# HOT PRESSING STUDIES IN Al-Cu-Mg-SiC SYSTEM

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FATMA MUAZZEZ ŞİMŞİR

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T.C. YÜKSEKÖĞRETİM KURULU
DOKÜMANTASYON MERKEZİ

Approval of the Graduate Scool of Natural and Applied Sciences

Prof.Dr. Tayfur ÖZTÜRK
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science.

Prof.Dr. Naci SEVİNÇ
Head of Department

This is to certify that we have read this thesis and that in our opinion it is fully adequate in scope and quality, as a thesis for the degree of Master of Science.

Assoc.Prof.Dr. Bilgehan ÖGEL
Supervisor

Assoc. Prof. Dr. Rıza Gürbüz
Co-supervisor

Examining Committee Members:

Prof.Dr. Şakir BOR
Prof. Dr. İshak KARAKAYA
Assoc.Prof.Dr. Rıza GÜRBÜZ
Assoc.Prof.Dr.. Bilgehan ÖGEL
Assoc.Prof.Dr. Tamer ÖZDEMİR

T.C. YÜKSEKÖĞRETİM KURULU
DOKÜMANTASYON MERKEZİ

# ABSTRACT

## HOT PRESSING STUDIES IN Al-Cu-Mg-SiC SYSTEM

Şimşir, Fatma Muazzez
MSc., Department of Metallurgical and Materials Engineering
Supervisor: Assoc. Prof. Dr. Bilgehan ÖGEL
Co-supervisor: Assoc. Prof. Dr. Rıza GÜRBÜZ

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In this study the primary aim was to produce Al-SiC metal-matrix composites comprising 40 volume percent SiC using a conventional hot pressing method.The matrix alloy was Al-5wt%Cu in all mixtures. All hot pressing experiments were carried out with a single end die and under nitrogen atmosphere. In the first part of the study, the effect of addition of elemental Mg powder to the microstructural development was investigated. For this purpose, both hot pressing and quenching experiments were carried out with Al-5wt%Cu and Al-5wt%Cu-5wt%Mg powder mixtures. Although the liquid phase was seen to wet prior Al particle boundaries, the residual pores which originate from Mg powder could not be eliminated. In the second part, several hot pressing parameters were changed to obtain sound Al-5wt%Cu-40vol% composites, which include hot pressing time, pressure, temperature and heating rate ($3^0$C/min). It was found that a slower heating rate to hot pressing temperature ($615^0$C) was most effective for a succesful hot pressing operation.

The specimens are characterised by SEM observations and three point bending tests. It was found that the flexural strength of Al-5wt%Cu-SiC composites decreases from 451 MPa to 128MPa with an increase in SiC content from 20vol% to 40vol%.

**Keywords**: Al-SiC composites, hot press, mechanical properties, flexural strength, three point bending test, powder metallurgy, wetting

# ÖZ

## Al-Cu-Mg-SiC SİSTEMİNDE SICAK PRESLEME ÇALIŞMALARI

Şimşir, Fatma Muazzez
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Tez Yöneticisi: Doç. Dr. Bilgehan Ögel
Ortak Tez Yöneticisi: Doç. Dr. Rıza Gürbüz

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Bu çalışmanın temel amacı hacimce %40 SiC içeren metal anayapılı kompozitleri sıcak presleme yöntemi ile üretmektir. Bütün karışımlarda ana yapı Al- %5Cu alaşımıdır. Bütün sıcak presleme deneyleri azot atmosferi altında yapılmıştır. Çalışmanın ilk kısmında, elementel Mg tozunun mikro-yapı üzerindeki etkisi incelenmiştir. Bu amaçla, Al-%5Cu ve Al-%5Cu-%5Mg karışımlarıyla hem sıcak presleme hem de su verme deneyleri yapılmıştır. Oluşan sıvı faz Al tane sınırlarını ıslatmasına rağmen, Mg tozu kaynaklı gözenekler yok edilememiştir. İkinci kısımda, gözenek içermeyen hacimce %40 SiC içeren Al-SiC kompozitleri üretmek amacı ile çeşitli sıcak presleme parametreleri değiştirilmiştir. Bu parametreler arasında sıcak presleme süresi, basınç, sıcaklık ve ısıtma hızı yer almaktadır. Yavaş bir ısıtma hızının ($3^0$C/dakika) başarılı bir sıcak presleme işlemi için etkin olduğu bulunmuştur. Numuneler tarama elektron mikroskobu kullanılarak ve üç nokta bükme

deneyleri yapılarak karakterize edilmiştir. SiC miktarı hacimce %20'den %40'a çıkarıldığında bükme dayancının 451 MPa'dan 128 MPa'ya düştüğü kaydedilmiştir.

**Anahtar Kelimeler**: Al-SiC kompozitleri, sıcak pres, mekanik özellikler, üç noktadan bükme dayancı, toz metalurji, ıslatma.



T.C. YÜKSEKÖĞRETİM KURULU
DOKÜMANTASYON MERKEZİ

*To Cennet & Fatih Şimşir*

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# CHAPTER 1

## INTRODUCTION

Metal-matrix composites (MMCs) are a class of advanced materials, which have been developed for weight-critical applications.

In recent years, extensive investigations have been carried out on particulate metal-matrix composites. These in general have consisted of high strength aluminium alloy matrices reinforced with ceramic particulate such as SiC and $B_4C$ **[1-4]**. Metal-matrix composite materials offer significant advantages over monolithic metals and alloys, and their utilization in defence and aerospace applications has been growing steadily. However, their adoption in other industries, has been relatively slow **[5]**.

The major difficulty in the production of a metal-matrix composite is inhomogeneous distribution of the reinforcement material, which affects mechanical properties adversely **[6,7,8]**. Method of production has a great importance in obtaining homogeneous mechanical properties and achieving reproducibility.

In order to achieve homogeneity in the distribution of reinforcement particles, several methods such as mechanical alloying or ball milling have been investigated. The method of consolidation is also used for obtaining compacts with controlled microstructures that is achieved by the simultaneous application of pressure and heat rather then sequentially as in conventional powder

metallurgy processing. However, production steps involves complex and cost increasing methods such as vacuum degassing, HIP'ing. Hence, a method that optimises the enhanced property and the cost has to be investigated **[9]**.

Classical powder metallurgy methods are not so suitable for producing aluminium alloy matrix composites with 20% or more reinforcement particles due to the problems encountered during deformation processing **[9]**. As a result, amount of reinforcement particulate is usually limited by 25 volume percent. On the other hand, a recent study showed that aluminium composites containing 0 to 30 percent SiC can be produced by conventional hot pressing without porosity **[10]**. In this method, hot pressing is carried out under nitrogen atmosphere and copper is added to form a liquid phase.

The aim of this thesis work is to apply the method developed by Ögel, Bolay and Kaya **[10]** to the aluminium matrix composites containing 40 volume percent or more SiC. The production of Al-40vol%SiC composites using a conventional hot pressing method would possibly eliminate the extrusion step, which is very difficult at high reinforcement contents. Also, conventional hot pressing is the most simple powder compaction method, which can be adapted for industrial applications relatively easily.

The research involves the following main steps:

- Affect of hot pressing parameters on Al – Cu powder mixture
- Examination of the hot pressing behaviour of Al – Cu – Mg powder mixture
- Application of the above results to the samples containing SiC
- Mechanical testing of Al-Cu-SiC composites by three point bending test.
- Fracture surface analysis of Al-Cu-SiC and Al-Cu-Mg-SiC composites

# CHAPTER 2

# THEORY

## 2.1 Metal-Matrix Composites

Metal-Matrix Composites (MMCs) are engineered combinations of two or more materials in which tailored properties are achieved. The development of MMCs has followed several different avenues over the last thirty years. Engineered MMCs consisting of dispersions of continuous or discontinuous fibre(s), whisker(s), or particle(s) in metal posses unique combinations of properties not achievable in monolithic materials. These properties may include high specific strength, machinability, and friction; wear resistance, low thermal expansion.

The initial activity concentrated on continuous reinforcements, particularly on carbon and boron fibres, [1]. The properties obtained by continuous reinforcement can be impressive but the high cost of reinforcement; together with the high processing costs have limited their commercialisation.

Reinforcement costs were reduced with the introduction of discontinuous short fibre, particularly short staple, polycrystalline $Al_2O_3$ fibres, [2]. Discontinuous fibres have found commercial application as selective reinforcement in the area of diesel pistons when the pistons are produced by squeeze casting. While the basic cost of short fibres is much lower than continuous ones, they require consolidation into a semi-rigid pre-form and infiltration by squeeze casting to form the part, which add to the final cost. In



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addition, MMCs reinforced with both continuous and short fibres have no secondary fabrication capability, so they cannot be rolled to plate or extruded into profiles. However, a secondary fabrication capability is important for many applications, which was provided by the addition of SiC whisker reinforcement [3]. Whiskers are more expensive than the short fibres, but give superior modulus and strength in the final composite. However, whiskers are in a very dangerous aspect ratio range causing diseases such as lung cancer.

Limitations with continuous fibres, short fibres and whisker-reinforced composites have led to the recent development of particulate reinforced metal matrix composites (PMMCs). Particulate reinforcement does not give as great an increase in properties as continuous fibres or whiskers, but the limited improvement is offset by lower cost and greater processing flexibility. Almost every potential ceramic reinforcement is available, as particulate, but the major effort has concentrated on SiC and $Al_2O_3$.

### 2.1.1 Processing of Metal Matrix Composites

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

- Solid state; (including powder metallurgy and diffusion bonding)
- Liquid state

Majority of commercially viable applications is now produced by liquid state processing because of inherent advantages of this processing technique over solid state techniques, that is, the liquid metal is generally less expensive and easier to handle than powders, and the composite material can be produced in a wide variety of shapes, using methods already developed in casting industry for reinforced metals (stir casting, co-spraying). [4]

The lowest cost option for production MMCs is stir casting. In this process particles are stirred into a bath of molten metal, which is then cooled to solid. Proprietary techniques have been developed which achieve a reasonably even distribution in the ingot, especially where coarse particles are used at the lower volume fractions. Further working of the material, for example extrusion, is often used to improve the quality of distribution and breakdown the ingot structure.

Co-spraying uses a modified form of co-spray process incorporate particles which can be incorporated, and the volume fractions which can be achieved, but the process, in general, gives great uniformity of product than in stir casting. Secondary working can be used to break down the sprayed structure of the material and close up any porosity. **[5]**

Liquid state processes are often suffer from a lack of reproducibility as a result of incomplete control of the processing parameters, and undesirable chemical reaction at the interface between molten metal and the reinforcement. On the other hand, segregation effects and brittle reaction product formation are at a minimum for solid-state processes.

## 2.2 Powder Metallurgy MMC Composites

Powder metallurgy techniques may be used for producing all of the main families of MMC materials. In case of continuously reinforced composites powder (slurry) impregnation routes have been used to produce multi-filament MMC development. Fully dense matrix structures and good fibre distributions can be achieved. However, it is difficult to achieve matrix property optimisation and to avoid fibre damage in processing. From this aspect, PM routes have disadvantages compared to melt infiltration techniques by which composite material can be produced by shorter high temperature exposure and less reliance on deformation processing.

In the case of discontinuously reinforce MMC there exist a direct competition between PM and other processing routes. PM route is versatile in terms of range of the matrix and the reinforcement types that it can encompass. In terms of ultimate commercial exploitation, cost could lead to other routes versus PM but those applications it is the only route that can provide the required MMC microstructure.

Powder metallurgy is the most common method for the fabrication of particle reinforced metal matrix composites. Preparation of metal matrix composites using powder metallurgy techniques offers several advantages over fusion metallurgy or fusion bonding. Lower temperatures can be used during PM based composite production compared to the production based on fusion metallurgy. The result is the fewer interactions between the matrix and the reinforcement. By minimizing undesirable interfacial reactions, improved mechanical properties are obtained. In some cases, PM techniques permit the preparation of composites that cannot be prepared by fusion metallurgy. **[6]**

In PM route, powdered metal is blended with the reinforcement then densified by die pressing, canning, extrusion and hipping. This is probably more expensive than the other techniques, but allows the greatest flexibility in choice of particle size and reinforcement concentration. As other techniques, secondary processing may be used to improve the distribution and break down prior particle boundaries of the constituent metal powders. **[7]**

Powder metallurgy aluminium alloys with enhanced physical and mechanical properties have been produced by a variety of manufacturing routes of metal matrix composites by powder metallurgy methods, ***Figure 2.1.***

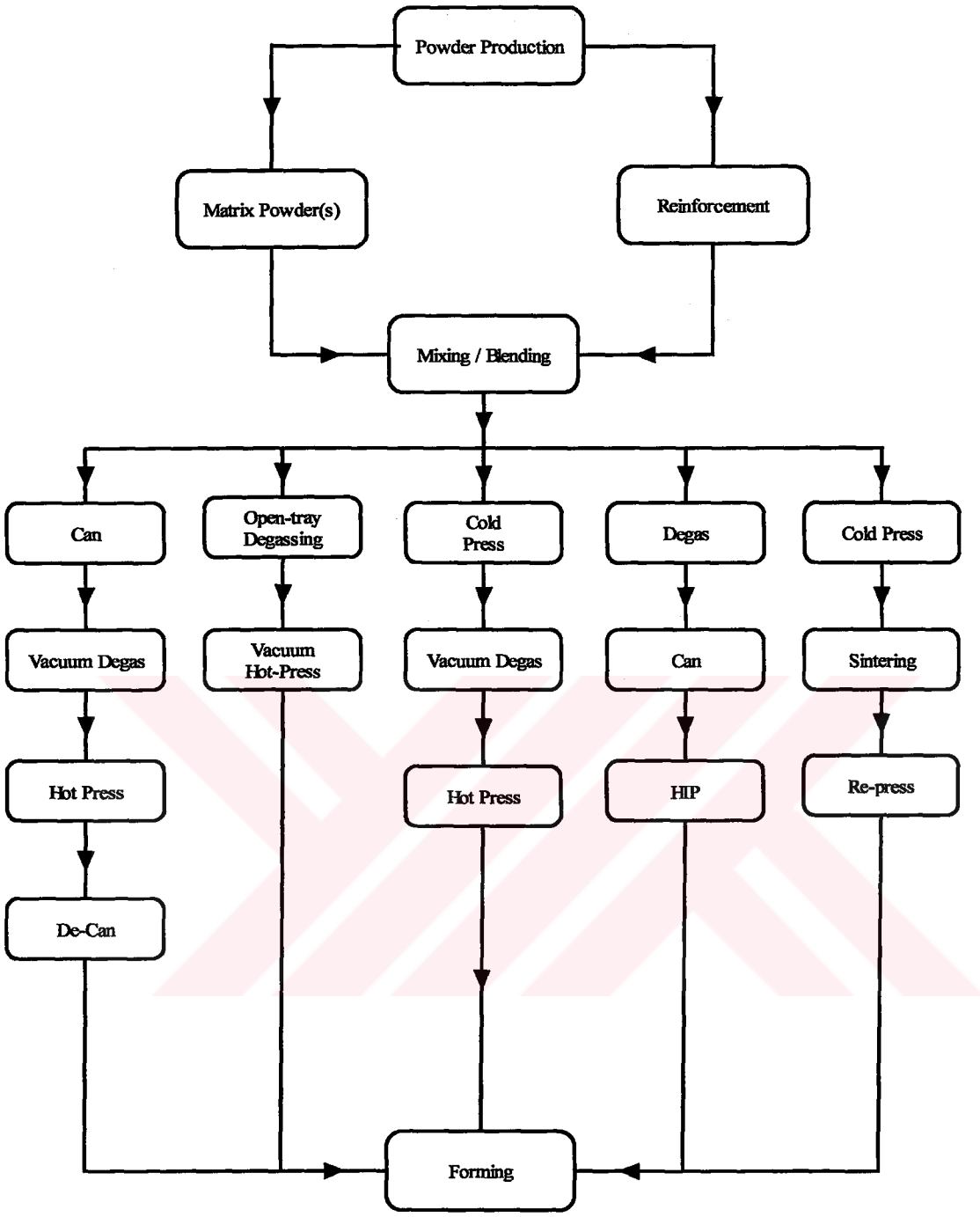


***Figure 2.1*** Manufacturing Routes of PM for Producing MMCs

## 2.3 Mechanical Properties of Particle Reinforced MMCs

The strength of a crystalline solid is determined by the stress required to either generate or move dislocations across a span of the lattice. Dislocation motion is controlled either by dislocation interaction some matrix structure defined by the presence of dispersoids (indirect dislocation-particulates interaction) or dislocation-dislocation interactions and direct dislocation particulates interaction. **[8]**

Metal matrix particulate reinforced composites generally defined as having a metal matrix containing a distribution of particles from 10μm to 200μm in size. In this sense the metal particulate composites differs from normal dispersion-hardened materials in which the particle size varies between 0.1μm and 2μm, the inter-particular distance is less then 5μm and the volume fraction of dispersoids is generally below 0.1. In mechanical point of view, while dislocation bowing between particles contributes to strengthening in dispersion and precipitation hardened materials, the same is not true for metal matrix particulate reinforced composites whose inter-particular distance is too large to yield any strengthening effect. **[9]**

The strengthening mechanism in metal matrix composites have been related to dislocation density in the matrix originating from differential thermal contraction **[10]**, geometric constraints and plastic deformation during processing. **[11]**

The generation of dislocations as a result of thermal contraction is caused by the large difference in the coefficient of thermal expansion of the matrix and the ceramic reinforcement. The misfit strains, which occur at the matrix-ceramic interface from thermal contraction during cooling, are capable of generating slip dislocations. For instance, thermal expansion coefficient of aluminium is ten times larger than that of SiC particulates. Hence, a misfit strain of about 1% is developed in aluminium matrix at the circumference of a SiC

particulate of 1μm diameter. This results in a dislocation density of $1.8\times10^{13}$ $m^{-2}$. These dislocations form a dislocation-induced substructure.

