

DOKUZ EYLÜL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED
SCIENCES

PRODUCTION AND CHARACTERIZATION OF
AL-BASED METAL MATRIX COMPOSITES BY
HIGH ENERGY BALL MILLING AND SQUEEZE
CASTING TECHNIQUES

by

Ahmet SAĞLAM

June, 2008

İZMİR

PRODUCTION AND CHARACTERIZATION OF AL-BASED METAL MATRIX COMPOSITES BY HIGH ENERGY BALL MILLING AND SQUEEZE CASTING TECHNIQUES

**A Thesis Submitted to the
Graduate School of Natural and Applied Sciences of Dokuz Eylül University
In Partial Fulfillment of the Requirements for the Degree of Master of Science
in Metallurgy and Materials Engineering, Metallurgy and Materials Program**

by

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M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled **“PRODUCTION AND CHARACTERIZATION OF AL-BASED METAL MATRIX COMPOSITES BY HIGH ENERGY BALL MILLING AND SQUEEZE CASTING TECHNIQUES”** completed by **AHMET SAĞLAM** under supervision of **ASSIST. PROF. DR. İSMAİL ÖZDEMİR** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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Ahmet SAĞLAM

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ABSTRACT

In this study, composites of an aluminium-copper alloy EN AW-2017 (3.9Cu, 0.6Mn, 0.7Mg, bal. Al, wt.%) containing 5 and 15 vol. % particulate silicon carbide reinforcement matrix alloy samples were successfully produced by high-energy ball milling and squeeze casting process.

The effect of squeeze pressure during solidification was evaluated with respect to microstructural characteristics using optical microscopy and SEM. This study presents the effect of SiC particles on the aging behavior, and mechanical properties. Squeeze cast microstructure showed relatively uniform reinforcement distribution.

The porosity level of composites was found to be below 0.5 vol.%. T6 heat treatment was applied for the matrix and composites. In composites, hardness increased with increasing volume fraction of SiC. After T6 heat treatment, The composite with 15 vol.% SiC_p exhibited higher increase in hardness in comparison to matrix and the composite 5 vol.% SiC_p.

Ultimate tensile strength, yield strength and elongation the produced of 5 vol.% SiC composite were found to be 213 MPa, 130 MPa and 3.73% respectively. For 15 vol. % SiC composites, ultimate tensile strength, yield strength and elongation were measured as, 208 MPa, 190 MPa and 3.16% respectively. Fracture behaviour showed that, the failure in composites occurs in both the matrix and particles simultaneously.

Keywords: Metal matrix composites; Squeeze casting; Al/SiC particulate composite; Fracture.

METAL MATRİKSİLİ KOMPOZİTLERİN YÜKSEK ENERJİLİ BİLYALI DEĞİRMENLE VE SIKIŞTIRMALI DÖKÜM YÖNTEMİYLE ÜRETİMİ VE KAREKTERİZASYONU

ÖZ

Bu çalışmada, EN AW-2017 (%3,9 Cu, %0,6 Mn, %0,7 Mg, ve %94,8 Al) matriksli hacimce % 5 ve % 15 oranında SiC partikül takviyeli metal esaslı kompozitlerin, yüksek enerjili bilyalı değirmenle ve sıkıştırırmalı döküm yöntemlerini kullanarak başarılı bir şekilde üretimi gerçekleştirilmiştir.

Katılaşma esnasındaki sıkıştırma basıncının, mikro yapı üzerindeki etkisi ışık mikroskobu ve taramalı elektron mikroskobu kullanılarak incelemelerde bulunulmuştur. SiC partiküllerinin yaşlanma davranışı ve mikro yapı üzerine etkisi incelenmiştir. SiC partiküllerinin mikro yapı içerisinde genelde homojen dağıldığı da görülmüştür.

Kompozit malzemelerimizin porozite oranlarını % 0,5'in altında olduğu saptanmıştır. Anayapı ve kompozit malzemeler için T6 ısıt işlemleri uygulanmıştır. Kompozit malzemelerde SiC partiküllerinin artan hacim oranıyla sertlik değerlerinde de artış gözlenmiştir. T6 ısıt işlemleri sonrası % 15 SiC içeren kompozitin, % 5 SiC içeren kompozit malzememize göre daha fazla sertlik artışı gözlenmiştir.

Üretilen % 5 SiC takviyeli kompozit malzememizin çekme mukavemeti, akma mukavemeti ve uzama değerleri sırasıyla 213 MPa, 130 MPa ve % 3,73 tür. % 15 SiC takviyeli kompozit malzemenin ise sırasıyla çekme mukavemeti, akma mukavemeti ve uzama değerleri de, 208 MPa, 190 MPa ve % 3,16 dır. Kompozitlerin kırılma davranışı, kompozitlerde hasarın anayapı ve katkı fazında aynı anda gelişerek meydana geldiği görülmüştür.

Anahtar Sözcükler: Metal anayapılı kompozit, Sıkıştırırmalı döküm, Al/SiC partiküllü kompozit, Kırılma.

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CHAPTER ONE

INTRODUCTION

The need for new engineering materials especially in the space and automotive industries has caused metal matrix composite (MMC) materials to develop rapidly. MMC materials are preferred more owing to the characteristics such as high durability and hardness, high abrasion resistance, high thermal performance, high thermal and mechanical fatigue resistance and creep resistance than monolithic materials (Tekmen, 2006).

Metal matrix composites (MMC_s) have found commercial use in some areas and are rapidly becoming strong candidates as a structural material for high-temperature and aerospace applications. The development of these materials started with the production of continuous fiber reinforced composites. The high cost and difficulty of processing these composites restricted their applications and led to the development of discontinuously reinforced composites. Among these, particulate metal-matrix composites are likely to reach the largest commercial application stage with their low cost, ease of fabrication, and improved properties. The main objective of using metal matrix composite system is to improve specific properties of structural components by replacing existing alloys. However, only in the past few years have these composites become realistic contenders as engineering materials. Mass-market products, like automobiles, now contain MMC components (Kaczmar, Pietrzak & Wlosinski 2000; Ozdemir, 2002).

Production and usage of the composites with metal matrix, with the technological development which has been acquired in the last 20 years, has increased especially in the automotive industry, space and aviation sector. Metal matrix composites are made of the combination of two diverse systems. At least one of these systems is made of metal or composition metal; and the other one is made of durable fiber, capillary crystal or particle shaped ceramic reinforcement such as SiC, TiC, B₄C and Al₂O₃ in order to obtain the characteristics such as high elasticity module, high

strength and high abrasion resistance which are unable to be obtained by one-component materials (Ahlatçı, Candan & Çimenoglu, 2006).

Metal-matrix composites (MMCs) have emerged as potential alternatives to conventional alloys in high-strength and stiffness applications. Cost is the key factor for their wider application in modern industry, although potential benefits in weight saving, increased component life, and improved recyclability should be taken into account. Today, even in those terms, MMCs are still significantly more expensive than their competitors. Cost reductions can be achieved only by simpler fabrication methods, higher production volumes, and cheaper reinforcements (Tekmen, 2006).

Parallel to commercialisation, there are research centres throughout the world that are actively researching further development and exploitation of net or near net shape fabric-based alloys with special emphasis on metal matrix composites MMCs. The search for cheaper, easily available reinforcements has led to the wider use of SiC particles. As a result, although they offer only moderate property improvements particle MMCs, are now dominating the MMC market. The particulate reinforced aluminium matrix composites not only have good mechanical and wear properties, but are also economically viable. Therefore, SiC-particulate-reinforced aluminium composites have found many applications in production process. This is evidenced by the publication of more than thousands papers in various engineering and scientific journals (Onat, Akbulut & Yilmaz, 2006).

Aluminum and aluminum alloys among many matrix alloys are commonly used in the process of production of metal matrix composites. This results from the fact that these alloys are light, and can be economically produced by various production methods, and that they have high durability and corrosion resistance. The most commonly used phases as fiber, whisker or particle for aluminum based MMC are silicon carbide, and zirconia. The most commonly used production methods in the process of producing aluminum based MMC are mixed casting, compressing casting, compo casting, liquid metal absorption, spraying and dust metallurgy. Compressing casting method is especially preferred due to the fact that it is economical and

improves the internal structure and mechanical characteristics of the composite material as it is hardened under pressure (Tekmen, 2006).

The aim of this study was to produce metal matrix compound of Al-3.9Cu-0.6Mn-0.7Mg (EN AW- 2017) reinforced with 5 vol.% and 15 vol.% SiC particles by the squeeze casting method with improved properties. The effect of volume content of SiC particles on the aging behavior, mechanical properties, aluminum alloy microstructure, and hardness of resultant composites were investigated using optical microscopy, scanning electron microscopy, tensile and hardness tests.

CHAPTER TWO

PRODUCTION METHODS OF MMC

2.1 Introduction

As metal matrix composite materials meet different needs by the combination of various characteristics, they are commonly used in many engineering applications especially in automotive, aircraft and space industries.

The development of metal matrix composites (MMCs) can be regarded as one of the major innovations in materials in the past 30 years and they have been predicted to find extensive applications in industry. Currently available MMCs are either too brittle (very low damage tolerance) or too expensive to be used in many engineering applications. The successes in exploiting the high strength or modulus of reinforcement are shadowed by the failure to exploit the ductility of the matrix. Many MMC researchers have followed conventional materials outlook and pursued a homogeneous microstructure, perhaps failing to appreciate the unique characteristics of MMC. Therefore, little effort has been made to tailor the architecture (morphology and spatial distribution) of the reinforcement (Peng, Fan, Mudher & Evans, 2001).

Especially in aerospace and automotive industries, the necessity of new engineering materials and the progress in modern technology are the most important motivations for the development of composites. Metal matrix composites (MMC) are quite attractive due to their high strength and hardness, high wear resistance, high thermal and mechanical fatigue resistance and creep resistance (Tekmen, 2006).

In recent years, stringent requirements of material quality in automotive and aerospace industries have necessitated the development of lightweight aluminum alloys. In this context, it is known that reinforcing of aluminum alloys with discontinuous second phase particles offers high strength, high modulus, superior wear resistance, good workability, desirable thermal expansion and isotropy. This

excellent combination of properties makes composites very attractive for industrial application. Metal matrix composites (MMC) are generally produced either by liquid metal metallurgy or powder metallurgy techniques. In the former, the particulate phases are mechanically dispersed in the liquid before solidification of the melt. Here with, squeeze casting method is known as a very promising route for manufacturing near net shape MMC components at relatively cost (Reihani, 2004).

The primary advantage of advanced composites is their ability to provide higher mechanical properties and tailored physical properties at less weight than that of conventional materials. In the case of MMC_s these advantage are available for high-temperature applications ($>316^{\circ}\text{C}$) where PMC_s are inadequate. Furthermore, because metals are more electrically and thermally conductive, MMC_s can be used in heat dissipation and electronic transmission applications (Clyne & Withers, 1995).

The addition of a reinforcement to metal can be characterized as follows. It can be ceramic in nature with high strength and high stiffness retention at high temperature, it can have physical attributes such as high heat transfer or low thermal expansion characteristics, or it can exhibit high wear resistance. Being very small in size, generally less than the diameter of human hair (0.13 mm), these reinforcements do not suffer from the flaws and defects generally attributed to bulk materials of the same chemistry. Although MMC_s with widely different properties exist, some general advantages of these materials over monolithic (unreinforced) metals and PMC_s can be cited. Compared with monolithic metals, MMC_s have higher strength to density and stiffness to density ratios, better fatigue resistance and superior temperature properties, lower CTE_s, and better wear resistance. The current disadvantages of MMC_s are those associated with higher material and fabrication costs, lack of material databases, and limited service experience. In addition, because of the complexity of these multiphase materials and their less mature technology, the costs of design, analysis, and quality control are greater. However, because of their unique engineered properties, the use of MMC systems is projected to accelerate rapidly. As this happens, the cost and experience factors will improve (Schwartz, 1996).

In the short term, application of aluminum based MMC in the automotive industry is currently made on motor parts and braking system parts which are made of steel and cast iron. In the long term, regarding the applications which require high rigidity, use of aluminum based MMC sheets instead of steel and aluminum are the potential applications. The fact that commercial vehicles are lighter, and intensive researches which have been made in order to reduce environment emission and increase fuel saving, increase of MMC (metal matrix composites) in automotive applications will continue in the future (Tekmen, 2006).

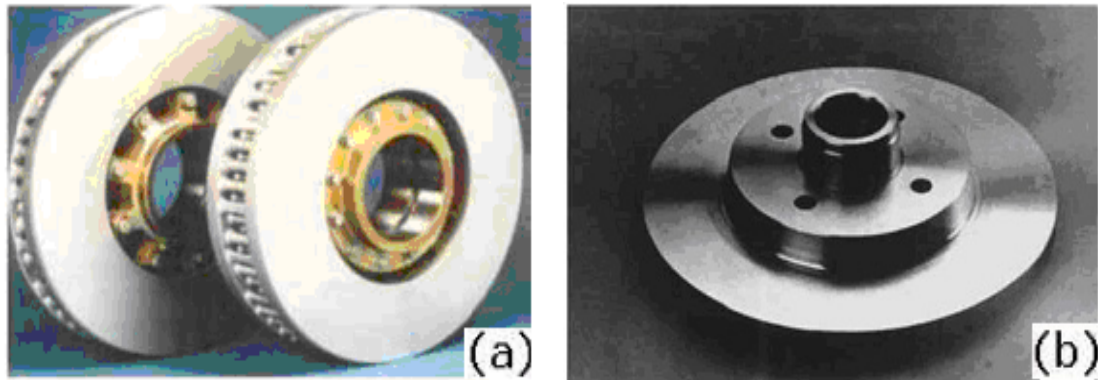


Figure 2.1 (a) Fast train brake disc produced from AlSi_7Mg based SiC particle reinforced composite material (Cayron, 2000), and (b) Car brake disc made from 20 vol.% SiC particle reinforced A359 aluminum alloy (Kaczmar & et. al., 2000).

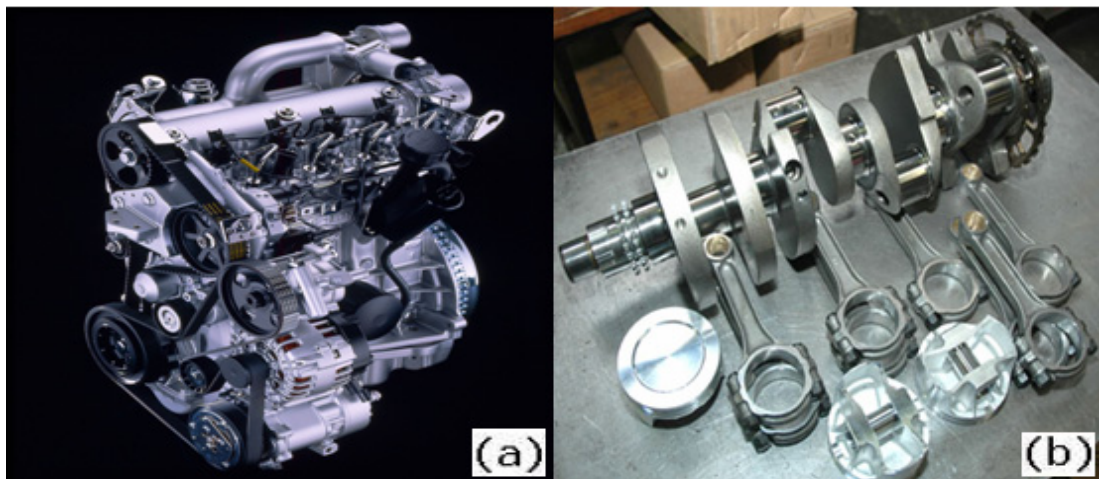


Figure 2.2 (a) Motor Block and (b) motor piston rod (Anonymous, 2007)

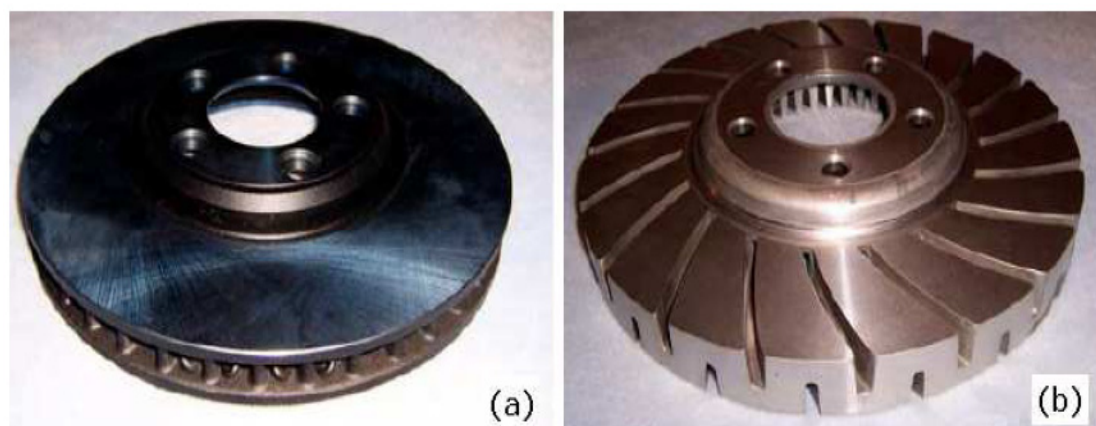


Figure 2.3 (a) Brake disc made from gray cast iron and used in 4-door sedan automobile. (Weight: 8.44 kg) (b) Brake disc made from Al-SiC composite used in sedan automobile (Weight: 3.32 kg) (Metalurji, 2007).

The fact that reinforcement phase is more expensive than aluminum alloy and the difficulty of processing the part owing to rigid reinforced phase is increasing the cost. Today, various MMC production methods are being developed in order to improve cost/performance characteristic. Usage rates of matrix and reinforcement phases used by the industrial firms in the process of MMC production have been presented in Fig. 2.4.

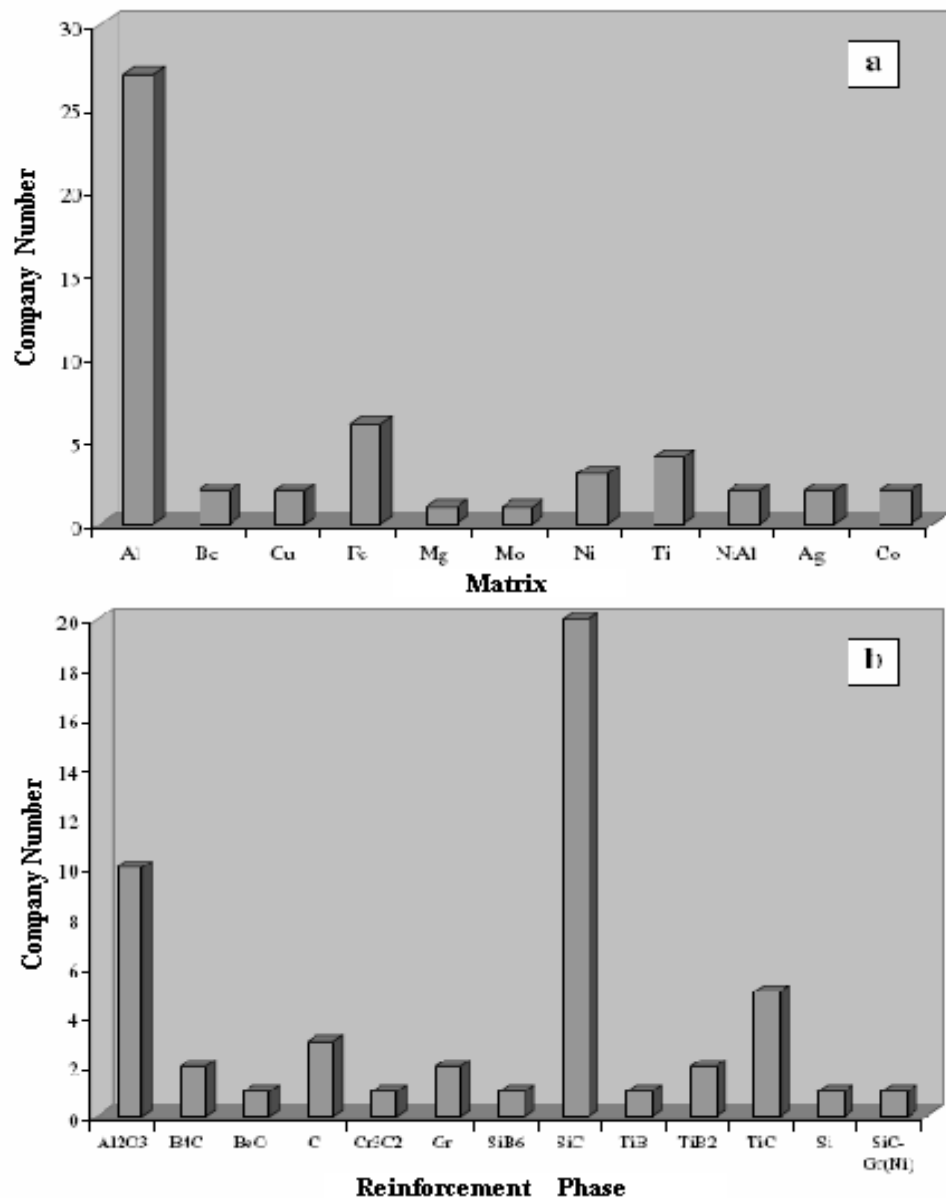


Figure 2.4 Usage rates of matrix and reinforcement phases used by industrial firms (Mortensen, ud).

