A THESIS

On

SYNTHESIS AND CHARACTERIZATION OF IMMISCIBLE ALUMINIUM BASED BEARING ALLOYS THROUGH P/M ROUTE

Submitted in the partial fulfillment of requirement for the award of the degree of

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IN

MATERIALS SCIENCE AND ENGINEERING

Submitted by

KOMAL MANMEET DEOL
Roll No.: 6040504

Under the guidance of

Dr.O.P.PANDEY

School of Physics and Materials Science

THAPAR INSTITUTE OF ENGINEERING AND TECHNOLOGY

(DEEMED UNIVERSITY)

PATIALA (PUNJAB)-147004

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CERTIFICATE

This is to certify that the thesis entitled " **Synthesis and Characterization of immiscible Aluminium based bearing alloys through P/M route"** submitted by **Miss Komal** in the partial fulfillment of the requirement for the award of the degree of **M. Tech** in **Materials Science and Engineering** from the **School of Physics and Materials Science, Thapar Institute of Engineering and Technology (Deemed University), Patiala,** is a record of candidate's own work carried out by her under my supervision and guidance. The matter embodied in this report has not been submitted in part or full to any other university or institute for the award of any degree.

(Dr.O.P.PANDEY)
Professor, SPMS
Thapar Institute of Engg. &Technology,
Patiala, Punjab (147004)

Countersigned by:

(Dr. O.P. Pandey)	**(Dr. T.P. Singh)**
Professor and Head, SPMS	Dean, Academic Affairs
Thapar Institute of Engg. & Technology,	Thapar Institute of Engg. & Technology,
Patiala, Punjab (147004)	Patiala, Punjab (147004)

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Komal
6040504

Dedicated To My Parents

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ABSTRACT

Aluminium bearing alloys are finding vast applications in various automobile industries these days. Aluminium P/M offers components with exceptional mechanical properties, low density, corrosion resistance, excellent machinability, good response to variety of finishing operations. In addition, aluminium P/M parts can be further processed to eliminate porosity and improve bonding yielding properties better than those of conventional wrought aluminium products.

Aluminium bearing alloys consisting of immiscible elements are being produced by conventional casting route. The problem of segregation of second element due to variation in densities always exists. The P/M route offers a better path to develop these alloys. In order to sinter these, a reducing or inert atmosphere is required. The current effort is to see a feasibility of producing such bearing alloys on industrial scale by creating a reducing atmosphere.

In this work, two compositions of conventional aluminium base bearing alloys were selected with a variation of Sn and Pb as a soft phase. Various series of alloys with variation in Sn and Pb% were selected for the study. P/M route was employed for the preparation of the alloys. The alloys were then studied for the variation in microstructure of different samples w.r.t the different sintering temperatures. This was done using an optical microscope at various magnifications. Microhardness test was also conducted on various samples using the Vickers hardness testing machine. Variation in microstructures and hardness was observed for different samples consisting of Pb and Sn as additives.

CHAPTER 1

INTRODUCTION

Lightweight metals are finding increased use in the Powder Metallurgy industry due to their unique physical and mechanical properties. Out of the lightweight metals most prevalent in the P/M industry (aluminium, titanium, beryllium), aluminium P/M parts enjoy the widest range of applications. Due to their high strength-to-weight ratio, corrosion resistance and superior finishing properties, aluminium parts are used in different machines, automotive, aerospace and other appliance applications. [1]

Powder Metallurgy technology provides a useful means of fabricating net shaped components that enables machining to be minimized, thereby reducing costs. Advances in P/M techniques that offer control of part shape and dimension with minimal scrap loss have significantly increased aluminium P/M parts application. Conventionally pressed and sintered aluminium powder metal parts have been commercially available for many years. Aluminium P/M parts are competitive with many aluminium castings, extrusions and screw machine products that require expensive and time consuming finishing operations. Also, P/M technology can be used to refine microstructures compared with those made by conventional ingot metallurgy, which often results in improved mechanical and corrosion properties. In addition aluminium P/M parts compete with other metal powder parts in applications where some of the attractive physical and mechanical properties of aluminium can be utilized.

BEARING MATERIALS

Machine parts are designed to reduce friction between moving parts or to support moving loads. There are two main kinds of bearings: the antifriction type, such as the roller bearing and the ball bearing, operating on the principle of rolling friction; and the plain, or sliding type, such as the journal bearing and the thrust bearing, employing the principle of sliding friction. . Ball bearings or roller bearings reduce friction more than sliding bearings do. Other advantages of antifriction bearings include ability to operate at high speeds with easy lubrication.

A **plain bearing** is a bearing which carries load by sliding. A plain bearing is often called a bushing or a babbit or journal bearing. Plain bearings are very widely used and appear in most kinds of equipment, notably as crankshaft and connecting rod bearings in automobile piston engines.

A typical plain bearing is made of two parts. A simple linear bearing can be a pair of flat surfaces designed to allow motion, for example a drawer and the slides it rests on.

Plain bearing may carry load in one of several ways depending on their operating conditions, load, relative surface speed (shaft to journal), clearance within the bearing, quality and quantity of lubricant and temperature (affecting lubricant viscosity). If full-film conditions apply, the bearing's load is carried solely by a film of fluid lubricant, there being no contact between the two bearing surfaces. In this condition they are known as fluid bearings. In mix or boundary conditions, load is carried partly by direct surface contacts and partly by a film forming between the two. In a dry condition, the full load is carried by surface to surface contact.

Plain bearings are relatively simple and hence inexpensive. They are also compact, lightweight, straightforward to repair and have high load-carrying capacity. However, if operating in dry or boundary conditions plain bearings may wear faster and have higher friction than rolling element bearings. Dry and boundary conditions may be experienced even in a fluid bearings when operating outside of its normal operating conditions, i.e., at startup and shutdown.

A common plain bearing design is to use a hardened and polished steel shaft and a soft bronze bushing. In such designs the softer bronze portion can be allowed to wear away, to be periodically renewed.

Plain 'self-lubricating' bearings utilize porous journals within which a lubricant is held. As the bearing operates and lubricant is displaced from the bearing surface, more is carried in from non-wear parts of the bearing. Dry plain bearings can be made of a variety of materials including PTFE (Teflon) and ceramic. Plain bearings manufactured from high performance polymers are now replacing traditional metal plain bearings.

Aluminum Bearing Materials

Aluminum alloys with desirable bearing properties are used in a wide variety of applications. Steel-backed and solid aluminum bearings are employed as connecting rod and main bearings in internal combustion engines and industrial compressors. Other aluminum bearing applications are in heavy tooling, such as boring mills, presses, lathes, milling machines, and grinding mills, and as hydraulic pump bushings. Aircraft landing gear assemblies, power shovels, and track rollers utilize solid aluminum bearings to withstand high-shock loads. Rolling mill bearings are cast of aluminum alloys to increase load and speed capability.

Aluminum bushings are normally employed for relatively light, low-speed duty, compared to bearings, and they are made from aluminum bearing or other alloys, depending on the frictional and mechanical properties required for the application.

In this project, we have made an attempt to add Sn and Pb separately to aluminium-copper alloy. The Al-Sn phase diagram (Figure 1) shows many of the features of an ideal sintering system. Al and Sn exhibit mutual insolubility in the solid phase, form no intermetallic phases and the liquids are miscible. Additionally, the melting point of tin (232^{O}C) is much lower than that of aluminium (660^{O}C). Since Sn is virtually insoluble in solid Al, the liquid will persist throughout the whole sintering cycle, rendering the system less sensitive to process variables than a transient system. A higher Sn alloy could be utilized as a bearing material, in which case the increased Sn content may be beneficial in both the elimination of porosity and as a solid lubricant.

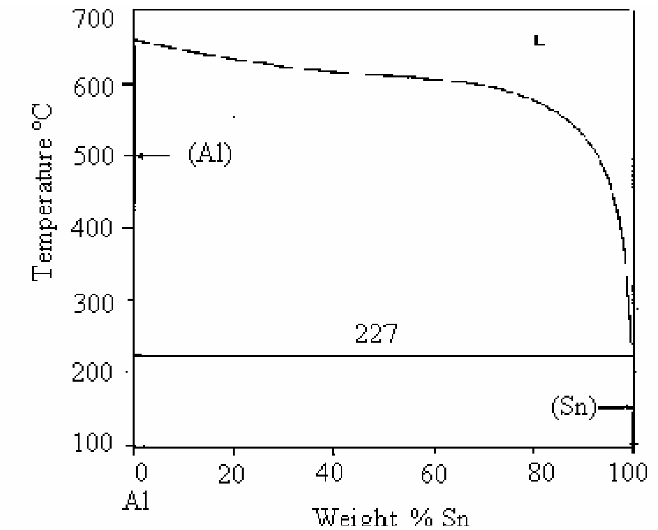


Figure 1: Al- Sn phase diagram

Increasing Sn content increases the green density. A similar result has been found with Pb additions and is due to the lubricity of the Pb. These powders are very soft and during compaction are extruded into the pore spaces. This acts to increase the green density. Addition of lead in small quantities improves the machinability of the aluminium alloy. Also Lead containing bearings have the advantage of good lubrication. However, their chief advantage is their low cost. Al-Pb phase diagram is shown in Figure 2 for reference.

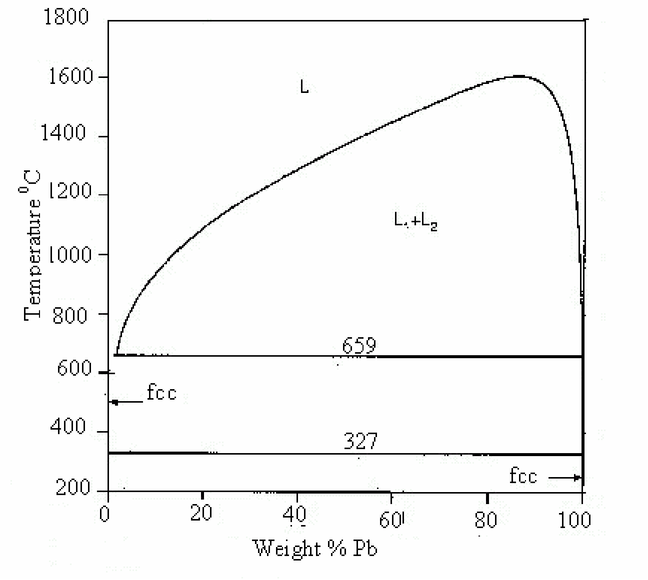


Figure 2: Al-Pb Phase diagram

CHAPTER 2

POWDER METALLURGY METHOD

Powder metallurgy method was used for the preparation of the alloys. The details of this technique are discussed below.

THE METHOD

Powder Metallurgy is a highly evolved method of manufacturing reliable net shaped components by blending elemental or pre-alloyed powders together, compacting this blend in a die, and sintering or heating the pressed part in a controlled atmosphere furnace to bond the particles metallurgically. The P/M process is a unique part fabrication method that is highly cost effective in producing simple or complex parts at, or close to, final dimensions. P/M processing provides the following advantages:

- Production of complex shapes to very close dimensional tolerance and minimum scrap loss and fewer secondary machining operations.
- Physical and mechanical properties of components can be tailored through close control of starting materials and process parameters.
- Particular properties can be improved through secondary processing operations such as heat treating and cold/hot forming.

There are many ways in which metals may be produced in powder form, comminution of solid metal - precipitation from solution of a salt; - thermal decomposition of a chemical compound; reduction of a compound, usually the oxide, in the solid state, electro deposition, and the atomization of molten metal. Of these the last three account for the bulk of the powders used.

2.1 STEPS INVOLVED

The Powder Metallurgy route involves preparation of powders either by the disintegration of the liquid melt into a wide range of droplets and solidification into powder particles or by chemical route. These powders are subsequently consolidated by extrusion, hot isostatic pressing etc. The production of powder is accompanied by a number of techniques such as centrifugal atomization, water atomization, inert gas atomization and vacuum atomization. However inert gas atomization is preferred over other atomization techniques because a high degree of control can be exercised so far as the powder chemistry, powder cleanliness and their size and size distribution is concerned.

2.1.1. ATOMISATION

In atomization, the molten metal is broken up into small droplets and rapidly frozen before the drops come into contact with each other or with a solid surface. The principal method is to disintegrate a thin stream of molten metal by subjecting it to the impact of high energy jets of gas or liquid. Air, nitrogen and argon are commonly used gases, and water is the liquid most widely used.

Centrifugal Atomization Process In addition, there are several other processes that are finding increasing application, an important one being centrifugal atomization in which droplets of molten metal are discharged from a rotating source. There are basically two types of centrifugal atomization processes: - in one a cup of molten metal is rotated on a vertical axis at a speed sufficient to throw off droplets of molten metal, or a stream of metal is allowed to fall on a rotating disc or cone; in the other a bar of the metal is rotated at high speed and the free end is progressively melted e.g. by an electron beam or plasma arc. This latter process is called the Rotating Electrode Process (REP), and the bar may be rotated either on a horizontal or on a vertical axis. A special advantage of these processes is that they can be carried out in a sealed vessel in a controlled atmosphere - even vacuum - and thus produce 'clean' powders of highly reactive metals. With the REP process the avoidance of contact with refractory is a potent means of reducing the number of non-metallic inclusions in the powder, and in components manufactured from the powder.

In **Inert gas atomization,** disintegration of the liquid metal is brought about by using high velocity gas jets. The interaction of the melt stream with the atomizing inert gas drastically affects the efficiency of atomization and also the size distribution of powder particles. The two main components of the gas atomization assembly used during atomization of the melt are the nozzle and the melt flow channel. The interaction of the liquid metal with the high energy jet leads to the formation of a wide range of small sized droplets. The selection of a particular nozzle design depends upon the efficiency and the desired size and size distribution of the droplets. The principle imperative behind nozzle designs is the generation of a gas jet with high kinetic energy and the efficient use of this energy to accomplish melt breakup. Furthermore, a high gas velocity renders small mean particle size and narrow size distribution. The gas atomization techniques can be classified into three categories:

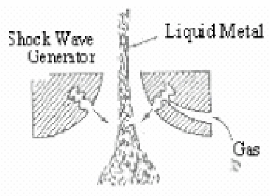
- Subsonic atomization,
- Supersonic atomization
- Ultrasonic atomization.

In **Subsonic gas atomization**, the gas at subsonic velocity leads to coarser powder particle size. The isentropic expansion of a compressed gas through a duct is the basic principle employed for the design of supersonic nozzles. Mainly three kinds of ducts are used to produce high velocity gas jets, namely convergent and convergent-divergent. These work on the isentropic expansion principle. This expansion work could be controlled by different design parameters and working conditions at the inlet and outlet of the nozzles. A highly pressurized gas enters the nozzle and expands isentropically. The thermodynamics and continuity of the flow in the nozzle help to obtain supersonic velocity of the gas. The minimum area in the passage is called throat of the nozzle. At a critical pressure, for a given nozzle designs, the gas obtains sonic velocity at the throat and further expansion imparts a supersonic velocity to the gas provided the exit pressure of the nozzle is equal to the atmospheric pressure. Otherwise the gas losses its energy if it exits the nozzle in over expanded or under expanded conditions. The three different types of gas atomizers used are as shown below,

In **Free fall gas atomizer** the liquid metal falls from crucible under gravity unconstrained into an atomization region where it interacts with few concentric or annular high velocity gas jets. The angle of incidence of the gas jet relative to the liquid stream is critical in the design so as to control splashing and back pressure on the melt flow once the gas jets converge below the nozzle exit. The gas jet travels a long distance before it interacts with the liquid stream and losses part of its kinetic energy, thus, reducing its atomization efficiency. In confined or closed coupled nozzle configuration, the gas-metal interaction takes place at the rim of the melt delivery tube as against the free fall design. Therefore, increased atomization efficiency is obtained from this nozzle configuration.

(a) Confined Gas Melt Atomization

(b) Free Fall Melt



(c) Ultrasonically superimposed gas jets

Figure 3: Spray nozzles used in Gas atomization (a) Confined (b) Free fall (c) Ultrasonic

The powder generated in the atomization process is then consolidated into near net shaped perform by using hot extrusion or hot isostatic pressing etc.

2.1.2. CONSOLIDATION OF POWDERS

COLD UNIAXIAL PRESSING

Elemental metal, or an atomized pre alloyed powder is mixed with a lubricant, typically lithium stearate (0.75 wt. %), and pressed at pressures of say, 600 M Pa (87,000 lb/in$_2$) in metal dies. Cold compaction ensures that the as compacted, or 'green', component is dimensionally very accurate, as it is moulded precisely to the size and shape of the die. Irregularly shaped particles are required to ensure that the as-pressed component has a high green strength from the interlocking and plastic deformation of individual particles with their neighbors. One **disadvantage** of this technique is the differences in pressed density that can occur in different parts of the component due to particle/particle and die wall/particle frictional effects. Typical as-pressed densities for soft iron components would be 7.0 g/cc, i.e. about 90% of theoretical density. Compaction pressure rises significantly if higher as-pressed densities are required, and this practice becomes uneconomic due to higher costs for the larger presses and stronger tools to withstand the higher pressures.

