one quarter that of steel and two-thirds that of aluminium, while its strength to weight ratio far exceeds either.

Table 2.1 shows the advantages of magnesium die casting alloy over aluminium alloy, mild steel, typical polymer and ceramic. Magnesium alloys have many advantages of the resources and recyclability aspects, which make them suitable for microelectronics and automotive components. The electromagnetic radiation (EMR) shielding capability of magnesium is a useful factor for such applications as mobile telecommunication products in place of polymer.

Table 2.1. Comparison of properties between AZ91D and other materials (Engineered Materials Handbook, v 4; Brandrup and Immergut, 1989; Murray et al., 1995)

Properties	Magnesium AZ91D	Aluminium A380	Mild Steel	Typical Polymer	Typical Ceramic
Mass Density (g/cm^3)	1.8	2.8	7.85	0.91-0.925	3.99
Young's Modulus (GPa)	45	71	210	0.055-0.17	393
0.2% Yield Stress (MPa)	160	200	440	N/A	N/A
Ultimate Tensile Strength (MPa)	225	275	540	15.2-78.6	206
Specific Young's Modulus (GPa/kg/dm^3)	25	26	27	0.059- 0.189	98
Specific Yield Stress (MPa/kg/dm^3)	88	73	57	N/A	N/A
Specific Ultimate Tensile Strength (MPa/kg/dm^3)	124	100	70	16.4-86.3	52

For the low-density properties of magnesium to be preserved, the alloying elements used must also be light. Aluminium is often present as it solid solution strengthens the magnesium, it also improves the castability of the melt and decreases the microporosity of the as-cast alloy.

The generic magnesium alloy contains aluminium and consists of two phases: α -Mg solid solution and β -Mg₁₇Al₁₂. The interdendritic β phase has a low melting point to be responsible for the low creep resistance of Mg-Al alloys. There can be as much as 10 vol% Mg₁₇Al₁₂ precipitated at the grain boundaries. The motion of dislocations is not restricted to the basal planes as in pure magnesium. The large quantity of the β phase and the extra slip systems in AZ91D both contribute to the low creep-resistance of the alloy.

Being resistant to both flexing and heat deformation and with minimal creep at higher temperatures (e.g. 150 °C), magnesium alloy housings eliminate leakage problems often encountered with metal-coated plastics. Magnesium alloy is able to maintain its strength at temperature in the range of -40 °C to 150 °C, which is suitable for many electronic and automotive components.

The high tolerance capability due to the low thermal distortion often renders secondary machining unnecessary and improves the precision (near net-shape) processing economics. The high fluidity when molten makes magnesium alloy ideal for die casting of thin wall or complex shape parts.

Magnesium has lower latent heat and reactivity for steel than aluminium has, which reduces die casting cycle time and tendency for die pick-up or erosion. Thus, the die used to cast a magnesium part lasts three to five times longer than that used for aluminium (Allsop and Kennedy, 1993).

Magnesium alloys also have excellent machinability with a tighter dimensional tolerance of ± 0.001 mm/mm, design freedom with a smaller draft angle than aluminium (0-1.5° vs. 2-

3°). In addition, magnesium can be cast into components whose wall thickness at 0.5-2.0 mm is typically 1/2 of aluminium (1-3.5 mm). The result is that magnesium components can be cast closer to near net-shape, which reduces the amount of material used and thus reduce the cost (Cole, 1998).

Despite the attractive properties of this ultra-light metal, there has been some reluctance to design and manufacture magnesium die-castings due to its high affinity with oxygen. However, magnesium alloys will only burn in contact with copious supply of air. With proper precautions and good housekeeping magnesium die-casting plant can be operated as safely as that for aluminium alloys. Another obstacle for magnesium die-casting is the relative high cost. Currently the cost ratio of Mg:Al is 2:1, however, with the increase of the numbers of new sources of magnesium supply, it is expected a 40% increase in magnesium production is very likely. The high content of porosity in magnesium die-casting is also a big problem, which affects the final properties of the component.

2.3 Magnesium Die Casting

Die-casting is a versatile process for producing engineered metal parts by forcing molten metal under high pressure into reusable steel molds. These molds, called dies, can be designed to produce complex shapes with a high degree of accuracy and repeatability. Parts can be sharply defined, with smooth or textured surfaces, and are suitable for a wide variety of attractive and serviceable finishes.

Die casting process is frequently the most practical and economic production method; it offers designers many advantages such as high rates of production, repeatability and lowest casting cost for producing metal casting. It also offers a high degree of dimensional accuracy from part to part, smooth or textured surfaces for minimal mechanical finishing and ability to produce parts with thin walls and great complexity.

Because of the hexagonal lattice structure, the ductility of magnesium is low, which makes

cold working difficult. Therefore, casting becomes the commonly used manufacturing process for magnesium alloys. Under the driving need of making net- shape magnesium component, the die casting process is selected because more complex shapes, thinner walls and greater dimensional accuracy can be produced by die casting than other metal forming process. At the same time, it can also achieve higher production rate and lower cost.

2.3.1 Common Magnesium Die Casting Alloys

Although there are a number of magnesium alloys which can readily be die cast, the standard magnesium-based die casting alloys used are AS, AZ, AM and AE series. Their nominal chemical composition is given in Table 2.2.

Table 2.2. Chemical composition of common magnesium alloys (wt%)
(Murray et al., 1995)

Material	Al	Mn	Zn	Si	Cu	Ni	Fe	RE	Other
AM60B	5.5-6.5	0.25	0.22	0.10	0.01	0.002	0.005	—	0.003
AZ91D	8.3-9.7	0.15	0.35-1.0	0.10	0.03	0.002	0.005	—	0.02
AS41A	3.5-5.0	0.2-0.5	0.12	0.5-1.5	0.06	0.03	—	—	0.3
AM50A	5.0	0.38	0.20	0.05	0.008	0.001	0.004	-	0.01

2.3.2 Designation System for Magnesium Alloys

The number of magnesium alloys that can be successfully cast by die casting is much more restricted than other casting method, for example, gravity-casting alloys. The current commercial magnesium die casting alloys contain aluminum as the main casting alloying element and other elements like zinc and manganese.

The standard magnesium-base die casting alloys used are AS, AZ, AM and AE series. In these series, the first two letters indicate the principal alloying elements. The greater alloying element presents will be the first letter, while the numbers, represent the nominal

compositions of the alloying elements in weight %. For example, AZ91 is a magnesium alloy containing approximately 9% aluminum and 1% zinc. The table below, Table 2.3, illustrates the designation system that is used by American Society for Testing and Materials for naming the magnesium alloys that can be cast.

AZ91 is the most commonly used alloy in the die casting industry. It has five variations, which are AZ91A, AZ91B, AZ91C, AZ91D and AZ91E. They have the same nominal composition except for iron, copper, and nickel contents, are die casting alloys used in the as-cast condition (F temper) and are selected for about 90% of all applications.

AZ91D is a high-purity alloy, which has much greater corrosion resistance than the others (Albright and Ruden, 1994a). It is the most commonly used magnesium die casting alloy. This Mg-Al-Zn alloy exhibits an excellent combination of strength, die-castability and corrosion resistance. It also has an acceptable short-term strength at elevated temperatures. However, its creep strength and bolt-load retention are poor (Luo and Pekguleryuz, 1994; King, 1998; Westengen).

AZ91A and AZ91B can be made from secondary metal, reducing the cost of the alloy. They must be used when maximum corrosion resistance is not required. AZ91E is a high-purity alloy with excellent corrosion resistance used in pressure-tight sand and permanent mold castings with high tensile strength and moderate yield strength. AZ91C is used in sand and permanent mold castings when maximum corrosion resistance is not required.

Table 2.3 Designation system for naming magnesium alloys

First part	Second part	Third part	Fourth part
Indicates the two principal alloying elements	Indicates the amounts of the two principal alloying elements	Distinguishes between different alloys with the same percentages of the two principal alloying elements	Indicates condition (temper)
Consists of two code letters representing the two main alloying elements arranged in order of decreasing percentage (or alphabetically if percentages are equal)	Consists of two numbers corresponding to rounded-off percentages of the two main alloying elements and arranged in same order as alloy designations in first part	Consists of a letter of the alphabet assigned in order as compositions become standard	Consists of a letter followed by a number (separated from the third part of the designation by a hyphen)
A - aluminum B - bismuth C - copper D - cadmium E - rare earth F - iron G - magnesium H - thorium K - zirconium L - lithium M - manganese N - nickel P - lead Q - silver R - chromium S - silicon T - tin W - yttrium Y - antimony Z - zinc	Whole numbers	Consists of a letter of the alphabet assigned in order as compositions become standard	F - as fabricated O - as annealed H10 and H11 - slightly strain hardened H23, H24 and H26 - strain hardened and partially annealed T4 - solution heat treated T5 - artificially aged only T6 - solution heat treated and artificially aged T8 - solution heat treated, cold worked and artificially aged

AZ91 alloys

AZ91 is the most commonly used magnesium alloy in die casting. It has three variations: AZ91A, AZ91B and AZ91D. They are selected for almost 90% of all applications. AZ91A and AZ91B are almost the same, except that there are wider limits for impurities for AZ91A. AZ91D is a high purity alloy that has much greater corrosion resistance than either A or B variation. AZ91 has excellent castability and strength. The mechanical properties are nearly the same for all the three variations. AZ91D is typically used for automotive and computer components, as well as for equipment, hand and power tools.

AM60 and AM50 alloys

Magnesium alloys can be divided into two groups: the *sand-casting* alloys that have a fine grain structure due to small additions of zirconium, and the *die casting* alloys, in which aluminium is the principal alloying elements. Aluminium improves the mechanical strength, corrosion properties and castability of magnesium castings. Ductility and fracture toughness are gradually reduced with increasing Al content. This has led to the introduction of a series of alloys with reduced Al contents. Due to high fracture toughness, impact resistance and good elongation combined with good strength and excellent corrosion resistance, AM60 and AM50 have found widespread application in parts like instrument panel supports, steering wheel armatures and brackets shown in Figure 2.3 (Albright and Ruden, 1994b).

Figure 2.3 Instrument panel support, steering column components and brackets
(Albright and Ruden, 1994b)

AS41 and AS21 alloys

By reducing the aluminium content to 4% and adding 1% silicon, considerable improvements of resistance to creep at elevated temperatures are obtained (Pettersen et al., 1996). Volkswagen introduced AS41 in the crankcase of the well-known Beetles in 1969. Further engine power enhancement of Volkswagen led to the introduction of AS 21 with only 1.9-2.5% aluminium, plus 0.7-1.2% silicon, which shows somewhat higher creep properties up to temperature around 150 °C. Because the die castability of AS41 and AS21 is inferior to that of AZ91, they are used to only a small extent.

AE41 alloys

By reducing the aluminium content to 4% and adding 1% rare earth elements (Ce, La, Nd and Pr), AE41 was made. Additions of RE elements strongly reduce the creep rate; the effect being more pronounced as the content of REs increases (Pettersen et al., 1996).

2.3.3 Die Casting Defects

Die casting is a casting process in which molten metal is forced under high pressure into

the cavity of a metal mold. With the upcoming of new alloys and more complex casting designs, the manufacturing technology is continuously being pushed to its limits.

As many alloys are particularly susceptible to porosity, hot tears and cracking during casting, the gating systems must be empirically developed through a trial-and-error process at high cost. Due to the fact that several combinations of gating, pouring parameters, mold configurations, mold temperatures are often necessary to make “good” castings, sometimes die casting defects such as dimensional deviations, surface defects and volume defects often surface (Rowley, 1993).

Dimensional Deviations

Dimensional defects usually refer to defects that affect external dimensions of casting. In such cases, there could be a change in overall size of the casting. Some of the likely causes that lead to the development of such defects include poor calculation of shrinkage allowance, varying cycling time or formation of flash effects.

- a. **Formation of flash** - Flash is a thin layer of metal that is created at zones that does not belong to the casting itself. The development of flashes occurs when liquid alloy penetrates into little gap along the parting lines. These parting lines are expanded during cavity fill. These types of defects could be attributed to the high die temperature, high melt temperature, bad mounting and alignment of die or die halves.
- b. **Incomplete filling** - Incomplete filling is sometimes known as misruns. These defects occur when the areas of the cavity are not totally filled. These are common sights in the edges of the cast or the thin zones of a casting. Some of the possible causes could be poor flow patterns, long cavity fill time, cold die or unsuitable alloy composition.

Surface Defects

Surfaces defects can be observed on the outer surfaces of the casting products.

- a. **Blisters** - Blisters or bubbles refer to the small bumps on the casting surfaces. These defects are generally smaller in size and are generated by the expansion of trapped gas. The low metal strength and the presence of the high pressure of entrapped gases induce the deformation of a thin metal layer, which forms blisters.
- b. **Solid bumps or protrusion** - These emerging bumps usually occur at the ending stage of the die casting process. Such defects could be due to insufficient surface preparation, presence of foreign material on the casting.
- c. **Flow lines** - Flow lines or heat waves are thin lines that mark the boundaries between the two flow patterns. The presence of these defects could be due to the long filling time of the cavity. Flow lines might develop into serious cold laps in a more severe circumstance.
- d. **Die crazing** - Die crazing is a fatigue destruction of the die surfaces due to thermal stress. It has a spider web like structure on the casting surfaces. Some of the possible reasons might be the usage of excessive spraying, improper heating of the die or high melting temperature.
- e. **Stained surface** - The presence of the lubricant that remains on the casting surfaces causes them to appear black or brown. The development of the stained surfaces could be due to the excessive use of die lubricant, low temperature and low die temperature.

Volume Defects

Volume defect refers to the presence of defects that affect the interior of the casting products.

- a. **Porosity** - Porosity is a common feature of metal casting, which may or may not be harmful depending on its location, size and connectivity. In addition to mechanical weakening of a component, porosity may cause leakage in parts intended for hydraulic applications and may cause unacceptable roughness in machined surfaces. For some

applications, small pores within the interior of a part may not interfere with the part's function, and there is then no need to eliminate them.

These defects occur when there are hidden gases or spherical voids inside the die casting. These gas holes can also be identified through x-ray, heat treatment and also naked eye. Some of the causes of such defects include short cavity filling time, low plunger temperature or unsuitable die lubricants.