2.2 Metal Matrix Composite Production Methods

A variety of processes have been and are being developed for the manufacture of MMCs. It is important to note from the outset that making the right choice of fabrication procedure is just as important in terms of microstructure and performance of a component, as it is for its commercial viability. However, before looking in detail at the various processing options, it is worthwhile dwelling for a moment on selection of the reinforcement. Clearly, the size, shape and strength of the reinforcing particles or fibres is of central importance. Often the choice between the continuous and discontinuous options is relatively straightforward, both in terms of performance and processing cost. However, within each category there exist wide variations in reinforcement size and morphology (Clyne & Withers, 1995).

As an example, consider particulate reinforcement. The most convenient form is SiC powder of about 3-30 nm diameter, which is cheap (largely because of the mature market for its use as an abrasive) and relatively easy to handle. Extensive research is currently being undertaken on the influence of particle size on properties, especially with respect to failure processes (Mortensen, Drevet & Eustathopoulos 1997). Fig 2.5 shows the morphology of the aluminum matrix powder produced by gas atomization (Ozdemir, Ahrens & Mücklich, 2008).

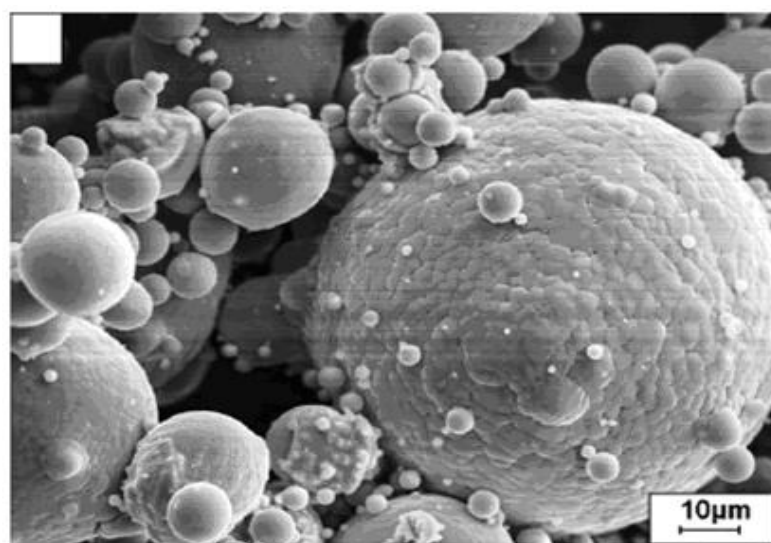


Figure 2.5 SEM micrographs show morphology of the as-received state aluminum matrix, topography (Ozdemir & et. al., 2008).

The rates of production methods preferred by the industrial firms in the process of MMC production have been presented in Fig. 2.6. Additionally; the rates of secondary processes applied to the produced composite materials by the firms have been presented in Fig. 2.7. Fig. 2.8 shows the typical as-received morphology of SiC produced by crushing and acidic washing.

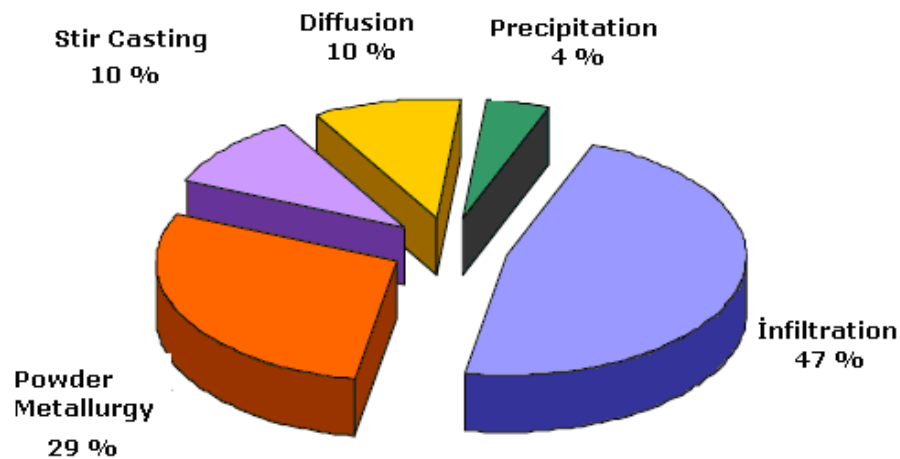


Figure 2.6 The rate of production methods preferred by industrial firms (Mortensen, ud).

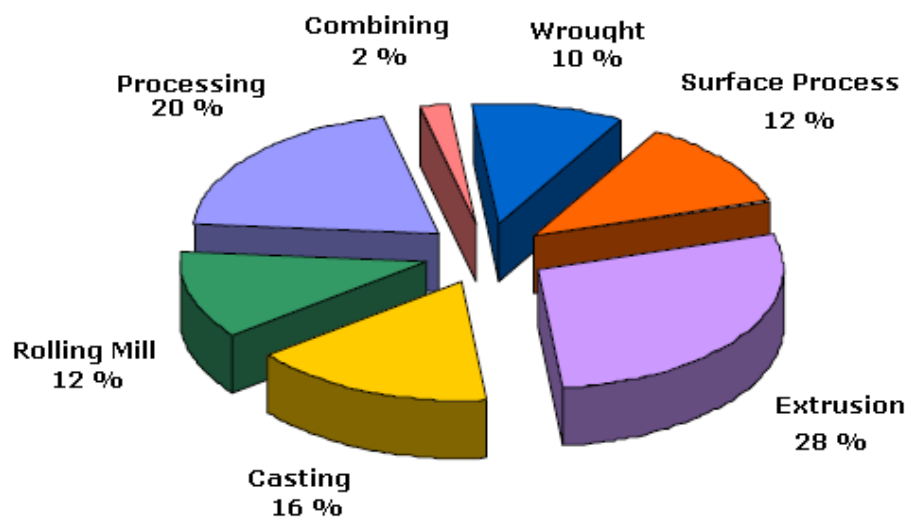


Figure 2.7 The rate of secondary treatments preferred by industrial (Mortensen, ud).

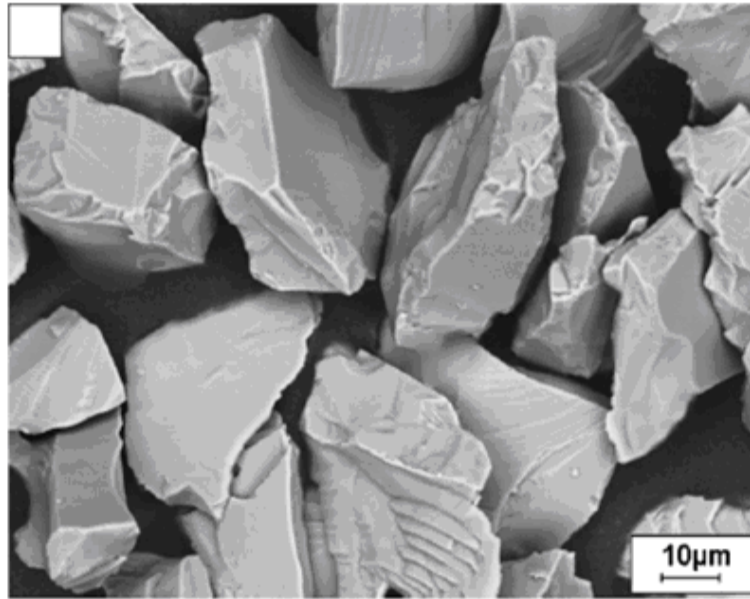


Figure 2.8 SEM micrographs show morphology of the as-received state SiC powders (Ozdemir & et. al., 2008).

Alumina has been produced for MMC use in the form of monocrystalline platelets of aspect ratio about 5-25 and diameter of the order of 10 nm. These can readily be assembled into preforms for melt infiltration, often with better control over volume fraction than is possible with equiaxed reinforcement. However, given the pressures to manufacture MMCs under economically attractive conditions, particularly for Al-based particulate MMCs aimed at bulk markets, the benefits of changing the type or nature of the reinforcement must first be clearly established and understood if such a change is accompanied by increased processing or raw materials costs (Clyne & Withers, 1995).

MMCs production methods can be classified as below;

1- Solid Phase Product Processing

- MMC Diffusion Bonding
- Powder Metallurgy

2- Vapour – Phase Product Processing

- Physical Vapor Deposition (PVD)

3- Liquid Metal Product Processing

- Stir Casting
- Compo Casting
- Spray Forming
- In-Situ MMC
- Liquid-Metal Infiltration
- Squeeze Casting

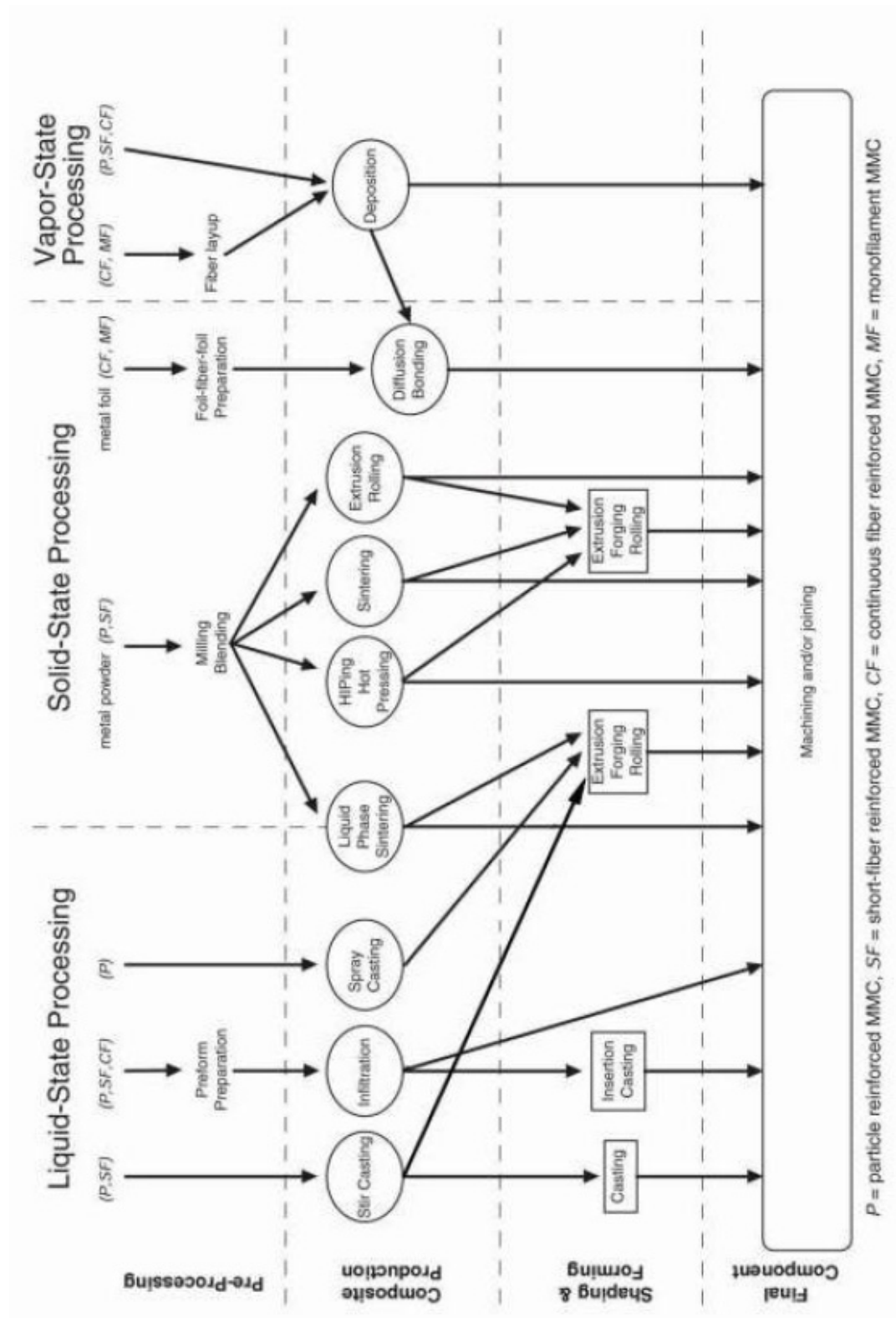


Figure 2.9 Flow chart indicating production methods of MMC (Clyne, 2001).

2.2.1 MMC Diffusion Bonding

Diffusion bonding is a common solid state welding technique used to join similar or dissimilar metals. Inter diffusion of atoms from clean metal surfaces in contact at an elevated temperature leads to welding. There are many variants of the basic diffusion bonding process; however, all of them involve a step of simultaneous application of pressure and high temperature. Matrix alloy foil and fiber arrays, composite wire, or monolayer lamina are stacked in a predetermined order. Fig. 2.10 shows a schematic of one such diffusion bonding process, also called the foil-fiber-foil process. The starting materials in this case were made by sputter coating the SiC fibers with titanium. Filament winding was used to obtain panels, about 250 μm thick. Four such panels were stacked and hot pressed at 900 $^{\circ}\text{C}$, under a pressure of 105 MPa for 3 hours. Vacuum hot pressing is a most important step in the diffusion bonding processes for metal matrix composites. The major advantages of this technique are the ability to process a wide variety of matrix metals and control of fiber orientation and volume fraction. Among the disadvantages are processing times of several hours, high processing temperatures and pressures that are expensive, and objects of limited size can be produced. Hot isostatic pressing (HIP), instead of uniaxial pressing, can also be used. In HIP, gas pressure against a can consolidates the composite piece contained inside the can. With HIP, it is relatively easy to apply high pressures at elevated temperatures over variable geometries (Chawla, 1998).

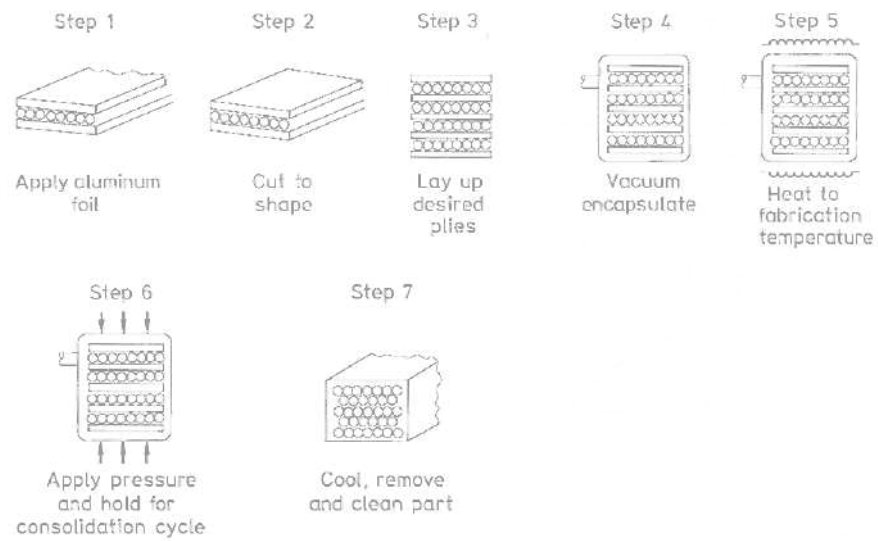


Fig. 2.10 Schematic of diffusion bonding process (Chawla, 1998)

Diffusion bonding is used for consolidating alternate layers of foils and fibers to create single or multiple-ply composites. After creep flow of the matrix between the fibers to make complete metal-to metal contact, diffusion across the foil interfaces completes the process. Pressure time requirements for consolidation can be determined from matrix flow stress, taking into consideration the above mentioned matrix flow processes. To avoid fiber degradation, care must be exercised to maintain low pressure during consolidation. Furthermore, obtaining complete flow in the interstices between the fiber midplane and the foil segments on either side is extremely difficult. This is accomplished at a temperature where the matrix is in either a soft or a superplastics state. Examples of this type of composite currently in use are aluminum or titanium alloys reinforced with SiC, B, or other fibers. Diffusion bonding is performed either by loading a vacuum pack in a hot press or by a hot isostatic pressing operation. In either case, thorough outgassing of the pack prior to consolidation is required (Chawla, 1998).

Roll bonding and extrusion have also been used for solid-state bonding of metallic foils and fibers and ceramic particulates in metallic matrix. Powder blends packed and evacuated in a container can be subjected to these consolidation methods. Laminated composites are ideally produced by high temperature roll bonding operations starting

from either foils of individual metals or from alloys. During roll bonding, both surface deformation and diffusion actively cause hard deformation and interdiffusion to produce strong interfaces.

Deposition processes for fiber composites generally yield a monotape that can be stacked in layers and diffusion bonded into a composite. Some degree of porosity is usually present in the monotape but this can be eliminated during diffusion bonding or during hot isostatic pressing. W-fiber reinforced Ni-base alloy monotapes produced by this method have been used for the fabrication of lightweight, hollow turbine blades for a high performance engine. The process involves placing monotapes around a bent steel core and diffusion bonding around a bent steel core and diffusion bonding several monotape layers. Near-net shape blades are produced after the steel core is removed by acid. This method is currently being applied to fiber-reinforced titanium matrix composites to improve fiber distribution compared and decrease composite mechanical properties (Suresh, Mortensen & Needleman, 1993).

2.2.2 Powder Metallurgy

Powder metallurgy is the most common method for fabricating metal-ceramic and metal-metal composites. With the advent of rapid solidification technology, the matrix alloy is produced in a prealloyed powder form rather than starting from elemental blends. After blending the powder with ceramic reinforcement particulates (or whiskers), cold isostatic pressing is utilized to obtain a green compact that is then thoroughly outgassed and forged or extruded. In some cases, hot isostatic pressing of the powder blend is required, prior to which complete outgassing is essential (Suresh & et. al., 1993).

Among the various metal working technologies, powder metallurgy (P/M) is the most diverse manufacturing approach. One attraction of P/M is the ability to fabricate high quality, complex parts to close tolerances in an economical manner. In essence, P/M takes a metal powder with specific attributes of size, shape, and

packing, and then converts it into a strong, precise, high performance shape. Key steps include the shaping or compaction of the powder and the subsequent thermal bonding of the particles by sintering. The process effectively uses automated operations with low relative energy consumption, high material utilization, and low capital costs. These characteristics make P/M well aligned with current concerns about productivity, energy, and raw materials. Consequently, the field is experiencing growth and replacing traditional metal-forming operations. Further, powder metallurgy is a flexible manufacturing process capable of delivering a wide range of new materials, microstructures, and properties. That creates several unique dimple applications for P/M such as wear resistant composites.

The applications of P/M are quite extensive. Consider the use of metal powders in the fabrication of tungsten lamp filaments, dental restorations, oil-less bearings, automotive transmission gears, armour piercing projectiles, electrical contacts, nuclear power fuel elements, orthopaedic implants, business machines, high-temperature filters, aircraft brake pads, rechargeable batteries, and jet engine components. Furthermore, metal powders find uses in such products as paint pigments, porous concretes, printed circuit boards, enriched flour, explosives, welding electrodes, rocket fuels, printing inks, brazing compounds, and catalysts. Worldwide market share for P/M parts are given in the following pie chart. Automotive industry is leading with more than 75% of the share. American cars contain more than 16 kg of P/M parts while European cars have 7 kg and Japan cars have 5 kg P/M parts (Türk **Toz Metalurjisi Derneği** [TTMD], 2008).

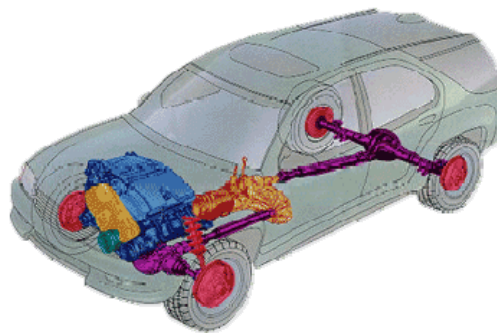


Figure 2.11 Distribution of P/M parts in a car (TTMD, 2008).

High energy ball milling of powder particles as a method for materials synthesis has been developed as an industrial process to successfully produce new alloys and phase mixtures in 1970's. This powder metallurgical process allows the preparation of alloys and composites, which cannot be synthesized conventional routes. A few parameters exists in high energy ball milling which on changing, we can produce a wide range of fine particles with different sizes and consequently with different physical properties. These parameters are (i) Type of mill, (ii) Milling atmosphere, (iii) Intensity of milling, (iv) Ball to powder weight ratio (BPR), (v) Milling time and (vi) Milling temperature (Nanospinel, 2007).

Brittle materials such as inter-metallic compounds, ferro-alloys, aluminium-silicon, aluminum-copper, etc. are crushed mechanically in ball mills. However, milling is not useful for many ductile metals since they do not easily fracture. Instead, the ductile particles cold-weld together and form larger particles. Milling is currently used for production of metallic flakes from ductile metals like aluminium and also MMC composite powder. In this case a lubricant is used for elimination of adhesion and cold welding (TTMD, 2008).