COLD ISOSTATIC PRESSING

Metal powders are contained in an enclosure e.g. a rubber membrane or a metallic can that is subjected to isostatic that is uniform in all directions, external pressure. As the pressure is isostatic the as-pressed component is of uniform density. Irregularly shaped powder particles must be used to provide adequate green strength in the as-pressed component. This will then be sintered in a suitable atmosphere to yield the required product. Normally this technique is only used for semi-fabricated products such as bars, billets, sheet, and roughly shaped components, all of which require considerable secondary operations to produce the final, accurately dimensioned component. Again, at economical working pressures, products are not fully dense and usually need additional working such as hot extrusion, hot rolling or forging to fully density the material.

2.1.3. SINTERING

Sintering is the process whereby powder compacts are heated so that adjacent particles fuse together, thus resulting in a solid article with improved mechanical strength compared to the powder compact. This “fusing” of particles results in an increase in the density of the part and hence the process is sometimes called densification. There are some processes such as hot isostatic pressing which combine the compaction and sintering processes into a single step. After compaction the components pass through a sintering furnace. This typically has two heating zones, the first removes the lubricant, and the second higher temperature zone allows diffusion and bonding between powder particles. A range of atmospheres, including vacuum, are used to sinter different materials depending on their chemical compositions. As an example, precise atmosphere control allows iron/carbon materials to be produced with specific carbon compositions and mechanical properties. The density of the component can also change during sintering, depending on the materials and the sintering temperature. These dimensional changes can be controlled by an understanding and control of the pressing and sintering parameters, and components can be produced with dimensions that need little or no rectification to meet the dimensional tolerances. In many cases all of the powder used is present in the finished product, scrap losses will only occur when secondary machining operations are necessary.

2.1.4. HOT ISOSTATIC PRESSING

Powders are usually encapsulated in a metallic container but sometimes in glass. The container is evacuated, the powder out-gassed to avoid contamination of the materials by any residual gas during the consolidation stage and sealed-off. It is then heated and subjected to isostatic pressure sufficient to plastically deform both the container and the powder. The rate of densification of the powder depends upon the yield strength of the powder at the temperatures and pressures chosen. At moderate temperature the yield strength of the powder can still be high and require high pressure to produce densification in an economic time. Typical values might be 1120°C and 100 M Pa for ferrous alloys. By pressing at very much higher temperatures lower pressures are required as the yield strength of the material is lower. Using a glass enclosure atmospheric pressure (15 psi) is used to consolidate bars and larger billets. The technique requires considerable financial investment as the pressure vessel has to withstand the internal gas pressure and allow the powder to be heated to high temperatures. As with cold isostatic pressing only semi finished products are produced, either for subsequent working to smaller sizes, or for machining to finished dimensions.

2.1.5. METAL INJECTION MOULDING

Injection moulding is very widely used to produce precisely shaped plastic components in complex dies. As injection pressures are low it is possible to manufacture complex components, even some with internal screw threads, by the use of side cores and split tools. By mixing fine, typically less than 20 μm diameter, spherical metal powders with thermoplastic binders, metal filled plastic components can be produced with many of the features available in injection moulded plastics. After injection molding, the plastic binder material is removed to leave a metal skeleton, which is then sintered, at high temperature. Dimensional control can be exercised on the as-sintered component, as the injected density is sensibly uniform so shrinkage on sintering is also uniform. Shrinkage can be large, due to both the fine particle size of the powders and the substantial proportion of polymer binder used.

2.2 CONTROL OF POWDER PROPERTIES

By varying the several parameters: design and configurations of the jets, pressure and volume of the atomizing fluid, thickness of the stream of metal etc. it is possible to control the particle size distribution over a wide range. The particle shape is determined largely by the rate of solidification and varies from spherical, if a low heat capacity gas is employed, to highly irregular if water is used.

2.3 PROCESS SUITABILITY

In principle the technique is applicable to all metals that can be melted, and is commercially used for the production of iron, copper, including tool steels, alloy steels, brass, bronze and the low-melting-point metals, such as aluminium, tin, lead, zinc and cadmium. The readily oxidisable metals, for example chromium-bearing alloys, are being atomized on an increasing scale by means of inert gas, especially argon.

2.4 ADVANTAGES OF USING ATOMISED POWDER

Atomization is particularly useful for the production of alloys in powder form, since the constituent metals are fully alloyed in the molten state. Thus each powder particle has the same chemical composition. Additionally the process is used to produce compositions such as copper-lead, in which the lead, though soluble in the liquid state, comes out of solution on solidification. If a casting of such an alloy is made, serious segregation of the lead results, but if the liquid is atomized, the end product is copper powder containing a very fine and uniform distribution of lead inclusions within each powder particle.

2.5 APPLICATIONS OF POWDER PRODUCED BY P/M METHOD

METALLIC AND NON-METALLIC POWDERS

Direct Applications

- Magnetic powders for copiers, non-destructive inspection
- Powders for agriculture and food industry
- Powders for colorants
- Abrasive powders
- Powders for welding electrodes
- Powders for flame cutting
- Powders for optical applications

Sintered parts production

- Structural parts
- Auto-lubricating parts
- Sintered parts from special materials

Special and new, advanced materials production

- Metals of high melting point
- Composite materials particulate reinforced of:
- Metallic/inter-metallic/ceramic matrix
- Friction materials
- Wear resistant materials
- Refractory materials
  - Alloys and super alloys
  - Intermetallics
- Porous materials
  - Filters
  - Ion exchangers
  - Metallic foams
- Magnetic materials
  - Soft/hard
  - Dielectromagnetic
- Tool materials
  - High speed steels
  - Cemented Carbides
  - Metal matrix composites reinforced with carbides, nitrides, diamond
- Crystalline/amorphous materials

CHAPTER 3

LITERATURE REVIEW

J.B. Fogagnolo et al [2] studied that the homogeneous distribution of the reinforcement phase is a prime requisite for a composite material to present its superior performance. Powder metallurgy can produce composite materials in the whole range of matrix reinforcement composition, without the segregation typical of the casting process, and mechanical alloying serves to optimize the particle mixing stage, enhancing the reinforcement distribution. This work investigates the use of mechanical alloying plus hot extrusion to obtain Al6061 matrix composites reinforced with Si_3N_4, AlN and ZrB_2, and compares the result with the same composite materials obtained by more conventional powder metallurgy techniques. The incorporation of the reinforcement does not suffice to produce a significant improvement of the mechanical properties of the conventional powder metallurgy composites. Mechanical alloying breaks the reinforcement particle clusters, eliminates most of the defects present in these particles, decreases their size and enhances their distribution, which together with the metallurgical phenomena that change the metallic matrix, such as work hardening and oxide and carbide dispersion, produces an increase of about 150% in the hardness of the powder, when compared with the hardness of the as received, non-reinforced aluminium powder alloy; and of 100% in the hardness and ultimate tensile strength of the consolidated materials, when compared with material of same composition processed by conventional powder metallurgy.

According to R Cook et al [3] P/M aluminium components are attracting interest in an increasing variety of industries due to the possibilities for weight saving in engineering parts. There are many processes for manufacturing from powder feedstocks that are either in production, becoming commercialized or still undergoing development. The nature of these processes and the required properties of the end products mean that powders of different particle size, shape, composition and microstructure must be produced. The requirements of various processes requiring aluminium and aluminium alloy powders for metal matrix composites, laser sintering, powder forging and metal injection moulding are discussed in relation to powder particle size and structure. The key requirement of the powder manufacturer is to supply cost effective materials for these different processes. This may require compromises to be made by the supplier and consumer while the techniques evolve from development to large scale production.

D.P. Bishop et al [4] studied a diffusion based technique of microalloying aluminium powder metallurgy product, which was examined to expand the range of feasible alloying additions. Thermodynamic calculations and diffusion rates for several elements suggested that tin and silver were the most promising; these elements were successfully alloyed into AA 2014 on both macroscopic and microscopic scale. The final microstructures were examined using X-ray diffraction, X-ray mapping and energy-dispersive electron probe microanalysis. Silver additions were homogenized throughout the alloy microstructure, whereas tin was concentrated in intergranular regions only. The results suggested that the technique was viable for a variety of microalloying elements. Also, the extent of alloying was predicted reasonably well using a mathematical mass balance model.

R. Q Guo et al [5] prepared Aluminium fly ash mixtures containing different weight % of fly ash and compacted at pressures from 138-414 Mpa. The compacts prepared at 414 MPa were sintered in Nitrogen atmosphere at 600, 625 and 645 ^{o} C respectively. The time of sintering ranged from 0.5-6 h. The densification parameter and the green densities of the compacts were determined as a function of weight percent of fly ash particles. Volume changes during sintering of green compacts were also evaluated as a function of increasing fly ash weight percent. Microscopic studies of green and sintered compacts were done to study the effectiveness of sintering. Green and sintered densities of the compacts were found to decrease with increasing weight percents of fly ash. Sintering results in slight decrease in density and increase in volume of green compacts within the range investigated. Strength of the sintered compacts decreased with the increasing weight percent of fly ash under the present experimental conditions; however the hardness was found to increase slightly up to 10wt% fly ash, beyond which it decreased.

According to B. Wang et al [6] cracking is one of the key elements in the fracture process of particulate-reinforced metal-matrix composite (MMC) materials. The present study quantitatively examined the amount of new surface area created by particle cracking and the number fraction of cracked particles in SiC-reinforced aluminum-matrix composite materials. These composite materials were fabricated by liquid-phase sintering and contained 9 vol % of 23, 63, or 142 μm SiC. The matrix properties were varied by heat treating to either an underaged or peak-aged condition. In general, the new surface area created by particle cracking (S_V) and the number fraction of cracked particles (F_{no}) were linearly dependent on the local strain along the tensile specimen. Multiple cracks were frequently observed in the composites containing large particles. It was found that the new surface area created by particle cracking per unit strain was higher for the case of high-strength matrices and was not systematically affected by particle size within the range studied. The number fraction of cracked particles was affected by both particle size and matrix strength. A higher number fraction of particles cracked in the composites reinforced with large particles and with high matrix yield strengths. These results are interpreted in terms of the size of the particle defects, which is a function of particle size, and the critical flaw size necessary to crack a given particle, which is a function of the stress on the particle. The new surface area created by cracking and the fractions of cracked particles was related and are in good agreement for the large and medium sized particles.

Department of Metallurgy and Materials Engineering [7] at the Catholic University of Leuven in Belgium worked on Al-Si-X hypereutectic pre-alloyed powder by means of hot pressing at predefined temperature levels. It is generally accepted that hypereutectic aluminium-silicon (Al-Si) based alloys processed by the conventional ingot metallurgy route are difficult to deform into their final shape through processes such as extrusion because of the existence of coarse primary silicon particles and cavities in the matrix. However, hypereutectic aluminium alloys processed using pre-alloyed powders through the powder metallurgy route offer considerable flexibility in composition and shaping. They also have a fine and homogeneous microstructure. Pre-alloyed aluminium powders produced by atomization and rapid solidification offer the advantage of extended solubility for elements such as Si, Fe, Cr, Mn and Ni that typically have a limited solubility in conventionally solidified aluminium alloys. There are, however, limitations in efficient consolidation of pre-alloyed powders while retaining the refined microstructure. Another challenge is achieving good interparticle bonding between pre-alloyed particles during sintering because of the oxide film present on the surface of the powder particles. In this work they sought to determine by experiment the minimum load requirements to achieve full densification of a multi component Al-Si-X hypereutectic pre-alloyed powder by means of hot pressing at predefined temperature levels. They concluded that Consolidation of air-atomized Al-17Si-5Fe-3.5Cu-1.1Mg-0.6Zr (wt %) powder into fully dense compacts was possible by means of hot pressing.

Experimental work revealed that a mechanical load of 80 MPa was needed at 450°C, whereas 458 MPa was needed to fully densify the powder when heating up to350°C.The onset of densification was influenced by the packing of the starting powder compact and the load during hot pressing. The Vickers hardness of the compacted material slightly decreased as function of hot pressing temperature. Significant microstructural changes were observed after hot pressing, when compared to the starting powder.

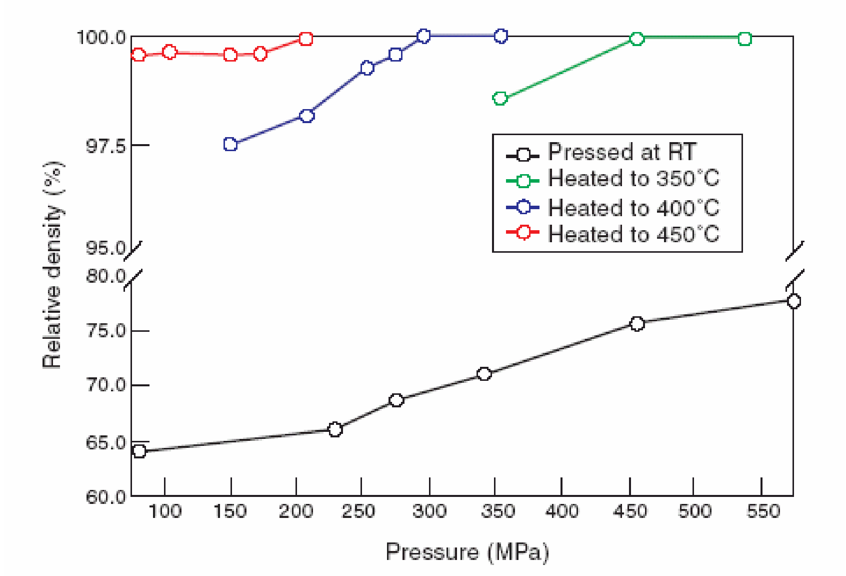


Figure 4: Variation of relative density with pressure for the Al-Si-X alloy powder during cold pressing.

The relative densities of various samples, densified using cold and hot pressing at different pressures, are shown graphically in Figure 4 and 5. A relative density of 80 per cent can be achieved by cold pressing when increasing the pressure up to 575 MPa, whereas relative density values above 98 per cent were measured under all investigated hot pressing conditions. Relative density as function of temperature during hot pressing is shown in Figure 6.

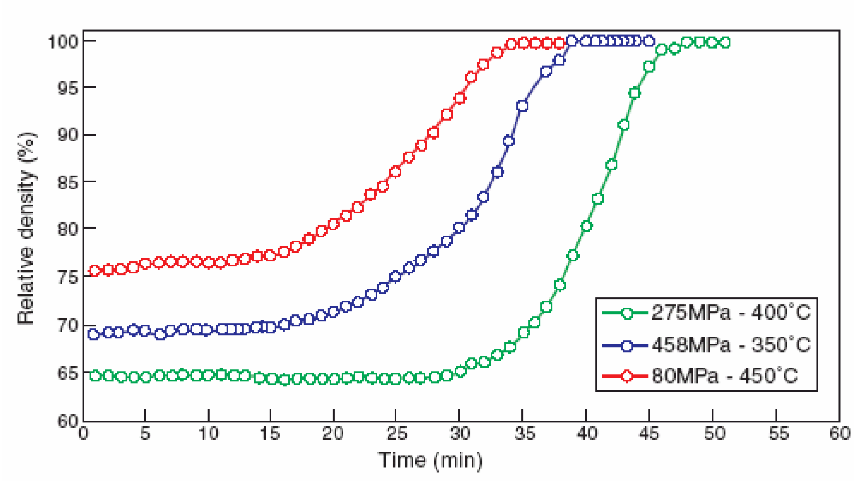


Figure 5: Variation of relative density with time during hot pressing

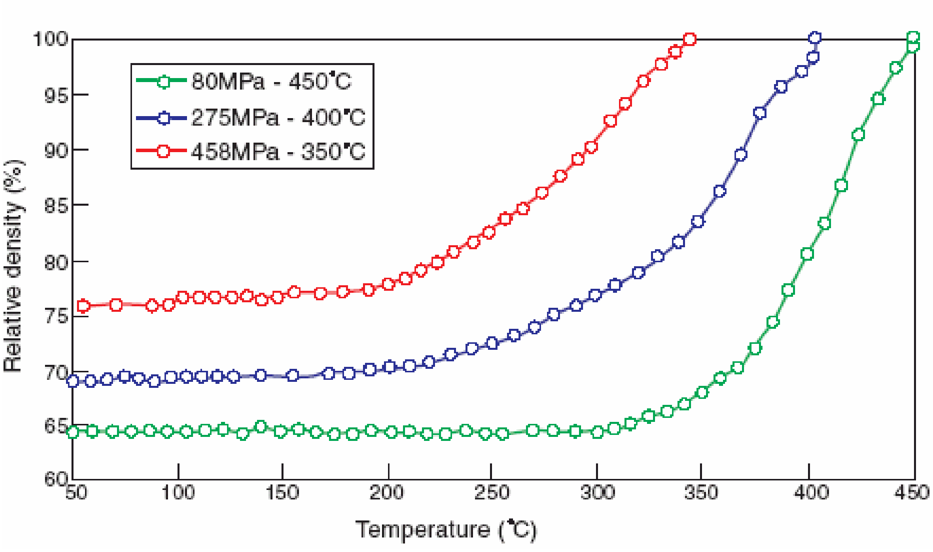


Figure 6: Relative density as function of temperature during hot pressing.