The presence of geometrical constraints also generates additional dislocations in the matrix. Increasing the size, and shape complexity of SiC particulate increases intensity of the dislocation generated at Al-SiC interface. Moreover, as particulate size or particulate surface curvature increases, the plastic zone size also increases.

### 2.3.1 Influence of Manufacturing Route on MMCs

The processing technique has a fundamental influence on the properties of a MMC. Manufacturing route imposes some constraints on the structure obtained, for instance;

- Particle size
- Nature of the reinforcement-metal interface
- Uniformity of the distribution achieved
- Segregation within the matrix

A comparative data for three main production processes are not available since different companies in the area use different alloys, heat treatments, reinforcement levels etc. ***Table 2.1*** gives a general overview of the relative performance and the cost of the three routes.

***Table 2.1*** Features of Main Processing Techniques for Producing MMC, **[14]**

| | STIR CAST | COSPRAY | PM |
|---|---|---|---|
| Particle size reinforcement, μm | 10-25 | 10-25 | 1-10 |
| Max. level of reinforcement available, % | 25 | 20 | 60 |
| Control of distribution in billet | LOW | BETTER | BEST |
| Mechanical Properties Achieved | LOWEST | INTERMEDIATE | HIGHEST |
| Cost | VERY LOW | LOW | HIGHER |

As can be seen from the table, PM route offers the greatest flexibility and provides the optimum properties but with the highest cost. Co-spray and stir casting techniques offers lower properties at rather lower prices. However, each class of materials tends to find its market dependent on the cost-property trade off. [12]

### 2.3.1.1 Matrix Alloy

Unlike continuous fibre reinforced composites where the properties are dominated by the fibres, the matrix alloy mainly influences particulate MMCs. In general, the higher the strength of the alloy the higher that of the MMC is. On the other hand, percentage strength improvement reduces with increasing alloy strength. Therefore, while it is possible to enhance a low strength alloy, some of the ultra high strength alloys are scarcely strengthening at all, ***Table 2.2.***

***Table 2.2*** Influence of Matrix Alloy on Strength of MMC, [14]

| | UNREINFORCED | | MMC (20 % SiC ) | | |
|---|---|---|---|---|---|
| Alloy Designation (SAE / AISI ) | Proof Strength ($MN/m^2$) | UTS ($MN/m^2$) | Proof Strength | UTS ($MN/m^2$) | Av.Str. Change, % |
| 1050 | 50 | 75 | 110 | 180 | +132 |
| 6061 (T6) | 275 | 310 | 340 | 420 | +30 |
| 2124 (T8) | 440 | 490 | 550 | 620 | +26 |
| 8090 (T8) | 430 | 530 | 490 | 560 | +9 |
| 7475 (T6) | 505 | 570 | 470 | 580 | -2 |

### 2.3.1.2 Reinforcement Level

A predictable rule of mixtures in order to affect certain properties such as Young's Modulus is varying the reinforcement level. ***Table 2.3*** and ***Figure2.2*** show how elastic modulus builds up quickly at even moderate reinforcement levels. Enhanced elastic modulus is the cause of a major attraction in use of particulate MMCs in structural applications.



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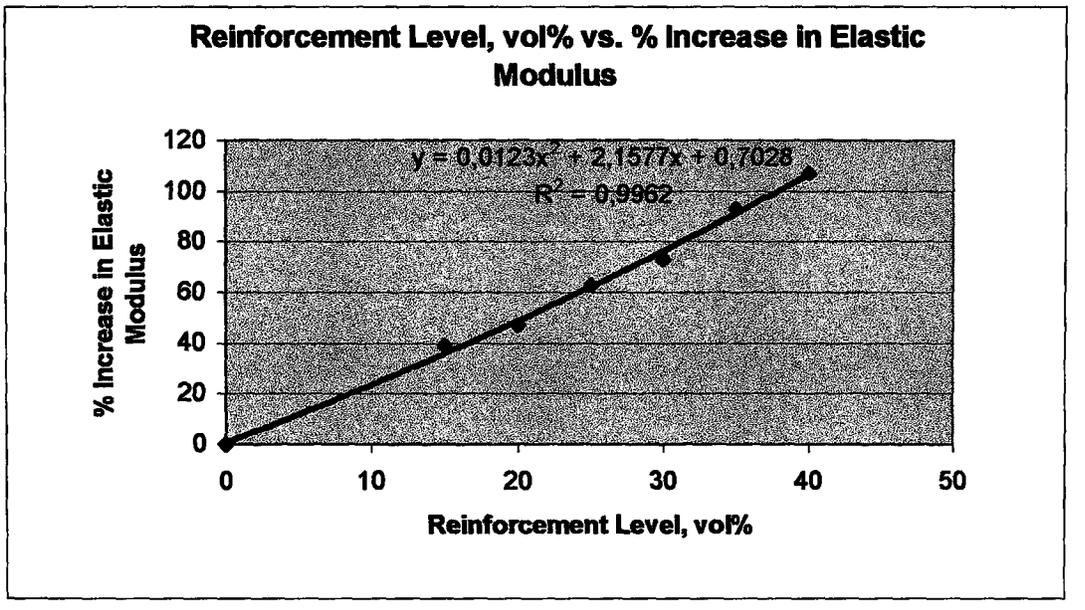


***Figure 2.2*** Reinforcement Level, Vol % vs. % Increase in Elastic Modulus

***Table 2.3*** The Effect on Elastic Modulus of 6061 Al Alloy Reinforced with SiC, [14]

| Reinforcement Level , vol% | % Increase in Elastic Modulus |
|---|---|
| 0 | 0 |
| 15 | 39 |
| 20 | 47 |
| 25 | 63 |
| 30 | 73 |
| 35 | 93 |
| 40 | 107 |

The heavily structure dependent properties such as ductility, fracture toughness, formability, and machinability tend to decrease with increasing levels of reinforcement. Among these, strength is more complex. Most of the alloys are strengthened by the reinforcement. Increasing levels of reinforcement give greater strength up to the point is reached where the material lack adequate

ductility and fractures at an early stage in the deformation process. Above this point, the strength achieved reduces with increasing level of reinforcement. [1]

#### 2.3.1.3 Reinforcement Size

Clearly strength increases with reducing the size of reinforcement particle. This is because of dislocation density and matrix grain size. Refinement of the reinforcement increases the dislocation density and gives finer matrix grain size following heat treatment. There are some other factors that seem to be enhanced by reduction in reinforcement size such as the ability to perform a secondary process on MMC (rolling, extrusion or forging) and the machinability. [14]

#### 2.3.1.4 Reinforcement Distribution

The major influence of reinforcement distribution is on the ductility and the fracture toughness of the MMC and hence indirectly on the strength. [15]

### 2.4 Production Steps in PM MMCs

#### 2.4.1 Mixing and Blending

Mixing and blending are the most common pre-compaction steps. There are several powder blends in use in PM. Powders can be blended to achieve control over the particle size distribution. Powders can be mixed to form new compositions. Either pre-alloyed or elemental powders are used for these new compositions. Because of alloying and increased work hardening rate, pre-alloyed powders are more difficult to press than elemental powders.

### 2.4.2 Sintering

Sintering is the mechanism by which solid bodies are bonded by atomic forces through the application of pressure and/or heat. In its larger sense it includes welding, brazing, soldering, seizing, or sticking of metals. Even at room temperature sintering is possible without application of pressure.

There are five different methods for sintering:

- Sintering with pressure alone, equivalent to moulding of powders
- Sintering with heat alone called thermal sintering or moulding without pressure
- Sintering with simultaneous application of pressure and heat
- Sintering of compacted powders with or without the production of a liquid phase

In the technical sintering of metal powders, several different processes can be distinguished:

- Sintering of homogeneous powders, ie, monometallic powders, solid solutions, intermetallic compounds
- Sintering of heterogeneous powders, i.e., poly-metallic or composite powders without formation of a liquid phase
- Sintering of powders with formation of a liquid phase
- Sintering of powders followed by impregnation with a liquid metal in which the powders may be in the loosely heaped condition or compressed into products

In the sintering process, time and temperature act in the same direction. The longer the time of heating, the better the mechanical coherence of a compact for a given temperature, the better coherence for a given period of heating is.

The most apparent difference between the microstructure of a metal made from powders and the one obtained by the fusion method is the presence of pores in the sintered structure. Porosity may constitute an obstacle in the development of satisfactory physical and chemical properties. Variation in the forming pressure affects the density of powder packing. The resulting porosity, which may be designated as "primary", is largely due to incomplete compaction. This kind of porosity can be controlled to a certain extend by changing certain conditions such as type and particle size of powder, compacting pressure, or shape of powder. In the case of reactivity of the atmosphere during sintering, or when sintering in vacuum, a new type of porosity may be created by the spontaneous evolution of the gases. The spherical shape of the pores distinguishes this porosity, which may be designated as "secondary". **[16]**

### 2.4.3 Hot Consolidation

Consolidation in powder metallurgy refers to the production of coherent metal structures using metal powders as the major raw material. Once suitable powders have been made, consolidation becomes the second vital step in the development of controlled microstructure materials vital to modern engineering and technology.

Hot consolidation of metal powders is the operation in which the step of applying pressure to the powder occurs at elevated temperature and is combined with the sintering step. It is expected that an operation in which pressure application and sintering are combined in one step should have economic advantages over two step processing, in which the powder is first cold pressed and then sintered.

The technology of metal powder consolidation includes two basic bodies of theory. These may be drawn upon to solve problems in existing systems. The critical concepts are vital to extend technology and thus create new systems.

The first body of theory concerns the mechanics of metal powder aggregates and is dealt with mechanical fundamentals of consolidation. This deals with the stage of consolidation in which metal powders are converted from more or less free-flowing granular particles into the green or unsintered state, having some of the characteristics of a rigid solid.

The second body of consolidation theory is concerned with the further conversion of metal powder aggregates from the weak green condition into coherent structural materials through the process of sintering. Thi is dealt with physical fundamentals of consolidation which explains the mechanism of sintering on dimensional changes and mechanical and physical properties of powder compacts.

For industrial, scientific, and economis reasons, the attainment of the desired final shape accompanied by adequate material properties is of great importance in the consolidation process. As is true in other industrial evolution, shape attainment in powder metallurgy is the result of historical interaction among scientific reasoning, engineering principles, and craft knowlegde, this subject is called shape fundamentals.

The production presses and tooling extends the shape principles of rigid tool compaction to the press requirements to provide the complex force-motion cycles necessary to densify powder metal parts into the green state. The practical requirements for sintering and development of full sintered properties begin with the technology of gas-metal thermochemistry in providing suitable protective atmospheres in which sintering can proceed. Once a protective atmosphere requirement is recognized and understood, the time-temparature cycle must be provided precisely by the furnace design.

Hot consolidation encompasses many diverse operations, includind uniaxial hot pressing and pressure sintering, hot isostatic pressing, ***Figure 2.3***, hot extrusion and hot forging.

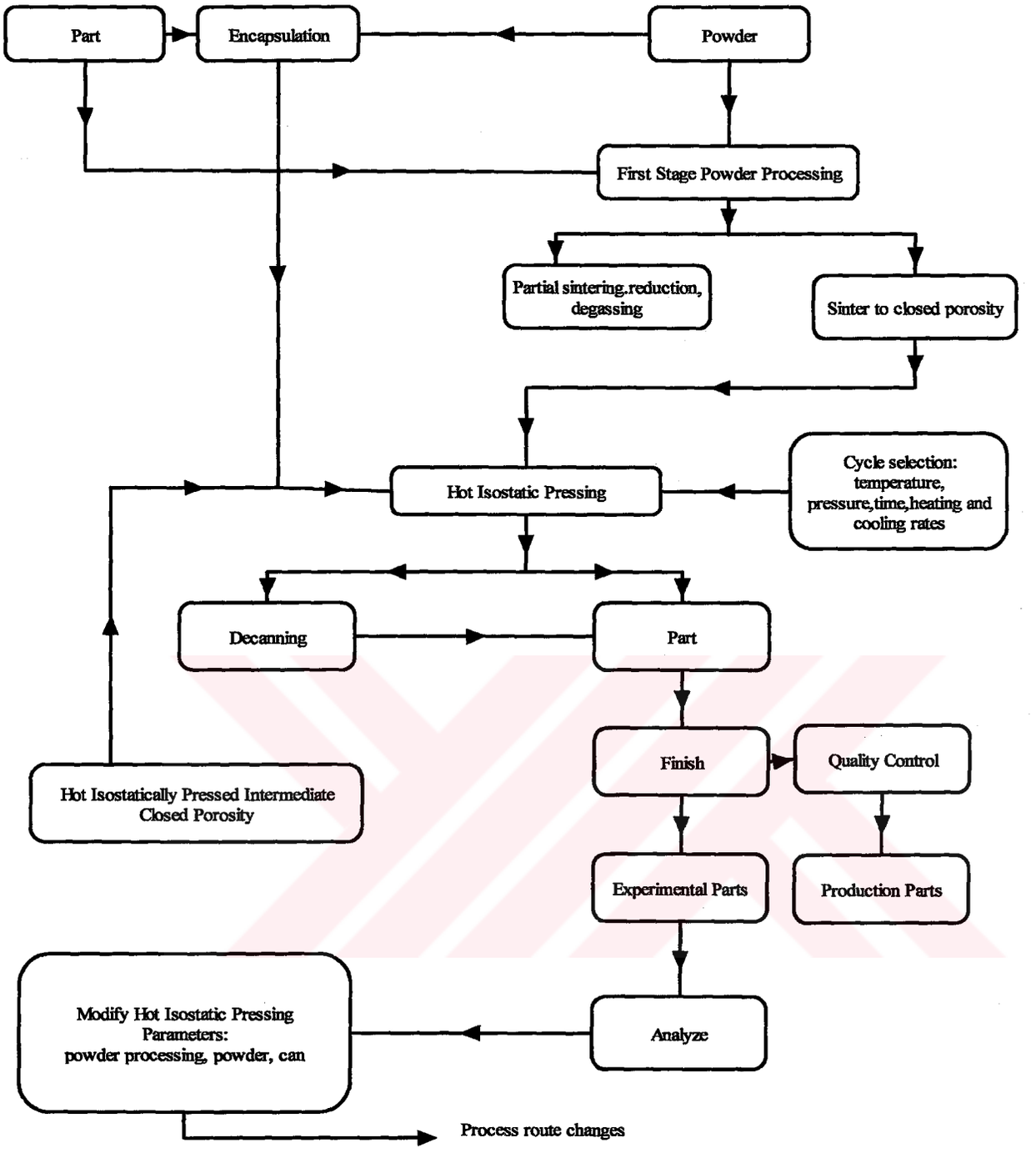


***Figure 2.3*** Flowchart for PM Manufacture of Metal Parts By HIP'ing

Some methods provide an uncontrolled atmosphere around the powder, such as that created by reaction of mold material with air, while other methods provide carefully controlled atmospheres or a vacuum. Selection of processing method is primarily determined by the physical properties of the material to be processed and by the composition of the hot pressing system, particularly the interaction of the work piece and tools.

### 2.4.4 Hot Pressing

Fully dense metal powder compacts with controlled microstructures can be produced by hot consolidation, in which pressure and heat are applied simultaneously rather than sequentially, as in conventional PM processing. Pressure is applied statically or dynamically to the heated powder in one or two opposing directions along a single axis or from all sides. A controlled atmosphere is required to protect the hot powders or pressed compacts from oxidation or nitridation by air.

Hot pressing of metal powder in closed dies is one of the oldest methods of PM hot consolidation. Altough hot pressing produces high quality products, it has not proved economically fesible for steel, brass, and other common metals.Disadvantages include; low production rates, hot die loadind, welding of the compact to the metallic die walls, tool wear, need for protective atmosphere. However, uniaxial hot pressing is used to fabricate cylindirical cemented carbide dies, rolls and wear parts. This method is also used to fabricate beryllium and silicon nitride, but hot isostatic pressing has increased usage for these materials as well as for PM superalloys, tools steels and etc. Use of hot isostatic pressing thecniques eliminates some of the problems caused by interaction of the powder compact and the die. Pressure can be applied more uniformly, thus producing a product with more homogeneous microstructure and properties. However, HIP is more expensive and needs proper equipment so it is important to develop alternative processing methods.



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There exist some factors that cause trouble in hot pressing; the wear of the die material, the tendency of compact surface particles to weld on die walls and punch facings and the tendency of very plastic metals to extrude into the clearance space between the die walls and the moving punches.

For hot pressing work, it is advisable to fit the punches into the die as closely as possible. Since the compaction cycle is slower than in cold pressing, particle consideration need to be given for the possibility of escaping of trapping gases. In many cases where clearance between punches and dies cannot easily be held to desired minimum, the dies that held together in a water-cooled wise may solve the problem. [17]

Apart from the fact that hot pressing improves the compactibility of a powder, another point of view may be taken into account. Basically, while moulding the particles into a definite shape, pressure has an important effect on making this shape coherent by creating strong particle bonds. On the other hand, temperature has an equally important effect on consolidating and homogenizing the structure by allowing for a sufficient atomic mobility and a place for interchange to transform the agglomerated particles into a recrystallised, uniform grain structure. In order to obtain desired results, the question of whether temperature and pressure are applied subsequently or simultaneously has to be answered.

#### 2.4.4.1 Process Principles

Altough the terms "pressure sintering" and "hot pressing" are used interchangeably, there exist a distinct difference between the two processes. In pressure sintering, the emphasis is on thermal processing; in hot pressing, applied pressure is the main process variable.

During hot pressing, a mold provides the compact with its desired shape. Press tools and machinary are similar to those used in small scale or special PM

operations. Facilities for appliying the requried heat usually can be adapted to the design futures of the press and the die.

A variety of techniques may be employed for hot pressing. They differ primarily in the methods used to bring a powder or prepressed compact in a desired temperature and in the material used for press tools.