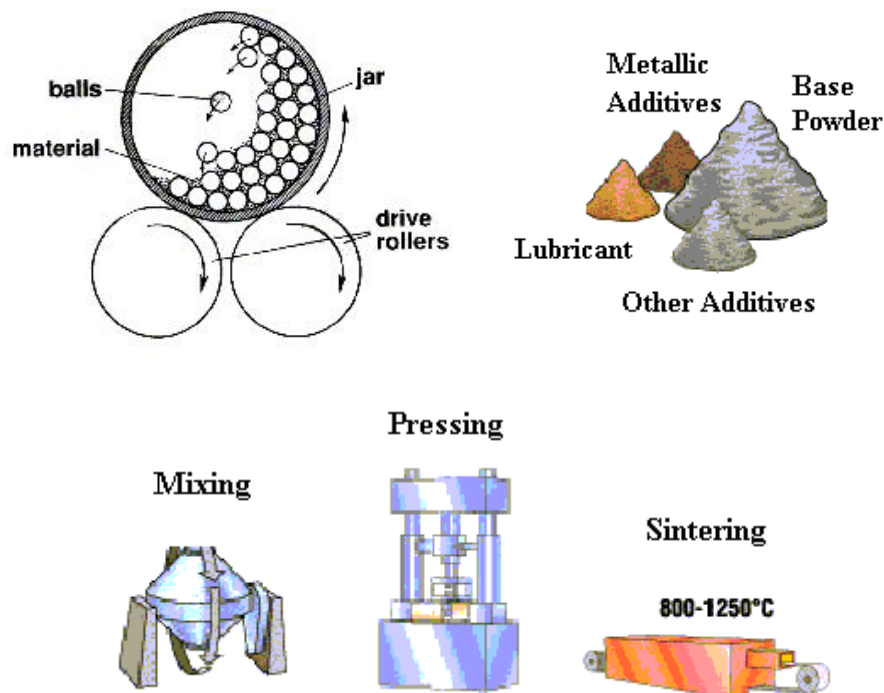


Figure 2.12 Production of Powder Metallurgy Process (TTMD, 2008).

Powder metallurgy (PM) is the most commonly used method in the production of discontinuous reinforced MMC (Delijie, 2002). The chart regarding the application of the method has been given in Figure 2.12. Accordingly, matrix and reinforcement phase powders are mixed and moulded as desired. Then, the mould is pressed for fastening the powders. With the purpose of making the business of powder fastening easier, heat treatment at a sufficient temperature below melting temperature is applied to the pressed powders in order that solid-phase diffusion can occur. Another option is direct application of hot press after mixing the powders (HIP). Copper, nickel, aluminum, cobalt, titanium and molybdenum alloys and steel are commonly used as matrix phase. SiC, graphite, Ni, Ti and Mo are used as particle or short fiber. This is cheaper than the other production methods as there are no melting and casting processes (Torralba, da Costa & Velasco, 2003).

2.2.3 Physical Vapor Deposition (PVD)

PVD constitutes a similar group of processes to CVD, the primary difference being that the vapour is formed by a technique which does not involve a chemical reaction. The vapour can be produced by simple heating (evaporation) or by bombarding a target with high energy ions (sputtering). It is also possible to generate a discharge which causes the deposit to be bombarded as it is formed (ion plating). Using this technique to clean the fibre before deposition of the coating leads to better adhesion. These processes can also be carried out with a reactive atmosphere. For example, deposited various coatings on wires by reactive evaporation and Kieschke and Clyne used reactive sputtering to form on SiC.

The process involves continuous passage of fibre through a region of high partial pressure of the metal to be deposited, where condensation takes place so as to produce a relatively thick coating on the fibre. The vapour is produced by directing a high power electron beam onto the end of a solid bar feed stock. Typical deposition rates are 5–10 μm per minute. Composite fabrication is usually completed by assembling the coated fibres into a bundle or array and consolidating in a hot press

or HIP operation. Composites with uniform distribution of fibre and volume fraction as high as 80% can be produced by this technique (Surappa, 2003).

2.2.4 Stir Casting

This involves incorporation of ceramic particulate into liquid aluminium melt and alloy the mixture to solidify. Here, the crucial thing is to create good wetting between the particulate reinforcement and the liquid aluminium alloy melt. The simplest and most commercially used technique is known as vortex technique or stir-casting technique. The vortex technique involves the introduction of pre-treated ceramic particles into the vortex of molten alloy created by the rotating impeller (Lloyd, 1991). Reports that vortex-mixing technique for the preparation of ceramic particle dispersed aluminium matrix composites was originally developed. Subsequently several aluminium companies further refined and modified the process which are currently employed to manufacture a variety of AMCs on commercial scale. Microstructural inhomogeneties can cause notably particle agglomeration and sedimentation in the melt and subsequently during solidification. Inhomogeneity in reinforcement distribution in these cast composites could also be a problem as a result of interaction between suspended ceramic particles and moving solid-liquid interface during solidification (Surappa, 2003).

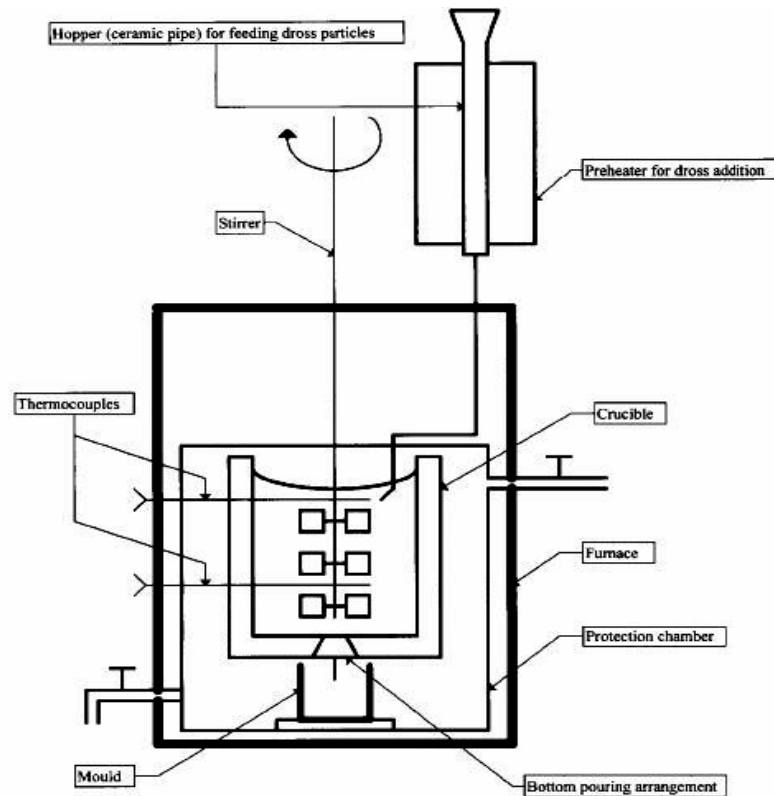


Figure 2.13 Stir Casting process (Kevorkijan, Torkar & Sustarsic, 1999)

Generally it is possible to incorporate upto 30% ceramic particles in the size range 5 to 100 μm in a variety of molten aluminium alloys. The melt–ceramic particle slurry may be transferred directly to a shaped mould prior to complete solidification or it may be allowed to solidify in billet or rod shape so that it can be reheated to the slurry form for further processing by technique such as die casting, and investment casting. The process is not suitable for the incorporation of sub-micron size ceramic particles or whiskers. Another variant of stir casting process is compo-casting. Here, ceramic particles are incorporated into the alloy in the semi solid state. Primary process of composite production whereby the reinforcement ingredient material is incorporated into the molten metal by stirring (Surappa 2003).

2.2.5 Compoasting

Variant of stir-casting in which the metal is semi-solid, i.e. casting process incorporating the reinforcing ingredient material to form the composite. (The semisolid state aids incorporation of the particles, in particular because the metal is more viscous.) Compo casting process is the stir casting process with reinforcement

in semi-solid alloy. Fig. 2.14 shows the schematics of compo casting process. At first, the alloy is melted by heater in semi-solid state. The reinforcements are thrown up to the molten alloy and then mixed by blade. Composite billet is obtained after water quench. Compo casting process is the suitable fabrication technique for the magnesium matrix composites because of low combustibility and oxidation for magnesium alloy.

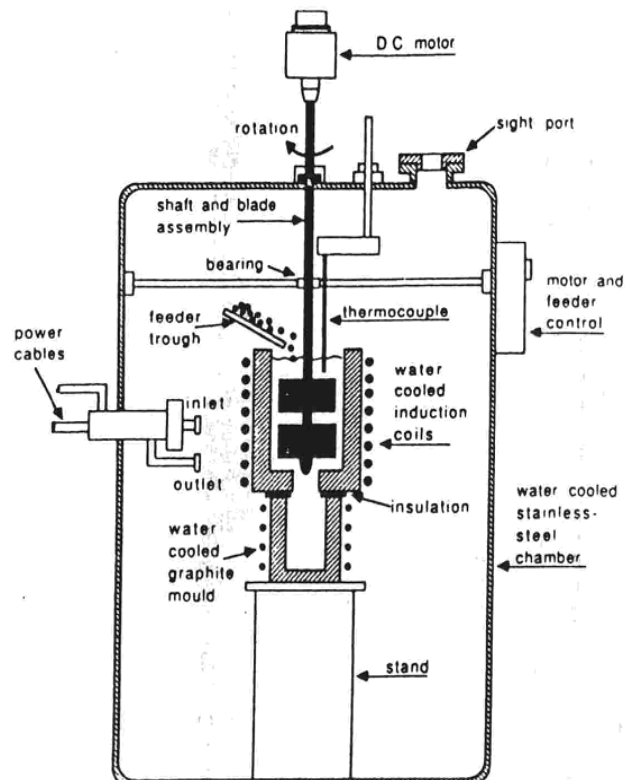


Figure 2.14 Compo-casting process
(Huda, El Baradie, & Hashmi, 1993).

In usual, as the compo casting composites has low volume fraction and the reinforcements in the composites have no bond, the composites can hot-form in order to make the products and improve the mechanical properties such as the hot-extrusion and semi-solid forming. As compo-casting composites can deform plastically, it is easy for preparation of the complex products. But the homogeneity for microstructure affect to the mechanical properties and reliability of the composites, heavily. In actual, the fraction of solid in semi-solid alloy affect to the homogeneity in the reinforcement distribution in the composites (Sasaki, 2003).

Arguably the simplest and most economically attractive method of MMC manufacture is to simply stir mix the liquid metal with solid ceramic particles and then allow the mixture to solidify. The slurry can be continuously agitated while the ceramic is progressively added. This can in principle be done using fairly conventional processing equipment and can be carried out on a continuous or semi-continuous basis. This type of processing is now in commercial use for Al-SiC_p composites and the material produced is suitable for further operations such as pressure die casting, but details of the conditions employed during the stir casting have not been published. Nevertheless, the main attractions and difficulties are fairly clear. The problems may be divided into three main areas - forming difficulties, microstructural inhomogeneities and interfacial chemical reactions (Clyne & Withers, 1995)

2.2.6 Spray Forming

Spray-forming of particulate MMCs involves the use of spray techniques that have been used for some time to produce monolithic alloys. A spray gun is used to atomize a molten aluminum alloy matrix. Ceramic particles, such as silicon carbide, are injected into this stream. Usually, the ceramic particles are preheated to dry them. Fig. 2.15 shows a schematic of this process. An optimum particle size is required for an efficient transfer. Whiskers, for example, are too fine to be transferred. The preform produced in this way is generally quite porous. The co-sprayed metal matrix composite is subjected to scalping, consolidation, and secondary finishing processes, thus, making it a wrought material. The process is totally computer-controlled and quite fast. It also should be noted that the process is essentially a liquid metallurgy process. One avoids the formation of deleterious reaction products because the time of flight is extremely short. Silicon carbide particles of an aspect ratio (length/diameter) between 3 and 4 and volume fractions up to 20% have been incorporated into aluminum alloys. A great advantage of the process is the flexibility that it affords in making different types of composites. For example, one can make in situ laminates using two sprayers or one can have selective

reinforcement. This process, however, is quite expensive, mainly because of the high cost of the capital equipment (Chawla, 1998).

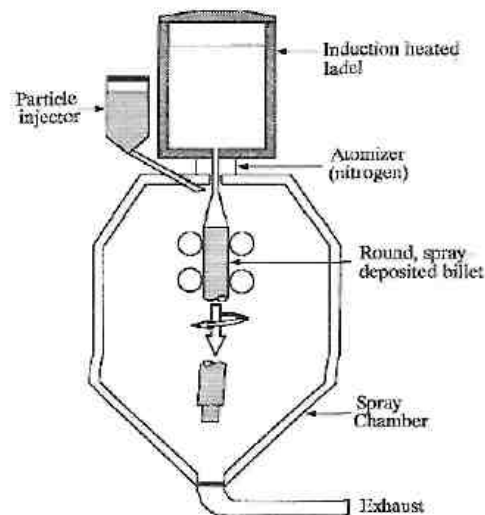


Figure 2.15 Schematic of the spray forming process (Chawla, 1998).

2.2.7 *In-Situ MMC*

In in situ techniques, one forms the reinforcement phase in situ. The composite material is produced in one step from an appropriate starting alloy, thus avoiding the difficulties inherent in combining the separate components as in a typical composite processing. Controlled unidirectional solidification of a eutectic alloy is a classic example of in situ processing. Unidirectional solidification of a eutectic alloy can result in one phase being distributed in the form of fibers or ribbon in the other. One can control the fineness of distribution of the reinforcement phase by simply controlling the solidification rate. The solidification rate in practice, however, is limited to a range of 1-5 cm/h because of the need to maintain a stable growth front. The stable growth front requires a high temperature gradient. The nickel alloy matrix has been etched away to reveal the TaC fibers. At low solidification rates, the TaC fibers are square in cross section, while at higher solidification rates, blades of TaC form. The number of fibers per square centimeter also increased with increasing solidification rate. A precast and homogenized rod of a eutectic composition is

melted, in a vacuum or inert gas atmosphere. The rod is contained in a graphite crucible, which in turn is contained in a quartz tube. Heating is generally done by induction. The coil is moved up the quartz tube at a fixed rate. Thermal gradients can be increased by chilling the crucible just below the induction coil. Electron beam heating is also used, especially when reactive metals such as titanium are involved. MMC in which the ingredient reinforcement material changes its phase or shape during primary composite processing (e.g. TiB₂ reacts to form TiC in some metals by adding carbon). To be an MMC, the constituents must, however, retain its local identity, although they change in shape and/or composition (otherwise, it is an alloy) (Chawla, 1998).

2.2.8 Liquid Metal Infiltration

This method is applied by injecting the liquid metal through the spaces between short fibers set regularly and called “preform” (Pech-Canul, M.I. & Makhoul, 2000). Preforms are generally designed in such a form that they take the shape of the last part after casting. Preforms are produced after the short fibers precipitate out from the liquid suspension. This process is also adapted for producing particle reinforced MMC. In order to protect the integrity and the form of the preforms, generally “fasteners” are used. Various silica and alumina based mixtures are used as high temperature fasteners. Applicability of the method is limited due to the fact that reinforcement phases are unable to be soaked by all melted materials and the fibers are decomposed at high temperatures. Infiltration process can be applied under atmospheric, inert gas or vacuum. Among these, production of MMC under vacuum is the most suitable one (Fig. 2.16), as surface activities of fibers under vacuum are high, wettability is better (Cornie, Chiang, Uhlmann, Mortensen & Collins, 1986).

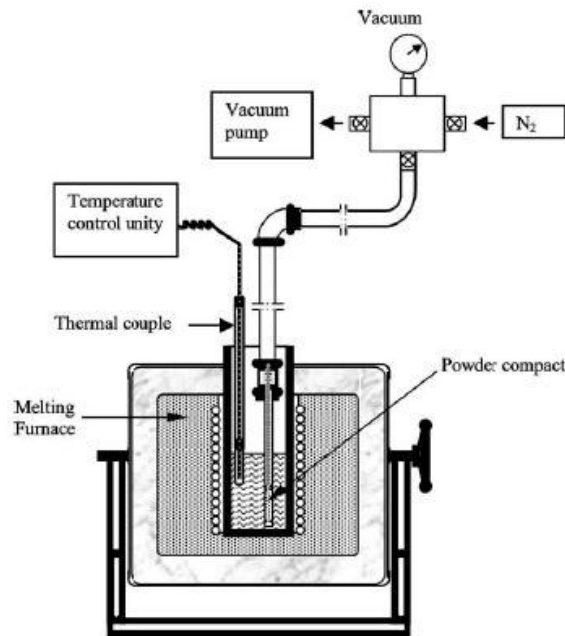


Figure 2.16 Vacuum infiltration contrivance
(Şahin & Acılar, 2003)

A primary process of composite production whereby the molten metal is made to fill, spontaneously or using external mechanical work, pores within a preform of the reinforcement ingredient (Mortensen & et. al., 1997).

2.2.9 Squeeze Casting Process

The term squeeze casting has come to be applied to various processes in which pressure is imposed on a solidifying system, usually via a single hydraulically activated ram. The technique has certain general characteristics, such as a tendency towards fine microstructure (as a result of the rapid cooling induced by good thermal contact with a massive metal mould) and low porosity levels encouraged by efficient liquid feeding. The primary distinction between this and conventional pressure die casting is that the ram continues to move during solidification, deforming the growing dendrite array and compensating for the freezing contraction (which is typically about 5%). In addition to this, the ram movement is usually slower, and the applied pressure often greater, than is typical of die casting. Squeeze casting can be applied to MMC_s as reheated powder mixtures or as stir cast material, but the most common pressure-assisted solidification process for MMC production is properly

termed squeeze infiltration. This involves the injection of liquid metal into the interstices of an assembly of short fibres, usually called a preform (Clyne, Withers, 1995).

Mechanical pressure infiltration process in which a preform is placed into a solid mould, and pressure infiltration is affected with a moving solid piston providing laminar flow of the molten matrix into the mould. Variants include direct squeeze casting, in which the piston surface represents a fraction of the final casting surface; and indirect squeeze casting, in which the piston acts on the ingate to the die (Tekmen, 2006).

Squeeze casting is really an old process, also called liquid metal forging in earlier versions. It was developed to obtain pore-free, fine-grained aluminum alloy components with superior properties than conventional permanent mold casting. In particular, the process has been used in the case of aluminum alloys that are difficult to cast by conventional methods, for example, silicon-free alloys used in diesel engine pistons where high-temperature strength is required. Inserts of nickel-containing cast iron, called Ni-resist, in the upper groove area of pistons have also been produced by the squeeze casting technique to provide wear resistance (Chawla, 1998).

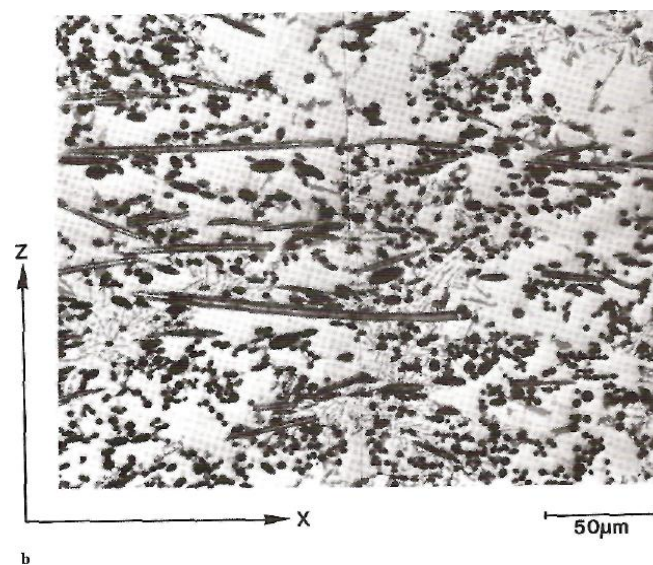


Figure 2.17 The micro-structure of Saffil alumina fiber/alumina matrix composite made by squeeze casting (Chawla, 1998).

Two variants of the process can be identified, depending on whether the preform simply fills a die cavity having a simple shape, such as a cylinder (with composite being subjected to further shaping operations), or is designed with a specific shape to form an integral part of a finished product in the as cast form. (The latter type of selectively reinforced casting is now of considerable industrial significance for applications such as pistons. A number of important process characteristics are common to both variants of process and these are now examined below (Clyne & Withers, 1995).

Squeeze casting has been particularly popular for every fine fibres, such as SiC whiskers, which are difficult to blend with metallic powders because of the size difference and the tendency for tenacious fibre agglomerates to be formed, which also makes stir mixing into melt impractical. Furthermore, many handling procedures lead to fine fibres becoming airborne, creating a risk of inhalation, which is hazardous in the case of whiskers. The preform preparation procedures described above must be carried out within a glove box when whiskers are involved (Clyne & Withers, 1995).

Cast structure obtained is more isotropic and has improved mechanical characteristics. The main advantage of the method is that metal matrix composites acquire fine grained and equal concentric structure without porosities and shrinkage (Kang, Zhang & Cantor, 1993). The sizes of the areas between dendrite and dendrite spaces as well as porosity and micro spaces are reduced by press casting (Chen, Shue, Chang & Lin, 2002).

By the solidification of the alloys under high pressure, in addition to high efficiency and the capacity of perfect final configuration, micro structural advantages such as fine matrix micro structure and better admixture/matrix wettability are also obtained. Additionally, solidification period is very short due to the high pressure, there is a reaction area between reinforcement phase and matrix alloy. Press casting method consists of the following steps as it is shown in Fig. 2.18:

1- Liquid metal as desired is poured into the heated mould. As the mould is exposed to repeated thermal and mechanical loading, damages such as thermal fatigue, cracking and corrosion may occur. Therefore, molding materials which have the characteristics such as high temperature hardness and high temperature resistance must be used. The most commonly used moulding material is H 13 hot work tool steel. (Fig. 2.18 a-b)

2- Pressure is applied by means of a stamper previously adjusted to the mould upper surface distance. (Fig. 2.18-c)

3-Pressure application is maintained until solidification is completed. This process both increases the speed of heat flow and removes the porosities resulting from macro and micro shrinkage. Additionally, gas porosities to be formed from the gases dissolved in the liquid metal are also prevented.

