Mechanical behaviour of magnesium nanocomposites at wide range of strain rates and temperatures

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**MECHANICAL BEHAVIOUR OF MAGNESIUM
NANOCOMPOSITES AT WIDE RANGE OF STRAIN
RATES AND TEMPERATURES**

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Table of Contents

Acknowledgments.....	i
Table of Contents	iii
List of Figures	vii
List of Tables	xii
Abstract	xiii
Chapter 1 Introduction	1
1.1 Background.....	1
1.1.1 Characteristics and structural applications of magnesium and magnesium alloys.....	1
1.1.2 Metal matrix composites.....	3
1.1.3 High strain rate behaviour of materials.....	5
1.1.4 High strain rate behaviour of materials under a wide range of temperatures	7
1.2 Objective and research scope.....	8
1.3 Thesis organization	9
Chapter 2 Literature review	10
2.1 Recent studies on magnesium matrix composites	10
2.1.1 Matrix.....	10
2.1.2 The reinforcement.....	11
2.1.3 The processing of magnesium matrix composites	13
2.1.4 Mechanical properties	15

Table of Contents

2.1.5 Dynamic behaviour of particle reinforced magnesium matrix composites..	24
2.2 Experimental techniques to study high strain rate behaviour	27
2.2.1 Historical background of the Hopkinson bar technique.....	28
2.2.2 Split Hopkinson Pressure Bar technique at low and elevated temperature..	30
2.2.3 Specimen geometry effects	32
2.2.4 Pulse shaper technique	34
2.3 Deformation of magnesium alloys and magnesium nanocomposites	35
2.3.1 Role of slip in deformation.....	35
2.3.2 Deformation by twinning	37
Chapter 3 Experimental techniques and data processing	40
3.1 Quasi-static experimental setup	40
3.2 The Split Hopkinson Pressure Bar	40
3.3 Theory of the Split Hopkinson Pressure Bar.....	43
3.3.1 One-dimensional stress wave analysis	43
3.3.2 Specimen stress	46
3.3.3 Specimen strain and strain rate.....	47
3.3.4 Summary of stress, strain and strain rate.....	49
3.4 Materials and specimen dimension	50
3.4.1 Materials fabrication	50
3.4.2 Specimen dimension and preparation	53
3.5 Pulse shaper.....	59
3.6 Dispersion of waves	64

Table of Contents

3.7 SHPB experiment at low/high temperature	65
3.7.1 Experimental setup of low/high temperature SHPB test	66
3.7.2 Temperature calibration	67
3.7.3 Modification of data-processing method with thermal gradient in Hopkinson bars	72
3.8 Data processing	73
3.8.1 SHPB signals calibration	73
3.8.2 Data acquisition	80
3.8.3 Experimental strain rate measurements	81
3.8.4 Data smoothing	82
3.9 Thermal softening effects	83
Chapter 4 Compressive mechanical behaviour of magnesium alloy AZ31B	86
4.1 Effect of strain rate.....	86
4.2 Effect of temperature	90
4.3 Strain hardening	94
4.4 Energy absorption capability	97
4.5 Microstructural analysis on strain rate effect.....	99
4.6 Constitutive modelling.....	101
Chapter 5 Compressive mechanical behaviour of 0.5 vol.% SiC _p /AZ31B.....	104
5.1 Effect of strain rate.....	104
5.2 Effect of temperature	107
5.3 Strain hardening	110

Table of Contents

5.4 Energy absorption capability.....	113
5.5 Constitutive modelling	114
Chapter 6 Compressive mechanical behaviour of 1.0 vol.% SiC _p /AZ31B.....	117
6.1 Effect of strain rate.....	117
6.2 Effect of temperature.....	121
6.3 Strain hardening	126
6.4 Energy absorption capability.....	129
6.5 Constitutive modelling	130
6.6 Effect of volume fraction of silicon carbide nanoparticles	132
Chapter 7 Conclusions and recommendations for future work.....	139
7.1 Conclusions	139
7.2 Recommendations for future work.....	142
Publications:	144
References	146

List of Figures

Figure 2-1: The process of (a) stir casting and [37] (b) squeeze casting[41]	14
Figure 2-2: A schematic diagram of the strain rate range and the techniques to develop them [127].....	28
Figure 2-3: Apparatus developed by Bertram Hopkinson [128]	28
Figure 2-4: A schematic diagram of the Kolsky Bar[129]	30
Figure 2-5: Schematic of slip systems in hexagonal magnesium crystal structure.....	36
Figure 2-6: A twinning transformation [194]	38
Figure 3-1: Schematic illustration of compressive split-Hopkinson pressure bar apparatus	43
Figure 3-2: Strain gauge data, after signal conditioning and amplification, from a compression split-Hopkinson pressure bar test of a magnesium alloy specimen	43
Figure 3-3: A differential element of the rod.....	44
Figure 3-4: A schematic diagram of the specimen under compressive loading	46
Figure 3-5: Schematic of experimental setup for ultrasonic vibration method [36].....	52
Figure 3-6: SEM micrographs of SiC _p /AZ31B nanocomposite (a) low magnification; (b) white spots are SiC nanoparticles.....	52
Figure 3-7: The same impact velocity (a) strain rate vs. time curve; (b) stress vs. strain curve.....	55
Figure 3-8: (a) Strain rate vs. time curve; (b) Stress-strain curve at the same strain rate (without filtering); (c) Hardening exponent n.....	57
Figure 3-9: Sample labelling for SHPB (left) and static (right) test	58
Figure 3-10: Marking sample for dimensioning	58
Figure 3-11: Measuring height of specimen	59
Figure 3-12: Measuring diameter of specimen	59

List of Figures

Figure 3-13: Incident and reflected strain pulses from oscilloscope for different pulse shapers of AZ91D	61
Figure 3-14: Incident and reflected strain pulses from oscilloscope for different pulse shapers of AZ31B.....	62
Figure 3-15: Strain gauge waveform on oscilloscope (dry run): comparison of different layers of pulse shaper	63
Figure 3-16: Sample of test results comparison	65
Figure 3-17: Schematic illustration of SHPB setup at (a) below zero temperature (b) elevated temperature	67
Figure 3-18: Thermocouple wires attached to specimen	68
Figure 3-19: AZ31B specimen temperature versus time	71
Figure 3-20: Schematic diagram of incident bar	75
Figure 3-21: Oscilloscope screenshot with waves captured.....	76
Figure 3-22: Schematic diagram of photodiode setup	77
Figure 3-23: Oscilloscope screenshot for measuring the travel time of the striker bar	78
Figure 3-24: ‘Bar together’ calibration test result.....	80
Figure 3-25: Average strain rate calculations during high strain rate testing	81
Figure 3-26: Engineering stress-time curve before and after filtering	82
Figure 3-27: True stress-true strain at $3300 \pm 300 \text{ s}^{-1}$ of AZ31B	84
Figure 3-28: True stress/Temperature variation of AZ31B for two strains at $3300 \pm 300 \text{ s}^{-1}$	85
Figure 4-1: Sample strain rates measured in SHPB tests on AZ31B	87
Figure 4-2: True stress-strain curves of AZ31B at different strain rates for different temperatures	88
Figure 4-3: Strain rate sensitivity of AZ31B.....	90

List of Figures

Figure 4-4: True stress-strain curves of AZ31B at different temperatures for different strain rates	91
Figure 4-5: The effect of temperature on the flow stress at 5% strain at all strain rates	92
Figure 4-6: The effect of temperature on failure strain at 0.001 s^{-1} and $3300 \pm 300\text{ s}^{-1}$.	92
Figure 4-7: Temperature sensitivity-true strain curves at different strain rates and temperatures	93
Figure 4-8: Effect of strain rate on the hardening behavior of AZ31B at $25\text{ }^{\circ}\text{C}$	95
Figure 4-9: Effect of temperature on the hardening behavior of AZ31B under various temperatures at 3300 s^{-1}	96
Figure 4-10: Energy absorption capability of AZ31B	99
Figure 4-11: Micrographs from Optical microscope (a) 10^{-3} s^{-1} (b) 700 s^{-1} (c) 1400 s^{-1} (d) 3300 s^{-1}	100
Figure 4-12: Scanning Electron Micrographs (a) 10^{-3} s^{-1} (b) 700 s^{-1} (c) 1400 s^{-1} (d) 3300 s^{-1}	101
Figure 4-13: Experimental and Johnson-Cook fitted curves at different strain rates and temperatures	103
Figure 5-1: Sample strain rates measured in SHPB tests on 0.5vol% $\text{SiC}_p/\text{AZ31B}$...	104
Figure 5-2: True stress-strain curves of 0.5vol% $\text{SiC}_p/\text{AZ31B}$ at different strain rates for different temperatures	105
Figure 5-3: Strain rate sensitivity of 0.5vol% $\text{SiC}_p/\text{AZ31B}$	107
Figure 5-4: True stress-strain curves of 0.5vol% $\text{SiC}_p/\text{AZ31B}$ at different temperatures for different strain rates.....	108
Figure 5-5: The effect of temperature on the flow stress at 5% strain at all strain rates	109
Figure 5-6: Temperature sensitivity-true strain curves of 0.5vol% $\text{SiC}_p/\text{AZ31B}$	110

List of Figures

Figure 5-7: Effect of strain rate on the hardening behavior 0.5vol% SiC _p /AZ31B at 25 °C.....	111
Figure 5-8: Effect of temperature on the hardening behavior of 0.5vol% SiC _p /AZ31B at 3300 s ⁻¹	112
Figure 5-9: Energy absorption capability of 0.5vol% SiC _p /AZ31B.....	114
Figure 5-10: Experimental and Johnson-Cook fitted curves at different strain rates and temperatures	116
Figure 6-1: Sample strain rates measured in SHPB tests on 1.0vol% SiC _p /AZ31B ...	118
Figure 6-2: True stress-strain curves of 1.0vol% SiC _p /AZ31B at different strain rates for different temperatures.....	119
Figure 6-3: Strain rate sensitivity of 1.0vol% SiC _p /AZ31B.....	121
Figure 6-4: True stress-strain curves of 1.0vol% SiC _p /AZ31B at different temperatures for different strain rates	122
Figure 6-5: The effect of temperature on the flow stress at 5% strain at all strain rates	123
Figure 6-6: The effect of temperature on failure strain at 0.001 s ⁻¹ and 3300±300 s ⁻¹	123
Figure 6-7: Temperature sensitivity-true strain curves at different strain rates and temperatures	124
Figure 6-8: The macroscopic failure of specimens at strain rates of (a) 0.001 s ⁻¹ (b) 3300 s ⁻¹	125
Figure 6-9: Fractograph of 1.0vol% SiC _p /AZ31B tested at 3300 s ⁻¹ under room temperature.....	125
Figure 6-10: Effect of strain rate on the hardening behavior of 1.0vol% SiC _p /AZ31B	126
Figure 6-11: Effect of temperature on the hardening behavior of 1.0vol% SiC _p /AZ31B under various temperatures at 3300 s ⁻¹	128

List of Figures

Figure 6-12: Energy absorption capability of 1.0vol% SiC _p /AZ31B	129
Figure 6-13: Experimental and Johnson-Cook fitted curves at different strain rates and temperatures	131
Figure 6-14: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at -30 °C.....	132
Figure 6-15: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 25 °C	133
Figure 6-16: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 100 °C	134
Figure 6-17: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 200 °C	135
Figure 6-18: Comparison of true stress-strain curves of AZ31B and AZ31B reinforced with 1.0vol.% SiC at 3300 s ⁻¹	136
Figure 6-19: Energy absorption of AZ31B and its nanocomposites at (a) 0.001/s and (b) 3300/s.....	137
Figure 6-20: Optical micrographs showing grain characteristics of (a) AZ31B (b) 1.0vol% SiC _p /AZ31B.....	138

List of Tables

Table 2-1: Ductility improvement results of different nanocomposites.	23
Table 2-2: Slip systems in HCP metals[183]	37
Table 3-1: Chemical composition of magnesium alloy AZ31B	51
Table 3-2: Solid cylindrical specimen dimensions for quasi-static compressive test...	53
Table 3-3: Specimen temperature after removal of thermocouple.....	69
Table 3-4: Temperatures of heated AZ31B specimen at 30s intervals	71
Table 3-5: Heater temperature and time required for 100 °C and 200 °C AZ31B specimen.....	72
Table 3-6: Temperature rise at 20% strain at $3300 \pm 300 \text{ s}^{-1}$ of AZ31B.....	84
Table 3-7: The reduction of flow stress at $\epsilon=20\%$	85
Table 4-1: Compressive strength properties of AZ31B at 25 °C	89
Table 4-2: Strain hardening exponent of AZ31B at 25 °C.....	95
Table 4-3: Strain hardening exponent of AZ31B at 3300 s^{-1}	97
Table 4-4: Parameters of the Johnson-Cook model for AZ31B.....	103
Table 5-1: Compressive strength properties of 0.5vol% SiC _p /AZ31B at 25 °C	106
Table 5-2: Strain hardening exponent of 0.5vol% SiC _p /AZ31B at 25 °C.....	111
Table 5-3: Strain hardening exponent of 0.5vol% SiC _p /AZ31B at 3300 s^{-1}	113
Table 5-4: Parameters of the Johnson-Cook model for 0.5vol% SiC _p /AZ31B.....	115
Table 6-1: Compressive strength properties of 1.0vol% SiC _p /AZ31B at 25 °C	119
Table 6-2: Strain hardening exponent of 1.0vol% SiC _p /AZ31B at 25 °C.....	126
Table 6-3: Strain hardening exponent of 1.0vol% SiC _p /AZ31B at 3300 s^{-1}	128
Table 6-4: Parameters of the Johnson-Cook model for 1.0vol% SiC _p /AZ31B.....	130

Abstract

As a result of environmental considerations, a key challenge for the automobile and aerospace industries is to reduce greenhouse gas emissions and to enhance fuel efficiency. This motivates the development of new lightweight alloys as replacement for steel and aluminium alloys. Magnesium and its alloys with low specific weight, high specific strength, vast resources, easy recyclability and biodegradation have attracted extensive interest in recent years. However, the low absolute strength and low ductility limit their usage. It has been found that the addition of stiffer and stronger second-phase reinforcements can enhance strength and ductility of magnesium alloys. Micro-sized reinforcements are effective in enhancing stiffness and strength but accompanied by an obvious decrease in ductility. Recent studies show that it is possible to obtain simultaneous enhancement in strength and ductility when ceramic nanoparticles are employed as reinforcements. The resulting nanocomposites have great potential to replace the existing metals and alloys for various automobile, aerospace and other applications.

There are many applications where materials are subjected to high strain rate loading. High strain rate deformation encounters in crash of automobiles or airplanes, where the structural material deforms under various strain rates. In defence and security sector, designing the bomb-proof shields and human protection against bullets and other penetrators need to consider high strain rate deformation of materials. In addition, high strain rate loading also arise in many aspects of everyday life. The dynamic response of a material is quite different from its static response. Limited data is available for high strain rate deformation of magnesium alloy and its nanocomposites, which makes it difficult for widespread usage of these lightweight materials.

In the present work, the compressive mechanical response of magnesium alloy AZ31B, AZ31B/0.5%SiC and AZ31B/1.0%SiC to quasi-static and dynamic loading over a wide range of temperature is investigated. The split Hopkinson pressure bar (SHPB) is used to study high strain rate behaviour of the materials in this work. The experimental investigation on the effect of specimen size and pulse shapers has been carried out to decide the optimal specimen dimension and the most suitable pulse shapers used in SHPB tests. The addition of silicon carbide nanoparticles can enhance the strength of the base alloy, while it slightly decreases the ductility of the material. It doesn't apply to all strain-rate and temperature conditions that higher volume fraction of nanoparticles results in higher strength. But this happens more at higher strain rate. Though ductility becomes worse after adding ceramic nanoparticles, the enhancement of energy absorption capability still exists especially at highest strain rate.

Chapter 1 Introduction

1.1 Background

With the increasing demand for economical use of scarce energy resources and ever-stricter control over greenhouse gas emissions, industries are searching for lighter yet stronger materials to replace steel, aluminium alloys and some plastic-based materials in structural applications. Magnesium is the lightest of all structural metals. Its specific strength and stiffness exceeds that of most commonly used metals and some plastic-based materials. Magnesium and magnesium-based materials attract more and more interest because of its gradually reducing cost.

1.1.1 Characteristics and structural applications of magnesium and magnesium alloys

Magnesium is the sixth most abundant element, constituting 2.7% of the earth's crust and 0.13% of the world's oceans. Although magnesium does not occur in its elemental form in nature, magnesium compounds can be found worldwide. The most common compounds are magnesite, dolomite, carnallite and seawater [1]. The total energy needed to produce magnesium from its various raw materials is much less than that of aluminum, zinc and even polymers, as long as the calculation is based on the volume.

Magnesium has a density of 1.74 g/cm^3 , which is approximately one-fourth the density of steel, two-thirds that of aluminum and slightly higher than that of reinforced plastics. The specific mechanical properties of magnesium can reach and even exceed the values of aluminum and steel. Moreover, most magnesium alloys show high impact resistance, very good machinability and processability. Because of these advantageous

properties, magnesium-based materials are found in all places where weight saving takes priority over other properties [2].

Magnesium and magnesium alloy have been successfully used in various structural applications due to their attractive properties. The extreme lightness makes them attractive in all parts that are moved or lifted during their manufacture or use. The low density of magnesium and magnesium alloys allows thicker sections to be used in parts, which eliminates the need for a large amount of stiffening and simplifies the parts and their processing. In addition, the low inertia resulting from the low density is especially advantageous in rapidly moving structures. While the most significant factor in the selection of magnesium and its alloys is light weight, there are other important properties for different uses, for example, elevated-temperature properties in aircraft and missiles. Historically, magnesium and magnesium alloys were mainly used for making military equipment, such as shelter frames, mortar bases, and missiles. They were used in aircraft and surface-vehicle frames, panels, floors, brackets and wheels as well as in engine components [1]. Nowadays, light metal automotive applications are the focus of interest due to the increasing cost of transportation and the necessity of reducing environmental pollution by lowering fuel consumption and exhaust gases. The driving force behind the development of magnesium alloys has shifted to automotive industry due to the possibility of mass-producing series parts at reasonable costs. Leading automobile makers such as Ford, Toyota, Audi, BMW, Hyundai and Mercedes-Benz have already used magnesium-based materials in their automotive parts. In the Toyota Lexus, Celica, Carina and Corolla, magnesium is used to make steering wheels [3]. In the Audi A4 and VW Passat, gearbox housing is made of magnesium [2]. In Hyundai Azera, magnesium is used in interior parts such as the seat frame, steering column housing, steering wheel and lock body [4]. In the Mercedes-

Benz SLK, the fuel tank cover is made of a magnesium alloy [5]. In the aerospace industry, weight reduction is also one of the most critical objectives as there is an increasing need for fuel efficiency and emission reduction. The total operating cost can be saved by reducing overall weight of the aircraft which results in fuel savings. Both civil and military aircraft have used magnesium-based materials. For example, magnesium alloys are used to make thrust reverser for Boeing 737, 747, 757 and 767. Military aircraft, such as F16, Tornado and Eurofighter Typhoon also benefit from the lightweight characteristics of magnesium-based materials for transmission casings. Besides, the widespread use of magnesium in aerospace applications is due to its high specific mechanical properties, ease of fabrication and capability to withstand (i) elevated temperature, (ii) exposure to ozone and (iii) bombardment of high-energy particles [5].

While high-pressure die casting is the dominant process for current magnesium automotive applications, wrought magnesium alloys are receiving more and more attention from academia and industries. Wrought magnesium products with improved mechanical and physical properties, crash performance and corrosion resistance are increasingly needed as the application of magnesium is expanding from interior components to more critical applications in chassis and body areas [6]. AZ31B with easy availability is one of the most commonly used magnesium alloys. Besides, AZ31B has better combination of strength and ductility than its respective group alloys such as AM50 and AZ60 which makes it potential for being used in structural applications.

1.1.2 Metal matrix composites

Metal composite materials have found application in many areas of daily life for quite some time but it is often not realized. For many researchers, the term metal matrix

composites is often equated to light metal matrix composites. The reinforcement of light metals can open up the possibility of application of these MMCs in areas where weight reduction has first priority. Historically, metal matrix composites (MMCs) were designed for use in aeronautic and space applications due to their lightness and high mechanical properties. The first commercial applications were at room temperature in the automotive industry for improving wear properties of car engines. Car makers such as Ford, Chrysler, Toyota and Lotus proposed SiCp/Aluminum brake or drum disks [7]. In traffic engineering, MMCs have also been used in fibre reinforced pistons and aluminium crank cases with strengthened cylinder surfaces as well as particle-strengthened brake disks [8].

1.1.2.1 Magnesium matrix composites

Although magnesium has become more and more attractive as an engineering material to replace conventional materials, the application of magnesium and magnesium alloys remain limited due to their insufficient strength, their low Young's modulus, the poor creep and abrasion resistance. However, these drawbacks can be partly or even completely overcome by a reinforcement of the matrix with an additional phase such as fibres or particles. Compared to use of long fibres, using short fibres or particles is more cost-effective and much less anisotropic. Moreover, particles as reinforcement components have the advantages of high availability, economic production, low price as well as a wide variety of adjustable material properties. The particles used as reinforcement in magnesium matrix composites are mostly ceramics and have their origin in the grinding industry. Nitrides, oxides, borides or carbides are normally used as ceramic particle reinforcements. The chemical reaction between reinforcement and matrix has to be taken into account while choosing the particles. Magnesium alloys generally shows chemical reactivity to most other materials while melting. Therefore,

the ceramic particles are often chemically attacked or even totally converted into a different phase. The particle reinforced magnesium matrix composites always show isotropic behaviour due to the homogeneous particle distribution. For a satisfactory distribution of ceramic particle reinforcement in the matrix, a short time of solidification and continuous stirring are required.