All hot pressing techniques have several common features. Apparatus and operations to consolidate powders and remove end products do not differ substantially from conventional cold pressing and sintering operations. Processing cycle varies greatly with the time required for the plastic deformation and creep under the particular stress and temperature conditions employed. Generally hot pressing requries:

- Machines that can provide the requried pressure for powder consolidation. Pressures should be capable of slow action in one or two directions and should provide ejection ram pressure and movement. Die and punch constructions shoul posses sufficient strengths and shape resentation capabilities under maximum powder consolidation stresses at hot pressind temparature.
- Loading facilities for loose powder or previously fabricated porous preforms are required for hot pressing opeartions. These components may be separate or an integral part of the die.
- Knock-out provisions and cooling receptacles or quench tanks are needed to hold the hot pressed product.

Additionally, mechanisms for monitoring and accuratelly controlling the temperature of the compact throughout the pressing cycle must be provided. Heat must be supplied either directly to the preform or compact or indirectly to the die or punches for transmission to the powder or compact. Type of power source depends on the mode of heating selected. A controlled atmosphere shoul be available throughout the entire hot pressing cycle.

### 2.4.4.2 Process Variables

The quality of the hot pressed compact is determined by the interrelationship of three process variables: pressure, tempearure and time. These variables can have a major effect on the microstructure, physical properties, dimensional accuracy and the surface conditions of the product. However, optimum results require an understanding of these variables, which contribute to the consolidation of the compact by particle rearrangement and plastic flow.

For powders heated to the low temperatures, pressure has the same effect as in cold pressing. Particles are brought closer together, and they may be rotated and pushed into openings, deformed, sheared, or fractured. At elevated temperatures, plastic deformation becomes the dominant mechanism. If a liquid phase is formed at the hot pressing temperature, consolidation is further enhanced by the isostatic action of compressive stress on the compact inside the dies. Additionally, diffusion rates are increased by the liquid phase, and densification is enhanced by good wetting between liquid and solid components of an alloy system.

The magnitude of the pressure used in hot pressing should be based on properties of the powder substance, such as hardness at ambient and elevated temperatures, particle size and surface conditiions, possible oxide films, capacity for plastic deformation at various temperatures, and the potential for liquid phase formation. When a liquid phase appears, pressure must be kept low to avoid squeezing out the liquid, which results in changes in the composition and produces porosity in the hot pressed body.

Because, most solid metals and metalloidal compounds soften and become more plastic with increased temperature, a corresponding decrease in specific pressure is required for consolidation at higher temperatures. For practical reasons, hot pressing temperatures should be sufficiently high to achieve complete densification with moderate pressures in a reasonably short

time. Under these conditions, metals that do not differ appreciably in microstructure or mechanical properties from fine-grained precision castings or forgings can be produced.

If the hot pressing temperature is kept below the temperature range for re-crystallisation and grain growth, pressures for complete densification must be much higher. Plastic behaviour of the material to be processed is affected by processing time as well as temperature and force. For practical reasons, the time during which the hot compact is maintained under pressure should be kept at minimum. However, short-processing times also may produce fully dense compacts. **[22]**

### 2.4.4.3 Process Equipment

Presses: Full densification of a powder compact is achieved by plastic deformation, creep, and diffusion; consequently, time is an important processing factor. Whether consolidation pressure is applied during heating or after the selected hot pressing temperature has been reached, time must be allowed for heat transfer and equilibrium to occur. Because production rates are low hydraulic or pneumatic presses are more suitable than mechanical presses.

Selecting of press type depends on the design of the hot pressing tooling. Presses range in size from a simple hydraulic jack built inside a heavy steel frame to heavy double action hydraulic presses that are capable of providing hydraulic or pneumatic side-ram action for lateral compaction or ejection.

Dies and punches: Selecting a suitable material for these press tools is one of the most critical aspects of hot pressing. Maintaining the physical integrity of dies and punches during the high temperature interval of the hot pressing cycle is essential. Punches must have adequate compressive strength to prevent plastic deformation and welding to die cavity wall.

If the compact is completely solid, only a fraction of this pressure is transmitted to the die wall, even when full densification is achieved. If the material to be processed develops a liquid phase at the hot pressing temperature, plastic deformation and creep of the die may occur, resulting in ejection difficulties and distortion. Consequently, dies must be designed for sufficient wall thickness to withstand the high hoop stresses reached under peak temperature and pressure loading.

Generally, metal dies and punches tend to plastically deform unless processing temperatures are kept low by water-cooling. Tools made of ceramics or graphite is subjected to brittleness, and frequently catastrophic failure occurs if overloaded.

Chemical reactivity of tool parts subjected to high temperatures is another important consideration. In graphite tools, oxidation occurs when a controlled atmosphere is not provided. Reaction of the tools with air inside the die cavity is less detrimental.

If metallic tools are allowed to heat to the maximum temperature of the cycle, interactions with the powder may occur. The high compressive stress may cause the compact to diffusion bond to the punch faces or weld to the die cavity wall. The presence of a liquid phase in the compact aggravates this effect. Sometimes, punches and compacts become inseparable and have to be removed from the hot die in one piece. Welded or bonded spots may only be broken with great force, if at all, and may cause irreparable damage. Under less severe conditions, erosion and abrasion of the die walls intensify with repeated pressings, leading to a loss of dimensional control of the compact as well as to progressively higher ejection force.

Die wall lubricants, such as colloidal graphite suspensions in oil or water, that are applied to the metallic die cavity surface to reduce wear.

Charging: Feeding of loose powder is only possible if the die is cold. Once the die becomes hot, powder particles heat up rapidly on entry to the die cavity.

Ejection: Ejection of the completed hot pressed component is a delicate operation. Stripping the compact from adjacent punches may not be feasible within the die assembly and may require delay until cooling to room temperature.

Heating: Bringing the powder compact to the specified temperature before or during pressing is the primary function of the hot pressing-process. This operation differs from conventional PM heating procedures. To achieve acceptable results, the hot pressing system should provide a means of directing or transferring heat to the material to be pressed, allow normal equilibrium conditions and monitor and control the temperature of the compact. To achieve these requirements, the powder compact can be heated directly, or the tools may be heated first, thus transferring heat to the compact.

Resistance heating of the powder is accomplished by passing a low-voltage, high-amperage current through the powder while simultaneously applying the pressure.

Atmospheric Environment: Because most metal and metal alloy powders react strongly with oxygen a controlled environment must surround the material at elevated temperatures. Depending on the powder to be consolidated and the hot pressing set up, the atmosphere may be a gas contained in the die cavity, entrance throat or vestibule and cooling receptacle. When then powder to be hot pressed is a metal or a compound that is subject to contamination by interstitial elements, use of vacuum offers the best protection. **[22]**

### 2.4.4.4 Advances in Hot Pressing

Hot pressing provides various advantages.

- Microstructural uniformity
- Fabrication of large multiple curvature shapes
- Considerable reduction in cost
- Development of non-oxide ceramics for high performance applications

All alloys or powder mixtures that can be sintered may be produced by this method, eliminating many limitations peculiar to cold pressing. Materials whose physical properties and structural quality exceed that of obtained by conventional cold pressing can be developed.

Hot pressing, generally, substitutes for sintering, and handles the losses due to the breakage of the green compacts. Hot pressing provides closer size control, since the volumetric changes caused by the shrinkage forces or by gas evolution occur in the hot pressing die.

Present knowledge show that physical characteristics obtained by the use of hot pressing are better. And in many cases they are comparable those of wrought materials. Because high densities are attained, surface treatments, plating or hardening are suitable for hot-pressed parts. Even complicated parts with re-entrant angles may be hot pressed if the alloy is sufficiently plastic and contains a liquid constituent. It can be said that hot pressing is less sensitive to the powders origin and particle characteristics. Moreover, hot pressing offers the possibility of utilizing the advantages of pre-alloyed powders, which, on account of their extreme hardness, exhibit poor compactibility at room temperature.

If sufficient time is allowed for plastic flow, there is little variation in density or hardness of hot-pressed pieces from one section to another.



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During production of aluminium powder compacts by hot consolidation, the hot pressing step is usually proceeded by cold pressing of the powder and followed by a hard working step, such as extrusion or forging. Aluminium alloy powder compacts can readily hot pressed in dies made of hot die steel, too. **[17]**

### 2.4.5 Degassing

For high performance aluminium alloys, produced by PM route, degassing of the powder prior to complete densification is critical to the final performance of the alloy. The presence of an aluminum oxide surface layer on the powder particle is prone to hydration. The mechanical properties of powder metallurgical aluminum alloys can be improved by uniformly distributing the aluminum oxide throughout the matrix. This successful distribution depends on the efficient removal of water from the hydrated oxide.

Oxidation of aluminum alloys first takes place during atomization and subsequently during storage. The oxidation takes place at room temperature even under a low partial oxygen pressure $P0_2 = 10^{-16}$bar. **[18]**

Generally, surface oxides are detrimental to the mechanical properties of the consolidated product, if they are not uniformly distributed throughout the matrix. This can be prevented by reducing the oxygen level to minimize the amount of oxides or hydrates and by properly processing the alloy powders. Essentially, oxidation in aluminum alloy powders is unavoidable. However, elimination of physically adsorbed and chemically bounded water is possible.

Powders should be degassed at temperatures equal or higher than the consolidation temperature; otherwise evolution of adsorbed gases/vapors from the powder particle surfaces during consolidation results in porosity or blistering.

Generally evacuating the container holding the loose powder to a pressure below 10-5 torr while the powder mass is heated degasses powders. [19] Although, high temperatures tend to degrade strength, a temperature of 450 $^0$C has been shown to be adequate to promote water vapor removal from the powder. Degassing of the powder is of the concern because of the effect of gas and water vapor on properties and economics because of safety hazards due to possible hydrogen gas evolution at high temperatures.

Various degassing techniques have been investigated. Soviet workers also examined degassing aluminum powders at high temperatures in a dry argon atmosphere. On exposure to air, re-hydration did not occur for 5 days. This result suggest that Al powder can be degassed in atmospheres of argon in an unconfined, open tray and then transferred to compaction apparatus without re-hydration upon exposure to air; removing a small volume of argon is easier than removing hydrogen formed by re-hydration. Such handling is simpler than containerization practices. Properties of materials degassed in this manner do not have as high mechanical properties as material subjected to vacuum degassing. [20]

## 2.5 Theoretical Aspects of Hot Pressing

During hot pressing both creep and sintering are diffusional processes. This similarity is most evident in dealing with pressure-assisted densification. Uniaxial hot pressing resembles die compaction with both an upper and lower punch. The rate of densification due to external stress can be estimated in terms of a surface energy enhanced driving force. The density of a hot pressed material will be enhanced by higher external stresses, longer times, higher temperatures and finer grain sizes. The action of an external stress is to promote grain and particle sliding by diffusional processes, to generate excess vacancies, and to cause pore collapse. The overt effect is a more rapid densification. [20]

The three concerns in discussing hot consolidation of powders are temperature, stress and strain. Full density processing is ineffective at temperatures below approximately one half of the absolute melting temperature. The effective stress is the stress at the interparticle contacts, which differs from the external stress because pores act as stress concentrators. Only at full density does the effective stress is equal to the applied stress. The strain rate is the third variable of the concern. A high strain rate allows less recovery and tends to promote less ductility, thus the fracture is more likely. Alternatively, a low strain rate provides for more plastic deformation of the compact with a higher final density. At high relative temperatures, with lower stresses, diffusional effects will dominate. The strain rate effect then becomes involved in the rate of work hardening and the dislocation recovery rate.

In the different stages of consolidation, there is a dependence on the stress and the grain size. A process controlled by diffusion has low stress sensitivity and is aided by fine grain size. Dislocation controlled densification has a high stress sensitivity and usually aided by a coarse grain size. In hot consolidation of powders, there are some factors that hinder densification. **[26]** These include a large grain size or a large particle size, low effective stresses, and low temperatures.

Diffusional creep is the dominant process in hot pressing to full density. Three mechanisms are recognized as being possible in final stage closure of isolated spherical pores. **[27]** Nabarro-Herring creep occurs when the vacancy flow is directed by the stress gradient between grain boundaries in tension and those in compression. The strain rate is given as,

$$de/dt = 13.3D_v\Omega\sigma/kTG^3 \quad \textit{(2.1)}$$

where de/dt is the strain rate, T is the absolute temperature, k is the Boltzmann's constant, $\Omega$ is the atomic volume, D is the diffusivity, G is the grain size and $\sigma$ is the effective stress. The rate of densification increases with the creep strain rate

and the porosity. ***In equation 2.1***, the diffusivity is highly sensitive to temperature; thus, provides an important controlling influence on the densification rate. An alternative process is Coble creep, where the diffusional flow is along grain boundaries. Again the stress directs the diffusional flow. For Coble creep directed densification the strain rate is given as;

$$de/dt = 47.5wD_b\Omega\sigma/kTG^3 \quad \textit{(2.2)}$$

where w is the grain boundary width, $D_b$ the boundary diffusivity. Finally, when both the stress and the temperature are high, the rate of densification depends on the diffusion rate for dislocation climb. The form introduced for this case is;

$$de/dt = (CubD_v/kT)(\sigma/U)^n \quad \textit{(2.3)}$$

where b is the burgers vector, C is a material constant, U is the shear modules, and n an exponent expressing the stress sensitivity. The form of ***equation 2.3*** is somewhat empirical but has been successful in explaining certain data. In the final stage of hot pressing, no particle sliding occurs. Consequently, formulae like ***equation 2.1*** through ***equation 2.3*** have been successful in explaining hot pressing behavior. Like cold pressing, hot consolidation will show a dependence on the amount of remaining porosity. High levels of porosity make the rate of densification higher because of a high effective stress. The rate of pore closure by diffusion combined with creep processes represented in ***equation 2.1*** through ***equation 2.3*** gives generalized densification equations such as;

$$d\rho/dt = (de/dt)A[\rho(1-\rho)/(1-(1--\rho)^{1/m})^n] \quad \textit{(2.4)}$$

where $\rho$ is the density (fractional), and A is a geometric constant. In most instances of hot pressing, several densification processes are simultaneously active. A linear combination of the rates of densification by the individual processes provides a first approximation to the total densification rate. The basic features of hot consolidation of metal powders can be combined into pressure

densification maps. **[27,28]** ***Figure 2.4*** gives two such maps for PM tool steel consolidated at either a constant temperature or constant pressure. The region of dominance of each mechanism is outlined in terms of density versus pressure and temperature. A lower initial compact density results in a higher effective stress. As density increases, further densification depends on diffusional processes. The position of the various regions depends on the material properties and the stress dependency as given in ***equations 2.1*** through ***2.4***. The pressure and temperature combination necessary to attain 99% density are shown in ***Figure 2.5*** for two particle sizes of tool steel. Densification is much more responsive to temperature changes then to pressure changes. Likewise, property variations are expected with the powder consolidation conditions. The data in ***Figure 2.6*** show that strength, ductility and toughness for a hot isostatically pressed material depend on the consolidation temperature. A low consolidation temperature results in incomplete densification and inferior properties. This example further demonstrates the sensitivity of dynamic properties. Hot pressing is a manifestation of stress enhanced densification of metal powders. **[20]**

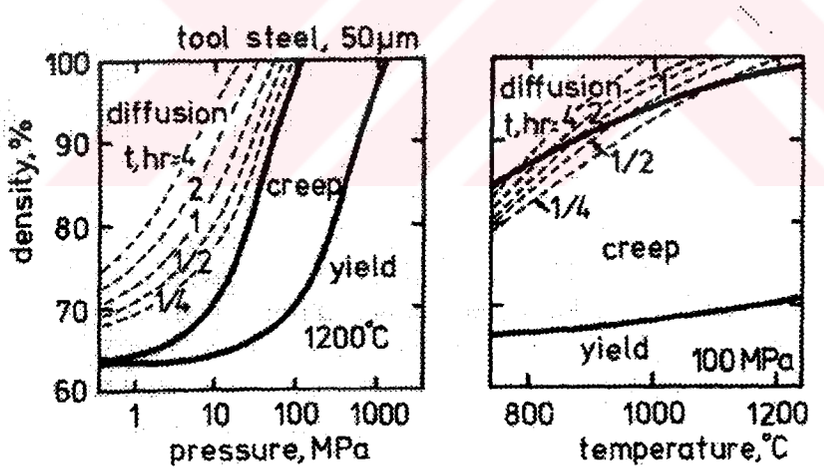


***Figure 2.4*** Densification Maps for The HIP'ing of a 50μm Tool Steel Powder, **[20]**

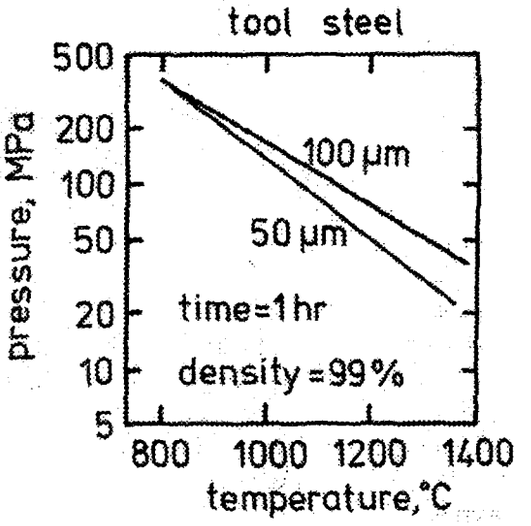


***Figure 2.5*** **The temperature and pressure combinations necessary to attain 99% density in one hour for two tool steel particle sizes, [20]**

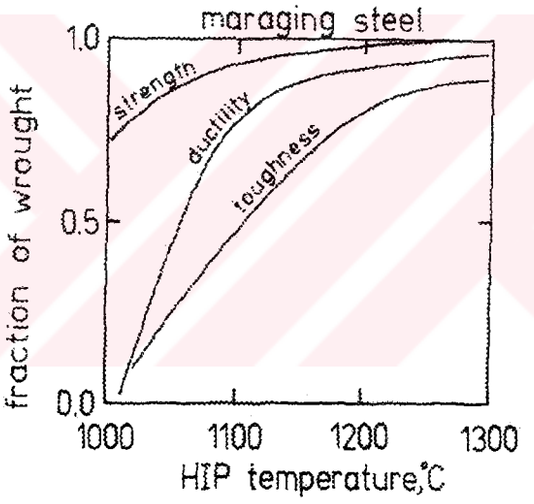


***Figure 2.6*** **Development of properties in a maraging steel by HIP'ing at 103 MPa and various temperatures, [20]**

Despite the obvious technological importance of the hot-pressing process, however, only a limited amount of fundamental information exists concerning the factors that affect and control the process. In contrast to conventional sintering, the added variable of pressure, as well as the pressure

temperature interaction, lead to process mechanisms which are potentially different and inherently more complex than pressureless sintering.