4-Stamper is taken up and the part is removed from the mould.(Fig. 2.18-d) (Tekmen, 2006).

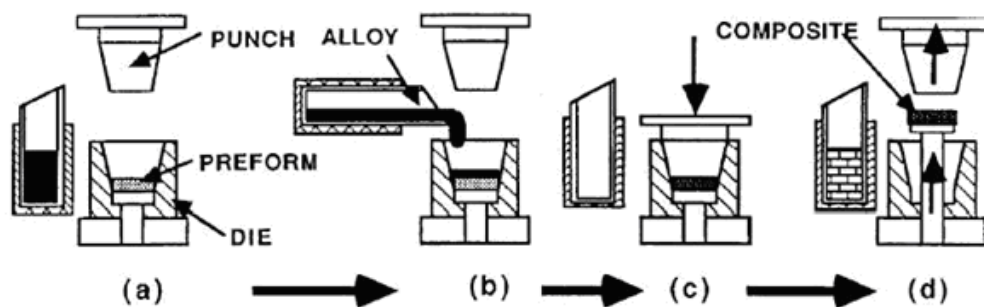


Figure 2.18 Squeeze casting technique (Coupard, Goni & Sylvain, 1999).

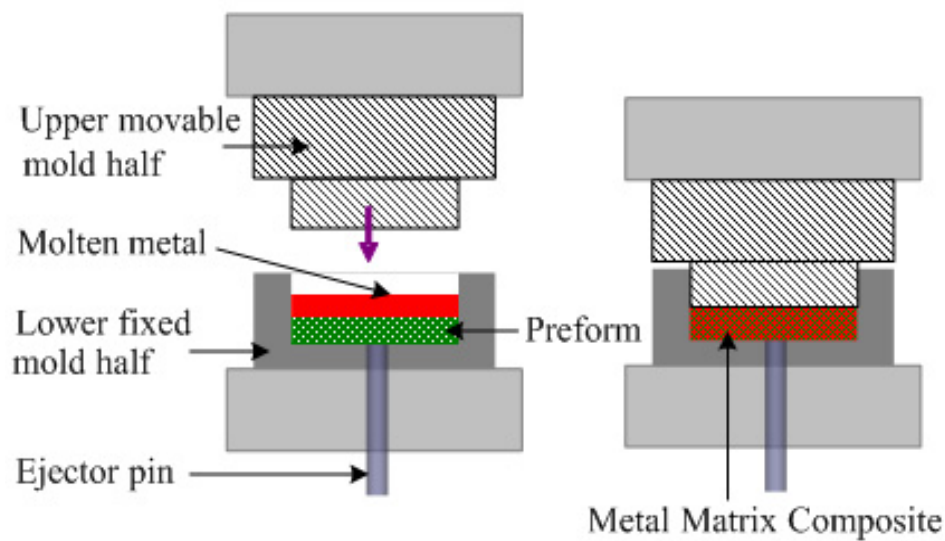


Figure 2.19 Schematic diagram of the squeeze casting apparatus (SubsTech, 2008).

In spite of the fact that press casting method is used within quite a wide production area, keeping process factors under control is very important. These changeable factors are preheat temperature of reinforcement and matrix phases, alloy elements, external cooling, solution quality, moulding temperature, the period passed until the process of pressure application (delay time), applied pressure, waiting time and the speed of pressure application. In the event that these changeable factors are used wrongly, moulding can be deformed, reinforcement phase can be decomposed, oxide inclusion and the other general casting failures may occur. The most disadvantageous side of this method is the size limitation of the part to be produced as high pressure is required for big size parts. Another limitation is the shape of the parts to be cast. However, despite these disadvantages, press method is the only method by which complex parts are produced in the shortest time (Ghomashchi & Vikhrov, 2000; Rajagopal, 1981).

2.2.9.1 The Effect of Production Parameters

It is possible to classify the production parameters which influence the characteristics of MMC produced by press casting method as follows:

- 1) Alloy characteristics
- 2) Casting temperature
- 3) Moulding temperature
- 4) Solidification cooling speed
- 5) The temperature of matrix alloy at the moment of reinforcement phase addition
- 6) The additional speed of reinforcement phase
- 7) Solution volume and quality
- 8) Magnitude and duration of the pressure applied
- 9) Pressure application delay time
- 10) Lubrication

Alloy Characteristics

Alloy characteristic is very important as it has a direct effect on moulding life and casting quality of the physical qualities such as composition of alloy, solution temperature, thermal conductivity, heat transfer coefficient and sticking on the moulding. Therefore, press casting method is generally used in the production of matrix alloyed MMC with low melting temperature such as aluminum and magnesium (Hu, 1998; Ghomashchi & Vikhrov, 2000; Rajagopal, 1981).

Casting Temperature

The temperature in which melted metal is moulded is extremely important in terms of both casting quality and moulding life. When compared to the traditional methods, viscosity of the solution may be lower in press casting method as moulding is filled with the solution as desired by the pressure which is applied.

Therefore, by this method, for the same material, production can be made at a relatively lower temperature. But very low casting temperature causes the viscosity level to be reduced, and therefore, moulding is not filled and cold folding failure occurs. On the other hand, very high casting temperature causes casting mechanism of the liquid metal to extrude the inner part of its interfaces and moulding to be compressed. Additionally, porosities may occur at the thick sections of the moulding

resulting from shrinkage and it may cause moulding life to be reduced. Casting temperature depends on the factors such as liquidity temperature, solidification range and moulding geometry. Casting temperature for aluminum alloys is generally 10 °C and 100 °C higher than liquidity temperature (Hu, 1998; Ghomashchi & Vikhrov, 2000; Rajagopal, 1981).

Moulding Temperature

Moulding and stamper temperature is important parameters as they influence heat transfer speed and accordingly the cooling behavior of the material. Moulding and stamper temperatures must have the capacity of preventing rapid solidification of the metal and must not cause porosities to occur resulting from shrinkage formed as a result of cold folding on the surface of casting, thermal fatigue of moulding and overheating the mould. Moulding temperature for many applications should be between 200 °C and 300 °C (Tekmen, 2006).

Solidification Cooling Speed

Chu and Wu (1999) investigated the effect of the cooling speed in press casting method on the characteristics of Al₂O₃ particle reinforced A356 matrix composite. They observed that when cooling speed was reduced, matrix alloy grain size on the other hand, bending durability and elasticity module were reduced (Tekmen, 2006).

The Temperature of Matrix Alloy at the Moment of Reinforcement Phase Addition

Guyer (1994), in his study, kept the temperature of aluminum alloy at nearly 780 °C before adding SIC particles. He observed that, at high temperatures, especially at a temperature over 820 °C, mixers dissolved into matrix alloys. In the event that the temperature drops below 700 °C at the moment of adding admixture phase, particles were not included into the solution (Tekmen, 2006).

The Additional Speed of Reinforcement Phase

Guyer(1994) observed that when particles were added at a speed higher than between 0.03 and 0.04 kg/min. speed, they got lumpy.

Solution Volume and Weight

When compared to other casting methods, solution quality is more important in press casting method because enteries required for removing the clinkers remained in the mould at the moment of pouring metal are only found in this method. On the other hand, the gas absorbed in the solution in press casting method is less important than the other methods because the pressure applied is so high that it can prevent gas formation and cause them to remain in the solution. As gas spaces can easily be removed by sufficient pressure, there is no need to apply long gas removing processes. Solution quantity is important in the course in which final shaped parts are produced. Final measures can be controlled by measuring casting weight or volume (Rajagopal, 1981).

Magnitude and Duration of The Pressure Applied

Pressure is one of the most important production parameters in press casting method. Since the pressure applied has an effect on heat transfer speed in the solidification temperature and on the interface of casting / moulding and accordingly on solidification behavior of casting, it considerably affects micro structural and mechanical characteristics of composite material. The effect of the pressure applied on solidification temperature of alloy has been given through Claucius-Clapeyron equation;

$$\frac{dT}{dP} = \frac{T_m \Delta V}{H_f}$$

P: Application pressure

T_m: Solidification temprature

ΔV : Solidification during bulk variation

H_f: Reduction hidden thermo

During solidification process, ΔV ve H_f values are negative due to shrinkage and heat dissipation. Thus, dT/dP value is positive and with the increase of the pressure applied, this indicates that solidification temperature has increased. The effect of the pressure applied and moulding temperature on solidification duration has been analyzed by mathematical modeling and the results have been presented in Fig. 2.20. Accordingly, 80 MPa pressure is seen to be sufficient for the speed of solidification and cooling. Moreover, it is observed that by the pressure rise in high moulding temperatures, whole solidification process gains speed (Hu, 1998).

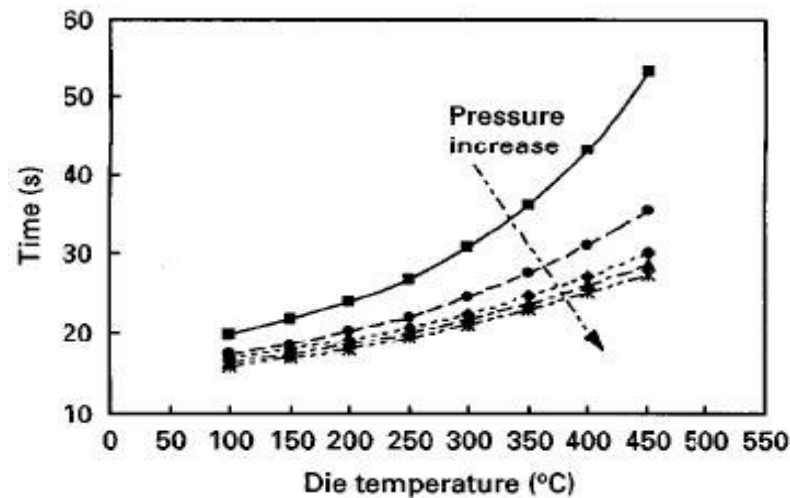


Figure 2.20 The effect of the pressure and mould temperature applied on the solidification duration. (■) 0 MPa, (●) 40 MPa, (◆) 80 MPa, (▲) 120 MPa ve (*) 160 MPa (Hu, 1998).

Furthermore, the effect of the pressure applied on heat transfer during solidification period is also very important. Moulding/casting interface is the most resistant area against heat transfer. Owing to the shrinkage of most metals and thermal expanding of moulding during solidification period, separation of casting from moulding wall is possible if the exterior solid layer formed at the beginning of

casting is powerful enough to retain the remained liquid metal. In conclusion, an air space forms between casting and moulding wall, and this increases heat transfer resistance. Generally, if the pressure is between 70-105 MPa for removing gas and shrinkage porosities, it is sufficient. However, in some cases, pressure is risen to high level due to the geometry of moulding. The pressure to be applied depending on the shape of moulding and section thickness must be in sufficient amount in terms of completing solidification under pressure. If the pressure is applied more than the required minimum duration, it can cause cracking and make it difficult to pull the stamper due to thermal expansion. Maximum pressure application must be 1 sec. for 1mm section thickness (Rajagopal, 1981).

Pressure Application Delay Time

Delay time is a period passed between the moment in which casting is made and the moment in which the stamper starts to apply pressure on the liquid metal. According to the practices carried out, optimum structure in casting is obtained by applying pressure at the zero viscosity temperature of the liquid metal (Rajagopal, 1981). Zero viscosity temperature is the temperature which forms at the moment when solid phase structure begins to form in double phased alloy and metal begins to lose its liquid viscosity characteristic and in general, it is between liquidus and solidus. However, some researches, in order that press casting is effective, defend the idea that pressure must be applied when the metal is completely in the liquid phase (Hu, 1998).

Lubrication

The most commonly used lubricants for aluminum alloys are water-based graphite lubricants. Lubricants are sprayed on the hot moulding and stamper before each casting process. Due to high pressure applied, the effect of thickness of oil layer on the solidification of casting can be neglected. But, owing to the excessive thickness, lubricant may separate from the surface of the mould and can stain the material surface (Hu, 1998).

CHAPTER THREE

WETTABILITY

3.1 Wettability

Archimedes discovered the concept of specific weight hundred years after Aristotle discovered that thin golden sheet or a little stick made from mahogany tree floated on the surface of water. As the specific weight of gold and mahogany tree is bigger than water, conditions on which these solid materials depend would not be the same. The contrast between Archimedes and Aristotle caused a long argument. This contrast ended in 1612 after Galileo discovered that the upper part of the solid material remained under water surface while this flat and thin solid material was floating on water. As Galileo did not know the concept of surface tension and was unable to measure the contact angle between solid material and liquid, his argument was not enough and satisfactory for the others; but his ideas regarding diffusion and wettability were true. The concept that Galileo discovered at that time was “capillary sinking” that we know now. Scientific researches regarding contact angle 200 years after Galileo time have been developed with the equation, the name of which is associated with Young (Good, 1993).



Figure 3.1 Wettability example (Rc-Boat-A-Holic, 2008)

Various mechanisms can assist or impede adhesion. A key concept in this regard is that of wettability. Wettability tells us about the ability of a liquid to spread on a solid surface. We can measure the wettability of a given solid by a liquid by considering the equilibrium of forces in a system consisting of a drop of liquid resting on a plane solid surface in the appropriate atmosphere. Fig. 3.2 and Fig. 3.3 shows the situation schematically.

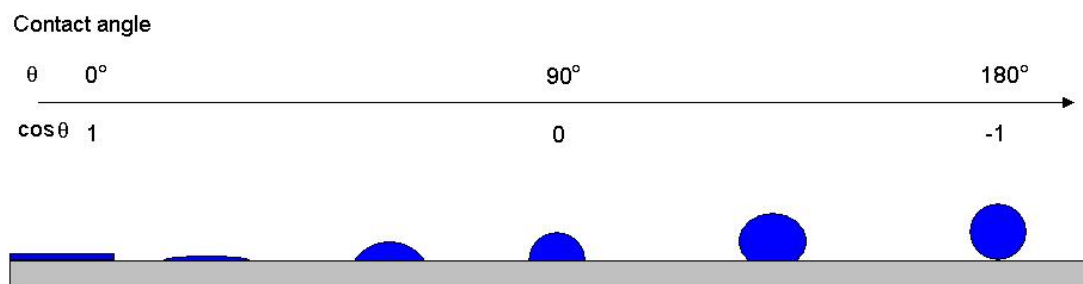


Figure 3.2 Symbolic picture displaying wettability angles (Kvs Instruments, 2008).

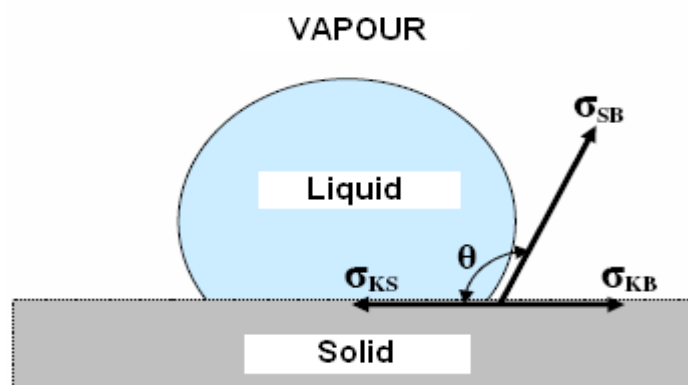


Figure 3.3 The surface energies of solid/vapour, liquid/solid, and liquid/vapour interfaces, respectively (Tekmen, 2006).

The liquid drop will spread and wet the surface completely only if this results in a net reduction of the system free energy. Note that a portion of the solid/vapor interface is substituted by the solid/liquid interface. Contact angle, θ of a liquid on the solid surface fiber is a convenient and important parameter to characterize wettability. Commonly, the contact angle is measured by putting a sessile drop of the

liquid on the flat surface of a solid substrate. The contact angle is obtained from the tangents along three interfaces: solid/liquid, liquid/vapor, and solid/vapor. The contact angle, θ can be measured directly by a goniometer or calculated by using simple trigonometric relationships involving drop dimensions. In theory, one can use the following expression, called Young's equation,

$$\sigma_{KB} = \sigma_{KS} + \sigma_{SB} \cdot \cos \theta + \pi_e$$

Where π_e is the specific surface energy, and the subscripts σ_{KB} , σ_{KS} , and σ_{SB} represent solid/vapor, liquid/solid, and liquid/vapor interfaces, respectively. If this process of substitution of the solid/vapor interface involves an increase in the free energy of the system, then complete spontaneous wetting will not result. Under such conditions, the liquid will spread until a balance of forces acting on the surface is attained; that is, we shall have partial wetting. A small θ implies good wetting. The extreme cases being $\theta = 0^\circ$, corresponding to perfect wetting, and $\theta = 180^\circ$, corresponding to no wetting. In practice, it is rarely possible to obtain a unique equilibrium value of θ . Also, there exists a range of contact angles between the maximum or advancing angle, θ , and the minimum or receding angle, θ . This phenomenon, called the contact-angle hysteresis, is generally observed in polymeric systems. Among the sources of this hysteresis are: chemical attack, dissolution, inhomogeneity of chemical composition of solid surface, surface roughness, and local adsorption (Tekmen, 2006).

It is important to realize that wettability and bonding are not synonymous terms. Wettability describes the extent of intimate contact between a liquid and a solid; it does not necessarily mean a strong bond at the interface. One can have excellent wettability and a weak van der Waals-type low-energy bond. A low contact angle, meaning good wettability, is a necessary but not sufficient condition for strong bonding.. What is normally neglected in such an analysis is that there is also a vertical force $\sin \theta$, which must be balanced by a stress in the solid acting perpendicular to the interface. This was first pointed out by Bikerman and Zisman in their discussion of the proof of Young's equation by Johnson. In general, Young's equation has been applied to void formation in solids without regard to the precise

state of internal stress. Analyzed the conditions for occurrence of these internal stresses and their effect on determining work of adhesion in particle reinforced composites (Tekmen, 2006).

Wettability is very important in PMCs because in the PMC manufacturing the liquid matrix must penetrate and wet fiber tows. Among polymeric resins that are commonly used as matrix materials, thermoset resins have a viscosity in the 1-10 Pa s range. The melt viscosities of thermoplastics are two to three orders of magnitude higher than those of thermosets and they show, comparatively, poorer fiber wetting characteristics and poorer composites. Although the contact angle is a measure of wettability, the reader should realize that its magnitude will depend on the following important variables: time and temperature of contact; interfacial reactions; stoichiometry, surface roughness and geometry; heat of formation; and electronic configuration (Chawla, 1998).

3.2 The Importance of Wettability in Al-SiC MMC

Since production of metal matrix composites by casting, cheapness of the method, and provides the opportunity to change the characteristics with various production parameters, it is commonly used. As is known very well, reinforcement phase and interface characteristics between matrix alloys have an important effect upon the mechanical behavior of MMC.

The fact that MMC have high elasticity module and durability as a result of transfer and distribution of external forces applied to reinforcement phase by the matrix, a strong interface between reinforcement phase and matrix is very important in this sense. (Hashim, Looney & Hashmi, 2001).

From the point of metallurgy, the fact that reinforcement phase are sufficiently soaked by the matrix alloy, low rate and speed chemical reactions in-between the interface, the fact that there is little or no diffusion between the phases; and therefore, reinforcement phase is very important in order that reinforcement phase is not

decomposed (Hashim & et. al., 2001). One of the biggest problems in the process of production of aluminum-based SiC reinforced composites is that SiC reinforcement phase reacts with liquid aluminum at high temperatures and that wettability of SiC reinforcement phase at low temperatures is not sufficient. As a result of the reaction of SiC reinforcement phase with aluminum, Al_4C_3 reaction product forms which influences the composite characteristics in matrix/reinforcement phase interface negatively (Fig. 3.4) (Urena, Escalera, Rodrigo, Baldonado & Gil, 2001).

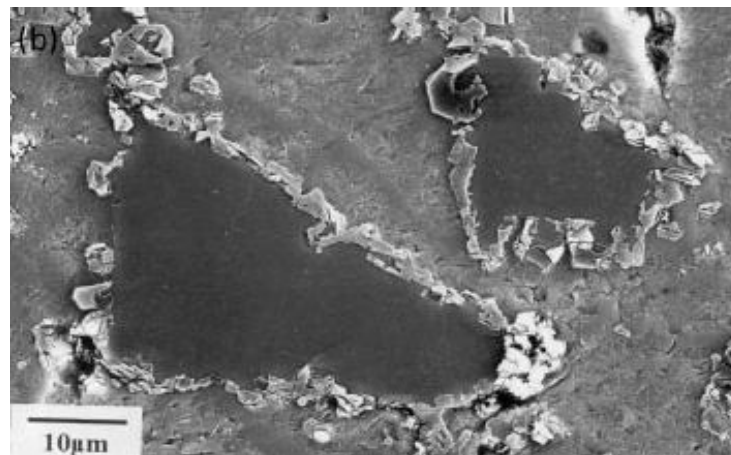


Figure 3.4 Al_4C_3 formation at the interface of matrix/reinforcement phase after heat treatment for 1 hour at 900 °C (Urena & et. al., 2001).

After the reaction experiment which has lasted for 20 minutes at 900 °C in unprocessed SiC particle reinforced composites, less wettability between SiC and Al has been observed (Urena & et. al., 2001).