The densification rate in the second stage is faster at higher heating rates and hot pressing temperature. The overall deformation rate is higher at higher hot pressing temperature and lower mechanical load because of the lower flow stress of the material at high temperatures. This also due to the low startup relative density of the compacts pressed with lower load and high temperatures.

Jung-Sik Kim et al [8] gave a new fabrication process for producing micro Al-based alloy components. The process is based on micro powder injection moulding technology, while the micromoulds are produced using MEMS technology. The process involves (1) fabrication of micromoulds from SU-8 masters, which are produced using a UV photolithography process; (2) making metallic paste by mixing 80 wt% of ultrafine Al (2.5 μm in average) powder, 5 wt% of Cu nanopowder (less than 60 nm), and 15 wt% of adhesive binder in about 30 ml of acetone; (3) mould filling with metallic paste and demoulding; and (4) sintering of the moulded component in an Ar atmosphere. The proposed process has been used to sinter Al–Cu powder microcomponents successfully without the help of either high pressure compression or mixing Mg with the Al powder. This research proposes a new approach to fabricate 3D micro metallic components to meet the needs in applications where metal, rather than silicon, microcomponents are required.

R.L. Orban [9] described ways to improve densification without reducing the productivity by Warm Compaction and High Velocity Compaction.

Warm compaction is based on the notable decreasing of yield strength of the ferrite steels at heating. Consequently, deformability of particles and implicitly, compressibility of such powders notable increases even at a moderate heating (up to 150^{0}C), allowing a higher densification at the same applied pressure. A lubricant resistant at these temperatures, able to assure a good lubrication, without to decrease the powder flowing capacity, and also a heating system of both powder and tooling is necessary, instead. Several companies developed such lubricants and heating systems (Fig. 7).

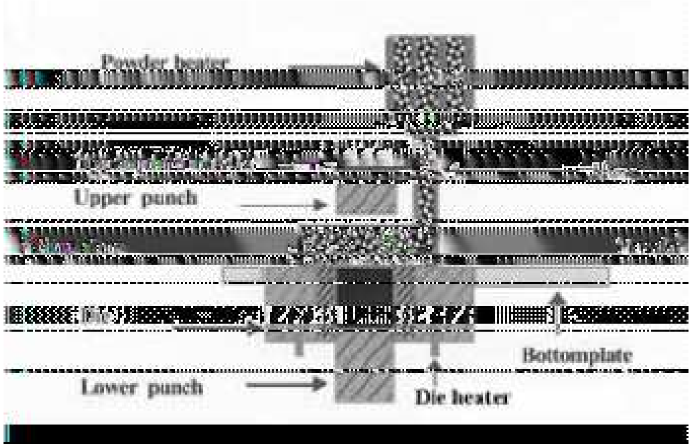


Figure 7: Schematic representation of a warm compaction system

Powders for warm compaction are usually delivered previously mixed with lubricant (e.g. Densmix [10]), assuring, at 150 ^{0}C and 700 MPa a porosity below ~3%. Investigations are performed to decrease the working temperature for the same densities obtaining, in order to improve die filling / increase tool life. Such a lubricant, working at 120 ^{0}C, was recently reported [11].

Although compaction by explosion (shock compaction) has been used for several years, it was not extended due to its difficulties and low productivity. The dynamic compaction idea was recently reconsidered; new technological variants, less dangerous and of high productivity, are being developed. So, Combustion Driven Compaction (CDC, Fig.8) uses the controlled release of energy from combustion of natural gas to compact powder. Another variant, High Velocity Compaction (HVC), realizes densification by intensive repeated shockwaves created by a hydraulically - operated hummer [12].

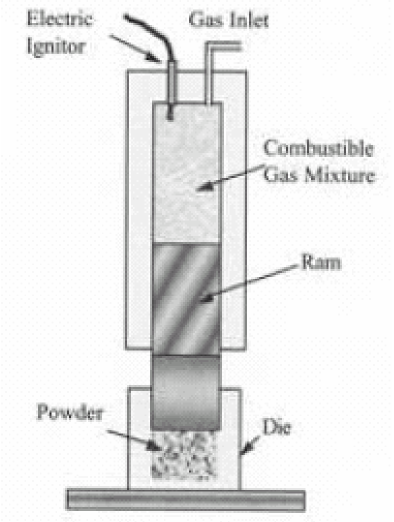


Figure 8: Schematic picture of the CDC process

Extending PM capability of complex parts processing

Although a notable progress in the complex PM parts production has been achieved by introducing the new computer controlled multi-level presses, realizing the compaction by a multi-punch die with separately operated punches, as this can compact only linear parts, researches are now focused on the complex 3-D parts realizing, especially by Powder Injection Molding/Selective Laser Sintering.

Selective Laser Sintering

Selective Laser Sintering (SLS) represents the latest development in the metal powder forming. It consists of monogranular powder layers successively realizing on the machine plate, able to sequentially move down by a step equal to the layer thickness (Fig.9). In each layer and between two successive layers, a laser beam, by a computer controlled sequential displacement in coordinates, joints particles upon a program to realize the required cross section of the part for that layer. The process is repeated until the whole part is “built”, layer-by-layer. At its end, the un-sintered particles are removed.

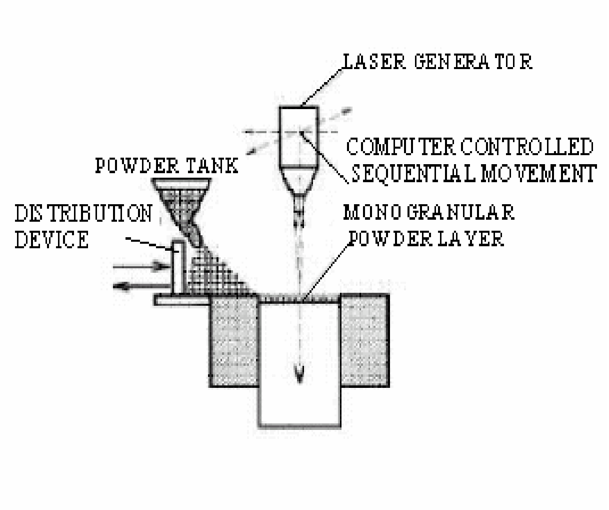


Figure 9: Schematic representation of SLS principle.

It is obvious that SLS allows large possibilities concerning the part geometry / dimensions (Figure 10) and processed materials. However, the obtained mechanical properties are modest and a supplementary sintering operation is usually necessary. Numerous researches are focused on SLS improving by the laser beam power/resolution/powder sinteribility increasing (e.g. by coating) [13]

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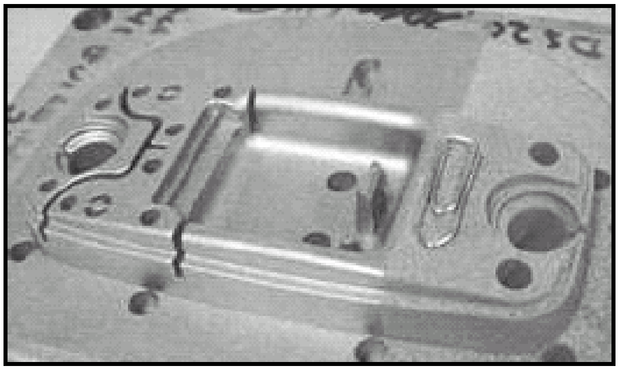
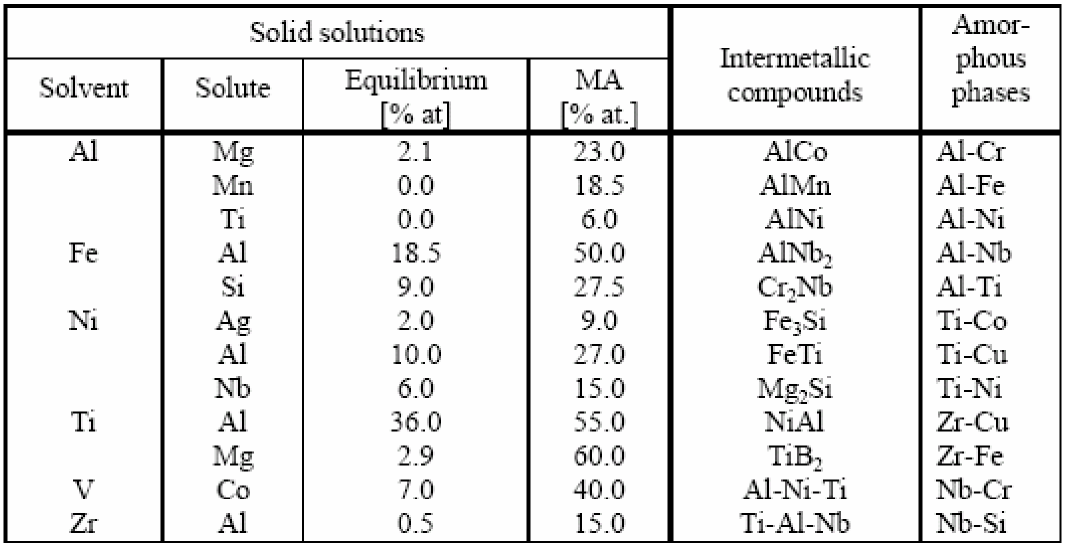


Figure 10: Example of a complex part realized by SLS

Mechanical Alloying

Mechanical Alloying (MA) is too a relatively new technology that enables a large variety of advanced materials obtaining. It consists of material milling in a high energy mill, process producing repeated powder particle deformation, work hardening, fracturing, clean surfaces forming, local heating, solid state welding, re-fracturing and so on, under the impact energy transferred to them from collisions of the chaotic moving balls [14]. Pure alloys/solid solutions of an extended solubility limit are obtained if components does not react each other, or if the reached temperature, produced by powder heating as a result of the mechanical energy of collisions partially transformation in heat, is too less for a possible reaction ignition. Intermetallics or other types of compounds are formed when components reacts together or with the milling atmosphere - if the ignition temperature of the reaction, T_{ig}, is reached by the particles. Finally, amorphous phases / nanocrystalline materials are obtained by a mechanical disordering at an enough long MA time. In Table below are given several examples of such advanced materials obtained by MA.

Table 1: Typical advanced materials produced by MA



Solid solutions				Intermetallic compounds	Amorphous phases
Solvent	Solute	Equilibrium [% at]	MA [% at.]		
Al	Mg	2.1	23.0	AlCo	Al-Cr
	Mn	0.0	18.5	AlMn	Al-Fe
	Ti	0.0	6.0	AlNi	Al-Ni
Fe	Al	18.5	50.0	$AlNb_2$	Al-Nb
	Si	9.0	27.5	Cr_2Nb	Al-Ti
Ni	Ag	2.0	9.0	Fe_3Si	Ti-Co
	Al	10.0	27.0	FeTi	Ti-Cu
	Nb	6.0	15.0	Mg_2Si	Ti-Ni
Ti	Al	36.0	55.0	NiAl	Zr-Cu
	Mg	2.9	60.0	TiB_2	Zr-Fe
V	Co	7.0	40.0	Al-Ni-Ti	Nb-Cr
Zr	Al	0.5	15.0	Ti-Al-Nb	Nb-Si

Intensive research are being performed for the MA process better understanding, its application to new components /nanocrystalline materials obtaining, for the resulted powder sintering to obtain bulk materials without to alter their microstructure, especially the nanocrystalline one, for the direct bulk MA processing technology and parameters establishment [15].

Vickers Hardness Test [16] is a standard method for measuring the hardness of metals, particularly those with extremely hard surfaces: the surface is subjected to a standard pressure for a standard length of time by means of a pyramid-shaped diamond. The diagonal of the resulting indention is measured under a microscope and the Vickers Hardness value read from a conversion table

Vickers hardness is a measure of the hardness of a material, calculated from the size of an impression produced under load by a pyramid-shaped diamond indenter. Devised in the 1920s by engineers at Vickers, Ltd., in the United Kingdom, the diamond pyramid hardness test, as it also became known, permitted the establishment of a continuous scale of comparable numbers that accurately reflected the wide range of hardnesses found in steels.

The indenter employed in the Vickers test is a square-based pyramid whose opposite sides meet at the apex at an angle of 136º. The diamond is pressed into the surface of the material at loads ranging up to approximately 120 kilograms-force, and the size of the impression (usually no more than 0.5 mm) is measured with the aid of a calibrated microscope. The Vickers number (HV) is calculated using the following formula:

$$HV = 1.854(F/D2),$$

with F being the applied load (measured in kilograms-force) and D2 the area of the indentation (measured in square millimeters). The applied load is usually specified when HV is cited.

The Vickers test is reliable for measuring the hardness of metals, and also used on ceramic materials. The Vickers testing method is similar to the Brinell test. Rather than using the Brinell's steel ball type indenter, and have to calculate the hemispherical area of impression, the Vickers machine uses a penetrator that is square in shape, but tipped on one corner so it has the appearance of a playing card "diamond". The Vickers indenter is a 136 degrees square-based diamond cone, the diamond material of the indenter has an advantage over other indenters because it does not deform over time and use. The impression left by the Vickers penetrator is a dark square on a light background. The Vickers impression is more easily "read" for area size than the circular impression of the Brinell method. Like the Brinell test, the Vickers number is determined by dividing the load by the surface area of the indentation (H = P/A). The load varies from 1 to 120 kilograms. To perform the Vickers test, the specimen is placed on an anvil that has a screw-threaded base. The anvil is turned raising it by the screw threads until it is close to the point of the indenter. With start lever activated, the load is slowly applied to the indenter. The load is released and the anvil with the specimen is lowered. The operation of applying and removing the load is controlled automatically.

Several loadings give practically identical hardness numbers on uniform material, which is much better than the arbitrary changing of scale with the other hardness machines. A microscope is swung over the specimen to measure the square indentation to a tolerance of plus or minus 1/1000 of a millimeter. Measurements taken across the diagonals to determine the area are averaged. The correct Vickers designation is the number followed "HV" (Hardness Vickers). The advantages of the Vickers hardness test are that extremely accurate readings can be taken, and just one type of indenter is used for all types of metals and surface treatments. Although thoroughly adaptable and very precise for testing the softest and hardest of materials, under varying loads, the Vickers machine is a floor standing unit that is rather more expensive than the Brinell or Rockwell machines.

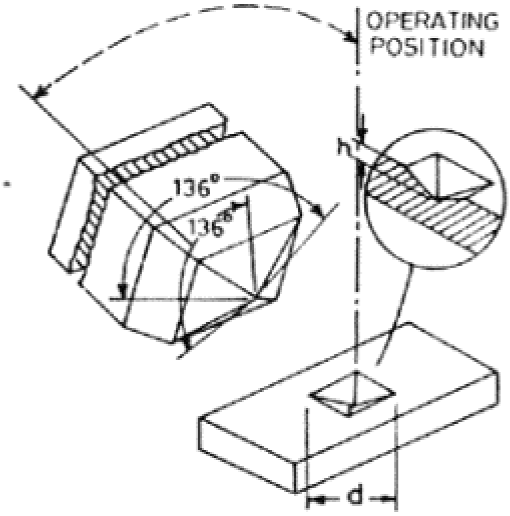


Figure 11: Vickers hardness Tester

J.P.Pathak et al [17] studied the compositions of conventional aluminium base alloys in which equal amounts of tin and lead as soft phase were incorporated separately. Impeller mixing and chill casting technique were employed for the preparation of the alloys. Mechanical properties of the as cast alloys were evaluated at room temperature. It is known that Al-Sn alloys have a very long history to be used as bearing materials. These alloys provide a good combination of strength and surface properties. The fatigue strength of cold worked and heat-treated Al-20% Sn-1% Cu alloy having reticular structure is close to that of Cu-30% Pb alloy with high seizure resistance. High Sn-Al alloys are used as linings bonded to a steel-backing strip. Aluminium-Tin alloys have good mechanical properties with conformability. It is well known that lead is more effective than tin as a soft phase alloying addition which confers the necessary antifrictional properties. A low modulus of elasticity is required in a bearing alloy to ensure good compatibility with the journal surface. Aluminium has a low modulus of elasticity and apart from indium, lead has the lowest of elasticity of all the soft phases alloying with aluminium. It has also been reported that that Al-Pb alloys are attractive and cheaper alternative to the commonly employed Al-Sn bearing alloys. However preparation of Al-Pb alloys is difficult due to solid insolubility of lead in aluminium and vice-versa. Wide miscibility gap in liquid state along with higher density difference and large solidification range make the alloy preparation very difficult. The aim of his investigation was to develop cheaper substitutes for the common but expensive bearing alloys, particularly, aluminium-tin alloys. To this end an attempt has been made to develop leaded aluminium alloys, which possess equivalent bearing property to that of conventional Al-Sn alloys.

