İSTANBUL TECHNICAL UNIVERSITY ★ INSTITUTE OF SCIENCE AND TECHNOLOGY

MECHANICAL PROPERTIES OF BORON CARBIDEPARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES

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ALÜMİNYUM MATRİSLİ BOR KARBÜR TAKVİYELİ KOMPOZİTLERİN MEKANİK DAVRANIŞLARININ İNCELENMESİ

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ABBREVIATIONS

XRD	**:** X-Ray Diffraction
LOM	**:** Light Optical Microscope
SEM	**:** Scanning Electron Microscope
UTS	**:** Ultimate Tensile Strength
MMC	**:** Metal Matrix Composites
EDS	**:** Energy Dispersive Spectroscopy

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ALÜMİNYUM MATRİSLİ BOR KARBÜR TAKVİYELİ KOMPOZİTLERİN MEKANİK DAVRANIŞLARININ İNCELENMESİ

ÖZET

Bu çalışmada, yüksek hacim oranlı (>% 50) B_4C partikül takviyeli Al alaşım matrisli kompozit malzemelerin mekanik özellikleri ve aşınma dirençleri incelenmiştir. Kompozit malzemeler, 7 mm çapında olup, gaz basınçlı infiltrasyon yöntemi ile üretilmiştir. İnfiltrasyon prosesi, 750 ^oC'de 765 kPa basınç altında yapılmıştır.

Kompozit malzemelerin aşınma davranışı, metal-metal ve metal-abrasif aşınma deney cihazı ile incelenmiştir. Deneyler, 28 N normal yük altında yapılmıştır. Deney sonuçları ağırlık kaybına göre değerlendirilmiştir. Metal-metal aşınma deneyleri, M2 kalite takım çeliğinden yapılmış disk üzerinde oda sıcaklığında kuru sürtünme koşullarında gerçekleştirilmiştir. Metal-abrasif aşınma deneyleri, oda sıcaklığı ile 600 °C arasındaki çeşitli sıcaklıklarda ve abrasif Al_2O_3 taneleri üzerinde yapılmıştır.

İncelenen kompozit malzemelerin mikroyapısal incelemeleri, B_4C takviye partiküllerin uçlarına yakın konumda bir miktar porozitenin mevcut olduğunu ortaya çıkarmıştır. Porozite hacim oranının matrise Mg ilavesi ile azalmıştır. Mikroyapısal karakterizasyon çalışmaları saf Al matriste AlB_{10} intermetaliğinin Al-%8 Mg alaşım matriste AlB_{10} , $B_{25}C$ ve $Mg_{0.5}Al_{14}$ oluştuğunu ortaya çıkarmıştır. İncelenen kompozit malzemelerin sertlik ve mukavemet değerleri Mg ilavesi ile artmıştır. Bununla beraber tokluk ve süneklik değerlerinde düşüş gözlemlenmiştir.

Kompozit malzemelerin metal-metal aşınma direnci, matrisin alaşımlandırılması ile artmıştır. Saf Al matriste şiddetli aşınma (severe wear) etkin aşınma mekanizması isen matrise Mg ilavesi ile aşınma mekanızması yumuşak aşınmaya (mild wear) dönüşmüştür. 8 wt% Mg içeren Al matrisli kompozitin aşınma yüzeyinde demir oksit tabakasının mevcudiyeti çıplak gözle görülebilir hale gelmiştir.

Metal-abrasive aşınma direnci matrise Mg ilavesi ile artmıştır. Sıcaklık arttıkça aşınma direnci azalmıştır. Bununla beraber Mg alaşımlı kompozit malzemelerde 300^oC den sonra aşınma direncinde belirgin bir artış olmuştur. Bu artış 500^oC den sonra tekrar düşme eğilimi göstermiştir.

MECHANICAL PROPERTIES OF BORON CARBIDE REINFORCED ALUMINUM MATRIX COMPOSITES

SUMMARY

In this thesis, mechanical and wear behaviour of high volume fraction (> 50 vol %) B_4C reinforced Al alloy matrix composites were investigated. Investigated composites having 7 mm diameter were fabricated by gas pressure infiltration technique. The infiltration process was carried out at 750 ^oC under a pressures of 1 765 kPa.

The wear behaviour of the composites was examined by metal-metal and metal-abrasive wear tests. Tests were carried out by applying a normal load of 28 N to the samples. Results of the wear tests were evaluated according to the weight loss of the samples. The metal-metal wear tests were performed on an unlubricated M2 quality tool steel disc (65 HRC) at room temperature. The metal-abrasive wear tests were conducted at various temperatures (20–600°C) by rubbing the samples on abrasive Al_2O_3 grains.

Microstructural examination revealed presence of porosities located near the intersecting points of the B_4C particles and intermetallic compounds precipitated in the matrix. The volume fraction of the porosity decreased with alloying the matrix with Mg. In pure Al matrix AlB_{10} and in Al-8%Mg matrix AlB_{10}, B25C ve $Mg_{0.5}Al_{14}$ type intermetallic compounds were observed. The strength of the investigated composites increased with increasing the amount of the Mg in the matrix. Alloying of the matrix decreased both ductility and impact resistances.

Metal–metal wear resistance of the investigated composites increased upon alloying of the matrix with Mg. The dominant wear mechanism which was severe wear for pure Al matrix composite, transformed to mild wear in Al-Mg alloy matrix composite. The worn surface of Al-8%Mg alloy matrix composite was covered with iron oxide layer which was easily be detected by a unaided.

Metal-abrasive wear resistance of the composites increased with increasing Mg content of the matrix. At elevated temperatures composites exhibited lower wear resistance than room temperature. Composites containing more than 1 wt Mg % in their matrix exhibited a slight increment in wear resistance in temperature range between 300 and 500°C due to dissolution of intermetallic compounds.

1. INTRODUCTION

The energy, communication, transportation, aeronautics and space industries require new materials with further superior properties than the present due to the economical difficulties, reliability and the ecological concerns. It is very difficult to meet these expects of hi-tech systems with the present traditional materials [1]. In recent years new investigations have been established on carbon-carbon and metal-matrix composites to manage to cope with these new requirements. Although these materials' production process is quiet expensive and low volume production for the industries require high technology like space-aeronautics-military applications, industries like transportation and energy sectors with high volume and low cost production applications render these materials wide spread in these fields. In spite of the information about these materials is quiet old; the applications are getting common in recent 15-20 years [2].

Metal-matrix composites consist of at least one metal and a reinforcement material as continuous fiber, intermetallics particles, compounds, oxide, carbide or nitride in order to achieve requirements and expected properties which can not be met by single compound materials [3]. Nowadays production of metal-matrix composites by liquid phase process is convenient in the production of metal matrix composites reinforced with particles. The driving force behind the development of metal matrix composites has been the attractive mechanical and physical properties and enhanced elevated temperature capabilities [4]. In addition, aluminum matrix composites reinforced with ceramic particles or whiskers have received considerable attention because they can be formed by standard metalworking practices. Apart from improved mechanical properties, other controlling attributes, such as coefficient of thermal expansion and wear resistance, are greatly improved by the addition of ceramic particles [5].

Ductile phases reinforced high-volume fraction of ceramic (>50%) are emerging as important class of materials with potential for structural applications where high

specific modulus, strength, and superior wear characteristics are desirable. However, the commercialization of these composites is limited principally due to the cost and the reliability of the final products [3].

With good properties, such as high hardness, high melting point, good thermal conductivity and good electrical conductivity, boron carbide ceramics are excellent candidates for neutron absorption materials, wear resistant materials, electrode materials and cutting tools [1]. Among the outstanding physical and mechanical properties of boron carbide is its hardness, which is second after diamond and c-BN. This specific property comes along with other attractive properties such as high impact and wear resistance, low density, high melting point, and excellent resistance to chemical agents as well as high capability for neutron absorption. However, its extreme sensitivity to brittle fracture (K_{IC}=3.2–3.7 MPa $m^{1/2}$) and the difficulties involved in fabricating dense B_4C materials have limited its use in industrial applications. These problems can be significantly reduced by the production of B_4C-based composites, especially B_4C-metal composites [6].

Aluminium metal matrix composites are being considered as a group of new advanced materials for its light weight, high strength, high specific modulus, low co-efficient of thermal expansion and good wear resistance properties. Combination of these properties is not available in a conventional material [3]. The use of Al metal matrix composite has been limited in very specific applications such as aerospace and military weapon due to high processing cost. For applications at low temperatures, aluminum or aluminum alloys is a material of choice. Suggested applications for B_4C–Al composites include its use as a structural neutron absorber, armour plate materials, and as a substrate material for computer hard disks [7].

The Al-B_4C system is of interest because of the potential for enhancement of mechanical properties and thermal stability of the matrix by the incorporation of B_4C particles. B_4C has lower density than other common commercial reinforcements, such as SiC, Al_2O_3, resulting in composites with higher specific stiffness (stiffness per unit weight) [5].

An important issue in the production of metal matrix composites is the chemical compatibility between the matrix and the reinforcement, particularly when using

liquid metal processes such as infiltration. Boron carbide-based ceramic–metal composites may, in principle, provide a useful combination of the elevated hardness of the ceramic with some toughness due to the metal component. One approach for the fabrication of such composites is based on the infiltration of the ceramic preform having a controlled fraction of porosity with a molten metal or alloy. Pressure infiltration process is one of the most important metal matrix composite production techniques because of its high capacity and high speed production opportunity and final shaping tolerances. Al, Mg and their alloys, as matrix materials in metal-matrix composites, are used commonly in pressure infiltration technique. Among these materials the Al alloys are used commonly because of their high mechanical properties, low electrical conductivity, low density, high corrosion resistance and their low cost according to other light metals such as Mg. 2xxx (AlCuMg), 5xxx (AlMg), 6xxx (AlMgCuSi), 7xxx (AlZnMgCu), 8xxx (AlLi) Al alloys used in composite production widely [6-8].

So far, the produced composites have the ratio of % 20–30 reinforcement material. During the literature survey there could not be come across with high ratios of reinforcement material (> %50) composites with Al matrix.

In this study, the aim is to examine the mechanical behaviours of Aluminum metal-matrix composites, with Al-Mg alloy matrix and high ratio reinforcement material, B4C, respect to Mg element changing in the range of % 1–8.

2. COMPOSITE MATERIALS

A composite, in general, is defined as a combination of two or more components differing in form or composition on a macroscale, with two or more distinct phases having recognisable interfaces between them [9].

Proper combination of materials into composites give rise to properties with transcends those of the constituents, as a result of the principle combined action. Materials of biological origin are generally composites [13]. Bone, for instance, achieves its combination of lightness and strength by combining crystals of apatite (a compound of calcium) with fibres of the protein collagen, whereas wood contains cellulose fibres surrounded by lignin and hemicellulose. Crushed rock aggregate used in concrete produces a composite structure, which reduces the cost and helps to improve the compressive strength. Structural weight savings (while retaining the reliability and strength), are achieved for aerospace, rocket applications etc. by the use of composite materials (Table 2.1) [10,13].

Composites are produced to optimise material properties, mechanical (mainly strength), and chemical and/or physical properties. In the latter, optimisation of thermal (thermal expansion, thermal conduction, specific heat, softening and melting points) as well as electrical (electrical conductivity, dielectric loss), as well as optical and acoustical properties can be noted. Since the early 1960s, there has been an increasing demand for materials that are stiffer and stronger, yet lighter in aeronautic, energy, civil engineering and in various structural applications [1].

Composites usually consist of a reinforcing material embedded in a matrix (binder). The effective method to increase the strength and to improve overall properties is to incorporate dispersed phases into the matrix, which can be an engineering material such as ceramic, metal or polymer [3,11-12]. Hence, ceramic matrix composites, metal matrix composites or polymer matrix composites or carbon matrix composites or even hybrid composites are obtained. In a composite, matrices, in general, are of

low modulus, while reinforcing elements are typically 50 times stronger and 20-150 times stiffer. Metal matrix composites and ceramic matrix composites structures are developed to provide rather high temperature applications (>316 oC), where polymer matrix composites are usually inadequate [12,13]. Furthermore, since metals are more conductive (electrically and thermally) metal matrix composites are also used in heat dissipation and electronic transmission applications. Each matrix type has different impact on the processing technique.

Table 2.1: Example of Composite Materials [10]

Matrix	Constituents	Areas of Application
1. Organic Matrix Comp.		
Paper, cardboard	Resin/fillers/cellulose	Printing, packaging
Particle panels	Resin/wood shavings	Woodwork
Fiber panels	Resin/wood fibers	Building
Coated canvas	Pliant resins/cloth	Sports/building
Impervious materials	Elastomers/bitumen	Roofing, earthworks
Tires	Rubber/canvas/steel	Automobiles
Laminates	Resin/glass fibres	Multiple areas
Reinforced plastics	Resins/Micro-spheres	
2. Mineral Matrix Comp.		
Concrete	Cement/sand/gravel	Civil Engineering,
Carbon-carbon comp.	Carbon/carbon fibres	Aviation, space, sports, biomedicine,
Ceramic Comp.	Ceramic/ceramic fibres	Thermomechanical items
3. Metal matrix Comp.	Aluminum/boron fibres	Space
	Aluminum/carbon fibres	
4. Sandwiches		
Skins	Metals, laminates, etc.	Multiple Areas
Cores	Foam, honeycombs, etc	

Composites are usually used for their structural properties where the most commonly employed reinforcing component is in particulate or fibrous form and hence the definition above can be restricted to such systems that contain continuous/discontinuous fibre or particle reinforcement, all in a continuous supporting core phase, the matrix [10,14]. A reinforcement phase usually exists with substantial volume fractions (10% or more). Generally, three types of composites can be described as: (a) particle strengthened, (b) discontinuous fibre reinforced and (c) continuous fibre reinforced composites; depending on the size and aspect ratio and volume fractions of reinforcing phases. In these, the function of each component can be different: in particle strengthened composites, the matrix bears the load and small dispersed particles obstruct the motion of dislocations in the matrix; and the load is distributed between matrix and particles [10,15]. In addition to these types of composites, one should also note the existence of another group of composite system, laminar composites (or simply laminates); where reinforcing agents are in the form of sheets bonded together and are often impregnated with more than one continuous phase in the system. The classification of composites mentioned above can be narrowed into two groups: macrocomposites and microcomposites. This classification depends on whether there are one or more dispersed distinguishable phases and on whether there is more than one continuous phase present (for macrocomposites), or whether all dispersed phases are between 10-1000 nm in size and there is only one continuous phase (for microcomposites). If the size of reinforcing component of a microcomposite is in the form of 'quantum dots' (i.e., being much smaller than 25 nm) then a new specific term is assigned to it by calling it a nanocomposite. The term flexible composite is used to identify composites based on elastomeric polymers where the usable range of deformation is much larger than the conventional thermoplastic or thermoset composites [16].