Early investigations suggested plastic flow as a mechanism of hot pressing [29]. Following that several investigators cited particle rearrangement by grain boundary sliding and particle fragmentation as important mechanism of hot pressing, particularly during early stages, as well as at very high pressures and / or lower temperatures. Later studies concluded that, hot pressing densification, beyond initial stages was essentially diffusion controlled, occurring by Nabarro-Herring diffusional creep. Also final stages of densification should occur by enhanced diffusion under the influence of stress.

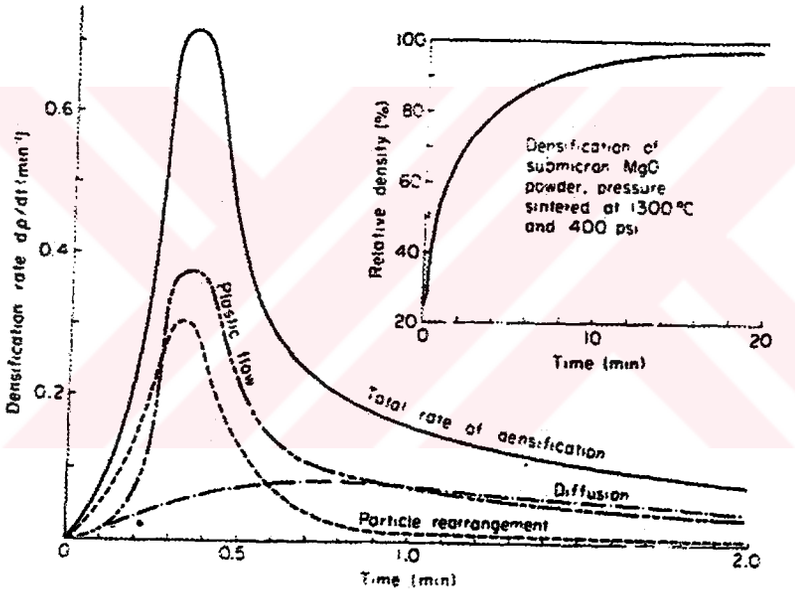


***Figure 2.7*** Hypothetical rate of densification vs. time curves for three densification mechanisms as well as total rate of densification, [28]

During those studies, defned hot pressing mechanisms was usually assigned to a somewhat definite interval in the densification sequence. However, it is now known that, all of the mechanisms contribute to some extent toward final densification during the entire hot pressing process. Any one meehanism



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may be a major contributor at a particular time, but is aided by the others, ***Figure 2.7.***

The process of densifieation is a continuous one, but the geometry changes are large, for that reason it is helpful to think of it as occurring in three sequential stages.

The first stage of powder packing is not well understood. The initial packing density depends on particle shape, on the size distribution, on the degree to which powder has been shaken down and etc. An applied pressure causes rearrangements, which increase the relative packing density. During stage 1 neck grow at the contact points between particles ***Figure 2.8.*** A number of mechanisms contribute to neck growth: plastic yielding and creep dominate when the pressure is large; when it is not densification involves various sorts of diffusion. **[19]**

The first stage (stage 0) describes the density reached by packing of the loose powder. The second (Stage l) describes the early phase of densification, while the porosity is still connected. The final stage (Stage 2) describes the final densification, when the residual porosity is in the form of small holes.

Final densification occurs in stage 2. By the time the relative density has reached 0.95, most of the necks have grown until they impinge and the individual pores have sealed off As in stage 1, a number of mechanisms contribute simultaneously to the shrinkage of pores.

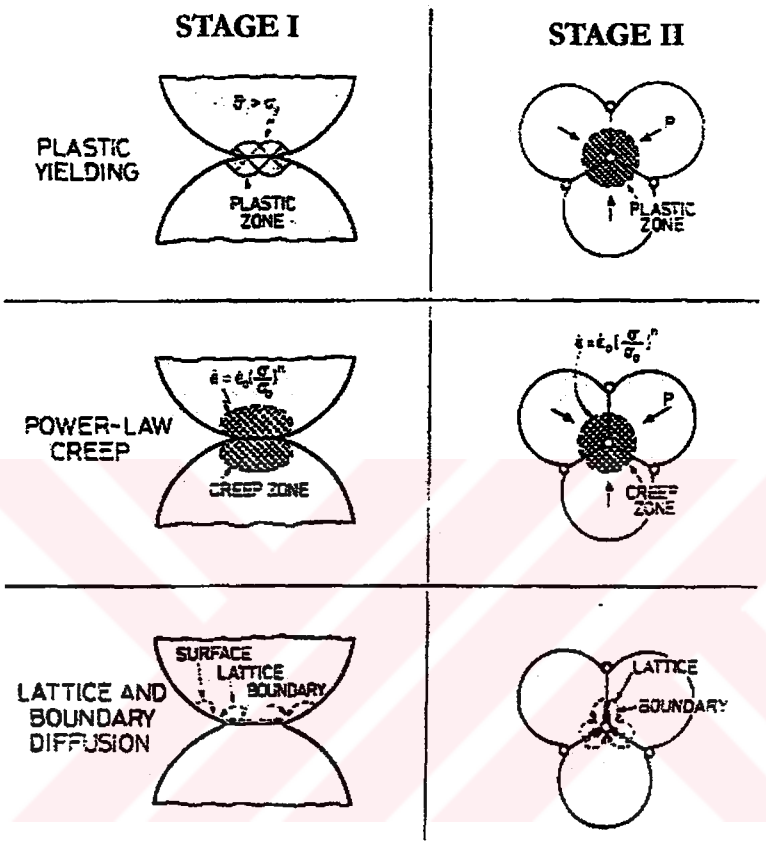


***Figure 2.8*** Stages of densification in hot pressing, [19]

# CHAPTER 3

# EXPERIMENTAL PROCEDURE

## 3.1 General

In this study, Al-SiC composites are produced by mixing pure aluminium, pure copper, pure magnesium and silicon carbide powders. The powder mixture is then hot pressed with a single-end die under nitrogen atmosphere.

Samples were characterised in terms of microstructure and flexural strength. The microstructural features are evaluated by using Scanning Electron Microscope, JEOL 6400.

A schematic diagram of the experimental procedure is given in ***Figure 3.1.***



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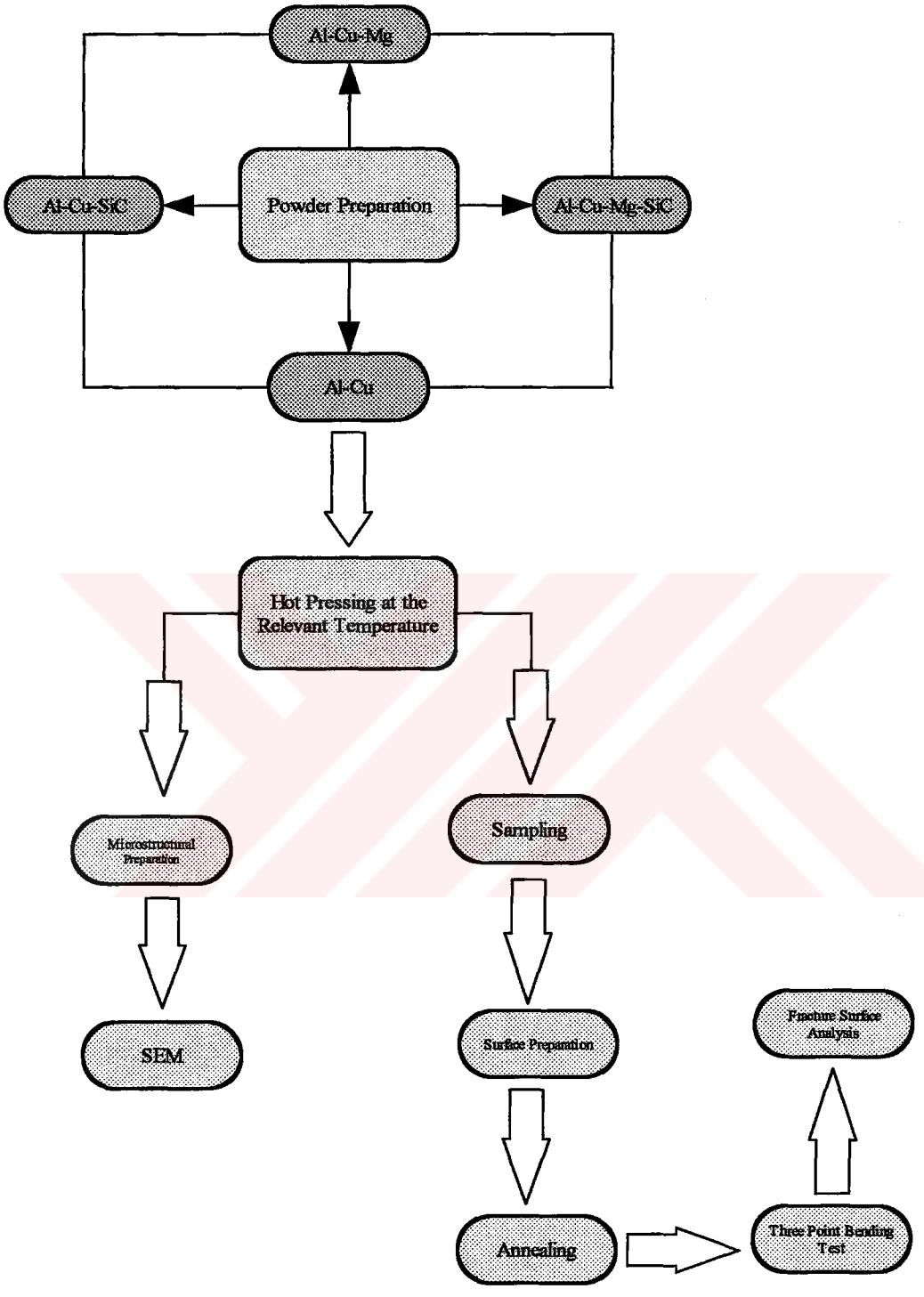



***Figure 3.1*** Experimental Procedure Flow-Chart

### 3.2 Details

#### 3.2.1 Raw Materials

During experiments commercial aluminium powder with an average particle size of 26μm is used. Copper powder is supplied from Merck with a size of ≦63μm and magnesium powder is supplied from Riedel Haen 131 12 (46 46 47) with a size of ≦100μm. SiC powder is commercial α-type with an average of 10μm size, (F-Grid 600).

#### 3.2.2 Mixing of Powders

The ingredients of each batch were weighed carefully, transferred into an agate mortar and pastle, and mixed there with isopropyl alcohol for about 5 minutes in order to blend powders thoroughly. Then, the mixtures are either cold compacted for quenching experiments or transferred to steel die for hot pressing.

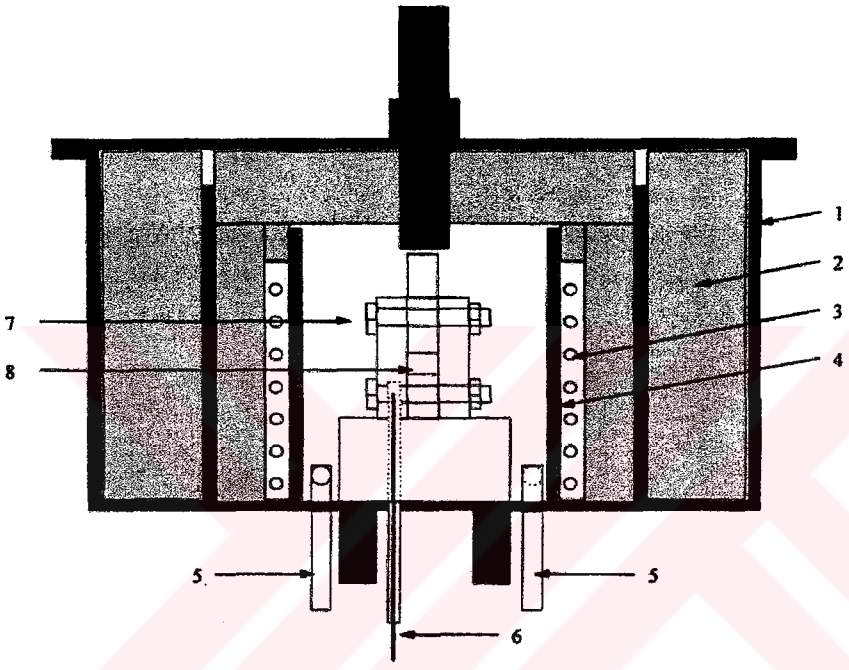
#### 3.2.3 Hot Pressing

After hand mixing, several powder mixtures were consolidated by hot pressing. It was similar to cold compaction using a single-end die but experiments are performed at an elevated temperature in the range 450°C to 600°C.

During heating the pressure was selected in the range 25-50 MPa with a manually controlled hydraulic press. Heating was performed by electrical resistance heaters. Furnace atmosphere was maintained by blowing nitrogen through inlets placed at the bottom of the hot press, (fig. 3.2). Temperature measurement was made by a K-type thermocouple, which was submerged inside the die wall. Die walls were isolated by fine $TiO_2$ powder in order to prevent sticking of the specimen into the walls. During hot pressing, the temperature of

the die was controlled within ±5°C. Specimen was held at the hot pressing temperature for 10 minutes then cooled down to 400°C under pressure and then cooled in air.

A schematic view of the hot press set up is given in ***Figure 3.2.*** It consists of a compaction die, heating elements surrounding the die and an isolation chamber for atmospheric control.




| | |
|---|---|
| 1. Isolation Chamber | 5. Nitrogen gas inlets |
| 2. Insulation Material | 6. Thermocouple |
| 3. Heating Elements | 7. Mould |
| 4. Ceramic Tube | 8. Specimen |

***Figure 3.2*** Schematic View of Hot Pressing Set-up

The hot press die manufactured from H13 type hot work tool steel. The die is designed to produce specimens having 30mm ×40mm × 10mm dimensions. The walls of the die were fixed with AISI 316 grade stainless steel nuts and bolts for easy removal of the die at the removing stage of the specimen.

The split die was also very helpful eliminate possible deformation of the specimen.

### 3.2.4 Cold Pressing

Several Al-Cu powder mixtures containing magnesium were also cold pressed. Cold pressed compacts were then quenched from relevant temperatures. For quenching experiments a vertical quenching furnace designed by ***Dr. Şakir Bor*** was used. A detailed description of quenched specimens can be seen in ***Table 3.1.***

### 3.2.5 Sample Descriptions

***Table 3.1*** gives a detailed description of the batches prepared in this study. The effect of magnesium on microstructural development was investigated at different temperatures. In order to analyse the microstructural properties properly several samples were cold compacted and then quenched from relevant temperatures. The effect of SiC on microstructural and mechanical properties was investigated by addition of 20vol%-60vol% SiC to Al-Cu and Al-Cu-Mg mixtures.

### 3.2.6 Metallographic Specimen Preparation

For microstructural characterisation under SEM, the samples were prepared by conventional metallographic methods. In order to reveal the microstructural details, either Keller's reagent (2 ml HF (48%), 3 ml HCl (concentrated), 20 ml $HNO_3$ (concentrated), 175 ml water) or 2% Nital was used. For SEM studies JEOL 6400 with Northern Tractor EDS analysis system was used.

*Table 3.1* Powder Mixtures Prepared for Microstructural Characterisation

| Composition | Sample Description | H.P Temp, °C | Quenching Temp, °C | Quenching Time, min |
|---|---|---|---|---|
| Al-5wt%Cu | As hot pressed | 600 | | |
| Al-5wt%Cu | As hot pressed | 550 | | |
| Al-5wt%Cu-15vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-20vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-30vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-40vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-60vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-5wt%Mg | As hot pressed | 430 | | |
| Al-5wt%Cu-5wt%Mg | As hot pressed | 430 | | |
| Al-5wt%Cu-5wt%Mg | As hot pressed | 615 | | |
| Al-5wt%Cu-5wt%Mg | Hot pressed & quenched | 430 | 500 | 5 |
| Al-5wt%Cu-5wt%Mg | Hot pressed & quenched | 430 | 550 | 5 |
| Al-5wt%Cu-5wt%Mg | Hot pressed & quenched | 430 | 550 | 15 |
| Al-5wt%Cu-5wt%Mg | Hot pressed & quenched | 430 | 600 | 5 |
| Al-5wt%Cu-5wt%Mg | Hot pressed & quenched | 430 | 600 | 15 |
| Al-5wt%Cu-5wt%Mg-4vol%SiC | As hot pressed | 615 | | |
| Al-5wt%Cu-5wt%Mg-40vol%SiC | As hot pressed | 580 | | |
| Al-5wt%Cu-5wt%Mg | Cold pressed & quenched | | 600 | 5 |
| Al-5wt%Cu-5wt%Mg | Cold pressed & quenched | | 600 | 15 |
| Al-5wt%Cu-5wt%Mg | Cold pressed & quenched | | 500 | 15 |
| Al-5wt%Cu-5wt%Mg | Cold pressed & quenched | | 550 | 5 |

### 3.2.7 Density Measurements

Density measurements of several Al-Cu-SiC composites were carried out. Dimension of the each specimen was measured by a micrometer and weighed. Then the amount of compaction was computed for each sample.