According to D.P.Bishop et al [18], sintered aluminum alloys are an attractive material for the automobile industry, both because of the low specific gravity and high strength-to-weight ratio of aluminum itself, and the fabrication advantages associated with powder metallurgy process. However, properties such as impact, stiffness, and wear resistance are often poor, thereby restricting the widespread use of these materials. Recent work by the authors has shown that hardness, wear resistance and tensile properties of a P/M Al-Cu–Mg ternary master alloy can be improved using a novel diffusion/supersolidus liquid phase sintering process. Improvements were due to in-situ microalloying during sintering, in particular, the influence of Ag and Sn. To complement this work, the present investigation addresses the response of a commercial alloy AA2014, to the microalloying process. Results show that sintered densities for the commercial alloy were relatively unaffected by the presence of either Ag or Sn, and were superior to the ternary master alloy. Hardness and tensile properties were also improved relative to those obtained for the ternary, and were comparable to wrought 2014. Examination of final microstructure of Ag modified AA2014 using TEM showed the presence of Ω as the principal precipitate, but only after extended sintering times. This particular precipitate is believed to contribute to enhanced hardness. The apparent absence of Ω for short sintering times was due to the presence of silicon in the commercial product. However, the corrosion behavior of the P/M AA2014 was superior to the wrought product and thus the process is presented as a potential P/M alternative to using ingot metallurgy techniques for microalloying.

M.Zhu [19] et al, reported the effect of mechanical alloying (MA) on the improvement of wear properties of Al-Pb binary alloys. The alloys with Pb ranging from 10 to 18 wt.% were prepared by vacuum sintering mechanically alloyed Al-Pb powder. Optical microscope (OM), scanning electron microscope, X-ray diffractometer and Vickers hardness indentation were used to characterize the microstructure and mechanical properties of Al-Pb alloys prepared. Wear properties of the alloys were characterized by measuring the bulk wear, the wear rate and the wear coefficient under both dry and oil-lubricated test conditions. And the influence of Pb content was evaluated. The obtained results of wear properties of mechanically alloyed Al-Pb alloys were compared with those of similar alloys prepared by powder metallurgy and casting. The work showed that MA is an effective way of improving microstructure and consequently the wear properties of Al-Pb alloys.

P.P.Rodriguez [20] et al studied that Cu-Al-Ni shape memory alloys processed by powder metallurgy show very good thermo-mechanical properties, much better than those found in alloys produced by conventional casting. They presented the microstructural characterization of these P/M alloys in order to find the microscopic mechanisms, linked to the P/M processing method, which are indeed responsible of such good thermo-mechanical behavior. Electron microscopy studies [scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM)] show that powder metallurgy processing creates a sub-grain structure characterized by the presence of low angle sub-boundaries. These sub-boundaries are found to be lying on {1 1 0} and {1 1 2} lattice planes and are composed by an arrangement of superdislocations. These sub-boundaries may improve ductility in two ways: acting as a sink of dislocations that promotes plastic deformation and decreasing stress concentration at grain boundaries. Moreover, since sub-boundaries act as weak obstacles for the movement of martensite plates, the improvement on ductility is accomplished by an adequate thermo-mechanical behaviour.

According to Dr.O.P.Pandey [21] two commercial grade aluminium based immiscible alloys were spray-deposited using convergent-divergent type of nozzle. The microstructural features of spray-deposited and as-cast alloys are compared. In spray formed alloys equiaxed grains were observed. The cell boundaries and intercellular regions were observed to be decorated with sub-micron sized particles whereas in normal casting the second phase was observed to be segregated along the grain boundaries. Also the spray deposited alloys showed microstructural inhomogeneity at the edges and at the top surface of the deposit. The rest of the preform revealed uniformity in the microstructure. Also, it was observed that for uniformity of Sn in aluminium matrix, the size of the atomized droplets and fraction of solid phase in spray strongly influence the microstructure of the deposit.

CHAPTER 4

EXPERIMENTAL WORK

The details about the various followed in the preparation and characterization of the samples is discussed in this chapter. Powder metallurgy route was followed for the preparation of different samples.

The main steps associated with the preparation and analysis of samples are summarized below:

- Powder preparation
- Compacting
- Sintering
- Characterization and Testing
  -Microstructural Characterization
  -Hardness measurement

Alloy Composition:

Four different series of Aluminium alloys were selected for the present study.
These are: -

1. Al- 4.5%Cu-10%Sn
2. Al- 4.5%Cu-8%Sn
3. Al- 4.5%Cu-10%Pb
4. Al- 4.5%Cu-8%Pb

All compositions are in wt%.

The elemental powders used are of following purity: -

- Al (melting pt 660.32 °C, 99.9% purity)
- Cu (melting pt 1084.62 °C, 99.5% purity)
- Sn (melting pt 231.93 °C, 99.9% purity)
- Pb (melting pt 327.46 °C, 99.5% purity)

4.1 SAMPLE PREPARATION

4.1.1 POWDER QUANTITY

Equivalent quantities of the various metal powders were taken by weight. The weighing was done in a very accurate weighing balance (Sigma). 25 gm batches were prepared for each sample.

For e.g. for **Sample A** (Al- 4.5%Cu-10%Sn), the quantities of the metal powder taken as:

Al =21.375gm
Cu =1.125gm
Sn =2.5gm

Similarly,
Sample B (Al- 4.5%Cu-8%Sn)

Al =21.875gm
Cu =1.125gm
Sn =2 gm

Sample C (Al- 4.5%Cu-10%Pb)

Al =21.375gm
Cu =1.125gm
Pb =2.5 gm

Sample D (Al- 4.5%Cu-8%Pb)

Al =21.875gm
Cu =1.125gm
Pb =2 gm

4.1.2 DRY MIXING

Each of the above mentioned samples were then mixed manually in a mortar-pastel. This dry mixing was carried out for about one hour so as to get a consistent mixture of the different metal powders w.r.t. their compositions. No binder or any other lubricant was added to the powdered samples.

4.2 COMPACTION

In this step a die-punch was used for compaction of the blended and mixed powders. Pellets of 20mm diameter and approximately 3mm height were made by compacting powders in die-punch using a hydraulic press. The compaction pressure for each pellet was 5 Ton. This compaction pressure was applied for 2 minutes and then released. The well-mixed powders of a particular sample were first put in the die. The die was filled with approximately same quantity of powder for every pellet to be made. A Silicon spray was sprayed into the die before the powder was put in the dye so as to provide proper lubrication between the die walls and the powder used. Then the punch was placed over the powder in the dye, filled up to the brim. A pressure/load of 5 ton was then applied on the die-punch by a hydraulic press. The pressure was applied for about 2 minutes and was released after that the pellets were ejected out after compaction was over. For each composition 10 pellets were made.

The dimensions (thickness) of the various pellets were measured using a Vernier Calliper. Three different readings were taken for each pellet at various sites of the pellet and then an average of these was taken as the thickness of a particular pellet.

4.3 SINTERING

Sintering is the process whereby powder compacts are heated so that adjacent particles fuse together, thus resulting in a solid article with improved mechanical strength compared to the powder compact. This “fusing” of particles results in an increase in the density of the part and hence the process is sometimes called densification.

The density of the component can also change during sintering, depending on the materials and the sintering temperature. These dimensional changes can be controlled by an understanding and control of the pressing and sintering parameters, and components can be produced with dimensions that need little or no rectification to meet the dimensional tolerances.

Sintering of the pellets was done in a calibrated muffle furnace. Though a vacuum or an inert atmosphere is required during sintering, but this was substituted by using small cast iron chips that created a reducing atmosphere in the furnace, when the pellets were heated and hence oxidation was avoided.

In order to study the sintering characteristics, each pellet of a particular composition was heated at a desired temperature. The heating was done at a constant rate for each set of experiments followed by furnace cooling. The sintering temperatures for each set of experiment were in between 350 to 700^{o}C at an interval of 50^{o}C. Thus each pellet was heated at a desired temperature in the muffle furnace and held at that temperature for one hour followed by furnace cooling until the temperature dropped down to the room temperature. During heating the pellets were first covered by a layer of iron chips that created a reducing atmosphere when the pellets were being heated inside the furnace. Carbon in the cast iron chips provides the reducing atmosphere. Once the pellets cooled down and the temperature brought down to the room temperature, the pellets were taken out from the furnace.

4.3.1 CHANGE IN VOLUME

Change in volume was then measured by measuring the changed dimension of the heated pellet by using a Vernier Calliper. This change in thickness was used to calculate the change in volume percentage of the pellet, before and after the heating, by comparing the thickness of the pellet before and after heating. A table was plotted, one for each composition giving the detail about the temperature at which a particular pellet was heated, its original thickness, thickness after heating, then percentage change in volume. Not much change was observed in the case of diameters of the pellets after sintering. So it was taken to be constant for individual pellets before and after sintering.

4.4 MOUNTING

The heated samples were then mounted. Mounting of the pellets is done so as to enhance the handling of the pellets, which are subjected to various grinding and polishing operations during characterization. The steps involved in the mounting are as following:

1. A hollow cylindrical ring of iron with the inner diameter greater than 20mm (approximately 25mm) and height about 1 inch was taken. The inside surface of this ring was then made smooth by rubbing an emry paper at the inside surface. This was done to facilitate the removal of the mounted pellet.

Grease was applied to a flat surface. A pellet was placed over it. Then the ring, with grease applied over the inside surface of the ring to help in removing the mounted pellet, was placed around the pellet. An acrylic powder was used to mount the pellet. The powder was poured onto the pellet surrounded by the ring. A binder fluid was added drop by drop on the poured powder, until the powder was fully immersed. Then it was left for some time till the acrylic powder hardened around the pellet and formed a mount. The mounted pellet was then taken out of the ring by applying a little force from the top of the mounted pellet. The mounts prepared above had an uneven surface. So the mount was first held over a grinding machine with a moving belt to obtain a smooth surface.

4.5 GRINDING/POLISING

The mounted pellets were then taken for the grinding/polishing operation. Each mounted pellet was first rubbed over a polishing machine with emry papers of different grits over it. Because the pellets being Aluminium alloy, aluminium being soft, it is rubbed over the 320 grit emry paper for a small time. Then it was rubbed over an emry paper of 400 grit and then over a very fine emry paper of 600 grit for a considerable time in order to get a smooth and clear surface of the pellet.

The pellet was then polished on a fine polishing machine using a diamond polish. This was done to get a well polished and a smooth surface required for the further characterization of the samples.

Similarly all the pellets were polished for a considerable time, over and over again until a very fine and smooth surface was obtained.

The polished samples were the washed under water and then dried. Keller's Reagent was used as an etchant. The polished pellets were etched to get a defined microstructure. The pellets were immersed in the etchant for about 5-6 seconds, washed under a stream of water and then blow-dried.

Keller's Reagent was made with the following composition:-

1. 2ml HF (48%)
2. 3ml HCl (conc.)
3. 5 ml HNO_3 (conc.)
4. 190 ml H_2O

4.6 CHARACTERIZATION

4.1 Microctructural Characterization

4.2 Hardness Measurement

4.6.1 MICROCTRUCTURAL CHARACTERIZATION

The well-polished and etched samples were then observed under an optical microscope at different magnifications. Micrographs were taken at magnifications of 100X, 200X, and 400X for all the pellets of different samples.

4.6.2.HARDNESS MEASUREMENT

Vickers Hardness Test

It is the standard method for measuring the hardness of metals, particularly those with extremely hard surfaces: the surface is subjected to a standard pressure for a standard length of time by means of a pyramid-shaped diamond. The diagonal of the resulting indention is measured under a microscope and the Vickers Hardness value read from a conversion table.

Microhardness test was done for the various pellets using the Vicker's Hardness machine. The readings of the indent obtained were taken and the corresponding VHN was obtained from a conversion table provided with the machine. Three to five readings were obtained for each sample at different phases for each sample, while observing from the microscope in the Vicker's Hardness machine. A constant load of 50 gm was applied every time on different samples. The results obtained were then tabulated and a graph prepared by taking the temperature at which the pellet was heated against the VHN.

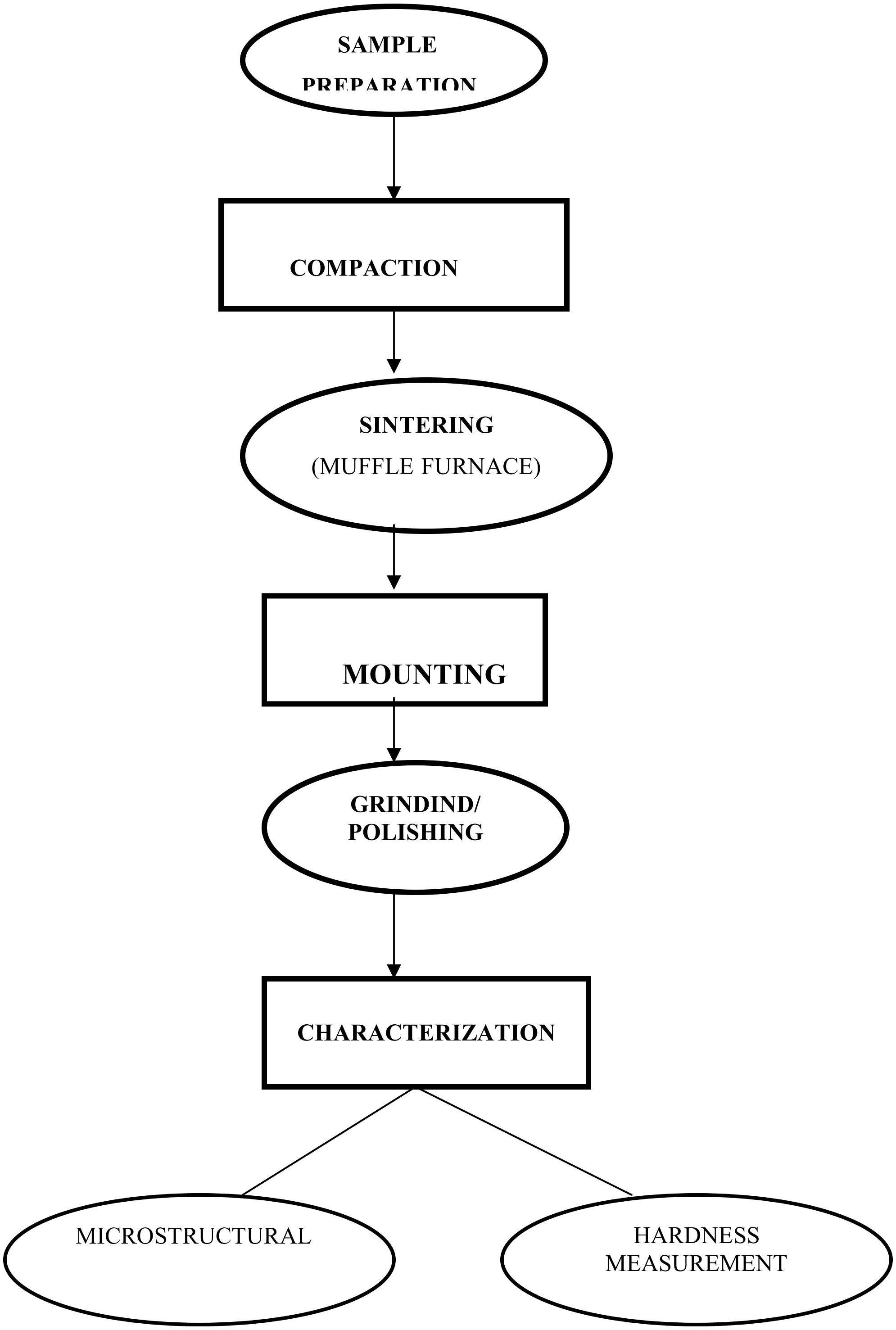


Figure 12: Flow Sheet showing the whole experimental work

CHAPTER 5

RESULTS AND DISCUSSION

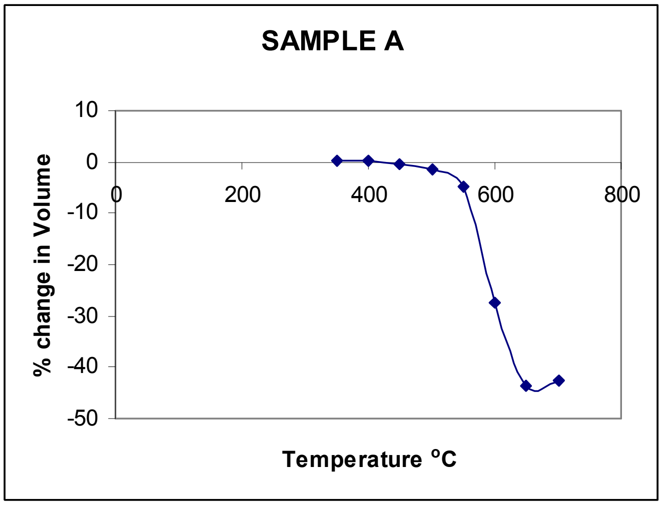
5.1 CHANGES IN VOLUME % WITH SINTERING AT DIFFERENT TEMPERATURES

Percentage change in volume after sintering the pellets at different temperatures was studied for all the samples of different series. The results are presented in tabular and graphical form for all the temperature ranges for each composition.