Of all the ceramic reinforcements, the most widely investigated and used reinforcing ceramics with pure magnesium and commercial grade magnesium alloys is the silicon carbide particulates. SiC particulates are thermodynamically stable in many molten Mg alloys. In addition, they have relatively good wettability with magnesium in comparison to the other ceramic reinforcements [9, 10].

1.1.3 High strain rate behaviour of materials

There are many applications in automobile and aerospace industry as well as in military application and daily life where materials are subjected to high strain rates. Impact conditions in automobiles and aerospace industry, armour penetration, rock blasting, shock and impact damage in daily life are characterized by high stresses and high strain rate loading. For example, high strain rate deformation happens in crash of automobiles and airspace, where the structural material of vehicle and aircraft deforms under various strain rates. In defence and security sector, human protection against bullets and other penetrators and design of the bomb-proof shield needs to consider the high strain rate deformation of materials. Dynamic fracture in nuclear pressure vessels and the mechanical responses of rocks and ores in blasting are other applications of high strain rates regime. In addition, high strain rate loading arise in many aspects of everyday life such as the impact of a golf ball and the dropping of a mobile. The high strain rate characterization of materials is not limited to the application mentioned

above but also important in studying the response of bones to impact and the response of biological tissues to dynamic pulses.

In studying the high strain rate behavior of materials, it is important to know the extent of the strain rate being developed during any situation. While considering the strain rate in mechanical testing, the dynamic loading region can be delineated as follows. (1) Creep rates (very low strain rates usually of the order of 10^{-8} to 10^{-4}). It is a time dependent deformation, which terminates eventually in fracture. (2) Quasi-static rates (low rates of strain, 10^{-4} to 10^{-1}). (3) Intermediate strain rates (10^{-1} to 10^2). (4) High strain rates (10^2 to 10^5) [11]. The knowledge of classification of strain rate under which the problem to be analyzed falls is necessary for accurate evaluation of the material response. Most of the situations encountered in daily life experience deformations in the time frame of the order of minutes to microseconds resulting in strain rates in the range between 10^{-4} and 10^8 s^{-1} . High strain rates are involved in most of the metal-working processes, such as extrusion (10^3 s^{-1}), machining (10^4 s^{-1}), explosion or metal forming (10^3 s^{-1}) [12]. Usually the strain rates encountered in crash applications are of the order of 10^2 s^{-1} to 10^3 s^{-1} , while higher strain rates are associated with localized deformation [13]. The falling of electronics devices on a hard surface may also experience the strain rate as high as 10^3 s^{-1} .

When the loading rate is high, the mechanical response of a material is quite different from its behavior under static loading. Such rate dependence of material behavior is observed for nearly all materials including metals, ceramics, glasses and polymers. In impact or dynamic loading, the sudden application of a force to material gives rise to two important effects, which dictate the material's response. These are the elastic and plastic stress wave propagation (inertia effect) and the changes in the inelastic mechanical properties of the materials (strain rate effect). When a material deforms in

dynamic state, these effects are very important as they have strong influence on the mechanical properties of the materials. The material behavior at high strain rates involves large deformations, localized heating of the material and failures through a variety of mechanisms that are usually absent in quasi-static loading.

1.1.4 High strain rate behaviour of materials under a wide range of temperatures

The temperature and strain rate are variables of fundamental importance in the mechanical response of materials, playing an important role on the deformation process. In several applications, like in machining operation, metal forming, high energy impact, or high energy deposition on metals, the materials are deformed at very high speed at high temperature condition[14]. In this case, the stress versus strain behavior comes as the result between the effects of hardening (due to strain and strain rate) and thermal softening.

Examples of materials serving under high or low temperatures can be easily found in industries. Due to the severe operating conditions encountered by many steel components during their service lives and the requirement for high-speed manufacturing, it is necessary to provide engineers with sufficient information to enable them to predict the high strain rate flow behavior of different steels under a wide range of temperatures[15]. In nuclear engineering applications, e.g., the pressure vessel of the advanced Neutron Source Research Reactor[16] or the heat exchange pipes in space nuclear power system radiators[17], 6061-T6 components are typically exposed to temperatures ranging from 100 to 300 °C and strain rates ranging from 1×10^2 to $5 \times 10^3 \text{ s}^{-1}$. A thorough understanding of the alloy's mechanical properties under elevated temperatures and strain rates is thus essential to ensure the safety and reliability of 6061-T6 structural components under such arduous operating conditions[18]. In automotive industry, vehicles are used under below-zero

temperature in some districts. Components like engine parts of airplanes may be also subjected at low temperature while airplanes are flying at high altitude.

1.2 Objective and research scope

Magnesium and its alloys have gained widespread attention in scientific research and commercial application as potential replacement of other materials for example steel and aluminium alloys. However, the poor ductility and low strength of magnesium alloys limit their usage. Recent studies show that when ceramic nano-particles (Al_2O_3 , Y_2O_3 , etc.) are incorporated into a magnesium matrix, it is possible to obtain simultaneous enhancement in strength and ductility [19, 20]. The resulting nanocomposites have great potential for usage in automobile and aircraft applications. Currently, investigations on these nanocomposites focus on their synthesis. Mechanical characterization of these materials are mainly performed under quasi-static loading, and studies on their dynamic behaviour in terms of stress-strain characteristics appear limited[21, 22]. The objective of the current research is to investigate the mechanical behavior of magnesium alloy AZ31b and its nanocomposites under wide range of strain rate and at below zero and elevated temperatures. The scopes of this study are as follows:

- To obtain stress-strain relation of magnesium alloy AZ31B and its nanocomposites under room temperature at quasi-static and high strain rates.
- To obtain stress-strain relation of magnesium alloy AZ31B and its nanocomposites under a wide range of temperatures at high strain rates.
- To study the strain-hardening, strain rate sensitivity, temperature sensitivity and energy absorption capability of magnesium nanocomposites.

- To investigate how the difference of volume fraction of silicon carbide nanoparticles in the composites affect the mechanical properties at high strain rates.
- To obtain Johnson-Cook model constants for magnesium alloy AZ31B and its nanocomposites.

1.3 Thesis organization

This report consists of seven chapters. A brief introduction of magnesium alloy, magnesium matrix composite and high strain rate behaviour of materials, objective and scopes of this work are presented in Chapter 1. A literature review of the split Hopkinson pressure bar, magnesium alloy and magnesium matrix composites is given in Chapter 2. The experimental setup, data analysis procedures and specimen preparation for this study are laid down in Chapter 3. Experimental results of AZ31B and its nanocomposites are discussed in Chapter 4, 5 and 6 respectively. The comparison between the materials investigated is presented in Chapter 6. Finally, Chapter 7 concludes the current research and makes some suggestions for future work.

Chapter 2 Literature review

The first part of this chapter gives a through literature review of light metal matrix composites especially their dynamic behaviors. A brief chronological development of the Hopkinson bar as an engineering tool is presented in the following parts.

2.1 Recent studies on magnesium matrix composites

Due to the increasing cost of transportation, light metals and light metal based composites are the focus of academia and industry interest. Particle reinforced light metals with their potential as high modulus and strength, low cost and easily fabricated materials are now being produced commercially. The physical and mechanical properties of these materials are dependent on several factors, e.g. type of matrix alloy, type of reinforcement, the processing method, shape, and amount and length scale of reinforcement. Different end properties resultant from different combination of these factors can serve different applications. As one of light metals, Al-based composites have been studied for several years due to their low density, wide alloy range, processing flexibility and heat treatment capability. Mg –based composites exhibit these features as well. Besides, Mg often achieves a larger property improvement with reinforcement because of lower density and elastic modulus.

2.1.1 Matrix

Magnesium is one of the lightweight metallic materials with the lowest density. It has been widely used as a matrix material because of high specific strength. In existing literature, not only pure magnesium but also commercial grade magnesium alloys are selected as the matrix materials. In terms of commercial grade magnesium alloys, AZ31 and AZ91 are two of the most commonly used matrix materials for the synthesis of magnesium matrix composites[5].

2.1.2 The reinforcement

For the synthesis of metal matrix composites, reinforcements of different types, shapes, amounts and length scales are selected to enhance the end properties of base materials.

2.1.2.1 Type and shape of the reinforcement

For magnesium matrix composites, the reinforcements intentionally incorporated into the matrix material can be classified into ceramic, metallic and intermetallic. The most commonly used type of reinforcements is ceramic due to its desirable properties. These properties include thermal stability, low density, and high levels of strength, hardness and elastic modulus. In addition, most ceramics are available as particles. Other shapes of reinforcements show some degree of anisotropic behaviours. A wide range of ceramic reinforcements have been investigated by researchers, such as zircon [23], boron carbide [24], mica [25], alumina [26], graphite [27], titanium diboride and silicon carbide [28]. While choosing reinforcement, there are some factors needed to be taken into consideration, namely

- i. The application: If the composite will be used in a structural application, a high modulus and strength, a low density and a good ductility are important. As angular particles can act as local stress raisers which reduce ductility, deciding particle shape is important as well.
- ii. The method of composite manufacture: While processing composites in the molten state, ceramic particles can spend considerable time in contact with the molten matrix. Whether the reinforcement is thermodynamically stable in the molten alloy matrix and the reaction between reinforcement and matrix should be considered. Reaction of the reinforcement can severely degrade the properties of the composite. The reinforcement has to be chosen after

considering the matrix alloy, the processing time and the processing temperature.

- iii. The cost: Particles are chosen as reinforcements mainly because of their low price. The particle reinforcement has to be readily available in the shape, size and quantities required at low cost, i.e. about 5USD/Kg.

With these considerations in mind, silicon carbide particulates have become the most widely investigated and used reinforcing ceramic. It is thermodynamically stable in many molten magnesium alloys and has relatively good wettability with magnesium, as compared to other ceramic reinforcements [9, 10].

2.1.2.2 Amount and length scale of the reinforcement

There is either an increasing or decreasing trend in the composite's properties with the increasing amount of reinforcements. Although the composite's strength and hardness values increase with the increasing amount of reinforcements, there is often a threshold amount whereby further addition of reinforcements will be detrimental to the mechanical properties of composites. The influence of the amount of reinforcements on the properties of a variety of magnesium-based composites has been investigated by several researchers [29-34].

The advancement of reinforcement fabrication technology has made different length scale reinforcements available including micrometre size, sub-micrometre size and nanosize. The research community and industry are thus motivated to adopt these length scale reinforcements in the fabrication of metal matrix composites. The influence of length scale of Al_2O_3 particulates on the properties of magnesium matrix was investigated by Hassan and his co-workers[35]. From the mechanical test results, it was found that the magnesium composites with the nanosized Al_2O_3 particles

yielded the best mechanical properties over their matrix magnesium, magnesium reinforced with micrometre-size and sub-micrometre-size particles. The superior specific strength values of the composite with nanosize particles make such composite systems most suitable for strength-based designs. In these designs, higher yield strength and tensile strength are required.

2.1.3 The processing of magnesium matrix composites

The reinforcing phases in magnesium matrix composites can be either fibres or particles in terms of the shape. In automotive applications, particulate-reinforced composites with relatively low material cost and suitability for automatic processing are preferable to fibre-reinforced composites[10]. Homogeneously distributing the particulate reinforcement is a key challenge in the processing of magnesium matrix composites.

Conventional processing methods used for aluminium matrix composites can be applied to fabricate magnesium matrix composites because of the similar melting temperatures of aluminium and magnesium alloys. The most commonly used conventional methods are stir casting, powder metallurgy and squeeze casting[10]. Stir casting is a mostly used technique of fabricating magnesium matrix composites which are reinforced by micro ceramic particles [36]. It is a cost-effective and an easily adaptable method. The reinforcing particles in powder form are mixed into molten magnesium by mechanical stirring. This method is more suitable for processing composites with up to 30% volume percentage of reinforcement [37, 38]. It has been used to produce magnesium composites with various matrix compositions, such as Z6[39], ZC63, ZC71 and AZ91[10]. Squeeze casting method is making the reinforcement into a preform and place them into a casting mould. Then the molten magnesium alloy matrix is poured into the mould and solidified under high pressure.

Squeeze casting method has the advantage of allowing for incorporating higher volume percentages (up to 50%) of reinforcement into the matrix[40].

Figure 2-1: The process of (a) stir casting and [37] (b) squeeze casting[41]

Pure magnesium matrix [42] and AZ91[43] matrix composites have been processed with this method. Various magnesium matrix composites have been produced through powder metallurgy [44-47]. During the powder metallurgical process, reinforcement powders and matrix materials are mixed, pressed, degased and sintered at a certain temperature in a vacuum or under a controlled atmosphere. This processing method allows for the incorporation of a relatively high volume fraction of reinforcement[10].

These conventional methods have been widely used to produce micro ceramic particle reinforced magnesium matrix composites. However, it is quite challenging for conventional methods to achieve uniform distribution and dispersion of nanoparticles in metal melts. Compared to micro particles, nanoparticles have larger surface-to-volume ratio and lower wettability in metal melts, which easily induce clustering and agglomeration [36]. More efficient and expensive fabrication methods are thus

necessary. Among these methods, there are disintegrated melt deposition method [48-50], ball-milling method [46, 51], semisolid process [52] and so on. Recently, ultrasonic vibration method was found to be efficient in dispersing nanoparticles in metal melts [53-55]. It was reported that high-intensity ultrasonic waves with micro scale transient cavitation and acoustic streaming could produce transient micro “hot spots” with temperatures of 5000 °C, pressures above 1000 atm and heating and cooling rates above 10^{10} K/s [54, 56]. Nanoparticle clusters can be broken and the particle surfaced can be cleaned due to the strong impact coupled with local high temperatures. Ultrasonic dispersion has been successfully used to distribute nano-sized particles into magnesium alloy melts [54, 57, 58].

2.1.4 Mechanical properties

Reaching an optimum of both mechanical and physical properties is the major attraction of developing metal matrix composites (MMCs). For instance, the increase in Young’s modulus and strength as well as lowering of creep rate can be expected by reinforcing magnesium alloys [59]. However, the improvement in some properties resulting from adding reinforcement phases is normally at the cost of some other properties [10]. Thus, the reduction in these properties and the additional cost should be considered while there are improvements in certain mechanical properties. Generally speaking, the development of metal matrix composites is to replace monolithic alloys, for example aluminium alloys and magnesium alloys. Metal matrix composites can be used for improving already existing parts or extending the field of application of matrix alloys [60]. Some properties listed below can be improved by the development of MMCs [61-63]:

- Young’s modulus
- Yield strength and tensile strength

- Strength at elevated temperature
- Creep
- Fatigue resistance
- Ductility
- Coefficient of thermal expansion

2.1.4.1 Elastic modulus

With the addition of a reinforcement phase, the elastic modulus is always significantly increased. Within a certain range, the elastic modulus of the magnesium matrix composite increases linearly with the increase of the reinforcement volume fraction in the composite[64]. The quantitative value of the elastic modulus somewhat depends on the method of measurement. Static values may be dependent on whether the measurements are made in tension or compression[65]. Most of these difficulties are because of the presence of thermal residual stresses caused by differences in the coefficient of thermal expansion between the ceramic particles and the matrix. Comparison between measuring values and theoretical expectations is also somewhat difficult because of uncertainty in the appropriate value for the elastic modulus of the reinforcement.

The rule of mixtures expression is

$$E_C = V_P E_P + V_m E_m \quad (2-1)$$

Where E_c , E_p , E_m are the elastic moduli of the composite, particle and matrix, respectively, and V_p , V_m are the volume fractions of the particle and matrix. The elastic modulus calculated by using this expression considerably overestimates the measuring one. As the rule of mixtures expression is most appropriate for continuous

reinforcement, it has been modified for discontinuous reinforcement in the Halpin-Tsai equation[66]

$$E_C = \frac{E_m(1+2sqV_P)}{1-qV_P} \quad (2-2)$$

where

$$q = \frac{\frac{E_P}{E_m} - 1}{\left(\frac{E_P}{E_m}\right) + 2s} \quad (2-3)$$

and s is the particle aspect ratio. The Halpin-Tsai equation gives a good representation of the results. The Eshelby equivalent inclusion method[67] is also used to calculate the elastic modulus and it is also in good agreement with the data. The volume fraction of reinforcement is the dominant factor in controlling Young's modulus, while the particle distribution doesn't affect considerably on the elastic modulus. The variations in the type and shape of the reinforcement can be accounted for by the different expressions.

The improvement in the elastic modulus is retained at higher temperatures. The temperature dependence of the composite Young's modulus reflects that of the matrix material.

2.1.4.2 Strength

Both the tensile strength and yield strength of the magnesium alloys are generally increased with the addition of a reinforcement phase. Within a certain range, the yield strength increases linearly with the increase in the volume fraction of the reinforcement[64]. McDanel [68] carried out the first extensive study on the strength of discontinuously reinforced Al alloys. SiC whisker and particle reinforcement in several different alloy matrices were investigated. Up to 60% increase in yield and

ultimate tensile strengths were reported, depending on the type of matrix alloy, the matrix alloy temper and the volume fraction of reinforcement. These findings were generally confirmed by subsequent work. However, there is an extremely large degree of scatter in experimental results, presumably reflecting differences in processing and the quality of the material. Humphreys [69] pointed out a more fundamental difficulty when considering the yield stress. Since composites work hardens extremely rapidly at low strains, the yield strength may not be equivalent to a conventional yield stress as the 0.2% proof stress. Fortunately, composites are now commercially available which will meet a strength specification[9].

Work hardening, particle strengthening, load transfer and grain refinement of the matrix alloy by the reinforcements are the key strengthening mechanisms in magnesium composites. Work hardening takes place when the composite is strained. A high density of dislocation in the matrix around the reinforcement is usually generated by the strain mismatch between the matrix and the reinforcement, thus strengthening the material. The dispersion of fine and hard particles in the matrix strengthens the material as it blocks the motion of dislocations drastically. Load transfer is another important strengthening mechanism. If the bonding between the reinforcement and the matrix is strong enough, the applied stress will be transferred from the soft matrix to the hard particle phases. The relatively soft matrix is protected by the secondary hard phases with much higher strength. The strength of magnesium is highly sensitive to its grain size. Therefore, grain refinement contributes to the great strength at room temperature for both pure magnesium and magnesium alloys matrix composite[10].

The strength of particle reinforced metal matrix composites has been extensively modelled mathematically [70-72]. The continuum shear lag model [73] gives the composite strength as

$$\sigma_c = (V_p \sigma_m s / 4) + V_m \sigma_m \quad (2-4)$$

where σ_m is the yield stress of the matrix, and s the aspect ratio. Equation above underestimates the strength for the typical aspect ratio used in particle MMCs which are in the 1-5:1 range. A better agreement can be achieved if the equation is modified to allow for end loading effects, suggested by Nardone and Prewo[74].

The cracking of the reinforcement can help to relax the stress built up in the magnesium composite when the composite is under an applied load[75]. It was observed in a fractured SiC/Mg composite that the SiC particulate fracture was the predominant form of localized damage under tensile loading[76]. The fracture of the composite was dominated by the cracking of the reinforcing particles. Crack propagation through the alloy matrix between particle clusters resulted in the final fracture. The size of ceramic particle reinforcements is important. A more cooperated deformation within the matrix can be produced by a finer secondary phase, which makes it easier for the stress built up around the small particles to be relaxed[77, 78]. Besides, a finer secondary phase offers more heterogeneous nucleation sites for the solidification of the magnesium matrix, which results in a finer matrix grain size. However, there isn't a monotonic increase in the strength of the magnesium matrix composite with decreasing particle size, as the particle strengthening relies greatly on the stress built up around the particles by lattice distortion.

Debonding along the reinforcement /matrix interfaces may also relax the stress built up in the metal matrix composite[79]. The composite may fail prematurely at the interfaces when the matrix and reinforcement interface is a relatively weak region of the material. In such case, the material's strength can be reduced due to the addition of any secondary hard phase. A research focusing on an AZ91 magnesium alloy

reinforced with 15 vol% TiB_2 , TiC , TiN , SiC , Al_2O_3 and AlN detected such an occurrence with AlN reinforcement[80]. The ultimate tensile strengths and hardness of these composites were compared. It was shown that the decrease in the tensile strength of AZ91, which was attributed to different powder size distribution, excessive chemical reactions and wetting conditions, was caused by the addition of AlN . The relative strength of the matrix and interface can also depend on temperature[81].

There are also crack initiation and propagation in the reinforcement-free areas in the matrix observed in composite materials. The matrix plays a more important role in determining the deformation and fracture behaviour of the composite in this situation. A comparison between pure magnesium and magnesium alloy AZ91 reinforced with 10-20 vol% of Al_2O_3 fibers showed that the AZ91 matrix composite always exhibited a higher tensile yield strength, but the pure magnesium matrix composite had a higher degree of strength improvement. This phenomenon was ascribed to the higher mechanical properties of AZ91 compared with pure magnesium[82]. Accordingly, processes which modify the microstructure of the matrix can influence the mechanical properties of the bulk composite[83].

2.1.4.3 Ductility

The limited ductility is the major limitation in the mechanical properties of composites. The tensile elongation decreases rapidly with the addition of reinforcing particles. It is shown in recent work that composite failure is associated with void formation and particle cracking in the matrix within clusters of particles[75, 84]. For coarser particles, particle fracture is more prevalent. This can be explained from two points of view. The coarser particles have a higher possibility of containing fracture initiating defects. In addition, the larger particle can be loaded more by conventional fibre loading and end loading mechanisms.