### 3.2.8 Three Point Bend Testing

In this part of the study, the aim was to obtain preliminary results of flexural bend strength. The precise procedures to be used in this testing are described in MPIF 15-62 and ASTM B312-64.

3-Point flexure test is based on the loading configuration shown in ***Figure 3.3***.This test method is used to determine flexure strength and modulus.

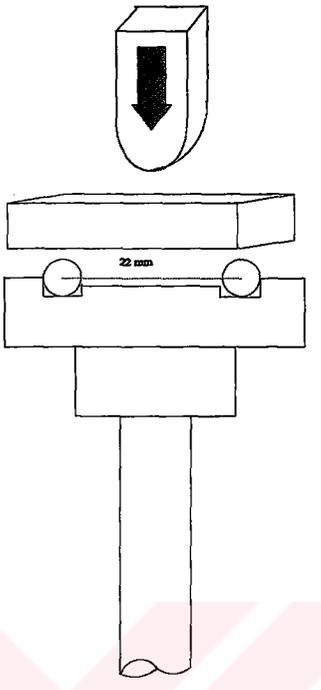



***Figure 3.3*** Schematic Drawing of 3 Point Bending Test Apparatus

Several specimens with different amounts of SiC were prepared to determine flexural strength and stiffness. Experimental results were compared to theoretical ones via rule of mixture of composites. Since Al-Cu alloys are known to be age hardenable, all the samples were annealed at $300^0$C for 10 minutes to eliminate a possible natural aging effect.

### 3.2.9 Fracture Surface Analysis

Besides microstructural characterisation fracture surface analysis were also carried out on Al-5wt%Cu-5wt%Mg-40%SiC specimens. The specimen was notched then fractured under impact loading. Fracture surfaces were observed under SEM. Fracture surfaces of Al-Cu-SiC specimens obtained via three point bending test were also investigated by SEM.

# CHAPTER 4

# EXPERIMENTAL RESULTS

In this study, the primary concern was to produce Al-SiC metal matrix composites comprising 40 vol% SiC. In a previous study **[10]**, Al-30vol%SiC composite could be produced by hot pressing method. Elemental copper powder was added to obtain Al-5wt%Cu matrix alloy and hot pressing was carried out in nitrogen atmosphere. However, it was not possible to increase the SiC content by this method, as the residual pores could not be eliminated during hot pressing **[30]**.

Therefore, the primary aim of the present study was to increase the SiC content to 40vol% by modifying the hot pressing method developed by **Kaya [30]**. The modification in this study includes:

- Addition of elemental Mg powder to the Al-5wt%Cu matrix alloy to improve the wetting properties of the liquid phase formed.
- Changing the hot pressing parameters of Al-5wt%Cu-40vol%SiC composites to eliminate or minimise the residual pores.

## 4.1 The Effect of Addition of Mg Powder on Al-Cu Powder Mixtures

Microstructural examination was carried out in two steps:

- Microstructural examination of Al-5wt%Cu system
- Microstructural examination of Al-5wt%Cu-5wt%Mg system

### 4.1.1 Aluminium-Copper System

Al-5wt%Cu system was examined for reference purposes. Previously, it has been shown that the addition of elemental Cu powder to Al powder aids in formation of a liquid phase and enhances densification during hot pressing. **[10]**

In Aluminium-copper system, first liquid appears at 548°C in the aluminium rich part of the phase diagram, ***Figure 4.1***. Presence of a liquid phase is very important in hot pressing because of two basic reasons. Firstly, it aids in elimination of pores. Secondly, higher rate of diffusion is achieved due to the presence of a liquid phase. As reported by Kaya **[30]**, the elemental copper powders are not eliminated when specimens are hot pressed below $550^0$C. Instead, an inter-metallic layer form around pure copper particles, which remain undissolved in the matrix. As a result, hot pressing of Al-5wt%Cu powder mixture could not be achieved, which can be related to the absence of a liquid phase and low diffusion rates. In view of these results, hot pressing temperatures were selected above 550°C **[10]**, which yielded pore free, sound specimens.

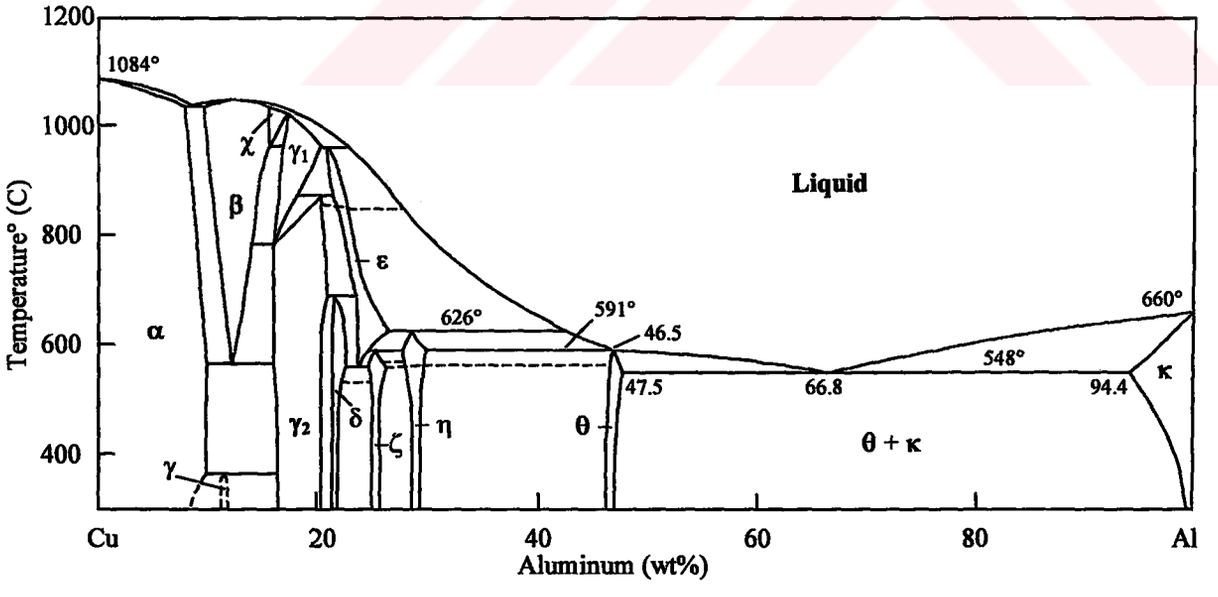



*Figure 4.1* Al-Cu Phase Diagram, **[38]**

The preliminary experiments of the present study were carried out in Al-5wy%Cu system. Microstructure of an Al-5%Cu sample that is hot pressed at 600°C is shown in figure 4.2. There is no residual porosity and grain boundaries of aluminium powder can be easily seen. Since the sample was cooled in the mould after hot pressing, in some regions $CuAl_2$ precipitates can be observed. In ***Figure 4.2***, $CuAl_2$ eutectics are seen as white contrasted precipitates along grain boundaries. In addition, hot pressing at this temperature caused small precipitates to form inside the grains as well. In conclusion, Al-5%Cu powder mixtures can be hot pressed by means of the liquid phase formed, under nitrogen atmosphere without porosity. **[10]**

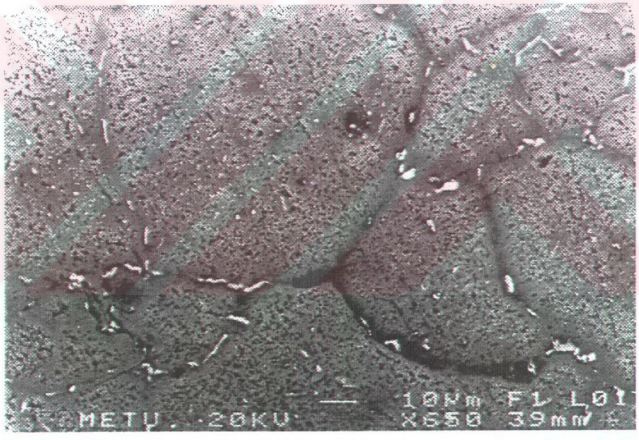



***Figure 4.2*** The SEM micrograph of the Al-5wt%Cu specimen after hot pressing at $600^0$

### 4.1.2 Aluminium-Copper-Magnesium System

#### 4.1.2.1 Hot Pressed Samples

At the second stage of the research, Al-5wt%Cu-5wt%Mg powder mixture was hot pressed in the liquid + solid region. The reason why magnesium is added to the system is to improve the wetting properties of the liquid phase, because Mg is known to have good surface-active properties.

When Al-Cu-Mg ternary phase diagram is investigated, it can be seen that the lowest temperature, where a liquid phase is present is 450$^0$C, ***Figure4.3***. Therefore, in order to observe the behaviour of the liquid phase, the hot pressing of Al-5wt%Cu-5wt%Mg powders are carried out in the range 430$^0$C – 615$^0$C.

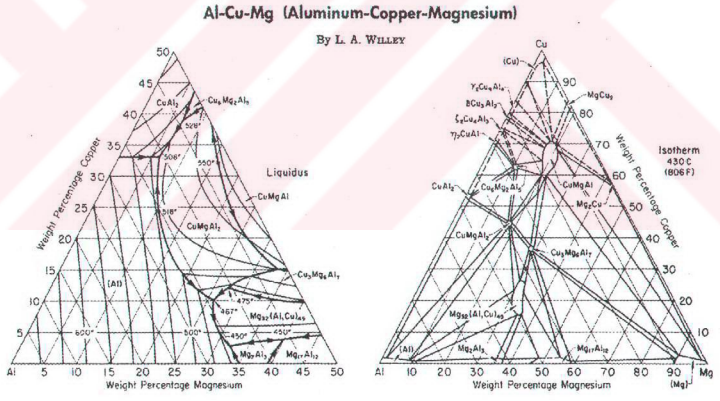



***Figure 4.3*** Al-Cu-Mg phase diagram & isotherm at 430$^0$C, **[38]**

Hot pressing of Al-Cu-Mg powder mixture at $430^0$ causes an appreciable amount of diffusion to take place between Al, Cu and Mg particles. In ***Figure 4.4***, a Cu powder particle can be seen in the Al matrix. After a hot pressing operation, the particle is not pure Cu anymore, but consists of several layers, due to diffusion between Al matrix and Cu powder particle. This appearance is typical for a solid-state diffusion. The layers around the copper powder most probably represent the different compositions of Al-Cu and/or Al-Cu-Mg intermetallics in Al-Cu-Mg system. However, no attempt was made to characterise these intermetallics, as it is beyond the scope of this study.

On the other hand, ***Figure 4.5*** shows the original site of a magnesium powder particle. The region with darker spots at the lower part of ***Figure 4.5*** is Mg powder particle site. As seen, the original shape of the Mg powder cannot be observed anymore, after a hot pressing operation at $430^0$C. It seems that diffusion of Mg in Al matrix is much faster with respect to that of Cu, even in solid state. Indeed, the spot analysis taken from either Al matrix or from precipitates in Al matrix indicated the presence of Mg element, after a hot pressing operation at $430^0$C.

Increasing the hot pressing temperature above solidus temperature ($450^0$C) complicated the microstructural development. The liquid formation sites in Al-5wt%Cu-5wt%Mg powder mixtures could not be determined. For this reason, quenching experiments were carried out in hot pressed samples. After a hot pressing operation at $430^0$C, several specimens were re-heated to a temperature range $500^0$C-$600^0$C and quenched in water.

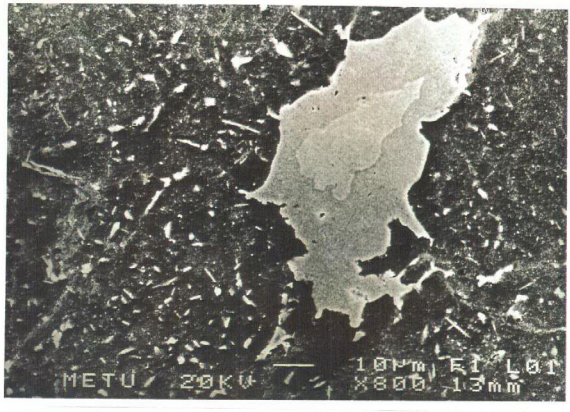



*Figure 4.4* The original site of a Cu particle in Al matrix, after hot pressing at $430^0$

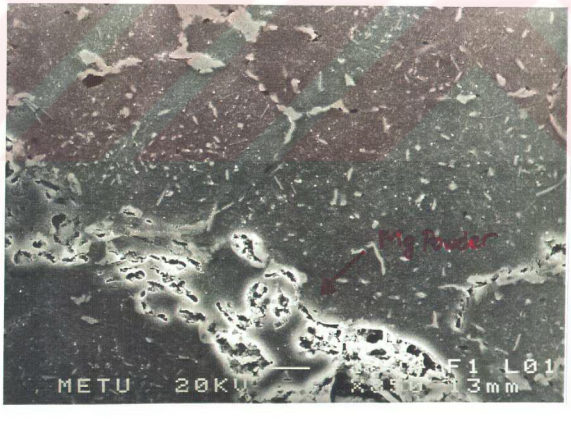



*Figure 4.5* The original site of a Mg particle in Al matrix, after hot pressing at $430^0$C



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First, a sample that is hot pressed at 430$^0$C was heated up to 600$^0$C then quenched to room temperature. During SEM studies it was observed that the sample contains pores, ***Figure 4.7***. This was an expected result since the solubility of Mg in Al is high. In ***Figure 4.6***, a porosity formed at the intersection of grain boundaries can be seen. However, more interesting to note that a continuous phase precipitation is observed at the original Al powder particle boundaries. A light contrasted phase in the form of a grain boundary network can be seen in ***Figures 4.6*** and ***4.7*** (shown as A). This is most probably a residue of a liquid phase, which is formed upon heating to 600$^0$C. It can be noted that complete wetting of the original Al powder particle boundaries takes place by the liquid phase at that temperature. The EDS spot analysis taken from the grain boundary network revealed that it is rich in Al, Cu, Mg. Considering the possible reflections from the matrix, it can be speculated that it is either a $CuAl_2$ or a $CuMgAl_2$ phase.

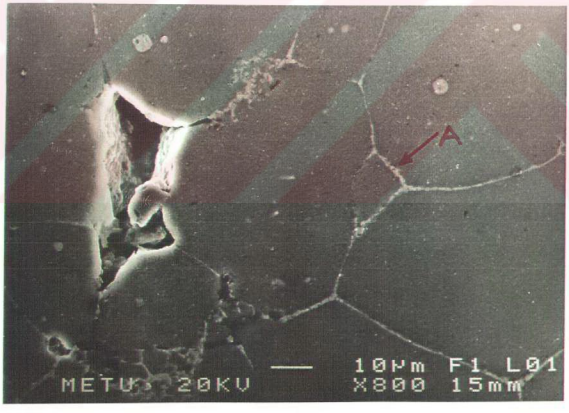



***Figure 4.6*** Pores formed in hot pressed (430$^0$C) Al-5wt%Cu-5wt%Mg specimen from after quenched 600$^0$C. The grain boundary phase is shown as A.

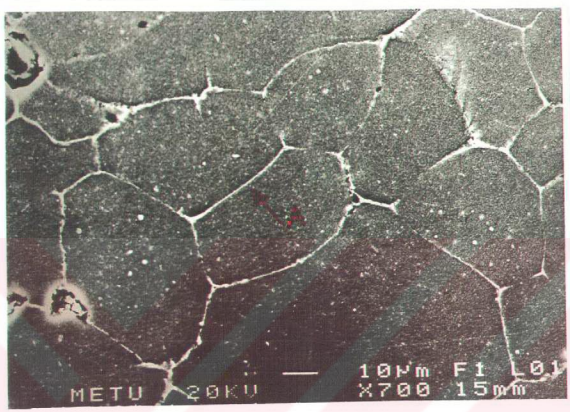



*Figure 4.7* The continuous grain boundary network phase in hot pressed (430) Al-5wt%Cu-5wt%Mg specimen after quenched from $600^0$C

As a second step, the hot pressed samples are quenched from a lower temperature, i.e., $550^0$C. Despite not clear enough, the grain boundary network was still evident, ***Figure 4.8***.

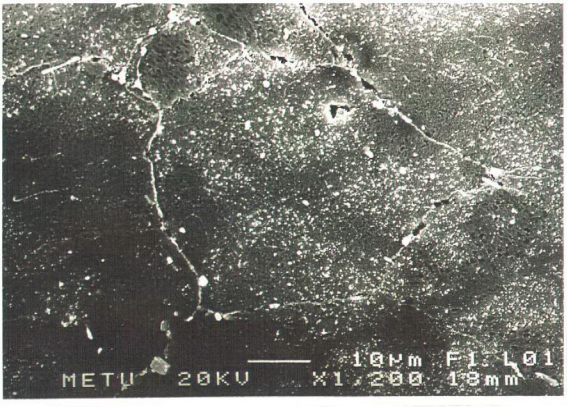



*Figure 4.8* The continuous grain boundary network phase in hot pressed (430$^0$C) Al-5wt%Cu-5wt%Mg specimen after quenched from 550$^0$C

Therefore, it can be concluded that when the solutionizing temperature were increased up to 600$^0$C, the undissolved copper particles are disappeared. Instead, a liquid phase is seen to spread along the grain boundaries.

### 4.1.2.2 Quenching Experiments on Cold Pressed Al-Cu-Mg Powder Mixtures

So as to understand the behaviour of Mg in our samples properly, some of them were not hot pressed. Instead, several Al-5wt%Cu-5wt%Mg powder mixtures were cold compacted at room temperature. Then quenching experiments were carried out on these samples in order to not disturb the liquid phase formation sites by an additional hot pressing step, ***Table 3.1***.