SAMPLE A: (Al- 4.5%Cu- 10%Sn)

Table 2: Change in Volume % on sintering at different temperatures for sample A

Pellet No.	Temp(^{0}C)	Pellet Thickness before sintering (mm)	Pellet Thickness after sintering for 1 hr (mm)	Change in volume (%)
A1	350	2.169	2.165	0.184
A2	400	3.008	3.004	0.132
A3	450	3.010	3.02	-0.33
A4	500	3.041	3.081	-1.31
A5	550	3.001	3.142	-4.69
A6	600	2.461	3.136	-27.42
A7	650	2.130	3.057	-43.52
A8	700	2.189	3.121	-42.57

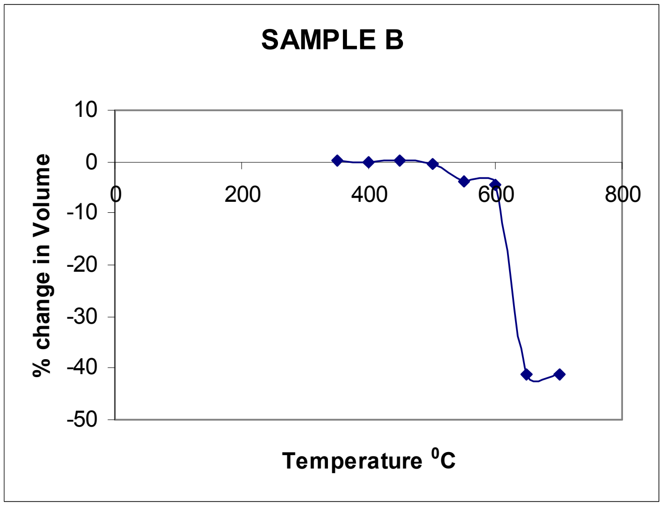


Graph 1: Change in Volume% (Y-axis) w.r.t temperature (X-axis)

SAMPLE B: (Al- 4.5%Cu- 8%Sn)

Table 3: Change in Volume % on sintering at different temperatures for sample B

Pellet No.	Temp(^{0}C)	Pellet Thickness before sintering (mm)	Pellet Thickness after sintering for 1 hr (mm)	Change in volume (%)
B1	350	3.028	3.021	0.231
B2	400	3.010	3.0093	0.033
B3	450	3.073	3.069	0.130
B4	500	3.042	3.056	-0.46
B5	550	3.065	3.18	-3.74
B6	600	3.014	3.153	-4.61
B7	650	2.172	3.070	-41.3
B8	700	2.162	3.054	-41.2

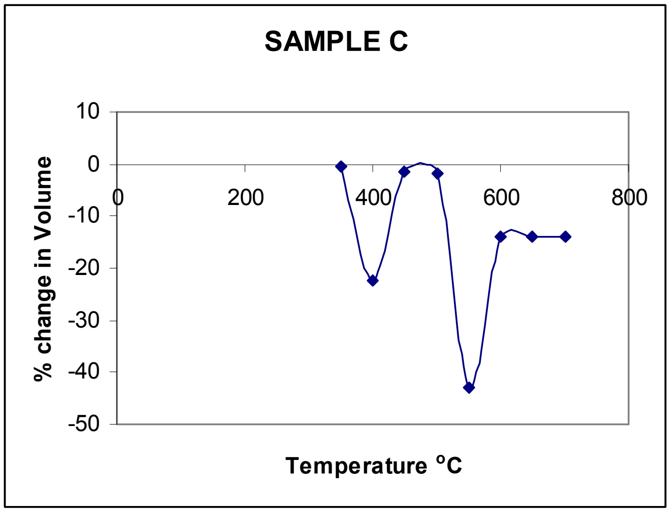


Graph 2: Change in Volume% (Y-axis) w.r.t temperature (X-axis)

SAMPLE C: (Al- 4.5%Cu- 10%Pb)

Table 4: Change in Volume % on sintering at different temperatures for sample C

Pellet No.	Temp(^{0}C)	Pellet Thickness before sintering (mm)	Pellet Thickness after sintering for 1 hr (mm)	Change in volume (%)
C1	350	3.062	3.078	-0.52
C2	400	2.458	3.006	-22.29
C3	450	3.02	3.06	-1.32
C4	500	3.018	3.069	-1.68
C5	550	2.16	3.088	-42.90
C6	600	3.017	3.442	-14
C7	650	3.030	3.449	-13.8
C8	700	2.734	3.118	-14.04

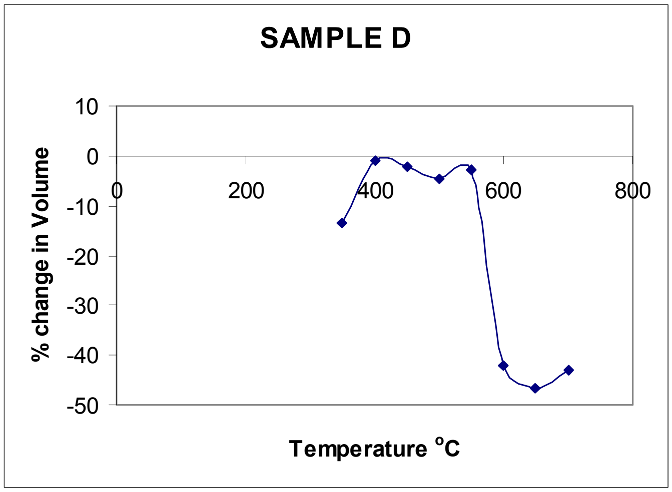


Graph 3: Change in Volume% (Y-axis) w.r.t temperature (X-axis)

SAMPLE D: (Al- 4.5%Cu- 8%Pb)

Table 5: Change in Volume % on sintering at different temperatures for sample D

Pellet No.	Temp(^{0}C)	Pellet Thickness before sintering (mm)	Pellet Thickness after sintering for 1 hr (mm)	Change in volume (%)
D1	350	2.144	2.432	-13.4
D2	400	2.106	2.125	-0.902
D3	450	2.096	2.142	-2.19
D4	500	2.097	2.129	-4.525
D5	550	2.12	2.177	-2.68
D6	600	2.116	3.009	-42.2
D7	650	2.125	3.116	-46.63
D8	700	2.108	3.014	-43



Graph 4: Change in Volume% (Y-axis) w.r.t temperature (X-axis)

Results:

The most important feature of sintering studies for all the samples containing Sn and Pb is that a slight contraction in volume in the initial stage of sintering i.e. in the temperature range of 350-400 °C or even in some cases upto 450 °C for the samples containing Sn was observed. This behavior was observed to change drastically after raising the temperature upto 700 °C, leading to expansion in the samples. Since Sn does not form any solid solution with aluminium and remains in the liquid state, this behavior is quite obvious. Moreover the Al_2O_3 layer which exists at the surface of Al, exhibit poor bonding with tin metal. The reducing atmosphere provided by the iron turnings (chips) is not sufficient enough to reduce the existing oxides into metallic form.

For samples containing Pb, in the initial stage the behavior observed was quite different as compared to samples containing Sn. It was observed that a drastic increase in volume followed by drastic contraction for the sintering temperature of 400 to 500°C. After that the sample again undergoes an expansion in volume for the sintering temperature of 550 °C. This typical characteristic was the dominating one for the samples containing higher amounts of Pb. Initially an expansion occurs at 400 °C because of the melting of Pb. The viscosity of Pb melt is on a higher side and does not allow it to fill the pores. So, it remains in the matrix as such. However, at higher temperatures, viscosity reduces sufficiently to allow the melt to flow into the pores, leading to a decrease in volume. As the temperature is increased, A sharp increase in volume is because of the oxidation of Pb, which is a common feature for both the samples containing lead.

5.2 MICROHARDNESS TEST

Microhardness of each sample was measured in the Al rich phase (white). The reason for this was to see the feasibility of providing hard matrix with proper distribution of the softer phase into it. Hardness measurement was done to see the feasibility of this material as a bearing material as hardness and wear characteristics are related to each other upto a greater extent. However, slight deviation in these interdependent characteristics exists for the samples that are being tested at different loads. A variation in hardness was observed for the samples sintered in the entire range of temperature.

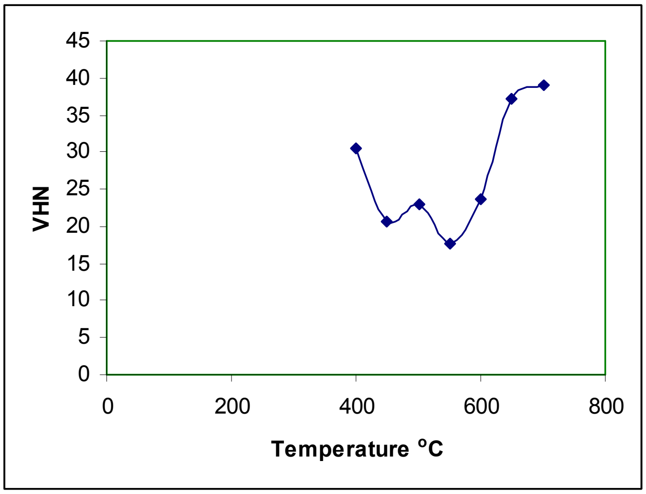
The readings obtained for various samples by using the Vicker's Hardness testing machine are tabulated below:

<u>**SAMPLE A:**</u> <u>**(Al- 4.5%Cu- 10%Sn)**</u>

Table 6: Change in VHN w.r.t. Sintering Temperature for sample A

PELLET NO.	SINTERING TEMPERATURE ^{o}C	AVERAGE DIA d	CORRESPONDING VHN	AVERAGE VHN
A1	350	no proper indent obtained	-	-
				30.45
A2	400	59	26.6	
		52	34.3	
				20.7
A3	450	67	20.7	
				23
A4	500	60	25.8	
		63.5	23	

		68	20.1	
				17.63
A5	550	74.5	16.7	
		78	15.2	
		66.5	21	
				23.7
A6	600	62.5	23.7	
				37.3
A7	650	57	28.5	
		54.5	31.2	
		53	33	
		40.5	56.5	
				39.1
A8	700	54.5	31.2	
		51.5	35	
		42.5	51.3	

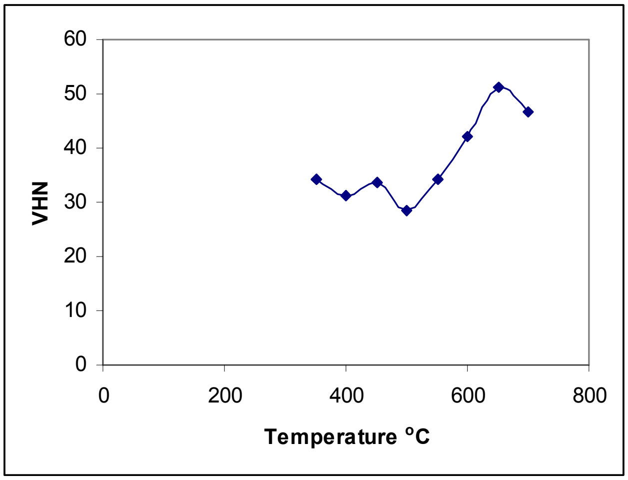


Graph 5: Variation in Microhardness wrt change in sintering temperature for Sample A

SAMPLE B: (Al- 4.5%Cu- 8%Sn)

Table 7: Change in VHN w.r.t. Sintering Temperature for sample B

PELLET NO.	SINTERING TEMPERATURE ^oC	AVERAGE DIA D	CORRESPONDING VHN	AVERAGE VHN
B1	350	53	33	34.1
		49	38.6	
		55	30.7	
				31.175
B2	400	53	33	
		57.5	28	
		55	30.7	
		53	33	
				33.7
B3	450	55.5	30.1	
		49	38.6	
		53.5	32.4	
				28.46
B4	500	59	26.6	
		53	33	
		60	25.8	
				34.26
B5	550	52.5	33.6	
		52.5	33.6	
		51	35.6	
				42.03
B6	600	46.5	42.9	
		48.5	39.4	
		46		
				51.36
B7	650	47.5	41.1	
		40.5	56.5	
		40.5	56.5	
				46.57
B8	700	45.5	44.8	
		40.5	56.5	
		48	40.2	
		45.5	44.8	



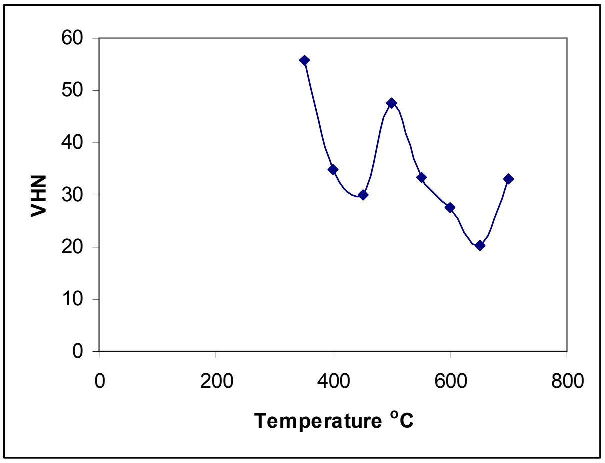
Graph 6: Variation in Microhardness wrt change in sintering temperature for Sample B

SAMPLE C: (Al- 4.5%Cu- 10%Pb)

Table 8: Change in VHN w.r.t. Sintering Temperature for Sample C

PELLET NO.	SINTERING TEMPERATURE °C	AVERAGE DIA D	CORRESPONDING VHN	AVERAGE VHN
C1	350	40.5	56.5	55.9
		39	61	
		43	50.2	
				34.9
C2	400	47.5	41.1	
		51	35.6	
		57.5	28	
				30
C3	450	53	33	
		63	23.4	
		52.5	33.6	

C4	500	40	58	47.6
		46	43.8	
		47.5	41.1	
				33.3
C5	550	45	45.8	
		58.5	27.1	
		56.5	29.1	
		54.5	31.2	
				27.5
C6	600	57	28.5	
		63.5	23	
		54.5	31.2	
				20.3
C7	650	62	24.1	
		67.5	20.3	
		74.5	16.7	
				33.1
C8	700	50	37.1	
		56.5	29.1	

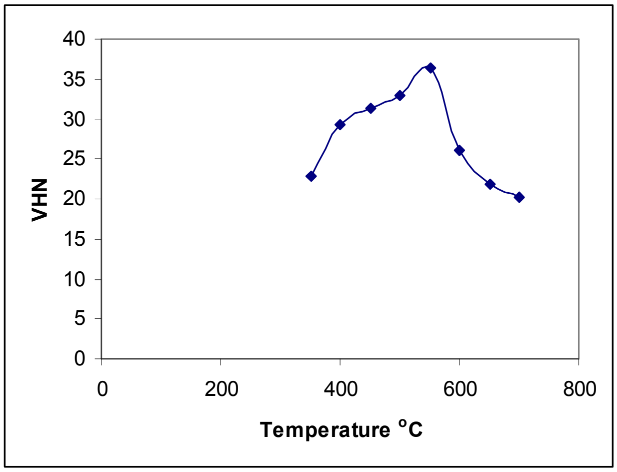


Graph 7: Variation in Microhardness wrt change in sintering temperature for Sample C

<u>Sample D:</u> <u>(Al- 4.5%Cu- 8%Pb)</u>

Graph 8: Variation in VHN wrt change in sintering temperature for Sample D

PELLET NO.	SINTERING TEMPERATURE ^{o}C	AVERAGE DIA D	CORRESPONDING VHN	AVERAGE VHN
D1	350	64.5	22.3	22.9
		63.5	23	
		63	23.4	
				29.25
D2	400	60	25.8	
		53.5	32.4	
		53	33	
		60	25.8	
				31.27
D3	450	51	35.6	
		60.5	25.3	
		54	31.8	
		53.5	32.4	
				33.03
D4	500	52	34.3	
		53.5	32.4	
		53.5	32.4	
D5	550	51	35.6	36.36
		50	37.1	
		50.5	36.4	
				26.06
D6	600	68	20.1	
		57	28.5	
		56	29.6	
				21.09
D7	650	63.5	23.0	
		68	20.1	
		64	22.6	
				20.3
D8	700	67.5	20.3	
		68.5	19.8	
		66.5	21	



Graph 8: Variation in Microhardness wrt change in sintering temperature for Sample D

Results:

For samples containing Sn, the hardness was observed to decrease initially with an increase in the sintering temperature followed by an increase in the hardness at high temperature for both the samples containing Sn. The decrease in hardness is because of the fact that initially Sn remains as a bigger droplet in the matrix of Al, due to its melting. At higher temperatures, oxidation followed by coalescence of these droplets leads to a slight increase and a decrease in hardness upto 600 °C. Once the sintering temperature is increased beyond 600 °C, the hardness was found to increase rapidly because at higher temperature, the oxidation phenomenon of both Al and Sn dominates in nature, which was also confirmed in the microstructure studies, being discussed in the next section.