High tensile and high modulus fibres (such as carbon, boron, silicon carbide and alumina) emerged in the 1970s, and were used to reinforce high-performance polymer, metal or ceramic matrices. A new group of advanced composite materials were than developed which were, in general, extremely strong and stiff. The matrix is one of the key factors reach to proper advanced composite materials structures [13]. In order to have advanced material properties, firstly densities of matrices should be as small as possible with as high as application temperatures for the

material. There usually exist a relationship between density and service temperature for different materials, as presented in Figure 2.1. As the figure shows, titanium and steel are not considered as advanced because of their relatively high densities. The arrow in the figure indicates the trend for advanced materials which has its peak at high application temperatures and low densities (two important criteria for the next generation spacecrafts being lighter and faster); hence silicone carbide ceramics and carbon-carbon composites are expected to be examples of such advanced materials [13].

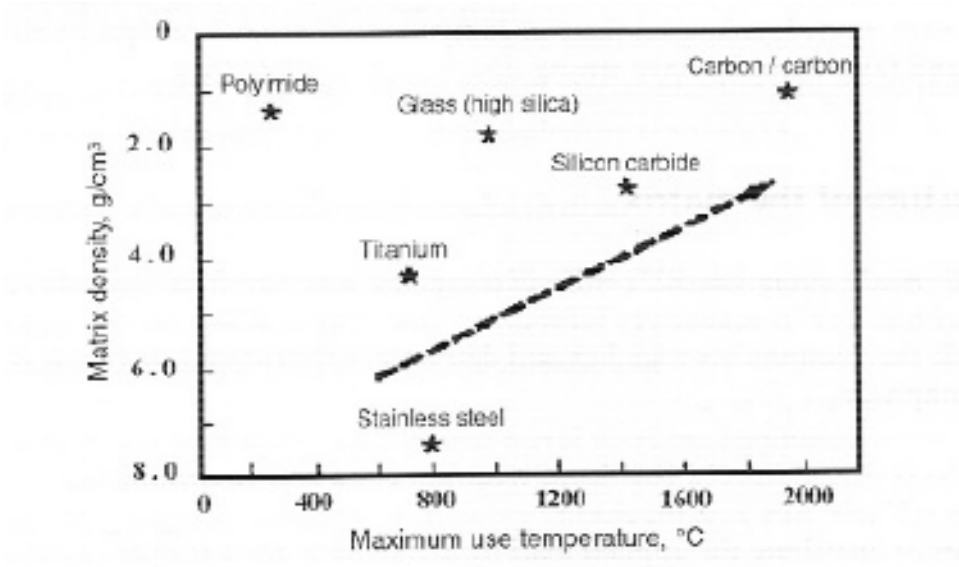


Figure 2.1: Densities versus Maximum Use Temperature for Some Materials [13]

The unique properties of advanced composite materials make them special for different applications. For example, advanced polymer composites are not only lightweight, but also offer excellent strength, stiffness and design versatility, which are particularly important in aerospace applications [13,18]. Some speciality polymers also have good chemical resistance and dielectric strength, like aramids, which gave rise to their use as electrical insulators in generators and transformers. Advanced ceramic materials provide a unique combination of high-temperature resistances, in addition to excellent wear and corrosion resistance and dimensional stability which are particularly important for parts which will be subjected to wear and cutting tools. Advanced metal composites and alloys obtained by rapid cooling of the melt have better strength and better electrical properties as well as improved corrosion resistance and enhanced magnetic properties [13-19].

The interfaces and interphases between different components in the composites, which is the boundary surface with a discontinuity, have a vital importance in determining the structural properties of the composite. The interfaces and interphases are expected to be proper, i.e., the interaction and adhesion between the components should be at the optimum level in order to distribute the load that is borne by the composite evenly [13,9].

Finally one should mention the three main forces that may affect the competition between composites and traditional engineering materials, both with similar mechanical properties: they are the cost, reliability and degree of complexities involved. The cost barrier in these is usually overcome by mass production and the degree of complexity is certainly more critical for composites which is due to the unisotropy existing (at least at microscale) for these structures. It is known that, in composites, thermoelastic properties as well as strength and failure modes have strong directional dependencies [13].

2.2 Structure of Matrix

The matrix usually comprises 30%-40% of composite structure. It has number of functions:[13]

- It binds the components together and determines the thermo-mechanical stability of the composite,
- It protects the reinforcements from wear/abrasion and environment,
- It helps to distribute the applied load by acting as a stress-transfer medium
- It provides durability, interlaminar toughness, shear compressive and transverse strengths to the system in general, and,
- It maintains the desired fibre orientations and spacing in specific structures.

As regards the toughness and strength of the composite, the role of the matrix is more subtle and complex than that of the reinforcements involved. Most of the reinforcing components like glass, graphite and boron fibres are all linear elastic and brittle solids, and whenever the stress on them is sufficient to cause unstable flaw growth, they fail catastrophically. And although both reinforcing component and the matrix are brittle, their combination can produce a material that is quite tough via a

synergism achieved by a combination of mechanisms that tends to keep cracks and flaws small, isolated and that dissipates mechanical energy effectively [13].

3. METAL MATRIX COMPOSITES

A composite material can be defined as a material consisting of two or more physically and/or chemically distinct, suitably arranged or distributed phases. It has characteristics that are not depicted by any of the components in isolation. Generally, one component acts as a matrix in which the reinforcing phase is distributed. The matrix component is, thus, the continuous phase. When the matrix component is a metal, we call such a composite a metal matrix composite. The reinforcement can be in the form of particles, whiskers, short fibers, or continuous fibers [20].

There are three entities that determine the characteristics of a composite, viz., reinforcement, matrix, and interface. Earlier on, the role of matrix was considered to be that of a medium or binder to hold the strong and stiff fibers or other types of reinforcement. This view was particularly prevalent in the polymer matrix composite area. Over the years, however, it has been realized that the matrix microstructure and consequently its mechanical properties have a considerable influence on the overall performance of a composite. This is particularly of the metal matrix composites because the act of the incorporating a reinforcement can result in changes in the microstructure of the metallic matrix and, consequently, in their structure sensitive properties such as strength and toughness [21].

Metals are chosen as the matrix material in metal matrix composite structures mainly because of following characteristics:

- They have higher application temperature ranges.
- They have higher transverse stiffness and strengths.
- In general, they have high toughness values.
- When present in metal matrices, the moisture effects and the danger of flammability are absolutely absent and they have high radiation resistances.

- They have high thermal and electric conductivities
- Metal matrix composites have higher strength-to-density, stiffness-to-density ratios, as well as better fatigue resistances, lower coefficients of thermal expansion and better wear resistances as compared with monolithic metals.
- They can be fabricated with conventional metal working equipment [22].

Some typical properties of some metals and some of their alloys are listed in Table 3.1.

Table 3.1: Some typical properties of some metals and their alloys [3].

	Density ρ ($g\ cm^{-3}$)	Young's Modulus (GPa)	Yield Strength (MPa)	Tensile Strength (MPa)
Pure Metals				
Aluminum	2.7	70	40	200
Copper	8.9	120	60	400
Nickel	8.9	210	70	400
Ti-6Al-4V		110	900	1000
Stainless Steel (304)		195	240	365
Plain Carbon Steel	7.9	210	250	420

3.1 Reinforcements

Metal Matrix composites offer a number of property advantages over conventional materials. They share with other composites the flexibility to combine the properties of both fiber and matrix to gain some of the advantages of each. Also, the combination of properties can be tailored to match composite properties to component requirements by varying the constituents and their volume fraction content. The properties achieved can surpass those possible with conventional materials [3,23].

Metal matrix composites offer a combination of properties that makes them superior to competing materials for some applications. A comparison of the similarities and differences helps to illustrate these advantages. The matrix serves as glue to bond fiber arrays into useful structural shapes for both polymer and metal matrix composites. The greater strength and stiffness of fibers are used to increase properties of the composite over those unreinforced matrix. The inorganic fibers used to reinforce polymers can also be used to reinforce metals. The uniaxial property contribution of the fiber is similar with both matrix materials, but the matrix contribution varies considerably [2,3].

Reinforcement increases the strength, stiffness, temperature resistance capacity but lowers density of metal matrix composites. The prime role of the fibres is to carry the load. The reinforcements can be divided into two major groups, continuous and discontinuous. However, they can be subdivided broadly into five major categories; continuous fibres, short fibres, whiskers, particulate and wire [24].

Table 3.2 provides examples of some important reinforcements used in metal matrix composites and their aspect (length/diameter) ratios and diameters [2,3,24].

Table 3.2: Typical Reinforcements Used in Metal Matrix Composites [2].

Type	Aspect Ratio	Diameter, μm	Examples
Particle	~1-4	1-25	SiC,B_4C,BN
Short fibre, whisker	~10-1000	0.1-25	SiC, Al_2O_3, C
Continuous fiber	>1000	3-150	SiC, Al_2O_3, C, B,W

3.1.1 Continuous Fibre Reinforcements

Continuous fibre in a composite are usually called filaments, the main continuous fibres including boron, graphite, alumina and silicon carbide. The fibre is unique for unidirectional load when it is oriented in the same direction of loading, but it has low strength in the direction perpendicular to the fibre orientation. Table 3.3 shows the characteristics of some continuous fibres [2,3,25].

Boron fibres are generally made by chemical vapour deposition (cvd) on a tungsten core. To retard reactions between the boron and the metal at high temperature, sometimes fibre coatings of silicon carbide or boron carbide have been used [3].

Carbon fibres are not suitable to form Al-based metal matrix composites due to the fibre degradation during processing.

Continuous fibre based on Al_2O_3 are produced by Du Pont (FP fibre) and Sumitomo. The FP fibre has a high purity and large grain size, which means that the fibre is extremely brittle and difficult to handle but may offer better metal matrix composites properties. Sumitomo's Al_2O_3 reinforced aluminum composite made by squeeze casting shows impressive strength-retention properties at elevated temperatures and is used widely in the automotive industry.

Silicon-carbide monofilament is made by the CVD process using a tungsten or carbon-core. Two silicon-carbide fibres are used widely due to their attractive combination of strength, stiffness and handling characteristics [24].

Table 3.3: Characteristics of Continuous Fibres [24].

Composition	Mean diameter (μm)	Young's Modulus (GPa)	Failure Strength (GPa)
B (cvd on W)	140	410	4.1
Al_2O_3 (-98%)	10-13	350	2.1
C	7	230	3.5
C	6.5	390	2.7
b-SiC coated C	dependent on C fibre used		
SiC (+O)	15	200	2.8
SiC (+O+Ti)	8-9	200	2.8
Al_2O_3-20SiO_2	10	160	1.6

3.1.2 Short Fibres

Disoriented short fibres are used have been used with some success as aluminum matrix composites reinforcement. The oxide fibres are used mainly for the reinforcement of automobile engine composites. Short fibres are still used for refractory insulation purposes due to their low strength compared with others, but they are cheaper than both fibres and whiskers [3].

3.1.3 Whiskers

Whiskers are characterised by their fibrous, single-crystal structures which have almost no crystalline defects. Numerous materials, including metals, oxides, carbides, halides and organic compounds, have been prepared under controlled conditions in the form of whiskers. Generally, a whisker has a single dislocation, which runs along the central axis. This relative freedom from dislocations means that the yield strength of a whisker is closed to the theoretical strength of the materials [2,3,24].

The method of vapour deposition is widely used in whisker preparation. The performance of whiskers at elevated temperatures is far better than that of any other fibres. Due to their outstanding specific mechanical properties, much work has been carried out on fabricating metal matrix composites using whiskers [3].

The physical properties of whiskers are responsible for different chemical reactivity with the matrix alloy. For instance, high-strength carbon fibres exhibit a much higher chemical reactivity toward liquid aluminum than do high modulus carbon fibres owing to their different states of crystallisation [24].

3.1.4 Wire

Metallic filaments are called wire, and they are characterised by their high elastic moduli. Among them, molybdenum and tungsten are the most popular. Presently stainless steel wire is creating interest. However the main disadvantage of metal filaments is that their density is higher than that of ceramic whiskers apart from of beryllium.

These fibres are generally more ductile than any other fibres, the aim being to use metallic filaments to fabricate composites for carrying tensile load with high toughness.

