Analysis of Mechanical and Metallurgical Properties of ALSICP Composites

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Abstract- A trend has been observed in the field of aluminum based composite materials to employ silicon carbide as reinforcement material in developing composites of unique properties. In the present study, an attempt has been made to fabricate the unreinforced Al and its composites were synthesized using the Powder Metallurgy (P/M) manufacturing route with blending, pressing and sintering allows the near net shape fabrication of precision parts. The composites are further solution heat treated at 5290°C for two hours and artificially aged at 1750°C for 18 hours. Optical Microscopy, Scanning Electron Microscopy has been carried out to analyze powder morphology and composite structure. An increasing trend towards micro-hardness and compressive strength with increase in weight percentage of silicon carbide has been observed.

Index Terms- Al-SiCp, Mechanical Properties, Microstructure, Powder Metallurgy.

I. INTRODUCTION

The Al-SiCp composites have seen most wide spread applications and hold the greatest promise for future growth because of their tailorable properties, good forming characteristics and the availability of comparatively low cost, high volume production methods. Aluminum based composite powders are highly compressible. Typically, green densities of more than 90 % of theoretical can be obtained utilizing low compacting pressures, (about 200MPa), allowing the use of presses with smaller capacity. Sintering of Al-SiCp composite parts is more energy efficient than for most other PM materials due to the relatively low sintering temperatures. Due to the low density of Al-SiCp composites, more than twice number of parts can be manufactured from unit weight of powder as compared to ferrous or copper based powders. During last 15 years various researchers have reported the fabrication of Al-SiCp composites and testing of their properties like tensile strength, hardness, wear resistance and microstructural characterization. Most of the researchers have observed an increase in tensile strength, hardness and wear resistance while decrease in ductility with increase in reinforcement content. Several researchers have studied the mechanical alloying of aluminum and SiC powders and identified improvement in mechanical properties of the Al-SiCp composites made from mechanical alloyed powders.

II. LITERATURE SURVEY

Mohanasundaram, et al. [1] have developed Al-SiCp composites by the powder metallurgy route and identified a significant improvement in tensile properties and wear resistance with increasing content of second phase. Madan [2] has fabricated 6061Al-SiCp and 6061Al-Al2O3 composites and tested their properties. The effect of fabrication method on the mechanical properties of the near net shape specimens was investigated by Ling, et al. [3]. Sinter /hot isostatically pressed compacts (sinter/HIPed) composites of up to 30 volume % SiC were produced with a significant improvement in ductility and ultimate tensile strength compared with the other fabrication methods. The poor mechanical properties of composites produced by the other methods are attributed to the weak bonding between adjacent particles and to internal porosity. The microstructural examination of fracture surfaces in representative materials confirmed that the sinter/HIPing technique yielded the best composites. For composites with reinforcement less than 10% by volume, the ductile fracture of the matrix appears to be the limiting factor. At higher volume fractions, the strength of interfacial bonds, initiation and growth of voids and
particle cracking all play an important role in controlling the mechanical properties.

Deevi and Sikka [4] have prepared Al-SiCp composites with 5 - 80 wt. % of SiC particulates using hot compaction. Increasing the SiCp content increased the yield and ultimate tensile strengths and reduced the tensile elongation at room temperature and at 450°C. The electrical resistivity and hardness of the composites increased with the increase of SiC content. The microstructure of the composites exhibited unique features with increases in SiC loading. SiC impinged into the Al particles, the extent and depth of impingement being severe above 50 volume % SiC content.

An Al-4 wt. %Cu, 10 volume % SiCp composite has been prepared using mechanically alloying technique [5]. The structural evolution of the mechanically alloyed powder mixture was monitored using X ray diffractionometry. The results showed that both the 0.2% yield and the ultimate tensile strengths increased with the duration of mechanical alloying. This increase was associated with the homogeneous distribution and refinement of the SiC particulates, the formation of oxides and the decreased grain size. Gingu and Orban [6] have studied the micro structural aspects of Al/(SiC+Cu) composite powders manufactured by mechanical alloying. Bhaduri et al. [7] used an attritor to mechanically alloy Al (7010) and SiC particulates with an addition of 2 wt. % stearic acid, which reduced cold welding of the Al particles. It was found that the equiaxed composite particles were formed. Several milling conditions (higher rpm and ball to powder ratio) have been used in the process whereas addition of SiC particulates retarded the process, due to possibility of the inhibiting effect on the formation and welding of lamellae in the initial stages of mechanical alloying.