2.4 Welding of Magnesium Alloys

The weldability of magnesium alloys is determined essentially by its physical, chemical, and metallurgical properties and the cast quality also determines the weldability of casting alloys. The physical and chemical properties should be taken into consideration when selecting a welding method. The effects of metallurgical properties, including the alloying elements should be considered in selecting filler metals.

2.4.1 Effect of Physical, Chemical and Mechanical Properties

Magnesium and its alloys have a hexagonal close-packed crystal structure. The amount of deformation that they can sustain at room temperature is limited compared with other metals such as steel and aluminum. However, it increases rapidly with the increase of temperature, and the metal can be severely worked between 200 °C and 315 °C (Kearns, 1992). However, its tensile strength decreases rapidly as temperature rises. Those forming operations, such as weld peening and anti-distortion straightening, are generally done at elevated temperatures.

Magnesium is an active element. When heated in air to its melting point of 651 °C (Carapella, 1945), magnesium will oxidize rapidly. This oxide (MgO) will inhibit wetting and flow during fusion welding, brazing and soldering (Lockwood, 1965). For this reason,

a protective shield of inert gas or flux must be used to prevent oxidization during exposure to elevated temperature. Unlike the oxidized layer of aluminum alloys, which is hard and non-porous and thus can prevent further oxidation, the oxide layer formed on magnesium surface will be recrystallized at high temperatures and become flaky (Kearns, 1992). The flaky magnesium oxide film tends to break up more readily during welding than does the aluminum oxide layer. Magnesium oxide is highly refractory and insoluble in both liquid and solid magnesium. The insoluble oxide layer will decrease the strength of the joint greatly.

Pure magnesium melts at 651 °C, about the same melting temperature as aluminum. However, magnesium boils at about 1090 °C (Carapella, 1945), which is low compared to other structural metals. Because magnesium has a high vapor pressure (1400 Pa at 727 °C) in liquid state, it tends to evaporate more easily than other elements during welding processing. Some researchers even believed that the evaporation of magnesium causes bubble in the weld and after cooling, porosity will appear in the weld (Wohlfahrt, 1998). Because of its comparatively low melting point, low latent heat of fusion and low specific heat, magnesium requires a relative small amount of heat to melt and the welding heat input is always lower than that used for aluminum, steel or titanium. The average coefficient of thermal expansion for magnesium alloys from 25 °C to 400 °C is about 16×10^{-6} /°C, about 1.2 times that of aluminum and twice that of the steel. That means the volumetric contraction from a liquid to a solid at its melting point is about 4.2% and from a liquid at its melting to a solid at room temperature about 9.1% (Jackson, 1972). During welding process, the uneven distribution of heat will cause high thermal stress. Thus, distortion is a potential problem for magnesium welding

2.4.2 Effect of Cast Quality

Wrought magnesium material does not present many quality-related problems for welding and it is reported that AZ31 alloy remains one of the most generally applicable and readily weld types (Lancaster, 1998). However, casting parts, particularly die-casting components,

demand a high level of manufacturing quality (Jutter, 1998). Due to the rapid filling processes, turbulence effects appear in the melt and gas bubbles under high pressure become entrapped in the matrix of the material. The rapid cooling of the molten metal also forces the gas to be in solution within the matrix. When the material is re-melted during welding, the gas is set free and pores will appear in the weld seam.

2.4.3 Microstructures of Magnesium Weldment

Microstructures of Mg-Al System Alloys

Mg-Al-Mn and Mg-Al-Zn base alloys are currently widely used engineering magnesium alloys. Because AZ91D and AM50A are the main materials in this research, information about these two alloys is useful for analysis of the metallurgy of weld. The phase diagram of Al-Mg system, which is a guide for us to understand the metallurgy of magnesium alloys, is shown in Figure 2.4.

Figure 2.4 Phase diagram of Al-Mg system. (Avedesian and Baker, 1999)

Under equilibrium conditions, AZ91 and AM50A alloys should solidify with the formation of only Mg-Al solid solution and secondary phase. However, the slow diffusion rate of aluminum under non-equilibrium solidification conditions leads to coring in hypoeutectic

Mg-Al solid solution and to the formation of eutectic phase, eutectic β phase and Al rich eutectic Mg-Al solid solution (Aghion and Bronfin, 1998). The morphology of eutectic phases formed under non-equilibrium cooling is distinguishable from typical eutectic morphology taking place under eutectic solidification. When the formation of eutectic is a secondary process, which is predominately primary solidification followed by the secondary solidification of a small amount of remaining liquid, typical lamellar symmetry is generated at a slow cooling rate. Under conditions of small amount of eutectic liquid and sufficiently rapid freezing rate, one of the eutectic phases- β phase will form as separate large particles with various shapes around the primary phase. The growth of one of the eutectic phases on to the existing crystals substitutes for independent nucleation is termed as divorced eutectic. For Mg-Al alloys, the tendency of forming divorced eutectic phase enhances with increasing cooling rate and decreasing aluminium content (Luo, 1997).

Due to the lower content of aluminium, the solidification temperature of AM50A is higher than that of AZ91D. The low aluminium content also results in a low content of eutectic precipitates in AM50A than AZ91D. Low content of brittle eutectic phases makes AM50A a higher ductility than AZ91D.

The microstructures of wrought magnesium alloys usually contain little precipitates (Closset, 2000). The strength of the alloys is influenced by the grain-extruded direction (Classet and Perey, 1998). Thus, for getting the best strength of the joint, the extruded direction of the base metal must be considered.

2.4.4 Welding Gas

In fusion welding, inert gases are used to form a protective shield for the fused material. The noble gases argon and helium or a mixture of the two is suitable for this purpose. Argon is the most widely used. Helium can also provide a satisfying protection. Because of the high prices per unit volume of helium and the requirement of two to three times more helium than argon for the same degree of protection, the use of pure helium has gradually decreased. Pure helium is also undesirable because it raises the current required for spray

arc transfer and increases weld spatter. Nitrogen and CO₂ are not suitable because they combine with magnesium to form nitrides or carbides, which can impair the mechanical properties of the weld point (Wohlfahrt, 1998).

2.4.5 Beam Welding Technology

Laser beam welding and electron beam welding are two welding methods that provide fast welding speed and allow high productivity. Compared with arc welding methods, beam welding has the advantages of decreasing the distortion of magnesium structure, narrowing the HAZ and reducing the probability of cracking formation in the weld.

Laser Beam Welding

The technology of using laser to join materials started in 1987 in Japan (Duley, 1999). Laser beam welding in the material processing was laid by the development of the CO₂ laser and Nd: YAG laser in the middle of the sixties. The main advantage of laser welding is the good focusing ability and the flexible beam handling.

Welding used laser was divided into gas laser and solid-state laser that differ from each other regarding beam generation and the kind of beam source. The beam source of gas laser is a gas mixture in cavity. In contrast, the laser medium of a solid-state laser is a crystalline stick made of Neodymium yttrium-aluminum garnet. The essential differences of both system groups can be found in the different wavelength λ of the emitted light. The wavelength of solid state laser light run up to $\lambda = 1.06 \mu\text{m}$, well the wavelength of a gas-laser beam comes to $\lambda = 10.6 \mu\text{m}$. This difference can effect the processing of different materials with variable absorption behavior.

The CO₂-laser welding qualified by more elevated initial performances up to more than 30kW higher efficiency, while the solid state laser welding demonstrate an initial performance of up to 4 kW. The advantage of solid state laser is due to the possibility of application of glass fiber optics for beam directing that allow a flexible application of laser systems. The advantage of using laser-welding methods is resulted from the good

absorption behavior of magnesium alloys. Much research work using solid state laser and CO₂ gas laser to weld magnesium alloys has been done in recent years and good welding results have been obtained with optimum welding parameters (Weisheit and Galun, 1998). Compared with arc welding, laser welding decreases the heat-input, narrows the weld size and heat-affected zone size and reduces the thermal stress in the welded plates. As a result, no hot cracking was found in the weld after welding. However, porosity problem still affects the quality of the weld.

Electron Beam Welding

Electron beam welding, like laser beam welding, is a fast welding method. The use of electron beam as a tool for material treatment in the process of melting, drilling and welding has been well known since the 50's. A conventional electron beam-welding machine consists of two main parts: the electron beam gun and the working chamber. A vacuum heated tungsten cathode emits electrons by thermic emission. The electrons are accelerated by an electrical potential, which is located between cathode and the drilled anode. After passing the hole of the anode, the electron beam diverges. Using a focusing system, a sufficient power density for material treatment is produced. Several electric-magnetic lenses focus the beam onto the workpiece in the vacuum-working chamber. In the case of non-vacuum electron beam welding machine, a vacuum chamber is not necessary because the beam is lead out to the atmosphere through an orifice system with increasing pressure. Because of the difference of the beam, the welding configuration is also different. For vacuum electron beam welded magnesium, the weld is narrow; while in welding with a non-vacuum electron beam, a wide weld is formed (Draugelates et al., 1998). Although electron beam welding method can be applied to join magnesium alloys, this method is rarely used in industry.

2.4.6 Magnesium Welding Defects

Defects that occurred during welding of magnesium alloys are reported as porosity, hot-cracking, grain growth in the HAZ and undercut formed in the weld.

Many researchers discovered the porosity problem in their research work using beam welding and arc welding methods (Weisheit et al., 1998). Some researchers thought that this problem was caused by the base metal containing some amount of shrinkage porosity. Wohlfahrt (Wohlfahrt, 1998) in his investigations also reported the formation of porosity in the magnesium weld produced. He thought that the formation of pores was due to the evaporation of magnesium during welding.

Cracking is a persistent problem that relates with the alloying elements the alloy system used. Wohlfahrt, (Wohlfahrt, 1998) reported the formation of hot cracking in welded magnesium joint. He thought that the hot cracking resulted from the alloy elements system used and he recommended an element range in Table 2.4 could be used as a direction for fusion welding of magnesium alloys.

Table 2.4 Weldable alloy groups and their maximum contents of alloying elements.

Alloy Group	Max. Proportion of alloying element
Mg-Al (Zn)	Al<10%, Zn<2%
MgZn	Zn<4%

Coarsening of grains may be a concern when wrought magnesium alloys such as AZ31 are welded. The coarse grains in the HAZ will greatly lower the strength of the joint. Undercut will occur in autogenously welded magnesium joint. To reduce the formation of the undercut during welding, filler rods and electrodes are always used.

2.5 Oxidation in Magnesium

Magnesium alloys, during typical applications, are subjected to environmental conditions ranging from indoor atmospheres, for household or electronic appliances, to intermittent salt splashes for some automotive components. As a result, a majority of studies devoted to

oxide growth on magnesium alloys describes room temperature phenomena and liquid environments (Avedesian and Baker, 1999). It has been agreed that the initial reaction of pure magnesium with oxygen proceeds in three stages: oxygen chemical absorption below the topmost magnesium layer, formation and coalescence of oxide islands and oxide thickening (F. Czerwinski, 2002). Contact with water vapour reaction is much slower and the oxide layer contains relatively large amounts of hydroxyl or hydroxide species (Fuggle et al., 1975). There are, however, manufacturing stages when magnesium alloys are exposed to high temperatures and detrimental contact with an oxidizing medium. This refers to heat treatment, welding and casting. An extremely high affinity of magnesium to oxygen made it prone to oxidation during manufacturing process. A description of the alloy oxidation behaviour is therefore of key importance for designing of material feeding and inert gas flow systems. It is also important for feedstock manufacturing in terms of morphology and its chemical modification against oxidation.

Chapter 3 Materials and Experimental Procedures

3.1 Materials

The materials used in the project were two ultralight magnesium alloys AZ91D and AM50A. Their chemical composition is shown in Table 3.1. AM50A was studied in ingot casting condition only, but AZ91D was studied both ingot casting condition and die casting conditions.

Table 3.1 Chemical composition of AZ91D and AM50A alloys (in wt.%)

Alloy	Al	Mn	Zn	Si	Cu	Ni	Fe	RE	Others
AZ91D	9.1	0.17	0.64	<0.01	0.001	0.001	<0.001	-	<0.01
AM50A	5.0	0.38	0.20	0.05	0.008	0.001	0.004	-	0.01

Figure 3.1 Geometry of the AZ91D and AM50A ingots used ($W_1 = 150\text{ mm}$, $W_2 = 100\text{ mm}$, $T = 60\text{ mm}$ and $L = 600\text{ mm}$).

3.2 Die Casting of Magnesium Alloy

Die casting of magnesium alloy AZ91D was carried out using a die casting machine from TOYO. The machine was operated in either vacuum condition or in air to study effect of vacuum on quality of the castings. The die-cast specimens were produced and

supplied by Professor Xiong Shou Mei’s research group in Tsing Hua University, Beijing. Die casting parameters are the same for both vacuum and air die casting samples, as shown in Table 3.2.

Figure 3.2 (a) and (b) Optical photographs of die-cast specimen of AZ91D.

Table 3.2 Die casting parameters

Casting pressure	66.7 MPa
Melt Temperature	680 °C
Die Temperature	150 °C
Slow Phase Speed	0.1 m/sec
Fast Phase Speed	1 m/sec

3.3 Microtensile Testing

3.3.1 Microtensile Tester

The microtensile tester used was MICROTTEST made by Deben UK Limited. It is equipped with a 300 N load cell. Deben MICROTTEST is capable of three-point bending and tensile / compression testing, but the project focused on microtensile testing. A photograph of the testing machine is shown in Figure 3.3(a) and the dimensions are shown in Figure 3.3(b). Some of the basic specifications are listed below:

- Tensile and compression testing
- Precision slides with symmetrical stainless steel leadscrew
- Vacuum feedthrough for connections
- Integrated 300 N load cell with accuracy of 1% of full scale range
- Standard speed range 0.1 mm/min to 1.5 mm/minute with 1119:1 gearbox
- Maximum travel distance 10 mm
- Linear scale for accurate position readout, resolution 3 microns, accuracy 10 microns
- Maximum sample size 50mmx15mmx10mm (L, B, H)

The experimental set up includes a laptop computer that has been installed with the software that controls the microtensile tester, sets drive parameters and displays the stress/strain curve live. Figure 3.4 shows a screen capture of the testing results during microtensile testing.