At high temperatures, there is too much metal-to-metal reaction, creating fabrication problems. Not much work has been done relatively at the present time on using metallic wire reinforcement to fabricate metal matrix composite materials [3,24].

3.1.5 Particulate

Particulates are the most common and cheapest reinforcement materials. These produce isotropic metal matrix composites, which show promise for applications in structural fields. Initially, attempts were made to produce reinforced aluminum alloys with graphite powder, but only low volume fractions of reinforcement had been corporated (<10%). Presently higher volume fractions of reinforcements have been achieved for various kinds of ceramic particles (oxide, carbide, and nitride). Their main properties are depicted in Table 3.4

Table 3.4: Characteristics of Ceramic Particles [3].

Particle	Size (μm)	Density (g cm^{-3})	UTS (Gpa)	E (Gpa)
Graphite	40-250	1.6-2.2	20	910
SiC	15-340	3.2	3	480
SiO_2	53	2.3	4.7	70
MgO	40	2.7-3.6		
Si_3N_4	46	3.2	3-6	360
TiC	46	4.9		320
BN	46	2.25	0.8	100-500
ZrO_2	75-180	5.65	0.14	210
B_4C	40-340	2.5	6.5	480
TiO_2	20	3.9-4.3		
Al_2O_3	40-340	3.97	8	460
Glass	30-120	2.55	3.5	110

The current trend seems to be to use SiC short fibres, whiskers, and particles in Al alloy matrix since SiC offers adequate thermal stability with Al alloys during synthesis and application. It also has good wettability with Al alloys. The density of SiC is close to alloy (2.8-3.3 g cm^{-3}), and reinforcements give substantial increases in the modulus and UTS. Dispersion of soft particles like graphite and mica in Al alloys do not contribute to the strength. On the contrary, these lower mechanical properties, but the composites offer special properties like adhesive wear resistance [3,24,25].

The USA leads the world in particulate production, with Alcan having made the greatest progress in research with particulates. It is observed from research that 20% silicon carbide in particulate form has shown improvements in yield and tensile strength of a similar percentage. There is especially no change in density, but stiffness has improved by up to 50%, which breaks the general "rule" that the specific thickness of all engineering metals, regardless of their density, is roughly the same [24,25].

The SiC-particulate-reinforced aluminum matrix composites have a good potential for use as wear-resistant materials. Actually, particulates lead to a favourable effect on properties such as hardness, wear resistance and compressive strength. A number of particulate reinforced systems have been in use industrially for many years, these including cermets, used in electronic industry for the tracks of precision variable resistors, and high-speed cutting-tool tips [3].

Because of their relatively higher costs to the other prevalent abrasive powders (SiC and Al_2O_3), not too many works has been done on boron carbide (B_4C) reinforced metal matrix composites. B_4C seems to be a better reinforcement material with its lower density and higher hardness according to SiC and Al_2O_3. Boron carbide is an outstanding reinforcement material for aluminum and its alloys meeting many of the mechanical and physical properties required of an effective reinforcement [2,24].

3.2 Important Metal Matrix Composites

A variety of metals and their alloys can be used as matrix materials. Below are the most common ones [2].

3.2.1 Aluminum Alloys

Due to their low density and gorgeous strength, toughness, and corrosion resistance, aluminum and its alloys are used in many applications in the field of aerospace. Table 3.5 shows the elastic modulus of some lightweight materials. In this respect of mention the alloys Al-Cu-Mg and Al-Zn-Mg-Cu alloys are very important precipitation hardenable alloys. Also aluminum-lithium alloys generate one of the most outstanding precipitation hardenable alloys. When lithium added to aluminum as a primary alloying element, it yields with an enhancement in the elastic modulus and decreasing in the density of the alloy. Naturally the aerospace technology is the main target for this kind of alloys [2].

Table 3.5: Elastic Modulus of Common Lightweight Metallic Alloys [26,27]

Alloy	Density (g cm^{-3})	Elastic Modulus (Gpa)
Aluminum Alloys	2.7	65
Magnesium Alloys	1.8	45
Ti Alloys	4.5	103
Steel	8.3	200

3.2.2 Titanium Alloys

Titanium is one of the most intriguing aerospace materials. The density of the titanium is 4.5 g cm^{-3} and the Young's modulus of 115 Gpa. However, the density of titanium can be in the range of 4.3 and 5.1 g. cm^{-3} and the modulus varies between 80-180 Gpa [2,28]. High strength/weight and modulus/weight ratios are important. Titanium has high melting point (1672 ^{o}C) respect to other matrix materials and stands strength at high temperatures with good oxidation and corrosion resistance. Consequently all these properties render titanium an important material for aerospace applications. Mainly the turbine parts and compressor blades in the jet engines are made of titanium alloys [2].

3.2.3 Magnesium Alloys

Recently a considerable progress has been achieved in magnesium alloy metal matrix composites. Some silicon carbide particles-reinforced magnesium alloy compositions have been used. Most significant benefit of magnesium alloys usage in composite materials is the increasing in the modulus (around 40% respect to unreinforced). These metal matrix composites also show good wear properties and they also have lower coefficient of thermal expansion. These properties make these materials an ideal choice for aerospace, automotive, bicycle and engineering industries [2].

3.2.4 Copper

Copper has a face centred cubic structure. Its use as an electrical conductor is quite ubiquitous. It has good thermal conductivity. It can be cast and worked easily. One of the major applications of copper in a composite as a matrix material is in niobium based superconductors [2,29].

3.2.5 Intermetallic Compounds

There are many other intermetallics, not necessarily ordered, available. Ordered intermetallic alloys possess structures characterized by long range ordering, in an example, different atoms occupy specific positions in the lattice. Because of their ordered structure, dislocations in intermetallics are much more restricted than disordered alloys. This results in retention of strength at elevated temperatures, a very desirable feature [2].

3.3 Aluminum Matrix Composites

Aluminum is the most significant matrix material for the metal matrix composites [30]. The Al alloys are quite attractive due to their low density, their capability to be strengthened by precipitation, their good corrosion resistance, high thermal and electrical conductivity, and their high damping capacity [31]. Aluminum matrix composites are one of the advanced engineering materials that have been developed for weight-critical applications in the aerospace, and more recently in the automotive

industries due their excellent combination of high specific strength and better wear resistance. [30]. Aluminum matrix composites have been widely studied since the 1920s and are now used in sporting goods, electronic packaging, armours and automotive industries. In table 3.6 markets for some metal matrix composites are given. They offer a large variety of mechanical properties depending on the chemical composition of the Al-matrix. They are usually reinforced by Al_2O_3, SiC, and C, however, SiO_2, B, BN, B_4C, AlN may also be considered. The aluminum matrices are in general Al-Si, Al-Cu, 2xxx or 6xxx alloys [32].

In the 1980s, transportation industries began to develop discontinuously reinforced aluminum matrix composites. These materials are significant for their mechanical properties and low costs [32].

Table 3.6: Markets for Some Metal Matrix Composites (in millions of $ US) [32].

Type	1988	1993	2000
Aluminum	12.0	35.0	78.3
Magnesium	2.0	8.9	20.0
Titanium	1.0	2.6	14.1
Copper	1.4	5.5	28.3

Aluminum matrix composites can be categorized into three types respect to its reinforcement; particulates, whiskers or discontinuous fibres, and continuous fibres.

The aluminum matrix composites with particulate reinforcement provide high stiffness with low cost. The properties are isotropic.

The whisker reinforced composites are more expensive the particulate reinforced ones, however, they provide mainly high strength. Respect to discontinuous based composites, and particulates, single crystal whiskers usually have a much greater tensile strength [2]

Although the continuous fibre based composites give the best combination of stiffness and strength, they cost very high due to the expensive continuous fibres and the production system [25].

Metal matrix composites have been attracting the attentions recently due to its outstanding mechanical and physical properties and increased elevated temperatures capabilities. In addition, aluminum matrix composites reinforced with the ceramic particles or whiskers have received significant attention since they can be formed by metalworking practices. Not only the improved mechanical properties, also other controlling behaviours, like coefficient of thermal expansion and wear resistance, can be gained by the ceramic particles. The incorporation of reinforcement into aluminum alloys has reportedly led to both strengthening and accelerated aging effects [33].

Among the ceramic reinforcement materials, boron carbide (B_4C) ceramics have been attracting the attentions due to its high hardness, high melting point, good thermal conductivity and good electrical conductivity. These properties make this material a great choice for neutron adsorption materials, wear resistance materials, electrode materials and cutting tools [6]. Boron carbide is used for a wide range of engineering applications because of its exceptional hardness just below that of diamond (9.5 + in Mohs' scale), outstanding elastic modulus, and low density (2.52 g cm^{-3}). These hardness and density values are much better than that of SiC and Al_2O_3, therefore boron carbide has become a better reinforcement material. However, due to the higher cost of boron carbide powder respect to common abrasive powders such as SiC and Al_2O_3, limited research has been done on B_4C reinforced metal matrix composites [34].

Although boron carbide has some outstanding physical and mechanical properties, its extreme sensitivity to brittle fracture (K_{IC} = 3.7 MPa $m^{1/2}$) and the difficulties involve in fabricating dense B_4C materials have limited its use in industrial applications [4]. These problems can be reduced considerably by the production of B_4C-based composites, essentially B_4C-metal composites. For applications at low temperatures, aluminum or aluminum alloys is a material of choice. Suggested applications for B_4C-Al composites include its use in as a structural neutron absorber, armour plate materials, and a substrate material for computer hard disks [35].

4. FABRICATION OF METAL MATRIX COMPOSITES

The problem of developing metal matrix and metal-polymer composite materials is one of the most important issues in present day materials technology, for its solution is pivotal to the development of a number of leading technologies. The development of new fibrous and lamellar composite materials with improved physico-chemical, electrical, thermal and other properties is a springboard for qualitative scientific and technological advances not only in aerospace and shipbuilding technologies, but also in mechanical, power, electronic, electrical, radio engineering, transport, construction and other industries [36].

Metal matrix composites can not be fabricated easily by the conventional method. It will be inexpensive if it can be successfully fabricated by the conventional casting method. There are several fabrication techniques available to manufacture the Metal matrix composite materials: there is no unique route in this respect. Due to the choice of matrix and reinforcement material and the types of reinforcement, the fabrication techniques can vary considerably [37].

Modern metallurgy uses various methods for fabricating alloys, semifinished products and articles made from them, including various types of casting, processes of powder metallurgy, plastic working, spraying, deposition and many others [38]. The choice of a particular fabrication method is mainly determined by the following factors.

1. Type source materials of matrix and reinforcing agents;
2. The possibility of introducing the reinforcing agent into the matrix without damaging it;
3. Forming a secure bond at the reinforcing agent-matrix interface;
4. Maximum realization in the material of the properties of its components;
5. Attaining the desired reinforcing agent distribution pattern inside the matrix;
6. Combining material fabrication processes with part manufacture;

7. Economic efficiency of the process [36].

4.1 Fabrication Route

Processing of metal-matrix composites can be broadly divided into two categories of fabrication technique:

(1) Solid state (including powder metallurgy and diffusion bonding)

(2) Liquid state

The past 30-year period of development and study of metal matrix composites has seen the development of dozens of procedures for composite material fabrication. According to the state of the matrix alloy in the process of composite material formation, methods can be divided into solid-phase, powder and liquid-phase ones. When solid-phase technology is used, the matrix material is present in the blank of the future composite material in the form of foils, fibre coatings, etc. in the case of powder technology; matrix material present in the form of powder is usually mixed with the reinforcing component. When liquid-phase technology is applied, the matrix melt is prepared individually, while composite material formation starts with the filling of cavities of the reinforcing skeleton with the melt. When the changeover from liquid-phase to solid-phase technology takes places, the matrix mobility during composite material formation diminishes. Naturally, such a classification is conditional, and some real processes combine the hallmarks of different methods [21].

A salient feature of the early stage of metal matrix composite material manufacture was development of specific procedures targeted at the fabrication of one particular composite material. As a rule, the following procedure was adopted. Taking a matrix/filler pair, a process for consolidation was developed so as to ensure the best possible fabrication of material in question. The next stage saw serious advances towards process unification: solid-phase methods were developed for systems with large fibres or for systems that allow high-quality deposition of matrix alloy on fibres. Powder methods were developed mainly for composite materials with dispersed filling particles, whiskers, etc. liquid-phase methods are good for fabricating various composite materials [37,39]. The main argument against them is

usually the claim of high reactivity of melt/filler systems. In fact this phenomenon does prohibit the application of liquid-phase methods to a number of systems, for-example Ti/C and Ni/C. However, the knowledge and experience accumulated in the field of controlling composite material component interactions by alloying the matrix and modifying the fibre surface, as well as the development of a wide spectrum of fillers, has allowed liquid-phase technology to be applied to a wide range of systems [38].

It should be noted that the main trend in recent years in the field of metal matrix composite materials has been revived interest in liquid-phase methods. This concerns in particular the method of forced infiltration.

A majority of the commercially viable applications are now produced by liquid-state processing because of inherent advantages of this processing technique over solid-state techniques. That is, the liquid metal is generally less expensive and easier to handle than are powders, and the composite material can be produced in a wide variety of shapes, using methods already developed in the casting industry for unreinforced metals. Conversely, liquid-state processes often suffer from a lack of reproducibility as a result of incomplete control of the processing parameters, and of undesirable chemical reactions at the interface between molten metal and reinforcement [37].

Also, they are often limited to low-melting point alloys, although some reinforced intermetallics have now been produced by liquid-state processes. Liquid-state processing technologies currently being investigated and developed utilize a variety of methods to physically combine the matrix and the reinforcement. Basically it is possible to sort liquid-state processing technologies into four major categories: Infiltration, dispersion, spraying, and In-situ fabrication [39].