Magnesium possesses only three independent slip systems due to its hexagonal close-packed (HCP) structure, which results in a limited ductility. The hard secondary phases in magnesium matrix composites have a two-fold effect. First, these phases can reduce the ductility of magnesium matrix composites by preventing plastic deformation. On the other hand, these particles can improve ductility of composites by producing a grain refining effect. Generally, the net effect of the hard particles is reducing ductility. This happens in both fibre-reinforced[85] and particle reinforced composites[86]. The reduced ductility is evident in the interactions between the reinforcement and the dislocations. It is obvious that the ductility of the composite material is reduced by the resistance to the dislocation motion. The superplastic behaviour of a fine-grained ($\sim 2\mu\text{m}$) WE43magnesium alloy containing spherical precipitates ($\sim 200\text{nm}$) was examined. It was observed that dislocations interact with the particles within the grains. The normalized strain rate of the particle-strengthened WE43 alloy was about 50 times lower than that of WE43 alloy. The existence of intragranular particles seems to diminish the superplastic flow[87].

However, the high brittleness of secondary particles does not necessarily lead to a low ductility of the composite with hard phases. In fact, superplasticity can result from grain refinement by the secondary phase, even with highly brittle secondary phases. For example, a total creep elongation of 200-350% was achieved in a ZK60A magnesium alloy reinforced with 17 vol% of SiC particles at the temperature range of 350-500 °C[88]. A lower temperature superplasticity with an elongation of 300% for the same material was obtained at 175-202 °C due to the refined grains[89]. This low-temperature superplasticity is important for magnesium matrix composite in practical industrial applications.

In recent years, researchers[19, 20, 32, 90-96] have also found that a simultaneous increase in the overall composite's strength and ductility can be achieved with the addition of nanosize discontinuous reinforcements (such as SiC, Al₂O₃, Y₂O₃, ZrO, MgO and CNTs) in certain amounts into magnesium-based matrix. It was noted that the composite synthesis method also influenced the composite's ductility value. The improvement in ductility or failure strain of some magnesium-based composites is listed in Table 2-1. The activation of a nonbasal slip system may be the cause of the increment in ductility of the magnesium composites [97, 98]. The fractographs of the composites exhibited improved ductility. A mixed mode type of failure with increased levels of plastic deformation was shown in fractographs as well. As reported in other studies [20, 99], the presence of the dispersed nanoparticles in the brittle magnesium matrix serves to

- i. change the local effective stress state from plane strain to plane stress in the neighborhood of crack tip,
- ii. dissipate the stress concentration that would otherwise exist at the crack front and
- iii. provide sites where cleavage cracks are opened ahead of the advancing crack front.

Considering all these factors, to maximum ductility for a particular volume fraction, the magnesium composite should have

- i. uniform particle distribution
- ii. a fine particle size
- iii. a high interfacial strength
- iv. control of particle shape

v. a ductile matrix

Table 2-1: Ductility improvement results of different nanocomposites.

Reinforcement			Type of Matrix	Fabrication Method	Increase in Ductility or Failure Strain
Type	Size	Amount			
SiC[95]	50nm	1.5 wt%	Mg4Zn	Melt casting (ultrasonic cavitation)	135% (D)
SiC[94]	50nm	0.5 wt%	Pure Mg	Melt casting (ultrasonic cavitation)	11% (D)
SiC[100]	50nm	1.5 wt%	Mg6Zn	Melt casting (ultrasonic cavitation)	3% (D)
SiC[101]	45-55nm	1.84 wt%	Pure Mg	Powder metallurgy (microwave sintering)	31% (D)
Al ₂ O ₃ [19, 35]	50nm	1.1 vol.%	Pure Mg	Disintegrated melt deposition	89% (D)
Al ₂ O ₃ [32]	50nm	1.5 vol.%	AZ31B	Disintegrated melt deposition	427% (FS)
Al ₂ O ₃ [32]	50nm	1.5 vol.%	AZ31	Disintegrated melt deposition	115% (FS)
Al ₂ O ₃ [90, 102]	50nm	1.1 vol.%	Pure Mg	Powder metallurgy (conventional sintering)	64% (D)
Y ₂ O ₃ [103]	29nm	0.6 wt%	Pure Mg	Powder metallurgy (conventional sintering)	258% (D)
Y ₂ O ₃ [104]	30-50nm	2.0 wt%	Pure Mg	Powder metallurgy (microwave sintering)	15% (D)
Y ₂ O ₃ [105]	30-50nm	0.7 vol.%	Pure Mg	Powder metallurgy (microwave sintering)	61% (FS)
ZrO ₂ [96]	29-68nm	0.66 vol.%	Pure Mg	Powder metallurgy (conventional sintering)	171% (D)
CNT[106]	40-70nm	1.0 vol.%	AZ31	Disintegrated melt deposition	68% (FS)

D: ductility; FS: failure strain.

2.1.5 Dynamic behaviour of particle reinforced magnesium matrix composites

Particle reinforced lightweight metal matrix composite have been applied in aerospace, military and civil engineering such as lightweight armours and protective coatings in defence due to their practical advantages, e.g. high specific strength, high specific stiffness, ease of fabrication, isotropy and low cost [107-111]. Therefore, a significant scientific effort has been devoted to investigating quasi-static mechanical behaviours of particle reinforced MMCs. As these advanced materials gain wider application, structural components made of these lightweight metal matrix composites will inevitably be subjected to dynamic loadings as well. For example, the impact of foreign objects (such as, birds and meteoroids) on structural members in aerospace applications can result in such loading conditions. When these materials are intentionally or inadvertently subjected to dynamic loadings, the effect of strain rate cannot be ignored. An understanding of how these materials respond to high strain rate loadings is thus necessary and can be used to dictate the potential uses[112].

The research on dynamic properties of various light metal matrix composites under high strain rate has been fruitful during the last decades in order to meet the demand in industries. The effect of strain rate on the mechanical response of aluminium matrices reinforced with ceramic particles has been studied for many years. The literature on rate dependent mechanical properties of aluminium matrix composites shows that strain rate sensitivity of composites is similar to that of the matrices. Wang et al. [109] studied the dynamic tension properties of aluminium matrix composites with 10 vol.% SiC particulate at high strain rates. Vaidya et al. [113] studied the mechanical response of 15 nominal volume percent SiC particle and B₄C particle reinforced Al alloy 6061 under dynamic loading conditions, using split Hopkinson pressure bar. It was found that the different strength of the reinforcing particles and bonding between

the particles and the matrix alloy resulted in different mechanical behaviour of the two composites. Compared to the unreinforced alloy, the stiffness and yield strength of both composites were enhanced at either low or high strain rates. Vaidya et al. [112] also investigated the strain rate effect on the strain-hardening behaviour of an Al-Li alloy (8090) and the same volume percent of SiC particle-reinforced 8090 composite. The strain-hardening doubled with increasing strain rate from 10^{-3} s^{-1} to 6500 s^{-1} . The strain hardening coefficient of the alloy was not affected by the presence of the SiC particles in the matrix. The effect of strain rate on the ultimate tensile strength, the hardening modulus and the failure strain of other aluminium alloys such as 2124, 6061, Al-5Cu and Al-Li alloys reinforced by silicon carbide particulates were investigated by some other researchers [114]. They found that the strain rate sensitivity of these materials was determined by the rate-dependent properties of their matrices. Zhang et al. [115] tested the monolithic 6092 aluminium alloy and its composites with 5 and 15 vol.% boron carbide particulate over a wide strain rate range of 10^{-4} to 10^4 s^{-1} . The strength of these metal matrix composites increase with increasing volume percentage of particulate reinforcement. An unreinforced 6061-T6 aluminium alloy and this alloy reinforced with 20 vol.% alumina particles were studied over a range of strain rates (10^{-4} to $6 \times 10^5 \text{ s}^{-1}$) by Yadav et al. [116]. An increased strength and the same strain hardening as the matrix for the composite was observed. Perng et al. [117] also studied high strain rate behaviours of 6061 aluminium alloy matrix composites with the Al_2O_3 particle and indicated that the strength of the composites increased with increasing strain rates, while the elongation of the composites was insensitive to strain rate. The results of an investigation on the effect of strain rates and strain hardening on the mechanical properties of aluminium matrix composites with 10-20 vol. % Al_2O_3 showed that the composites exhibited high strain rate sensitivity and increasing the particle content led to an increase in strain rate sensitivity [110]. In addition, research

on aluminium matrix composites reinforced with high content particles was compared to that on aluminium matrix composites with low content particles by Tan et al. [118]. On the other hand, finite element method has been also used to extensively study the effects of particle shape, particle distribution, residual stress and volume fraction on flow stress of these composites[119-121].

Given the light weight and the high performance of magnesium alloys reinforced with ceramic particles, the study of the stress-strain response of these materials at high strain rates and even different temperatures has aroused increasing interest in recent years. Essa et al.[122] has studied the effect of the strain rate and temperature on the mechanical behaviour of a magnesium matrix composite. The reinforcement was 10 μm SiC particles in a 12vol.%. The quasi-static and impact testing was performed in the temperature range from -150 to 200 $^{\circ}\text{C}$. It was found that magnesium-based composites exhibit higher specific properties than unreinforced magnesium alloys up to 700 s^{-1} strain rate. Essa et al. [123] also conducted tensile split Hopkinson bar testing on another magnesium alloy which was ZC71 reinforced with the same reinforcement and the same volume fraction. The stress-strain response of both the base and the reinforced materials were found to be dependent on the presence of reinforcement, the applied strain rate and the test temperature. It is shown in recent studies that a simultaneous enhancement of magnesium matrix composites 'strength and ductility can be achieved when the reinforcement particle size decreases to nano length-scale. However, studies on mechanical characterization of these materials under dynamic loading are limited[21]. Habibi et al. [124] investigated the effect of strain rate change on tensile and compressive response of hybrid Mg/Al-CNT nanocomposites. An improvement in mechanical response of Mg in both quasi-static and dynamic regimes was observed due to the presence of hybrid Al-CNT particles. Chen et al. [125]

fabricated AZ31 Mg alloy composites with different volume fractions of 50 nm Al_2O_3 nanoparticles and conducted tensile testing on these materials at strain rates spanning 10^{-4} - 10^3 s^{-1} . A significantly increased ductility and yield stress of the nanocomposites were observed for both low and high strain rates, indicating the positive effect of nanoparticles. Effect of ceramic nanoparticles on dynamic mechanical properties of magnesium-based composites has also been investigated by Shen et al.[126] using split Hopkinson pressure bar. The reinforcement they used was 20nm β -phase SiC in the volume fraction of 5 to 15%. The yield and ultimate tensile strength increased with reinforcement volume percentage up to 10%.

2.2 Experimental techniques to study high strain rate behaviour

In order to study the strain rate behaviour of the materials, the testing techniques should be capable of producing a high enough strain rate and also should be able to provide a well-defined material response. Standard testing techniques being used for quasi-static and low strain rate tests are not capable to perform high strain rate tests due to presence of waves and effects of inertia involved at high strain rate. It is important to develop a testing technique able to achieve a well-defined and relatively simple state of stress (i.e. one-dimensional state of stress) in the gauge length that is also comparable to that from other tests. There are a variety of the techniques used to study the mechanical behaviour of the materials at high strain rates. These include drop-weight machines, Split Hopkinson bars, Taylor impact and plate impact. A schematic diagram of the strain rate range and techniques developed for them is shown in Figure 2-2 [127]. The Split Hopkinson Bars is one of the most widely used experimental techniques to study the behaviour of materials at high strain rate under compressive, tensile and torsional loading.

Figure 2-2: A schematic diagram of the strain rate range and the techniques to develop them [127]

2.2.1 Historical background of the Hopkinson bar technique

John Hopkinson who is the founder of Hopkinson pressure bar came up with an idea of using a pressure bar to evaluate the material response to impulse loads in 1872. In 1914, Bertram

Figure 2-3: Apparatus developed by Bertram Hopkinson [128]

Hopkinson [128] developed a technique for determining the peak pressure obtained during an impact which leads scientists to develop later versions of Hopkinson bar. The device consisted of a long cylindrical bar with a smaller cylindrical specimen and

a ballistic pendulum. Based on this pioneering work, Kolsky [129] added a second pressure bar to Hopkinson's design in 1949. Instead of attaching the specimen at the far end of the bar, Kolsky sandwiched the specimen between two bars. The strain data at both the input and output interfaces of the test specimens can be collected after the addition of the second bar. From the collected data, Kolsky was able to derive the expression for calculating specimen stress, strain and strain rate. The method was known as 'Split Hopkinson Pressure Bar' which became one of the preferred methods for testing materials at strain rates from 10^2 s^{-1} to 10^4 s^{-1} . The concept of using electrical condensers units to measure the displacements of the surface of the pressure bar caused by the wave propagation was introduced by Davies[130]. The usage of condensers in measuring the strain enhanced the accuracy of the Hopkinson's original concept. Significant improvement to strain measurement came in mid 1950's to early 1960's with the use of strain gauges [131-133]. Hauser and Simmons[133] used strain gauges in their study of static and dynamic loading of iron and mild steel at elevated temperatures. It was found that the use of strain gauges significantly increased the accuracy, repeatability and resolution of the data. With the development of the high-speed computer data acquisition in 1970s, the split Hopkinson pressure bar underwent the most significant improvements. The digital storage oscilloscope and high bandwidth signal analysers have enabled researchers to collect more highly resolved data with higher precision.

Figure 2-4: A schematic diagram of the Kolsky Bar[129]

2.2.2 Split Hopkinson Pressure Bar technique at low and elevated temperature

In a wide range of engineering applications, it is necessary to predict evolution of the material response during an impact loading under variable temperature. It is then required to define correctly the strain rate and temperature dependence of the flow stress. The split Hopkinson pressure bar system (SHPBs) has mostly been used to study the plastic flow stress of various engineering materials that undergo large strains at high strain rates. The need for extra computations and the requirement for additional equipment may be the reasons for the limitation of the SHPBs in use at low and elevated temperature[134]. In systems of SHPB currently in use, the standard cylindrical specimen is in contact with the pressure bars and heating or cooling it creates temperature changes in the bars, which leads to changes in their mechanical characteristics, creating errors in the obtained results. On the other hand, the fact that the bars are very long means that it is not practical to heat or cool[135].

There are two general methods for the compression SHPB in low or high temperature experiments. In one the specimen and small portions of the pressure bars adjacent to the specimen are heated [136-139]. Other systems are employed to preheat or cool the

specimen, while the incident and transmission bars are not in contact with the specimen [140-144]. Heating or cooling the specimen while in contact with bars results in heating or cooling the ends of the incident and transmission bars. A temperature gradient is thus established in the two pressure bars. If the temperature gradient is large enough that it can't be ignored, it becomes necessary to correct the measured signals to compensate for the temperature-dependent wave velocity and Young's modulus. One of the approaches to moderate these problems is to use maraging steel or Inconel alloy bars in testing up to 600 °C as they retain their elastic properties and mechanical impedance with temperature up to this temperature[134, 145]. Another method is to use short ceramic bars which have their impedance matched to the pressure bars. Another problem with heating or cooling specimen alone and then inserting the pressure bars in contact with the specimen is the contact time. As soon as the pressure bars come in contact with the heated or cooled specimen, the specimen loses its temperature by heat conduction to or from the bars. To moderate this problem, controlling the time between bringing the pressure bars in contact with the specimen and the arrival of the pulse is critical [140-142, 144].

For high-temperature tests in the compression SHPB, a heating system is needed to preheat the specimen. In order to perform high temperature tests, various heating devices have been designed. The examples are furnace[140], furnace and vacuum chamber[137], tubular-shaped furnace[141], specially built vacuum furnace[146], small resistive tube furnace[142], electric combustion tube furnace[138], IR image furnace[134], electric-resistance-type oven[139], induction furnace[136], high frequency induction coil[143], in situ induction coil[147] and infrared spot heater[144].

To reach low temperatures in the specimen a climatic chamber was specially designed to be coupled to the Hopkinson bar device [148, 149]. Thus a portion of the input and

output bars remained inside it. The chamber has two circular holes through which the bars pass during the test. Liquid nitrogen flows inside the chamber, controlled by an electro-valve connected to a low temperature thermocouple placed close to the specimen. A fan inside the chamber ensures a uniform distribution of temperature. The specimen was cooled during 20 min before testing to reach the thermal equilibrium with the environment.

2.2.3 Specimen geometry effects

The general theory of the Hopkinson bar testing technique has been established well for decades. However, there are still some issues associated with Hopkinson bars needed to be solved for more reliable results. Among them, specimen geometry has been found to be very influential on the stress-strain behaviour of the materials in Hopkinson bar testing due to friction and inertia. Specimen diameter, specimen thickness, specimen gauge length and specimen length to diameter ratio have been investigated by several authors.

The influence of the specimen length-to-diameter ratio l/d was studied by Lindholm[132]. l/d ratio was found to have small effect on stress at small strains and have almost no effect at large strains. Higher strain rate and higher stress can be achieved while using smaller diameter specimens. Yeakley and Lindholm [150] found that varying either l/d ratio or length of the specimen within reasonable bounds had no significant effect on stress-strain curves obtained. If inertial or frictional effects are significant, specimen dimensions will be influential. It was found by Davies and Hunter [151] in an experimental technique that the inertial effects may be ignored when the slenderness ratio l/d was equal to $\sqrt{3/4\nu_s}$, where ν_s is Poisson's ratio of the specimen. Yokoyama and Mayama [152] also did a detailed work in this regard. The cylindrical compression specimens made from AL6061-T6 were tested at strain rates

above 1000 s^{-1} . In this work, the optimum specimen slenderness ratio was found to be between 0.50 and 0.75 when diameter ratio of specimen and the bar was 0.5.

The effect of specimen size on stress-strain behaviour was investigated by Gunsekera, Havranek and Littlejohn[153]. They found that an increase in specimen diameter gave an overestimation of the deformation stresses because of more barrelling. By conducting some simulated experiments, Buchar, Bilek and Dusek [154]found that increasing the length of specimens gave a better approximation to the actual mechanical behaviour of the materials. Rodriguez et al. [155]used specimen diameter of 21.6, 14, 10 and 7 mm to analyse the diameter effect while keeping l/d ratio fixed at 0.5 under an impact velocity of 15-16 m/s. The results show there are larger inaccuracies in the stress-strain curve although a larger plastic strain is reached for smaller diameters specimens. In addition, the strain rate is more uniform for smaller diameter and strain rate increases as the diameter decreases. Gorham [156] analysed how specimen size affected the critical strain rate for copper. It was found larger specimens were associated with low values of critical strain rate and the vice versa. Meyer, Herzig and Kruger [157]studied the effect of specimen size on flow stresses under compressive loading over a wide range of strain rates. The specimens were made of Ti-6-22-22S and aluminium 7075 T6. Larger specimens with same slenderness ratio display lower stresses and for the same strain rate larger specimens lead to less plastic deformation. Li et al. [158]measured the compressive behaviour of AA2024 alloy specimens with various aspect ratio l/d where d=3.5 mm. They found the effect of specimen geometry was in the same order of magnitude as strain rate sensitivity of AA2024 alloys.

2.2.4 Pulse shaper technique

In order to facilitate the dynamic stress equilibrium and determine material behaviour at a constant strain rate, it is necessary to use some pulse shaping techniques in SHPB testing[159, 160]. Moreover, the impact of the striker on the incident bar results in a rectangular-shaped incident pulse. The shape of the incident pulse is suitable for testing perfectly plastic materials, while it is not quite proper for materials with considerable work hardening[161]. In addition, the pulse shaping technique also physically filters out the high frequency components of the waves[162]. There are four pulse-shaping techniques that have so far been developed to adjust the profile of the incident wave, i.e. pulse shaper technique[163], dual-pulse shaper technique[164], three-bar technique[165] and conical striker bar technique[166].

Among these pulse-shaping methods, using pulse shapers is simpler and has been widely used to shape the incident pulse. The materials used to make pulse shapers are usually stainless steel[165], copper[163, 167, 168], plastic[169], polymer[170], Plexiglas[171], brass[159] and paper[172]. The typical pulse shaper is a thin disk with a diameter of 2 to 3 mm and a thickness of 0.5 to 1 mm[173]. The presence of a pulse shaper turns the shape of incident wave from rectangular to trapezoidal as it increases the rise time of incident wave. The necessities and advantages of shaping the incident pulse have been discussed by Franz et al.[159] and Follansbee[160]. The pulse shaper they used was in a diameter slightly larger than that of bars and a thickness of 0.1-2.0mm. The pulse shaper was placed between the striker and the incident bar. They found that using pulse shapers minimized the dispersion effects and helped to achieve dynamic stress equilibrium in the specimen. In order to determine the proper dimensions of the pulse shaper under different test conditions, Vecchio and Jiang [174] have used a high-strength, high work-hardening rate material to fabricate pulse

shapers and tested a lot of specimens within a wide range of strength and work-hardening rate. In addition, Li and Xu [175] have investigated the pulse shaper diameter for testing a special concrete in split Hopkinson bar technique. They found that a certain diameter of pulse shaper can only offer one optimum nearly constant strain rate. Furthermore, several researchers [176-180] have employed pulse shapers with different materials and dimensions in SHPB test to determine dynamic behaviour of different materials. Besides experimental methods, Naghdabadi et al. [181] also performed simulations in LS-DYNA software to study parameters affecting the incident pulse shape. The pulse shape used was a thin copper disk.