A similar characteristic in the behavior of hardness was also observed for the samples containing Pb. The important feature in these samples containing lead was that the hardness at higher sintering temperature was observed to decrease. This behavior indicates that Pb exhibits better wettability as compared to samples containing Sn.

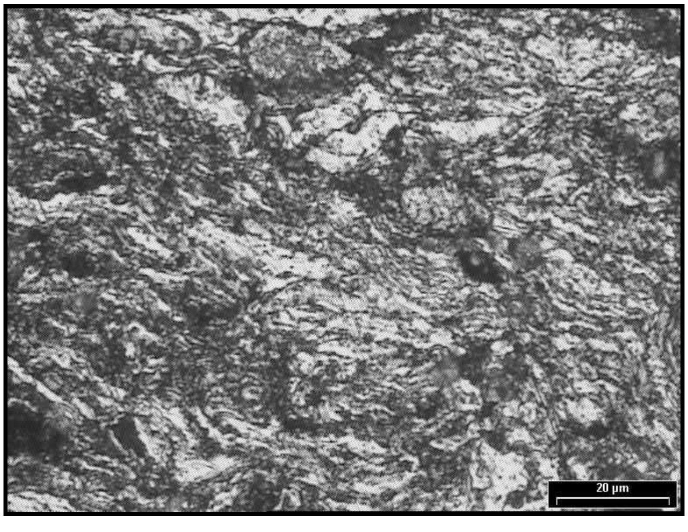
5.3 MICROSTRUCTURAL ANALYSIS

Microstructural features are the key features that provide the backbone for the wear characteristics of all the materials. A detailed study of the microstructural features of all the samples was carried out for the samples sintered at different temperatures.

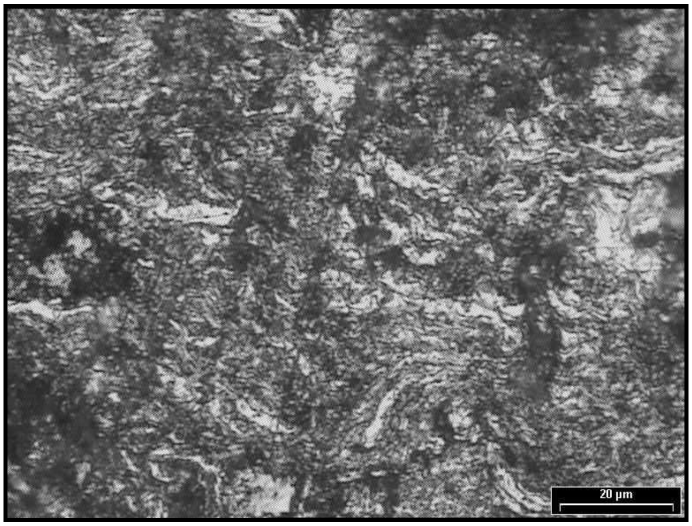
5.3.1 MICROSTRUCTURAL ANALYSIS OF SAMPLES CONTAINING TIN

In order to compare the structural features, the micrographs taken at higher magnification are presented here. The typical feature observed in each sample is that flow pattern was observed for each sample. This flow pattern is because of the fine Al flakes that were taken as the raw material for this study. The micrographs indicate that a uniform distribution of the second phase (Sn) exists for all samples sintered at different temperatures. Moreover this flow pattern is also because of the unidirectional compaction of the powder mass. As the sintering temperature was increased, a grain growth was observed. This growth phenomenon was more prominent for the samples sintered at 600 ^{0}C and above. Since Cu was also used (4.5%) to provide a harder matrix. So this growth phenomenon also dominates because of the diffusion of Cu in the matrix.

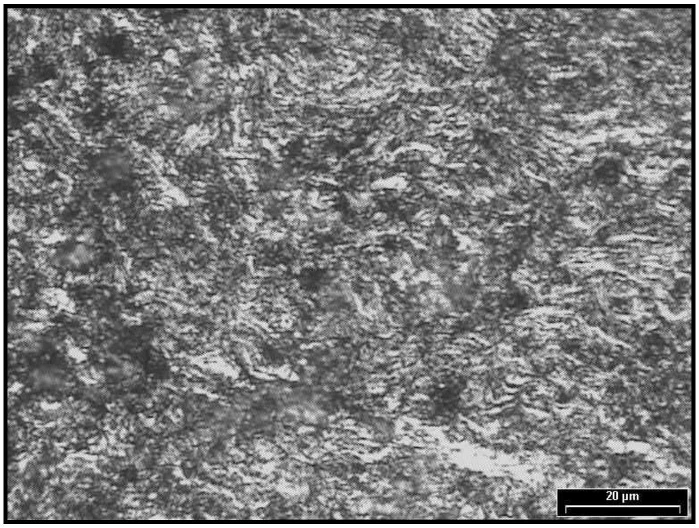
5.3.1.1 OPTICAL MICROGRAPHS FOR SAMPLE A (Al- 4.5%Cu- 10%Sn)



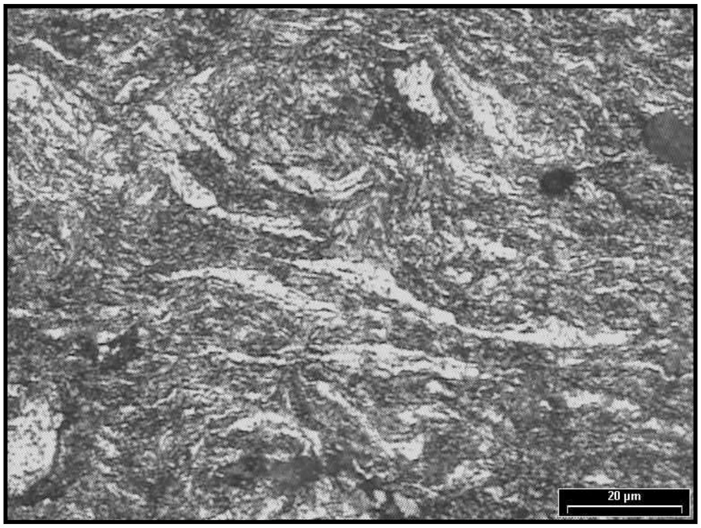
Sample A1: Sintering temperature 350 ^{0}C



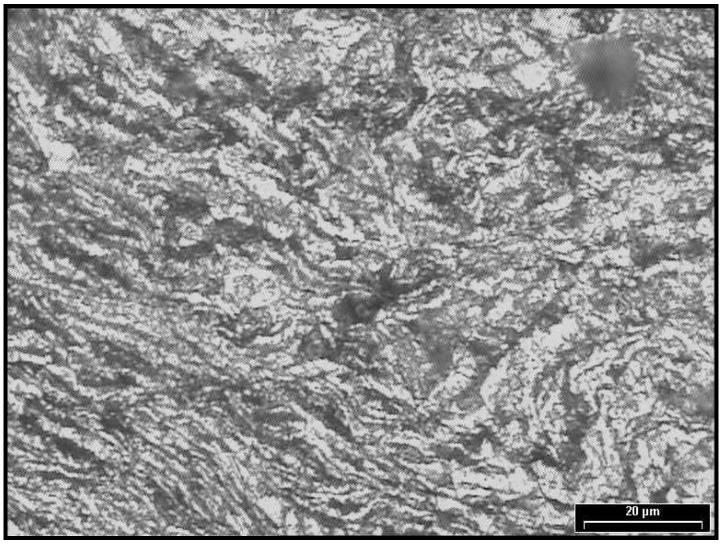
Sample A 2: Sintering temperature 400^{0}C



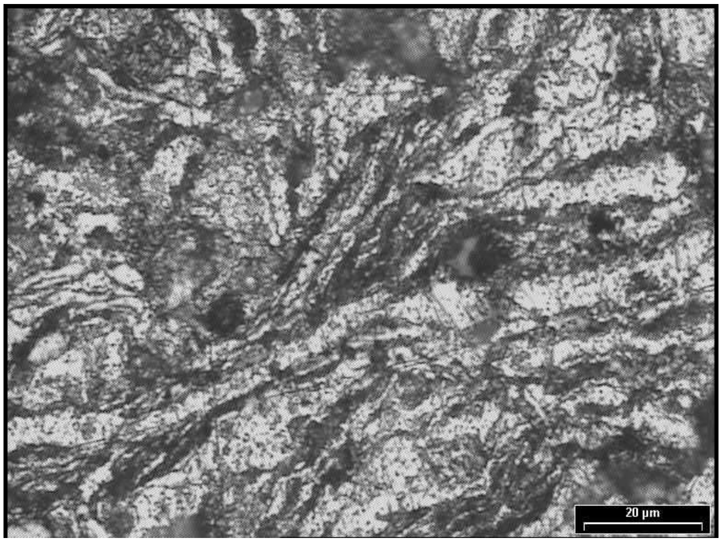
Sample A 3: Sintering temperature 450^{0}C



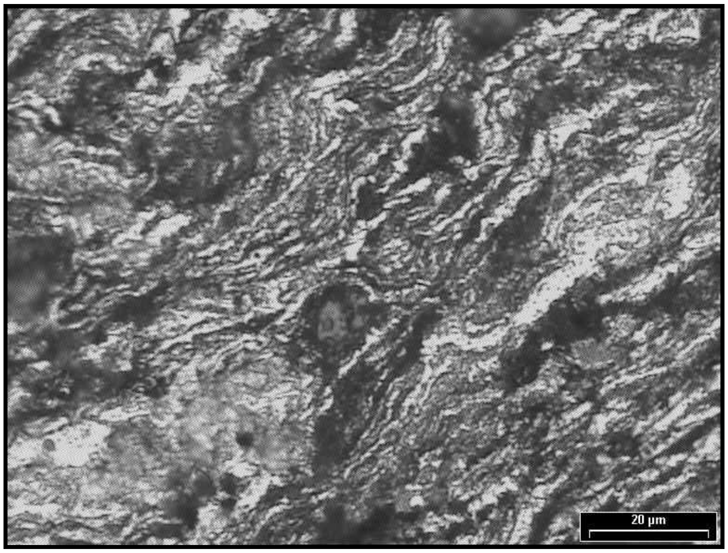
Sample A 4: Sintering temperature 500^{0}C



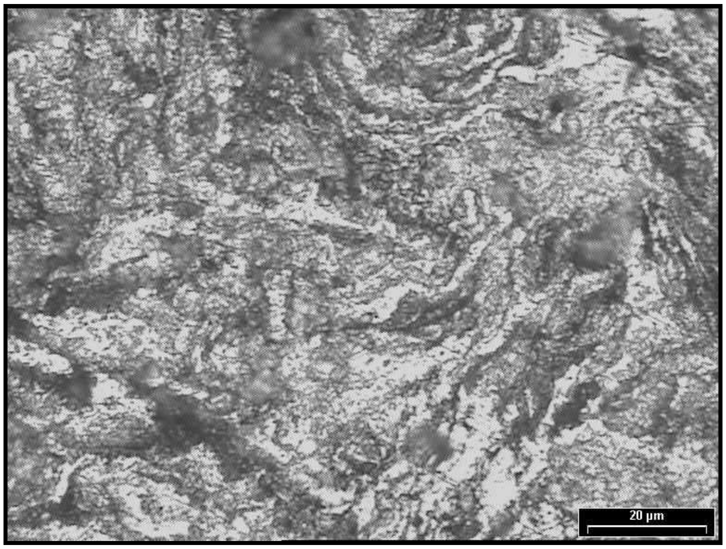
Sample A 5: Sintering temperature 550^{0}C



Sample A 6:Sintering temperature 600 ^{o}C

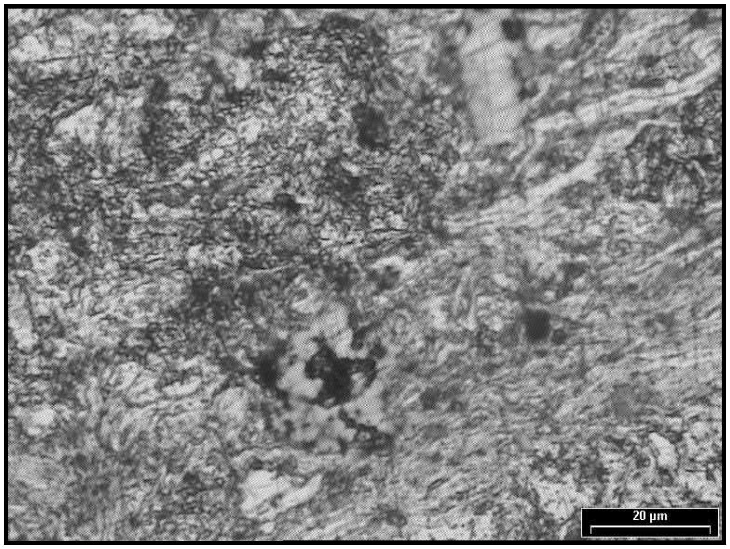


Sample A 7 Sintering temperature 650 ^{0}C

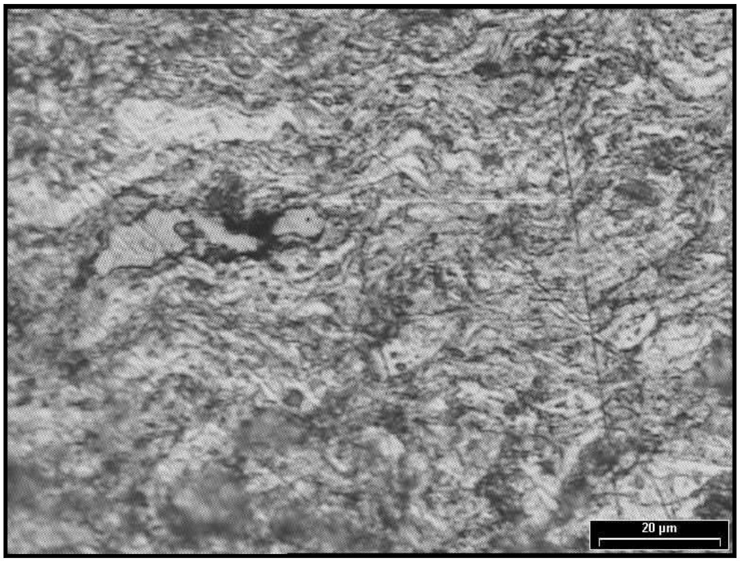


Sample A8: Sintering temperature 700 ^oC

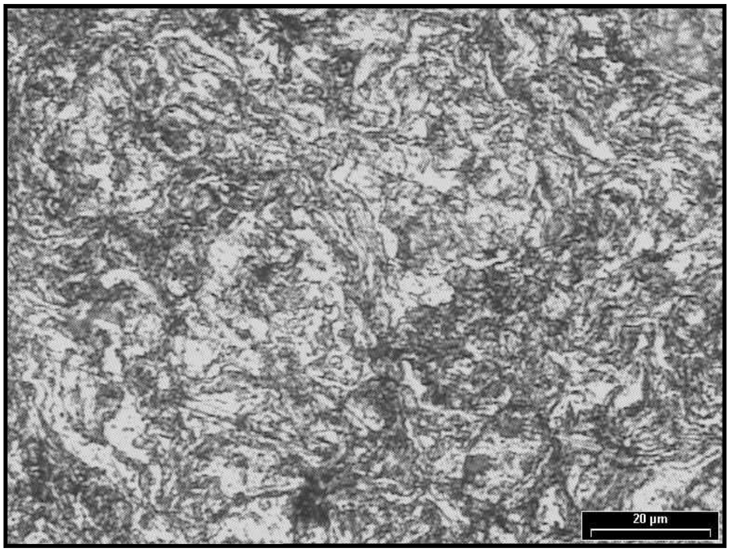
5.3.1.2 OPTICAL MICROGRAPHS FOR SAMPLE B (Al- 4.5%Cu- 8%Sn)



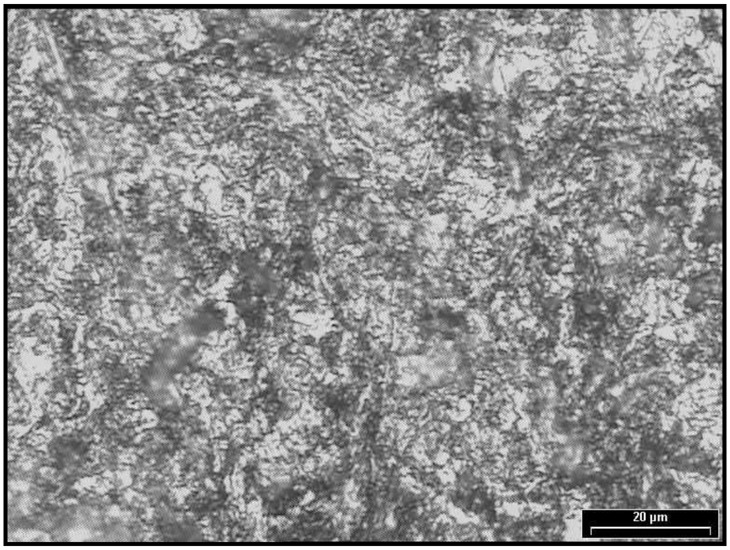
Sample B1: Sintering temperature 350 ^oC