4.2 Solid State Fabrication Methods

There are several ways to fabricate metal matrix composites using solid state materials such as diffusion bonding, hot rolling, extrusion, drawing, explosive welding, powder metallurgy route, pneumatic impaction etc, but among them diffusion bonding and the powder metallurgy route are commonly used [36].

4.2.1 Diffusion Bonding

This technique is usually used to produce fibre reinforced metal matrix composites with sheets and foils of matrix material. In this technique primarily sheet metal and metal alloys and the fibre form reinforcement material are chemically surface treated for supporting interdiffusion. Afterwards fibres are placed on the metal in demanded orientation and bonding occurs by press forming directly. Sometimes the fibres are coated by plasma spraying or ion platting to increase the bonding strength before diffusion bonding. Secondarily machining performed [22,36].

In this technique types of the fibre determine the bonding conditions. For instance, in the case of monofilament and fibre bundles like, C, SiC Al_2O_3, they are wound onto a metal with good thermal conduction. In this case various coating methods can be applied such as plasma spraying, chemical coating, electro-chemical platting, cvd and pvd coating. Plasma spraying is the simplest method among these coating techniques. Its low cost allows producing sheets of large width with satisfying adhesion between fibre and the matrix [2,22,36].

4.2.2. The Powder Metallurgy Technique

The powder metallurgy technique is the most widely used method for producing discontinuous reinforced metal matrix composites. In this process metal powders and reinforcement materials are first mixed and put into mould of the demanded shape. Afterwards pressure is applied in order to give a compact shape (cold pressing). In order to achieve the bonding between the powder particles, the compact is then heated to temperature below melting point. This temperature must allow diffusion between the particles and the compact.

This method offers several advantages over diffusion bonding. Firstly, the process allow to work under lower temperatures relatively. This yields with less interaction between matrix and the reinforcement material and results with improved mechanical properties. Secondly the shape tolerances are very much higher respect to fusion metallurgy. Thirdly a wide range of reinforcement levels can be used. This extended range of reinforcement volume loadings is important to address customised

applications requiring higher levels of stiffness and lower levels of coefficient of thermal expansion [2,3,36].

4.3 Liquid State Fabrication Methods

4.3.1 Infiltration Process

In recent years, metal matrix composites containing high volume fractions of reinforcements have been developed for several applications, including multifunctional electronic packaging, wear-resistant components, and creep-resistant applications. Among all the techniques to fabricate these MMCs, the liquid infiltration process is one of the most economical and versatile [40].

Infiltration process involves holding a porous body of the reinforcing phase within a mold and infiltrating it with molten metal that flows through interstices to fill the pores and produce a composite. Such processes are not new; liquid-metal infiltration has been used for many years in powder metallurgy in order to strengthen porous metal parts of iron and steel with copper. The major difference between infiltration of solid metal by liquid metal and infiltration of ceramic by liquid metal regards wetting. Liquid metal generally does not spontaneously wet the reinforcement. Therefore, it is often forced into the preform by application of an external force that overcomes the capillary and fluid-drag forces [41].

The main parameters in infiltration processes are the initial composition, morphology, volume fraction and temperature of the reinforcement, the initial composition and temperature of the infiltrating metal, and the nature and magnitude of the external force applied to the metal [22-40].

4.3.1.1 Pressure – Driven Infiltration:

A strategy to overcome poor wetting is to supply mechanical work to force the metal into a preform that it does not wet. Although the primary purpose of an externally applied pressure is overcome the capillary forces, higher pressures can provide additional benefits such as increased processing speed, control over chemical reactions, refined matrix microstructures [38,39-41].

Pressure is generally applied by a gas or mechanically. In the case of pressure application by gas, the metal is forced into the preform of reinforcing phase by an inert gas, such as Ar. The process was first patented in 1970 and, has been widely used since then to produce both reinforced aluminum alloys and intermetallics composites [22].

The technique utilizing mechanically applied pressure involves a force that is exerted on the molten metal by the piston of a hydraulic press, and subsequently maintained during solidification. The pressures involved are generally higher than those in gas-driven processes [38]. This type of infiltration process is currently the most widely investigated for commercial applications, and has been directly adapted from established processes designed to cast unreinforced metals. Composites produced by this method are generally featuring a pore-free matrix. However, application of pressure may induce preform deformation or breakage during infiltration [42].

A major advantage of infiltration processes is that they allow for near-net shape production of parts fully or selectively reinforced with a variety of materials. If cold dies and reinforcements are used, or if high pressures are maintained during solidification, matrix – reinforcement chemical reactions can be minimized and attractive, defect-free matrix structures can be achieved [43].

A limitation of infiltration process is the need for the reinforcement to be self-supporting, either as a bound preform or as a dense pack of particles or fibers. Heterogeneity of the final product may result from preform deformation during infiltration or from clustering of fibers that are detrimental to the composite mechanical properties. Also, tooling may be expensive if high pressures are used [44].

4.3.2 Dispersion Process

In dispersion process the reinforcement is incorporated in loose form into the metal matrix. Because most metal reinforcement systems exhibit poor wetting, mechanical force is required to combine the phases, generally through stirring. This method is currently the most inexpensive manner in which to produce MMCs, and lends itself to production of large quantities of material, which can be further processed via

casting or extrusion [22]. The simplest dispersion process in current use is the vortex method, which consists of vigorous stirring of the liquid metal and addition of particles in the vortex. Skibo and Schuster have patented a process for mixing SiC particulates in molten aluminum under vacuum, with a specially designed impeller that has the advantage of limiting the incorporation of impurities, oxides, or gases because of the vacuum and reduced vortexing. Ingots of such composites are now commercially produced in large quantities by Duralcan, San Diego, CA. Other methods being investigated include the bottom-mixing process, where a rotating blade is progressively lowered into an evacuated bed of particles covered with molten aluminum, and the injection of particles below the surface of the melt using a carrier gas. Mixing of particles and metal can also be achieved while the alloyed metal is kept between solidus and liquidus temperatures. This process is known as compo-casting [39].

Critical to the success of dispersion process is the control over generally undesirable features such as porosity resulting from gas entrapment during mixing, oxide inclusions, reaction between reinforcement and metal favoured by long contact times, as well as particle migration and clustering during and after mixing. Furthermore, such processes are not suited for long fibers or oriented reinforcement because of the difficulties in stirring and the need for secondary deformation processing to improve the reinforcement distribution and close any pores [42].

4.3.3 Spray Process

In these processes, droplets of molten metal are sprayed together with the reinforcing phase and collected on a substrate where metal solidification is completed. Alternatively, the reinforcement may be placed on a substrate, and molten metal may be sprayed onto it. Spray-forming processes have been utilized for unreinforced metal for more than 20 years because the high solidification rate of the droplets yields materials with little segregation and a refined grain structure [41]. The various composite spray processes differ in the method of the spraying the metal, and differ in the way the reinforcement is mixed with the metal. The critical parameters in spray processing are the initial temperature, size distribution and velocity of the metal drops, the velocity, temperature, and the feeding rate of the reinforcement, and the position, nature, and temperature of the substrate collecting the material. Most

spray-deposition process use gases to atomize the molten metal into fine droplets. The particles can be injected within the droplet stream and the atomizing gas [43].

One advantage of the spray-deposition techniques resides in the resulting matrix microstructures that feature fine grain size and low segregation. Also, because liquid metal and reinforcement contact only briefly, interfacial reaction is minimized, allowing the production of thermodynamically metastable two-phase materials such as iron particles in aluminum alloys. Simple forms, such as ingots or tubes, can be produced by these processes, although they lack in part-shape versatility. Other drawbacks include the amount of residual porosity and the resulting need to further process the materials. Also, this process is not as economical as dispersion or infiltration processes, because of high cost of the gases used and the large amounts of waste powder to be collected and disposed [41-43].

4.3.4 In-Situ Process

The term in-situ composite was first used for materials produced by solidification of polyphase alloys. When polyphase alloys solidify directionally with a plane front, they may exhibit a fine lamellar or rod-like structure of β phase in an α phase matrix, and the interface spacing is a function of the growth rate [38]. Extensive work has been done in this field since the 1960s. Applications have been proposed for these materials in the areas of optics and electronics, but their generally low growth rates, and problems resulting from the gradual coarsening of the structure at elevated temperatures, restricted their possible use [39]. The recent emphasis on processing temperature materials, however, has given a new impetus to research on in-situ composites, because intermetallics alloys may be produced by controlled solidification or by other in-situ processes such as chemical reaction between melt and solid or gaseous phases. Recently a wide range use of in situ process has been very common. By the time nanocompsite technology has been developed the importance of the in situ process increases day by day. Especially in the production of polymer nanocomposites this technique serves perfectly in various situations. [22].

A major advantage of in-situ composite materials is that the reinforcing phase is generally homogeneously distributed, and spacing or size of the reinforcement may be adjusted in several cases by solidification or reaction time. It can be very easily

made on substrates such as glass, silicon and titania. In this technique shrinkage and cracking during drying heating reduce to very low ratios. However, the choice of systems and the orientation of the reinforcement are limited, and the kinetics of the processes, or the shape of the reinforcing phases, are sometimes difficult the control [22,39].

5.EXPERIMENTAL

5.1. Materials

In this study, commercially pure aluminum (Al) and Al-Mg alloys were used as the matrix material. Chemical compositions of the matrix materials are given in Table 5.1. The reinforcement material was used as boron carbide (B_4C) particles. The average size of the boron carbide particles were about 27.19 μm. All these materials were used as purchased without any purification and modification.

Table 5.1: Chemical Compositions of the Materials Utilized as Matrix in this Study

Alloy Type	Element, wt %		
	Al	Mg	Si
Pure Al			
Al	99.78	-	0.092
Al-Mg Alloy			
Al-% 1 Mg	98.69	1.078	0.060
Al-% 2 Mg	97.66	2.104	0.070
Al-% 4 Mg	95.67	4.048	0.070
Al-% 8 Mg	91.69	7.962	0.074

5.2 Composite Production

In this study, the composites were fabricated by pressure infiltration of pure Al and Al-Mg alloy melts into B_4C particle performs. Schematic view of the pressure infiltration apparatus used for the fabrication is shown in Figure 5.1. The apparatus composed of three parts; heating unit, vessel and lid assembly. Fused silica tubes with 9 mm outer diameter, 7 mm inner diameter and 250 mm lengths were used as perform holder. The infiltration temperature was 750 ^oC and Al and Al-Mg alloys were infiltrated into boron carbide particle performs under argon pressure of 765 kPa. The vessel was pressurised at an approximate rate of 170 kPa/s. the applied pressure was sustained for 2 minutes. Then the pressure was released from the vessel

at a rate of approximately 210 kPa/s. The composites having the diameter of 7 mm were detached afterwards from the vessel and cooled in air. The applied pressure to infiltrate performs were over the value of minimum threshold, which is 200 kPa, required for the infiltration of the pure Al.

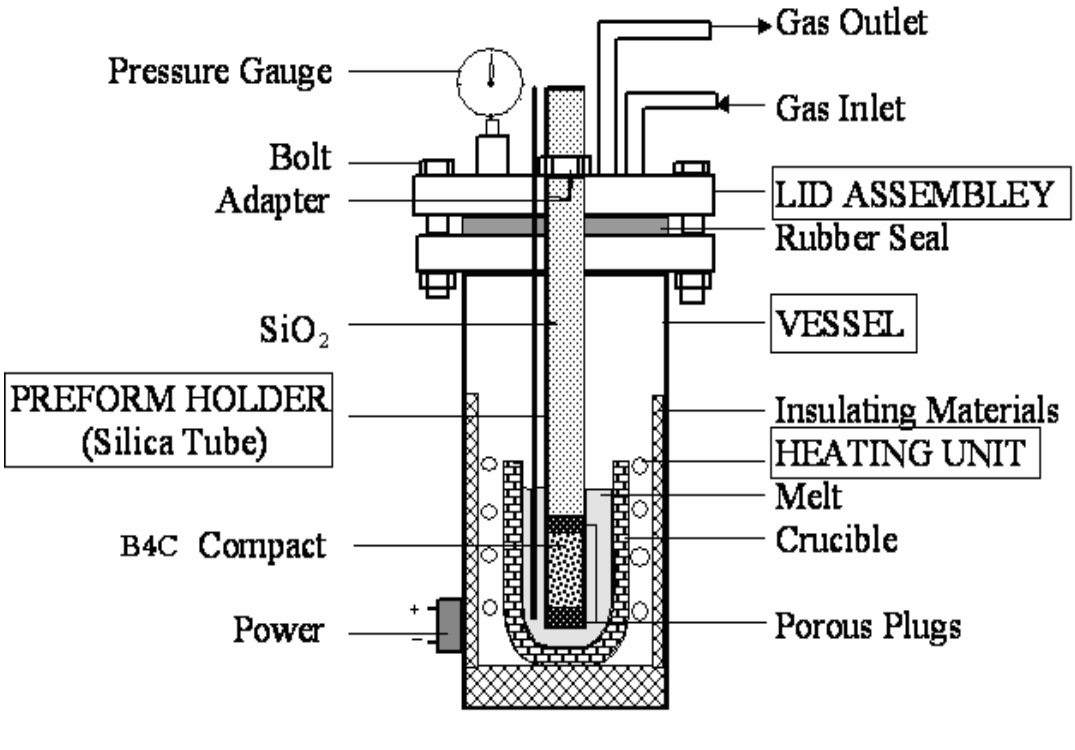


Figure 5.1: The schematic view of the pressure infiltration apparatus [30].