Ionic and covalent solid materials like SiC are characterized by stable electronic structure of atoms and interatomic powerful bond. Interaction of such materials with liquid metal is likely to occur only by partial or complete separation of interatomic bonds and reacting with solid phase (Kennedy & Karantzalis, 1999).

The biggest disadvantage of metal matrix composites is that they have low thickness values. Less thick interface area than matrix alloy causes crack to be developed. Low thickness of the interface is resulting from the covalent bond

structure in the interface. Therefore, using proper coating material which has metallic bond structure is very important for high thickness (Rajan, Pillai, & Pai, 1998).

3.3 The Factors Which Influences Wettability in Al-SiC Composites

Oxide layer to be formed on the surface of the solution reduces the wettability of reinforcement phases by melted alloy. This oxide layer, especially, prevents reinforcement particles added from the top from being mixed up in the alloys. Due to high oxygen affinity of aluminium (50 mm thick oxide layer forms as a result of holding for 4 hours at 400 °C), it is nearly impossible to prevent oxide formation in aluminum-based systems. According to the studies of Zhou and Xu (1997), the main reason of weak wettability in Al/SiC composites is a thin gas layer on the surface of ceramic particle. They expressed that the gas layer prevented liquid aluminum from making a contact with SiC particles and when particle concentration in the solution exceeds a certain critical value, the gas layer has a function like a bridge and causes all the particles to be removed out of the solution (Tekmen, 2006).

Another element which affects wettability is particle size. The smaller the size of the particle is the less wettability becomes. This result from the increase of surface energy required for the liquid metal surface to spread on small radius reinforcement phase. Moreover, since small size particles have high surface areas, adding to matrix alloy is difficult (Hashim & et. al., 2001).

3.4 Methods Which Increase Wettability in Al-SiC Composites

The main principle of increasing wettability is to increase surface energy of solid material, to reduce surface tension of liquid alloy or to reduce interface energy between reinforcement phase and matrix. In order to do this, alloy element must be added, ceramic reinforcement phase must be coated or heat treatment must be applied (Tekmen, 2006).

3.4.1 Adding Alloy Element

The interface between pure metals (M) and oxides (MO) is evaluated in two different categories depending on the (ΔG°) sign of standard Gibbs free energy of the reaction between the two phases. According to the experimental studies with Al-SiC composites, adding magnesium, lithium, silisium, calsium, titanium or zirconium in matrix alloy reduces surface tension of melted metal, interface energy of solid-liquid material; and therefore, has increased wettability (Saiz, Cannon & Tomsia, 2001, Candan 2002, Candan 2006).

Magnesium in aluminum-based composites, adding reinforcement phase to matrix alloy is known that they are more effective and have improved the characteristics in distribution of reinforcement phases when compared to other elements (Kobashi & Choh, 1993).

To sum up the effect of magnesium in Al-Si alloys:

- 1) Magnesium as an effective oxygen remover reacts with the oxygen on the surface of SiC, and causes the gas layer on the surface to become thinner and thus, increases wettability and prevents particles from getting lumpy.
- 2) Magnesium reduces aluminum oxide layer in liquid aluminum and on admixture phase/matrix interface and increases wettability. Moreover, it increases wettability of MgAl_2O_4 spinel formed at the interface.
- 3) As magnesium is a very reactive element, it creates thermodynamically stable oxides and causes wettability to increase (Tekmen, 2006).

3.4.2 Heat Treatment of Reinforcement Phase

Before adding ceramic reinforcement phase to matrix alloy, cleaning the surface by removing the gases absorbed on the surfaces of heat treatment and reinforcement phase is very important with regard to wettability. By pre-heating process to be

applied to SiC particles at 900 °C, surface impurities are removed, gases are desorbed and SiO₂ layer is formed on the surface (Fig. 3.5).

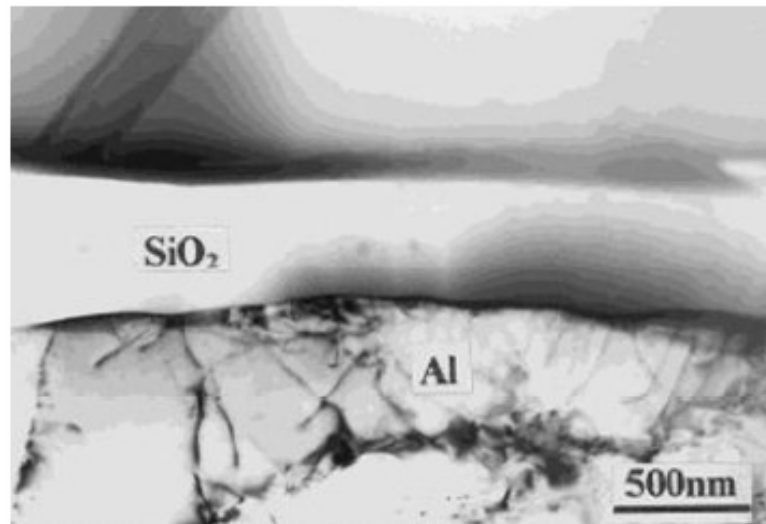


Figure 3.5 TEM image displaying SiO₂ layer at the interface of Al/SiC. (Gu, Jin, Mei, Wu & Wu, 1998).

SiO₂ has the function of protective layer between liquid aluminum and SiC and forms Al₄C₃ and thus, prevents SiC from decomposing. However, SiO₂ coating, as a result of the chemical reactions occurred at the interface of matrix/reinforcement phase, reduces the interface energy of solid-liquid material and increases wettability and therefore, it has been applied by many researchers (Urena & et. al., 2001).

In addition to this, cleaner surface of reinforcement phase provides better interaction between reinforcement phase and matrix alloy and increases wettability. Ultrasonic cleaning, various etching techniques and heating treatments under suitable atmosphere are the techniques which are used for surface cleaning (Warren & Andersson 1984). Another method for coating SiC particles with SiO₂ is sol-jel technique (Rams, Campo & Urena, 2004). It has been observed that in SiC particle reinforced aluminum-based composites coated with SiO₂ by sol-jel technique, SiO₂ coating increased wettability at 725 °C played the role of protective layer between aluminum and SiC reinforcement phase. The biggest advantage of SiO₂ coating by this technique is that porosities and surface structure and therefore interface reactions

can be controlled by heat treatment to be applied to sol-jel coating (Rams & et. al., 2004).

3.4.3 Coating Reinforcement Phase

One of the most effective methods of preventing interface reactions and of increasing wettability of reinforcement phase is coating reinforcement phase. Coating layer also prevents liquid metal from diffusing reinforcement phase. Another quality which is expected from coating is to form a strong bond between reinforcement phase and matrix. However, formation of brittle phases at the interface which affects the mechanical characteristics negatively is an undesirable situation. In the process of coating reinforcement phase, chemical vapor deposition (CVD), physical vapor deposition (PVD), electrolytic coating with and without current, plasma spray and sol-jel techniques are used (Chou, Kelly & Okura, 1985).

CHAPTER FOUR

INTERFACE

In composites the role of the interface is crucial. Stiffening and strengthening rely on load transfer across the interface, toughness is influenced by crack deflection/fibre tensile-out, and ductility is affected by relaxation of peak stresses near the interface. Unfortunately, however, a great deal of confusion surrounds the question of how best to characterise, and then optimise, the mechanical response of the interface to stresses arising from an applied load. In this chapter, a brief outline is given of the meaning and significance of interfacial bond strength, followed by a summary of the methods used to measure interfacial mechanical properties, with particular reference to fibrous MMCs. Some attention is then devoted to interfacial chemical reactions. Finally, the production and characteristics of fibre coatings are briefly examined (Tekmen, 2006).

In the previous two chapters it has become clear that many important phenomena can take place at the matrix/reinforcement interface. For polymer-based composites, although the chemistry involved may be complex, the objectives in terms of interfacial properties are often the rather straight forward ones of a high bond strength (to transfer load efficiently to the fibres) and a good resistance to environmental attack. In designing ceramic composites, on the other hand, the aim is usually to make the interface very weak, as the prime concern is in promoting energy dissipation at the interface so as to raise the toughness. For MMCs, a strong bond is usually desirable, but there may be instances where inelastic processes at the interface can be beneficial. Furthermore, the avoidance of environmental attack is often less important than the need for control over chemical reactions between the constituents themselves, occurring during fabrication or under service conditions at high temperature (Chawla, 1998).

It's reported that the problem of how to characterize the mechanical performance of the interface has been approached in two different ways. The most common

approach has been to establish critical stress levels at which inelastic processes initiate there. This is complicated by the fact that various combinations of normal and shear stresses might trigger the same process. Furthermore, a critical interfacial stress value does not always lead directly to useful predictions about the mechanical performance of the composite. The alternative approach, which has received considerable attention recently, involves measuring an interfacial fracture toughness. This may require careful experimental procedures, but it offers promise for establishing correlations with composite performance. Before examining this in detail, it is instructive to consider the nature of the interfacial stresses and the interfacial processes they bring about (Tekmen, 2006).

4.1 Composite Performance

Relatively few quantitative correlations have been established for MMCs between interfacial characteristics and composite performance indicators. However, poor interfacial bonding has been shown to result in a reduced elastic modulus and work-hardening rate and this is readily explained in terms of reduced load transfer. Examples of the progressive impairment of load transfer, and associated reductions in elastic modulus, have also been detected as a consequence of prior plastic strain and of progressive interfacial reaction.

A technique giving information about inelastic processes, which can be particularly useful for long fibre MMCs, is to monitor the changing Poisson's ratios during loading, via strain gauge measurements. The three Poisson's ratios exhibited by a uniaxial composite can be predicted analytically for elastic loading. Referring to Ti-SiC laminae. For the as-fabricated composite under axial loading, (a), ν_{32} is close to the elastic value (~ 0.30) up to a strain of about 0.5%, above which it increases. This corresponds the onset of plastic deformation in the matrix, which will raise the apparent Poisson's ratio. Under transverse loading, (b), a value of ν_{23} close to that for elastic deformation (~ 0.23) is again observed initially, but inelastic behaviour sets in at a low strain ($\sim 0.1\%$) and (c) ν_{23} falls sharply beyond this point. This is due to the onset of interfacial damage, since the debonding and opening up of

interfaces generates longitudinal extension without the corresponding lateral contraction (Clyne & Withers, 1995).

Substantial heat treatment (giving $\sim 5\text{-}6\ \mu\text{m}$ interfacial reaction layer) changes the behaviour. The axially loaded plot, is similar to that of but with the expected reduction in ductility and very limited plastic flow in the matrix. The transversely loaded plot, (d), however, differs significantly from that for the as-fabricated specimen, having substantially higher values of v_{23} in both regimes. In the elastic portion, this may also reflect the elastic properties of the reaction zone, but for the inelastic regime the change is presumably due to the reduced capacity of the interface to open up without causing failure of the specimen.

Clear correlations between interfacial parameters and composite strength or toughness properties are more difficult to establish. However, some simple analyses have been developed. One approach is to assume that an interfacial region, being composed of brittle constituents, will contain a defect equal in size to the thickness of the region. This has been used to explain the deterioration in axial tensile strength of long fibre composites usually observed on increasing the thickness of an interfacial zone (Clyne & Withers, 1995).

4.2 Importance of Reinforcement Phase/Matrix Interface

The effect of reaction products (Fig. 4.1) formed as a result of interface nature and the reactions realized on the characteristics of metal matrix composites is great. Increasing durability of composites by reinforcement phase depends on durability of interface bond between reinforcement phase and matrix. Composite characteristics such as rigidity, reinforcement thickness, fatigue durability, creeping, thermal expansion coefficient and thermal conductivity depend on interface characteristics. Chemical bond forms when reinforcement phase and matrix interact and exchange electrons. Such chemical bond can be metallic, ionic or covalent. The interface with metallic bond is more ductile than the other bond types and preferred in metal matrix composites (Cayron, 2000).

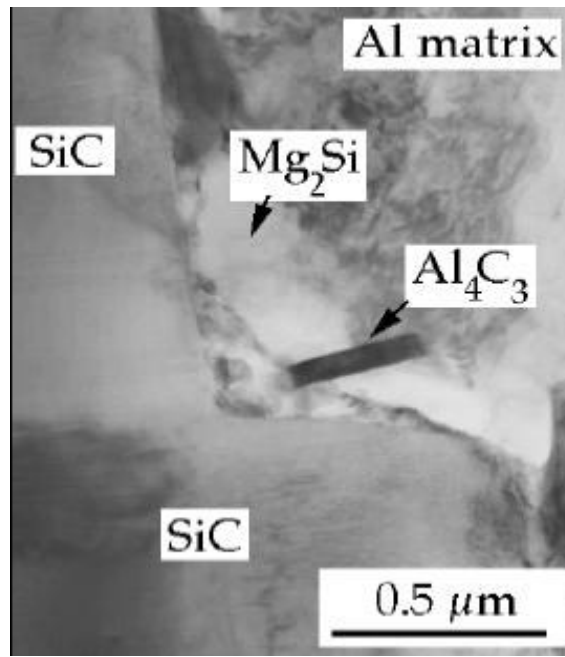


Figure 4.1 Reaction products formed at the interface of Al/SiC (Cayron, 2000).

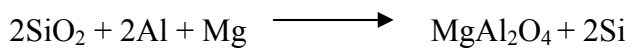
Some chemical reactions form at the interface of matrix/reinforcement phase in the process of production of metal matrix composite. In such cases, interface reaction products with more different characteristics than reinforcement phase and matrix form. Type of the formed chemical products and the power of the chemical reaction depend on process temperature, pressure, chemical compound of matrix alloy and the surface chemistry of reinforcement phase. The interface between matrix and reinforcement phase is a critical area which is affected in the process of production. If interface characteristics are not kept under control, they may affect composite characteristics negatively. Interface chemical reactions, decomposition of reinforcement phase and reduction of wettability are the primary problems which may emerge related to the interface (Tekmen, 2006).

4.3 Oxidation of Reinforcement Phase

Due to the fact that reinforcement phase is being used in high temperature applications for different purposes, a good deal of researches have been made regarding oxidation of this material (Suery & Salvo, 1995). Perfect oxidation resistance of SiC, SiO₂ coherent layer formed on its surface and this layer prevents

further oxidation. SiO_2 layer formed on SiC surface has two functions in Aluminum-based MMC.

One of the functions is to prevent SiC from interacting with aluminum and (ii) the other function is to increase the wettability of SiC by aluminium (Gu & et. al., 1998). reaction product is obtained as a result of the reaction of SiC particles with Mg which is indicated below.



As it is seen in Fig. 4.2 size of MgAl_2O_4 crystals is approximately 100 nm and it does not cause a permanent layer at the interface of reinforcement phase/matrix . The results obtained also have indicated that MgAl_2O_4 formation, although reaction of SiO_2 with liquid aluminum develops very fast, has developed quite slowly (Gu & et. al., 1998).

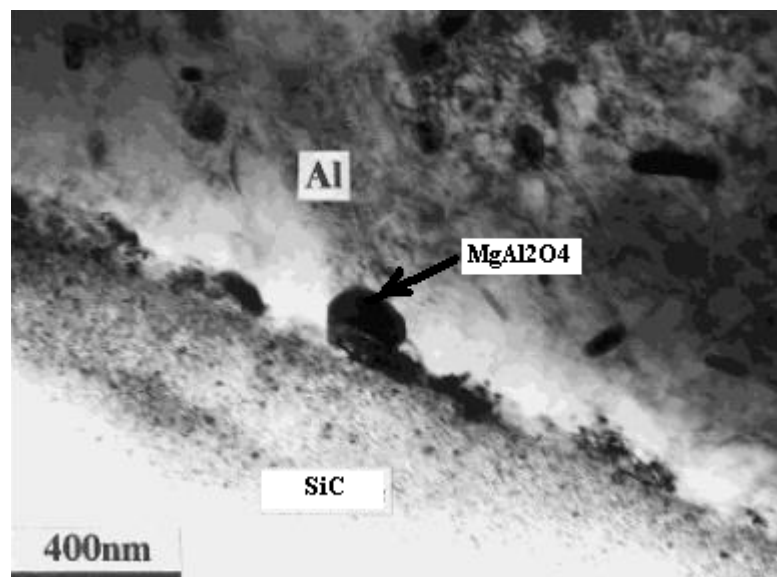


Figure 4.2 TEM image displaying MgAl_2O_4 crystals formed at the interface of Al/SiC (Gu & et. al., 1998).

Magnesium is an active element in aluminum alloys and is diffused into Al/SiC interface with the purpose of forming particle. If Mg amount in matrix alloy is over

4%, an intensive MgO layer will form on the surface of SiC (Shi, Ochiai, Gu, Lee & Wu, 2001).

If there is enough Mg in matrix alloy, MgAl_2O_4 and Si cores are nucleated on SiO_2 and this layer enlarges until SiO_2 finishes and in the course of time, SiO_2 layer completely turns into MgAl_2O_4 layer. MgAl_2O_4 layer formed plays the role of a protective layer between Aluminum and SiC and prevents Al_4C_3 formation (Lee, Ahn, Shim, Shi, & Lee, 1999).

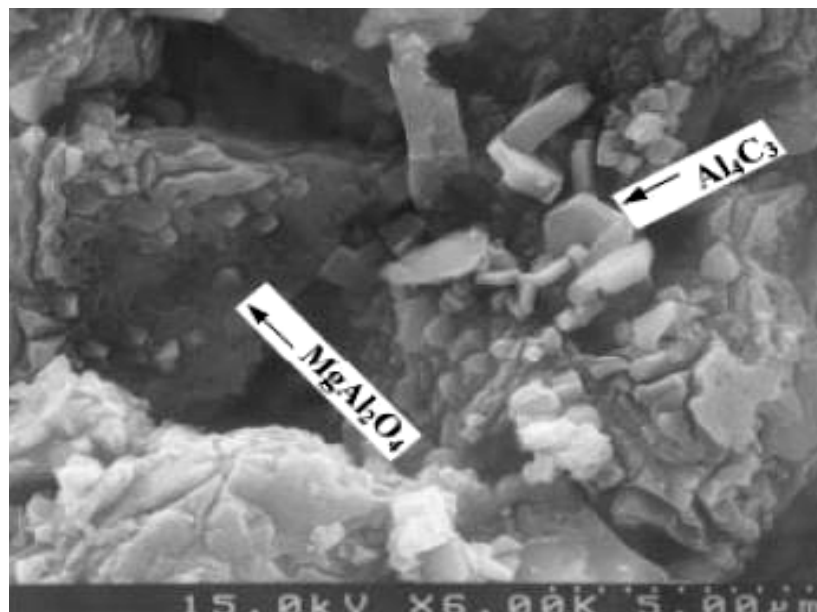


Figure 4.3 SEM image belonging to SiC particles extracted from composite material after heat treatment for 1 hour at 800 °C (Shi & et. al., 2001).

CHAPTER FIVE

EXPERIMENTAL WORKING

5.1 Material

In this study, the materials used in this work and their chemical compositions are listed in Table 5.1. The alloy EN AW-2017 (3.9Cu, 0.6Mn, 0.7Mg, bal. Al, wt.%), and the composites reinforced with 5 and 15 vol.% particulate silicon carbide (SiC_p) produced by the squeeze casting technique.

Table 5.1 Chemical compound of matrix alloy used in the process of MMC production.

Chemical Composition (wt %)				
Matrix Alloy	Mg	Cu	Mn	Al
	0.7	3.9	0.6	Bal.



Figure 5.1 High energy ball milling unit (Nims, 2008).

5.2 Production of Metal Matrix Composites

Before applying squeeze cast, gas atomized Al-Cu matrix powder and SiC particles with 5 and 15 vol. % content were blended for 15 minutes by using mixer. Some part of blended composite powder was also ball milled shortly (up to 2h) by using lab type of high energy ball milling unit (Fig. 5.1). MMCs have been produced

by press casting method. For composite production melting furnace system specially designed for previous (Tekmen, 2006) and this study indicated in Figure 5.2.

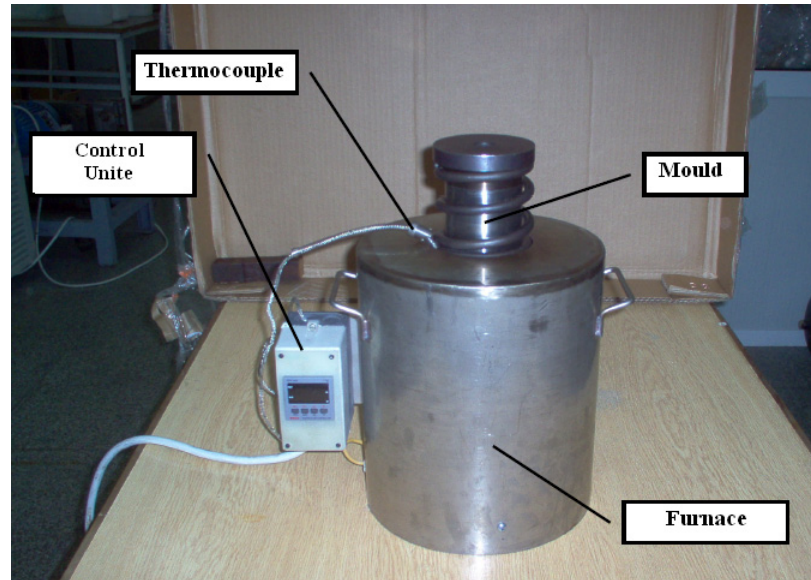


Figure 5.2 Our furnace designed for melting our aluminum matrix material.