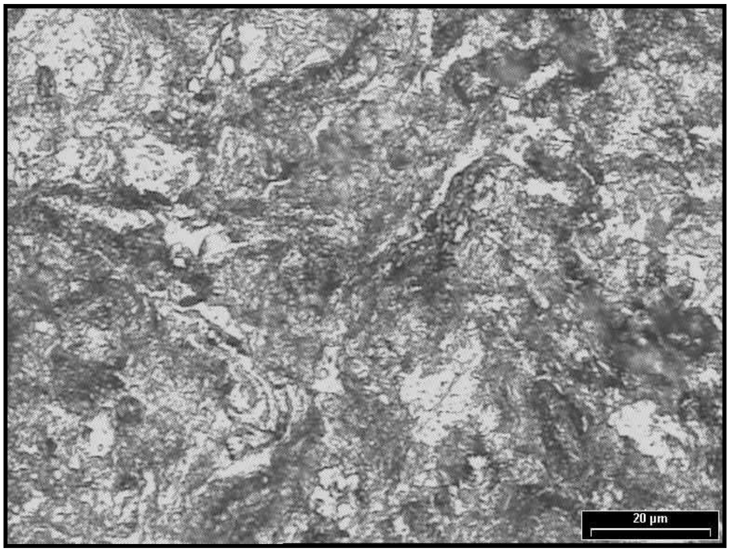
Sample B2: Sintering temperature 400^{o}C



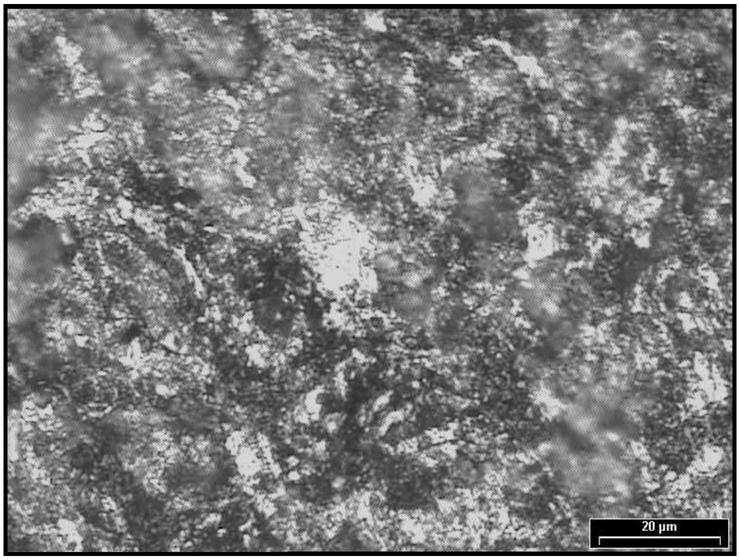
Sample B3: Sintering temperature 450 ^{o}C



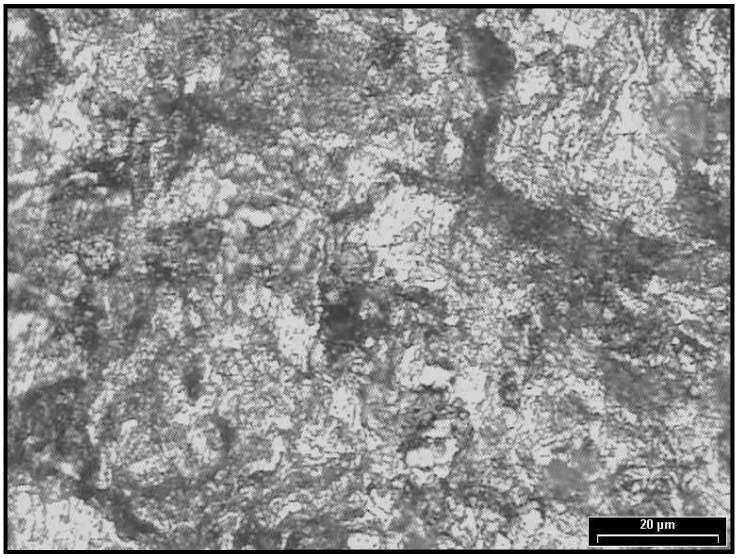
Sample B4: Sintering temperature 500 ^oC



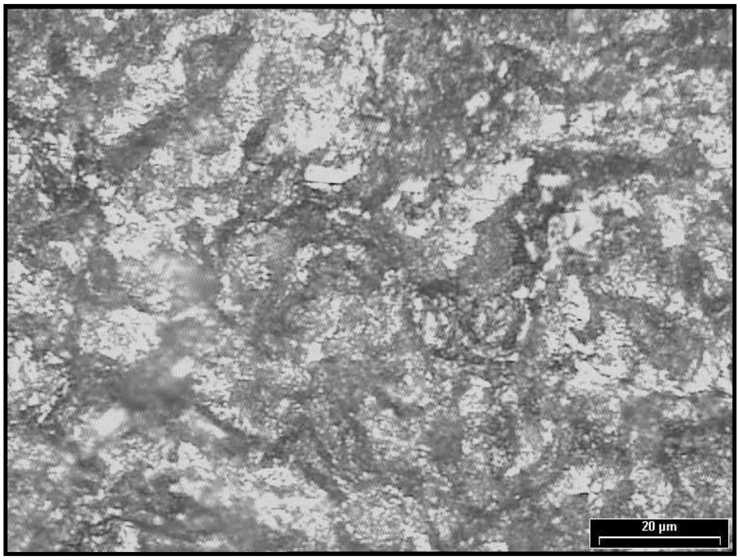
Sample B5: Sintering temperature 550 ^oC



Sample B6: Sintering temperature 600 °C



Sample B7: Sintering temperature 650 °C



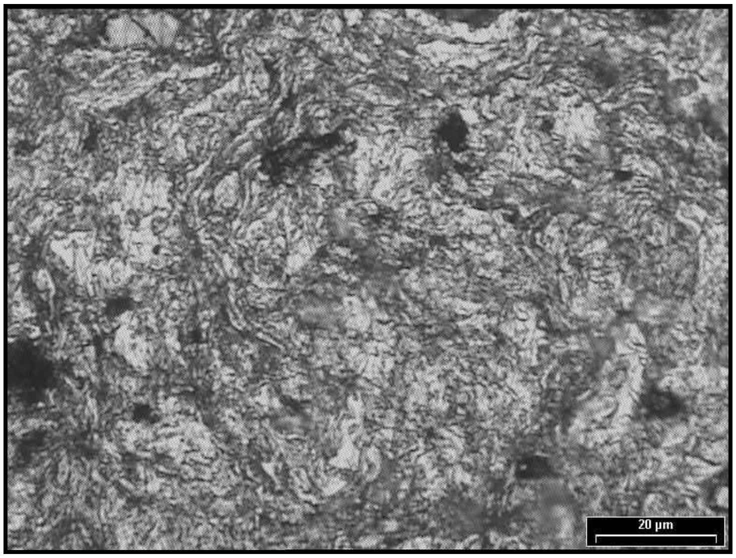
Sample B8: Sintering temperature 700 °C

5.3.2 MICROSTRUCTURAL ANALYSIS OF SAMPLES CONTAINING LEAD

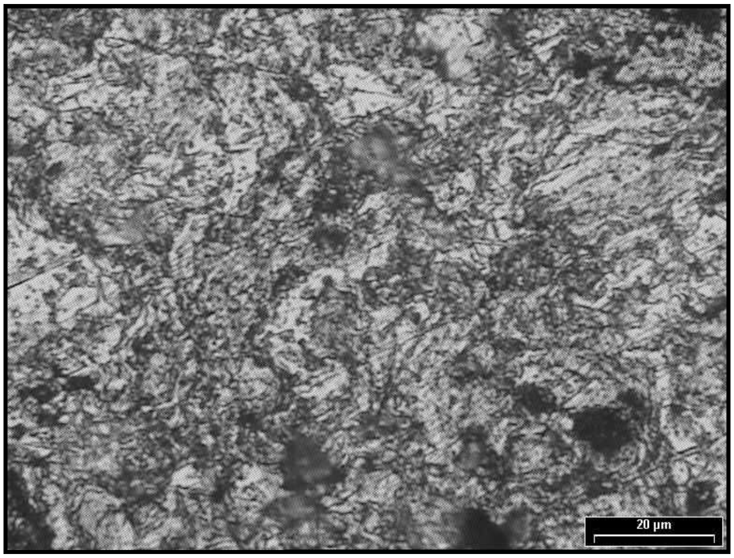
Here also the structural features observed are similar to that of samples containing Sn. Moreover, the distribution of lead particles in the matrix of Al is more uniform as can be seen on samples C5 and C8. The fine distribution of lead was observed even for samples sintered at higher temperatures. This particular feature exists for both the samples containing 10% and 8% Pb respectively. This particular feature is more important for the development of good bearing material as lead can provide better wear characteristics by the smearing action, which leads to provision of uniform thin film at the interphase.

The hardness characteristics and the structural features indicate that Pb can act as a better bearing material as compared to Sn.

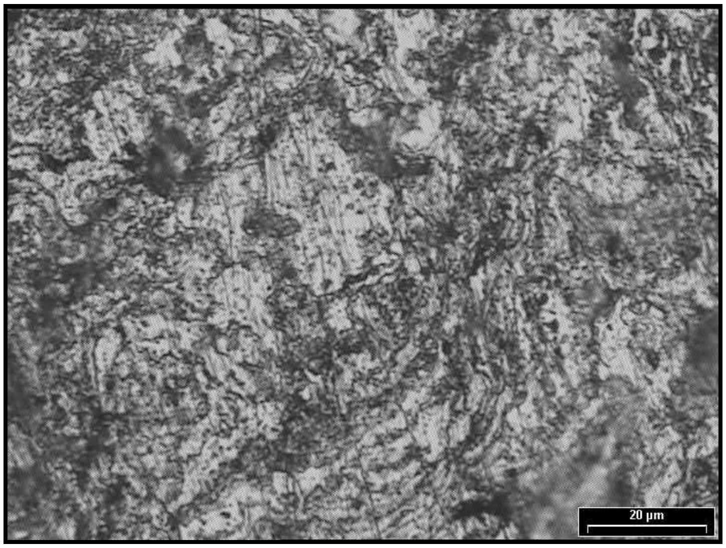
5.3.2.1 OPTICAL MICROGRAPHS FOR SAMPLE C (Al- 4.5%Cu- 10%Pb)



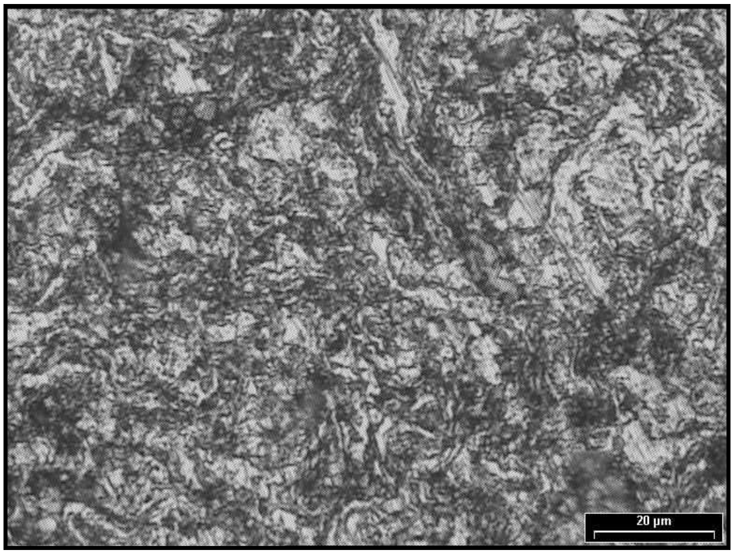
Sample C1: Sintering temperature 350 ^{o}C



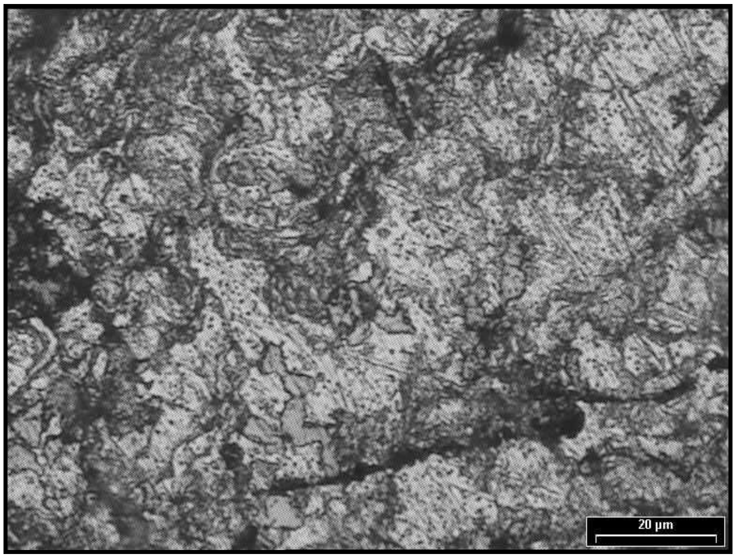
Sample C2: Sintering temperature 400 ^{o}C



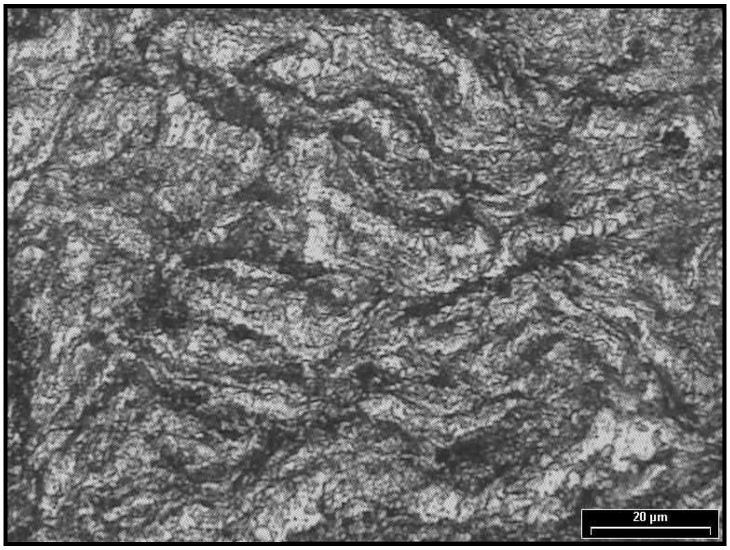
Sample C3: Sintering temperature 450 ^{o}C



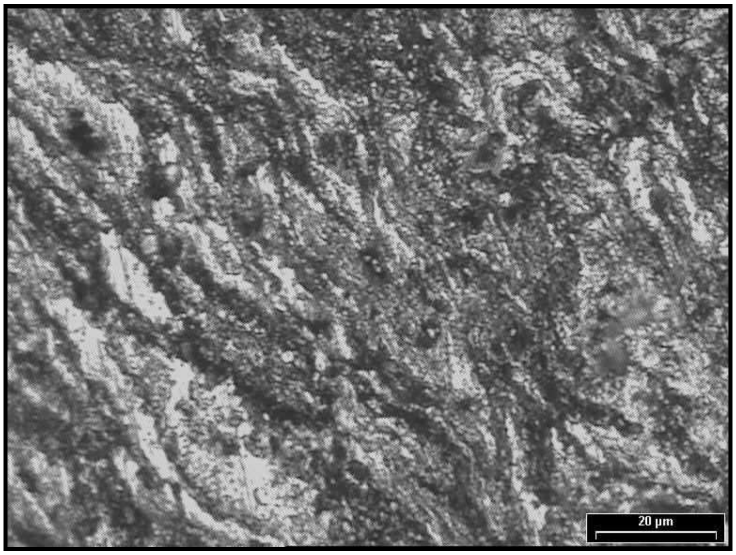
Sample C4: Sintering temperature 500 ^{o}C



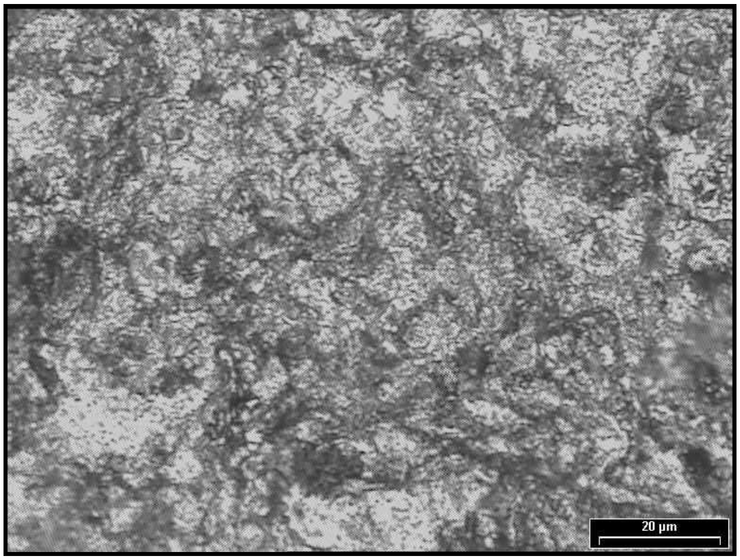
Sample C5: Sintering temperature 550 ^{o}C