5.3 Characterization of Microstructures

The microsturctural characterizations of different types of composites were carried out by using several techniques; microscopic examination, X-Ray diffraction (XRD) analyses, and micro hardness measurements.

5.3.1 Microscopic Examination

Metallographic samples were prepared from the longitudinal cross sections of the investigated composites by gentle grinding and polishing. All samples were grinded gently with 120, 240, 400, 600, 800, 1000, and 1200 mesh SiC emery papers. Afterwards samples were gently polished by Al_2O_3 solutions and then carried on polishing by 6 μ and 1 μ diamond solutions. Microstructures of the composites were

investigated by computer equipped no brand Chinese trademark light optical microscope (LOM) and Jeol JSM 7000F type field emission Scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS).

In order to find the volume fractions of B_4C particles and the porosity, image analyse was carried out manually. The image analyse of each compositions were done over their snapshots which was taken from at least five different regions of the composites.

5.3.2 XRD Analysis

Identification of the phases in Al-B_4C and Al/1-8 wt% Mg-B_4C composites were performed by using a Philips PW 3710 type X-ray diffractometer. X-ray diffraction (XRD) analyses were performed by using Cu Kα radiation with an incident beam angle of 2^o. Diffraction angle was between 10-90^o, with a step increment of 0.02^o and a count time of 1s.

5.3.3 Microhardness Experiments

Microhardness tests were performed on metallographic samples by utilizing Shmadzu HMV Microhardness tester. Metallographic samples were used in order to find the hardness of the composites was measured by using a Vickers indenter. Test load for each sample was 500 g. Due to the trace of the indenter cover both matrix and reinforcement material, the hardness value stands for the bulk hardness of the composite.

5.4 Mechanical Tests

Room temperature mechanical properties of the composites were determined by compression and impact test. The lengths of the composites, which have 7 mm diameters, were about 14 and 30 mm.

5.4.1. Compression Test

Compression test of the composites were carried out by using Dartec compression test machine. Samples of the compression test were 7 mm in diameter an 14 mm in length.

5.4.2. Impact Test

The impact tests were performed by using Zwick B5113 charpy-type impact test equipment having capacity of 50 J. The measurement sensitivity of the equipment was 0.2 J. The diameter and length of the impact test samples were 7 mm and 30 mm, respectively.

5.5 Wear Tests

The wear behaviour of the Al matrix B_4C reinforced composites were investigated by metal-metal and metal-abrasive wear test. The tests were carried out by applying a normal load of 28 N which was corresponding to pressure of 0.73 MPa, to the samples. The results of the wear tests were evaluated by wear loss method. In this technique the weights of the composites were measured during tests at certain intervals with an electronic balance having a resolution of ± 0.1 mg. The weight loss of the composites was determined at 2000 m and 5.5 m intervals during metal-metal and metal-abrasive wear tests, respectively.

Worn surfaces of the composites were investigated by Jeol JSM 7000F type field emission Scanning electron microscope (SEM).

5.5.1 Metal-Metal Wear Tests

Metal-metal wear test were performed on a by pin on disc type wear equipment depicted in Figure 5.2. The composites having diameter of 7 mm were used as the pin. The constant speed rotating disc (0.45 m/s) is made of AISI M2 quality tool steel with a hardness of 65 HRC. The experiments were carried out at room temperature having the humidity of % 40-50. The total sliding distance was set as 12000 m and the weight loss was measured at every 2000 m.

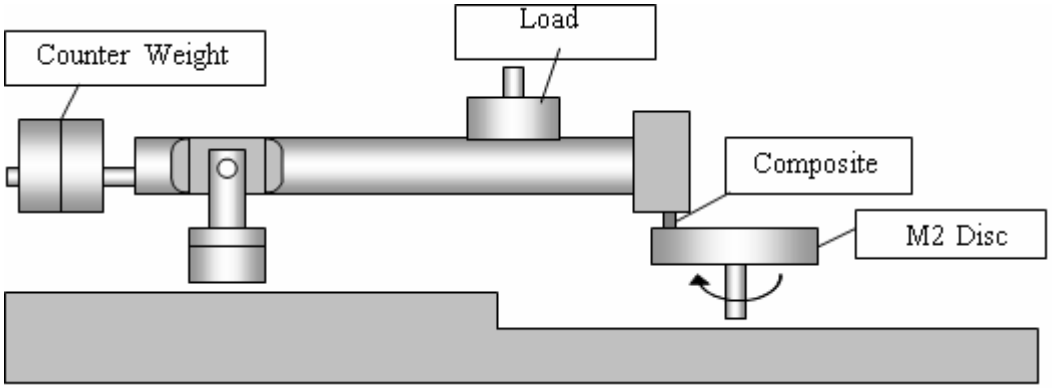


Figure 5.2: The schematic views of the metal-metal wear test equipment [30].

5.5.2 Metal-Abrasive Tests

Metal-abrasive wear tests were conducted at room (25^{o}C) and elevated (up to 600^{o}C by 100^{o} gradually enhancement) temperatures by rubbing the composites on 150 mesh Al_2O_3 abrasive bands as shown in Figure 5.3.

Metal-abrasive wear tests were performed at constant sliding speed of 0.18 m/s. the total sliding distance was 5.5 m for each room and elevated temperatures.

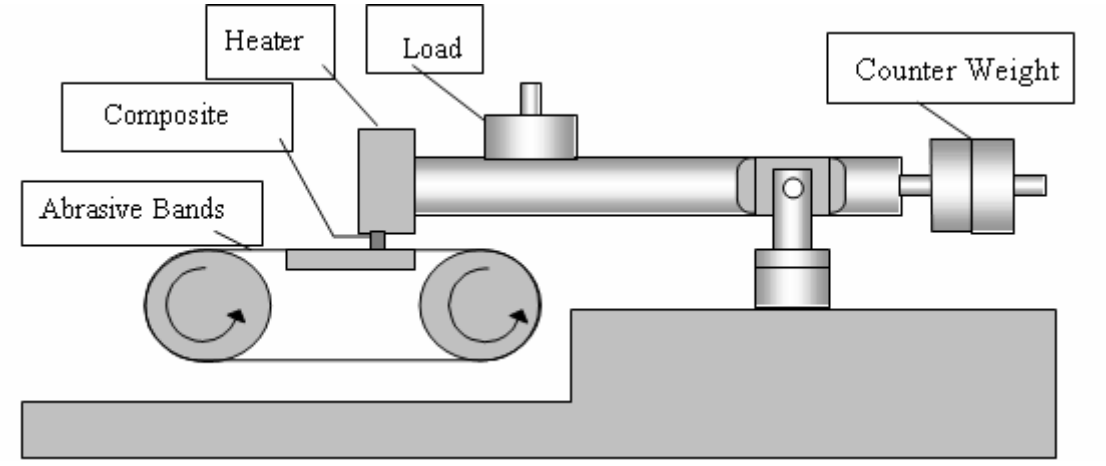


Figure 5.3: The schematic views of the metal-abrasive wear test equipment [30].

In order to examine the high temperature microstructure of the Al/1-8 % wt Mg alloy matrix composite; the samples were hold at each temperature for 2 hours and water

quenched. The water quenched samples were then prepared for metallographic investigation by light optical microscope.

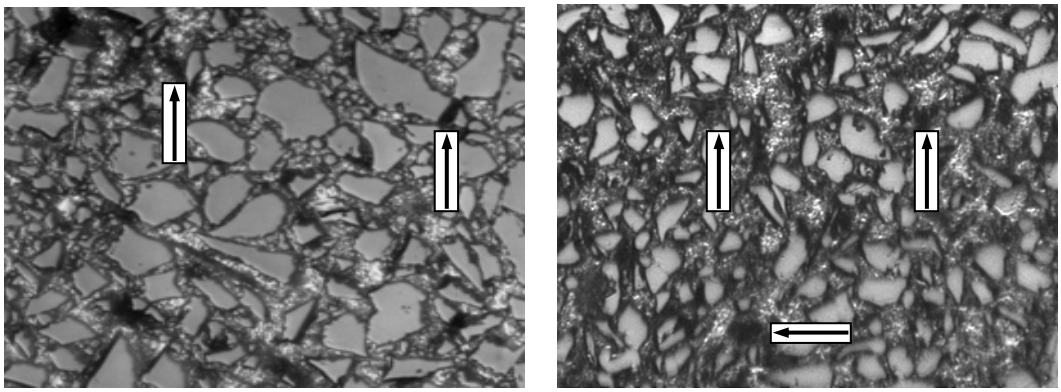
6. RESULTS AND DISCUSSION

6.1. Metallographic Investigation

The results of light optical microscope (LOM) and scanning electron microscope (SEM) examinations conducted on pure Al and Al/1-8 wt % Mg matrix composites reinforced with B_4C particles are discussed below.

6.1.1 Pure Al Matrix Composites

LOM micrographs of the pure Al matrix composites are shown in Figure 6.1 at various magnifications. It can easily be seen that the B_4C particles are distributed homogenously in the matrix and no segregation takes place at a particular region. However presences of the porosity at tips of the B_4C particles are observed. The volume fractions of the porosity, reinforcement material and the matrix are presented in Table 6.1. The volume fraction of the B_4C particles was found about 50 %.



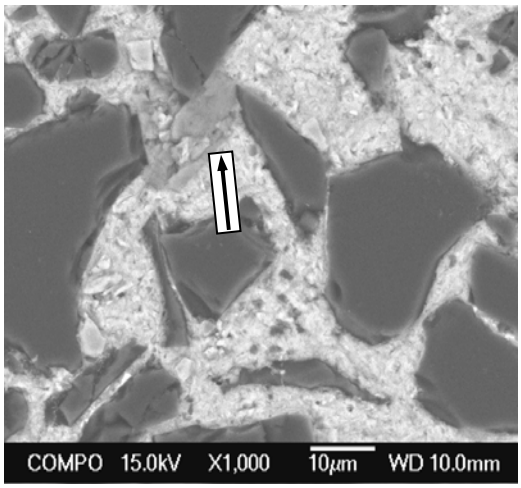
(a) **(b)**

Figure 6.1: Microstructures of pure Al matrix composites at different magnifications (a) 40X (b) 20X. Arrows show some of the porosities in the microstructure.

Table 6.1: % Volume fractions of the microstructure constituents of pure Al matrix composite

Microstructure Constituent	Volume Fraction, %
Matrix	32,9
Particle	53,2
Porosity	13,9

SEM examination revealed presence of light coloured intermetallic compounds in the microstructure as presented in Figure 6.2a. This type of intermetallic compound was not detected by the LOM. EDS analyses revealed that intermetallic compound was composed of two major elements; Aluminum and boron (Figure 6.2b).



(a)

Element	Weight%
B	53.48
O	2.16
Al	44.36
Totals	100.00

(b)

Figure 6.2: (a) SEM micrographs of pure Al-B_4C composite (b) EDS analyses resulted with the intermetallic shown by arrows.

In literature it has been reported that [45-47] intermetallic precipitation starts at 450-500^oC with the formation of Al_4BC. At 600^oC AlB_2 forms due to rapid depletion of the aluminum content in the composite. According to Frage et al. [45] AlB_2 and B_4C are the main phases present in between 600 and 700^oC.

6.1.2 Al-Mg Alloys Matrix Composites

The LOM micrographs of Al-Mg alloy matrix composites are presented in Figure 6.3. B_4C particles were distributed in the matrix quite homogenously. The porosity in Al-Mg matrix composites is very low with respect to pure Al matrix composites. By increasing of the Mg content in the matrix the porosity decreased significantly. The volume fractions of the porosity, matrix and reinforcement for the Al.1-8 wt% Mg matrix composites are given in Table 6.2. The presence of 8 wt% Mg in the matrix decreased the porosity in the order of 95% when compared to 1 wt% Mg