The matrix alloy and the composite samples were prepared in an furnace under an argon gas protective atmosphere by melt stirring technique. For the right and desired alloy, as it is seen in Figure 5.4, the mould specially designed for squeeze cast experiment is made of H13 hot work tool steel and after it is processed, it has been solidified by heat treatment in ISTAS firm. (50 HRC). Moulding and stamper has been lubricated with a special graphite-based lubricant. After lubrication process is over, our moulding is placed in our specially designed furnace, and we keep on heating until moulding temperature rises to 400 °C (Fig. 5.6). The argon atmosphere was maintained over the melt to reduce the oxidation. After inner temperature of our moulding comes closer to 400 °C, we pour aluminum matrix material in the mould carefully, we keep on heating until inner temperature of the mould becomes 700-750 °C. As soon as we observe that temperature of the mould becomes 750 °C by thermocouple, temperature of the semi-solid matrix alloy was measured as 600 °C. Fig. 5.5 shows we place our stamper in the mould (approximately with 5 sec. delay time) by means of a 100 ton capacity hydraulic press (Dirinler Machine) and casting is solidified under 100 MPa pressure. The effect of pressure on the properties of composites was also studied by increasing pressure from 100 MPa to 120 MPa.

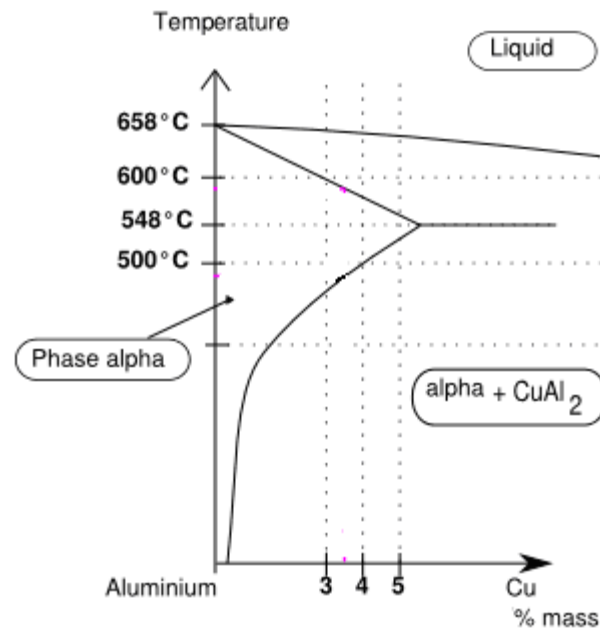


Figure 5.3 Al-Cu Phase diagram (Wikimedia, 2008).

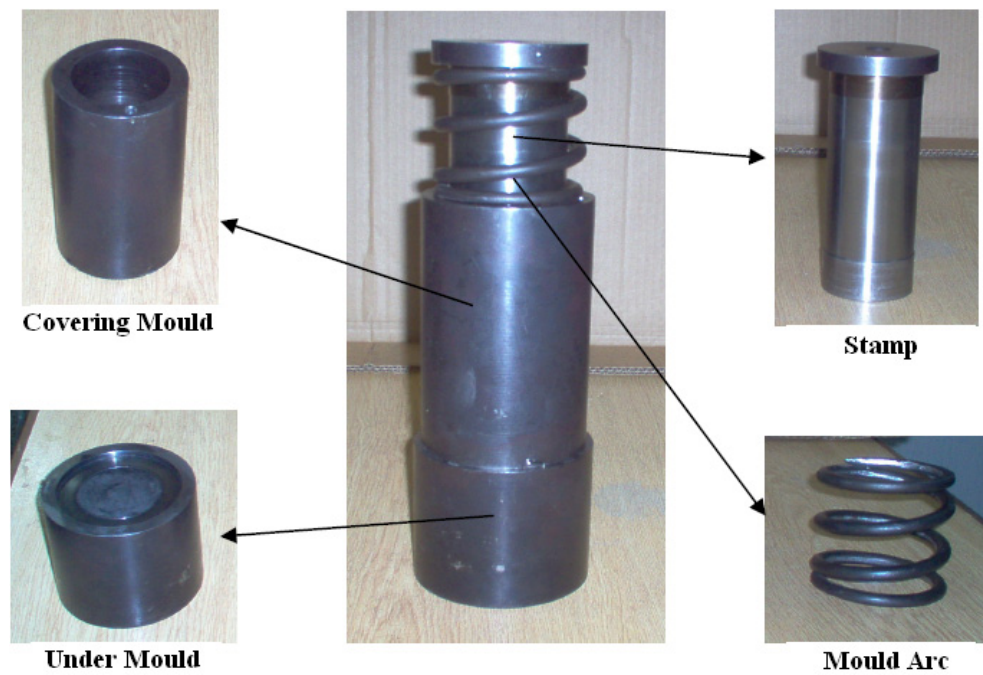


Figure 5.4 Parts of the mould used in squeeze casting.



Figure 5.5 Squeeze casting mechanism and hydraulic press.

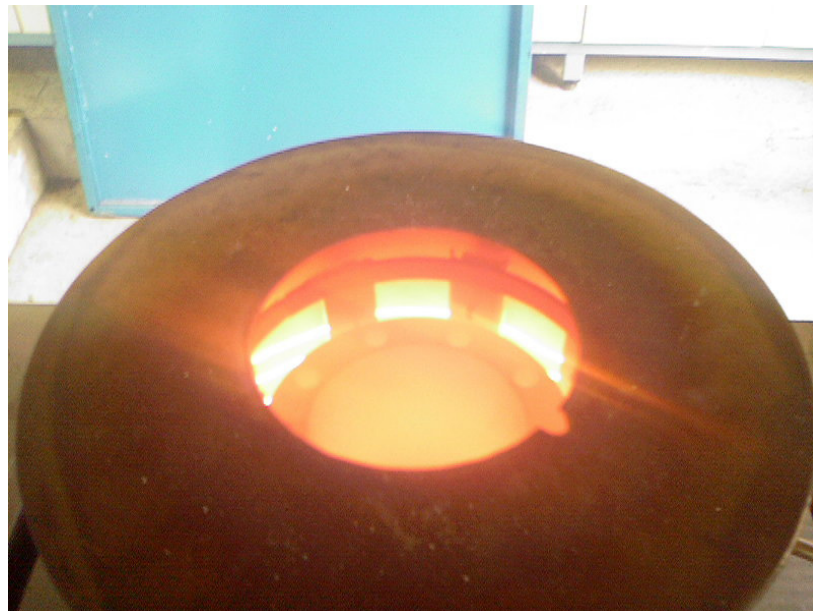


Figure 5.6 Heating moment of the material and mould.

5.3 Tensile Tests

The composites produced by squeeze casting were cut to prepare samples for mechanical tests and metallographic examinations. Four samples of each cast were prepared further by using diamond blades in CNC machine for tensile testing. Tensile test samples, have 8.0 mm diameter with a gauge length of 51.2 mm, were prepared on a computerized AG-50kNG Shimadzu universal testing machine. Tensile tests were carried out to evaluate the mechanical properties of the produced composites, and to find out the effects of the reinforcement content, and squeeze casting process on the strength and ductility.

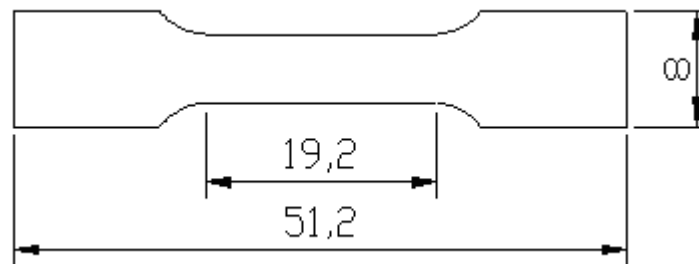


Figure 5.7 The view and the dimension of tensile sample prepared from the composite material produced. (Thickness: 3 mm)

5.4 Aging Behaviour

An aging study was conducted on specimens taken from the matrix alloy and composites in order to determine the time required to attain peak hardness. The aging process essentially involves a solution treatment of the sample for 2 h at 525 °C then quenching in cold water followed by aging at two different conditions. These conditions were: (1) aging at room temperature (natural aging T4 heat treatment); (2) artificial aging at 160 °C up to 24 h; All specimens were immediately refrigerated until testing to prevent room-temperature aging.

5.5 Microstructural Examination

The specimens of all the materials were prepared for metallographic examinations using 240–400–600–800–1000–1200–2400 mesh emery papers, followed by polishing techniques. Microscopic methods were used to study the composite structure, particle distribution and porosity. Microstructure and fracture surfaces (after tensile tests) were observed by means of optical and scanning electron microscopy (SEM). For the examination of the elements and phases in the composite microstructures, Optical microscope (Nikon ECLIPSE ME600D), energy dispersive spectroscopy (EDS) (JSM–6060 JEOL) and a scanning electron microscope with microprobe unit (SEM-microprobe) (JSM–6060 JEOL) were used.

The porosity content of the samples was evaluated from the difference between the calculated and experimentally measured density of each sample by using the Archimedean principle. The calculated densities of the samples were determined from the chemical analysis. The porosity is calculated as where ρ_T is the theoretical density measured from rule of mixture, and ρ_E is the density measured by water displacement method.

$$\text{Porosity (\%)} = \frac{\rho_T - \rho_E}{\rho_T} \times 100$$

Hardness measurement was carried out using a Vickers hardness tester. Before testing, specimen surfaces were polished using emery papers down to 2400 mesh. At least 5 measurements were taken for each sample and the average was taken as the hardness value. Micro hardness measurements were carried out by using a HSV-30 vickers hardness tester under 80 gr load.

CHAPTER SIX

EXPERIMENTAL RESULT

6.1 Material

The matrix Al alloy powder used for composite production was shown in Fig 6.1a. As seen, the matrix powder produced by gas atomisation is spherical in shape. The matrix cross section after etching can be seen in Fig 6.1b. The reinforcement particles was shown in Fig. 6.1c.

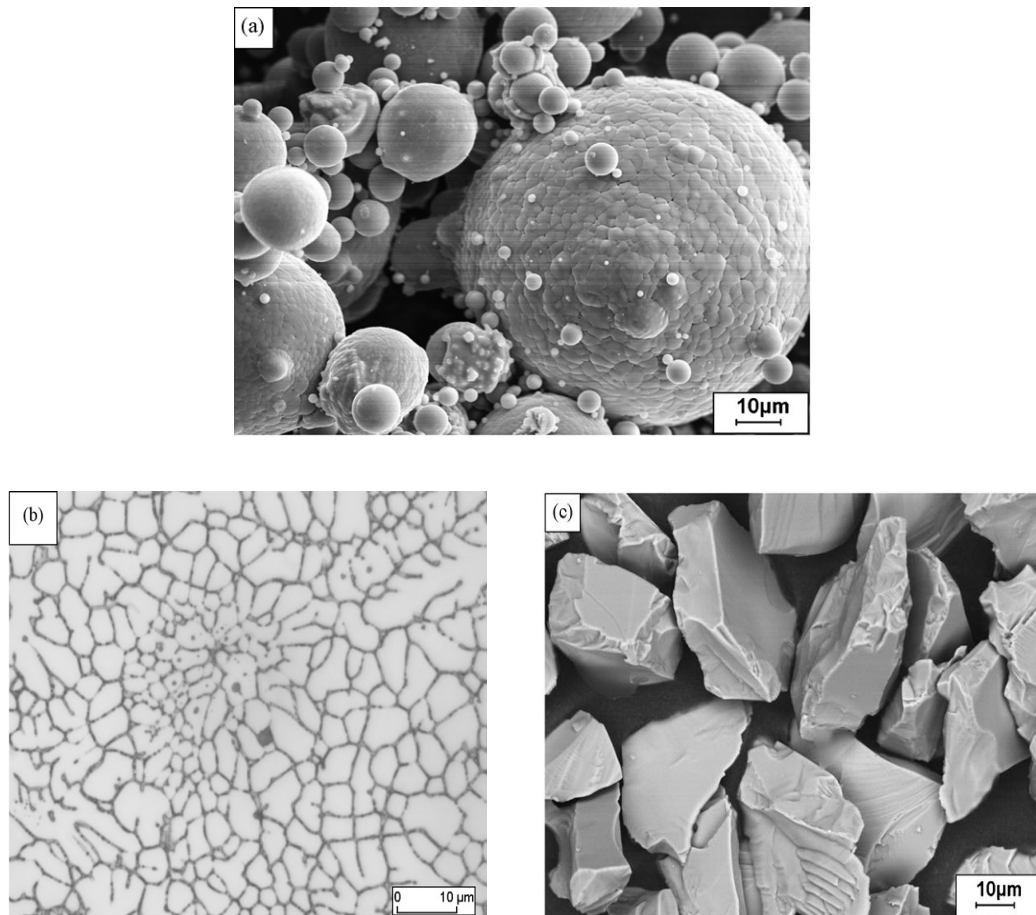


Figure 6.1 SEM micrographs show morphology of the as-recived state (a) aluminum matrix, topography; (b) aluminum matrix, etched; (c) SiC powders (Ozdemir & et. al., 2008).

6.2 Micro Structure and Interface

Fig. 6.2 shows typical cast composite sample obtained squeeze cast process. Cylindrical squeeze cast specimen with a diameter of 75 mm and thickness of 15 mm was obtained. After visual inspection, no casting failure and macro porosity have been observed.

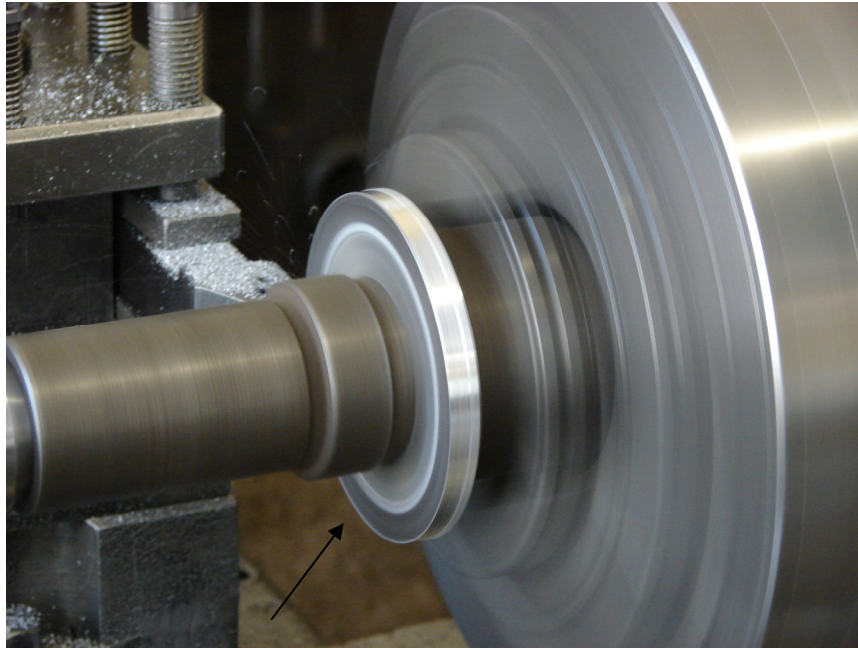


Figure 6.2 After squeeze casting view of the composite material produced.

Optical view of typical microstructure of composite containing 5 % SiC particles was given in Fig. 6.3a and 15 % SiC particles was given in fig. 6.3b. Studying the figures, SiC particle distribution in composite material produced is seen to be homogeneous. As seen from figs, After squeeze cast, both microstructures of composites exhibited almost pore free structure. The increase in reinforcement content resulted in more agglomeration in some areas.

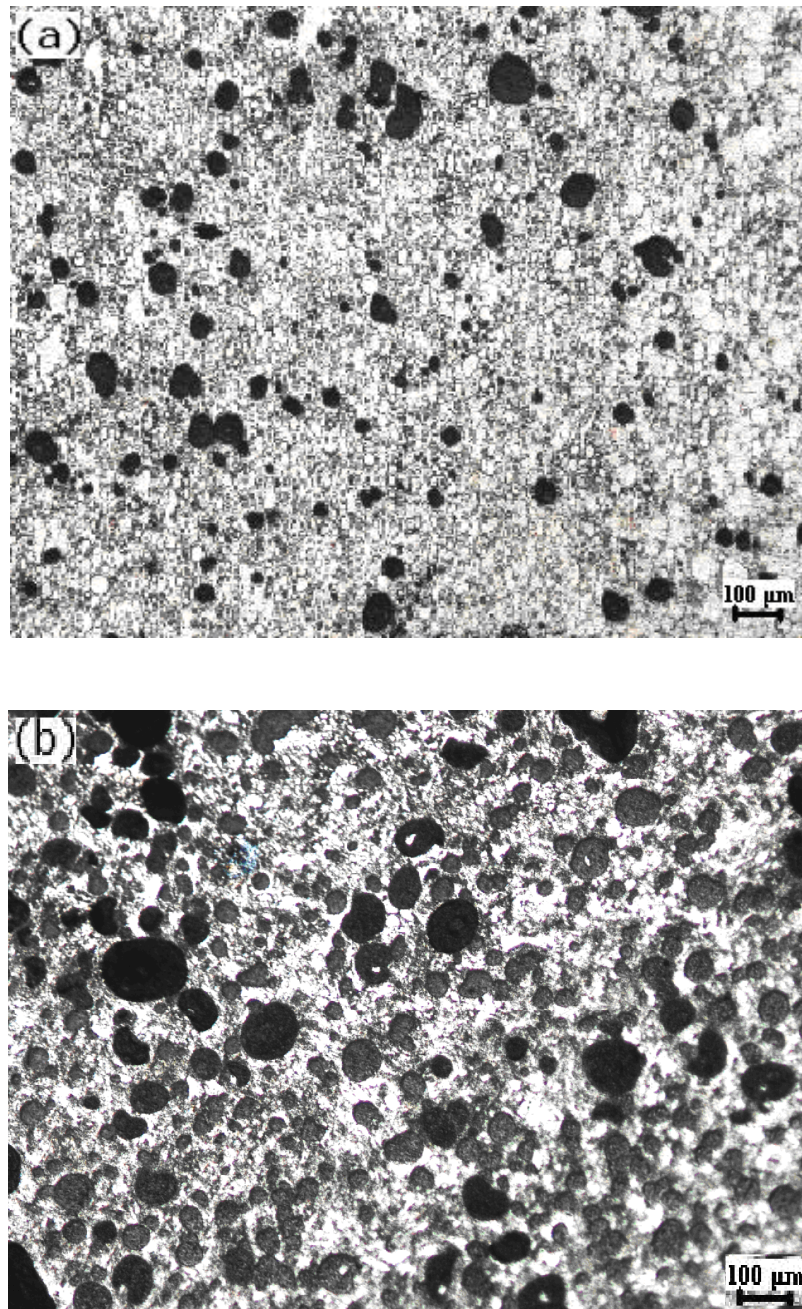


Figure 6.3 Optical picture showing general internal structure of the composite containing (a) 5 vol. % and (b) 15 % SiC particles.

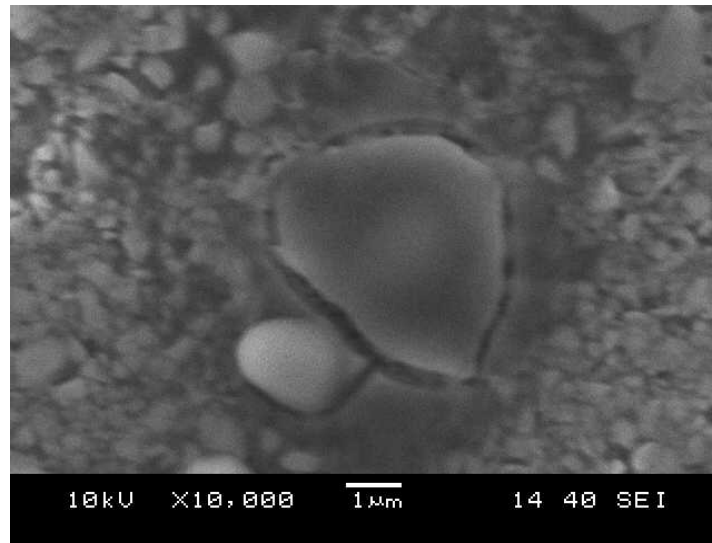


Figure 6.4 Al/SiC interface SEM pictures

SEM view of general micro structure belonging to 15 vol.% SiC reinforced aluminum matrix composite material was given in Fig. 6.4. The most important factor in the fabrication of particle reinforced composites is the uniform dispersion of the reinforcement. Therefore, the appearance of microstructure could give an insight into the quality of composites. Fig. 6.5 shows the representative microstructure of aluminum 15 vol.% SiC composite made by the squeeze casting method. The region with dark contrast corresponds to SiC particles. A uniform distribution of the second phase particles is revealed in the matrix phase. It is known that as the reinforcement is increased in volume, porosity and agglomeration of SiC particles become more evident.