The software of the MICROTTEST is developed to control the microtest tensile stages. It is graphical in nature and allows control of all necessary parameters. It allows live viewing of the graph plotted of force against extension or time. Sampling time is also a variable and it can be set between 100 ms and 5 s per sample. Communication between the module and PC is via RS-232. Other software available to assist in using this

microtensile tester to get results will be using an integrated video capture which allows for simultaneous video and data capturing with the data (force and extension) stamped on images.

(a)

(b)

Figure 3.3 (a) Deben microtensile tester with load cell of 300 N. (b) Dimensions of Deben microtensile tester.

Figure 3.4 A screen capture of microtest software

3.3.2 Specifications of Microtensile Specimens

All specimens used for the microtensile testing were fabricated by Electric Discharge Machining process using 0.2 mm diameter wire cut. The sample size is limited by the constraints of the load capacity of the microtensile tester. The specimen’s dimensions were carefully chosen to make it possible to break the specimens with a load of less than 300 N.

Both smooth and notched specimens were used. The notches were especially introduced to make it possible to monitor the deformation and fracture processes, which normally start from the notch root. Geometric dimensions of the notched specimens are illustrated in Figure 3.5 and some specimens are shown in Figure 3.6.

Figure 3.5 Dimensions of notched specimens (all measurements in mm). The notch angle can be varied from 45° to 15°, 30°, 75° or any other degrees.

Figure 3.6 Samples fabricated using EDM wire cut process.

3.3.3 Metallographic Preparation of Surfaces for in-situ Observation

The preparation of the surfaces of the specimens for the in-situ observation involved a few steps. First, SiC paper grinding was carried out to smoothen the surfaces which might be damaged during the EDM wire cutting. The samples are very small and delicate, hence forces applied have to be moderate in order to remove any unwanted surface material to reveal the true structure of the specimens.

The grinding processes were carried out in several steps for both AZ91D and AM50A specimens progressively from 400, to 600, 1000 and finally 1200 grits silicon carbide paper. Careful cleaning of the specimens was performed between the steps to prevent cross contamination of coarse abrasive particles in the finer steps. Besides, to avoid the formation of directional grinding, these operations were performed with a rotation of the specimen by 90° after each step to eliminate scratches formed in the previous step. Although grinding produces a flat, smooth surface, it is suitable for macroscopic examination only.

Secondly, both AZ91D and AM50A specimens were polished with 1 μm and then 0.5 μm diamond suspension on a polishing cloth covered on a disc rotating at about 100 rpm. The polishing lubricant used was an oil based one to prevent the polished surfaces from corrosion. During polishing, it should be noted that excessive pressure may cause heating and flowed surfaces, so the pressure had to be carefully adjusted. When the polished surface became scratch-free and mirror-like, it could be used for in-situ observation. However, some surfaces were etched to reveal the microstructures before the microtensile test and in-situ observation were carried out. The etching technique is described later in Section 3.7.

3.3.4 Geometric Measurements of Microtesnile Specimens

After grinding and polishing, measurements of thickness, width, and notch net cross-sectional width of each specimen were carried out using a combination of digital vernier calipers and Carl Zessis optical microscope (shown in Figure 3.7). The thickness and width were measured using vernier calipers, taking at least 3 readings before averaging them out. The notch width was measured on the Carl Zessis optical microscope to get the true width under the notch as shown in Figure 3.8.

Figure 3.7 Carl Zessis model optical microscope used.

Figure 3.8 The location of the true width under the notch measured using optical microscope.

3.4 Techniques for in-situ Observation

The microtensile tester is so small in size (Figure 3.3) that it can be placed either under an optical microscope or inside the chamber of a scanning electron microscope (SEM), making it possible to monitor the whole deformation and fracture processes when the mechanical testing is going on. Figure 3.9 shows how a pre-polished specimen was installed onto the jaws of the tester. The two ends of the specimen were tightened using Allen key.

Figure 3.9 Positioning of sample onto microtensile tester

Carl Zeiss optical microscope shown in Figure 3.7 is used for the optical in-situ observation. Due to the height of the microtensile tester on the stage of the microscope, only 5 times objective lens was able to focus on the sample without hitting it. Hence, there is a limitation with the magnification of the sample while using optical microscope.

For in-situ observation under the SEM, the microtensile tester module shown in Figure 3.3 was mounted onto a specially prepared base inside the chamber of Joel Scanning Electron Microscope Model JSM-5600LV and a feed-thru (RS 232 port) was used to pass the load cell signals to the notebook computer placed outside the SEM chamber.

The experimental set-up is shown schematically in Figure 3.10. Figures 3.11 and 3.12 show how the microtensile tester is attached to the SEM in preparation for observation during testing in it. Figure 3.13 shows a completed set-up for the in-situ SEM observation.

Figure 3.10 Schematic set-up of the in-situ observation under SEM.

Figure 3.11 Microtensile tester is connected through RS 232 port before it is mounted into the SEM chamber.

Figure 3.12 Microtensile tester fitted onto stage inside the SEM chamber.

Figure 3.13 Completed set up for SEM observation (the tester module inside the chamber cannot be seen but its is connected to the notebook computer by the cable)

3.5 Tensile Split Hopkinson Bar Test

Magnesium alloy AM50A was tested using tensile split Hopkinson bar. The configuration of a dynamic Hopkinson bar tension specimen with threaded ends is shown in Figure 3.14.

Figure 3.14 Dimensions of impact tension specimen with threaded ends

The apparatus consists principally of a striker tube, input bar, output bar, anvil bar, absorber bar, gun barrel and a gas chamber mounted and aligned to a rigid base, as shown schematically in Figure 3.15. Both the anvil bar and input bar are screwed together to form a stepped bar. All the bars and striker tube are made from cold-drawn bearing steel. The diameter of both input and output bars is 12 mm and their lengths are 2500 mm and 1500 mm respectively. The length and diameter of both anvil and absorbing bars are 500 mm and 20 mm accordingly.

Figure 3.15 Schematic diagram of the Hopkinson bar set-up

Two waves are produced at the impact surface by an axial impact with the striker tube propelled from the gun barrel by the compressed air released from the gas chamber. One is the tensile wave which travels down the input bar. The other is a compressive wave that travels in the anvil bar through the interface of the anvil and the absorbing bar in an undispersed manner. The compressive pulse continues to propagate until it reaches the free end of the absorbing bar. From there, it reflects and propagates back as tensile pulse. As the absorbing bar is not fastened to the anvil bar, the pulse cannot enter the anvil bar. The functions of an absorber bar are to absorb the impact energy to ensure that both the anvil and input bar are almost stationary during the experiment and to eliminate the compressive wave in the anvil bar. Higher striker tube velocity leads to higher strain rate.

The tensile wave will propagate along the input bar to the input bar/specimen interface. At the interface, the tensile wave is partially reflected and partially transmitted through the specimen to the output bar. This is due to the impedance mismatch. The incident, reflected and transmitted wave signals are obtained by the strain gauges mounted onto the input and output bars. It will convert these signals into voltage signals that are displayed on the oscilloscope as shown in Figure 3.16.

Figure 3.16 Typical set of oscilloscope traces from tensile Hopkinson bar test

3.6 Metallographic Observation

The metallographic specimens of the magnesium alloys were cold-mounted in epoxy resin and then grounded and polished for metallographic observation of the microstructures.

Although certain information may be obtained from as-polished specimens, the microstructure is usually visible only when after etching. Only features, which exhibit a significant difference in reflectivity, can be viewed without etching. This is the true of microstructure features with strong colour differences cause relief formation. Cracks, pores, pits, shrinkages, and nonmetallic inclusions may be observed in the as-polished condition. In most cases, a polished specimen will not exhibit its microstructure because light is uniformly reflected. Since small differences in reflectivity cannot be recognized by human eye, some means of producing image contrast must be employed. This has become known as grain contrast etching. Both magnesium alloy samples after the last step of polishing of 0.5 diamond suspensions, the microstructures were ready to be viewed under the optical microscope or SEM.

Etching was carried out using 2% nital. The specimens were then washed in running water and swiped with soap and cotton wool to remove any etchant left on the surface before being blown dry with compressed air. Care must be taken while blowing the specimen with compressed air, as the force may bend the specimens while drying.

Selected broken halves of Hopkinson bar specimens were sectioned along the axis direction and prepared for examination of microcracks. The sectioning of the specimens is illustrated in Figure 3.17.

Figure 3.17 Sectioning of Tensile- and Creep-Tested Specimens

3.7 Fractographic Observation and EDX

Joel Scanning Electron Microscope Model JSM-5600LV was used to examine the microstructure of the samples at higher magnifications. The SEM was also used to examine fracture surfaces of the broken specimens. Elemental analysis was carried out using an energy dispersive x-ray (EDX) spectrometer mounted onto the SEM.

3.8 Microhardness

Matzusawa Microhardness tester was used for the Vickers microhardness testing in the project with a load of 25 grams and loading time of 15 seconds. Samples were polished to a scratch-free surface condition prior to testing.

MTS Nano Indenter XP system and software called Test Work were used to carry out nanoindentation tests of various parts of the microstructures. The results obtained include hardness, Young's modulus and contact stiffness.

3.9 Laser Welding

The laser used was Lumonics JK704 neodymium yttrium aluminum garnet (Nd:YAG) laser with the technical specifications shown in the Table 3.3. It combines 20 kW peak power with low divergence resonator optics to produce high intensity laser spot. The laser machine was incorporated into the MAHO LASERCAV five-axes machine centre to control movement of samples. The magnesium alloy specimen plates were clamped onto the custom-made jig fixture.

The laser power was varied from 250 W to 330 W and welding speed ranging from 10 mm/s to 20 mm/s were chosen for laser welding of 3 mm by 2 mm of 1mm thickness AZ91D magnesium alloy plates. Laser power of 250 W and welding speed of 10 mm/s were selected for laser surface treatment area of 10 mm by 10 mm on 3 mm thick AZ91D magnesium alloy plates.

Prior to welding and surface treatment, surfaces of AZ91D samples were ground using 1000-grit SiC paper to remove the oxidized film and then degreased with acetone. Argon was used as the shielding gas during the welding or surface treatment processes. Pulse rate of 160 Hz and pulse duration of 0.3 ms were kept constant throughout the experiments.

Table 3.3 Technical Specifications of MAHO Nd: YAG laser system

Wavelength:	1.064 μm (near infrared)
Power output:	0 – 400 W continuous-wave
Pulse energy:	0 – 55 J
Pulse duration:	0.3 – 20 ms
Pulse rate:	0.2 – 500 Hz
Maximum pulse output:	20 kW
Beam diameter:	10 mm
Beam divergence:	3 – 30 mrad

CNC controller:	5 simultaneous axes
Working volume:	700(L) x 600(W) x 500(H) mm

3.10 Tensile Testing

A Instron 5569 model machine of 50 kN capacity was used to carry out tensile testing of laser welded specimens at a speed 0.01 mm/min. The specimens used were 1 mm thick, 20 mm wide and 60 mm long.

3.11 Oxidation Testing

3.11.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) from TA instruments was used to the study the effect of oxidation on as-cast AZ91D ingot. In the project, weight change kinetics of bulk specimens weighing around 10 mg was studied under isothermal conditions in the temperature range of 400-500 °C for time intervals up to 60 min. The specimens were inserted into the heating chamber at room temperature and heated at a rate of 10 °C/min.

All the tests were conducted in air. Temperature and sample weight were monitored continuously using a computer throughout each test. After the TGA tests, surfaces of the samples were carefully observed under the optical microscope and scanning electron microscope.

3.11.2 In-situ Observation of Oxidation

An innovative method was devised to make it possible to observe oxidation of Mg alloys directly under the optical microscope. This was achieved through the careful integration of a heating stage (THMS600), a temperature controller (TMS 93), and a

high-quality optical microscope (Zessis Axioscope), as illustrated in Figure 3.18. Because of the insertion of heating stage under the optical microscope, long work-distance lens had to be used, as shown in Figure 3.19.

Samples were cut into thin foils to make it easier for heat conduction and thus to achieve uniform temperature on the observed surface. The thin foil surface was carefully ground and polished before the heating experiment. The oxidation testing temperature was ramped from room temperature to an elevated temperature of up to 500 °C at a rate varying from 5 °C/min to 100 °C/min. During the heating process, the surface was monitored continuously using the optical microscope and optical micrographs were taken at intervals to show the whole oxidation process.

Figure 3.18 Heating stage and controller setup with optical microscope

Figure 3.19 THMS600 working distances

3.13 XRD Analysis

X-ray diffraction analyzer (Philips Model No. PW1830) was used for analyzing the phases in laser treated samples. X-ray diffraction patterns were obtained using copper target as a source of X-ray with wavelength $\lambda = 1.5404 \text{ \AA}$ (Cu K α 1). The scanning angle was in the range of 10° - 100° and scan speed of $0.01^\circ/\text{s}$. The detected peaks were then compared with the reference materials peaks obtained from the Powder Diffraction File (PDF) to determine the phases present in laser treated AZ91D surface.

Chapter 4 In-situ Observation of Fracture Processes in AZ91D Magnesium Alloy

4.1 Introduction

This chapter describes results obtained for in-situ observation of failures in AZ91D ingot and die castings. Preparation of the specimens and microtensile testing methods are described in details in Chapter 3. A large part of the chapter focus on describing the deformation and fracture processes using SEM and optical images. Finally, the results are analyzed to propose a fracture model.

4.2 Study of AZ91D Ingot

4.2.1 Microstructure of AZ91D Ingot

Figure 4.1 shows the microstructure of etched AZ91D magnesium alloy. The major strengthening intermetallic is β ($\text{Mg}_{17}\text{Al}_{12}$) phase. The body centered cubic (b.c.c.) structure of $\text{Mg}_{17}\text{Al}_{12}$ is incompatible with the h.c.p. structure of magnesium matrix thus cracks may easily form at the intersections of the 2 zones. Table 4.1 shows the chemical compositions of the different phase in the microstructure.

Figure 4.1 Microstructure of AZ91D magnesium alloy.