Quenching experiments were done at different temperature and time combinations. ***Figure 4.9*** shows the microstructure of a cold pressed sample, which was solutionized at 500$^0$C about 15 minutes then quenched into water. As can be seen, light contrasted areas are present in the quenched sample. The EDS

analysis studies have indicated that the light contrasted areas are rich in Cu and Mg. On the other hand, the tree like precipitates are rich in Al. Therefore, it can be depicted that the original site is transformed to liquid phase during heating stage into which a substantial amount of Mg is diffused.

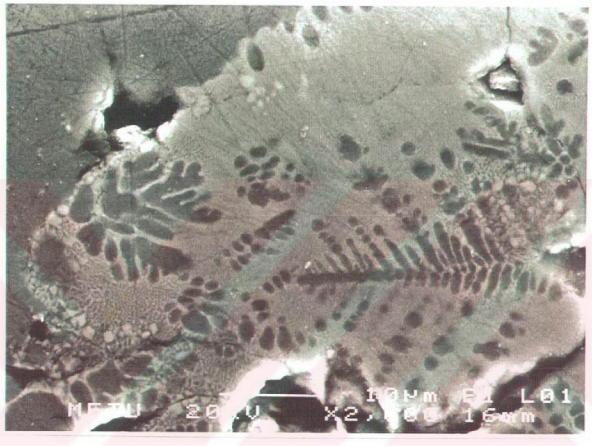
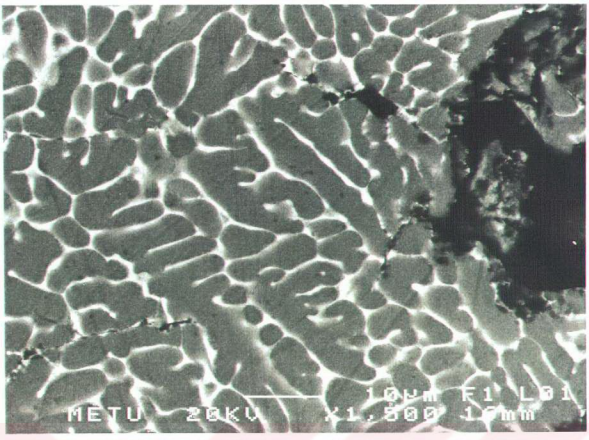
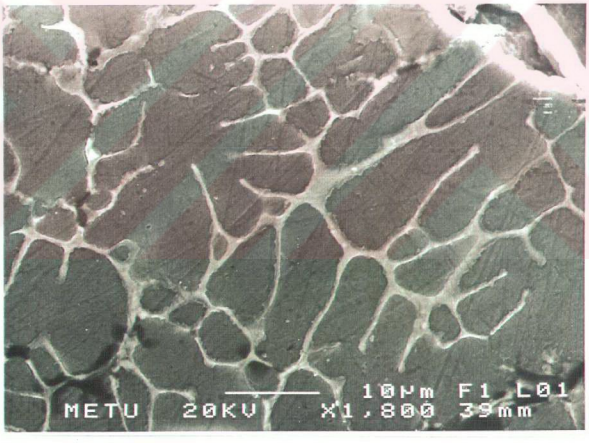


***Figure 4.9*** The SEM micrograph of the Al-5wt%Cu-5wt%Mg powder mixture. The light appearing region was formerly a Cu particle.

A further increase in temperature to $600^0$C has revealed that the light contrasted phase, which is rich in Cu and Mg spreads along the grain boundaries, ***Figures 4.10a*** and ***4.10b***.




*(a)*




*(b)*

***Figure 4.10 a &b*** The spread of liquid phase along prior Al particle boundaries after quenching from 600$^0$C (cold compacted Al-5wt%Cu-%wt%Mg)

EDS point analysis has indicated that the light contrasted phase at the grain boundaries has a very similar composition with respect to the light contrasted specimen quenched from $500^0$C. ***Figures 4.10a, 4.10b*** and ***4.11*** show the grain boundary phase at different details. It can be dictated that this phase is a residue from the liquid phase present at $600^0$C.

***In Figure 4.11***, a micrograph of the same sample taken in back scattered electron mode of SEM can be seen.

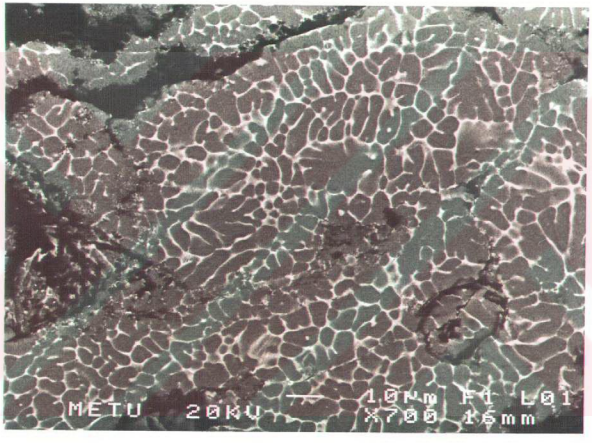



***Figure 4.11*** The BSE image of the grain boundary network phase in Al-5wt%Cu-5wt%Mg sample after quenched from $600^0$C

### 4.1.3 Hot Pressing Studies on Al-Cu-SiC System

On contrary to samples containing magnesium, pores were never observed in Al-Cu matrix alloy. Hence, experiments were concentrated on Al-5wt%Cu-40vol%SiC mixtures. By changing hot pressing parameters, several experiments were carried out. The details of the hot pressing studies on Al-5wt%Cu-SiC mixtures can be seen in Table 4.1.

***Table 4.1*** The details of hot pressing parameters applied to Al-Cu-SiC powder mixtures

| Composition | H.P Temp, $^0$C | H.P rate, $^0$C/min. | Powder size, μm Al | Powder size, μm SiC | Pressure, MPa | (*) Result of H.P. |
|---|---|---|---|---|---|---|
| Al-5wt%Cu-15%SiC | 590 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-15%SiC | 590 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-15%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-15%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-20%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-20%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-30%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-30%SiC | 615 | 15 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-40%SiC | 590 | 3 | 26 | 10 | 50 | × |
| Al-5wt%Cu-40%SiC | 590 | 3 | 26 | 10 | 50 | × |
| Al-5wt%Cu-40%SiC | 590 | 3 | 26 | 40 | 25 | × |
| Al-5wt%Cu-40%SiC | 615 | 3 | 100 | 40 | 50 | × |
| Al-5wt%Cu-40%SiC | 615 | 3 | 26 | 5 | 50 | × |
| Al-5wt%Cu-40%SiC | 615 | 3 | 26 | 10 | 50 | ✓ |
| Al-5wt%Cu-40%SiC | 615 | 15 | 26 | 10 | 25 | × |
| Al-5wt%Cu-40%SiC | 615 | 3 | 100 | 10 | 25 | × |
| Al-5wt%Cu-40%SiC | 615 | 10 | 26 | 10 | 50 | × |
| Al-5wt%Cu-60%SiC | 615 | 3 | 26 | 10 | 50 | × |

(*) The success criteria is to obtain pore free, microstructurally sound specimens

*Figures 4.12* and *4.13* shows the microstructural appearance of Al-5wt%Cu-15vol%SiC and Al-5wt%Cu-30vol%SiC specimens after hot pressing at $600^0$C.

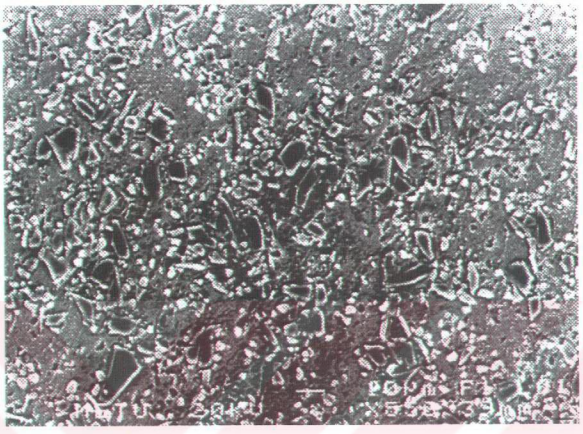


*Figure 4.12* The microstructural view of a Al-5wt%Cu-15vol%SiC specimen after hot pressed at $615^0$C

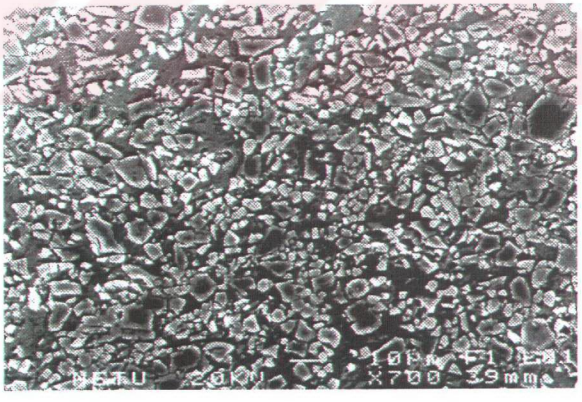


*Figure 4.13* The microstructural view of a Al-5wt%Cu-30vol%SiC specimen after hot pressed at $615^0$



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Several parameters, which act on the final quality of the composites, are studied. Among these, temperature, pressure, hot pressing time, heating rate and SiC powder size can be mentioned.

During hot pressing studies of the Al-Cu-SiC powder mixtures, no problem was encountered in the production of composites up to 30-vol% SiC. However, in Al-5wt%Cu-40vol%SiC composites, either the amount of pores was very high or the specimen could not be hot pressed at all. ***Figure 4.14*** shows the microstructure of Al-5wt%Cu-40vol%SiC composite. Increasing Al powder size caused to incompact samples. Similarly, increasing and decreasing the SiC powder size lead to formation of pores. Better results were obtained at higher temperatures and pressures, especially when the heating rate was kept low. As far as the microstructures of the samples concerned, the optimum results were obtained when the heating rate was $3^0$C/min. since the liquid formation temperature of Al-Cu mixtures lie at $550^0$C, the heating rate was decreased to this level between $550^0$C to hot pressing temperature.

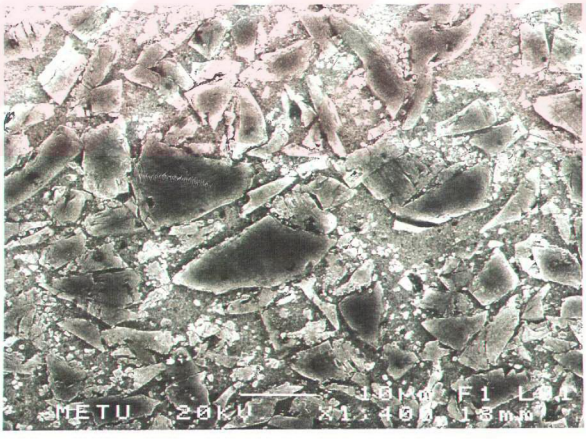


***Figure 4.14*** Microstructure of Al-5wt%Cu-40vol%SiC composite

### 4.1.3.1 Density Measurement Results

First, the theoretical densities of Al-5wt%Cu-SiC samples were calculated then these results were compared to the experimental densities. In Al-5wt%Cu-20vol%SiC samples 99% compaction was satisfied. Similarly, in Al-5wt%Cu-30vol%SiC sound samples were obtained. However, increasing the SiC content to 40vol% reduced the compaction amount to 87%. ***Table 4.2*** shows the experimental densities together with theoretical densities. Percent compaction values were also tabulated.

***Table 4.2*** The Density Measurements

| %SiC | $\rho_{th}$ (g/cm$^3$) | $\rho_{exp}$ (g/cm$^3$) | Avg. $\rho_{exp}$ (g/cm$^3$) | % Compaction |
|---|---|---|---|---|
| 20 | 2.79 | 2,62 | 2,75 | 99 |
| 20 | 2.79 | 2,79 | 2,7 | 95 |
| 20 | 2,79 | 2,79 | 2,53 | 87 |
| 20 | 2,79 | 2,79 | | |
| 30 | 2,84 | 2,68 | | |
| 30 | 2,84 | 2,66 | | |
| 30 | 2,84 | 2,65 | | |
| 30 | 2,84 | 2,53 | | |
| 30 | 2,84 | 2,83 | | |
| 40 | 2,89 | 2,51 | | |
| 40 | 2,89 | 2,55 | | |
| 40 | 2,89 | 2,55 | | |

### 4.1.5 Hot Pressing of Al-Cu-Mg-SiC System

In this study, the preliminary concern was to produce Al-SiC metal matrix composite comprising 40 vol% SiC. However, preliminary experiments have indicated that the hot pressing parameters, which were successful in the production of Al-5%Cu-15vol%SiC, ***Figure 4.12***, and Al-5%Cu-30%SiC, ***Figure 4.13***, did not work for Al-5%Cu-40vol%SiC composites. After hot pressing operation, all the specimens containing 40-vol% SiC were still in powder form. As a result, elemental magnesium powder is added to Al-Cu-SiC

powder mixtures. Mg is known to be a good surface-active element and improves wetting of SiC with liquid aluminium.

The hot pressing temperature of Al-5wt%Cu-5wt%Mg-40vol%SiC were selected as 615$^0$C, which was identical to that of Al-Cu-SiC mixtures. However, behaviour of Mg powder during hot pressing was very similar to that of Al-Cu-Mg matrix alloy. Residual pores were seen in the matrix. The porosity caused by magnesium can be seen in ***Figure 4.15***. On the other hand, ***Figure 4.16*** shows a pore free region of the same sample. It can be seen that SiC particles are distributed more or less homogeneously within the matrix.

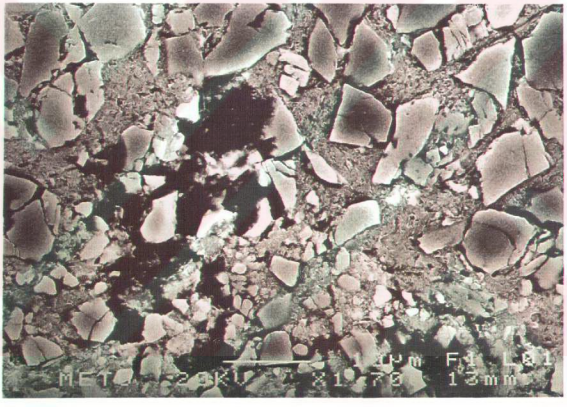

***Figure 4.15*** A porosity at the original site of the Mg powder in hot pressed (615$^0$C) Al-5wt%Cu-5wt%Mg-40%SiC sample

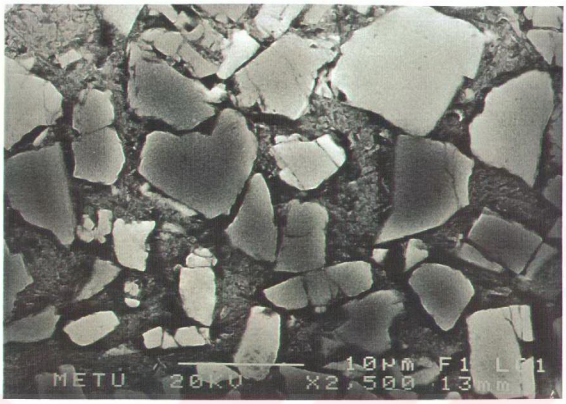


*Figure 4.16* A pore free region in hot pressed (615$^0$C) in Al-5wt%Cu-5wt%Mg-40%SiC sample

### 4.1.6 Three Point Bending Tests

The sound specimens obtained from section 4.1.1 and 4.1.3, were subjected to three point bending tests. The aim was to compare the bending properties of Al-5wt%Cu-SiC samples in the range 20 vol%SiC to 40 vol%SiC.

The criterion of success in hot pressing of samples in section 4.1.3 was their microstructures. However, together with the microstructural properties the mechanical properties should also be concerned.

The stress-strain behaviour of brittle materials is not usually ascertained by tensile test for two reasons. First, it is difficult to prepare and test specimens having the required geometry; and second, there is a significant difference in results obtained from tests conducted in compressive and in tensile modes. Therefore, transverse bending test, which is more suitable for brittle materials, is most frequently employed. In this type of testing, a rod specimen having a

rectangular cross section is bent until fracture using three point loading scheme. At the point of loading, the top of surface is placed in a state of compression, whereas the bottom surface is in tension. Stress is computed from the specimen thickness, the bending moment, and the moment of inertia of the cross section.

For a rectangular cross section, the flexural strength is

$$\sigma_f = 3PL / 2bh^2 \qquad (4.1)$$

where P is the load at fracture, L is the distance between supports, b is the width of the specimen and, h is the height of the specimen. The bending test results obtained can be seen in ***Table 4.3***. After obtaining these data, the standard deviation of experimental stiffness and flexural strength was calculated for 20vol%, 30vol% and 40vol% SiC containing composites, ***Table 4.4***. The data given in ***Table 4.3*** is plotted in ***Figures 4.17***, ***4.18*** and ***4.19***. As seen from ***Figure 4.17***, the flexural strength of the composite decreases with an increase in SiC content from 20 vol% to 40 vol%. However, the flexural strength of the matrix alloy Al-5wt%Cu is lower than the composite containing 20 vol%SiC. As far as the stiffness of the composites is concerned, an increase in SiC content caused an increase in stiffness as well. ***Figure 4.18*** shows the same data as a bar chart.