Sample C6: Sintering temperature 600 ^{o}C

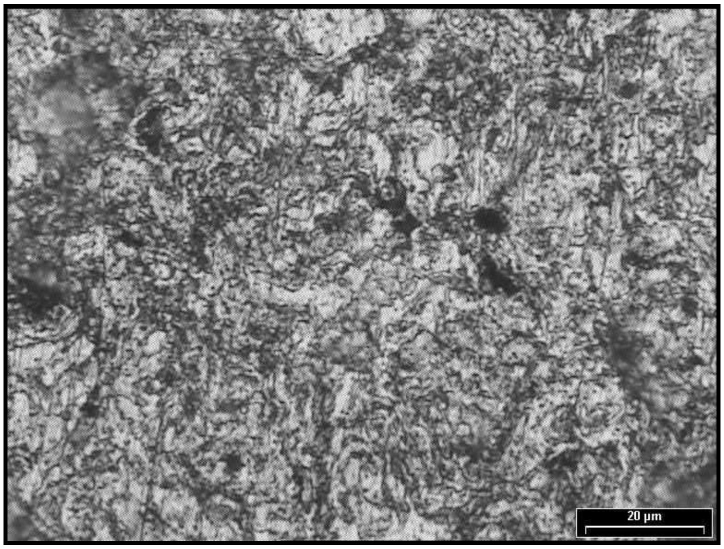


Sample C7: Sintering temperature 650 °C

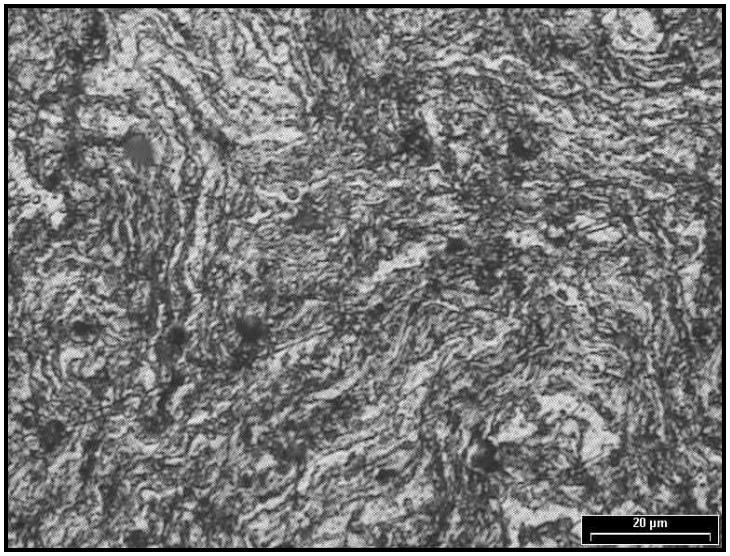


Sample C8: Sintering temperature 700 °C

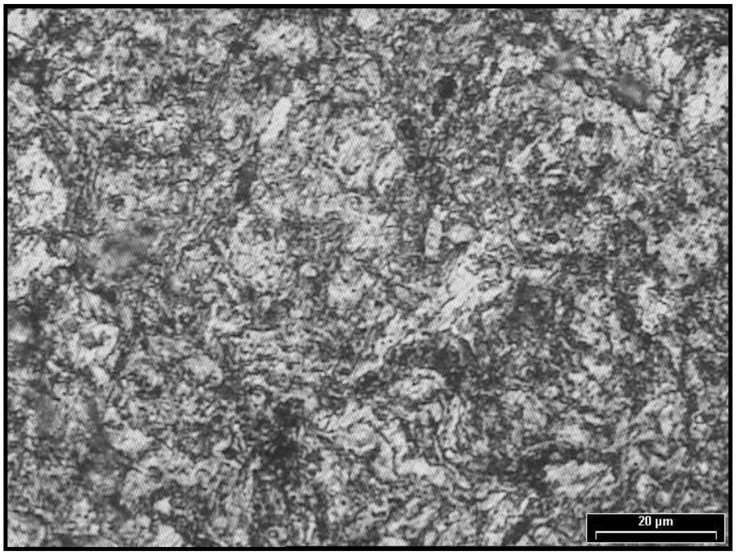
5.3.2.2 OPTICAL MICROGRAPHS FOR SAMPLE D (Al- 4.5%Cu- 8%Pb)



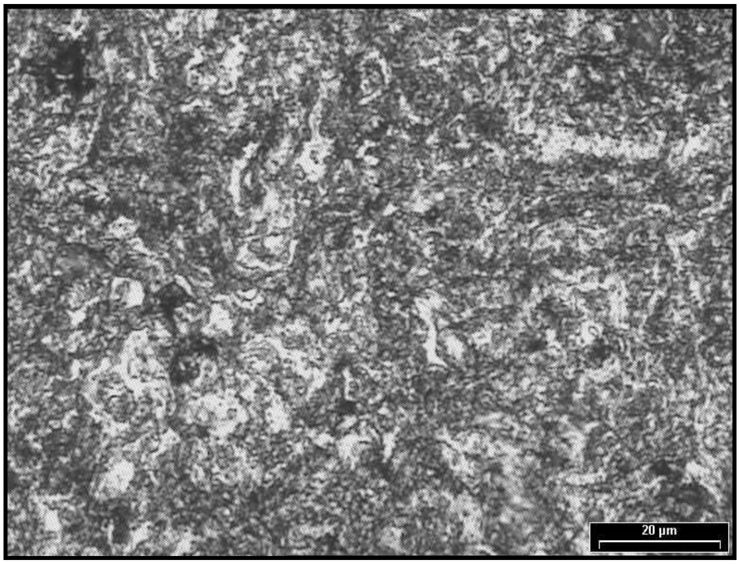
Sample D1: Sintering temperature 350 ^{o}C



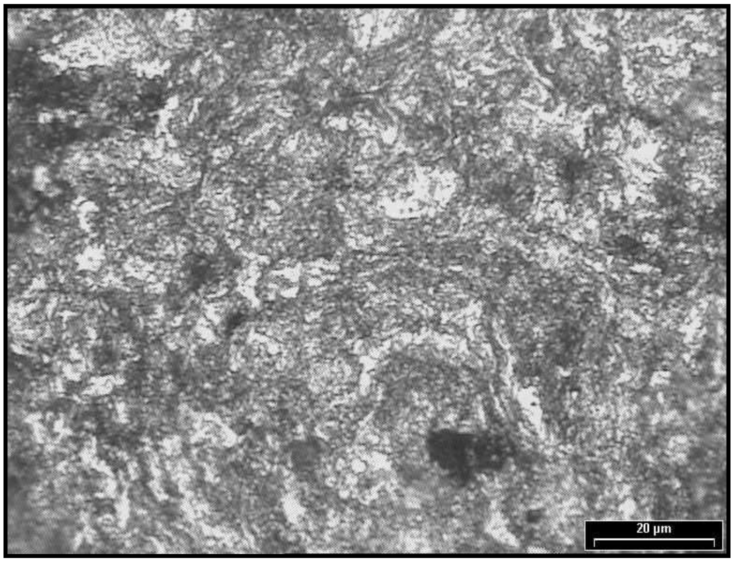
Sample D2: Sintering temperature 400 ^{o}C



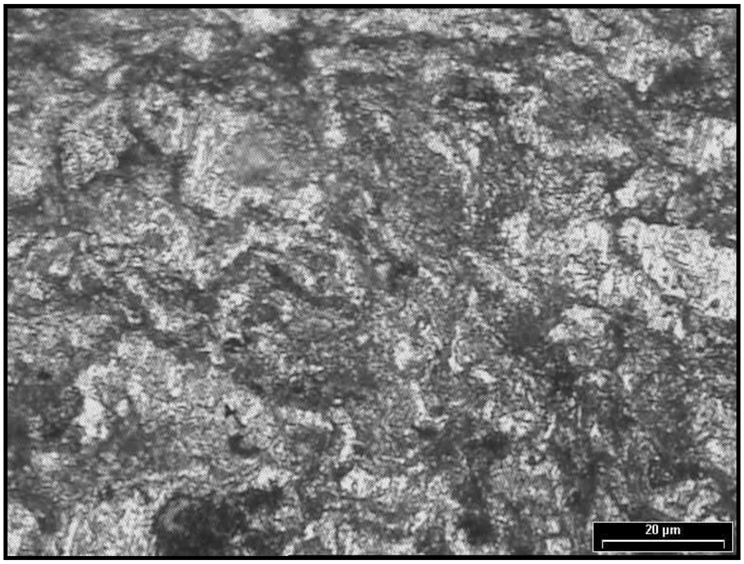
Sample D3: Sintering temperature 450 °C



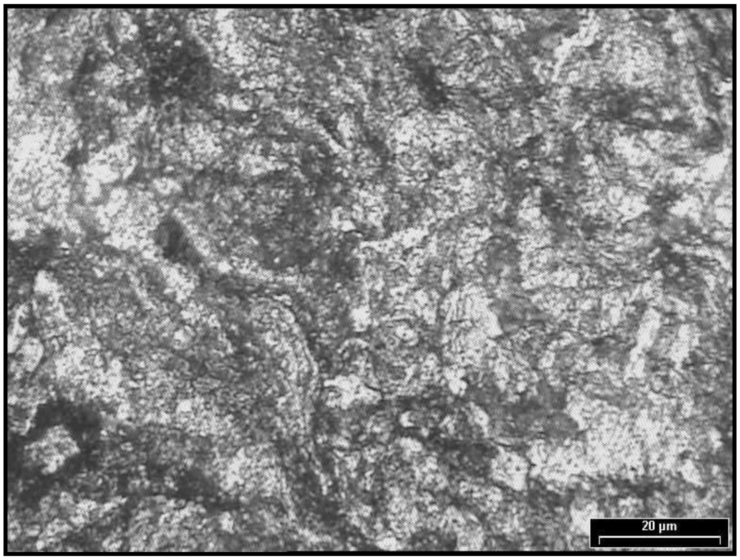
Sample D4: Sintering temperature 500 °C



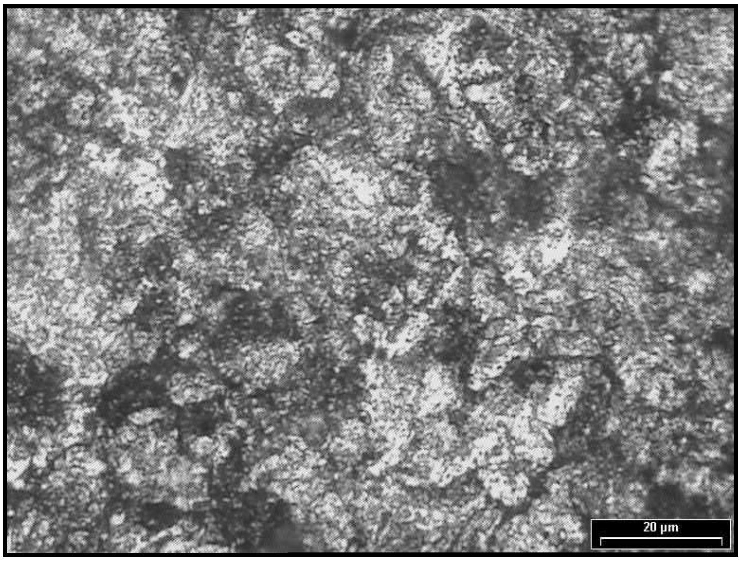
Sample D5: Sintering temperature 550 ^{o}C



Sample D6: Sintering temperature 600 ^{o}C



Sample D7: Sintering temperature 650 °C



Sample D8: Sintering temperature 700 °C

CHAPTER 6

CONCLUSION

The aluminium alloys containing tin and lead as additive element were studied in this work. The alloys were made by the powder metallurgy route consisting of the various steps such as powder blending, compaction, sintering followed by characterization of the prepared samples. The main aim of this work was to prepare such materials which can be used as a bearing alloy and hence should have good wear properties.

From the above study following conclusions can be drawn:

1. P/M method can be used to develop different bearing materials with close tolerance limit at defined temperature.
2. Pb provides better uniformity in structure as compared to Sn for Al based alloys.
3. The variation in volume as well as hardness is because of the non-conducive atmosphere (less reducing atmosphere) provided by the iron turnings.
4. The structural features indicate that the distribution of the second phase can be observed more uniformly by P/M method.

CHAPTER 7

SUGGESTIONS FOR FUTURE WORK

From the above study the following suggestions can be given:

1. A detailed study for increase in holding time that can provide better sinterability for these samples are required.

2. Studies on samples containing Pb and Sn both should be done to see the feasibility of better bonding of Pb and Sn.

REFERENCES

1. Handbook of Materials Sciences, Volume 7.

2. J.B. Fogagnolo, M.H. Robert, F.Velasco, J.M. Torralba; *Aluminium Matrix Composites Reinforced with Si_3N_4, AlN and ZrB_2 Produced by Conventional Powder Metallurgy and Mechanical Alloying.*

3. R Cook, H S Meeks, I T H Chang, C L Falticeanu; *Aluminium and Aluminium Alloy Powders for P/M Applications.*

4. D.P. Bishop, J.R. Cahoon, M.C. Chaturvedi, G.J. Kipouros and W.F. Caley; *Diffusion-based microalloying of aluminium alloys by powder metallurgy and reaction sintering.* Journal of Materials Science, Volume 33(August 1998) 3927-3934.

5. R. Q Guo, P. K Rohatgi and D Nath; *Preparation of aluminium–fly ash particulate composite by powder metallurgy technique.* Journal of Materials Science**,** Volume 32 No.15 (Jan 1997) 3971-3974.

6. B.Wang, G.M.Ianowski and B.R. Patterson; *SiC Particle Cracking in Powder Metallurgy Processed Aluminum Matrix Composite Materials*. Metallurgical and Materials Transactions Abstracts, Volume 26A (Sep 1995)

7. Department of Metallurgy and Materials Engineering at the Catholic University of Leuven in Belgium; *PM brings out the best form from complex aluminium alloys.* Pmetal-powder.net (July/Aug 2004)

8. Jung-Sik Kim, Kyle Jiang and Isaac Chang; *A net shape process for metallic*

microcomponent fabrication using Al and Cu micro/nano powders. Journal of Micromechanics and Microengineering, Volume16 (2006) 48–52

9. R..L.Orban; *New research Directions in Powder Metallurgy*. Romanian Reports in Physics, Volume 56, No. 3, P. 505 - 516, 2004

10. *Iron and Steel Powders for Sintered Components*. North American Höganäs, 2002, 393 p.

11. U.Engstrom; Materials Properties and Processes Robustness Obtained with Warm Compaction Improved DENSMIX Powder. *Advances in Powder Metallurgy and Particulate Materials*, MPIF Ed., USA, 2002, Part 4, 4-118-4-129.

12. P.Skoglund et al; High Density PM Components by High Velocity Compaction. *Advances in Powder Metallurgy and Particulate Materials*. MPIF Ed., USA, 2002, Part 4, 85-95.

13. J Kotila et al; Direct Metal Laser Sintering in 20 μm layer thickness. *Advances in Powder Metallurgy and Particulate Materials.* MPIF Ed., USA, 2002, Part 4, 164- 170.

14. R.L Orban; Mechanical alloying: an overview. *Proceeding of the Second International Conference on Advanced Materials and Technologies "MATEH'98*. UT Press Ed., Cluj-Napoca, Romania, 1998, vol. 2,. 583-588.

15. T, Aizawa et al; Nano-structured Materials via Bulk Mechanical Alloying. *Scripta Materialia*, No. 44, 2001, 1751-1755.

16.Online text from www.answers.com

17.J.P.Pathak, S Mohan; *Tribiological behaviour of conventional al-Sn and equivalent Al-Pb alloys under lubrication.* Bulletin of Materials Science, Volume 26 No.3 (April 2003) 315-320

18.S.P.Bishop, J.R.Cahoon, M.C. Chaturvedi, G.J.Kipouros and W.F.Caley; *On enhancing the mechanical properties of aluminium P/M alloys.* Received 27 September 1999; revised 13 April 2000. Available online 18 August 2000.

19.M.Zhu, Y.Gao, C.Y.Chung, K.C.Luo and B.L.Li; *Improvement of the wear behaviour of Al-Pb alloys by mechanical alloying.* Received 20 December 1999; revised 14 March 2000; accepted 14 March 2000. Available online 14 August 2000.

20.P.P.Rodriguez, A.Ibarra, V.Recarte, et al; *Influence of thermo-mechanical processing on the microstructure of Cu-based shape memory alloys produced by P/M.* Received 12 June 2003;Revised 15 September 2003. Available online 1 June 2004.

21.O.P.Pandey; Microstructural features of Spray-Deposited Immiscible Bearing Alloys, Al-43Sn and Al-7Sn-1.3Cu-1.3Ni. Journal of Materials Science and Technology, Volume 19 No.5 (2003).