Table 4.1 EDX analysis of AZ91D Magnesium Alloy

Symbol	Averaged Weight Percentage (Wt%)		Phase	Remarks
	Mg	Al		
A	71.1	28.9	Mg ₁₇ Al ₁₂	Along Grain Boundary
B	94.5	5.5	Primary α	Lower in Al Content
C	88.4	11.6	Eutectic α/β	Medium Al Content

4.2.2 Mechanical Properties of AZ91D Ingot

Nine specimens with three different notch angles (15°, 45° and 75°) were tested for the in-situ observation. All the specimens were carefully polished before the testing, as described in Chapter 3. The results on the in-situ observation are shown in the next section, and this section summarizes the mechanical properties only.

During the microtensile tests, the load Vs displacement curves were recorded using a computer system. A data sampling rate of 100 ms was carefully chosen to ensure not to cause data overflow and in the meantime record sufficiently large number of data points for further analyses. A typical raw data record all the useful data including time, displacement and force acting on the microtensile specimen automatically throughout the testing.

An EXCEL program was written to input the important data of displacement and force to calculate stress and strain and to plot stress vs. strain curves. The program can also be used to obtain maximum load, fracture load, etc. Figure 4.2 shows a typical stress-strain curve obtained.

Figure 4.2 Typical stress-strain curve obtained.

The stress-strain curves for all the specimens were plotted and the maximum stresses tabulated. The results were split by the different methods of calculating the cross sectional area of the specimens which would be used in the calculation of the maximum stresses.

As calculation of stress is done by dividing force over area, there were 2 considerations for the area calculation. The results will be split between the smooth section and the notch section, as shown in Table 4.2. Results for the smooth section are “nominal” applied stresses because the method ignores the presence of the notch. Results in notch

section on the other hand consider the notch into the area, but they are also nominal values because the stress concentration caused by the notch is ignored.

From the results obtained, it can be seen that the overall notch section's results displayed a higher maximum stress as compared to the overall result of the smooth section for all 3 notch angles. This is due to the fact that area considered from the notch section is smaller than the smooth section, thus resulting in higher values.

Other than the trend seen in the maximum stress level, both sections' results also show a trend in the amount of stress for a given notch angle. It shows that the smaller the notch angle, the higher the fracture stress level.

Table 4.2 Tabulation of microtensile test results.

RESULTS for the smooth section:			
	Sample Name	Max Stress (MPa)	Failure Strain
15 degrees	A1	196	0.019
	A2	101	0.021
	A3	89	0.133
45 degrees	B1	83	0.012
	B2	97	0.049
	B3	102	0.010
75 degrees	C1	82	0.011
	C2	74	0.010
	C3	96	0.019
RESULTS for notch (nominal):			
15 degrees	A1	294	0.019
	A2	163	0.021
	A3	138	0.133
45 degrees	B1	133	0.012
	B2	152	0.049
	B3	167	0.010
75 degrees	C1	125	0.011
	C2	115	0.010
	C3	149	0.019

The decrease in notch angle increases in stress triaxiality and stress concentration, and therefore smaller notch angle usually tends to reduce the fracture stress. However, the phenomenon observed can be explained in terms of sampling process. Small notch angle leads to localized plastic deformation zone and therefore makes it less likely to “sample” large defects in the magnesium alloy.

4.2.3 In-situ Observation of Fracture Processes

The microtensile test was performed under the optical microscope and the SEM. During each microtensile testing experiment, an in-situ observation of the fracturing process was performed with pictures taken and recorded using a computer. The pictures were taken starting from 0 s and in different timings until the specimen broke. For the SEM pictures, due to the ability of the machine to go into higher magnification as compared to the optical microscope, it is able to investigate into the different regions of the specimen and perform a more detailed observation on the microstructure especially the intermetallic ($\text{Mg}_{17}\text{Al}_{12}$) during the fracturing process.

After the specimen has broken, the fracture surfaces are observed using the SEM to investigate more into the fracturing process. In this report, results from specimen Mg-D-A2 for optical microscope and specimen Mg-D-A3 for SEM are chosen to demonstrate the experimental technique and its application to study of fracture mechanisms.

4.2.4 In-situ Observation under Optical Microscope

Specimen Mg-D-A2 with a 15 degree notch was tested under the optical microscope and the following pictures were taken. The pictures were taken in different timing (and thus different and progressively higher stress levels), as shown below in Figures 4.4(a)-(f). The pictures taken in sequence were then processed to make a movie to show the whole deformation and fracture process.

Figure 4.4(a) Optical micrograph taken at 0 second (just before loading started)

Figure 4.4(b) Optical micrograph taken after loading for at 30 s.
Note the crack starting to form at the bottom right of the notch (arrowed).

Figure 4.4(c) Optical micrograph at loading for 40 s. Note that the crack has grown and wrinkling (slip lines) forming at both sides of the notch.

Figure 4.4(d) Optical micrograph at loading for 47 s.
Crack further grew and the crumpling becoming very obvious.

Figure 4.4(e) Optical micrograph at loading for 49 s with the specimen nearing to fracture.

Figure 4.4(f) Optical micrograph taken after loading for 49.5 s showing the specimen has fractured.

4.2.5 In-situ Observation under SEM

Specimen Mg-D-A3 with a 15 degree notch was tested using the microtensile tester inside the SEM. Again, many pictures were taken in different timings. Figures 4.5(a)-(c) show the overall picture of the specimen during loading process. A video was made to show the whole deformation and fracture process and it will be shown during the oral presentation.

SEM can be used to get pictures of much higher magnifications, so 4 regions of interest were identified and investigated at higher magnifications, as shown in Figures 4.6-4.9.

After fracture occurred, the fracture surface was observed using the SEM, as shown in Figures 4.10-4.12.

Figure 4.5(a) SEM picture taken at 0 seconds (just before loading)

Figure 4.5(b) SEM picture taken at 35 s showing crack initiated at the bottom left of the notch.

Figure 4.5(c) SEM picture taken at 40 s after the specimens had fracture. Note the secondary cracks (arrowed) at the sides of the main crack.

Region 1

Figure 4.6(a) SEM picture zooming into region 1.

Figure 4.6(b) SEM picture of zone 1 taken at 20 s. Note the intermetallics shown.

Figure 4.6(c) SEM picture of zone 1 taken at 30 s. Note the micro cracks forming within and at the boundaries of the intermetallics.

Figure 4.6(d) SEM picture of zone 1 taken at 35 s.
Note how the cracks propagate through the intermetallics.

Figure 4.6(e) SEM picture of zone 1 taken at 35 s at higher magnification.

Figure 4.6(f) SEM picture of zone 1 taken at 40 s.

The specimen had fracture at this point.

Region 2

Figure 4.7(a) SEM picture zooming into region 2.

Figure 4.7(b) SEM picture of zone 2 taken at 40 s.

Region 2 shows no crack formations throughout the entire experiment.

Region 3

Figure 4.8(a) SEM picture zooming into region 3.

Figure 4.8(b) SEM picture of zone 3 taken at 30 s.
Slight deformation to the intermetallic seen.

Figure 4.8(c) SEM picture of zone 3 taken at 35 s. Cracks forming at 3 separate locations.

Figure 4.8(d) SEM picture of zone 3 taken at 40 s. Specimen has fractured.

Region 4

Figure 4.9(a) SEM picture zooming into region 4.

Figure 4.9(b) SEM picture of zone 4 taken at 20 s. Note the intermetallics.

Figure 4.9(c) SEM picture of zone 4 taken at 35 s. Cracks initiating from intermetallics.

Figure 4.9(d) SEM picture of zone 4 taken at 40 s. Specimen had fractured.

Specimen Mg-D-B4 with a 45 degree notch was tested using the microtensile tester inside the SEM. Figures 4.10 (a)-(d) show the overall fracture process of the specimen. SEM can be used to get pictures of much higher magnifications; so many different

regions of interest such as intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) and eutectic α/β were identified and investigated at higher magnifications, as shown in Figures 4.11- 4.15.

(a)

(b)

(c)

(d)

Figure 4.10 (a), (b), (c) and (d), Fracture process of 45° notched AZ91D magnesium alloy.

(a)

(b)

(c)

Figure 4.11 (a), (b) and (c) Shows that eutectic α/β acting as crack arrestors, thus only the cracking of the intermetallics occurs.

(a)

(b)

Figure 4.12 (a) and (b) Shows multiple cracking in the brittle intermetallics (Mg₁₇Al₁₂). Eutectic α/β have a higher resistance to cracks.

(a)

(b)

(c)

Figure 4.13 (a), (b) and (c) Lines were observed at fracture areas, this is a new phenomenon.

(a)

(b)

Figure 4.14 (a) Intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) is brittle and cracked into bits (Indicate by arrows). Figure 4.14 (b) 45° shearing and tearing of the eutectic α/β

(a)

(b)

Figure 4.15 (a) and (b) The brittle intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) and eutectic α/β particles are strong barriers to dislocation movement and slips, causing build up of local stresses

4.2.6 Study of Strain Lines

A more in-depth study has been conducted on the new phenomenon shown in Figure 4.13 and below in Figure 4.16. 2 Types of different strain lines have been observed, namely Type A and Type B. Type A lines are very fine lines forming at grain boundaries and usually propagate within the grains in primary α . Type B lines are made of thicker slip lines that propagate across grains which are usually 45 degree to the loading direction. 4 different types of tensile specimens; un-notched, 15° notched, 45° notched, 75° notched, were prepared and tested to study the effect of strain on the forming of the strain lines. All specimens were loaded at a constant strain of 0.1 mm/min.

Figure 4.16. Two different types of strain lines after tensile loading on AZ91D magnesium alloy.

Un-notched samples

There were no type A strain lines observed, only some type B strain lines can be observed as shown in Figure 4.17 (a) and (b). Intergranular fracture was the predominant fracture process was shown in Figure 4.17 (c).

(a)

(b)

(c)

Figure 4.17. Micrographs of un-notched samples.

15 degrees notched samples

Large amount of type A strain lines observed due to the high stress concentration. There is lesser amount of type B slip lines observed as compared to the un-notched samples. The type A slip lines are usually on the primary α , along on the intersection with intermetallics β or eutectics α/β as shown in Figure 4.18 (a) and (b). The type A strains lines (Shown in Figure 4.18 c) also effects the eutectic α/β phase too. Figure 4.18 (d) and (e) also shows the inter-crossing of type B strains lines that form a web pattern.

(a)

(b)

S11

(c)

(d)

911

(e)

Figure 4.18 Micrographs of 15 degrees notched samples.

45 degree notched samples

There are moderate amount of both type A and type B strain lines. Figure 4.19 shows the formation of type B strain lines while Figure 4.20 shows the formation of type A strain lines. Large amount of type A strain lines were observed in the primary α along the intermetallics β .

(a)

(b)

Figure 4.19 Formation of type B strain lines under the notch.

Figure 4.20 Formation of type A strain lines under the notch.

(a)

(b)

Figure 4.21 Shows large amount of type A strain lines along the intermetallics β .

75 degrees notched sample

Lesser amount of type A strain lines observed due to the low stress concentration. However larger amount of type B slip lines were observed due to the large amount of plastic deformation.

Figure 4.22 Micrographs showing the formation of large amount of type B strain lines due to the large plastic deformation.

Figure 4.23 Micrograph of the formation of type B strain lines, it affects the intermetallics as well.

4.2.7 Observation of Fracture Surfaces Using SEM

(b)

Figure 4.24 SEM picture of fracture surface of specimen Mg-D-A3. Dimples under notch show sign of ductile failure at the initiation stage.

(a)

(b)

Figure 4.25 SEM picture of fracture surface of specimen Mg-D-B3.
Note the secondary crack.

(a)

(b)

Figure 4.26 SEM picture of fracture surface of specimen Mg-D-C3 showing intergranular fracture facets.

4.3 Study of AZ91D Die Cast

4.3.1 Background

Magnesium alloys have poor hot workability due to their h.c.p. crystal structures. Therefore, they are usually die-cast into required shape. In any die casting process, the melt has to be injected into the die cavity at a very high velocity to avoid cold shut. Even higher filling velocity has to be used for magnesium alloys than for aluminium alloys because magnesium alloys have lower specific heat and cool more rapidly. The very high velocity used in magnesium die casting tends to cause a strong turbulence and therefore lead to the gas entrapment.

Gas pores are common defects in die castings especially in magnesium die castings. No matter how the die casting process is optimized, there always exists porosity in the die cast parts because of the high filling velocity and the turbulence it causes. Recent efforts have been initiated to apply the vacuum die casting process to the production of components for use in the aerospace and automotive industry. The primary difference in

the processing of vacuum die casting process is that the entire process must be performed under relatively high vacuum to achieve the melt cleanliness and very effective for reducing porosity in the casting and producing high quality parts in high volume.

By controlling the vacuum, the pressure differential between the die cavity and the molten metal can be varied allowing for differential fill rates necessitated by part design and gating requirements. This results in tight control of the fill rate which also directly influences the soundness of the casting. Through proper part design, die design and the use of the vacuum die process, voids, shrinks, and gas pockets can be greatly reduced or eliminated in critical areas.

When experimental work was carried out to study fracture of die-cast magnesium alloys, attention was focused on in-situ observation of fracture process of AZ91D die-cast and vacuum die-cast. The die-cast specimens are produced by Professor Xiong Shou Mei research group in Tsing Hua University (Beijing).

2 samples were produced from a TOYO die-cast machine. The sample A1-4 (TA) is produced under vacuum condition, while the A26-4 (TB) without vacuum. Both specimens were cut into different sections and named according to Figure 4.27. Sections 01, 02 and 03 were used for microstructural studies while section 04 and 05 were used for in-situ microtensile testing

Figure 4.27 Diagram of die-cast specimens

4.3.2 Microstructures of Die-cast AZ91D

The microstructures of different sections from the vacuum and normal die-cast samples were ground and polished. The polished samples were then etched and examined under the optical microscope. Optical micrographs from different sections and different die-cast conditions were taken and compared.

Vacuum Die-Cast - Specimen TA01

(a)

(b)

Figure 4.28 Micrographs of the section near the gate of vacuum die-cast specimen.

(a) Near the external side, no porosity is observed. (b) Near the center, rings of porosities are observed.

Specimen TA02

(a)

(b)

Figure 4.29. Micrographs of the section near the end of vacuum die-cast specimen. (a)

Near the external side, no porosity is observed. (b) Near the center, large porosities are observed.