*Table 4.3* Three Point Bending Test Results

| % SiC | $P_{max}$ (kg) | $\Delta X_m$ (mm) | S (kg/mm) | $\sigma_f$ (Mpa) |
|---|---|---|---|---|
| 0 | 360 | 1,69 | 259,16 | 268,40 |
| 0 | 430 | 1,88 | 265,35 | 320,59 |
| 0 | 390 | 0,56 | 217,90 | 393,82 |
| 20 | 174 | 1,44 | 162,77 | 381,41 |
| 20 | 196 | 1,44 | 181,68 | 455,90 |
| 20 | 336 | 1,59 | 250,84 | 481,95 |
| 20 | 338 | 1,75 | 254,34 | 485,45 |
| 30 | 141 | 0,67 | 221,54 | 302,43 |
| 30 | 155 | 0,69 | 229,96 | 307,42 |
| 30 | 182 | 0,94 | 204,58 | 377,80 |
| 30 | 242 | 0,87 | 322,35 | 409,92 |
| 40 | 242 | 0,64 | 239,90 | 120,10 |
| 40 | 244 | 0,83 | 336,05 | 136,34 |

*Table 4.4* Some Statistical Data Derived from Table 4.2

| $S_{avg}$ (kg/mm) | % SiC | Min | + error | - error | std | $\sigma_f^{avg}$ (Mpa) | min | max | Std | + error | - error |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 247,47 | 0 | 217,9 | 265,4 | 17,9 | 29,6 | 327,6 | 268,4 | 393,82 | 63 | 66,2 | 59,2 |
| 212,41 | 20 | 162,77 | 254,3 | 41,9 | 49,6 | 451,18 | 381,4 | 485,45 | 48,34 | 34,3 | 69,8 |
| 244,61 | 30 | 204,58 | 322,4 | 77,7 | 40,0 | 349,39 | 302,4 | 409,92 | 53,03 | 60,5 | 46,9 |
| 287,98 | 40 | 239,9 | 336,3 | 48,4 | 48,1 | 128,22 | 120,5 | 136,34 | 11,48 | 8,1 | 7,7 |

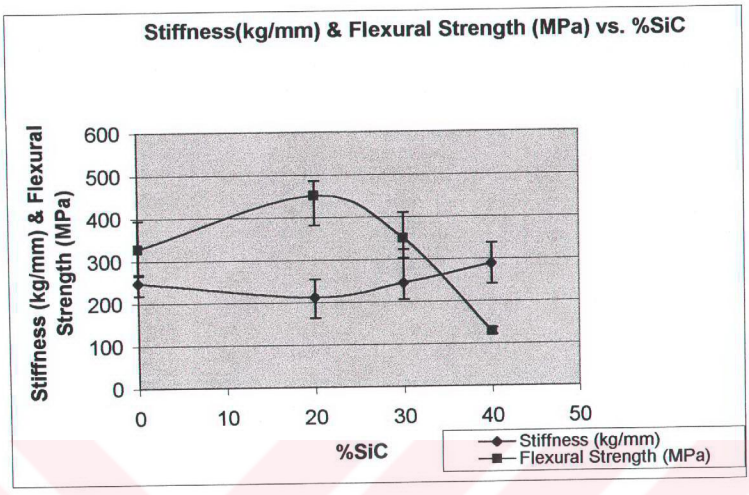


*Figure 4.17* Stiffness & Flexural Strength vs. %SiC

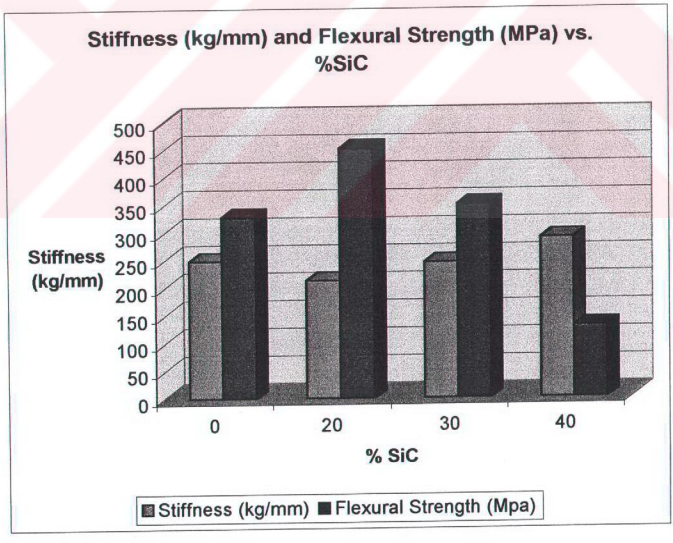


*Figure 4.18* Bar Chart form of Chart 4.1

The volume fraction of two different phases in a given composite influences the mechanical behaviour. A mathematical expression has been formulated for the dependence of a flexural strength on the volume fraction of the constituent phases for a two phase composite. This rule of mixture equation predict that the flexural strength should be greater than or equal to a lower bound represented by;

$$\sigma_f^C = \sigma_f^m \sigma_f^p/(\sigma_f^m V^p + \sigma_f^p V^m) \quad \textit{(4.2)}$$

In ***Equation 4.2***, $\sigma_f$ and V denote the flexural strength and volume fraction respectively, where as the superscripts c, m and p represent the composite, matrix and particulate phases. ***Figure 4.19*** shows the experimental behaviour of Al-Cu-SiC composites as far as the bound of rule of mixture is concerned.

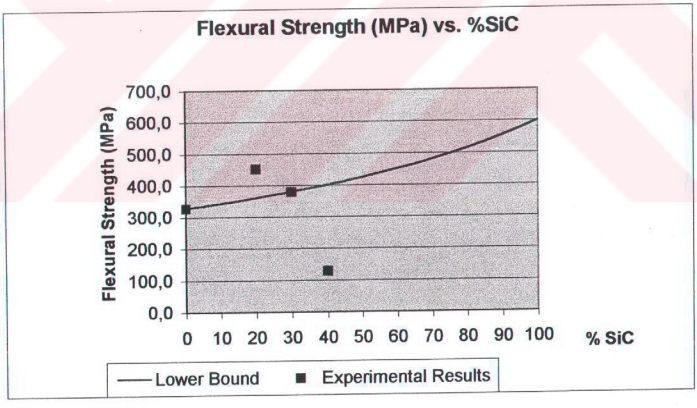


***Figure 4.19*** Rule of mixtures & experimental flexural strength data

### 4.1.6 Fracture Surface Analysis

Fracture surfaces of selected specimens were observed under SEM. The aim was to gain information about the fracture type, the crack path, and the particle-matrix bonding.

The fracture surfaces of Al-5%Cu specimen are given in ***Figure 4.20***. Micrograph ***4.20*** was taken from the middle part of the fracture surface. The dimples on the surface are typical for ductile fracture, which indicates extensive plastic deformation.

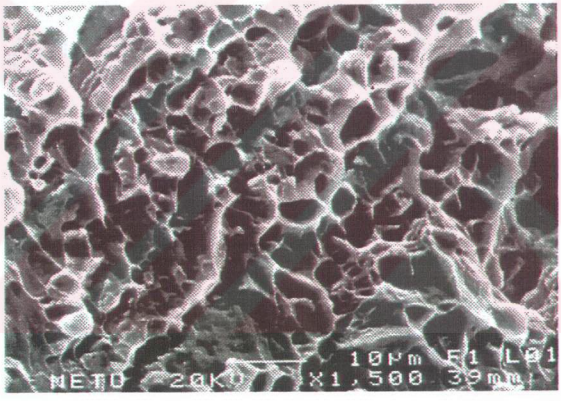


***Figure 4.20*** Fracture surface of Al-5%Cu

Addition of SiC particles into Al-Cu matrix restricted the plastic deformation, ***Figures 4.21***.

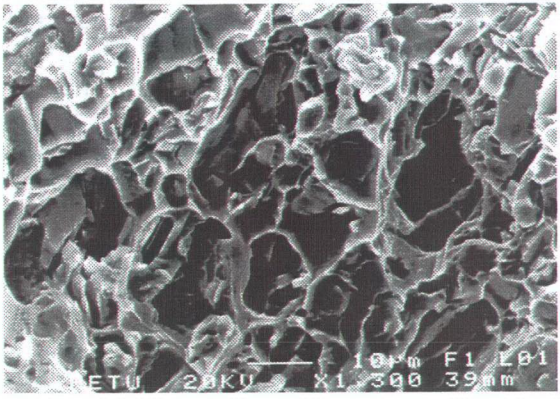


*Figure 4.21* Fracture Surface of Al-5%Cu-15%SiC

Addition of magnesium to the system did not affect the fracture behaviour very much. However, void formation was observed in Al-5wt%Cu-5wt%Mg-40%SiC samples. In ***Figure 4.22***, the fracture surface of Al-5wt%Cu-5wt%Mg-40%SiC can be seen. Although the traces of plastic deformation are still visible on the surface, the deformation is localized around SiC particles. The SiC particles embedded in the matrix can be observed in ***Figure 4.23***. The flat surfaces of the SiC particles indicate that they were broken during crack propagation and left embedded in the matrix.

Therefore, it can be stated that specimens show localized plastic deformation uniformly throughout the failed surface. High concentration of SiC particles limited the gross plastic deformation. ***Figure 4.24*** shows the regions where the matrix could not reach.

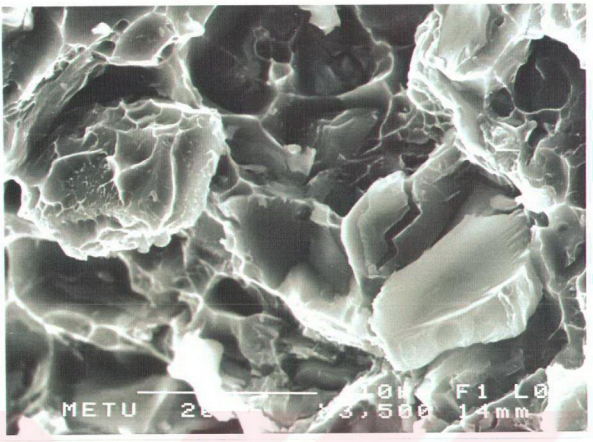


*Figure 4.22* Fracture surface of Al-5wt%Cu-5wt%Mg40vol%SiC

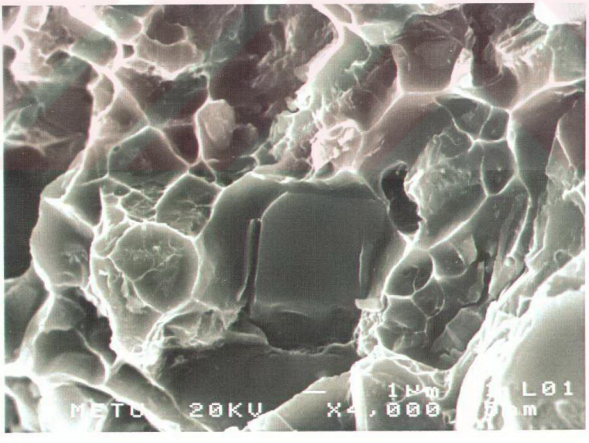


*Figure 4.23* A SiC particle embedded in the matrix of Al-5wt%Cu-5wt%Mg-40vol%SiC

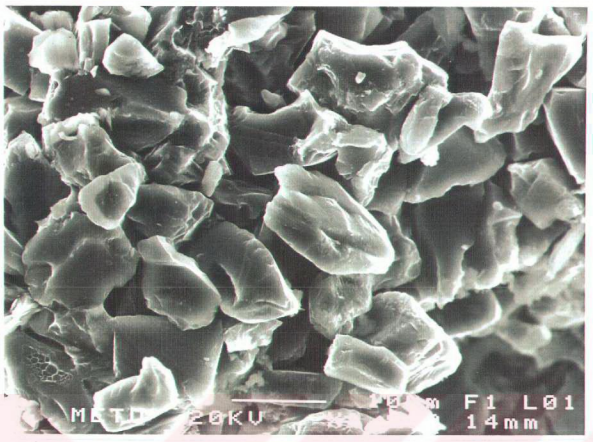


***Figure 4.24*** An agglomeration of SiC particles, in which the matrix alloy could not penetrate in Al-5wt%Cu-5wt%Mg-%40SiC

# CHAPTER 5

# DISCUSSION

Among the various metal working technologies, powder metallurgy is the most diverse manufacturing approach. One attraction to powder metallurgy is its ability to fabricate high quality, complex parts to close tolerances in an economical manner. The process efficiency uses automated operations with low energy consumption, high material utilization and low capital costs. **[20]**

The PM process relies on a different philosophical approach to component fabrication than the one encountered in traditional metalworking. Specifically, the versatility of PM gives expanded latitude to the processing of materials: not only is the material chemistry, heat treatment & microstructure variable, but also the distribution of phases & micro-constituents (including porosity) is controllable.

Lightweight metals are finding increased use in PM industry due to their unique physical and mechanical properties. One of the lightweight metals prevalent in PM industry, the aluminium based PM parts enjoy the widest range of applications, due to their high-strength to weight ratio (specific strength), corrosion resistance and superior finishing properties **[22]**. Therefore, aluminium powder metallurgy has been a topic of interest for many years, since the production with any other element would make the use of this new structure as a suitable matrix material for many composites.

Investigations have been carried out to sinter aluminium with a suitable element based on a suitable process. Since aluminium powder contains a thin but stable oxide layer, it was considered that complete sintering of aluminium powder is not possible. However, in a recent study **[32]**, the oxide scale broken up by plastic deformation of Al powder itself in the compaction process, making it possible for sintering to be achieved to a certain extend.

On the other hand, it is a well-known fact that hot consolidation is very effective in obtaining dense and compact bodies from powder mixtures **[33]**. The pressure applied to the powder at an elevated temperature and is combined with a sintering step. Thus, hot pressing eliminates the cold pressing and the sintering steps.

In hot pressing, both creep and sintering are diffusional processes. In this study, a uniaxial hot press setup with an upper punch was used. The rate of densification due to an external stress can be estimated in terms of a surface energy enhanced driving force. Higher external stresses, longer times, higher temperatures and finer powder sizes will enhance the density of a hot pressed material. The action of an external stress is to promote grain and particle sliding by diffusional processes, to generate vacancies, and to cause pore collapse. The overall effect is a more rapid densification.

Recent studies **[10,30,31]**, Al-5%Cu, Al-5%Cu-15%SiC, Al-5%Cu-30%SiC mixtures were hot pressed successfully. In this study, magnesium added to the system because it a good surface-active element and Al-Cu-Mg alloys are heat treatable **[34,37]**.

The eutectic liquid formation in Al-Cu-Mg system is an important fact to mention. As it can be seen from Al-Cu-Mg ternary phase diagram ***Figure 5.1***,the following invariant reactions occur in the temperature range 450 – 548 $^{0}$C, ***Table 5.1***.

*Table 5.1* Invariant Reactions of Al-Cu-Mg System

| Reaction | Temperature $^0C$ | Composition | |
|---|---|---|---|
| | | %Cu | %Mg |
| Liq → Al + $CuAl_2$ | 548 | 33 | 0 |
| Liq → Al + $CuAl_2$ + $CuMgAl_2$ | 508 | 30 | 6 |
| Liq → Al + $CuMgAl_2$ (quasi binary) | 518 | 24,5 | 10,1 |
| Liq + $CuMgAl_2$ → Al + $CuMg_4Al_6$ | 467 | 10 | 26 |
| Liq → $CuMg_4Al_6$ + Al + $Mg_5Al_8$ | 450 | 2,7 | 32 |
| Liq → Al + $Mg_5Al_8$ | 450 | 0 | 33 |

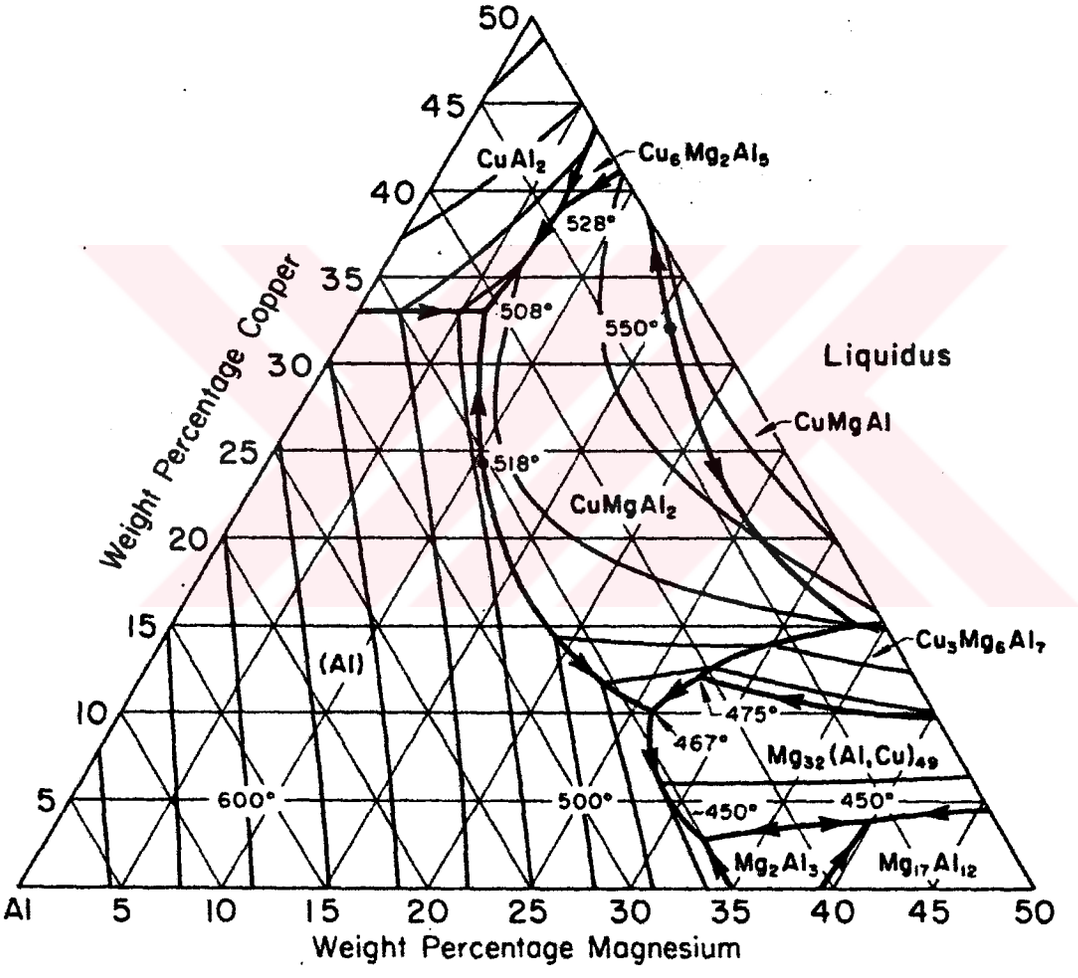


*Figure 5.1* Al-Cu-Mg Ternary Phase Diagram

Therefore, in order to form a liquid phase, powder mixture should be hot pressed at 450 $^0$C and above. However, it is important to note that elemental powder mixtures behave different with respect to pre-alloyed powders with an increase in temperature. In elemental powder mixtures, the powder particle interacts with its surrounding at the initial stages of heating, in a similar manner that the pure metals are brought into contact. This may cause appearance of several phases at the powder matrix interface, as shown in ***Figures 4.4-4.8 & 4.9-4.11***.