2.3 Deformation of magnesium alloys and magnesium nanocomposites

Slip and twinning are two basic deformation modes which have been identified during the plastic deformation of crystalline solids. To identify whether slip or twinning will be the predominant mechanism for deformation will depend on which mechanism requires the lowest stress[182]. This stress is not constant for a given material, but varies with strain rate, temperature, the distribution of nanoparticles and loading direction of the crystal[183]. Hexagonal crystals such as magnesium have a limited number of slip systems[184]. Thus, twinning becomes an important deformation mechanism as it can provide additional slip systems for deformation[185].

2.3.1 Role of slip in deformation

Although the slip mechanisms are the same in single and polycrystalline materials, their stress strain behaviour differs substantially[186]. The displacements across grain boundaries must be matched so that the grains can deform in concert. Slip will then spread from grain to grain. The yielding takes place progressively in polycrystalline materials, which results in an absence of a sharp yield point on the stress-strain curve.

The yield strength is therefore higher in polycrystalline materials than if the material is just a single grain. Both slip plane and slip direction are needed to define the slip system.

Slip in hexagonal close-packed (HCP) metals is restricted to only a few active slip systems which generally led to brittleness in magnesium[187]. Slip occurs on the basal, prism and pyramidal planes in magnesium alloys as shown in Figure 2-5 [188]. The deformation mode in any slip system is controlled by the local stress upon the slip system. When an external force F , is applied to a crystal, a shear stress can be resolved onto the given plane in a given direction. Slip occurs when the resolved shear stress on the slip plane reaches the critical resolved shear stress (CRSS) for that particular system.

Table 2-2 shows all available slip systems in HCP metals [183]. However, their activation depends on the given set of applied conditions, i.e. temperature, strain rate etc.

Figure 2-5: Schematic of slip systems in hexagonal magnesium crystal structure

Table 2-2: Slip systems in HCP metals[183]

Structure	Slip Plane	Slip Direction	Number of non-parallel planes	Slip directions per plane	Number of geometrical slip systems	Number of independent slip systems
HCP	{0001} Basal	$\langle 11\bar{2}0 \rangle$	1	3	3	2
	{1010} Prismatic	$\langle 11\bar{2}0 \rangle$	3	1	3	2
	{1011} Pyramidal	$\langle 11\bar{2}0 \rangle$	6	1	6	4

It has been observed that magnesium slips readily on the basal plane at ambient temperatures [182, 189, 190]. Pyramidal slip cannot be activated even at 200 °C [191] but they can be activated at temperature above 225 °C [192]. The activation of pyramidal slip systems is relatively difficult at room temperature due to larger burger vectors and small pyramidal plane distance. Increasing the temperature will ease the activation process of pyramidal slip system [193]. At higher temperature of more than 250 °C, slips occur on the pyramidal and prismatic slip planes [184]. These additional slip planes explain the increasing ductility at elevated temperature.

2.3.2 Deformation by twinning

In addition to slip, twinning is another mechanism of deformation at room temperature. Twinning is a plastic deformation process that occurs when a crystal is subjected to a sufficiently large shearing force. Twinning is a sudden localized shear process that involves a certain volume within a crystal. It is shown in Figure 2-6 that twinning has taken place along the plane K_1 and the twinning direction η_1 [194]. Twin initiation or growth occurs when the macroscopic stress across the twin plane resolved in the

direction of the shear reaches a critical value[192]. The onset of twinning occurs when three conditions have been fulfilled simultaneously:

- i. The critical resolved shear stress(CRSS) of a twin system exceeds than that of any other slip system.
- ii. The CRSS satisfies the sense of a twin shear.
- iii. The ratio of the CRSS of a twin system is greater than that of any other slip system.

Figure 2-6: A twinning transformation [194]

Two important twinning systems could accommodate the plastic deformation in magnesium crystals which are “tension/extension twinning” $\{1012\}\langle 1120 \rangle$ system and “contraction twinning” system $\{1011+1012\}\langle 1120 \rangle$ [195]. “Tension/extension twinning” in magnesium can be activated by a tensile stress parallel to the c-axis (perpendicular to the basal plane) when the c/a ratio is less than 1.73[182]. Magnesium is almost ideally hexagonal close packed and has a c/a ratio of 1.628. Similar to tension twinning, the contraction twinning can only be activated by a compression stress parallel to the c-axis when the c/a ratio is greater than 1.73 [182, 196, 197].

Twinning is much more operative in compression than in tension, which has been confirmed by optical microscopy [182].

It has been observed that deformation by twinning on pyramidal planes occur at ambient temperatures [192]. Twinning becomes a more dominant mode of plastic deformation as temperature decreases [182]. Due to the limited slip system at room temperature in HCP magnesium alloys, twinning has been theoretically and experimentally proven to be the dominant model that is responsible for plastic deformation [189]. If the stress required for twinning rises less rapidly with decreasing temperature than the flow stress for slip, a transition to twinning will be expected. There is low mobility of lattice dislocations at low temperatures, which inhibits slip from occurring. The stress required for twinning varies slowly with temperature as twinning is not a thermally activated process. Under impact loading conditions, HCP magnesium metals deform solely by twinning [185, 192]. Research show that when the strain rate is relatively low (496/s-964/s), the microstructure is predominated by high density of twinning, while the volume fraction of twin decreases as the strain rate increases to 2120/s [196]. However, experimental findings for the past forty years have shown that high strain rate is accompanied by an increase in propensity of twinning [185].

Chapter 3 Experimental techniques and data processing

The strain rate and temperature dependence of AZ31B and its nanocomposites were studied by performing quasi-static and high strain rate compression experiments. Universal testing machine and split Hopkinson pressure bar were used in this work.

3.1 Quasi-static experimental setup

Quasi-static compression tests were conducted to compare with the dynamic tests at a strain rate of 0.001 s^{-1} . Quasi-static compression tests at room temperature ($25 \text{ }^{\circ}\text{C}$) were carried out using INSTRON-5500 machine with maximum load of 150 KN. High temperature compression tests at $100 \text{ }^{\circ}\text{C}$ and $200 \text{ }^{\circ}\text{C}$ were carried out using INSTRON-5569 machine with a close chamber. The chamber was heated by a built-in heater and the temperature in the chamber was set and controlled by a temperature control unit with an accuracy of $\pm 1 \text{ }^{\circ}\text{C}$. The maximum load allowed on this machine is 50KN. Specimen displacement was measured by a video extensometer, while specimen temperature was measured by a thermocouple. The heating temperature was set twenty Celsius degree higher than the target one. It was observed that after about 15 minutes the desired temperature can be achieved and remain stable.

3.2 The Split Hopkinson Pressure Bar

Due to the complexity of data analysis, the Split Hopkinson Pressure Bar has not been standardized[198]. There are usually five components used in a typical compressive Split Hopkinson Pressure Bar[199]:

- i. a compressive gas launcher/gun tube or alternative propulsion device[200],

- ii. three long symmetrical cylinder bars with a uniform cross section and usually made from the same materials,
- iii. bearing and alignment fixtures/supporters to allow the bars to move freely,
- iv. a pair of strain gauges mounted on both the incident and transmitted bars, and
- v. associated instrumentation and data acquisition system to control, record and analyze the strain wave data in the bars[201].

All bars used in the SHPB apparatus are usually made from the same material, such as AISI-SAE 4140 steel[202], maraging steel[203, 204], aluminum[205-207], titanium[208], magnesium[209], or polymeric materials[210-212]. The selection of the bar material is dependent on the specimen material. The general rule for selecting bar materials is that the bar can remain elastic during SHPB testing, i.e. the yield strength of the bar is higher than the maximum compressive strength of specimen at the corresponding strain rate. The wave impedance properties which are related to the bar sensitivity for the strain measurement should be also taken into consideration.

To ensure the one-dimensional stress wave in the bars, the cross section of the bars should be uniform for the entire length with a straight neutral axis. Moreover, both end faces of the bars should be perpendicular to the central axis of the bar. The end faces need to be machined with high accuracy. Song et al. [213] suggested the straightness of the bar was 0.05mm per 305mm length.

The specimen material also decides the bar diameter. Generally, 10 to 25 mm is selected[162]. A larger bar diameter is selected for a certain size of specimen in order to obtain the bulk properties. In contrast, smaller diameter has also been used to achieve a high strain rate by Kamler et al.[214].

The length to diameter ratio of the bar depends on test validity, strain rate and strain level of specimens. As for the first factor, one-dimensional wave propagation requires a length-to-diameter (L/D) ratio of at least 10. In order to allow separation of the incident and reflected waves which occur in the incident bar, the L/D ratio of the bar should exceed 20 as strain gauges are usually mounted in the middle of the bar. The second factor decides the bar diameter. By using a smaller diameter, the length of the specimen can be shortened to achieve a higher strain rate. The last factor determines the bar length. An L/D ratio of 100 or more may be required for a strain of more than 30% which also depends on the specimen size. Thus, the bar length-to-diameter ratio is commonly in the range of 20 to 100 based on all three factors above[215].

The SHPB apparatus used in this study consists of a striker bar, an incident bar, a transmitted bar and an absorbing bar at the end as shown in Figure 3-1. A gas gun is used to drive the striker bar. Two diametrically opposing strain gauges are mounted in the middle of the incident and the transmitted bar to monitor the strain on respective bars. The cylindrical specimen is placed between the incident and the transmitted bar. In this study, all bars are made of spring steel 65Si2MnWA and have a diameter of 12.7 mm. The length of the incident, transmitted, and absorbing bars is 1 m each and that of the striker bar is 300 mm. 4-layer A4 paper was used as a pulse shaper. The signals measured by strain gauges are amplified 500 times by DC-92D dynamic strain meters, and recorded in a DLM2024 oscilloscope. A typical Oscilloscope trace obtained from Split Hopkinson Pressure Bar test of magnesium alloy specimen is shown in Figure 3-2.

Figure 3-1: Schematic illustration of compressive split-Hopkinson pressure bar apparatus

Figure 3-2: Strain gauge data, after signal conditioning and amplification, from a compression split-Hopkinson pressure bar test of a magnesium alloy specimen

3.3 Theory of the Split Hopkinson Pressure Bar

3.3.1 One-dimensional stress wave analysis

The SHPB procedure is based on one-dimensional stress wave propagation theory. One-dimensional theory is based on the mechanics of materials method and explains

the propagation of a longitudinal wave along an infinitesimally thin elastic straight rod.

The basic assumptions of this theory are:

- i. The material is linear elastic
- ii. Only axial displacement occurs and it is uniform throughout the cross-section
- iii. Uni-axial stress exists along the whole rod
- iv. The stress and strain fields in the specimen are uniform in its axial direction and the specimen inertia effect and the friction effects in compression are negligible.

In the SHPB experiment, the rod is subjected to an impact on its longitudinal axis. Therefore a differential element 'dx' of a finite long rod of cross sectional area A_0 , density ρ and Young's modulus E , is taken into study. Figure 3-3 shows the differential element of the rod along with the coordinates taken for the analysis.

Figure 3-3: A differential element of the rod

Using the Hook's law, the forces in the element resisting compressive loading can be expressed in terms of the displacement u ,

$$F_x = A_0 E \frac{\partial u}{\partial x} \quad (3-1)$$

Force acting, on the differential element 'dx' when subjected to uni-axial compressive stress, are shown in Figure 3-3. Assuming perfectly elastic conditions in the pressure bar, the compressive force for F_1 and F_2 can be expressed as:

$$F_1 = A_o E \frac{\partial u_1}{\partial x} \quad (3-2)$$

$$F_2 = A_o E \frac{\partial u_2}{\partial x} \quad (3-3)$$

The forces on the element shown in Figure 3-3 are given by the addition of forces acting on the element $\sum F = ma$.

$$A_o E \frac{\partial u_1}{\partial x} - A_o E \frac{\partial u_2}{\partial x} = A_o dx \rho \frac{\partial^2 u_1}{\partial t^2} \quad (3-4)$$

Where ρ is the density of the pressure bar material and $\frac{\partial^2 u_1}{\partial t^2}$ is the particle acceleration within the bar. Other than the uniaxial deformation assumptions made earlier, it is also assumed that the bar material is homogeneous across the cross section. Therefore, Equation 3.4 can be simplified in to the equation of motion.

$$c_o^2 \left(\frac{\partial u_1}{\partial x} - \frac{\partial u_2}{\partial x} \right) = \frac{\partial^2 u_1}{\partial t^2} dx \quad (3-5)$$

In Equation 3.5, c_o is the longitudinal wave velocity in the differential element, calculated from equation

$$c_o = \sqrt{E/\rho} \quad (3-6)$$

Using momentum conservation, u_2 can be expressed in terms of u_1 :

$$u_2 = u_1 + \frac{\partial u_1}{\partial x} dx \quad (3-7)$$

Differentiating Equation 3.7 gives the rate of change at interface 2 in terms of displacement:

$$\frac{\partial u_2}{\partial x} = \frac{\partial u_1}{\partial x} + \frac{\partial^2 u_1}{\partial x^2} dx \quad (3-8)$$

By substituting Equation 3.8 in Equation 3.5, the equation of motion for bar reduces to

$$c_0^2 \frac{\partial^2 u_1}{\partial x^2} = \frac{\partial^2 u_1}{\partial t^2} \quad (3-9)$$

Equation 3.9 is the simplified equation of motion for the single differential element ‘dx’ of homogeneous, uniaxially deforming cylindrical rod and will help in simplifying the specimen stress, strain and strain rate.

3.3.2 Specimen stress

The average stress of the specimen can be expressed in terms of the forces acting on it.

A schematic drawing of the specimen is shown in Figure 3-4.

Figure 3-4: A schematic diagram of the specimen under compressive loading

The pressure bars exert forces on the specimen surface from both sides therefore the average force acting on the specimen is given by

$$F_{ave}(t) = \frac{F_1(t) + F_2(t)}{2} \quad (3-10)$$

Hence, the average stress on the specimen is

$$\sigma_{ave}(t) = \frac{F_{ave}(t)}{A_s} \quad (3-11)$$

Where A_s is the cross sectional area of the specimen and is given by the following equation

$$A_s = \frac{\pi D_s^2}{4} \quad (3-12)$$

When the specimen is in dynamic equilibrium, forces at the ends of the pressure bars can be expressed in terms of incident and reflected pressure bar strains

$$\mathbf{F}_1(t) = EA_o[\boldsymbol{\varepsilon}_I(t) + \boldsymbol{\varepsilon}_R(t)] \quad (3-13)$$

$$\mathbf{F}_2(t) = EA_o\boldsymbol{\varepsilon}_T(t) \quad (3-14)$$

where A_o is the cross sectional area of the bars. Substituting Equation 3.10, 3.13 and 3.14 into Equation 3.11, the average stress on the specimen in terms of the pressure bar strains is expressed as

$$\sigma_{ave}(t) = E \frac{A_o}{2A_s} [\boldsymbol{\varepsilon}_I(t) + \boldsymbol{\varepsilon}_R(t) + \boldsymbol{\varepsilon}_T(t)] \quad (3-15)$$

It is assumed that the specimen is in force equilibrium and the specimen is deforming uniformly. A simplification can be made equating the forces on each side of the specimen (i.e., $F_1=F_2$). Comparing Equation 3.13 and 3.14, we can obtain

$$\boldsymbol{\varepsilon}_I(t) + \boldsymbol{\varepsilon}_R(t) = \boldsymbol{\varepsilon}_T(t) \quad (3-16)$$

Therefore, the expression for the average specimen stress given in Equation 3.15 can be reduced to the following equation

$$\sigma_{ave}(t) = E \frac{A_o}{A_s} \boldsymbol{\varepsilon}_T(t) \quad (3-17)$$

From Equation 3.17, it can be deduced that the specimen stress is proportional to the amplitude of the strain transmitted through the specimen into the transmitted bar.

3.3.3 Specimen strain and strain rate

In order to derive the equations for the strain rate and the strain, the equation of motion (wave equation) is needed:

$$c_o^2 \frac{\partial^2 u_1}{\partial x^2} = \frac{\partial^2 u_1}{\partial t^2} \quad (3-18)$$

From elementary wave theory, it is known that the solution to the wave equation can be written as:

$$\mathbf{u} = \mathbf{f}(x - c_o t) + \mathbf{g}(x + c_o t) = \mathbf{u}_I + \mathbf{u}_R = \mathbf{u}_1 \quad (3-19)$$

for the incident bar, where f and g are functions describing the incident and reflected wave shapes, and c_o is the longitudinal wave speed in the pressure bars as mentioned in the last section.

By definition, the 1-D strain is given by

$$\epsilon = \frac{\partial u}{\partial x} \quad (3-20)$$

Therefore, differentiating Equation 3.19 with respect to x , the strain in the incident rod is given by

$$\epsilon = f' + g' = \epsilon_I + \epsilon_R \quad (3-21)$$

Differentiating Equation 3.19 with respect to time and using equation 3.21 gives:

$$\dot{u}_1 = c_o(-f' + g') = c_o(-\epsilon_I + \epsilon_R) \quad (3-22)$$

for the incident bar.

The time derivative of the displacement in the transmitted bar, $u_2 = h(x - c_o t)$, yields:

$$\dot{u}_2 = -c_o \epsilon_t \quad (3-23)$$

in the transmitted bar.

Equation 3.22 and 3.23 are true everywhere, including at the ends of the pressure bars.

The strain rate in the test specimen is:

$$\dot{\epsilon}_s = \frac{\dot{u}_1 - \dot{u}_2}{l_s} \quad (3-24)$$

Where l_s is the instantaneous length of the specimen, and $\dot{u}_1$ and $\dot{u}_2$ are the velocities at the incident bar-specimen and specimen-transmitted bar interface, respectively.

Substituting Equation 3.22 and 3.23 into 3.24 gives:

$$\dot{\epsilon}_s = \frac{c_o}{l_s} (-\epsilon_I + \epsilon_R + \epsilon_T) \quad (3-25)$$

Substituting Equation 3.16 into 3.25 yields:

$$\dot{\epsilon}_s = \frac{2c_o \epsilon_R}{l_s} \quad (3-26)$$

The specimen strain can be obtained by integrating Equation 3.26

$$\epsilon_s(t) = \frac{2c_o}{l_s} \int_0^t \epsilon_R(t) dt \quad (3-27)$$

Equation 3.26 and 3.27 form the basis for calculation of the specimen strain rate and strain.

3.3.4 Summary of stress, strain and strain rate

When the specimen is deforming uniformly, the strain rate within the specimen is directly proportional to the amplitude of the reflected pulse. Likewise, the stress within the specimen is directly proportional to the amplitude of the transmitted pulse. The strain, strain rate and average stress in the specimen are calculated by the following well known Hopkinson equations derived in previous sections.

$$\begin{cases} \dot{\epsilon}_s(t) = \frac{d\epsilon_s}{dt} = \frac{2c_o}{l_s} \epsilon_R(t) \\ \epsilon_s(t) = \frac{2c_o}{l_s} \int_0^t \epsilon_R(t) dt \\ \sigma_{ave}(t) = E \frac{A_o}{A_s} \epsilon_T(t) \end{cases}$$

The Split Hopkinson Pressure Bar (SHPB) equations are valid only with certain assumptions:

- 1) One dimensional stress
 - The bars remain elastic and straight throughout the test.
 - The elastic wave propagation in the bars is linear.
- 2) Force balance

- All stresses in the specimen are in equilibrium throughout the test.
 - Deformation in the specimen is homogeneous.
- 3) Stresses and velocities at the ends of the specimen are propagated into the bars in an un-dispersed manner.
 - 4) Stress and strain along the specimen are uniform.

The first assumption must be enforced due to the fact that the Split Hopkinson Bar is governed by the elastic wave equation. The second assumption indicates that the specimen should be short enough to achieve the equilibrium and the constant pressure across the specimen. The third assumption should exist so that the strain calculated at the strain gauge is similar to the actual strain at the interface. The last assumption is due to the fact that the wave propagation time along the specimen is very short as compared to the time of the test and many reflections of wave can take place within the specimen.

3.4 Materials and specimen dimension

3.4.1 Materials fabrication

Of various designations used for metals, standards published by American Society for Testing and Materials (ASTM) are the most widely recognized. In ASTM designation, a four-part letter-number system is expressed as follows:

- 1) The first two letters indicate the main alloying elements in an order of reducing content, where: A-aluminium, B-bismuth, C-copper, D-cadmium, E-rare earth metals, F-iron, 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.
- 2) The second part usually consisting of two numbers represents the weight percentage of the alloying elements respectively.

- 3) A letter following the numeric part indicates the revision or modification of the same alloy (A, B, C...).
- 4) A letter followed by a number indicates the condition or temper, where: F-As fabricated, O-Annealed or wrought products, H-Strain hardened, T- Heat treated other than F, O and H.

The matrix material used in this work is as-cast AZ31B alloy manufactured by JIANGXI Royal Metals Co.China in the form of ingot. AZ31B indicates magnesium alloy with roughly 3% weight aluminium and 1% zinc and the alloy is the B modification. The chemical composition of it is given in Table 3-1 .