Specimen TA03

(a)

(b)

Figure 4.30 Micrographs of the section near the middle section of vacuum die-cast specimen. (a) Near the external side, no porosity is observed. (b) Near the center, no porosity is observed.

Normal Die-Cast - Specimen TB01

(a)

(b)

Figure 4.31 Micrographs of the section near the gate of die-cast specimen. (a) Near the external side, no porosity is observed. (b) Near the center, a ring of porosities are observed.

Specimen TB02

(a)

(b)

Figure 4.32 Micrographs of the section near the end of die-cast specimen. (a) Near the external side, no porosity is observed. (b) Near the center, a ring of porosities are observed.

Specimen TB03

(a)

(b)

Figure 4.33 Micrographs of the section near the middle section of die-cast specimen. (a) Near the external side, no porosity is observed. (b) Near the middle section, small amount porosity is observed.

From the comparison of the optical micrographs of different sections of both the normal and vacuum die-cast specimens of AZ91D magnesium alloys, vacuum die-cast process have better porosity control over the normal die-casting methods. In addition, porosity control is the best in the middle area within the same die-casting process.

4.3.3 Mechanical Properties of Die-cast

Specimens from different sections of each sample were tested for the in-situ observation. Specimens were cut into a dog bone shape with a 45° notch, similar to those used in the Microtensile testing of the as-cast section. All the specimens were carefully polished before the testing, as described in Chapter 3. The results on the in-situ observation are shown in the next section, and this section summarizes the mechanical properties only. The results calculated are consider nominal as stress concentration caused by the notch is ignored

Table 4.3. Tabulation of microtensile test results.

Sample	Young's modulus (MPa)	UTS (MPa)	0.2% proof line (Yield strength Mpa)
TA04-1	19122	184	164
TA05-1	17551	230	200
TB04-1	13989	192	190
TB05-1	15610	178	170
TA04-2	29578	210	192
TA05-2	13450	212	210
TB04-2	23369	204	186
TB05-2	19555	173	173
TA04-3	19521	205	161
TA05-3	23408	196	182
TB04-3	19821	192	152
TB05-3	16209	182	163

Figure 4.34 Comparison of UTS of vacuum and normal die-cast specimens.

From the results obtained, it can be seen that the results of the vacuum die-cast displayed a higher maximum stress as compared to the results of normal die-cast. This may be due to the fact that there were less porosity in vacuum die-cast samples as compared to the normal die-cast samples as observed in the previous section.

4.3.4 Optical Microtensile testing

Specimen TA05-2

Figure 4.35 Optical micrographs showing the fracture process of vacuum die-cast sample TA at different loads and time. The fracture is brittle.

Specimen TB05-2

Figure 4.36 Optical micrographs showing the fracture process of vacuum die-cast sample TB at different loads and time. The fracture is brittle.

4.3.5 SEM Microtensile testing

Specimen TA04-3

Figure 4.37 SEM micrographs showing the fracture process of vacuum die-cast sample TA at different loads and time. The fracture is brittle.

After fracture

Figure 4.38 SEM micrographs of vacuum die-cast sample TA after fracture. Cracks in the intermetallics can be observed.

Specimen TB05-3

Figure 4.39 SEM micrographs showing the fracture process of vacuum die-cast sample TB at different loads and time. The fracture is brittle.

After fracture

Figure 4.40 SEM micrographs of vacuum die-cast sample TB after fracture. Cracks in the intermetallics can be observed in (a) and (b). Strain lines were also observed in (c) and (d).

4.3.6 Observation of Fracture Surfaces Using SEM

Specimen TA4-03

(c)

Figure 4.41. Fractographs of sample TA near the notch showing transgranular fracture.

(a)

(b)

(c)

(d)

Figure 4.42 Fractographs away from the notch showing intergranular fracture facets

4.4 Discussion

4.4.1 Comparison of Optical Microscope and SEM Observation

From the comparison of the pictures between the optical microscope and the SEM (for specimens Mg-D-A2 and Mg-D-A3), it can be seen that for the optical microscope, it is possible to observe slip lines and thus “crumpling” of the specimens below the notch root. This is because the slip lines produce high optical contrast for optical observation. The slip lines do not show large topographic differences, so they cannot be observed easily under the SEM, though SEM can be used to get images of much higher magnifications.

For optical image, it is not easy to observe the region of stress near to the notch as the surface turns dark. However, SEM is generally a much better tool for observing the crack formation. SEM can also reveal the intermetallics compounds clearly. Figure 4.43 compares images obtained using the optical microscope and SEM.

Figure 4.43 Comparison of optical microscope picture and SEM picture.

4.4.2 Comparisons of As-Cast and Die-Cast Ingot

By comparing the 45° notched samples from both the as-cast and die-cast specimen, it is clear that the die-cast specimens have an higher overall UTS than the as-cast specimens. This maybe due to the finer intermetallics structures in the die-cast specimen. It can also be seen in the result that vacuum die-cast displayed a higher maximum stress as compared to the results of normal die-cast. This may be due to the fact that there were less porosity in vacuum die-cast samples as compared to the normal die-cast samples.

4.4.3 Analyses of Fracture Process

Theoretical strength of a solid is about 10% of its Young's modulus. Magnesium alloy has a Young's modulus of about 45 GPa, so its theoretical strength is expected to be as high as 4500 MPa. In fact, the actual experimental values obtained (Table 4.2) in the project are significantly lower. However, this is not surprising. It was found in the early 20th century by Griffith that the actual strength of brittle solids was several orders of magnitude lower than their theoretical strength.

Many experiments were carried out to explain the discrepancy, and the research results are represented by the Griffith theory. As reviewed in Chapter 2, Griffith (1921, 1924) introduced an energy-balance argument and derived the following equation:

$$\sigma_f = \left(\frac{2E'\gamma}{\pi a} \right)^{\frac{1}{2}} \quad (4.1)$$

where σ_f is the fracture stress, E' is Young's modulus, γ is the surface energy need to create new fracture surface, and a is the half length of microcrack.

The Griffith equation clearly points out the role of defects in decreasing the strength of brittle materials, but also indicates that the final fracture event occurs at some critical tensile stress determined by the material properties and the length of the pre-existing defect.

The in-situ observation revealed clearly the deformation and fracture process. First, plastic deformation occurred at the notch root, as can be seen from the dark slip lines in the optical images. In the 2nd stage, the plastic deformation and the progressively higher loading led to cracking of the brittle intermetallic particles $Mg_{17}Al_{12}$ (especially those located at the grain boundaries) in Figures 4.14, because they were strong barriers to dislocation movement and thus caused build-up of local stress. More examples of cracked particles are shown in Figures 4.29-4.30. In the final stage, when both applied stress increased and the crack length increased, brittle fracture propagation suddenly occurred when the Griffith equation was satisfied.

Fig. 4.44 Illustration of how stress forms at intermetallics.

Fig. 4.45 SEM picture of specimen Mg-D-C3 showing cracked intermetallics.

Fig. 4.46 SEM picture of specimen Mg-D-C3 showing secondary cracks associated with cracked particles.

Chapter 5

In-situ Observation of Fracture Processes and Dynamic Properties of Magnesium Alloy AM50A

5.1 Introduction

This chapter describes results obtained for in-situ SEM observation of fracture and dynamic properties in AM50A. Preparation of the specimens and microtensile testing method are described in detail in Chapter 3 and Section 4.2. Microstructures of the magnesium alloy were studied carefully and the results are also presented in this chapter.

5.2 Study of AM50A Ingot

5.2.1 Microstructure of AM50A Ingot

Figure 5.1 and Figure 5.2 show both optical and SEM micrographs respectively of the AM50A microstructure in the as-cast condition. The microstructure was found to contain intermetallic β phase ($\text{Mg}_{17}\text{Al}_{12}$), eutectic α/β and primary α . The β phase ($\text{Mg}_{17}\text{Al}_{12}$) in the AM50A magnesium alloy can be seen distributed evenly through out in the matrix however they are much smaller in size as compared to those of the AZ91D magnesium alloy.

EDX analyses of various phases present in the AM50A magnesium alloy were carried out. The results are shown in Figure 5.2(c) and Table 5.1. Typical EDX spectra are shown in Figure 5.3. It can be seen from the results that the intermetallic phase is predominantly $\text{Mg}_{17}\text{Al}_{12}$. Large amount of manganese was also detected in some of the rod-like particles. This is not surprising, considering that Mn is one of the two major

alloying elements in the alloy, as shown in Table 3.1.

Under SEM, two different regions in the primary α were discerned: the darker region contains a much higher amount of aluminium than the lighter region.

Figure 5.1 An optical micrograph showing microstructures of AM50A ingot in the as-cast condition.

Figure 5.2(a) SEM micrograph of AM50A at low magnification.

Figure 5.2(b) A close-up of microstructure in Figure 5.2(a) showing rod-like structure (arrowed) in the matrix.

Figure 5.2(c) A further close-up of the $Mg_{17}Al_{12}$ intermetallics, showing 2 regions of the primary α , a darker region (B) and a lighter region (A). Various microstructures identified as A to F were analyzed using EDX (Table 5.1)

Figure 5.2(d) SEM micrograph at high magnification clearly showing the unsmooth $Mg_{17}Al_{12}$ intermetallics and needle-like eutectic α/β .

Table 5.1 EDX analyses of composition of phases in AM50A

Symbol	Averaged Weight Percentage (wt%)			Phase	Remarks
	Mg	Al	Mn		
A	100	-	-	Primary α	Lighter region
B	98	2	>0.1	Primary α	Darker region
C	68	32	-	Mg ₁₇ Al ₁₂	Intermetallics
D	67	33	-	Mg ₁₇ Al ₁₂	Intermetallics
E	90	10	-	Eutectic α/β	Higher Al Content
F	42	42	16	Mn Complex	Rich in Al

Figure 5.3(a) EDX spectrum for lighter region of primary α .

Figure 5.3(b) EDX spectrum for darker region of primary α .

Figure 5.3(c) EDX spectrum for Mg₁₇Al₁₂ intermetallics.

Figure 5.3(d) EDX spectrum for eutectic α/β .

Figure 5.3(e) EDX spectrum for rod-like intermetallics.

5.2.2 Microtensile Results

Specimens with three different notch angles (15° , 45° and 75°) were tested for the in-situ observation. All the specimens were carefully polished before the testing, as described in Chapter 3. During the microtensile tests, the load against displacement curves was recorded using a computer system. A data-sampling rate of 100 ms was carefully chosen to ensure not to cause data overflow and in the meantime record sufficiently large number of data points for further analyses.

For AZ91D and AM50A magnesium alloys, their h.c.p. slip systems do not allow much deformation leads to the limitation of deformability and work strengthening. A major strengthening intermetallic is β ($\text{Mg}_{17}\text{Al}_{12}$) phase. The body centered cubic (b.c.c.) structure of $\text{Mg}_{17}\text{Al}_{12}$ is incompatible with the h.c.p. structure of magnesium matrix, leading to the fragility of the $\text{Mg}/\text{Mg}_{17}\text{Al}_{12}$ interface. In addition, $\text{Mg}_{17}\text{Al}_{12}$ itself is relatively soft and has poor strength. Therefore, microcracks tend to initiate in the $\text{Mg}/\text{Mg}_{17}\text{Al}_{12}$ interface and even within the $\text{Mg}_{17}\text{Al}_{12}$ particles if the particles are large in size. The AM50A alloy contains very fine $\text{Mg}_{17}\text{Al}_{12}$ particles, so its ductility is expected to be better than that for AZ91D, and this is confirmed by the testing results.

The stress-strain curves for all the specimens were plotted and the maximum stresses tabulated. The results were split by the different methods of calculating the cross sectional area of the specimens which would be used in the calculation of the maximum stresses.

As calculation of stress is done by dividing force over area, there were two considerations for the area calculation. The results will be split between the smooth section and the notch section, as shown in Table 5.2. Results for the smooth section are “nominal” applied stresses because the method ignores the presence of the notch. Results in notch section on the other hand consider the notch into the area, but they are

also nominal values because the stress concentration caused by the notch is ignored.

From the results obtained, it can be seen that the overall notch section's results displayed a higher maximum stress as compared to the overall result of the smooth section for all 3-notch angles. This is due to the fact that area considered from the notch section is smaller than the smooth section, thus resulting in higher values.

Table 5.2 shows that the failure stresses decrease with decreasing notch angle. This is understandable because smaller angle leads to greater stress concentration at the notch root. However, it is noted that the trend is different for AZ91D (Chapter 4).

Table 5.2 Tabulation of microtensile test results

Notch is ignored	Sample ID	Max Stress (MPa)	Nominal Yield Stress (MPa)	Failure Strain	Strain rate (/s)
15degrees	AM01	66.9	56	0.034	0.0001667
45degrees	AM02	83.9	29	0.055	0.0001667
45degrees	AM05	93.5	30	0.07	0.0001667
75degrees	AM03	115.8	45.4	0.06	0.0001667
Notched	Sample Name	Max Stress (MPa)	Nominal Yield Stress (MPa)	Failure Strain	Strain rate (/s)
15degrees	AM01	110.1	89	0.034	0.0001667
45degrees	AM02	135.6	45	0.055	0.0001667
45degrees	AM05	154.7	60.2	0.07	0.0001667
75degrees	AM03	183.4	72	0.06	0.0001667

5.2.3 In-situ Observation of Fracture Process

The microtensile test was performed under the SEM. During each microtensile testing experiment, an in-situ observation of the fracturing process was performed with pictures taken and recorded using a computer. The pictures were taken starting from initial loading and in different stress range until the specimen broke. With SEM it is able to investigate into the different regions of the specimen and perform a more detailed observation on the microstructure especially the intermetallic during the fracturing process, due to the ability of the machine to go into high magnification.

Figures 5.4 show the fracture process of the AM50A magnesium alloy under stress levels. Loading directions is horizontal.

Figure 5.4(a) At 80 MPa

Figure 5.4(b) At 140 MPa

Figure 5.4(c) At 155 MPa

Figure 5.4(d) At 110 MPa

Figure 5.5(a) SEM micrograph showing the close-up of the notched area at 150 MPa. Note that wrinkling (slip lines) forming under the notch.