***Figure 4.4*** depicts that how a solid-state reaction occurs at the powder-matrix interface. Layered structure of the copper particle is typical for solid state-diffusion. On the other hand, ***Figure 4.5*** shows the original site of a magnesium particle, however, the original shape of Mg powder cannot be observed, anymore. This may be explained in terms of diffusion coefficients of copper and magnesium in aluminium. Diffusion of magnesium is much faster than that of copper in aluminium matrix at 430$^0$C, as can be seen from ***Table 5.2***.

***Table 5.2*** Diffusion Coefficients of Cu & Mg in Al at 430$^0$C

| Diffusing Species | Host Metal | Temp, $^0$C | D, m$^2$/sec |
|---|---|---|---|
| Cu | Al | 430 | $5.98*10^{-15}$ |
| Mg | Al | 430 | $2.19*10^{-14}$ |

It is interesting to note that a continuous phase precipitation is observed at the original Al powder particle boundaries when the hot pressed sample is quenched from 600$^0$C, ***Figure 4.7***. This is probably a residue of the liquid phase formed at 600$^0$C. The grain boundary network is still evident when the quenching temperature is decreased to 550$^0$C. This result indicates that the liquid phase formed in Al-Cu-Mg mixtures is quite active and immediately wets the Al powder particle boundaries.

Liquid formation sites are determined via quenching cold compacted samples from different temperatures. The light contrasted regions in ***Figures 4.9-4.11*** are the original copper site transformed to liquid into which substantial amount of Mg is diffused during heating. Therefore, it can be stated that the diffusion of Mg into the Al matrix is quite fast.

Actually, in equilibrium phase diagrams invariant reactions shift to lower temperatures while starting from elemental powders. This deviation can be explained by considering the following facts. To form a solid phase from a liquid phase, it is necessary to have an under-cooling. This under-cooling supplies both the bulk free energy and the surface energy for formation of the solid phase. However, while heating elemental powders from room temperature, isothermal reactions occur at lower temperatures. The reason is that powders are fine and have extremely large surface area. This large surface area has a very high surface energy, which can be used for the solid to liquid transformation. In brief, the large specific surface area is the driving force for sintering.

Hence, an Al-5%Cu-5%Mg alloy, which should not form a liquid at 450 0C, can cause local melting, when prepared from elemental powders. That's to say; at any composition local melting may cause the formation of liquid phase.

In this study, amount of Mg added to the system is chosen 5%, because in that composition range Al-Cu-Mg phase diagram can be estimated by Al-Cu phase diagram. ***Figure 5.2*** illustrates the Al-rich end of the Al-Cu-Mg phase diagram with the liquidus, solidus and solvus shown for a 4%Cu, 1%Mg alloy.

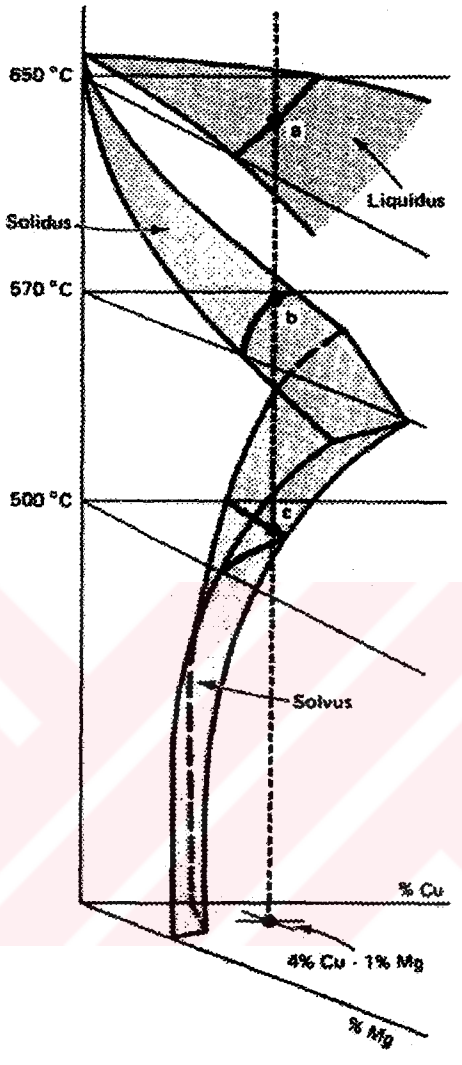


*Figure 5.2* Al-rich end of Al-Cu-Mg Phase Diagram

In view of above results, hot pressing temperatures were chosen as 430 $^0$C. The isothermal section shows the inter-metallic phases found in the system at 430 $^0$C, ***Figure 5.3***.

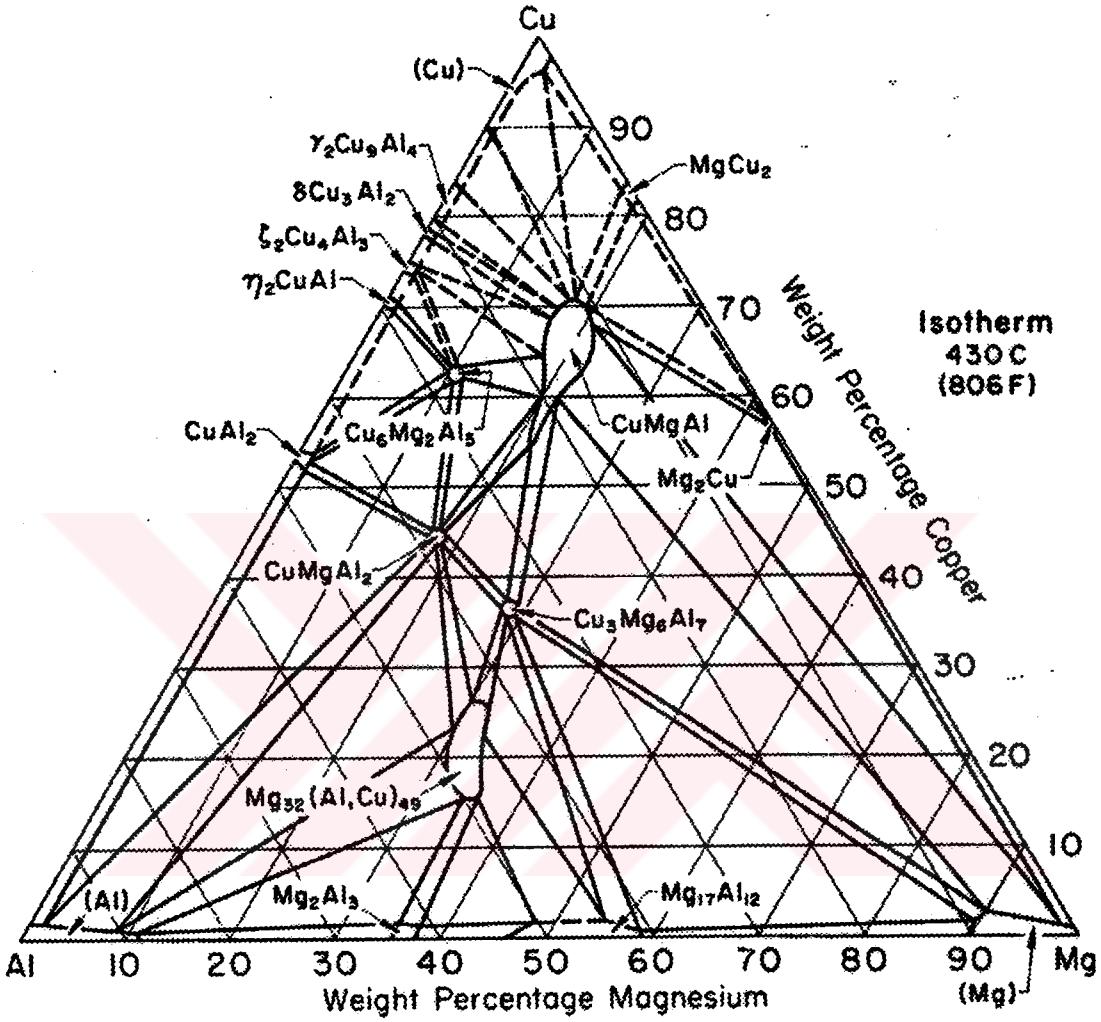


*Figure 5.3* 430 $^0$C Isotherm of Al-Cu-Mg Phase Diagram

Behaviour of magnesium during hot pressing is very similar to that of metal powders during sintering. To prevent porosity formation during the sintering of pure metal powders;

- The solubility of matrix in added powder should be high
- Powders should locate at grain boundaries
- Diffusion coefficients of the matrix and the added powders should be equal

Unfortunately, Mg powder does not satisfy these properties. Even the solubility of Mg in Al is higher than that of Al in Mg. Hence, it can be concluded that Mg powder is not suitable for hot pressing applications as far as porosity formation is concerned. Introduction of SiC powder to the system did not change the effect of Mg. The porosity caused by Mg can be seen in ***Figure 4.6***.

In contrast to samples containing magnesium and SiC, the others that do not contain Mg but SiC has given promising results. However, increasing amounts of SiC deteriorates desired microstructural, thus mechanical properties. The optimum results were obtained when hot pressing at $615^0$C with a heating rate of $3^0$C/min and SiC content less than or equal to 30 volume percent, ***Table 4.1***. This result coincides with the theory of hot pressing; higher pressures, longer times of heating give rise to more compact bodies.

As it can be seen from ***Figures 4.17*** and ***4.18*** flexural strength of the matrix Al-5wt%Cu is less than that of composites containing 20 and 30vol % SiC. This was not an unexpected result since addition of SiC enhances the work hardening effect. However, further increase in SiC to 40 vol% leads to formation of agglomerates and hot pressing operation becomes harder. Hence, a drastic decrease of flexural strength was observed.

The data obtained for flexural strength lie in a reasonable range as far as the rule of mixtures concerned. As it can be seen from ***Figure 4.19***, there are some deviations from rule of mixtures. This might be explained in terms of by the nature of rule of mixtures. Rule of mixtures does not concern the following;

- The interface
- Shape of the reinforcement
- Size of the reinforcement
- Distribution of the reinforcement

Therefore, the experimental deviations are reasonable as far as the rule of mixtures concerned. Furthermore, the samples containing 40 volume percent SiC contain pores, ***Table 4.2***. It is a known fact that bending test gives a rough description for flexural strength when the specimens contain defects. In conclusion the results obtained lie in a reasonable range and give a preliminary idea about the behaviour of flexural strength and stiffness.

In regards to fracture behaviour of Al-Cu-SiC mixtures, it was investigated that the fracture was mainly a quasi-cleavage type due to presence of SiC particles at the matrix particle interface. Generally, micro-void coalescence is the predominant fracture mode in particle reinforced MMCs. The stages of micro-void coalescence consist of void nucleation, growth and coalescence. The void nucleation process can be influenced by a variety of factors including total volume fraction and local fraction of particles, matrix deformation characteristics and interfacial bond strength. **[35]** In particle-reinforced materials, it has been demonstrated that void nucleation and the final ductility may be sensitive to both the size and the distribution of reinforcing particles. In particular, it has been suggested that the local volume parameter in determining the fracture of materials containing dispersion of particle at high volume percentages. **[36]**

In this study, it can be stated that the specimens show localized plastic deformation throughout the failed surface. High concentration of SiC particles limited the gross plastic deformation both with the addition of Mg and without.

# CHAPTER 6

# CONCLUSION

- Al-5wt%Cu powder mixture can be hot pressed with the help of the liquid phase formed, under nitrogen atmosphere without porosity.

- Introduction of Mg to Al-Cu system results in formation of pores due to high solubility of Mg in Al. Mg powder seems to be not suitable for hot pressing applications as far as the porosity formation is concerned.

- The conventional hot pressing of powder mixtures prepared from pure Al, pure Cu, and SiC powders under nitrogen atmosphere yielded pore free structures when the SiC content is less than or equal to 30 volume percent.

- Addition of Mg powder to Al-5wt%Cu-40vol%SiC system did not work. The results obtained were similar to that of Al-5wt%Cu-40vol%SiC system with additional pores.

- For Al-5%Cu-40vol%SiC mixtures, optimum hot pressing parameters were obtained at $615^0$C hot pressing temperature, 50 MPa pressure and $3^0$C/min hot pressing rate with 10μm SiC powder size and 26μm Al powder size.

# REFERENCES


[1] Basu A. and Humphreys F.J., "The microstructure & Deformation of Al-Mn-SiC particulate MMCs", *3rd International Conference on Aluminium Alloys*

[2] Nair S.V., Tien J.K. and Bates R.C., *International Metals Review, 30, pp 275-288, 1985*

[3] Humphreys F.J., *Materials Science and Engineering,* **A 135**, pp 267-273, 1991

[4] Nieh T.G. and Korlak R.F., *Scripta Metall.,* **18**, pp 25-28, 1984

[5] Mordfin L., *3rd International Conference on Aluminium Alloys*

[6] Harrington W.C., "Commercial Processing of MMCs", *Materials Science and Engineering,* **A244**, pp 75-79, 1998

[7] Wang N., Wang Z.R., Weatherly, "Formation of Magnesium Aluminate(Spinel) in Cast SiC Paericulate Reinforced Aluminium MMCs", *Metall. Trans.,* **A23**, pp 1423-1430, 1992

[8] Kulkarni M.D., Robi D.S., Prasad R.C., Ramahrishan P., "Fracture Toughness and Fractography of Cast and Extruded 7075 Al-SiC Particulate Composites", *Scripta Metall. Mater.,* **31**, pp 237-242, 1994

[9] Tan M.J., Koh L.H., Khur K.A., Boey F.Y.C., Murakoshi Y., *Journal of Materials Proceedings Tech.,* **37**, pp 391, 1993

[10] Ögel B., Bolay K., Kaya G., " The microstructure and Mechanical Properties of Al-Cu & Al-Cu-SiC Powders", *Sintering Science & Tech.*, 2000

[11] Chou T.W., Kelly A. and Okura A., *Composites,* **16,** pp 187, 1985

[12] Dinwoodie J., Moore E., Langman and *Proc. 5th International Conference on Composite Materials,* pp671, 1985

[13] Suresh S., Martensen A., Needlemann A., *Fundamentals of MMCs,* 1993, USA, Butterworth, Helnemann

[14] Begg A.R., *Powder Metallurgy,* **36,** pp 107, 1993

[15] Erich D.L., *International Journal of Powder Metallurgy,* **23,** pp 45, 1987

[16] İbrahim I.A., Mohammed F.A., Cavernia E.J., *Journal of Materials Science,* **26,** pp 1156, 1991

[17] Majumbar B.S., Yegneswaran A.H, *Materiaks Science and Engineering,* **68,** pp 85, 1984

[18] Flom Y., Arsenault R.J., *Journal of Metals,* **25,** pp 31, 1986

[19] Ashby M.F., *Powder Metallurgy: An Overview*

[20] German R.M., *Powder Metallurgy Science,* Princeton, NJ, Powder Metal Industries Federation, 1984

[21] Goetzel C.G., *Treatise on Powder Metallurgy,* **1**, NY, Interscienc Publishers Inc., 1949

[22] ASM Handbook, 9th Edition, **7**, *Powder Metallurgy*

[22] Hausner H.H., *Perspectives in PM,* **1**, NY, Plennium Press,1967

[23] Lenel F.V., *Powder Metallurgy,* Princeton, Met.Powder Ind. Fed., 1980

[24] Hausner H.H., *Handbook of PM,* NY, 1973

[25] Pickens J.R., *Journal of Materials Science,* **16**, pp 1437, 1981

[26] Coble R.L., *Powder Met. Inter.*, **10**, pp 128-130, 1978

[27] Notis M.R., Smoak R.H. and Krishnamachari, *Sintering & Catalysis*, NY, Plenium Press, pp 493-507, 1975

[28] Ashby M.F., Arzt E., Easterling K.E., *Met. Trans.*, **14A**, pp 211-221, 1983

[29] Springs R., Dutta S., *Mat. Science & Eng.*, **6**, pp 369, 1973

[30] Kaya G., *MSc. Thesis*, METU, 1999

[31] Bolay K., *MSc. Thesis*, METU, 1998

[32] Wantanabe T., Yamada K., *Inter. J. Powder. Metall.*, **4**, pp 337, 1968

[33] Tweed T.H., *Mat. Science & Eng.*, **A135**, pp 73

[34] Hunt E, Pitcher P.D., *Proc. Int. Conf. Of Light Metals*, pp 687, 1990

[35] Lu L., Lai M.O., Yap S.H., *Mat. Science & Tech.*, **13**, pp 192, 1997

[36] Lewandowski J.J., *Mat. Science & Eng.*, **A107**, pp 241, 1989

[37] Şimşir F.M., Güngor Ö., Ögel B., *Proc. of Inter. Metall. & Mater. Cong.*, **3**, pp 1781, 2000

[38] Van Horn K., *Aluminum*, **1**, 1967



T.C. YÜKSEKÖĞRETİM KURULU
DOKÜMANTASYON MERKEZİ