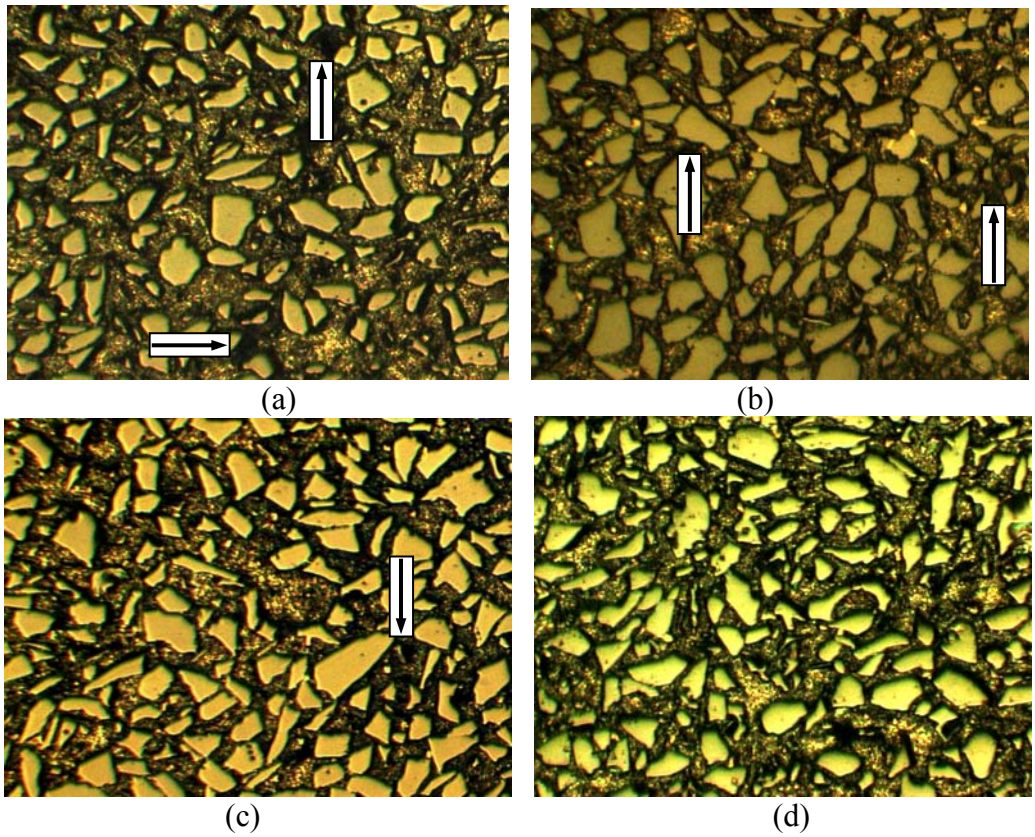


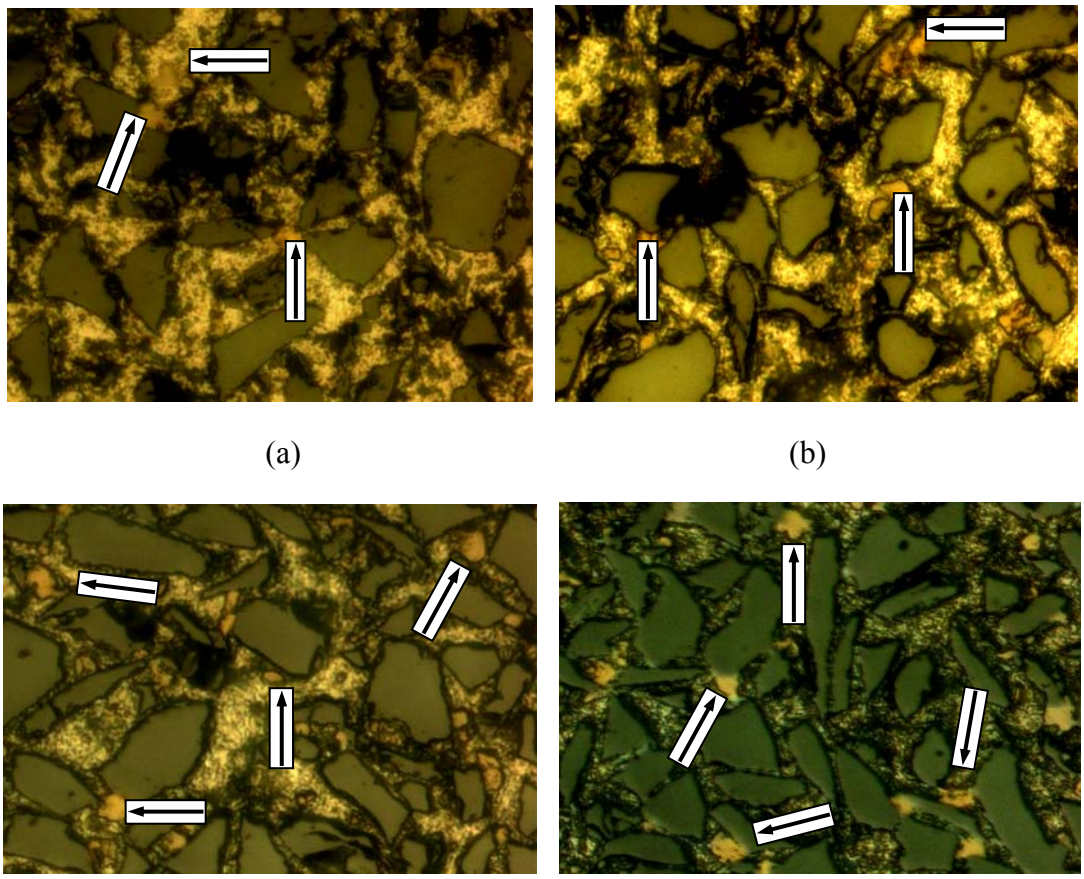
Figure 6.3 : LOM micrographs of (a) Al-% 1, (b) Al-%2, (c) Al-%4 ve (d) Al-%8 Mg alloy matrix composites at 20X magnification. Arrows point some porosities in the matrix

High magnification LOM studies revealed presence of intermetallic compounds in the Al-Mg matrix, similar to pure Al matrix composites (Figure 6.4). The yellowish colour particles occur by the addition of Mg element in the matrix. The volume fraction of these yellowish particles enhance with addition of Mg content in the matrix. These intermetallic compounds appeared in light grey colour on the SEM micrographs (Figure 6.5a). The EDS analyses revealed that these intermetallic

compounds are composed of Al, B, and Mg (Figure 6.5b). Although only Al and B were detected in the intermetallic compound formed in pure Al matrix, alloying the matrix with Mg resulted with penetration of Mg atoms to Al and B rich intermetallic compound.

Table 6.2: Volume fractions of microstructure constituents in Al-Mg alloy compsoites

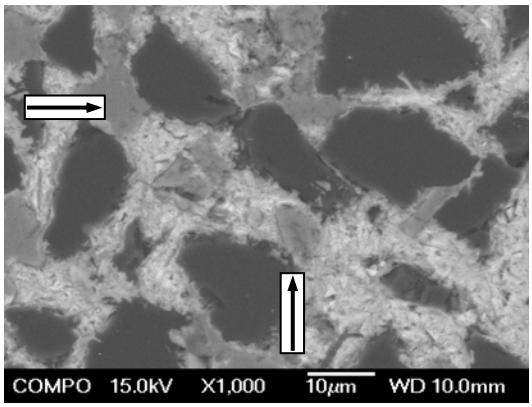
Matrix	Volume Fraction (%)			
	B_4C	Porosity	Matrix	Intermetallic
Al-% 1 Mg	58.1	10.9	31	-
Al-% 2 Mg	56.8	6.4	35.8	1,0
Al % 4 Mg	59.6	1.2	37.7	1,5
Al-% 8 Mg	58.2	0.6	38.2	3



(a) (b)

(c) (d)

Figure 6.4: LOM micrographs of (a) Al-% 1 Mg, (b) Al-%2 Mg, (c) Al-%4 Mg ve (d) Al-%8 Mg alloy matrix composites at 40X magnification. Arrows point the intermetallic compounds.



Element	Weight%
B	52.66
Mg	25.17
Al	22.17
Totals	100.00

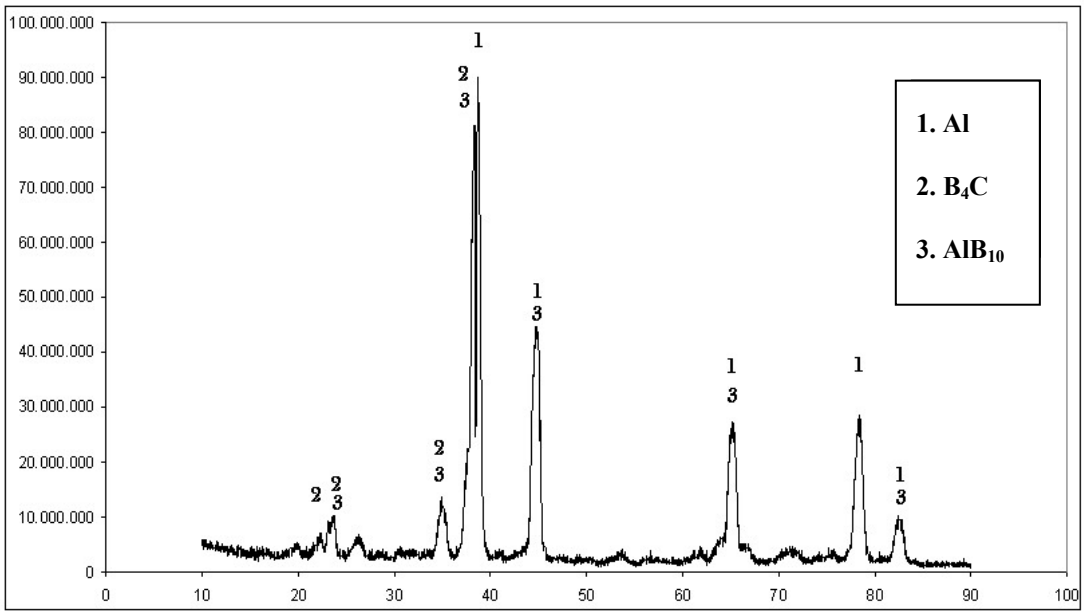
(a) **(b)**

Figure 6.5: (a) SEM micrograph of Al-8wt % Mg composite (b) EDS analyses results conducted on the intermetallic compounds shown by arrows.

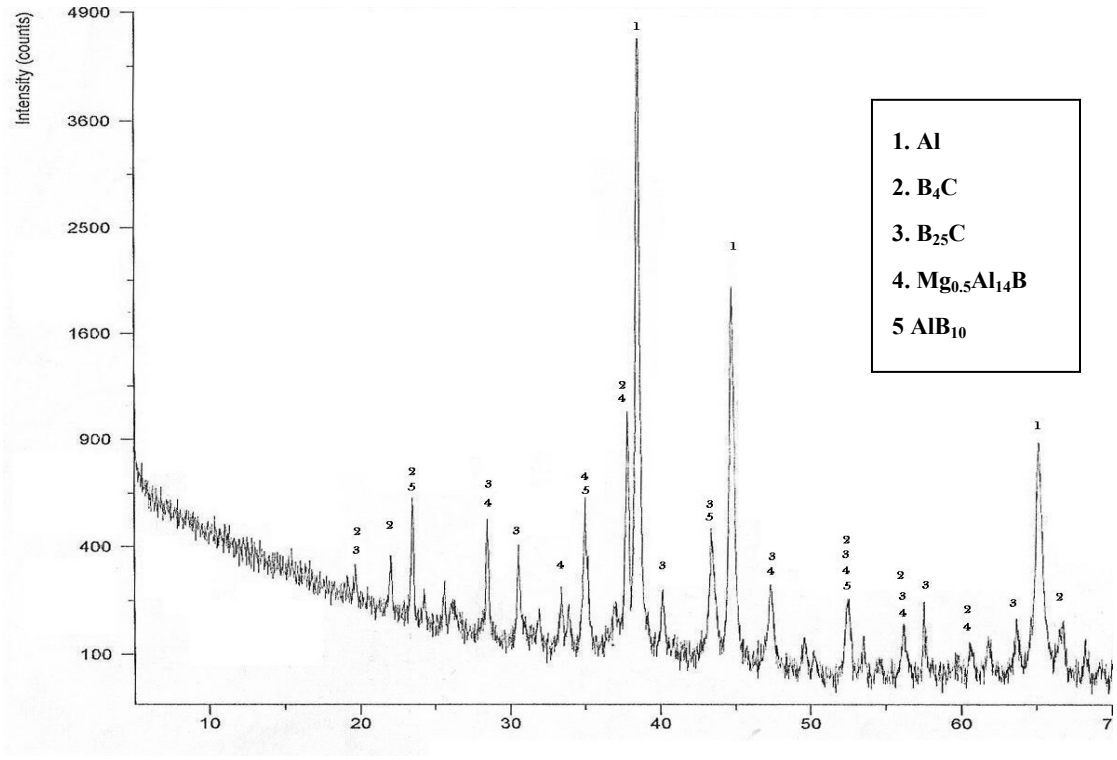
6.2 XRD Analyses

XRD patterns obtained from the pure Al and Al-8 wt% Mg alloy matrix composites are shown in Figure 6.6. The pattern belongs to pure Al matrix composites shows that composite composed of Al, B_4C and AlB_{10}. However, the presence of AlB_{10} in the composite is quiet abnormal. According to Viala et al. [46] AlB_{10} is not formed even at 1000^oC in Al-B4C composites. Halverson et al [47] reported formation of AlB_{10} c at temperatures above 1200^oC in Al-B_4C cermets. Beside these two studies Lee et al. [4] declared the precipitation of AlB_{10} at 800^oC due to interfacial reactions between Al and B_4C. The peaks of most expected AlB_2 type intermetallic compound did not appear on the XRD patterns.

The addition of Mg to the matrix resulted in formation of two additional intermetallic compounds ($B_{25}C$ and $Mg_{0.5}Al_{14}B$). Peaks of AlB_2 did not appear on the X-ray patterns as in pure Al matrix composites. As pointed out in Figure 6.5 (b) in Al-Mg alloy matrix composites intermetallic compound composed of Mg, Al, and B elements and therefore, XRD studies revealed precipitation of $Mg_{0.5}Al_{14}B$ type intermetallic compound. AlB_{10} still presents in the matrix along with $B_{25}C$ intermetallic compound.



(a)



(b)

Figure 6.6: X-Ray patterns of (a) pure Al matrix composite and (b) Al-8 wt% Mg matrix composite.

6.3 Results of Hardness Measurements

The hardness of the composites are listed in Table 6.3. It is obvious that Mg addition increased the hardness of the composites considerably. The increase of hardness by addittion of Mg can be attributed to the solid solution hardening of the matrix as well as intermetallic precipitation discussed in section 6.2. Figure 6.7 shows that increase of hardness with increasing Mg content is almost linear.