Table 3-1: Chemical composition of magnesium alloy AZ31B

Element	Mg	Al	Zn	Mn	Fe	Cu	Si	Ni	Ca
wt%	Balance	2.98	0.99	0.5	0.0026	0.0005	0.019	0.0005	0.001

SiC nanoparticles with an average diameter of 60 nm were selected as the reinforcement. The volume fractions of SiC particles were 0.5 and 1.0 vol.%, respectively. The SiCp/AZ31B composites were fabricated by ultrasonic vibration in Harbin Institute of Technology, which was described by Nie et al.[36]. Figure 3-5 shows the setup for ultrasonic vibration. The transducer has a maximum power of 2 kW and frequency of about 20 kHz. Briefly, the fabrication procedures were as follows: (1) melting of 1 kg AZ31B alloy in a mild steel crucible under an atmosphere of inert gas containing CO₂ and SF₆; (2) submergence of the aluminum foil containing SiC nanoparticles beneath the surface of the melt once the melt temperature reaches 700 °C; (3) ultrasonic process of the melt by using an ultrasonic probe at 480W power level for 20 minutes under a controlled temperature of the melt at about 700 °C (4) elevation of

the melt to 720 °C pouring temperature (5) cast and solidification of the nanocomposite into a mould (450 °C) under 100MPa pressure. It can be noted that the ultrasonic vibration was only used for dispersing nanoparticles in molten AZ31B, not for solidifying AZ31B melts. SiC nanoparticles are almost distributed uniformly in the composites as shown in Figure 3-6[36].

Figure 3-5: Schematic of experimental setup for ultrasonic vibration method [36]

Figure 3-6: SEM micrographs of SiC_p/AZ31B nanocomposite (a) low magnification; (b) white spots are SiC nanoparticles

3.4.2 Specimen dimension and preparation

3.4.2.1 Quasi-static compressive test specimen dimension

In this present study the specimen dimension for quasi-static compressive tests were processed according to ASTM standard E9-89a [216]. The dimensions of specimens in solid cylindrical form for quasi-static compressive tests on AZ31B and its nanocomposites are shown in Table 3-2. The specimens were manufactured from AZ31B, AZ31B/0.5%SiC and AZ31B/1.0%SiC ingots.

Table 3-2: Solid cylindrical specimen dimensions for quasi-static compressive test

Material	Length(mm)	Diameter(mm)	Approx L/D Ratio
AZ31B	16.00±0.1	8.00±0.1	2.0
AZ31B/0.5 vol.%SiC	16.00±0.1	8.00±0.1	2.0
AZ31B/1.0 vol.%SiC	16.00±0.1	8.00±0.1	2.0

3.4.2.2 Compressive SHPB test specimen dimension

The rapid particle accelerations imposed during high strain-rate testing cause longitudinal and radial inertia, which can influence the measured stress-strain behaviour[160]. Corrections have been derived for the errors due to both longitudinal and radial inertia[151]. This analysis states that:

$$\sigma(t) = \sigma_m(t) + \rho_s \left(\frac{l_s^2}{6} - v_s \frac{d^2}{8} \right) \frac{\delta^2 \epsilon(t)}{\delta t^2} \quad (3-28)$$

where l_s is the specimen length, d is the specimen diameter, ρ_s is the specimen density, v_s is the Poisson's ratio of the specimen and σ_m is the measured stress. This expression predicts that errors are minimized when the strain rate is held constant or when the term inside the brackets is set to zero by choosing specimen dimensions as

$$\frac{l_s}{d} = \sqrt{\frac{3v_s}{4}} \quad (3-29)$$

As materials tested by SHPB are magnesium alloy AZ31B and its nanocomposites in this study, the Poisson's ratio is equal to 0.35. According to Equation 3.29, the optimum sample aspect ratio l/d is 0.5 to minimize the effects of inertia. Further work by Gorham[217], Bertholf and Karnes[218] confirmed these results. It can be noted that this aspect ratio is less than that determined to be the most favourable for the minimization of errors due to friction in ASTM E9, which specifies that $1.5 < l/d < 2.0$ [160]. However, samples with an aspect ratio of 0.5 are not expected to introduce any serious errors because the total strain in a split Hopkinson pressure bar is typically limited to approximately 25% to reduce the area mismatch between the specimen and the pressure bar. Thus, all specimens processed for SHPB testing have the aspect ratio of 0.5 in this work.

In addition to specimen aspect ratio, the detailed specimen dimension and bar to specimen diameter ratio have influence on the mechanical behaviours measured in SHPB. Specimens made of magnesium alloy AZ31B were manufactured in three different dimensions with the same aspect ratio of 0.5. Split Hopkinson pressure bar testing were performed on these specimens which had diameters of 6, 8, 10 mm and lengths of 3, 4, 5 mm, respectively. The ratio D_s/D_b between specimens and Hopkinson bar diameter was thus 0.47, 0.63 and 0.79 respectively. The stress-strain curves for different sizes at nearly the same strain rate are plotted and discussed below. The effect of the specimen dimension on the specimen strain rate is analyzed at same striking velocity and same strain rate. For each kind of dimension, 4 to 5 tests were performed at each strain rate to get at least three valid tests. The results presented in this study are the average values of these repeating tests. To smooth the pulses, 8-layer papers were used as pulse shapers and a low-pass filter was applied to the raw data to remove noise usually associated with high strain rate tests.

Figure 3-7(a) presents the strain rate versus time curves for three different specimen sizes at same impact velocity ($=11.42\text{m/s}$). During early deformation, the strain rate increases rapidly, attains a uniform value for most of the test duration and then decreases to zero. The average strain rates are shown in the figure. At the same impact velocity, higher strain rates were obtained for smaller size specimens and the initial non-uniform deformation region was relatively less for smaller specimen indicating that the smaller specimens attain equilibrium earlier. It is also observed that the strain rate is relatively uniform for smaller specimens compared with larger size specimens. Stress-strain curves for the same impact velocity are plotted in Figure 3-7(b). Higher stresses were obtained for smaller-dimension specimens. It is interesting to note that the yield strength of the specimen with diameter of 10mm and length of 8mm is much lower than the specimen with diameter of 6mm indicating more energy absorption during deformation before plastic deformation starts. The yield stress of 6mm-diameter specimen is almost twice than that of 10mm-diameter specimen.

Figure 3-7: The same impact velocity (a) strain rate vs. time curve; (b) stress vs. strain curve

In Figure 3-7, strain rates are not the same therefore the effect of strain rate is also contributing to the results. To eliminate the strain rate effects, specimens are deformed at same strain rate. Figure 3-8(b) shows the stress-strain relationship for three sizes at the same strain rate of about $1500 \pm 50 \text{ s}^{-1}$. It is observed that the smaller specimens show higher stresses. An increase of about 30% yield stress is noticed for the 6 mm diameter specimen as compared with the 10mm-diameter specimen. Some oscillations are present in the stress-strain curve (without filtering) for smaller-dimension specimens. However, strain did not show a regular trend. It might be due to the reason that the specimens with 8 and 10 mm diameter are not broken. Oscillations in the stress-strain curve for 6 mm specimen might be due to smaller ratio (0.47) of $D_{\text{specimen}}/D_{\text{bar}}$. The hardening exponent n was obtained from $\lg \sigma \sim \lg \epsilon$ curve. Deformed at the same strain rate, the alloy shows higher hardening behaviour for larger size specimen which can be seen from Figure 3-8(c). Higher hardening behaviour means more ability for the material to strengthen during deformation.

Figure 3-8: (a) Strain rate vs. time curve; (b) Stress-strain curve at the same strain rate (without filtering); (c) Hardening exponent n

In conclusion, optimum specimen geometries for compression SHPB tests were chosen as diameter=8mm and thickness=4mm for all samples tested in this work after considering the above factors.

3.4.2.3 Specimen preparation

In order to make the ends of specimens to be flat and perpendicular to the sides for perfect contact with the bars' ends, wire electrical discharge machining (EDM) was used to cut the rods into required cylindrical specimens. Due to the machine's constraint, the axial faces of the specimens may not be smooth and flat. As the axial faces of the specimens may not be smooth and flat because of the machine's constraint, the 800-Grit sandpaper is used for grinding. Acetone was used to clean the specimens'

ends after grinding. Specimens' diameter and thickness were measured by using a digital Vernier calliper. The procedures of measurement of samples are as follows:

1. Label the specimen as shown in Figure 3-9. 'T' refers to top of the specimen for the static test sample.

Figure 3-9: Sample labelling for SHPB (left) and static (right) test

2. On one face of the specimen, mark the centre of the cylindrical specimen. Draw 4 lines through the central point to divide the face into 8 equal parts, as shown in Figure 3-10.

Figure 3-10: Marking sample for dimensioning

3. Measure the height at each line by using a dot as reference and rotate the specimen until the dot returns to original position as shown in Figure 3-11. 8 measurements have been taken.

Figure 3-11: Measuring height of specimen

4. Take average of the 8 measurements as the height of the specimen.
5. Measures the diameters by using the same dot mentioned above as reference and rotate the specimen as shown in Figure 3-12. 3 measurements have been taken.

Figure 3-12: Measuring diameter of specimen

6. Take average of the 3 measurements as the diameter of the specimen.

3.5 Pulse shaper

In conventional SHPB test, the incident (ϵ_I), reflected (ϵ_R) and transmitted (ϵ_T) strain pulses are measured by the strain gauges. The strain, stress and strain rate are then computed by use of these strains as follows:

$$\dot{\epsilon}_s(t) = \frac{d\epsilon_s}{dt} = \frac{c_b}{l_s} (\epsilon_I(t) - \epsilon_R(t) - \epsilon_T(t)) \quad (3-30)$$

$$\epsilon_s(t) = \frac{-2c_o}{l_s} \int_0^t \epsilon_R(t) dt \quad (3-31)$$

$$\sigma_{ave}(t) = E \frac{A_b}{A_s} \varepsilon_T(t) \quad (3-32)$$

where c_b , A_b and E_b are the wave speed, the cross-sectional area and Young's modulus of the pressure bars, respectively; while h_s and A_s are the length and cross-sectional area of the specimen.

Assuming dynamic stress equilibrium in the specimen which is $\varepsilon_I + \varepsilon_T = \varepsilon_s$ and substituting Equation 3.32 to 3.30, we can obtain[219]:

$$E_b A_b \varepsilon_I(t) - A_s \sigma_s(t) = \frac{h_s E_b A_b}{2c_b} \dot{\varepsilon}_s(t) \quad (3-33)$$

Equation 3.33 shows that the incident pulse should be proportional to the stress in the specimen in order to achieve a constant strain rate in the specimen. In other words, the incident pulse must mimic the work hardening behavior of the specimen. However, the incident pulse in the conventional SHPB test is nearly rectangular for proper testing of only perfectly plastic specimens. Instead of direct impact of striker and incident bars in the conventional SHPB test, a pulse shaper is usually placed between the striker and incident bars to possibly shape the incident pulse for mimicking the specimen work-hardening behavior. In the pulse-shaped SHPB test, the dimensions and material of the pulse shaper can be properly determined to achieve the following conditions:

- 1) Constant strain rate
- 2) Dynamic stress equilibrium
- 3) Minimum dispersion effect

A thin disk made of soft materials such as mild steel, copper and aluminum is usually selected as pulse shaper[181]. In the current study, aluminum and copper discs of 1mm thickness and 12mm diameter as well as 8-layer A4 paper with the density of 80g/m² were investigated as pulse shapers. In order to compare the effect of different pulse

shapers, incident, reflected and transmitted strain pulses of magnesium alloy AZ91D and AZ31B are plotted in Figure 3-13 and Figure 3-14, respectively. It can be noted that without pulse shapers, the incident pulse is flat topped with short rise time, which affects the response of the reflected pulse. The strain rate will be thus varying appreciably as the strain rate is dependent on the shape of the reflected pulse. Compared to aluminum and copper disks, the paper pulse shaper is the most useful and can effectively increase the rise time of the incident pulse resulting in a uniform strain rate. Hence, paper pulse shaper was selected.

Figure 3-13: Incident and reflected strain pulses from oscilloscope for different pulse shapers of AZ91D

Figure 3-14: Incident and reflected strain pulses from oscilloscope for different pulse shapers of AZ31B

In order to decide how many layers of paper should be used, experiments were conducted using different number of layers of paper as pulse shaper in between the striker and incident bar. Although paper pulse shaper with more layers can increase the rise time of incident wave more so as to reduce unreliable data during initial loading, it also significantly alters the shape of the waveform as shown in Figure 3-15. It was then decided that a 4-layered paper pulse shaper will be used for all SHPB tests in this work.

Figure 3-15: Strain gauge waveform on oscilloscope (dry run): comparison of different layers of pulse shaper

3.6 Dispersion of waves

Hopkinson bar equations are based on the assumption that one-dimensional wave propagation occurs within the bars. The calculation of stress, strain and strain rate, which has been explained in Section 3.3.4, can only be applied at the bar-specimen interfaces. The signals should be corrected and reconstructed back to the bar-specimen interfaces using a characteristic of the longitudinal wave propagation in the bar, which is naturally dispersed [220-225].

Efforts have been made by several authors to overcome this problem. Davies [220] used the Pochhammer-Chree solution to show the dispersion that takes place in a trapezoidal wave as it travels along the bar. He expressed the periodic wave by a Fourier series and let each component travel with its phase velocity. Some 25 years later Follansbee and Frantz [222] repeated the calculation made by Davies on the dispersion of a trapezoidal wave. Similar calculations were used to predict dispersion of a measured pulse in a SHPB test in order to improve the accuracy of dynamic stress strain curves. The dispersion correction improved the shape of the stress-strain curve by reducing its oscillations, but not eliminating them. The method for treating the dispersion effects was based on Fourier series of the stress pulse in the time domain. Another more efficient method is to consider the pulse as a single pulse and use FFT technique to transform it to the frequency domain. The shape of the pulse after travelling a distance x is obtained by adding the appropriate phase angle to each component of the pulse and taking the inverse transform. Various techniques were employed to minimize the dispersion effects from the strain pulses. Smaller diameter bars and an increase in the pulse rise time [226], elimination of high-frequency components by using low pass filter [227] are some of the techniques to minimize the dispersion effects. Besides, a deformable metallic disc (pulse shaper) sandwiched

between the striker and the input bar [226] [170, 228] can help to attenuate the high-frequency oscillations resulting from the impact thus producing a smooth incident pulse.

In the current study, low pass filter was used to eliminate high-frequency components and paper pulse shaper was used to increase the rise time of incident pulse. A sample of comparison between signals measured from strain gauges, signals filtered and signals with dispersion correction is given in Figure 3-16. It can be seen from the figure that the difference is mainly on the starting part of the curves, namely small strains. Thus, the curves from other test results in this thesis were only filtered by FFT method instead of dispersion correction.

Figure 3-16: Sample of test results comparison

3.7 SHPB experiment at low/high temperature

3.7.1 Experimental setup of low/high temperature SHPB test

SHPB tests at -30°C (243K) temperature were conducted by using 1.8kg of 9mm pellet size dry ice. Styrofoam box filled with 1.8kg of dry ice was slotted between incident and transmitted bar as shown in Figure 3-17(a). The specimen attached centrically to the transmitted bar. The tip of thermocouple wire was positioned at the center of the specimen and held in position by the incident bar. Elevated temperature SHPB tests were conducted by using an induction heater around the specimen as shown in Figure 3-17(b). The heating coil was slotted into the transmitted bar followed by placement of specimen on the end of the transmitted bar. The tip of thermocouple wire was clamped between the specimen and incident bar. A temperature-time calibration of the specimen was conducted before doing real tests which was presented in section 3.7.2. The effect of cooling and heating on the strain gauges was also considered. The temperature at a distance of about 100 mm from the specimen-bar end was measured to be room temperature. It guarantees that the strain gauges mounted in the middle of the bars were not affected by cooling or heating the specimen.

Figure 3-17: Schematic illustration of SHPB setup at (a) below zero temperature (b) elevated temperature

3.7.2 Temperature calibration

3.7.2.1 Low temperature calibration

A thermocouple was used to measure the temperature on the specimen surface in low temperature SHPB testing. As the inside temperature of the specimen may not be at equilibrium with the surface, temperature calibration of the specimen was done to obtain how much time required for the whole specimen to reach $-30\text{ }^{\circ}\text{C}$. The procedures of temperature calibration are as follows:

- 1) Clamp the specimen firmly by using a bench clamp. Be caution not to deform the specimen.
- 2) Drill a 1mm blind hole in the centre of the specimen by using an electric drill.
- 3) Insert the wire tip of the thermocouple into the blind hole in the specimen and secure it with thermal tape as shown in Figure 3-18.

Figure 3-18: Thermocouple wires attached to specimen

- 4) Place the Styrofoam box containing dry ice in between the incident and transmitted bar.
- 5) Apply lubricant between the interface of the specimen and the transmitted bar to secure the specimen.
- 6) Sandwich the specimen between the incident bar and transmitted bar.
- 7) Cover the Styrofoam box and allow the temperature of central part of the specimen to drop to -30°C .
- 8) Record the time taken for the central part of specimen to reach -30°C and remain stable for a while and the amount of the dry ice used.
- 9) Repeat step 4-8 by using roughly the same amount of dry ice for three times and take the average time as reference for real testing.

The dry ice used in this work was roughly 1.8kg and the average time taken to reach -30°C was about 8m20s.

During the period between the removal of thermocouple to the firing of the striker bar, there may be some fluctuation of the specimen temperature. In order to keep the specimen temperature within 1 Celsius degree different from -30°C , the temperatures

were recorded at different intervals after the thermocouple is removed which is shown in Table 3-3. As long as the striker is fired within 15 seconds after the removal of the thermocouple, the specimen temperature still stays at around 30 °C.

Table 3-3: Specimen temperature after removal of thermocouple

Time interval	Temp. of Specimen
After 5s	-30.1 °C
After 10s	-30.2 °C
After 15s	-30.3 °C

3.7.2.2 High temperature calibration

In high temperature split Hopkinson pressure bar testing, an induction heater is used to heat up the specimen and a thermocouple is applied to measure the temperature of the specimen. As the thermocouple is in contact with the surface of the specimen, it can only measure the temperature of the specimen surface. The inside temperature of the specimen may or may not be at equilibrium with the surface. A temperature calibration is thus necessary in order to ensure the entire specimen is heated up to the desired temperature for the experiment. The calibration was carried out on one specimen prior to doing SHPB test to determine the temperature of the heater and the amount of time required to bring the specimen to the desired temperature. The specimen used for calibration is made from the same material and in the same dimension as the investigated specimen. The preparation and calibration procedures are as follows:

- 1) Clamp the specimen firmly by using a bench clamp. Be caution not to deform the specimen.
- 2) Drill a 1mm blind hole in the centre of the specimen by using an electric drill.

- 3) Insert the wire tip of the thermocouple into the blind hole in the specimen and secure it with thermal tape as shown in Figure 3-18.
- 4) Use thermal tape to seal one end of the induction heater heating coil to reduce loss of heat to the surrounding.
- 5) Insert the specimen and secure it in the position from the other end of the heating coil. Make sure the specimen is not in contact with the heating coil.
- 6) Switch on the heater and set the heating temperature to 150 °C.
- 7) Start the stopwatch and record the temperature shown on the thermocouple from 0th second at every 30 seconds interval until a stable temperature is reached.
- 8) Switch off the heater.
- 9) Repeat step 5-8 using a different heating temperature after the specimen cool down to room temperature (i.e. 25 °C).

The specimens were tested at 100 °C and 200 °C. The heating temperature was set at 150 °C, 130 °C, 230 °C and 240 °C in calibration, respectively. The temperatures for each experiment were recorded. The data of AZ31B specimen temperature calibration is shown in Figure 3-4 as an example.

Table 3-4: Temperatures of heated AZ31B specimen at 30s intervals

Time (s)	Specimen Temperature (°C)			
	Expt No. (Heater Temperature)			
	Expt 1 (150°C)	Expt 2 (130°C)	Expt 3 (230°C)	Expt 4 (240°C)
0	23.0	23.0	23.5	23.7
30	67.5	58.0	111.1	123.0
60	84.8	70.7	138.7	147.1
90	97.5	79.9	155.7	166.2
120	104.4	86.9	167.3	178.9
150	109.3	91.4	176.2	187.5
180	112.7	97.3	181.4	190.4
210	114.9	96.8	181.6	194.0
240	115.9	98.1	184.0	196.6
270	115.4	98.9	185.0	196.9
300	117.1	100.2	185.0	197.6
330	120.1	99.9	185.6	198.0
360	118.8	99.9	187.0	195.8
390	118.4	99.8	185.2	195.9
420	118.6	99.7	185.5	200.3
450	118.3	99.4	185.7	198.5
480	119.9	99.8	185.5	197.7
510	118.8	100.0	185.8	197.0
540	118.3	99.0	185.3	197.3
570		99.9	186.3	197.0
600		99.3	186.6	199.7
630			185.6	198.7
660				197.8

Figure 3-19: AZ31B specimen temperature versus time

The calibration temperature vs. time was also plotted as shown in Figure 3-19. It can be used as reference while conducting elevated temperature SHPB testing. When the

heater temperature was set at 130 °C, a steady temperature of 100 °C was achieved after approximately 300s. When the heater temperature was set at 240 °C, a steady temperature of 200 °C was reached after about 400s. In conclusion, to achieve 100 °C and 200 °C of AZ31B specimen, the following heating time and temperatures are needed:

Table 3-5: Heater temperature and time required for 100 °C and 200 °C AZ31B specimen

To achieve:	100 °C	200 °C
Heater Temp.	130 °C	240 °C
Time (s)	>300	>400

3.7.3 Modification of data-processing method with thermal gradient in Hopkinson bars

When the mechanical impedance along the bars is constant, the deformation and stress in the specimen can be determined from recorded waves using one-dimensional elastic wave theory. The Hopkinson equation can be used without further modifications if either the whole apparatus is at the same temperature or only the specimen is at the testing temperature and the rest of the system is at room temperature. However, portions of the incident and transmitted bars were also heated while heating the specimen in this work. Changing the temperature of the specimen introduced a temperature gradient in the bars which may affect the propagation of the elastic waves. A variation of temperature along the bars can affect the bars' elastic constants and thus the impedance and the speed of elastic waves. A nonconstant impedance along the bars which causes reflections of the elastic waves will make the interpretation of the recorded wave data very difficult.