Figure 5.5(b) SEM micrograph showing the close-up of the notched area at 155 MPa. Note that more wrinkling (slip lines) had formed under the notch. Crack had also start to initiate from the wrinkling lines. Intermetallics as shown by

the arrow have also cracked.

Figure 5.6(a) SEM micrograph showing the extended range of the winking affecting the specimen at a loading stress of 135 MPa where the crack is propagating across the specimen.

Figure 5.6(b) A close-up of the previous micrograph. Note the cracking of the intermetallics affected by the slip lines.

Figure 5.7(a) A SEM micrograph showing that the eutectic α is more resistant to fracture than the $\text{Mg}_{17}\text{Al}_{12}$ intermetallics.

Figure 5.7(b) Another SEM micrograph showing crack in the $\text{Mg}_{17}\text{Al}_{12}$, intermetallics but not in the eutectic α .

Figure 5.8 An SEM micrograph of a brittle fracture of the $\text{Mg}_{17}\text{Al}_{12}$ intermetallics.

Figure 5.9(a) Multiple cracking that leads to tearing of the specimen.

Figure 5.9(b) Large deformation along the tear.

Figure 5.9(c) Further close-up of the crack propagation.

5.2.4 Observation of Fracture Surface Using SEM

After tensile testing, the fracture surfaces were examined under the scanning electron microscope. Dimples were observed under notch root, indicating failure at the initiation stage as shown in Figure 5.10(d) and Figure 5.12(d). This shows plastic deformation occurred at the notch root. Column structures were observed on the fracture surfaces.

(a)

(b)

Figure 5.10 Fractographs near the tip of the notch. Dimples were observed.

Figure 5.11 Fractographs near the tip of the notch showing columnar structures.

Figure 5.12 Fractographs away from the tip of the notch showing transgranular facets.

5.3 Study of Dynamic Tensile Properties

5.3.1 Background

Understanding behaviors of material at high strain rates is important for applications such as blast loading, structural impacts, terminal ballistics and metalworking. One of the most widely used experimental configurations for high strain-rate material

measurement is the Split Hopkinson Pressure Bar (SHPB) or Kolsky apparatus. Other techniques such as Pressure-shear Plate impact does exist but is far more expensive and complex compared to SHPB.

In the past, the first use of a long thin bar to measure the pulse shape induced by an impact was considered due to Hopkinson (1914) In this experiment, Hopkinson documented that the displacements in the pressure bar are directly related to the stresses and that the length of the wave in the bar is related to the duration of the impact as obtained from the velocity of sound traveling in the bar as long as the pressure bar remains elastic. The pressures were estimated by measuring the momentum, with a ballistic pendulum, acquired by a small section of the bar placed in contact with bar at the far end. This method has been well established after the critical work of Davies (1948). The experimental setup, with two long bars and a short specimen, known as the split Hopkinson pressure bar has been set up by Kolsky (1949).

Kolsky's original SHPB analysis is based on these three assumptions: the waves propagating in the bars can be described by the one-dimensional wave propagation theory, the stress and strain fields in the specimen are uniform in its axial direction and the specimen inertia effect and the friction effect in the compression test are negligible. Subject to the assumptions and limitations of the technique, dynamics stress-strain curves in compression can be obtained at strain rates exceeding 10^3 s^{-1} . Its yields the highest possible strain rates in an uniaxial compression test under uniform deformation conditions. In the past decades, these assumptions have been extensively studied.

There have been various advances in experimental procedures based on the Hopkinson bar. In 1961, the use of strain gages to measure surface displacements on the elastic pressure bars was first reported. In 1963, a technique for performing elevated-temperature tests was described (Chiddister and Malvern, 1963).

The split Hopkinson bar technique, which has been initially used in compression, has

been extended to tension (Harding et al., 1960) and torsion (Duffy et al., 1971). The principle of the SHPB in tension is similar to those in compression. The primary differences are the methods of generating a tensile loading pulse, specimen geometry and the method of attaching the specimen to the input and output bar.

In the past decades, metals are often used to study with SHPB as plastic behaviour and rate dependence are the main interest. In the present day, SHPB is also applied to many non-metallic materials such as concrete, rocks, salt-rock, polymers and polymeric foams (Chiu and Neubert, 1967; Dioh et al., 1993; Gong et al., 1990).

The aim of this part of work was to determine the reliable tensile stress-strain characteristics of Magnesium AM50A alloy at high strain rates of strain. The impact tension tests were conducted using the tensile version of the split Hopkinson bar. Magnesium AM50A alloy was tested at room temperature. The influence of strain rate on tensile strength is examined.

5.3.2 Method of Data Analysis

The equations for the analysis of the compression split Hopkinson bar assume that stresses and velocities at the end of the specimen are propagated down the bars in an undispersed manner. The stress and strain are assumed to be uniform along the specimen as the wave-transit time in the short specimen is small compared to the total time of the test.

Analysis of the tensile split Hopkinson bar test is almost identical to that of the compression test. The major differences are the actual or effective gauge length of the specimen and the change in sign of the tensile pulse.

The displacement, u_1 & u_2 , and the strain, ε_1 & ε_2 , at the end of the specimen are related

by

$$u_1 = \int_0^t c_o \varepsilon_1 dt \quad (1)$$

$$u_2 = \int_0^t c_o \varepsilon_2 dt \quad (2)$$

where c_o is the elastic-wave velocity in the Hopkinson bars. In terms of the incident, reflected and transmitted waves,

$$u_1 = c_o \int_0^t (\varepsilon_I - \varepsilon_R) dt \quad (3)$$

$$u_2 = c_o \int_0^t \varepsilon_T dt \quad (4)$$

where stresses and strains are assumed positive in compression.

The average strain in the specimen is

$$\varepsilon_S = \frac{u_1 - u_2}{L} \quad (5)$$

or, in terms of the strain pulses

$$\varepsilon_S = \frac{c_o}{L} \int_0^t (\varepsilon_I - \varepsilon_R - \varepsilon_T) dt \quad (6)$$

where L is the length of the specimen. The forces at the ends of the specimen are obtained from

$$P_1 = EA(\varepsilon_I + \varepsilon_R) \quad (7)$$

$$P_2 = EA \varepsilon_T \quad (8)$$

where E and A are Young's modulus and the cross-sectional area of the Hopkinson bars.

The average force is calculated from

$$P_{av} = \frac{EA}{2}(\varepsilon_I + \varepsilon_R + \varepsilon_T) \quad (9)$$

If it is assumed that the forces are equal at both ends of the specimen, i.e., $P_1 = P_2$, then from (6), (7) and (8)

$$\varepsilon_I + \varepsilon_R = \varepsilon_T \quad (10)$$

$$\varepsilon_S = \frac{c_o}{L} \int_0^t (\varepsilon_T - \varepsilon_R - \varepsilon_R - \varepsilon_T) dt \quad (11)$$

For the specimen of cross-sectional area A_s , the strain, stress and strain rate in the specimen become:

$$\varepsilon_S = \frac{-2c_o}{L} \int_0^t \varepsilon_R dt \quad (12)$$

$$\sigma_S = E \frac{A}{A_s} \varepsilon_T \quad (13)$$

$$\dot{\varepsilon}_S = \frac{-2c_o}{L} \varepsilon_R \quad (14)$$

Thus, the stress-strain behaviour of the specimen is determined simply by measurements made on the elastic pressure bars in a split Hopkinson pressure bar test.

The above equations relate strain gauge measurement to stress-strain behaviour in the deforming specimen and require that two conditions be met. The first is that wave propagation within the pressure bars must be one-dimensional and the second condition is that the specimen must deform uniformly.

5.3.3 Split Hopkinson Pressure Bar Results

Figure 5.13 The strain rate in AM50A, obtained by using equation (14)

Figure 5.14 shows a direct comparison of tensile stress-strain curve for the AM50A alloy obtained from static and impact testing at different strain rates. It is observed that tensile strength (UTS) obtained from the static tensile testing is around 167 MPa. The tensile

strength is observed to increase with increasing strain rate. Fracture occurs only when strain rates are at 1150 s^{-1} to 1350 s^{-1} . Figure 5.15 shows both initial and fracture appearance of the AM50A alloy.

Figure 5.14 Stress-strain curves for AM50A obtained from static and impact testing.

(a) Untested specimen

(b) Strain rate of 1050 s⁻¹

(c) Strain rate of 1150 s⁻¹

(d) Strain rate of 1350 s⁻¹

Figure 5.15 Initial and fracture appearances of AM50A at various strain rates. A shearing of 45 degrees can be observed in the highest strain rate of 1350 s⁻¹.

5.3.4 Observation of Fracture Surfaces Using SEM

Figure 5.16 Fracture surface of the specimen after undergoing a strain rate of 1050 s^{-1}

(c)

(d)

Figure 5.17 Fracture surface of the specimen after undergoing a strain rate of 1350 s^{-1} . Steps-like fracture surface can be observed.

(a)

(b)

(c)

(d)

Figure 5.18 Fracture surface of the specimen after undergoing a strain rate of 1350 s^{-1} .

5.3.5 Observation of Sectioned Surfaces Using SEM

Figure 5.19 Selected broken halves of Hopkinson bar specimens were sectioned along the axis direction and prepared for examination of microcracks

(a)

(b)

(c)

(d)

(e)

(f)

Figure 5.20 Section surface of specimen after undergoing a strain rate of 1350 s^{-1} .

Cracks in intermetallics were observed near the fracture surface. Cracks in eutectic α/β were observed, such cracks were not observed in tensile testing.

5.4 Discussion

Based upon the experimental results obtained so far, it is clear that the fracture model proposed to the as-cast AZ91D is applicable to AM50A as well. The in-situ observation revealed clearly the deformation and fracture process. First, plastic deformation occurred at the notch root, as can be seen from the dark slip lines in the optical images. In the 2nd stage, the plastic deformation and the progressively higher loading led to cracking of the brittle intermetallic particles $\text{Mg}_{17}\text{Al}_{12}$ because they were strong barriers to dislocation movement and thus caused build-up of local stress. In the final stage, when both applied stress increased and the crack length increased, brittle fracture propagation suddenly occurred when the Griffith equation was satisfied.

The dynamic stress strain relation of magnesium alloy AM50A has been obtained using the Hopkinson bar apparatus. The strain rate ranges between 600 /s and 1300 /s. The magnesium alloy AM50A displays about 50% higher in tensile stress at the strain rate of about 1300/s than at static. Fast shearing is the fracture mode at the highest strain rates.

Chapter 6

Effect of Oxidation on Failure Mechanism of AZ91D Magnesium Alloy

6.1 Introduction

The continuing needs to reduce weight are driving the usage of magnesium and its alloys in automotive and light truck industries. Besides light weight property, high temperature oxidation and corrosion resistance are also critical for demanding applications like power train. In order to satisfy the needs of today, the corrosion resistance and high temperature properties of the widely used magnesium alloy such as AZ91D has to be promoted.

This chapter describes results obtained for in-situ observation of failures in magnesium alloy after oxidation in Thermalgravimetric Analysis (TGA). As-cast magnesium alloy AZ91D was exposed to air in the temperature range from 400 °C to 500 °C for time intervals up to 1 hour. Thermalgravimetric measurements revealed 3 different stages of reaction. Firstly the formation of an initial protective oxide, followed by an incubation period and finally degrading to a non protective oxide (F. Czerwinski, 2002; Zeng et al., 2001). In-situ observation was also conducted using a heating stage to study the change in surface morphology. Microtensile specimens were also prepared and heated to different temperature range causing oxidization. Microtensile testing method described in Chapter 3 are conducted to observe the fracture process and mechanical properties after oxidization. A large part of the chapter focuses on describing the deformation and fracture processes of oxidized AZ91D using SEM and optical images.

6.2 Thermogravimetric measurements results

Results of the Thermogravimetric measurements revealed the character of the alloy weight change versus the time. As seen in Figure 6.1(a), (b) and (c), as-cast AZ91D is exposed to air at 420 °C, 450 °C and 470 °C for 40 minutes. From the results of the Thermogravimetric measurements, there is a slow initial weight loss followed by a rapid weight gain in all 3 different temperatures. There is also a distinct relation between the temperature and the

weight gain rate. The rate of weight gain increase with higher temperature (F. Czerwinski, 2004).

(a) 420 °C

(b) 450 °C

(c) 470 °C

Figures 6.1 (a), (b) and (c). Thermogravimetric measurements of weight change versus time for as-cast AZ91D.

Chapter 6 Effect of Oxidation on Failure Mechanism of AZ91D Magnesium Alloy

(a)

(d)

(b)

(e)

(c)

(f)

Figures 6.2 (a), (b), (c), (d), (e) and (f). Optical micrographs of AZ91D. (a), (b) & (c) showing the initial microstructure of AZ91 and (d), (e) & (f) showing after exposing to air at 420 °C for 40 minutes.

(a)

(d)

(b)

(e)

(c)

(f)

Figures 6.3 (a), (b), (c), (d), (e) and (f). Optical micrographs of AZ91D. (a), (b) & (c) showing the initial microstructure of AZ91 and (d), (e) & (f) showing after exposing to air at 450 °C for 40 minutes.

(a)

(d)

(b)

(e)

(c)

(f)

Figures 6.4 (a), (b), (c), (d), (e) and (f). Optical micrographs of AZ91D. (a), (b) & (c) showing the initial microstructure of AZ91 and (d), (e) & (f) showing after exposing to air at 470 °C for 40 minutes.

Figure 6.5. Intermetallics cracking and decomposing after undergoing heating at 470 °C.

Figure 6.6. Oxides growing at the grains boundaries.

(a)

(b)

Figure 6.7 (a) A cauliflower - like oxide growing at the intermetallics.
(b) A close-up of “Cauliflower”.

(a)

(b)

Figure 6.8 (a) Flowers pattern on the surface on primary α .
(b) A close-up of the “Flower”.

6.3 In-situ Observation Results

(a) 22 °C

(d) 435 °C

(b) 420 °C

(d) 440 °C

(c) 430 °C

(e) 450 °C

Figure 6.9. Micrographs of the in-situ observations of as-cast AZ91D exposed to air from 22 °C to 450 °C.