Table 6.3: Microhardness of pure and Al-Mg matrix composites

Matrix	B_4C Reinforcement size (μm)	Hardness (HV)
Pure Al	27,19	222.5±6
Al-% 1 Mg		265.8±20.0
Al-% 2 Mg		312.11±11.1
Al % 4 Mg		341.75±13.08
Al-% 8 Mg		505.8±14.7

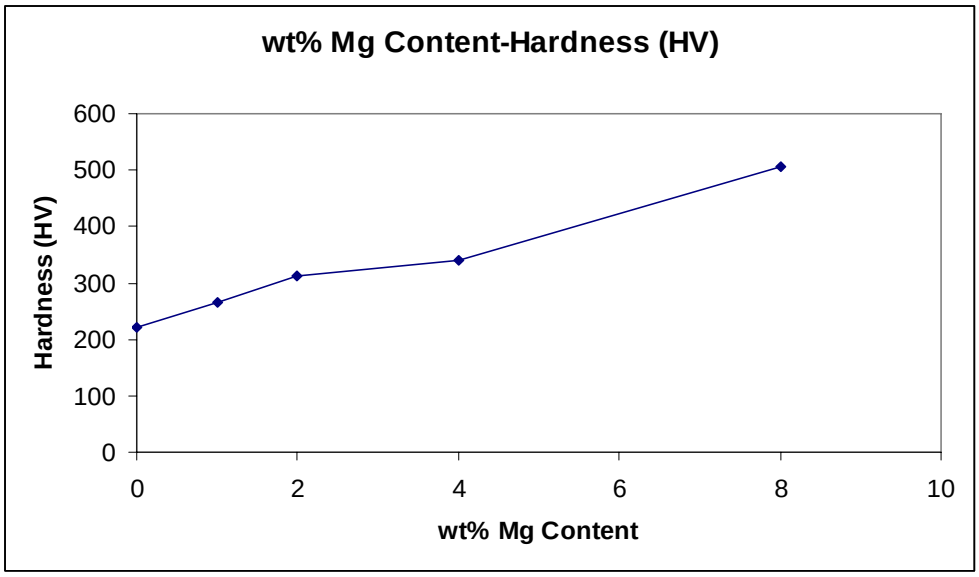


Figure 6.7: Hardness of the composite vs. Mg content of the matrix

6.4 Mechanical Properties

The results obtained from unaxial compression and impact tests are given in Table 6.4. The compression test and impact test results are plotted as a function of Mg content of the matrix in Figure 6.8.and Figure 6.9, respectively. The increase of Mg content of the matrix yielded an improvement in compression strength, however, caused a reduction in impact resistance.

Table 6.4: Compression strengths and impact resistances of the composites

Matrix	Compression Strength MPa	Impact Resistance (J/cm^2)
Pure Al	454.56	2.51±0.38
Al- 1 wt% Mg	459.08	1.265±0.10
Al- 2 wt% Mg	475.92	0.74±0.098
Al- 4 wt% Mg	574.96	0.6948±0.078
Al- 8 wt% Mg	618.89	0.6254±0.112

Compression strength increased very slightly up to Mg content of 2 wt%. Further addition of Mg increased the strength from about 450 MPa to 620 MPa, probably due to significant reduction in porosity and precipitation of intermetallic compounds. On the other hand Mg addition resulted in a dramatic reduction in impact resistance up to about 2 wt% Mg. At higher Mg contents impact resistance was kept almost constant.

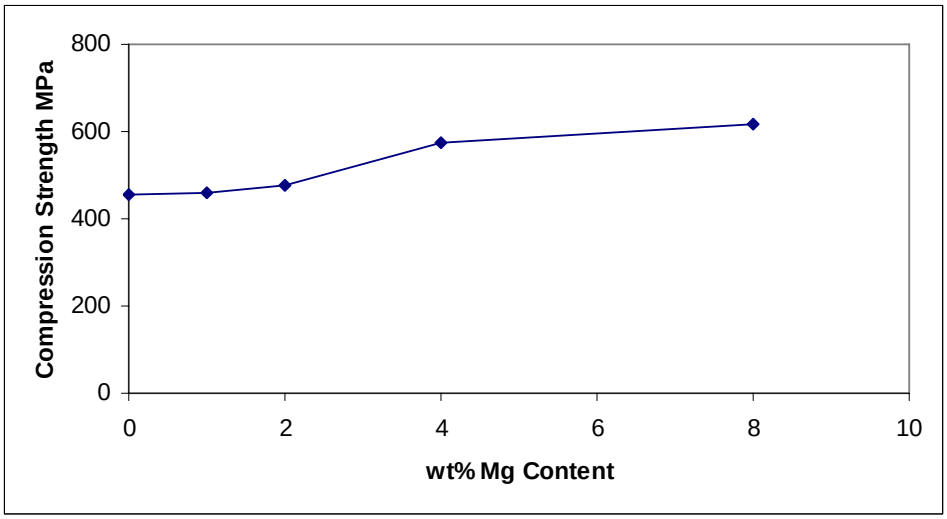


Figure 6.8: Variation of compression strength respect to Mg content

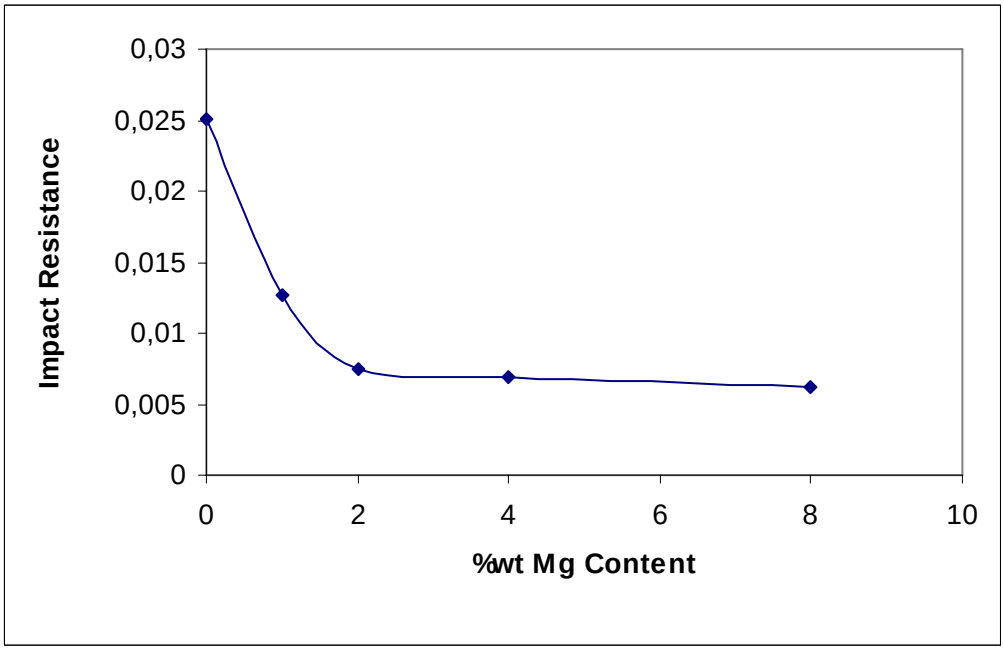


Figure 6.9: Variation of impact resistance respect to Mg content

6.5 Wear Test Results

6.5.1 Metal-Metal Wear Characteristics

The results of the metal-metal wear test are given as the weight loss-sliding distance graphs in Figure 6.10. The values for weight losses for each composite are listed in Table 6.5. Weight loss of the investigated composites increased parabolically; by having two regions which were called as “running-in” and “steady state” periods.

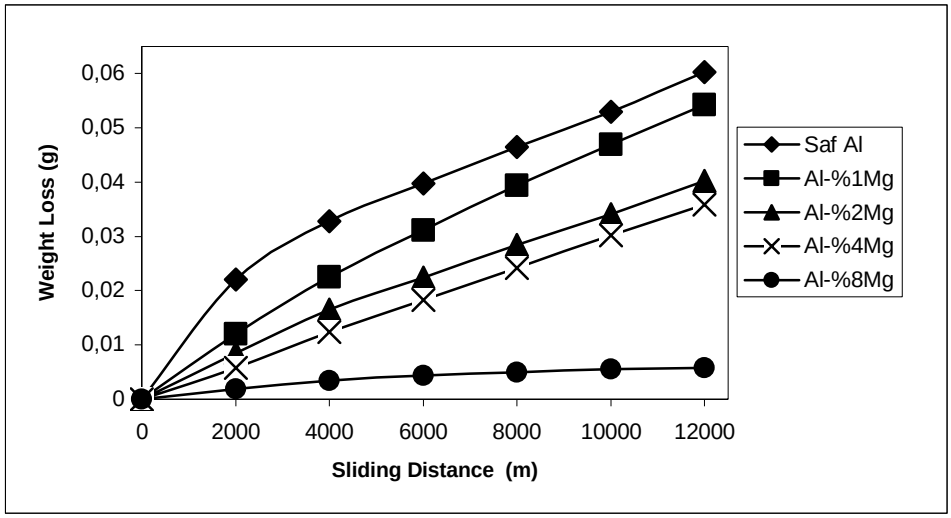


Figure 6.10: Weight Losses versus Sliding Distance Graph for Produced Composites

During running-in period, the weight loss increased remarkably with the increasing sliding distance. Transition from running in period to steady state period was achieved at sliding distance in between 2000 m and 4000 m, above which wear progressed linearly with increasing sliding distance. These two periods could not be observed clearly for the composites containing more than 2 wt% Mg. Figure 6.11 present the variation of wear rate obtained from the slope of steady state region with respect to Mg content of the matrix. Wear rate decreased significantly after 4 % Mg content of the matrix.

Table 6.5: Weight loss values for the each composite

Sliding Distance	2000 m	4000 m	6000 m	8000 m	10000 m	12000 m
Matrix	Weight Loss (gr)					
Pure Al	0.0221	0.0.328	0.0397	0.0465	0.053	0.0603
Al-%1 Mg	0,012	0,0225	0,0312	0,0394	0,0469	0,0543
Al-%2 Mg	0,0085	0,0165	0,0224	0,0284	0,0341	0,0402
Al-%4 Mg	0,0058	0,0124	0,0183	0,0242	0,0302	0,0359
Al-%8 Mg	0,0019	0,0034	0,0044	0,0049	0,0055	0,0058

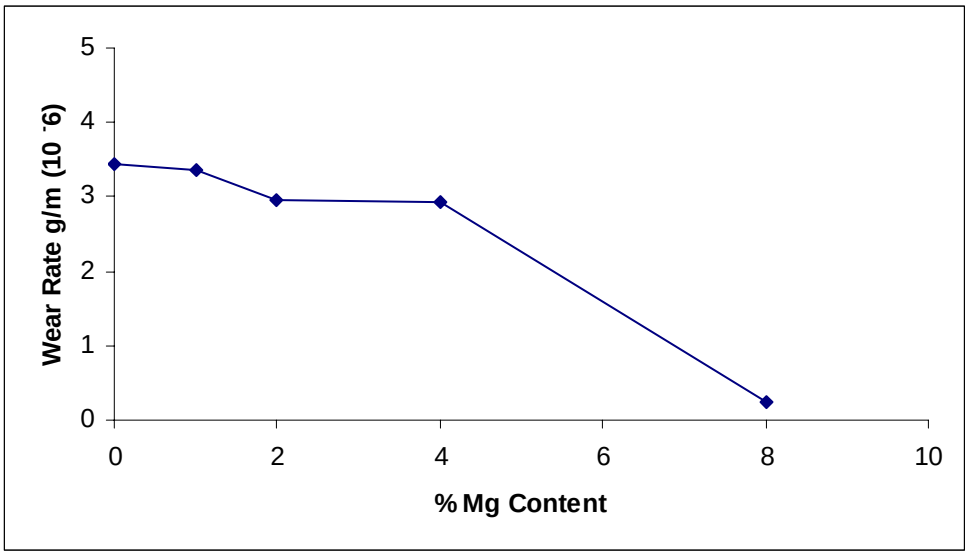
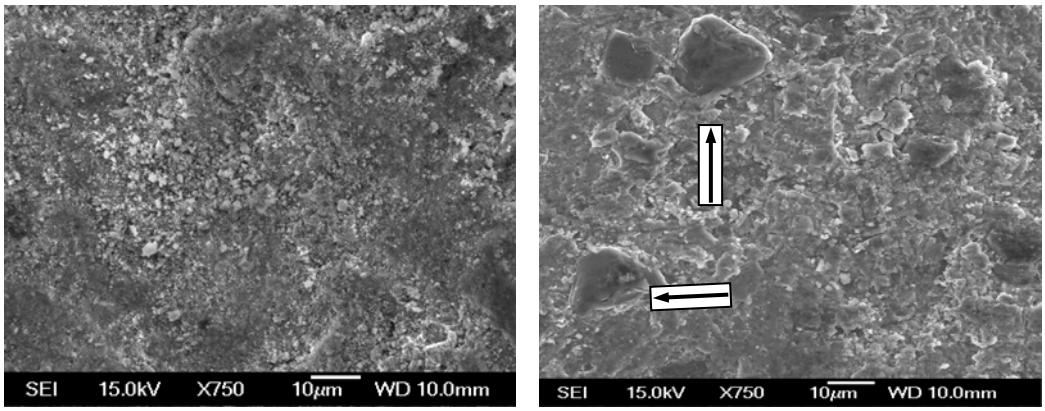


Figure 6.11: Variation of wear rate vs. Mg content of the matrix

Worn surface SEM micrographs of pure Al matrix and Al-8 wt% Mg matrix composites are presented in Figure 6.12. The boron carbide particles detected on worn surface of pure Al matrix composites and worn surface characteristics was indicating the dominant wear mechanism as severe wear. However, the worn surface of the Al-8 wt% Mg matrix composite was completely different from that of pure Al matrix composite and covered with a red-brown oxide layer, which is the typical characteristic of mild wear. The EDS analyses (Table 6.6) proved that for the Al-8 wt% Mg composite the worn surface mainly consists of Fe, Mo, Al, and Cr elements, which were detached from the counter-face tool steel disc. The EDS analyses for pure Al matrix composite describe a severe type wear.