When tests are conducted at mildly elevated temperatures ($\sim 200^\circ\text{C}$), a possible change in the elastic constants along the bars due to temperature gradient caused by heating the specimen is small and can be neglected. The data are processed like in the room-temperature test ignoring the temperature gradient in the bars. When tests are conducted at higher temperatures, the effect of temperature should be corrected. Although the highest heating temperature in this work was 473K (200°C), the modified SHPB equations[229] were still used to obtain strain, stress and strain rate of the specimen at elevated temperature more accurately as shown below:

$$\varepsilon_e(t) = \sqrt[4]{\frac{E}{E_A} \frac{2C_0}{L_s}} \int_0^t \varepsilon_R(t) dt \quad (3-34)$$

$$\dot{\varepsilon}(t) = \sqrt[4]{\frac{E}{E_A} \frac{2C_0}{L_s}} \varepsilon_R(t) \quad (3-35)$$

$$\sigma_e(t) = \sqrt[4]{\frac{E_A}{E}} E\left(\frac{A}{A_s}\right) \varepsilon_T(t) \quad (3-36)$$

where E_A is the elastic modulus of the bars at elevated temperature which has been obtained by conducting tensile tests on the specimens made from the same bar material. E_A/E is equal to 0.95 at 100°C , while E_A/E is equal to 0.98 at 200°C .

3.8 Data processing

3.8.1 SHPB signals calibration

The most crucial step in the data processing is calibration of SHPB signals. When (i) a new test setup, (ii) the bars have been changed and (iii) a new strain gauge has been mounted, the bar calibration must be conducted in order to determine the strain correction factor and the stress correction factor.

3.8.1.1 'Bar apart' testing for the strain correction factor

The strain correction factor is determined by impacting the striker bar on the input bar and the output bar separately. Based on the one-dimensional wave propagation theory, the stress in the bar which is produced by the impact of the striker bar is given by[230]:

$$\sigma = \rho \times c_0 \times \dot{u} \quad (3-37)$$

where ρ is the density of the bar, c_0 is the actual longitudinal bar wave velocity and $\dot{u}$ is the particle velocity in the stress pulse. The particle velocity is given by[230]:

$$\dot{u} = V/2 \quad (3-38)$$

where V is the striker bar velocity. Assuming the bar remains elastic during testing, the strain in the bar can be calculated as:

$$\varepsilon^{calculated} = \frac{\sigma}{E} = \rho \times c_0 \times \frac{V}{2E} = V/2c_0 \quad (3-39)$$

The strain gauge signals (in voltage) need to be converted into strain signals as the measure strain to compare with the calculated strain. The strain correction factor is then calculated from the ratio of the calculated strain to the measured strain as follows:

$$K_\varepsilon = \varepsilon^{calculated} / \varepsilon^{measured} \quad (3-40)$$

In the real experiment, the incident, reflected and transmitted waves can be corrected as:

$$\varepsilon_i^{calculated} = K_\varepsilon \times \varepsilon_i^{measured} \quad (3-41)$$

where subscript i is used to denote incident, reflected and transmitted waves.

Calculation of longitudinal bar wave velocity c_0

‘Bar apart’ tests were carried out to obtain bar wave velocity in this work. A wave is generated inside the input bar once the striker impact the input bar. The wave reflection occurs and can be recorded by strain gauge and shown on oscilloscope. c_0 can then be calculated as :

$$c_0 = \frac{\text{distance travelled by wave}}{\text{time taken}} \quad (3-42)$$

The strain gauges were mounted in the midpoint of the bars, as shown in Figure 3-20. The distance l_1 from start of the incident bar to the center line of the strain gauge and the distance l_2 from end of the incident bar to the center line of the strain gauge were measured by using a measuring tape. The time taken for the wave to travel across the bar can be obtained from the oscilloscope.

Figure 3-20: Schematic diagram of incident bar

In order to minimize the systematic error due to the time which cannot be counted accurately when the wave travelled inside the strain gauge, 8 waves including 4 incident waves and 4 reflected waves were used. The duration time t was recorded on the oscilloscope. An example of the oscilloscope screenshot with the 8 waves captured is shown in Figure 3-21.

Figure 3-21: Oscilloscope screenshot with waves captured

Thus,

$$c_0 = \frac{l_1 + l_1 + l_2 + l_2 + l_1 + l_1 + l_2 + l_2 + l_1 + l_1 + l_2 + l_2 + l_1 + l_1}{t'}$$

$$= \frac{8l_1 + 6l_2}{t'} = 4968.47 \text{ m/s}$$

Measurement of striker bar velocity

The impact velocity of striker bar was measured by using photodiodes and laser beam. The photodiode setup is shown in Figure 3-22. The distance d between two laser torches were measured. The time t taken for striker bar to travel from photodiode 1 to photodiode 2 was measured via the oscilloscope. A sample oscilloscope screenshot for the measurement of striker bar travelling time is shown in Figure 3-23. Thus, the impact velocity V of the striker bar can be calculated as:

$$V = \frac{d}{t} \quad (3-43)$$

Using Figure 3-23 for example, Impact velocity of Striker bar $V = 40\text{mm}/4.95\text{ms} = 8.08$ m/s. According to Equation 3.36,

$$\varepsilon_{\text{calculated}} = \frac{V}{2c_0} = 8.13 \times 10^{-4} \quad (3-44)$$

Figure 3-22: Schematic diagram of photodiode setup

Figure 3-23: Oscilloscope screenshot for measuring the travel time of the striker bar

By using the same pressure of the gas to achieve the same velocity (8.08m/s), the measured incident strain can be obtained as $\epsilon_i^{measured}$. According to Equation 3.37, strain correction factor $K\epsilon=0.9835$.

3.8.1.2 'Bar together' testing for the stress correction factor

The stress correction factor is determined by impacting the striker bar on the coupled input-output bars. A pulse is generated after the input bar is impacted by the striker. This pulse is partially reflected back to the input bar and partially transmitted to the

output bar on the input-output bars interface. If the interface is perfectly aligned, there will be no reflected wave and the shape of incident and transmitted waves should be similar.

The stress correction factor is the ratio of the amplitude of the incident wave to the transmitted wave:

$$K_{\sigma} = \varepsilon^{incident} / \varepsilon^{transmitted} \quad (3-45)$$

Then the calculated average stress in the specimen can be corrected as:

$$\sigma_s^{corrected} = K_{\sigma} \times \sigma_s^{measured} \quad (3-46)$$

Before conducting ‘Bar together’ empty test to get stress correction factor, the optimum orientation of the striker bar and incident bar should be found. The striker bar and the incident bar were divided into and marked at four equal angles, i.e. 0°, 90°, 180°, 270°. There were 16 orientations altogether. ‘Bar together’ empty tests were conducted at each orientation to find out the optimum orientation, i.e. the most incident wave transmitted to the transmitted bar. ‘Bar together’ test was conducted at the optimum orientation to get the stress correction factor, which is shown in Figure 3-24. According to Equation 3.42, the stress correction factor K_{σ} is equal to 0.927.

Figure 3-24: 'Bar together' calibration test result

3.8.2 Data acquisition

The strain on incident and transmitted bars were measured by two diametrically opposing strain gauges, amplified by dynamic strain meters and then input to digital oscilloscope. The incident and reflected strain pulse were recorded and plotted on the same channel (Channel 1) while the transmitted strain pulse was recorded and plotted on Channel 2. The output data from the oscilloscope were in the form of voltage histories and opened in Excel. The Excel file contained two column data, while the first column presented incident and reflected voltage signals recorded in Channel 1 and the second column presented transmitted voltage signals recorded in Channel 2. As only the first pulse of incident, reflected and transmitted waves is needed, it is important to select the start and ending point of them. Usually these three pulses start from zero and end at zero. But it is not necessary to take transmitted pulse data up till zero as the data is useless after unloading or fracture of the specimen. In next step, these selected voltage signals were converted in to incident (ϵ_I), reflected (ϵ_R) and transmitted (ϵ_T) strain pulses by multiplied by the gain factor which was dependent on

the strain gauge factor, the amplification of strain meters and the type of Circuit Bridge to connect strain gauges.

The specimen stress $\sigma(t)$ can then be calculated by using transmitted strain ϵ_T based on Equation 3.32. The strain rate $\dot{\epsilon}(t)$ can be calculated by using reflected strain ϵ_R according to Equation 3.26. In order to get the specimen strain, the reflected strain ϵ_R needs to be integrated as shown in Equation 3.27.

3.8.3 Experimental strain rate measurements

Strain rate in SHPB experiments is an average value as a constant strain rate cannot be achieved. In high strain rate tests, the strain rate first increase steeply, then reach a plateau and finally decrease sharply as can be seen in Figure 3-25. In present study, the strain rate is calculated by taking the average value of the plateau region (between A and B) of the strain rate vs. time curve.

Figure 3-25: Average strain rate calculations during high strain rate testing

3.8.4 Data smoothing

The raw strain signals captured during SHPB tests are usually accompanied by some noise. Although the signals have already passed through ‘High Pass Filter’ of the dynamic strainmeter, the raw data from oscilloscope need to be smoothed by a fast Fourier transform low pass filter which is included in a Matlab code. The cut-off frequency is calculated as [231]:

$$f_c = 0.3\sqrt{2}c_0/(2\pi av) \quad (3-47)$$

where a is the bar’s radius. One of the comparisons between original data and filtered data is shown in Figure 3-26 as an example.

Figure 3-26: Engineering stress-time curve before and after filtering

3.9 Thermal softening effects

In an adiabatic test, assuming there are no heat losses and that all the mechanical work of deformation is converted into heat, then the temperature rise ΔT corresponding to a strain ε in the sample is given by

$$\Delta T = \frac{1}{C_p} \int_0^\varepsilon \sigma(\varepsilon) d\varepsilon \quad (3-48)$$

where C_p is the volume specific heat of the specimen [232].

The subject of adiabatic plastic deformation has been reviewed by Rogers [233] and more recently by Stelly and Dormeval [234]. In spite of the obvious importance of adiabatic softening, few attempts have been made to evaluate the magnitude of the effect on actual stress/strain behaviour. Follansbee [234] calculate the temperature rise in a specimen of Nitronic 40 stainless steel compressed by 20% in a SHPB test at 5000 s⁻¹ should be 62 °C. He estimated that such a temperature rise could account for a reduction in flow stress of 168MPa, i.e.14% of the observed flow stress, at 20% true strain.

To calculate the temperature rise at 20% strain under -30 °C, 25 °C, 100 °C and 200 °C of AZ31B, a selection of mean curves of true stress against true strain for all test temperatures at the highest strain rate was used as shown in Figure 3-27. The variation in flow stress was to be found to be less than $\pm 5\%$ about the mean at any strain level as can be seen from the repeatability figures. The specific heat of magnesium is 1.02 J/g °C. The density of magnesium is 1738 kg/m³. So the volume specific heat of magnesium is 1.77×10^6 J/Kg °C. The value of temperature rise at 20% strain under -30 °C, 25 °C, 100 °C and 200 °C at the strain rate of 3300 s⁻¹ is shown in Table 3-6.

 Figure 3-27: True stress-true strain at $3300 \pm 300 \text{ s}^{-1}$ of AZ31B

 Table 3-6: Temperature rise at 20% strain at $3300 \pm 300 \text{ s}^{-1}$ of AZ31B

Temperature	-30 °C	25 °C	100 °C	200 °C
ΔT (°C)	30	27	24	22

Having established the manner of the flow stress decreases with temperature at a given strain rate, it is possible to calculate the reduction in flow stress $\Delta\sigma$ attributable to adiabatic heating effects. Values of $\Delta\sigma$ due to a temperature rise ΔT in the sample were determined by

$$\Delta\sigma = \left. \frac{\partial\sigma}{\partial T} \right|_{\varepsilon=5\%} \times \Delta T \quad (3-49)$$

For a particular test condition, values of ΔT were evaluated according to Equation (3-48), while $\left. \frac{\partial\sigma}{\partial T} \right|_{\varepsilon=5\%}$ was obtained from appropriate curves such as in Figure 3-28. The gradient at 5% strain was used since 5% true strain is large enough for the flow stress at this point to be undisturbed by yield point effects and yet small enough that no significant temperature rise will yet have taken place during compression. Values of

$\Delta\sigma$ are shown in Table 3-7. It can be seen from Table 3-7 that the predicted temperature rise leading to thermal softening accounts for a total reduction in stress of 9.75MPa, 8.77MPa, 7.80MPa and 7.15MPa at a true strain of 20% under -30 °C, 25 °C, 100 °C and 200 °C respectively. The reduction are 2.6%, 2.4%, 2.3% and 2.4% of the actual observed flow stresses under -30 °C, 25 °C, 100 °C and 200 °C respectively. Thus, the true stress-strain curves will be more accurate if the adiabatic condition is taken into consideration.

Figure 3-28: True stress/Temperature variation of AZ31B for two strains at $3300 \pm 300 \text{ s}^{-1}$

Table 3-7: The reduction of flow stress at $\epsilon=20\%$

Temperature	-30 °C	25 °C	100 °C	200 °C
$\Delta\sigma(\text{MPa})$	9.75	8.77	7.80	7.15

Chapter 4 Compressive mechanical behaviour of magnesium alloy AZ31B

The experimental results of magnesium alloy AZ31B under compression are presented and discussed in this chapter. Tests were performed at strain rates ranging from 10^{-3} s^{-1} to 3300 s^{-1} and temperatures between -30°C and 200°C . In each testing condition, five tests were carried out to ensure the reproducibility. The data shown in this chapter are the representative results of at least three consistent tests under each condition. In dynamic compressive tests, the strain rates were controlled by velocity of the striker bar as the dimension of specimens remained the same.

4.1 Effect of strain rate

Figure 4-1 shows strain rates obtained in SHPB tests on AZ31B specimens. The strain rate usually begins with a sharp increase then keeps at a small variation for a long range and finally decreases rapidly to zero. The gradual decrease observed in the plateau region of the strain rate vs. time curve is usually due to the hardening of the alloy under compression. The average strain rate values shown in the figure are average values of the plateau portion of strain rate vs. time curves, as explained in the previous chapter.

Figure 4-1: Sample strain rates measured in SHPB tests on AZ31B

To illustrate the strain rate dependence of the mechanical behavior of AZ31B, the true stress-strain curves of this material at temperatures of $-30\text{ }^{\circ}\text{C}$, $25\text{ }^{\circ}\text{C}$, $100\text{ }^{\circ}\text{C}$ and $200\text{ }^{\circ}\text{C}$ are plotted in Figure 4-2. Generally speaking, for a given temperature, the flow stress (*stress required to sustain plastic deformation at a particular strain*) increases with increasing strain rate within dynamic range. At room temperature, both the 0.2% offset yield strength (*draw a line parallel with the modulus slope with 0.2% offset, the stress/strength at the intersection with the graphs will be the 0.2% yield stress/strength*) and flow stress at 5% strain first increase and then decrease with the increasing of strain rate as shown in Table 4-1. The maximum values are achieved at the strain rate of 2257 s^{-1} . However, the peak compressive strength (*the maximum stress in the stress-strain curve beyond which the stress starts decreasing*) undergoes a large increase of about 79.5% with strain rate increasing from 733 s^{-1} to 2257 s^{-1} , while there is a slight decrease of about 5.7% with strain rate increasing from 2257 s^{-1} to 2717 s^{-1} . It can be noted that, for a given strain before unloading, the corresponding stress obtained in

high strain rate tests is always higher than quasi-static tests. The ultimate compressive strength (value of uniaxial compressive stress reached when the material fails completely) has a significant increase of 80.9% when the strain rate increases from 0.001 s^{-1} to 3200 s^{-1} under room temperature as can be seen from Table 4-1. The initial slope of the true stress-strain dynamic curve is steeper than that in the quasi-static one. The difference of true stress-strain curves between quasi-static and dynamic loading condition become more obvious with the increase in temperatures.

Figure 4-2: True stress-strain curves of AZ31B at different strain rates for different temperatures

Table 4-1: Compressive strength properties of AZ31B at 25 °C

Magnesium alloy AZ31B						
Strain rate [s ⁻¹]	10 ⁻³	733	1370	2257	2717	3200
0.2% Offset yield strength [MPa]	40±3	45±3	50±3	70±3	60±3	50±3
Flow stress at 5% strain [MPa]	100±3	135±3	145±3	195±3	160±3	155±3
Ultimate compressive strength/Peak compressive strength [MPa]	210±5	195±3*	290±5*	350±5*	330±5*	380±5
Failure strain/Maximum strain reached	0.150±0.002	0.073±0.001	0.131±0.001	0.180±0.002	0.180±0.002	0.280±0.005

*Failure of specimens didn't occur. This is the maximum stress reached during testing.

To quantitatively evaluate the strain rate sensitivity of flow stress, the strain rate sensitivity (SRS) parameter is introduced[235]:

$$m = \left(\frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right)_{\epsilon, T} = \frac{\Delta \ln \sigma}{\Delta \ln \dot{\epsilon}} = \frac{\ln \sigma_1 - \ln \sigma_2}{\ln \dot{\epsilon}_1 - \ln \dot{\epsilon}_2} \quad (4-1)$$

where σ_1 and σ_2 are the true stress tested under the same temperature T corresponding to a constant strain ϵ at strain rate $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$ respectively. Figure 4-3 shows curves of relationship between $\ln(\text{stress})$ and $\ln(\text{strain rate})$ calculated for $\epsilon = 0.02, 0.04$ and 0.06 under four temperatures within high strain rates. The difference of the slopes indicates the variation of strain rate sensitivity m . AZ31B shows different strain rate sensitivity at low, room and high temperature. There is not much difference between m at 100 °C

and 200 °C. Strain rate sensitivity at strain levels between 4% and 6% shows similar trend at each temperature, while strain rate sensitivity at 2% strain is quite different from other strain levels. In addition, the strain rate sensitivity almost keeps constant under -30 °C and 25 °C at larger strains($\geq 2\%$).

Figure 4-3: Strain rate sensitivity of AZ31B

4.2 Effect of temperature

Temperature effect on compressive true stress-strain curves for strain rates of 0.001 s^{-1} , $700 \pm 50\text{ s}^{-1}$, $1400 \pm 100\text{ s}^{-1}$ and $3300 \pm 300\text{ s}^{-1}$ is shown in Figure 4-4. At a constant strain rate, the flow stress decreases with temperature increasing. The stress-strain relationship between -30 °C (243K) and 25 °C (298K) only show small difference, while

significant thermal softening is seen at 100 °C(373K) and 200 °C(473K). Temperature has the most significant effect at quasi-static loading. The effect of temperature on the flow stress is shown in Figure 4-5. It can be noted that the flow stress at 5% strain decreases with temperature increasing. For all strain rates, the maximum value of the flow stress at 5% strain is achieved at the lowest temperature, while the minimum value is achieved at the highest temperature, i.e. 200 °C(473K). Besides, AZ31B can bear slightly more strength at below zero temperature than at elevated temperature under dynamic loading. The effect of temperature on failure strain at strain rate of 0.001 s^{-1} and $3300 \pm 300 \text{ s}^{-1}$ is shown in Figure 4-6. It is obvious that temperature doesn't have much influence at high strain rate, while there is a 21.2% increase of failure strain at 0.001 s^{-1} when temperature increases from 25 °C (298K) to 200 °C (473K).

Figure 4-4: True stress-strain curves of AZ31B at different temperatures for different strain rates

Figure 4-5: The effect of temperature on the flow stress at 5% strain at all strain rates

Figure 4-6: The effect of temperature on failure strain at 0.001 s⁻¹ and 3300±300 s⁻¹

The temperature sensitivity of the deformed specimens can be quantified as follows[18]:

$$t_{\sigma} = |(\sigma_2 - \sigma_1)/(T_2 - T_1)| \quad (4-2)$$

where σ_1 and σ_2 are obtained in tests conducted at temperatures T_1 and T_2 , respectively. The temperature sensitivity with variation of true strain at different high strain rates under a wide range of temperature is shown in Figure 4-7. It can be noted from this figure that at the constant strain rate, the temperature sensitivity decreases as temperature increases. Within a given temperature range, the temperature sensitivity increases as strain rate increases. However, there is not a consistent tendency for temperature sensitivity with variation of true strain.

Figure 4-7: Temperature sensitivity-true strain curves at different strain rates and temperatures

4.3 Strain hardening

The strain-hardening of metals means their load bearing capacity increases during deformation. Strain hardening is the strengthening of a material by plastic deformation. This strengthening occurs because of dislocation generation and dislocation movements within crystal structure of the material. The measure of the ability of materials to get strength is known as strain hardening exponent n which is calculated by

$$n = d\log(\sigma_t)/d\log(\varepsilon_t) \quad (4-3)$$

where σ_t is true stress and ε_t is true strain. Strain hardening exponent n is graphically the slope of the line fitted to true stress-true strain data on log-log scale. Figure 4-8 shows the strain hardening behaviour of AZ31B at various strain rates under room temperature (25 °C). A linear relationship between $\log(\text{true stress})$ and $\log(\text{true strain})$ exists only within a certain region at all strain rates. The linear portion is usually when the strain is small. The hardening exponent is dependent on the strain. It first increases and then decreases with strain at all strain rates. The hardening exponent decreases slightly with strain rate increasing due to heat generation during dynamic compression as shown in Table 4-2. The heat generated by adiabatic compression is more at 3300 s^{-1} than at 700 s^{-1} and 1400 s^{-1} .