As-cast AZ91D was prepared by grinding and polishing to obtain a scratch free surface. The sample was then placed on a LINKAM THMS600 heating plate. The sample was heated in air from room temperature to 450 °C. The sample was first ramp at 100 °C per minute to 400 °C and then 2 °C per minute to 450°C. During the test, optical microscope was used to take pictures at different temperature as shown below in Figures 6.9 (a) - (f). The pictures taken in sequence were then processed to make a movie to show the whole oxidation process of AZ91D magnesium alloy. (Note: The movie will be shown at the oral presentation).

Magnesium alloy AZ91D exhibited protective behavior until 410 °C (Huang et al., 2003; Liu et al.,2004). From Figure 6.9 (b) and (c), it shows clearly that initial oxidation of AZ91D magnesium alloy occurs at around 430 °C. During the initial stage, oxidation only occurs in small portions around the intermetallics ($Mg_{17}Al_{12}$) in Figure 6.9 (c). The preferential sites for oxide growth are the intermetallics and the intersection of grain boundaries (Shown in Figure 6.5 and 6.6) (Zeng et al., 2001; Chen et al., 2005). After 430 °C, the increase in temperature cause a sudden acceleration with oxygen thus accelerate the growth of the dark particles, MgO (showing in Figures 6.10) around the intermetallics and spreading to cover nearly 90% of the sample.

During the acceleration of the oxidization process, the intermetallics also turned black and decomposed as the intermetallics have a melting temperature at around 435 °C. The Mg ions decomposed from the intermetallics may have reacted to form MgO at such high temperature thus accelerating the growth of the MgO on the surface layer. Oxide grew at the decomposed intermetallics site into a cauliflower-like morphology shown in Figure 6.8 (a) and (b).The oxidation of Mg to MgO is the predominant process and upon gradual oxidation, Al reacts with oxygen and Mg to form $MgAl_2O_4$ spinel. The results suggested that the oxidation of AZ91D alloy initiated at the eutectic α/β phase melting temperature which is critical importance of the non-protective oxide formation (F. Czerwinski 2002).

(a)

(b)

Figures 6.10. The oxide layer covering the AZ91D alloy.

(a) Oxide layer covering the surface. (b) A close-

6.4 Microtensile Testing of Oxidized AZ91D

6.4.1 Microtensile Results

Nine microtensile specimens with 45° notch angles were tested for tensile strength observation. All the specimens were carefully grinded and polished before the tensile test as described in Chapter 3. The specimens were then heated in a Lenton furnace (model WHT4/30). 3 samples were heated to 420 °C at a rate 10 °C /min and held at 420 °C for 10 minutes. 3 other samples were heated to 450 °C at a rate of 10 °C /min and held at 450 °C for 10 minutes. And the remaining 3 were tested under normal tensile conditions. All samples were tested at a loading rate of 0.1 mm/min.

Figure 6.11. Influence of temperature and oxidation on tensile properties of AZ91D magnesium alloy.

The stress-strain curves for all the specimens were plotted and the maximum stresses tabulated in Figure 6.11. It can be seen that the overall UTS for specimens heated to 420 °C and normal tensile specimens displayed a higher maximum stress as compared to the result of specimens heated to 450 °C. This is clear from the tensile results that the high temperature decreased the failure stress of AZ91D. And the temperature between 350 °C - 450 °C is critical. This is understandable because of the earlier observations in Section 4.3 that melting

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of intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) and severe oxidation and taken place, lowering the mechanical properties of AZ91D magnesium alloy.

6.4.2 In-situ Observation of Fracture Process

The microtensile test was performed under the optical microscope and the SEM. During each microtensile testing experiment, an in-situ observation of the fracturing process was performed with pictures taken and recorded using a computer. The pictures were taken starting from 0 s and in different timings until the specimen broke. For the SEM pictures, due to the ability of the machine to go into higher magnification as compared to the optical microscope, it is able to investigate into the different regions of the specimen and perform a more detailed observation on the microstructure especially the intermetallic ($\text{Mg}_{17}\text{Al}_{12}$) during the fracturing process. After the specimen has broken, the fracture surfaces are observed using the SEM to investigate more into the fracturing process.

6.4.3 In-situ Observation under Optical Microscope

Two specimens each heated to 420 °C and 450 °C respectively were tested under the optical microscope. The pictures were taken in different timing (and thus different stress levels), as shown below in Figures 6.12 and Figures 6.14. The pictures taken in sequence were then processed to make a movie to show the whole deformation and fracture process.

For the specimens heated to 420 °C only some minor surface darkening due to surface oxidation were observed. The intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) not affected by the heating are intact, thus the fracture process (Shown in Figures 6.12) are similar to the fracture process of normal unheated tensile specimens (As describe in Chapter 4). Similar plastic deformations were observed under the notched root during the fracture process.

For the specimens heated to 450 °C severe oxidation was observed on the surface and on the intermetallics. The intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) affected by the heating are decompose and melted (Shown in Figures 6.14). There are voids left behind by the melted intermetallics and

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thus it provides an easy path for the fracture process. No plastic deformation was observed and the fracture process is brittle.

Figures 6.12 (a), (b), (c), (d), (e) and (f). Show the fracture surface of AZ91D magnesium alloy heated to 420 °C under stress levels. Loading direction is indicated by white arrow.

Figures 6.13(a), (b), (c), (d), (e) and (f). Show the close-up of the oxidation process of the AZ91D magnesium alloy heated to 420 °C under stress. The loading direction is horizontal.

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Figures 6.14 (a), (b), (c), (d), (e) and (f). Show the fracture process of the AZ91D magnesium alloy heated to 450 °C under stress levels. Loading directions is horizontal.

6.4.4 In-situ Observation under SEM

The specimen is heated to 450 °C before being tested under the SEM for in-situ observation. Again, the pictures were taken in different timing (and thus different and progressively higher stress levels), as shown below in Figures 6.15. The pictures taken in sequence were then processed to make a movie to show the whole deformation and fracture process. SEM can be used to get pictures of much higher magnifications; different regions of interest were identified and investigated at higher magnifications, as shown in Figures 6.16.

It can be seen in Figure 6.15 (a) that there are voids on the specimens after the heating process. These voids are left over by the melting and decomposition of the intermetallics at the high temperature. These voids provide an easy path for the crack propagation. No plastic deformation had been observed under the notch root and the fracture process is brittle and fast.

Figures 6.15 (a), (b), (c), (d), (e) and (f). The fracture process of the AZ91D magnesium alloy heated to 450 °C under stress levels. Loading directions is horizontal.

(a)

(c)

(b)

(d)

Figures 6.16. Close-up of the fracture process of the AZ91D magnesium alloy heated to 450 °C under stress levels. Loading directions is horizontal.

6.4.5 Fractography

After tensile testing, the fracture surfaces were examined under the scanning electron microscope. Samples heated to 450 °C were observed. Intergranular fracture was observed in Figures 6.17, thus indicating the melting and decomposing of intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) weaken the grain boundaries. Figures 6.18 clearly show the voids left behind after the melting and decomposing of intermetallics ($\text{Mg}_{17}\text{Al}_{12}$). Oxidation in the voids can also be seen.

(a)

(b)

Figures 6.17. Fractography showing intergranular fracture for specimens heated to 450 °C

(a)

(b)

Figures 6.18. Internal oxidation in the voids cause by the melting and decomposing of intermetallics ($Mg_{17}Al_{12}$)

6.5 Discussion

The temperature 420 °C is critical for AZ91D magnesium alloys. Comparing normal tensile specimens and 420 °C there is not much different in the mechanical strength, however for specimens heated to 450 °C, there is a great reduction in mechanical strength (about 50 %).

Even though minor oxidation take place on the surface of the alloy before 420 °C but it does not affect the mechanical strength much. The voids left behind by the melted intermetallics ($\text{Mg}_{17}\text{Al}_{12}$) when heated above 420 °C provide an easy path for the fracture process thus lowering the mechanical strength. The preferential sites for oxide growth are the intermetallics and the intersection of grain boundaries.

Chapter 7

Welding of AZ91D Magnesium Alloy using Nd: YAG Laser

7.1 Introduction

This chapter describe the usage of Neodymium-doped Yttrium Aluminum Garnet (Nd:YAG) laser on the welding of AZ91D magnesium alloys. This chapter will focus on how different parameter setting affects the welding quality and on mechanical properties of the welded joint mainly the tensile properties and the microstructure of the welded and heated affected zone (HAZ) of the material.

7.2 Laser Welding

As the lightest structural material available so far (Haferkamp et al., 2000), magnesium alloys have the potential to replace steel and aluminium in many structural applications (Leong et al, 1998; Sanders et al., 1999). Thus, magnesium alloys have already found considerable applications in aerospace, aircraft, automotive, electronics and other fields, especially magnesium die-castings in the automotive industry (Westengen, 2000).

Magnesium alloys have also been employed in nuclear energy industrial equipments due to the low tendency to absorb neutrons, adequate resistance to carbon dioxide and good thermal conductivity (Froes et al., 1998). Many magnesium alloy components are used at high rotational speeds to minimize inertial forces (Pastor et al., 2000). To date magnesium alloys have not usually been welded except for some repaired structures because of the occurrence of defects such as oxide films, cracks, and cavities (Haferkamp et al., 2000). However, the wider application of magnesium alloys needs reliable welding processes. Magnesium alloy components may be joined using mechanical fasteners as well as a variety of welding methods including tungsten-arc inert gas (TIG), metal-arc inert gas (MIG), plasma arc, electron, laser, friction, adhesive, explosion, stud, ultrasonic, and spot welding.

Today TIG and MIG processes are the main methods for magnesium alloys, especially for the removal and repair of casting defects. However, low welding speeds, large heat

affected zone (HAZ) and fusion zone (FZ), high shrinkages, variations in microstructures and properties, evaporative loss of alloying elements, high residual stress and distortion of arc-welded joints have caused attention to be drawn towards laser welding due to the low and precise heat input, small HAZ, deep and narrow FZ, low residual stress and weldment distortion, and high welding speed.

The effectiveness of laser welding depends greatly on the physical properties of the material to be welded. Magnesium alloys possess certain inherent characteristics such as low absorptivity of laser beams, strong tendency to oxidize, high thermal conductivity, high coefficient of thermal expansion, low melting and boiling temperatures, wide solidification temperature range, high solidification shrinkage, a tendency to form low melting-point constituents, low viscosity, low surface tensions, high solubility for hydrogen in the liquid state, and absence of color change at the melting point temperature (Cao, 2005).

During laser welding of magnesium alloys, therefore, some processing problems and weld defects can be encountered such as an unstable weld pool, substantial spatter (Leong et al., 1998; Sanders et al., 1999; Watkins, 2003), a strong tendency to drop-through for large weld pools (Haferkamp et al., 2001), sag of the weld pool (especially for thick work piece), undercut (Lehner et al., 1999), porous oxide inclusions, loss of alloying elements (Leong et al., 1998; Sanders et al., 1999), excessive pore formation particularly for die castings (Reinhart and Schaller, 1999), liquation and solidification cracking (Marya and Edwards, 2000). Nonetheless, crack-free laser welded joints, with low porosity and good surface quality, can be achieved for wrought magnesium alloys using appropriate laser processing conditions (Weisheit et al., 1997).

Magnesium alloy welding has been insufficiently documented (Marya et al., 2001) but the relative arc weldability of most magnesium alloys has been rated based largely on the susceptibility to cracking and to some extent on joint efficiency (Avedesian and Baker, 1999). The relative laser weldability of magnesium alloys, however, has not been systematically investigated yet. Research into stable laser welding has involved identifying and controlling the processing parameters influencing process stability and reproducibility to reliably produce defect-free welds at high welding speeds. The following discussion focuses on some important processing variables and their

influences on welding process and quality during the keyhole mode welding of magnesium alloys.

The weldability of magnesium alloys is reported to be significantly better with the Nd:YAG laser due to its shorter wavelength, which in turn reduced the threshold irradiance required for keyhole mode welding and produced a more stable weld pool (Leong et al., 1998; Sanders et al, 1999; Watkins, 2003).

7.3 Butt Welding of 3 mm Thick Plates

7.3.1 Welding parameters and microstructure

6 samples were laser welded with varying speeds and spot sizes on 3 mm thick AZ91D plates. All the samples were cleaned thoroughly of dust and foreign particles prior to butt welding. The following table show the parameters used. The gas pressure, laser power and pulse frequency were maintained constant while the welding speed and spot size of the laser beam were varied. This test was conducted to find out the relation of how welding speed will vary with the spot size of the beam used.

Table 7.1 Butt Welding of 3 mm Plates

Sample Name	Gas	Gas pressure (Bar)	Laser power (W)	Welding Speed (mm/s)	Pulse frequency (Hz)	Spot Size (mm)	Remarks
LW-01	Ar	3.5	330	25	160	1	
LW-02	Ar	3.5	330	20	160	1	
LW-03	Ar	3.5	330	15	160	1	
LW-04	Ar	3.5	330	10	160	2	Incomplete fusion
LW-05	Ar	3.5	330	20	160	2	Incomplete fusion
LW-06	Ar	3.5	330	15	160	2	Incomplete fusion

The following Figure 7.1 shows the 6 butt welded samples prior to in-depth investigations of the weld and the various metallurgical defects.

Figure 7.1 Butt Welded 3 mm AZ91D plates

It was found that using 2 mm spot size will result in incomplete fusion of the plates. It is due to the low power density from the 2 mm spot size. Incomplete fusion resulted in weak butt welded joint. Hence, spot size of 2 mm was not used in further butt welding processes in this project. The welded zones are also being investigated. LW-01 was chosen for further research on the welded zone and HAZ using optical microscope and SEM. The sample was sectioned horizontally and polished for investigation.

Figure 7.2 LW-01

Figure 7.3 Optical micrographs of weld

As shown in Figure 7.3, slide blow and porosities were observed in the weld. Slide blow arises when the shielding gas (argon) was directed at the plates at one particular direction during the welding process. As the shielding gas was directed onto the plate at a particular direction, it caused surface turbulence and in turn caused entrapment of gases inside the weld which led to the formation of porosities inside the weld.

Figure 7.4 Optical micrographs of weld toe and HAZ zone

As shown in Figure 7.4, there were no visible cracks observed in the weld toes and HAZ. Further observations were done on the weld root. As shown in Figure 7.5, porosities and cracks were found. The formations of cracks were due to large freezing temperature range, large solidification shrinkage, high coefficient of thermal expansion and low melting point intermetallics constituents.