Table 6.6: EDS analyses for pure Al and Al-Mg matrix composites

Al-8 wt% Mg Matrix Composite			Pure Al Matrix Composite		
Element	Weight%	Atomic%	Element	Weight%	Atomic%
C	4.99	10.83	B	40.77	55.98
O	39.34	64.02	C	13.14	16.24
Al	2.14	2.07	O	17.63	16.36
V	1.46	0.74	Al	14.47	7.96
Cr	2.19	1.09	Cr	0.46	0.13
Fe	42.37	19.75	Fe	11.68	3.11
Mo	3.33	0.90	Mo	0.91	0.14
W	4.18	0.59	W	0.93	0.08
Totals	100.00		Totals	100.00	



(a) **(b)**

Figure 6.12: SEM micrographs of worn surfaces (a) Al-8 wt% Mg (b) Pure Al matrix composite. Arrows point out the B_4C particles

6.5.2 Metal-Abrasive Wear Characteristics

The metal-abrasive wear test results are given in Table 6.7. The analysis of the test results were made on the basis of relative wear resistance by assuming the room temperature relative wear resistance of pure Al matrix composite as 100 %. The relative wear resistance of other composites at other temperatures were then calculated by dividing the weight loss values to the room temperature weight loss of pure Al matrix composite.

Figure 6.13 shows that at all testing temperatures wear resistance of the composites increased with the increasing Mg content. The composite containing 8 wt% Mg demonstrated the highest wear resistances at all temperatures. For certain Mg content wear resistance tended to decrease with increasing test temperature. Composites containing Mg higher than 1 wt% Mg, exhibited complex variation of wear resistance with respect to test temperature. The wear resistances of these composites decreased rapidly till 300^{o}C and tended to increase slightly in between 300 and 500 °C. At 600°C wear resistance of the composites decreased again.

Table 6.7: Weight loss values for metal-abrasive wear tests

Temperature	25°C	100°C	200°C	300°C	400°C	500°C	600
Matrix	Weight Loss 10^{-3} (g)						
Pure Al	49.2	78.6	84.1	101.4	92	102.5	105.2
Al-%1 Mg	41.8	82.2	82.2	82.4	87.2	94.3	80.5
Al-%2 Mg	39	70.5	74.05	77.7	78.7	68.4	98.6
Al-%4 Mg	30	56.7	70.2	69.6	53.8	54.3	83.8
Al-%8 Mg	18.9	30.8	37.3	58.7	46.3	44.9	98.7

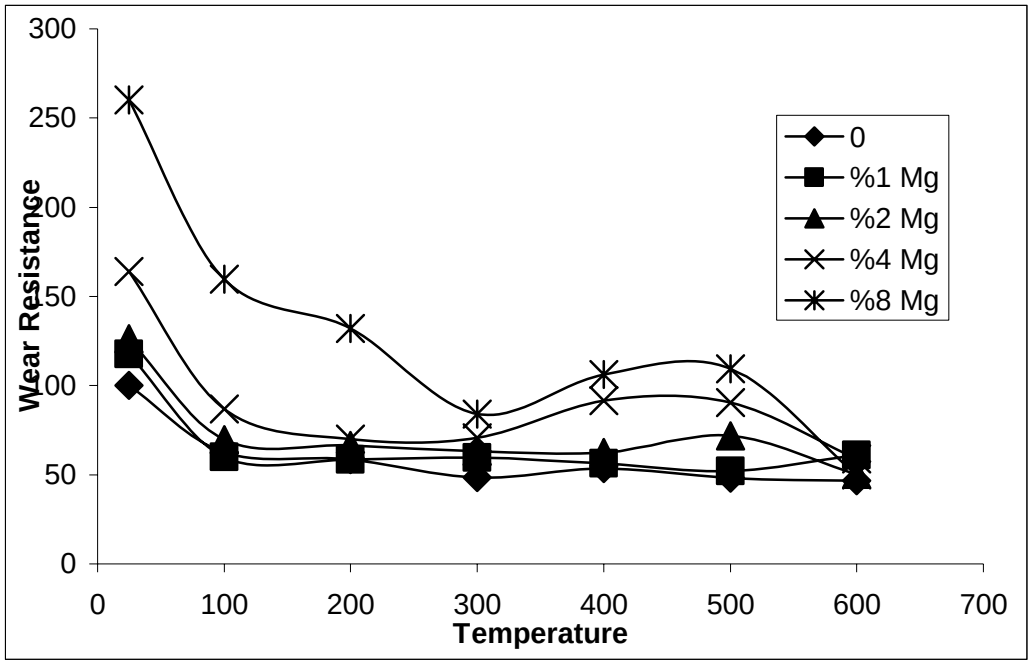
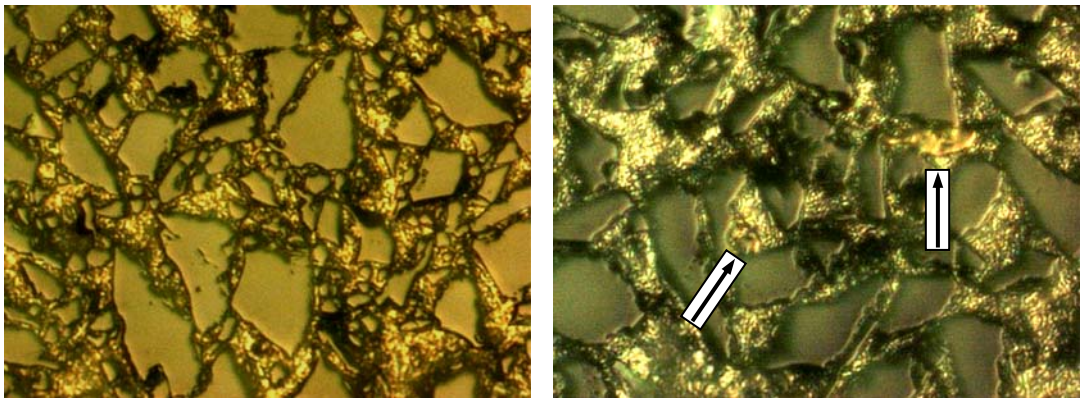


Figure 6.13: Variations of the wear resistance respect to temperature

In order to analyse this behaviour observed in between 300 and 500°C, samples were heated up to each temperature and kept in the furnace for 2 hours. These samples then cooled down rapidly in the water in order to maintain the elevated temperature microstructures at room temperatures. The samples then were prepared for the metallographic investigation as mentioned previously.

The metallographic investigations showed no considerable difference in between the room temperature and elevated temperature microstructures of pure Al-matrix composites. However, an intermetallic compound was precipitated at 600°C as can

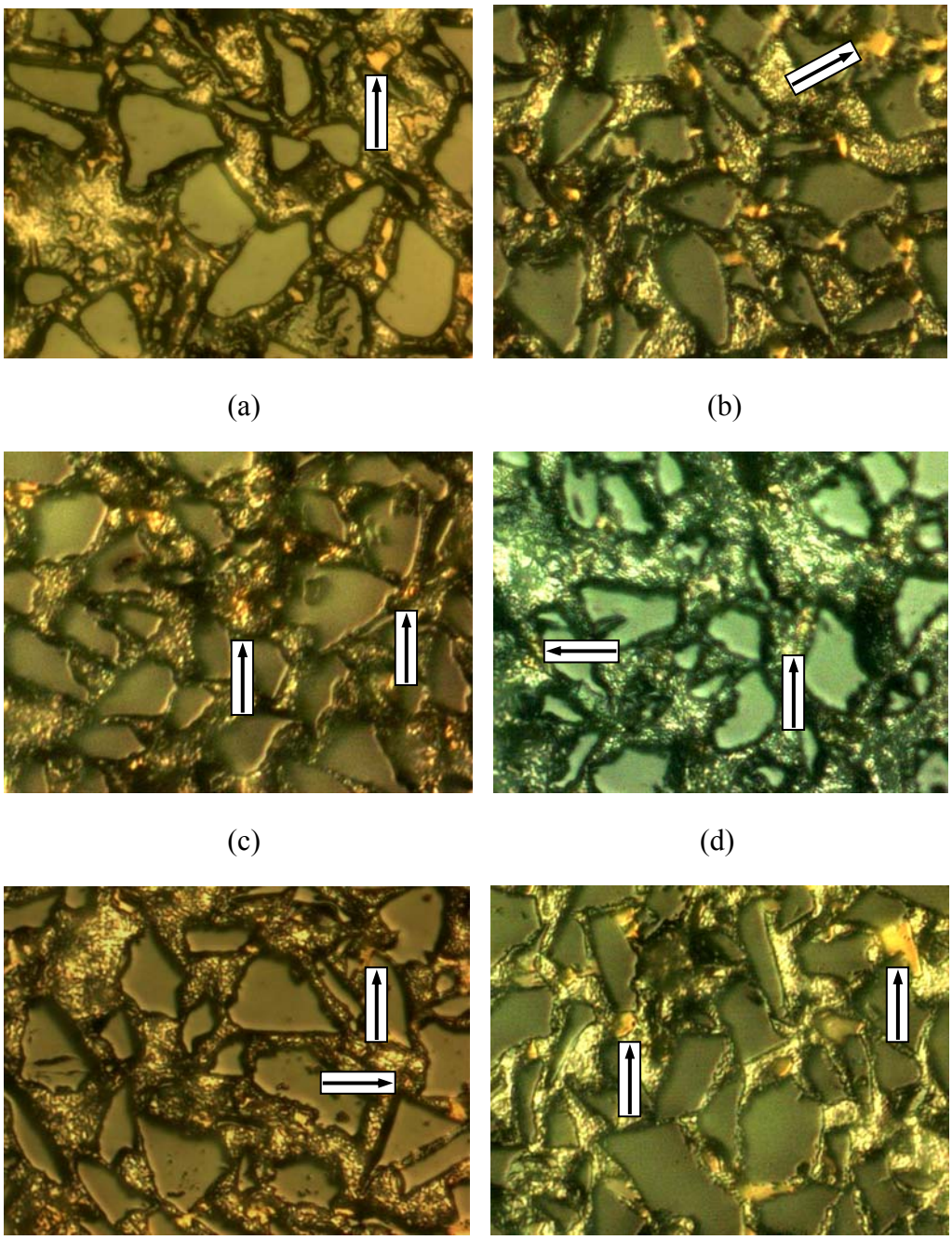
be seen in Figure 6.14b. As mentioned previously it was reported [45-47] that AlB_2.precipitation reaction starts at 600°C in Al-B_4C systems.



(a) **(b)**

Figure 6.14: The microstructures of pure Al matrix composites at different temperatures (a) Microstructure at room temperature (b) Microstructure at 600°C. Arrow points the intermetallic compounds.

Al-Mg matrix composites exhibited different precipitation behaviour than pure Al matrix composites. Figure 6.15 illustrates the microstructures of Al-8 wt% Mg composite at elevated temperatures. The microscopic examinations showed that, at 300°C the intermetallic compounds particles start to dissolve in the matrix. At 400°C they were almost completely dissolved and started to re-appear at 500°C. At 600°C intermetallic compounds became clearly visible by an optical microscope. Therefore, improved wear resistance of Al-Mg alloyed matrix composites in between 300 and 500 °C can be attributed to the dissolution of intermetallic compounds. Their re-appearance at 600°C resulted in a reduction in wear resistance.



(a) (b)

(c) (d)

(e) (f)

Figure 6.15: The microstructures of Al-8 wt% Mg matrix composite at temperatures (a) 100°C (b) 200°C (c) 300°C (d) 400°C (e) 500°C (f) 600°C. Arrows points the intermetallic compounds.

7. CONCLUSION

The results of this study conducted on B4C reinforced pure Al matrix and Al-Mg matrix composites produced by gas pressure infiltration technique can be summarised as follows.

1. The amount of the porosity; which were mainly located by the tips of the B_4C particles; decreased with increasing Mg content of the matrix. The X-Ray patterns revealed precipitation of AlB_{10} intermetallic in both pure Al and Al-Mg matrixes. Al-Mg alloy matrixes contain $B_{25}C$ and $Mg_{0.5}Al_{14}B$ type intermetallic compounds in addition to AlB_{10}.

2. The hardness of the composites increased linearly by addition of Mg. Mg content in the matrix also increased the strength of the composite especially if the Mg content is higher than 2 wt%. On the other hand, the impact resistance of the composite decreased dramatically with increasing Mg content.

3. On M2 quality tool steel, wear progressed in two sequential periods namely “running-in” and “steady state”. In the earlier period (running-in period) the composites exhibited high wear rate. In the steady state period, wear rate tended to increase slightly. The decrease of wear rate during the metal-metal wear tests is due to the formation of iron rich transfer layer on the contact surfaces. For the investigated range of Mg, the metal-metal wear resistance of the composites increased when the Mg content of the matrix is higher than 4 wt%.

4. The metal-abrasive wear resistance of the investigated composites decreased with increasing test temperature and Composites containing more than 1 wt% Mg in their matrix exhibited a slight increment in wear resistance in temperature range between 300 and 500°C, due to dissolution of intermetallic compounds decreasing the Mg content of the matrix.

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