Figure 4-8: Effect of strain rate on the hardening behavior of AZ31B at 25 °C

Table 4-2: Strain hardening exponent of AZ31B at 25 °C

$\varepsilon =$	0.01~0.04	0.05~0.1	0.11~0.16
0.001 s^{-1}	0.9903	1.1162	0.4618
$\varepsilon =$	0.01~0.03	0.04~0.06	0.07~0.08
$700 \pm 50 \text{ s}^{-1}$	0.4626	0.8150	0.3052
$\varepsilon =$	0.01~0.02	0.03~0.09	0.10~0.13
$1400 \pm 100 \text{ s}^{-1}$	0.4096	0.8175	0.3267
$\varepsilon =$	0.01~0.04	0.05~0.11	0.12~0.20
$3300 \pm 300 \text{ s}^{-1}$	0.5431	0.7422	0.3083

The effect of the temperature on the hardening behaviour of AZ31B is also investigated. Figure 4-9 shows hardening behaviour of AZ31B at 3300s^{-1} for various temperatures. The hardening exponent increases and then decreases with true strain at low and elevated temperatures, as observed for room temperature. There are three regions regarding the alloy's hardening exponent. The ranges of each region are almost the same for four temperatures, while the range of strain region for different strain rates at room temperature is different from each other. It means the temperature doesn't have as much effect on strain hardening behaviour as the strain rate. The values for each temperature and strain range are given in Table 4-3. For the same strain range, the maximal value of hardening exponent is achieved at room temperature while the minimal value is achieved at the highest temperature which is 200°C .

Figure 4-9: Effect of temperature on the hardening behavior of AZ31B under various temperatures at 3300 s^{-1}

Table 4-3: Strain hardening exponent of AZ31B at 3300 s⁻¹

ϵ T	0.01~0.04	0.05~0.13	0.15~0.20
-30 °C	0.5012	0.5857	0.1679
25 °C	0.5434	0.7340	0.1973
100 °C	0.4681	0.6578	0.4486
200 °C	0.3979	0.5718	0.5298

4.4 Energy absorption capability

Energy absorption is an increasingly important function of structural materials as structural crashworthiness is now an essential requirement in the design of automobiles, aircraft and rotorcraft. The crashworthy structure is designed to absorb the impact energy in a controlled manner before the energy gets transmitted into the passenger compartment in the event of a crash. Metals can absorb impact energy in a controlled manner because of the plastic deformation characteristics, which makes them most commonly used for crashworthy structural applications[236]. The axial crush[236] and bending crush[237] are two common tests[238] used for examining the energy absorbing capabilities of materials or structural. Axial crush tests can be carried out under either quasi-static loading or impact loading. The static axial collapse tests can be performed between the parallel steel plates of a hydraulic press at very low crosshead speed. In this work, Instron machine was used to carry out static axial crush tests. The corresponding dynamic tests were conducted by using Split Hopkinson Pressure Bar. The energy absorbed by the collapsed specimen during the axial crushing

process can be calculated by using the definition of area under the stress-strain curve up to failure strain. The energy absorbed by AZ31B alloy during compression under both static and dynamic loading was calculated by using equation:

$$E = \int_0^{\epsilon_f} \sigma(t) d\epsilon \quad (4-4)$$

where ϵ_f is the failure strain.

To quantify the energy absorption capability of AZ31B alloy and to see how it changes with temperature under quasi-static and dynamic loading, the energy absorbed by AZ31B specimens are calculated and plotted against homologous temperature T^* which is defined by:

$$T^* = \frac{T - T_0}{T_m - T_0} \quad (4-5)$$

where T_0 (-273K) is the absolute zero and T_m (893K) is the melting temperature in Kelvin. The results are given in Figure 4-10. As the specimens were only broken into pieces at 0.001 s^{-1} and $3300 \pm 300 \text{ s}^{-1}$, the energy absorption of the alloy was only calculated for these two strain rates. It can be found that the alloy can absorb much more energy under dynamic loading. The effect of temperature on the energy absorption is less significant at high strain rate, especially when the temperature is higher than room temperature (25 °C). There is a 62.7% decrease of energy absorbed when the temperature increases from 25 °C to 200 °C under quasi-static loading, while the decrease under dynamic loading is only 11.5% in the same temperature range.

Figure 4-10: Energy absorption capability of AZ31B

4.5 Microstructural analysis on strain rate effect

Optical and scanning electron microscopy was performed on selected specimens at quasi-static and high strain rates. The sample preparation includes sectioning, molding (embedding the sectioned specimens in a thermosetting resin), grinding, polishing and etching. The surface parallel to the impact direction was ground with 800, 1200, 2400 and 4000 grit abrasive paper. 1 μm diamond paste was used for polishing. The composition of the etchant used in this work is 4.2g picric acid + 10ml acetic acid + 70ml ethanol + 10ml H_2O . The optimum etchant time was about 6-8 s. Micrographs from optical microscope and scanning electron microscope are shown in Figure 4-11 and Figure 4-12.

Figure 4-11: Micrographs from Optical microscope (a) 10^{-3} s^{-1} (b) 700 s^{-1} (c) 1400 s^{-1} (d) 3300 s^{-1}

A large number of twins can be seen at strain rate of 10^{-3} s^{-1} . Twin density decreases as the strain rate increases. Some intersecting twins as well as voids are observed at all strain rates, while micro-cracks parallel to twins and along grain boundaries are only observed at 3300 s^{-1} strain rate, which seems to be the result of coalescence of voids. Some localized shear bands are also formed and the grains are refined in the specimens tested at 3300 s^{-1} strain rate. The refined grains are about 4 to 5 times smaller than the size of those at quasi-static strain rate. Grains within the shear bands are severely deformed and cannot be clearly identified. The alloy partly melts at high strain rates and deforms plastically because of thermal effects. Twinning contributes to plastic deformation as well.

Figure 4-12: Scanning Electron Micrographs (a) 10^{-3} s^{-1} (b) 700 s^{-1} (c) 1400 s^{-1} (d) 3300 s^{-1}

4.6 Constitutive modelling

In order to utilize the experimental results in simulations, the experimental data must be evaluated. The most common approach is to use existing material models (e.g. Johnson-Cook Model [239], Zerilli-Armstrong Model [240], Huh-Kang-Han Model [207], etc.) to quantify constitutive behaviour of the material investigated. In the present study, the Johnson-Cook model is used to model the constitutive response of AZ31B as a function of strain rate and temperature.

In this model, the flow stress is described as a function of the plastic strain, the strain rate and the temperature which is given by the equation as follows:

$$\sigma = (A + B\varepsilon_{eps}^n)(1 + C \ln \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})(1 - (T^*)^m) \quad (4-6)$$

where ε_{eps} is the effective plastic strain, $\dot{\varepsilon}$ is the strain rate, $\dot{\varepsilon}_0$ is the reference strain rate (usually taken as 1 s^{-1}), T^* is homologous temperature which is defined in $T^* = \frac{T - T_{room}}{T_{melt} - T_{room}}$. A, B, C, n and m are five material constants which are determined by the experimental data. This model is widely used as it can be easily implemented into commercial numerical codes. The terms in the first set of parentheses impose a power-law relationship on the true stress versus effective plastic strain. The terms in the second parentheses introduce a logarithmic dependence on the strain rate and the final term gives the stress an exponential decay as temperature increases. The estimated values for the constitutive parameters as well as the lower and upper bounds of 95% confidence are given in Table 4-4. The constitutive parameters required by the Johnson-Cook model are fit to the experimental results using a MATLAB built-in curve fitting tool based on nonlinear regression analysis. The model fitted curves at different strain rates and temperatures are shown in Figure 4-13. The experimental data considered were reduced to (i) after the constant strain rate was achieved and (ii) before the stress reached the peak compressive stress. It can be observed that reasonable agreement with the experimental data is obtained except in the beginning portion of the flow curves at 700 s^{-1} . The reason might be that the duration of constant strain rate at 700 s^{-1} is shorter than that at higher strain rate. Overall, best agreement with experimental data is obtained at highest strain rate (3300 s^{-1}) and lowest temperature ($25 \text{ }^\circ\text{C}$). The Johnson-Cook model only captures constant log-linear strain rate sensitivity while in fact it changes with strain rate.

Table 4-4: Parameters of the Johnson-Cook model for AZ31B

Parameter	Estimate	95% Confidence	
		Lower bound	Upper bound
A[MPa]	73	67	81
B[MPa]	1610	1350	1953
n	0.8409	0.7412	0.9314
C	0.012	0.010	0.014
m	0.9313	0.9147	0.9500

Figure 4-13: Experimental and Johnson-Cook fitted curves at different strain rates and temperature

Chapter 5 Compressive mechanical behaviour of 0.5 vol.% SiC_p/AZ31B

The experimental results of AZ31B reinforced with 0.5 vol.% SiC nanoparticles are presented and discussed in this chapter. Tests were performed at strain rates ranging from 10^{-3} s^{-1} to $2200 \pm 200 \text{ s}^{-1}$ and temperatures between -30°C and 200°C . In each testing condition, five tests were carried out to ensure the reproducibility. The data shown in this chapter are the representative results of at least three consistent tests under each condition.

5.1 Effect of strain rate

Figure 5-1 shows strain rates obtained in SHPB tests on 0.5vol% SiC_p/AZ31B specimens. The shapes of strain rate-time curves are similar to the shape of AZ31B. The strain rate begins with sharp increase then decreases slowly for a long range and finally decreases rapidly to zero. The average strain rate values shown in the figure are average values of the plateau portion of strain rate vs. time curves.

Figure 5-1: Sample strain rates measured in SHPB tests on 0.5vol% SiC_p/AZ31B

To illustrate the strain rate dependence of the mechanical behaviour of AZ31B reinforced with 0.5 vol.% silicon carbide nanoparticles, the true stress-strain curves of this material at -30 °C, 25 °C, 100 °C and 200 °C are plotted in Figure 5-2. Generally speaking, the flow stress increases with increasing strain rate under all temperatures. For a given strain before unloading, the corresponding stress obtained in high strain rate tests is always higher than quasi-static tests. However, for a given stress, the corresponding strain obtained in high strain rate tests is much smaller than quasi-static tests. The initial slope of the true stress-strain dynamic curve is steeper than that in the quasi-static one. The maximum value of 0.2% offset yield strength and flow stress at 5% strain are also achieved at 2200 s⁻¹. Although the specimens are broken under quasi-static loading, the ultimate compressive strength at 0.001 s⁻¹ is 68% lower than peak compressive strength at 747 s⁻¹ as can be seen in Table 5-1.

Figure 5-2: True stress-strain curves of 0.5vol% SiC_p/AZ31B at different strain rates for different temperatures

Table 5-1: Compressive strength properties of 0.5vol% SiC_p/AZ31B at 25 °C

0.5vol% SiC _p /AZ31B					
Strain rate [s ⁻¹]	10 ⁻³	747	1450	2200	3130
0.2% Offset yield strength [MPa]	20±2	80±2	100±2	100±2	100±2
Flow stress at 5% strain [MPa]	90±2	220±5	250±5	290±5	230±5
Ultimate compressive strength/Peak compressive strength* [MPa]	160±3	270±5*	400±5*	500±5*	520±5
Failure strain/Maximum strain reached	0.185±0.002	0.070±0.001	0.135±0.002	0.210±0.002	0.275±0.002

*Failure of specimens didn't occur. This is the maximum stress reached during testing.

Figure 5-3 shows curves of relationship between $\ln$ (stress) and $\ln$ (strain rate) calculated for $\epsilon=0.02$, 0.04 and 0.05 under four temperatures. The difference of the slopes indicates the variation of strain rate sensitivity m . AZ31B reinforced with 0.5 vol.% SiC shows different strain rate sensitivity at low, room and high temperature. Strain rate sensitivity at different temperature shows different trend. Strain rate sensitivity decreases with strain rate increasing at -30 °C and 200 °C, while it increases with strain rate increasing at 25 °C and 100 °C. Strain rate sensitivity m doesn't show much difference when strain changes from 0.002 to 0.005 at -30 °C, 25 °C and 100 °C. The biggest difference of m between different strains happens at 200 °C.

Figure 5-3: Strain rate sensitivity of 0.5vol% SiC_p/AZ31B

5.2 Effect of temperature

Figure 5-4 shows temperature effect on compressive true stress-strain curves for strain rates of 0.001 s^{-1} , $700 \pm 50 \text{ s}^{-1}$, $1400 \pm 100 \text{ s}^{-1}$ and $3300 \pm 300 \text{ s}^{-1}$. At a constant strain rate, the flow stress decreases with temperature increasing. The stress-strain relationship between -30°C and 25°C only show small difference, while significant thermal softening is seen at 100°C and 200°C . Temperature effect at $700 \pm 50 \text{ s}^{-1}$ is less evident than its effect at $1400 \pm 100 \text{ s}^{-1}$ and $3300 \pm 300 \text{ s}^{-1}$. The effect of temperature on the flow stress is shown in Figure 5-5. The flow stress at 5% strain first increases, reaches the maximum value at room temperature (25°C) and then decreases with temperature

increasing. The lowest value of flow stress at 5% strain for all strain rates is achieved at the highest temperature (200 °C).

Figure 5-4: True stress-strain curves of 0.5vol% SiC_p/AZ31B at different temperatures for different strain rates

Figure 5-5: The effect of temperature on the flow stress at 5% strain at all strain rates

The temperature sensitivity with variation of true strain at high strain rates under a wide range of temperature is shown in Figure 5-6. It can be noted that at the constant strain rate, the temperature sensitivity increases as strain becomes larger. The most obvious increase happens at 1400 s^{-1} and 3300 s^{-1} in the temperature range of $25 \text{ }^{\circ}\text{C} \sim 200 \text{ }^{\circ}\text{C}$. For a given strain and strain rate, temperature sensitivity increases with temperature increase. Flow stress is less sensitive in temperature at lower strain rate. However, temperature sensitivity doesn't have the same variation tendency with change of strain rate for all temperature ranges.

Figure 5-6: Temperature sensitivity-true strain curves of 0.5vol% SiC_p/AZ31B

5.3 Strain hardening

Figure 5-7 shows the strain hardening behaviour of 0.5vol% SiC_p/AZ31B at various strain rates under room temperature (25 °C). A linear relationship between log (true stress) and log (true strain) exists only within a certain region of strain at all strain rates. The nonlinear portion lays in large strain. The hardening exponent decreases with strain at 0.001 s⁻¹, while it increases at 700 s⁻¹ and 1400s⁻¹ as shown in Table 5-2. The hardening exponent has a slight decrease at the highest strain rate which is 3300 s⁻¹. The maximal value of strain hardening exponent is achieved under quasi-static loading.

Figure 5-7: Effect of strain rate on the hardening behavior 0.5vol% SiC_p/AZ31B at 25 °C

Table 5-2: Strain hardening exponent of 0.5vol% SiC_p/AZ31B at 25 °C

$\epsilon =$	0.01~0.06	0.07~0.11	0.13~0.19
0.001 s ⁻¹	0.7638	0.5119	0.0084
$\epsilon =$	0.01~0.03	0.04~0.07	N.A.
700±50 s ⁻¹	0.3141	0.6228	N.A.
$\epsilon =$	0.01~0.03	0.04~0.13	N.A.
1400±100 s ⁻¹	0.2814	0.4898	N.A.
$\epsilon =$	0.01~0.10	0.11~0.20	N.A.
3300±300 s ⁻¹	0.4374	0.3233	N.A.

The effect of temperature on the hardening behaviour of 0.5vol% SiC_p/AZ31B is investigated as well. Figure 5-8 shows hardening behavior of 0.5vol% SiC_p/AZ31B at 3300 s⁻¹ for various temperatures. There are roughly three regions of hardening behavior under room and elevated temperatures. The hardening exponent first increases and then decreases with strain increasing. There are only two regions of hardening behavior under temperature of 200 °C. The hardening exponent increases with strain increasing under this temperature. The values of strain hardening exponent for each temperature and strain range at 3300 s⁻¹ are given in

Table 5-3. Within the same strain range, the maximal value of the exponent is obtained at room temperature and the minimal value is obtained at the highest temperature except for the last strain range.

Figure 5-8: Effect of temperature on the hardening behavior of 0.5vol% SiC_p/AZ31B at 3300 s⁻¹

Table 5-3: Strain hardening exponent of 0.5vol% SiC_p/AZ31B at 3300 s⁻¹

$\epsilon =$	0.01~0.06	0.07~0.16	0.17~0.2
-30 °C	0.4816	0.5932	0.2963
$\epsilon =$	0.01~0.04	0.05~0.11	0.12~0.2
25 °C	0.5327	0.6513	0.3546
$\epsilon =$	0.01~0.11	0.12~0.2	N.A.
100 °C	0.5225	0.4434	N.A.
$\epsilon =$	0.01~0.10	0.11~0.14	0.15~0.2
200 °C	0.4311	0.4148	0.5338

5.4 Energy absorption capability

To quantify the energy absorption capability of 0.5vol% SiC_p/AZ31B and to see how it changes with temperature under quasi-static, the energy absorbed by specimens made of 0.5vol% SiC_p/AZ31B are calculated and plotted against homologous temperature which has been already defined in Equation 4.5. The melting temperature of 0.5vol% SiC_p/AZ31B is taken the same as AZ31B which is 893K. As the specimens under other strain rates were not broken, the energy absorption is only calculated for strain rate of 0.001 s⁻¹ and 3300 s⁻¹. The results are given in Figure 5-9. The material can absorb more energy when the temperature is lower. The decrease of energy absorbed under quasi-static loading is about 64.9% from room temperature to the highest temperature, while the decrease under 3300 s⁻¹ is roughly 16.6%.

Figure 5-9: Energy absorption capability of 0.5vol% SiC_p/AZ31B

5.5 Constitutive modelling

In the present study, the Johnson-Cook model is used to model the constitutive response of 0.5vol% SiC_p/AZ31B. The estimated values for the constitutive parameters as well as the lower and upper bounds of 95% confidence are given in Table 5-4. The constitutive parameters required by the Johnson-Cook model are fit to the experimental results using a MATLAB built-in curve fitting tool based on nonlinear regression analysis. The model fitted curves at different strain rates and temperatures are shown in Figure 5-10. The experimental data considered were reduced to (i) after the constant strain rate was achieved and (ii) before the stress reached the peak compressive stress. Although Johnson-Cook model was proposed originally for metals, it can be observed that reasonable agreement with the experimental data of 0.5vol% SiC_p/AZ31B is obtained. The reason might be that the nanoparticles distribute almost homogeneously

in the base alloy and the volume fraction of nanoparticles is very small. Overall, better agreement with experimental data is obtained at higher temperature. The Johnson-Cook model only captures constant log-linear strain rate sensitivity while in fact it changes with strain rate.

Table 5-4: Parameters of the Johnson-Cook model for 0.5vol% SiC_p/AZ31B

Parameter	Estimate	95% Confidence	
		Lower bound	Upper bound
A[MPa]	105	98	116
B[MPa]	1500	1430	1560
n	0.8215	0.7900	0.8405
C	0.015	0.014	0.016
m	0.79	0.76	0.81

Figure 5-10: Experimental and Johnson-Cook fitted curves at different strain rates and temperatures

Chapter 6 Compressive mechanical behaviour of 1.0 vol.% SiC_p/AZ31B

The experimental results of AZ31B reinforced with 1.0 vol.% SiC nanoparticles are presented and discussed in this chapter. Tests were performed at strain rates ranging from 10^{-3} s^{-1} to 3300 s^{-1} and temperatures between $-30 \text{ }^{\circ}\text{C}$ and $200 \text{ }^{\circ}\text{C}$. In each testing condition, five tests were carried out to ensure the reproducibility. The data shown in this chapter are the representative results of at least three consistent tests under each condition. The comparison between AZ31B, 0.5 vol.% SiC_p/AZ31B and 1.0 vol.% SiC_p/AZ31B is shown in this chapter as well.

6.1 Effect of strain rate

Figure 6-1 shows strain rates obtained in SHPB tests on 1.0vol% SiC_p/AZ31B specimens. The shapes of strain rate-time curves are similar to the shape of AZ31B. The strain rate begins with sharp increase then decreases slowly for a long range and finally decreases rapidly to zero. The strain rate-time curve is more likely to have a plateau region at higher strain rates. The average strain rate values shown in the figure are average values of the plateau portion of strain rate vs. time curves.

Figure 6-1: Sample strain rates measured in SHPB tests on 1.0vol% SiC_p/AZ31B

To illustrate the strain rate dependence of the mechanical behavior of 1.0vol% SiC_p/AZ31B, the true stress-strain curves of the material at temperatures of -30 °C, 25 °C, 100 °C and 200 °C are plotted in Figure 6-2. Generally speaking, the flow stress increases with strain rate increasing. The lowest values of both yield strength and flow stress at 5% strain are achieved at 0.001 s⁻¹ under room temperature as can be seen in Table 6-1. This phenomenon can be applied to other temperatures. The highest values of yield strength and flow stress at 5% strain under all temperatures are achieved at the highest strain rate which is 3300 s⁻¹. There is a large increase of 75% in yield strength with strain rate increasing from 710 s⁻¹ to 3183 s⁻¹ under room temperature. The increase of flow stress at 5% strain is 52.6%. The difference between stress-strain curves under quasi-static and dynamic loading is larger at higher temperature. It can be noted that before strain of 0.05, for a given strain before unloading, the corresponding stress obtained in high strain rates is always higher than quasi-static tests. The ultimate

compressive strength obtained at 0.001s^{-1} is 51.8% lower than the one obtained at 3183s^{-1} as shown in Figure 6-2(b).