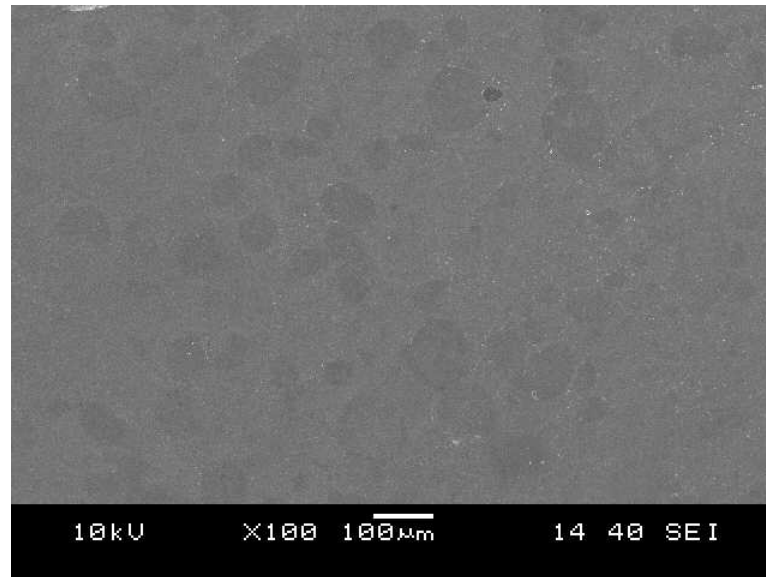


Figure 6.5 SEM picture showing general internal structure of the composite material produced.

6.3 Mechanical Properties

6.3.1 Hardness and Tensile Tests

Average values obtained following micro Vickers hardness measuring in matrix phase of 5 % and 15 % SiC reinforced composite have been respectively given in Table 6.1 and 6.2. Average hardness values of the composites solution treated at 525 °C for 2h subsequently quenched and then artificially aged at 160 °C for 4, 8,12,16, and 24 hours were given in Table 6.1. In Table 6.2, the hardness values for the composites in T4 condition, i.e., solution heat treatment at 525 °C for 2 h, quenched and following natural aging for 7 days, was obtained. Micro hardness curves according to these processes were given in Fig. 6.6.

Table 6.1 The effect of aging period on average hardness values of the composite material produced.

Heat Treatment period	Hardness Value (HV)	
	%5 SiC	%15 SiC
	Value	Value
4 Hour	113 $\pm$ 3	103.0 $\pm$ 3
8 Hour	139.50 $\pm$ 1	161.5 $\pm$ 5
12 Hour	147.04 $\pm$ 2	153.8 $\pm$ 5
16 Hour	124.10 $\pm$ 3	133.6 $\pm$ 3
24 Hour	114.30 $\pm$ 2	108.5 $\pm$ 3

Table 6.2 Average hardness values of the composite material produced after Squeeze cast (F) and T4 heat treatment

Composite Material Speciality	Average Hardness Value
5 % Al/SiC (F)	86 HV
15 % Al/SiC (F)	82 HV
5 % Al/SiC (T4)	128 HV
15 % Al/SiC (T4)	112 HV

Force-stroke curves obtained as a result of tensile test of four different samples were given in Figure 6.7. Tensile test results values following the experiments and analyses applied with the purpose of determining certain characteristics of composite material produced was shown in Table 6.3.

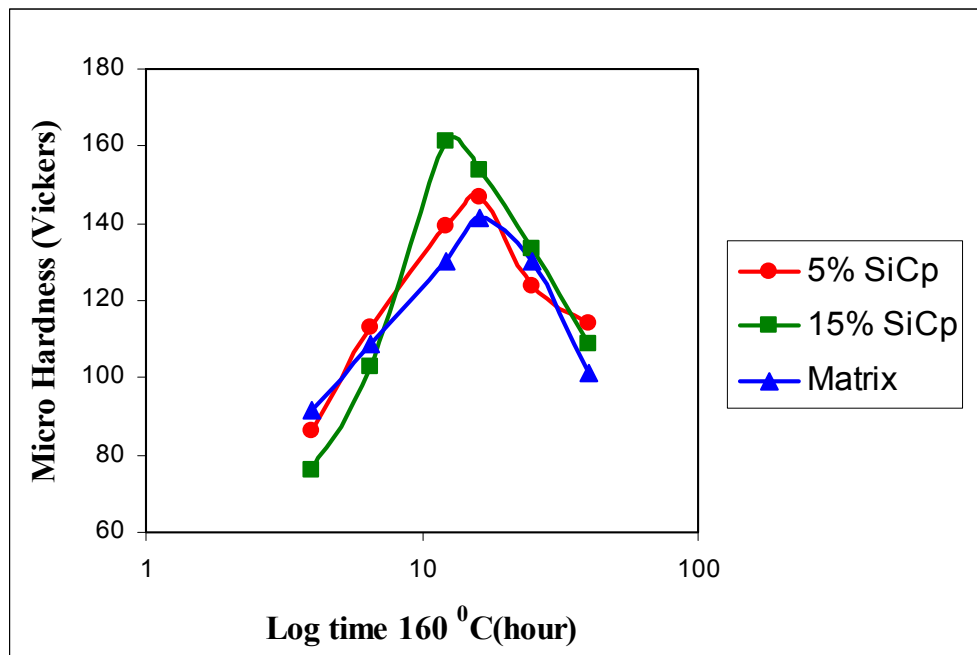


Figure 6.6 Micro hardness results average values

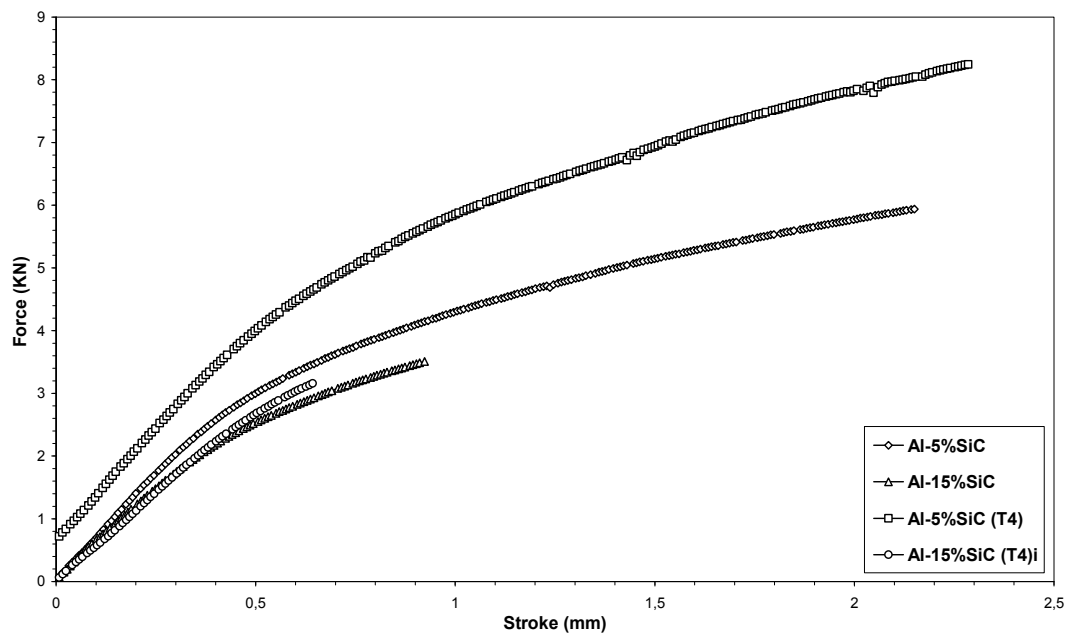


Figure 6.7 Force-stroke curves obtained as a result of tensile test of four different samples

Table 6.3 Average values of the composite material produced according to the results of tensile tests.

	5% Al/SiC	15% Al/SiC	5%Al/SiC(T4)	15%Al/SiC(T4)
UltimateTensile Strength, (MPa)	213	208	260	217
Yield Strength (MPa)	130	190	133	188
Elongation, at fracture, (%)	3,73	3,16	3,03	2,36

6.4 Fracture

Following tensile test applied with the purpose of determining fracture behaviour of composites produced, fractured surfaces of the samples were analyzed by SEM. Ductile fracture and grains of matrix phase are seen to have damaged due to brittle fracture as seen in Fig. 6.8a, Fig. 6.8b. Fig 6.8a, represents the fracture surface of the 15 vol.% SiC particle reinforced composite and shows relatively brittle fracture and a limited amount of ductile fracture of the surrounding matrix. It is observed that damage in some areas has occurred in the areas where SiC particles are intensive (Fig. 6.8b).

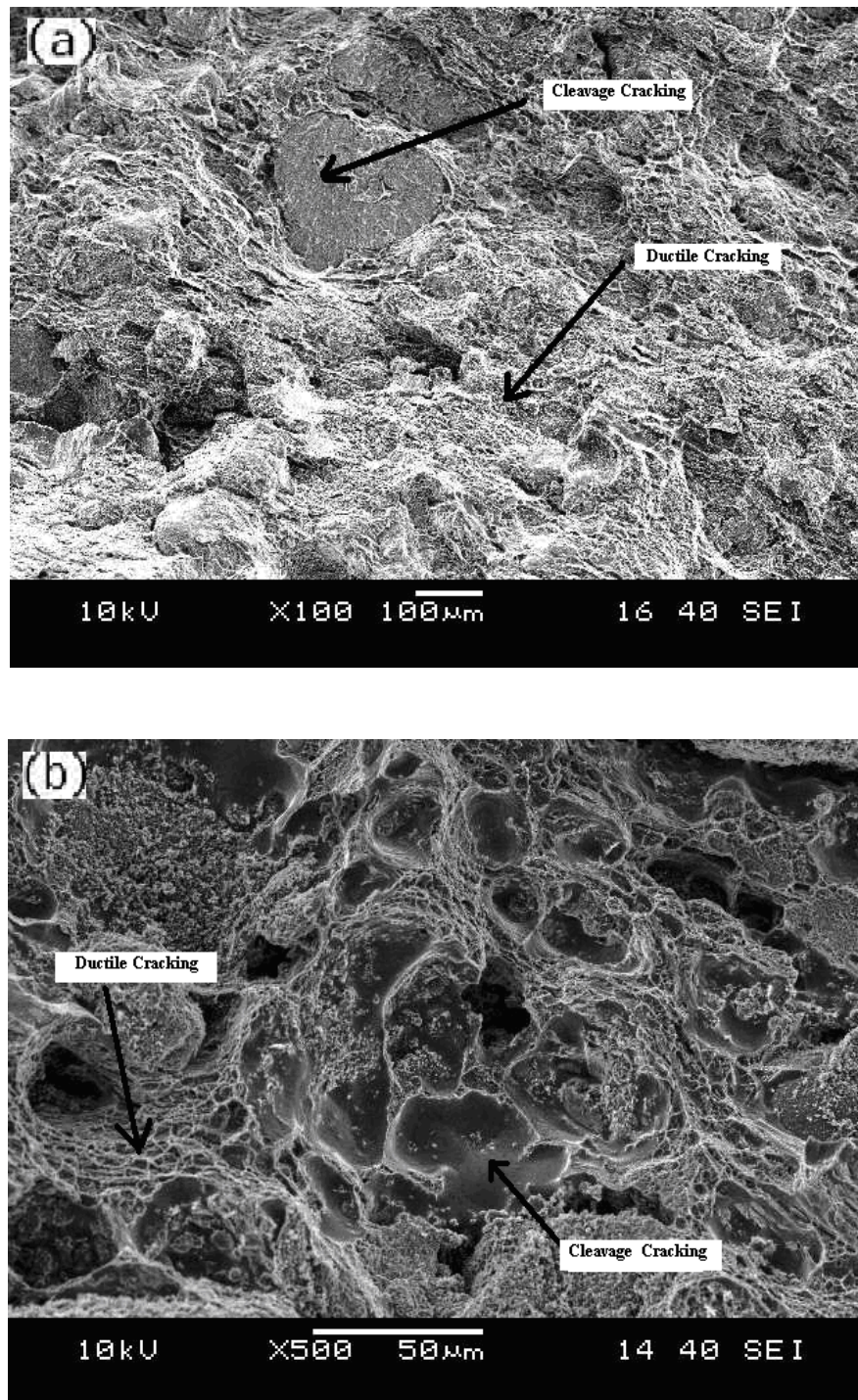


Figure 6.8 SEM view belonging to fracture surface after 15 vol.% SiC tensile test.
(a) Cleavage and ductile fracture, (b) fracture in the area particles are intensive.

It is observed that general fracture behavior of composite material does not occur at the interface of SiC/matrix, but occurs as brittle fracture of SiC particles and ductile fracture of matrix phase.(Fig. 6.9a). It is seen that some SiC particles have been pulled out fracture surface in Fig. 6.9b.

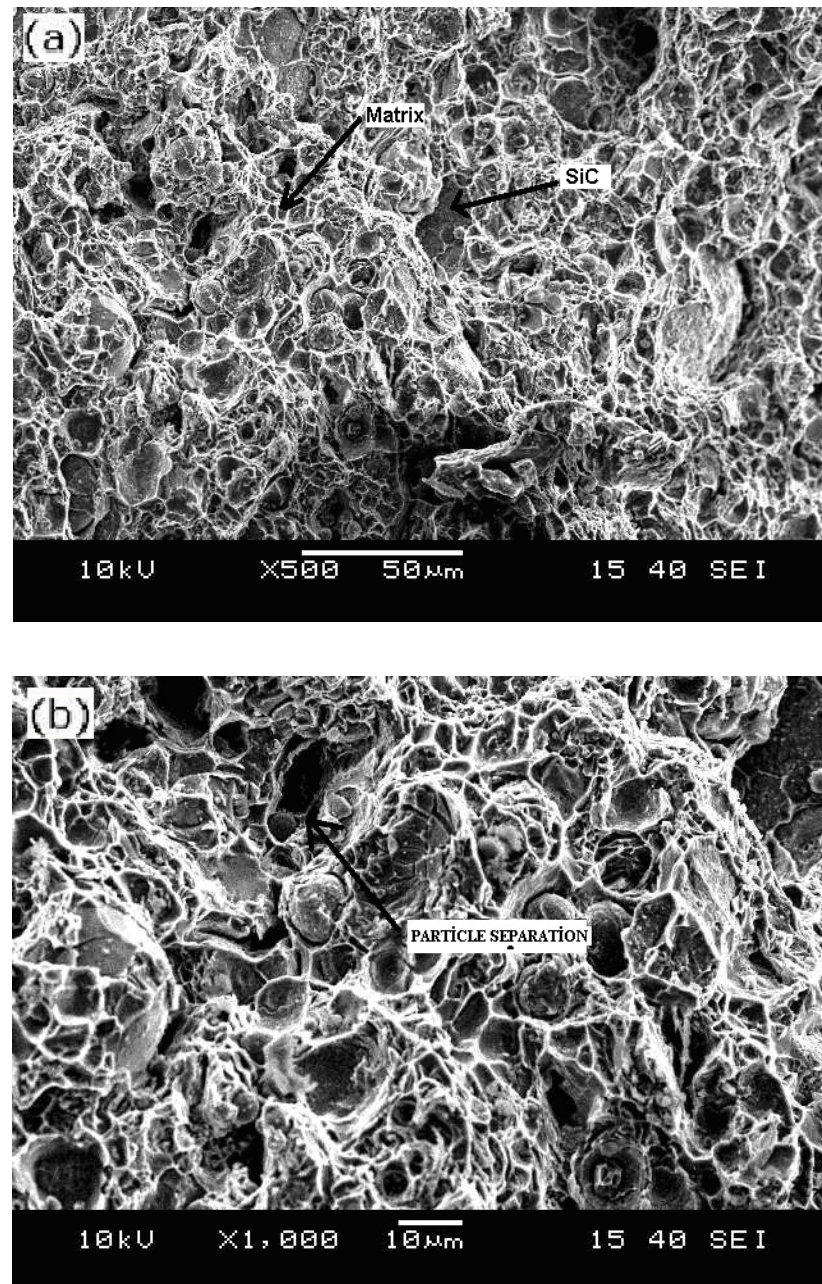


Figure 6.9 SEM view belonging to fracture surface after 5 vol.% SiC tensile test (a) ductile fracture of matrix phase and brittle fracture of SiC particles, (b) SiC particle separation.

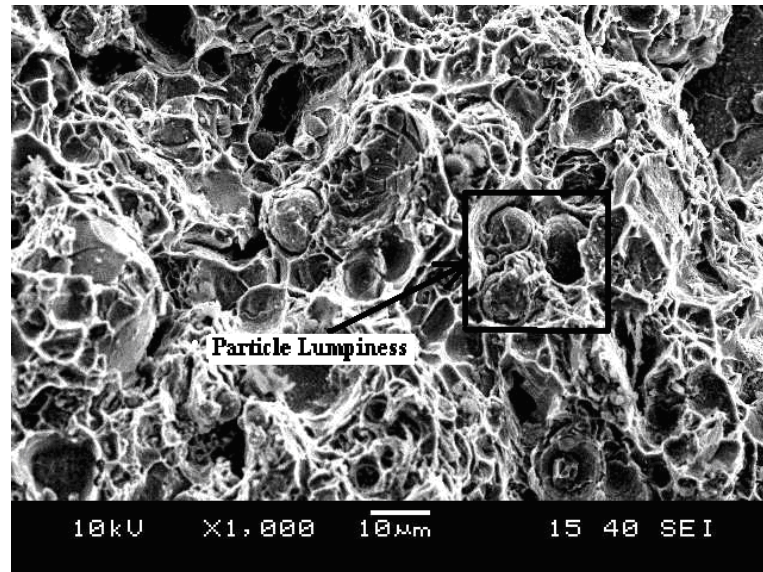


Figure 6.10 Regarding SEM view belonging to fracture surface after 5 vol.% SiC tensile test, local particle lumpiness.

Fig. 6.10 shows 5 vol.% SiC, SEM view belonging to fracture surface local particle lumpiness. Homogeneous ductile fracture has been observed in 15 vol.% SiC reinforced aluminum composite material we have produced. More homogeneous and ductile fracture is observed in our 15 vol.% SiC reinforced material than the fracture surfaces of 5 vol.% SiC reinforced aluminum composite materials in Fig. 6.11.

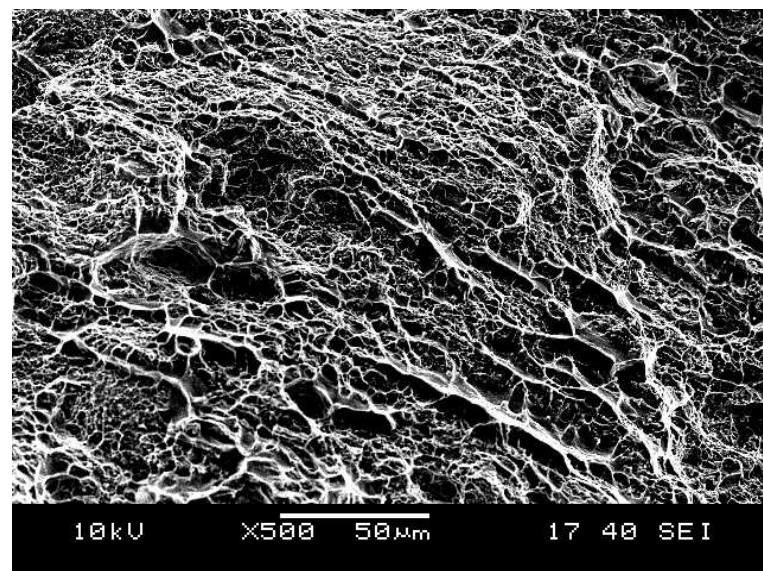


Figure 6.11 Homogeneous ductile fracture has been observed in our 15 vol.% SiC reinforced aluminum composite material.

CHAPTER SEVEN

DISCUSSION

7.1 Production and Composite Characteristics

Type of matrix phase in metal matrix composites, chemical compatibility of matrix and reinforcement phase, type of reinforcement phase, its amount, its distribution, interface characteristics of matrix/reinforcement phase, production method of composite and its parameters, its microstructure and the phases found in it, many other factors such as application of thermo mechanic and heat treatment play an important role in terms of determining the mechanical characteristics of MMC (Ahmad, 2001; Cöcen, Önel & Ozdemir, 1997). Therefore, matrix nature of composite material produced, SiC particle distribution, porosity quantity and interface characteristics have been analysed and their effect on mechanical characteristics have been researched.

7.1.1 Microstructure

In aluminum-based SiC particle reinforced composites produced by casting method, it is stated that there is a critical speed value with regard to determining the processes in which SiC particles are pushed by the dendrites during solidification process or absorbed by them and it is also stated that this value depends on surface energy, particle shape, thermal conductivity and the amount of the impurities in the solution (Samuel, Gotmare & Samuel, 1995).

Another important factor which affects especially the mechanical characteristics in metal matrix composites produced by casting method is porosity proportion, size and distribution. Hence, low proportion of porosity holds great importance. In general, porosity formation results from i) the fact that gases remain in the solution in the process of mixing, ii) that gas spaces form as a result of sudden reduction of hydrogen solution in the process of solidification, iii) tensile formed in the process of solidification and iv) that especially in MMC, particle reinforcements create suitable

areas at the interface of matrix/particle in order that porosity can nucleate and that the sections remain between particle piles are fed insufficiently (Hashim, Looney & Hashmi, 1999; Ahmad, Hashim & Ghazali, 2005). Primary ways of reducing the porosity proportion in the structure are applying gas removing processes before casting, casting under vacuum condition, pressure casting and application of thermomechanic treatments such as after casting forging or extrusion.

In this study, it has been observed that porosity in 5 vol.% and 15 vol.% SiC particle reinforced aluminum-based composite is % 0.49. High pressure applied in squeeze casting method prevents porosity formation resulting from the gas and shrinkage in solidified metal (Skolianos, Kiourtsidis, & Xatzifotiou, 1997; Cornie & et. al., 1986). In the present study, when the composite slurry solidified under at 100 MPa pressure, it is observed that SiC particles are piled in some areas shown in Fig. 6.3, but liquid aluminum alloy has fed these areas due to the pressure applied and no spaces are formed. If we apply more pressure like nearly 120 MPa or over, as it is observed in other studies, cracking and rupture may occur to SiC_P as seen in Fig. 7.1.