Figure 7.5 Optical micrographs of weld root

7.3.2. The Microstructure of Laser Treated Surface

In conventional solidification condition, the microstructure of the substrate is composed of primary α -Mg dendrites and eutectic mixture of α -Mg and β -Mg₁₇Al₁₂ in varying proportions depending on aluminum concentration and cooling conditions. (Liu et al., 2005)

Upon laser welded, the surface of the sample was rapidly heated and cooled. Since melting and solidification occur within a very short interaction time and remain confined only to the top surface, the underneath acts as an infinite heat sink without any noticeable change in microstructure (Greenwald et al., 1978).

The weld consists of the network dendrites and columnar dendrites shown in Figure 7.8.

Figure 7.6 SEM micrographs of a laser welded butt joint

The weld boundaries are very well-defined and melting of intermetallics is observed as shown by the arrows in Figure 7.6. Further investigation of the intermetallics is shown in Figure 7.7. The intermetallics are observed to be diffused out. It is due to the high heat intensity of the laser beam that caused the melting and diffusion of the intermetallics.

Figure 7.7 SEM micrographs of different magnification of the intermetallics near the boundary

Figure 7.8 SEM micrographs of the weld (a) 1000X and (b) 2000X magnification

7.3.3 XRD Studies Results

XRD result analysis show that the weld has a microstructure consisting of Mg and $Mg_{17}Al_{12}$, as shown in Figure 7.9, which indicate a composition of the melted layer with rapid solidification has been fabricated by laser surface melting and the laser melting processing did not change the compositions of base metal.

Figure 7.9 X-ray diffraction profiles of the melting surface and the AZ91D substrate.

7.3.4 Investigate of Welding Bead on 3 mm Thick Plate

5 different welding speeds: 70 mm/s, 40 mm/s, 25 mm/s, 15 mm/s and 5 mm/s were experimented on 3 mm thick plates. Laser power of 330 W with 1 mm diameter spot size was kept constant. Samples were grounded with 1000 Grit SiC paper prior to

welding and shielding gas, argon, was introduced 10 s earlier into the chamber before the welding commenced.

Weld A, 70 mm/s

Figure 7.10 Micrographs of 70 mm/s bead on plate

Figure 7.11 Microhardness test of weld A, 70 mm/s

Weld B, 40 mm/s

Figure 7.12 Micrographs of 40 mm/s bead on plate

Figure 7.13 Microhardness test of weld B, 40 mm/s

Weld C, 25 mm/s

Figure 7.14 Micrographs of 25 mm/s bead on plate

Figure 7.15 Microhardness test of weld C, 25 mm/s

Weld D, 15 mm/s

Figure 7.16 Micrographs of 15 mm/s bead on plate

Figure 7.17 Microhardness test of weld D, 15 mm/s

Weld E, 5 mm/s

Figure 7.18 Micrographs of 5 mm/s bead on plate

Figure 7.19 Microhardness test of weld E, 5 mm/s

As can be seen from the microhardness test conducted on all the different welding speed, the microhardness value is significantly higher inside the weld. From the data obtained, 25 mm/s welding speed was selected to be used to butt weld the 1 mm AZ91D plates due to its penetration depth and width.

Another test was conducted to compare the depth, vaporization depth and width of the weld with two different laser welding power. The following table shows the data obtained.

Table 7.2 Results for W01 and W02 samples

Specimen Name	Welding speed (mm/s)	Spot Size (mm)	Power (J/s)	Power Density (J/mm ²)	Depth (μm)	Width (μm)	Vap. Depth (μm)	Cracks
W01-1	5	1	250	50.00	191.39	1065.90	62.16	yes
W01-2	10	1	250	25.00	176.21	949.30	53.25	yes
W01-3	25	1	250	10.00	129.28	1075.07	43.09	yes
W01-4	50	1	250	5.00	141.97	1062.50	62.10	yes
W01-5	100	1	250	2.50	85.06	764.32	28.55	yes
W02-1	1	1	50	50.00	96.36	804.86	25.38	no
W02-2	2	1	50	25.00	67.17	807.38	0.00	no
W02-3	5	1	50	10.00	72.97	792.14	24.11	yes
W02-4	10	1	50	5.00	40.72	735.13	13.37	yes
W02-5	20	1	50	2.50	182.67	638.84	182.67	yes

Figure 7.20 Optical micrographs of W01 and W02 samples

Figure 7.21 Graphical comparisons of results of W01 and W02 samples (a) comparison of depth, (b) vaporization depth (c) weld width

A high laser power will produce a deeper weld depth as it could penetrate deeper into the substrate. The vaporization depth and width of the weld also increases with a higher laser power.

7.4 Tensile Test of Butt Welded 1 mm Thick Plates

7.4.1 Welding parameters

6 sets of 1 mm thick AZ91D plates (LC01 to LC06) were butt welded for tensile test using Instron tensile tester. The shielding gas used was argon, released into the chamber 10 seconds prior to welding. The welding parameters were shown in Table 7.3. An Instron tensile tester was used to conduct the tensile test on a strain rate of 1 mm/min.

Table 7.3 Butt welding parameters

Specimen Name	Welding Speed (mm/s)	Spot size (mm)	Power (J/s)	Power Density (J/mm ²)	UTS (MPa)	Comments
LC01	20	1	200	10.00	32.60	
LC02	15	1	200	13.33	46.00	
LC03	10	1	200	20.00	97.30	Through weld
LC04	20	1	250	12.50	32.20	
LC05	15	1	250	16.67	86.40	
LC06	10	1	250	25.00	104.80	Through weld

LC06, which produced the highest ultimate tensile strength (UTS), was chosen for in-depth observation using SEM and optical microscope.

Figure 7.22 SEM micrographs (top view) of LC06, (a) 50X magnifications (b), 100X magnifications (c), 200X magnifications (d) and 1000X magnifications.

Figure 7.23 Optical micrographs of LC06

As can be observed from Figure 7.23, metallurgical defects like voids and cracks were found inside the weld.

18 sets of AZ91D 1 mm plates (WI-01 to WI-18) were butt welded with varying welding speed and laser power. The welding speeds used were 10 mm/s, 15 mm/s and 20 mm/s. Laser powers used were 270 W, 300 W and 330 W. An Instron tensile tester was used to conduct the tensile test on a strain rate of 1 mm/min.

Table 7.4 Butt welding Parameters

Specimen Name	Welding Speed (mm/s)	Spot Size (mm)	Power (J/s)	Focus Distance	Power Density (J/mm ²)	UTS (MPa)	Through weld (Yes/No)
WI-01	10	1	270	Surface	27.00	97.1	yes
WI-02	10	1	270	Surface	27.00	99.9	yes
WI-03	10	1	300	Surface	30.00	93.2	yes
WI-04	10	1	300	Surface	30.00	85.2	yes
WI-05	10	1	330	Surface	33.00	47.3	yes
WI-06	10	1	330	Surface	33.00	50.1	yes
WI-07	15	1	270	Surface	18.00	18.2	no
WI-08	15	1	270	Surface	18.00	21.9	no
WI-09	15	1	300	Surface	20.00	40.6	yes
WI-10	15	1	300	Surface	20.00	38.4	yes
WI-11	15	1	330	Surface	22.00	39.2	yes
WI-12	15	1	330	Surface	22.00	35.4	yes
WI-13	20	1	270	Surface	13.50	28.8	no
WI-14	20	1	270	Surface	13.50	25.8	no
WI-15	20	1	300	Surface	15.00	22.7	no
WI-16	20	1	300	Surface	15.00	23.3	no
WI-17	20	1	330	Surface	16.50	21.7	no
WI-18	20	1	330	Surface	16.50	16.6	no

7.4.2 In-situ Microtensile testing

The butt welded plate of parameter similar to WT01 and WT02 is sectioned into thin stripes for microtensile test using Deben Microtensile tester. Laser power used was 270 W; welding speed was 10 mm/s and 1 mm diameter spot size. The samples are grinded on the top and bottom surface to ensure perfect weld geometry. The motor speed is 0.1 mm/min. Micrographs of 5X magnification using the optical microscope are taken at regular interval to monitor the changes and showing the whole fracturing process

Figure 7.24 Optical micrographs of WT02 during microtensile test

From the microtensile test, the fracture point is at the substrate instead of it breaking at the laser welded boundaries or weld. The results showed that the laser weld is able to withstand tensile testing more than the substrate. From Figure 7.24, cracks and voids can be seen clearly inside the substrate and intermetallics. The fractography showed that brittle was the mode of fracture.

Figure 7.25 SEM fractography of WT02

Similar microtensile testing was conducted inside the SEM. Higher magnification was possible and thus produced a more detailed result. Micrographs were taken during the microtensile testing process. Laser power used was 270 W; welding speed was 10 mm/s and 1 mm diameter spot size.

Figure 7.26 SEM micrographs

As can be seen from Figure 7.26, despite the formation of cracks near the weld boundaries, the fractures still occurred at the substrate instead of the weld boundaries. The fractures that occurred were all brittle. The SEM results obtained demonstrated that the weld was stronger than the substrate.

7.5 Discussion

Butt welds of AZ91D magnesium alloy were produced using a Nd:YAG laser and with Argon as protective gas. The main weld defect was porosity in the fusion zone. Hot cracking was also observed. Optimum tensile properties were obtained for welds made with a welding speed of 10 mm/s, power of 250K and a spot size of 1 mm.

The microhardness of the weld has significantly increased compared with the substrate. XRD result analysis show that the weld has a microstructure consisting of Mg and $Mg_{17}Al_{12}$ indicating a composition of the melted layer with rapid solidification has been fabricated by laser surface melting.

From the in-situ microtensile test, the fracture point is at the substrate instead of it breaking at the laser welded boundaries or weld. The results showed that the laser weld is able to withstand tensile testing more than the substrate. The fractography showed that brittle was the mode of fracture. Both SEM and optical microscope in-situ tensile testing demonstrated that the weld was stronger than the substrate.

Chapter 8

Conclusions and Recommendations for further Researches

8.1 Conclusions

(1) The digitalized microtensile device is demonstrated to be an effective tool for in-situ observation of deformation and fracture process using small samples. Both optical microscope and SEM can be used to carry out the in-situ observation. SEM is generally a much better tool for observing the crack formation, but optical microscope can be used to observe slip lines more clearly than SEM because of the optical contrast.

(2) The testing results for notched specimens of AZ91D magnesium alloy with different notch angles show that the smaller the notch angle, the higher the fracture stress level. This is quite unexpected, because the decrease in notch angle increases in stress triaxiality and stress concentration, and therefore smaller notch angle usually tends to reduce the fracture stress. However, the phenomenon observed can be explained in terms of sampling process. Small notch angle leads to localized plastic deformation zone and therefore makes it less likely to “sample” large defects in the magnesium alloy.

(3) The in-situ observation revealed clearly the deformation and fracture processes in the magnesium alloys, AZ91D (In both as-cast and die-cast conditions) and AM50A (in as-cast conditions). First, plastic deformation occurred at the notch root, as can be seen from the dark slip lines in the optical images. In the 2nd stage, the plastic deformation and the progressively higher loading led to cracking of the brittle intermetallic particles $\text{Mg}_{17}\text{Al}_{12}$ (especially those located at the grain boundaries), because they were strong barriers to dislocation movement and thus caused build-up of local stress. In the final stage, when both applied stress increased and the crack length increased, brittle fracture propagation suddenly occurred when the Griffith equation was satisfied.

(4) The dynamic stress strain relation of magnesium alloy AM50A has been obtained using the Hopkinson bar apparatus. The strain rate ranges between 600 /s and 1300 /s. The magnesium alloy AM50A displays about 50 % higher in tensile stress at the strain rate of about 1300 /s than at static. Fast shearing is the fracture mode at the highest strain rates.

(5) As observed using the in-situ heating stage, the temperature 420 °C is critical for AZ91D magnesium alloys. Comparing normal tensile specimens and at 420 °C there is not much different in the mechanical strength, however for specimens heated to 450 °C, there is a great reduction in mechanical strength (about 50 %).

(6) Even though minor oxidation take place on the surface of the alloy before 420 °C but it does not affect the mechanical strength much. The voids left behind by the melted intermetallics ($Mg_{17}Al_{12}$) when heated above 420 °C provide an easy path for the fracture process thus lowering the mechanical strength. The preferential sites for oxide growth are the intermetallics and the intersection of grain boundaries.

(7) The laser melted layer in AZ91D magnesium alloys consists of the network dendrites and columnar dendrites are resulted from consuming convection and high cooling rate of the surface. The microhardness of the melted zone has significantly increased compared with the substrate. XRD result analysis show that the laser surface melting coating has a microstructure consisting of Mg and $Mg_{17}Al_{12}$ indicating a composition of the melted layer with rapid solidification has been fabricated by laser surface melting.

(8) Butt welds of AZ91D magnesium alloy were successfully produced using a Nd:YAG laser and with Argon as protective gas. The main weld defect was porosity in the fusion zone. Hot cracking was also observed. Optimum tensile properties were obtained for welds made with a welding speed of 10 mm/s, power of 250 W and a spot size of 1 mm.

(9) From the in-situ microtensile test, the fracture point is at the substrate instead of it breaking at the laser welded boundaries or weld. The results showed that the laser weld is able to withstand tensile testing more than the substrate. The fractography

showed that brittle was the mode of fracture. Both SEM and optical microscope in-situ tensile testing demonstrated that the weld was stronger than the substrate.

8.2 Recommendations for further Researches

- (1) The microtensile tester can be improved further. For example, it is possible to install a CCD camera or even a high-speed camera to record the whole deformation and fracture process continuously.
- (2) Further researches can be carried out using the system, for example, to study effect of strain rate on the fracture process. When the materials are heat-treated to produce different microstructures, the testing device can be used to understand how heat treatment can be utilized to improve properties of the materials and causes for the improvement.
- (3) Nd:YAG laser can be used to join dissimilar joints of aluminium and magnesium alloys. The fracture process of the weld can be tested on the in-situ Microtensile tested to observe the fracture process. In addition, focused ion beam is to be used to extract TEM samples from the tested samples for nanoscale analyses of the fracture processes.

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