Figure 6-2: True stress-strain curves of 1.0vol% SiC_p/AZ31B at different strain rates for different temperatures

Table 6-1: Compressive strength properties of 1.0vol% SiC_p/AZ31B at 25 °C

1.0vol% SiC _p /AZ31B					
Strain rate [s^{-1}]	10^{-3}	710	1377	2350	3183
0.2% Offset yield strength [MPa]	65 ± 3	80 ± 3	100 ± 3	140 ± 3	140 ± 3

Flow stress at 5% strain [MPa]	140±3	190±3	220±5	290±5	290±5
Ultimate compressive strength/Peak compressive strength* [MPa]	260±5	260±5*	420±5*	505±5*	540±5
Failure strain/Maximum strain reached	0.175±0.00 2	0.074±0.00 1	0.135±0.00 1	0.220±0.00 2	0.212±0.00 2

*Failure of specimens didn't occur. This is the maximum stress reached during testing.

Figure 6-3 shows curves of relationship between $\ln(\text{stress})$ and $\ln(\text{strain rate})$ calculated for $\epsilon=0.02, 0.04$ and 0.06 under four temperatures. The difference of the slopes indicates the variation of strain rate sensitivity m . Strain rate sensitivity of this material at different strain levels shows similar trend at each temperature. Strain rate sensitivity increases from 0.001 s^{-1} to 1400 s^{-1} , while it decreases from 1400 s^{-1} to 3300 s^{-1} . The decrease of strain rate sensitivity at higher strain rate is more significant at higher temperature.

Figure 6-3: Strain rate sensitivity of 1.0vol% SiC_p/AZ31B

6.2 Effect of temperature

Temperature effect on compressive true stress-strain curves for strain rates of 0.001 s^{-1} , $700 \pm 50 \text{ s}^{-1}$, $1400 \pm 100 \text{ s}^{-1}$ and $3300 \pm 300 \text{ s}^{-1}$ is shown in Figure 6-4. At a constant strain rate, the flow stress decreases with temperature increasing although the difference of flow stress at 700 s^{-1} is not obvious. Temperature has more significant effect on stress-strain relationship under quasi-static loading than under dynamic loading. Within dynamic range, temperature has the most obvious influence on stress-strain curves at the highest strain rate. Stress-strain curves under 200 °C is the most different from the curves under other temperatures at each strain rate. The effect of temperature on the

flow stress at 5% strain is shown in Figure 6-5. It can be noted that the flow stress at 5% strain decreases with temperature increasing. The highest value is achieved at the lowest temperature (either -30 °C or 25 °C), while the lowest value is achieved at the highest temperature, i.e. 200 °C. Temperature has the most significant effect on flow stress at strain rate of 0.001 s^{-1} . Within high strain rate range, temperature has more influence on flow stress at higher strain rate. There is a 35.7% decrease in flow stress at 5% strain from -30 °C to 200 °C at 3300 s^{-1} , while the decrease at 700 s^{-1} is only 20%. The effect of temperature on failure strain at strain rate of 0.001 s^{-1} and 3300 s^{-1} is shown in Figure 6-6. It can be seen that temperature has more influence at high strain rate. The increase of failure strain at 3300 s^{-1} is about 26% when temperature increases from 25 °C to 200 °C, while there is a slight decrease at 0.001 s^{-1} when temperature increases from 25 °C to 200 °C.

Figure 6-4: True stress-strain curves of 1.0vol% SiC_p/AZ31B at different temperatures for different strain rates

Figure 6-5: The effect of temperature on the flow stress at 5% strain at all strain rates

Figure 6-6: The effect of temperature on failure strain at 0.001 s⁻¹ and 3300±300 s⁻¹

The temperature sensitivity with variation of true strain at different high strain rates under a wide range of temperature is shown in Figure 6-7. It can be noted from this

figure that temperature sensitivity increases when strain increases at 3300 s^{-1} , while the tendency is not applicable at the other strain rates. Within a temperature range, the value of temperature sensitivity becomes larger when strain rate increases especially at larger strain.

Figure 6-7: Temperature sensitivity-true strain curves at different strain rates and temperatures

Figure 6-8 exhibits different macroscopic deformation or failure modes of AZ3B reinforced with 1.0vol. % SiC nanoparticles. This material displays different deformation and failure modes from 0.001 s^{-1} to 3300 s^{-1} under different temperatures. At all temperatures specimens tested at 700 and 1400 s^{-1} strain rate keep intact. At 243K, specimens are broken into pieces under strain rate of 3300 s^{-1} . At room temperature, specimens tested at 0.001 s^{-1} and 3300 s^{-1} are failed with multiple cracks at about 45° angle to the loading direction. Examination of dynamic fracture surfaces at 3300 s^{-1} revealed shear bands which can be attributed to twinning shear (see Figure 6-9). At 373 and 473K, specimens deform plastically with some fine cracks observed

under 0.001 and 3300 s⁻¹ strain rate. Failure modes in a typical ductile fracture characteristic for room and high temperature and a typical brittle behavior for low temperature can be noted from Figure 6-8.

Figure 6-8: The macroscopic failure of specimens at strain rates of (a) 0.001 s⁻¹ (b) 3300 s⁻¹

Figure 6-9: Fractograph of 1.0vol% SiC_p/AZ31B tested at 3300 s⁻¹ under room temperature

6.3 Strain hardening

Figure 6-10 shows the strain hardening behaviour of 1.0vol% SiC_p/AZ31B at various strain rates under room temperature (25 °C). Strain hardening exponent n is graphically the slope of the line fitted to true stress-strain curve on log-log scale. The hardening exponent depends on the strain. The relationship between $\log$ (true stress) and $\log$ (true strain) is almost linear under quasi-static loading, while the linear relationship between $\log$ (true stress) and $\log$ (true strain) exists only within a certain region at high strain rates. Within dynamic range, the hardening exponent first increases and then decreases with strain increasing. The hardening exponent decreases with strain rate increasing as can be seen in Table 6-2.

Figure 6-10: Effect of strain rate on the hardening behavior of 1.0vol% SiC_p/AZ31B

Table 6-2: Strain hardening exponent of 1.0vol% SiC_p/AZ31B at 25 °C

$\epsilon =$	0.014~0.02	0.03~0.16	N.A.
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Compressive mechanical behavior of 1.0 vol.% SiC_p/AZ31B

0.001 s ⁻¹	1.001	008319	N.A.
$\epsilon =$	0.014~0.02	0.03~0.07	N.A.
700±50 s ⁻¹	0.3981	0.6943	N.A.
$\epsilon =$	0.014~0.03	0.04~0.08	0.09~0.13
1400±100 s ⁻¹	0.3717	0.6896	0.5580
$\epsilon =$	0.014~0.03	0.04~0.11	0.13~0.20
3300±300 s ⁻¹	0.3911	0.5440	0.2734

The effect of temperature on the hardening behavior of 1.0vol% SiC_p/AZ31B is also investigated. Figure 6-11 shows hardening behavior of 1.0vol% SiC_p/AZ31B at 3300 s⁻¹ for various temperatures. The relationship between log (true strain) and log (true stress) is almost linear under 200 °C. The variation of hardening exponent with strain is more obvious under lower temperature. The exponent first increases and then decreases with strain increase under -30 °C, 25 °C and 100 °C. The values of hardening exponent for each temperature and strain range are given in Table 6-3. For the same strain range, the maximal value is achieved at the lowest temperature (-30 °C) while the minimal value is achieved at room temperature.

Figure 6-11: Effect of temperature on the hardening behavior of 1.0vol% SiC_p/AZ31B under various temperatures at 3300 s^{-1}

Table 6-3: Strain hardening exponent of 1.0vol% SiC_p/AZ31B at 3300 s^{-1}

$\epsilon =$	0.01~0.04	0.05~0.11	0.13~0.17
-30 °C	0.4588	0.6081	0.1866
$\epsilon =$	0.01~0.04	0.05~0.13	0.15~0.20
25 °C	0.4019	0.5360	0.2379
$\epsilon =$	0.01~0.13	0.15~0.20	N.A.
100 °C	0.4505	0.3393	N.A.
$\epsilon =$	0.01~0.17	0.18~0.20	N.A.,
200 °C	0.4328	0.6203	N.A.

6.4 Energy absorption capability

To quantify the energy absorption capability of 1.0vol% SiC_p/AZ31B and to see how it changes with temperature under quasi-static and dynamic loading, the energy absorbed by 1.0vol% SiC_p/AZ31B specimens are calculated and plotted against homologous temperature which has already been defined in Equation 4.5. The melting temperature is taken the same as AZ31B which is 893K. The results are given in Figure 6-12. As the specimens are only broken into pieces at 0.001 s⁻¹ and 3300 s⁻¹, the energy absorption of the material was only calculated for these two strain rates. The nanocomposite can absorb more energy under dynamic loading as the other two materials. Temperature has less effect on the energy absorption at high strain rate. There is a decrease of 18.6% when temperature increases from 25 °C to 200 °C at 3300 s⁻¹, while the decrease at 0.001 s⁻¹ is much more significant in the same temperature range.

Figure 6-12: Energy absorption capability of 1.0vol% SiC_p/AZ31B

6.5 Constitutive modelling

In the present study, the Johnson-Cook model is used to model the constitutive response of 1.0vol% SiC_p/AZ31B. The estimated values for the constitutive parameters as well as the lower and upper bounds of 95% confidence are given in Table 6-4. The constitutive parameters required by the Johnson-Cook model are fit to the experimental results using a MATLAB built-in curve fitting tool based on nonlinear regression analysis. The model fitted curves at different strain rates and temperatures are shown in Figure 6-13. The experimental data considered were reduced to (i) after the constant strain rate was achieved and (ii) before the stress reached the peak compressive stress. Although Johnson-Cook model was proposed originally for metals, it can be observed that reasonable agreement with the experimental data of 1.0vol% SiC_p/AZ31B is obtained. The reason might be that the nanoparticles distribute almost homogeneously in the base alloy and the volume fraction of nanoparticles is very small. Overall, better agreement with experimental data is obtained at higher temperature and higher strain rate. As the volume fraction of SiC nanoparticles of the material investigated in this chapter is a bit higher than 0.5vol% SiC_p/AZ31B, the fitting for 1.0vol% SiC_p/AZ31B is a bit worse than that for 0.5vol% SiC_p/AZ31B especially at lower strain rate.

Table 6-4: Parameters of the Johnson-Cook model for 1.0vol% SiC_p/AZ31B

Parameter	Estimate	95% Confidence	
		Lower bound	Upper bound
A[MPa]	100	90	114
B[MPa]	1315	1405	1280
n	0.7155	0.7011	0.7405
C	0.0130	0.0100	0.01876
m	0.8191	0.7900	0.8598

Figure 6-13: Experimental and Johnson-Cook fitted curves at different strain rates and temperatures

6.6 Effect of volume fraction of silicon carbide nanoparticles

Compressive true stress-strain plots for magnesium alloy AZ31B and its nanocomposites at various temperatures under quasi-static and high strain rate loading are shown in Figure 6-15, Figure 6-16, Figure 6-17 and Figure 6-17 respectively. It can be noted from Figure 6-14 that both AZ31B reinforced with 0.5vol.% SiC and AZ31B reinforced with 1.0vol.% SiC have higher yield strength and maximum compressive strength than their base ally under -30 °C. Two kinds of AZ31B nanocomposite have similar stress-strain relationship at lower strain rates, while higher ultimate compressive strength with higher volume fraction is observed at the highest strain rate (3300 s^{-1}).

Figure 6-14: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at -30 °C

Although AZ31B nanocomposites have higher flow stress than AZ31B under room temperature under dynamic loadings as can be seen in Figure 6-15, both AZ31B nanocomposites have lower flow stress than AZ31B under quasi-static loading. Besides, AZ31B reinforced with 1.0vol.% SiC doesn't show obviously higher strength than AZ31B reinforced with 0.5vol.% SiC at both strain rates of 700 s^{-1} and 1400 s^{-1} under room temperature.

Figure 6-15: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 25 °C

AZ31B nanocomposites display higher strength under dynamic loading at 100 °C, while the stress-strain relationship of AZ31B and its nanocomposites is similar under quasi-static loading as shown in Figure 6-16.

Figure 6-16: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 100 °C

AZ31B reinforced with 1.0vol.% SiC has the highest strength among the three materials at 700 s^{-1} and 1400 s^{-1} at 200 °C though AZ31B reinforced with 0.5vol.% SiC has the highest strength under quasi-static loading and the highest strain rate instead of AZ31B reinforced with 1.0vol.% SiC as shown in Figure 6-17. From the figures discussed above, AZ31B nanocomposites have higher strength than AZ31B of all temperatures under dynamic loading. However, this doesn't apply to their behaviors under quasi-static loading. Furthermore, higher volume fraction of silicon carbide nanoparticles contributes more to the increase of strength at higher strain rate.

Figure 6-17: True stress-strain curves from quasi-static and high strain rate tests on AZ31B and its nanocomposites at 200 °C

As the specimens are only fractured at 3300 s⁻¹ within high strain rate range, the comparison of stress-strain relationship between AZ31B, AZ31B reinforced with 0.5vol.% SiC and AZ31B reinforced with 1.0vol.% SiC at 3300 s⁻¹ is shown in Figure 6-18. It can be noted that AZ31B nanocomposites have higher yield strength and ultimate compressive strength/peak compressive strength than its base alloy under all temperatures. The effect of silicon carbide nanoparticles on the increase of strength is more obvious under room temperature than other temperatures. However, the addition of nanoparticles has negative effect on ductility. AZ31B has better ductility than its nanocomposite under all temperatures. However, strength of the material doesn't increase with increasing the volume fraction of nanoparticles under all temperatures. The energy absorption capability of these three materials is calculated for 0.001 s⁻¹ and

3300 s⁻¹ as shown in Figure 6-19. It is obtained that AZ31B can absorb more energy after the addition of SiC nanoparticles under dynamic loading although AZ31B nanocomposites don't show better energy absorption capability at all temperatures under quasi-static loading. The effect of nanoparticles on energy absorption capability is most obvious under room temperature under dynamic loadings. Higher energy absorption of AZ31B nanocomposite indicates better resistance against impact or crash. It can be seen that higher volume fraction of nanoparticles doesn't result in better energy absorption capability.

Figure 6-18: Comparison of true stress-strain curves of AZ31B and AZ31B reinforced with 1.0vol.% SiC at 3300 s⁻¹

Figure 6-19: Energy absorption of AZ31B and its nanocomposites at (a) 0.001/s and (b) 3300/s

Reduction in grain size associated with the presence of nanoparticles can be observed in Figure 6-20. The grain refinement can be attributed to the ability of the nanoparticles to nucleate recrystallized grains and inhibit grain growth [241, 242].

These microstructural characteristics may be the reasons for the improved mechanical properties of AZ31B nanocomposites compared to their monolithic counterpart.

Figure 6-20: Optical micrographs showing grain characteristics of (a) AZ31B (b) 1.0vol% SiC_p/AZ31B

Chapter 7 Conclusions and recommendations for future work

7.1 Conclusions

The aim of the current research is to investigate mechanical behaviour of magnesium alloy AZ31B and its nanocomposites with different volume fractions of silicon carbide nanoparticles. Specimens made from AZ31B, AZ31B reinforced with 0.5 vol.% SiC nanoparticles and AZ31B reinforced with 1.0 vol.% SiC nanoparticles were tested under quasi-static and dynamic loading between temperature of -30 °C and 200 °C. In summary, the following conclusions can be drawn from the work presented in previous chapters.

1. Mechanical properties of AZ31B are strain rate and temperature dependent. For a given temperature, the flow stress increases with increasing strain rate within dynamic range. However, the highest value of flow stress at 5% strain is achieved at 2257 s⁻¹ instead of 3200 s⁻¹ at 25 °C. For a given strain before unloading, the corresponding stress obtained in high strain rate tests is always higher than quasi-static tests. The difference of true stress-strain curves between quasi-static and dynamic loading condition become more significant with the increase in temperatures. AZ31B shows different strain rate sensitivity at low, room and high temperature. Strain rate sensitivity at strain levels between 4% and 6% shows similar trend at each temperature, while strain rate sensitivity at 2% strain is quite different from other strain levels. At a constant strain rate, the flow stress decreases with temperature increasing. The stress-strain relationship between -30 °C(243K) and 25 °C(298K) only show small

difference, while significant thermal softening is seen at 100 °C(373K) and 200 °C(473K). Temperature has the most significant effect at quasi-static loading. At a constant strain rate, the temperature sensitivity (up to strain of 6%) decreases as temperature increases. Within the given temperature range, the temperature sensitivity generally increases as strain rate increases. However, there is not a consistent tendency for temperature sensitivity with variation of true strain. The effect of strain rate and temperature on strain hardening of AZ31B is also studied. It is found that the hardening exponent decreases slightly with strain rate increasing. Temperature doesn't have as much effect on strain hardening behaviour as the strain rate. The energy absorption of the alloy is calculated for 0.001 s^{-1} and $3300 \pm 300 \text{ s}^{-1}$. The effect of temperature on the energy absorption is less significant at high strain rate, especially when the temperature is higher than room temperature (25 °C).

2. The experimental results of AZ31B reinforced with 0.5 vol.% SiC nanoparticles are presented and discussed. The flow stress increases with increasing strain rate under all temperatures. For a given strain before unloading, the corresponding stress obtained in high strain rate tests is always higher than quasi-static tests. For a given stress, the corresponding strain obtained in high strain rate tests is much smaller than quasi-static tests. The stress at 5% strain undergoes an increase of 31.8% with strain rate increasing from 747 s^{-1} to 2200 s^{-1} at 25 °C. Strain rate sensitivity decreases with strain rate increasing at -30 °C and 200 °C, while it increases with strain rate increasing at 25 °C and 100 °C. At a constant strain rate, the flow stress decreases with temperature increasing. The stress-strain relationship between -30 °C and 25 °C only show small difference, while significant thermal softening is seen at 100 °C and 200 °C. At the constant strain rate, the temperature sensitivity

increases as strain becomes larger. The most obvious increase happens at 1400 s^{-1} in the temperature range of $25\text{ }^{\circ}\text{C}\sim 200\text{ }^{\circ}\text{C}$. However, temperature sensitivity shows different variation tendency against change of strain rate within different temperature range. The hardening exponent has a slight decrease at the highest strain rate which is $2200\pm 200\text{ s}^{-1}$. The highest value of strain hardening exponent is achieved under quasi-static loading. The effect of temperature on the hardening behaviour of 0.5vol% SiC_p/AZ31B is investigated as well. Within the same strain range, the highest value of the exponent is obtained at the lowest temperature and the lowest value is obtained at the highest temperature. Regarding energy absorption, only 0.001 s^{-1} is considered as the specimens under dynamic loading are not broken. The material can absorb more energy when the temperature is lower.

3. With respect to AZ31B reinforced with 1.0 vol.% SiC nanoparticles, the flow stress increases with strain rate increasing as AZ31B and AZ31B/0.5%SiC show. The lowest values of both yield strength and flow stress at 5% strain are achieved at 0.001 s^{-1} under room temperature. The highest values of yield strength and flow stress at 5% strain under all temperatures are achieved at the highest strain rate which is $3300\pm 300\text{ s}^{-1}$. The difference between quasi-static stress and dynamic stress is larger at higher temperature. Strain rate sensitivity of this material at different strain levels shows similar trend at each temperature. Strain rate sensitivity increases from 0.001 s^{-1} to $1400\pm 100\text{ s}^{-1}$, while it decreases from $1400\pm 100\text{ s}^{-1}$ to $3300\pm 300\text{ s}^{-1}$. The decrease of strain rate sensitivity at higher strain rate is more significant at higher temperature. Temperature has more significant effect on stress-strain relationship under quasi-static loading than under dynamic loading. Within dynamic range, temperature has the most obvious influence on stress-strain curves at the

highest strain rate. Stress-strain curves under 200 °C is the most different from the curves under other temperatures at each strain rate. Temperature sensitivity increases when strain increases at 3300 s^{-1} , while the tendency is not applicable at the other strain rates. Within a temperature range, the value of temperature sensitivity becomes larger when strain rate increases especially at larger strain. This material displays different deformation and failure modes from 0.001 s^{-1} to $3300\pm300\text{ s}^{-1}$ under different temperatures. Within dynamic range, the hardening exponent first increases and then decreases with strain increasing. The hardening exponent decreases with strain rate increasing. The variation of hardening exponent with strain is more obvious under lower temperature. The exponent first increases and then decreases with strain increase under -30 °C, 25 °C and 100 °C. The nanocomposite can absorb more energy under dynamic loading as the other two materials. Temperature has less effect on the energy absorption at high strain rate.

4. The addition of silicon carbide nanoparticles can enhance the strength of the base alloy, while it slightly decreases the ductility of the material. It doesn't apply to all strain rates and temperature conditions that higher volume fraction of nanoparticles results in higher strength. But this happens more at higher strain rate. Though ductility becomes worse after adding ceramic nanoparticles, the enhancement of energy absorption capability still exists especially at highest strain rate.

7.2 Recommendations for future work

There are a couple of possible extensions from the present work which are listed below:

1. As only two kinds of nanocomposites have been studied, other volume fractions of nanoparticles can be used in AZ31B alloy. Strength, ductility and energy absorption of new nanocomposites will be studied to find out the optimal volume fraction of silicon carbide nanoparticles.
2. A relationship between mechanical properties and microstructural changes occurred of AZ31B reinforced with SiC nanoparticles can be built using Scanning Electron Microscopy.
3. Some explicit dynamic software such as ANSYS LSDYNA could be used to simulate high strain rate behaviour of AZ31B nanocomposites.

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