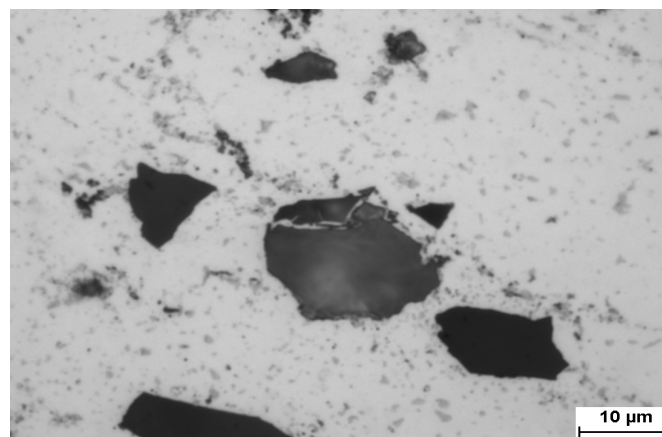


Fig. 7.1 An example of particle fracture emerged as a result of excessive pressure applied in the process of squeeze casting.

Microstructure of the composites which were produced by applying stir casting and after extrusion (Tekmen & Cöcen, 2003), analyzed in previous studies, and squeeze casting in this study have been indicated in Figure 7.2. Porosity formed as a result of particle piling in the structure following stir casting process. In spite of the fact that porosity level is considerably reduced following extrusion, porosities are

observed in some areas especially as a result of particle piling. This indicates that extrusion process is effective in removal of the porosities, the size of which are below a certain level (Tekmen, Ozdemir, Cöcen & Onel, 2003). Likewise, porosity level is reduced at the rate of respectively 97 % and 64 % by squeeze casting method when compared to stir casting method and extrusion treatment. In the present study it has been shown that the porosity content is significantly reduced to low level 0.49%.

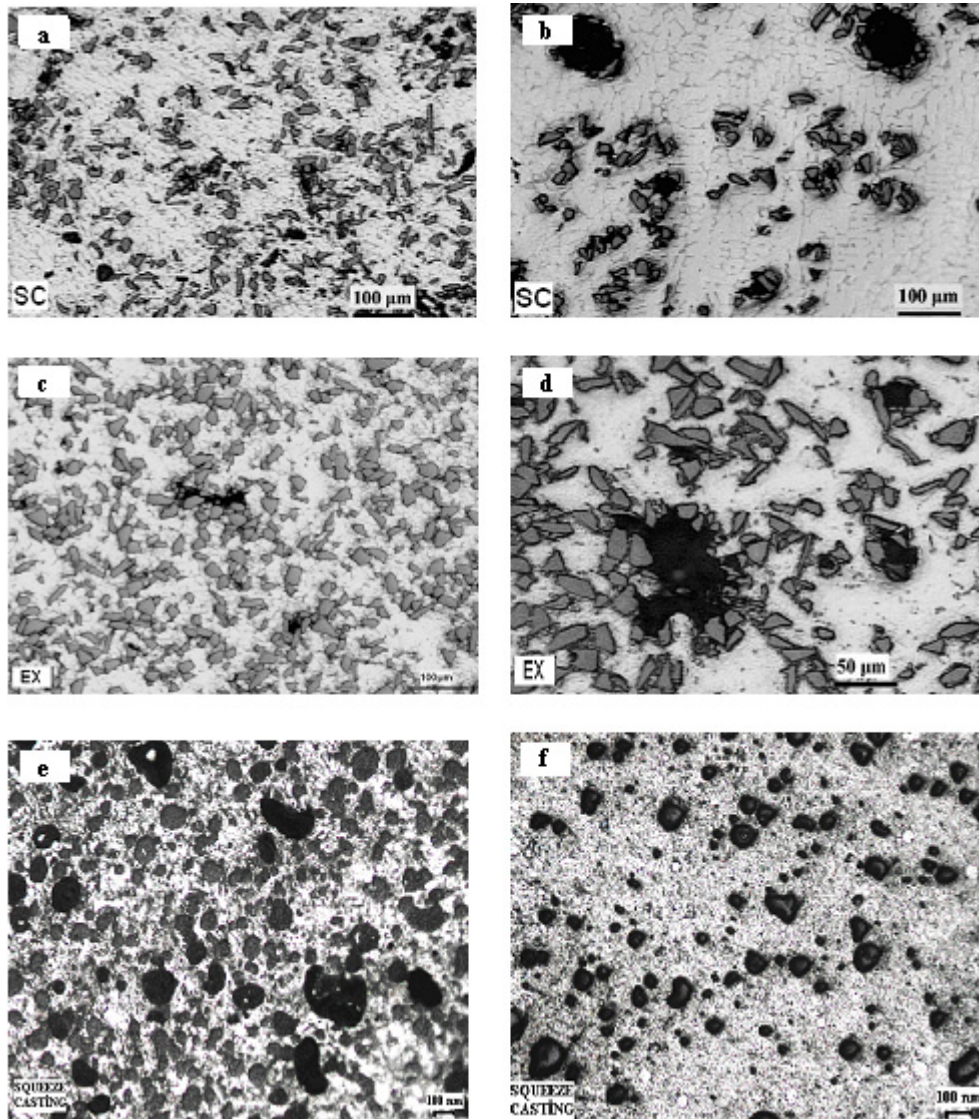


Figure 7.2 Microstructure of the composite material after stir casting (a,b), extrusion (c,d) (Tekmen & Cöcen, 2003) and squeeze casting (e,f) (Present study).

According to the EDS results of the 5 vol.% SiC composite in T4 condition after tensile test, matrix phase has homogeneously melted.

SiC has not separated from matrix phase and created a strong bonding. As it can be understood from Fig. 7.3, 7.4 and 7.5 reinforcement particles seem to be well bonded to matrix.

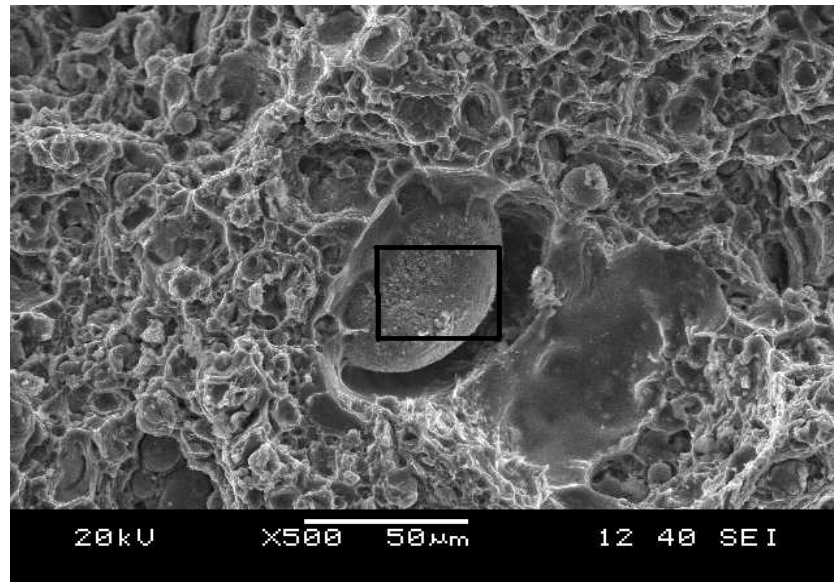


Figure 7.3 A profile from the EDS area of our 5 vol.% SiC reinforced composite material we have produced.

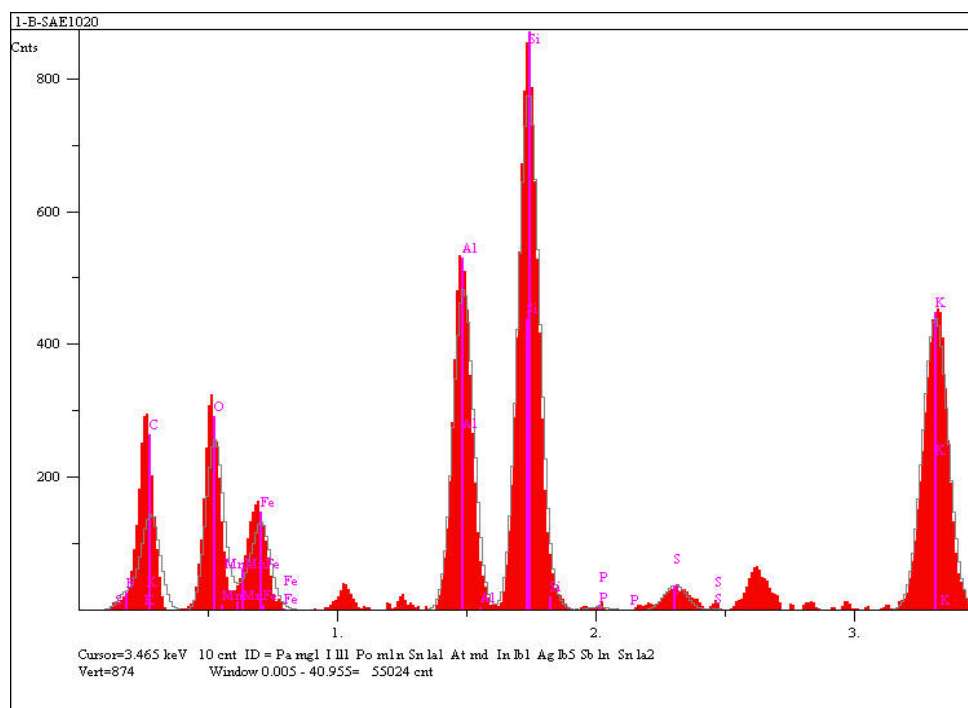


Figure 7.4 Analysis of the EDS area of our 5 vol. % SiC reinforced composite material we have produced.

El.	Line	Intensity (c/s)	Error 2-sig	Conc	
C	K α	0.19	0.086	11.193	wt. %
Al	K α	28.79	1.073	37.516	wt. %
Si	K α	22.17	0.942	47.612	wt. %
Cu	K α	0.50	0.141	3.679	wt. %
				100.000	wt. % Total
kV		20.0			
Takeoff Angle		35.0°			
Elapsed Livetime		100.0			

Figure 7.5 % percentage weighted analysis of the EDS area of our 5 vol. % SiC reinforced composite material we have produced.

7.1.2 Mechanical Characteristics

Increase of durability in MMC by reinforcement phase depends on the size, shape, amount of reinforcement phase and the distance between the particles. It is possible to explain durability increase by Orowan mechanism. It is generally accepted that for a matrix and reinforcement phase given, when the rate of reinforcement phase increases, yield and tensile strength of the material also increases (Stefanos, 1996; Vogelsang, Arsenault & Fisher, 1986). When movement of dislocations is prevented by SiC particles, the required shear stress in order to around also determines yield stress. As a result of the continuation of dislocation movement between particles, a dislocation ring forms around SiC particles. Each dislocation moving on sliding plane adds one ring and causes dislocation intensity to increase. This dislocation rings causes internal stress and thus, causes matrix phase deformation hardening. However, in the conclusion that particle size and the distance between particles are relatively larger, increase of dislocation stress occurs due to the increase of internal stress in the course of heat treatment and solidification owing to the difference between thermal expansion equations of matrix and reinforcement phases by the mechanism explained above (Ahmad, 2001; Kainer, 2006; Dieter, 1988). Reinforcement phase addition to matrix alloy in metal matrix composites increases rigidity and durability, but reduces ductility (Qin, Chen, Zhang, Wang & Wang, 1999; Llorca & Gonzalez 1998). The main factor which increases ductility in MMC

is microstructure of matrix phase, porosity level and matrix/reinforcement phase interface resistance (Gustafson, Panda & Raj, 1997).

Matrix phase microstructure and SiC particle distribution, which have considerable effect on the mechanical characteristics of the composite, depend on solidification speed of casting, and when the solidification speed increases, the distance between dendrites reduces and more uniform SiC particle distribution is obtained (Samuel, Liu & Samuel, 1993). On the other hand, when the distance between dendrite arms increases, tensile strength and strain values reduce (Askeland & Phule, 2002). Thus, it has been observed that, in some areas of fracture surfaces, the damage occurs in the areas where SiC particles intensively exist. (Figure 6.8b) This also accounts for low ductility of the composite produced. Yet, according to the study in which the effect of microstructural factors in particle reinforced MMC on ductility, particle piling has restricted deformation in matrix phase, improved particle rupture and thus, caused ductility to be reduced (Llorca & Gonzalez, 1998). In this study, with application of pressure during solidification dendrite structure significantly reduced. It is well known that coarse dendritic structure have negative effect on mechanical properties.

One of the most important factors which affect the mechanical characteristics of metal matrix composites is porosity (Li, Y.M. & Li, R.D. 2001; Samuel & et. al., 1995). Since the wettability of SiC particles by aluminum alloy is less, microporosities formed at the interface of matrix/reinforcement phase reduce interface strength of matrix/reinforcement phase. Also, the porosity resulting from shrinkage of matrix phase and dissolved gases reduces the area on which load is applied and affects the mechanical characteristics of the composite negatively (Caceres, 1995; Tekmen & et. al., 2003). Moreover, it is also known that according to the studies, due to the fact that liquid metal is unable to be transmitted to the areas between SiC particle piles formed for various reasons in the course of production some gaps are formed. It is stated that these porosities formed as a result of particle piling have affected the mechanical characteristics of the composite (Zhou & Xu, 1997; Tekmen & et. al., 2003). Ahmad et. al. (2005) has examined the effect of

porosity level on the mechanical characteristics in A356 matrix SiC particle reinforced composite, he has observed that increased porosity level has reduced tensile strength, yield strength and strain values. Ahmad (2005) has discovered that tensile strength, yield strength and strain values of the composite with 20 vol.% SiC particle reinforced by volume and 4 % porosity level are respectively 129.9 MPa, 77.4 MPa and % 1.45. When the results which Ahmad (2005) have discovered and the ones obtained in this study are compared, it is observed that production of composite material by squeeze casting method have considerably increased the mechanical characteristics. The primary reason of this is that porosity level, which has a considerable effect on the mechanical characteristics by solidification of casting under pressure in this study, has considerably been reduced.

The effect of the interface of matrix/reinforcement phase on the mechanical characteristics of the composites is another factor. Thus, due to the products emerging as a result of the reactions occurring at the interface, tensile strength and strain values are caused to reduce (Salvo, Esperance, Suery, & Legoux 1994; Suery, 1998).

Tekmen et. al. (2003), has discovered that tensile strength, yield strength and strain values of 12 μ m size and 20 vol.% by volume SiC particle reinforced and AlSiMg matrix alloy composite they have produced by mix casting and extrusion method are respectively 182 MPa, 110 MPa and 7.9 %. When these results are compared to the results of the tensile test of the composite produced by squeeze casting method in this study, a considerable increase is observed in yield strength, tensile strength and strain values. Thus, in the other study, it has been stated that grain decrease, reinforcement phase and its distribution in both matrix and reinforcement phase by extrusion process are more uniform. Additionally, high plastic deformation applied by extrusion treatment may have relatively increased dislocation intensity and tensile strength. The increase in yield strength obtained in this study, indicates that matrix phase has began plastic deformation later. In other words, no interface separation of matrix/reinforcement phase occurs in elastic area. The primary reason of this is that when compared to the after extrusion, 0.49 %

porosity level by (1.19 %) squeeze casting method has been reduced at the rate of 59 %. Another reason of the increase in the yield strength is that SiC particles are mixed with matrix phase before melting process, placed in the mould and the temperature value is adjusted in the mould and melting is adjusted homogeneously.

In another study, Chen et.al. (2002), has discovered that tensile strength, yield strength and strain values of 12 μ m size and 21 % by volume SiC particle reinforced and A383 aluminum-based composite they have produced by stir casting method under 75 MPa pressure value are respectively 232 MPa, 121 MPa and 3%. Although yield strength and strain values of our 5% SiC reinforced composite material are very close to the results obtained in this study respectively 133 MPa, %3.03, there is a difference in tensile strength 260 MPa. The reason of this is probably the difference of SiC particle size +25 μ m and volume proportion in the composite produced in this study. Thus, in a study which has been made for determining the effect of reinforcement phase size 5 μ m or 13 μ m on the mechanical characteristics of 7075 aluminum matrix SiC particle reinforcement composites, in the event of using 5 μ m size SiC particle , tensile strength has increased at the rate of 10% (Doel, Loretto & Bowen, 1993). In another study, when volumetric proportion of reinforcement phase is risen to 25% from 15%, tensile strength of 15% by volume SiC particle reinforced composite with 6061 matrix has increased at te rate of 7% (Ahmad, 2001).

Sukumaran et. al. (2008) has discovered that tensile strength, yield strength and strain values of 23 μ m size and 10 vol.% by volume SiC particle reinforced and Al 2124 alloy composite they have produced by squeeze casting is respectively 421 MPa, 388 MPa and % 2. When these results are compared to the results of the tensile test of the composite produced by squeeze casting method in this study, a considerable decrease is observed in yield strength, tensile strength but increase elongation to fracture. As a consequence Al 2124 has difficult chemical composition.

Homogeneous ductile fracture has been observed in 15 vol.% SiC reinforced aluminum composite material we have produced by squeeze casting method. The

reason is that it occurs under equal pressure by affecting the composite material and the mould homogeneously in the temperature formed as a result of squeeze casting method and since there is a homogeneous melting in the internal structure. However, this ductile fracture has not been observed homogeneously but locally in our 5 vol. % SiC reinforced aluminum composite material as our other sample in this study. Finally, as it is seen in Figure 6.11, more homogeneous and ductile fracture has been observed in our 15 vol. % SiC reinforced material when compared to fracture surface of aluminum composite material.

One of the primary factors which affect fracture behavior in metal matrix composites is the size of the reinforcement phase, its shape and distribution, interface strength and ductility of matrix phase. Damage generally occurs as a result of the growth of the gaps around reinforcement phase and at the interface of matrix/reinforcement phase and as fracture of relatively larger particles (Chen & et.al., 2002; McDanel, 1985; Davidson, 1991). In this study, following examining the fracture surfaces, it is observed that fracture does not occur at the interface of matrix/reinforcement phase, but as ductile fracture of matrix phase and brittle fracture of SiC particles. Also, it has been observed that in some areas, damage has occurred in the areas where SiC particles are intensively found.(Figure 6.8b). The reason may be that tensile stress is quite intensive in the areas in which particles are more intensive. In fact, when particle proportion increases, the amount of load to be transferred to this area and consequently, stress concentration increases (Lloyd, 1991).

Due to the fact that wettability is less in aluminum-based SiC particle composites, it is stated that the gaps formed at the interface of matrix/reinforcement phase has caused SiC particles to separate from matrix under low loads and fracture to begin at the interface (Caceres, 1995). In this study, on the other hand, as it is seen in Figure 6.11, regarding the fracture surfaces of 15 vol.% SiC reinforced composite material, there is no sign with look to fracture starting at the interface of matrix/reinforcement phase and also there are no SiC particle gaps on the fracture surfaces and ductile fracture has been observed.

CHAPTER EIGHT

CONCLUSION

In this study, 5 % and 15 % by volume SiC particle reinforced Al-Cu alloy matrix composite has been produced by squeeze casting method. The reinforcement content, squeeze cast pressure, temperature and milling period was found to affect the strength, ductility, hardness and fracture of the composites. Aging behaviour of the composites is also studied in T4 and T6 conditions. The following conclusions can be drawn from the experimental results:

- 1) The fabrication of aluminum base composites using squeeze casting method results in a homogenous dispersion of the reinforcement particles in the aluminum matrix. Application of squeeze casting processing of the composites leads to break up of SiCp. The matrix is almost pore free. It has been observed that porosity in 5% and 15% volume SiC particle reinforced aluminum-based composite was calculated as 0.49 %.
- 2) An Al2017-5vol%SiC composite produced by squeeze casting has been shown that tensile strength 213 MPa, yield strength 130 MPa and elongation 3.73%, and an Al2017-15vol%SiC composite made by squeeze casting has been shown that tensile strength 208 MPa, yield strength 190 MPa, and elongation 3.16%.
- 3) After T4 heat treatment, it has been shown ultimate tensile strength, yield strength and elongation of the produced for 5 vol.% SiC composite was found to be 260 MPa, 133 MPa and 3.03% respectively and ultimate tensile strength, yield strength and elongation of the produced for 15 vol.% SiC composite was found to be 217 MPa, 188 MPa and 2.36% respectively.
- 4) When we examine the average results of micro hardness values of 5 vol.% and 15 vol.% SiC reinforced composites, micro hardness values of after squeeze casting are

respectively as follows: 86 Hv and 82 Hv, after T4 treatment, peak hardness values was found to be 128 Hv, 112 Hv.

5) The fracture surface of the SiC particle reinforced composite shows relatively brittle fracture and a limited amount of ductile fracture of the surrounding matrix. A fracture surface consisting of a mixture of dimple fracture. The appearances of fracture surface indicate good bonding between the matrix and the SiC particles. But some particle pull out also is present in the structure.

6) Increasing the volume fraction of SiC_p increases yield strength of the squeeze composites increases whereas the tensile strength and elongation is slightly decreased.

7) In comparison to blended powders, short period of milling composite powders exhibited improved microstructure after squeeze casting. Increasing squeeze cast pressure from 100 MPa to 120 MPa lead to more uniform distribution of reinforcement particles but it cause particle cracking as well.

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