Sankar and Singh [8] have synthesized the 7075 Al/SiC particulate composite powders by mechanical alloying in argon atmosphere in a high-energy attritor mill and 2 wt. % of stearic acid was used as process control agent. Powder samples were withdrawn periodically and characterized to find out the sequence of phase formation and the extent of alloying with time by X ray diffraction analysis. The surface morphology and nature of alloying of the composite powder was observed using scanning electron microscope. After 12 hour of milling, homogeneous equiaxed powders were obtained. One interesting observation of the XRD analysis is the absence of peak corresponding to Al4C3, which is often seen in the composite prepared by the liquid metallurgy route and is undesirable because of low strength and brittleness. Angers, et al. [9] have investigated the properties of 2024 Al/SiCp composites prepared by low energy ball milling using tumbler ball mill. The process parameters studied were milling time (between 1 and 24 hours) and the volume proportions of SiC (between 5 and 35 %). It was reported that as compared to high-energy attritor, the risk of contamination by the balls and container material is significantly reduced in the case of low energy ball milling. Composites containing up to 25 volume % SiCp exhibited superior mechanical properties and homogeneous distribution of reinforcement particles but their ductility decreased with increase in SiCp content. Mechanical properties and stress-strain behavior of different types of commercially fabricated aluminum matrix composites, containing up to 40 volume % discontinuous SiC whisker, nodule or particulate reinforcement were evaluated by McDanels [10]. The elastic modulus of the composites was found to be isotropic, to be independent of type of reinforcement, and to be controlled solely by the volume percent of SiC reinforcement. The yield/ tensile strengths and ductility were controlled primarily by the matrix alloy and temper condition. Ductility decreased with increasing reinforcement content. AMC Ltd. Have used a powder metallurgy approach involving mechanical attrition and hot isostatic pressing to achieve an exceptionally uniform dispersion of SiC particles in aluminum matrix and consistent mechanical properties [11]. Microstructure and deformation behavior of 12 volume % SiCp /6061 Al composites have been studied by Cheng, et al. [12]. It was reported that the load transfer between matrix and reinforcements, grain refinement of metal matrix and dislocation strengthening are the main strengthening mechanisms of Al-SiCp composites.

The ductile tearing of SiCp/Al interfaces and the SiC particle cracking are the dominant failure modes of Al-SiCp composites. The effect of reinforcement particle size, matrix to reinforcement particle size ratio and volume fraction of the reinforcement (0-20 vol.%) on the microstructure and mechanical properties of Al-6Cu-0.4Mn/ SiCp composites.
manufactured by powder metallurgy was investigated by Slipenyuk et al. [13].

In the present work Al-SiCp composites have been fabricated using powder metallurgy process. Mixture of six different compositions viz. 5, 10, 15, 20, 25 and 30 weight percent of SiC particulates in aluminum matrix were prepared using horizontal ball mill. The changes in powder particle morphology during mechanical alloying of aluminum and SiC particles after each four hour intervals were studied. The Al-SiCp composites were fabricated using isostatic compaction as well as direct compaction of powders and subsequent sintering in vacuum. The physical and mechanical properties of the Al-SiCp composites were measured and microstructural analysis was also done using scanning electron microscopy.

III. EXPERIMENTAL PROCEDURES

A horizontal ball mill (also called tumbler ball mill) was fabricated for mechanical alloying of aluminum and SiC particulates. The container of the mill was made of the same material as the powder to be milled (i.e. Al-15 weight % SiCp composites) to prevent contamination of powders from the container walls. The ball mill was designed for milling a total powder charge of 0.50 kg per run. Following are the specifications of the horizontal ball mill:

- Outer diameter = 300 mm
- Width = 105 mm
- Rotation speed = 78 rpm

The mill was filled with balls and powder charge up to about 20 to 35% of its total volume and the milling was done for required period of time (12 to 15 hours). Due to the combined action of centrifugal force and the friction between balls and container wall, the balls move together with the container wall until the gravitational force is balanced by the centrifugal force and subsequently fall down in free space causing an impact with the powder particles. Figure 1 shows the photograph of the horizontal ball mill.

Study of changes in powder particle morphology during mechanical alloying of Aluminum and SiC powders

The mixtures of aluminum with 5 to 30 weight percent of SiC particulates were prepared and ball milled using different ball mills in the argon atmosphere. 0.5 wt.% of Mg was also added to increase the wettability of SiC particulates with aluminum powders. The powder samples were taken from the ball mills at four hours intervals of milling to study the change in powder particle morphology during mechanical alloying. The parameters used for mechanical alloying in horizontal ball mill are shown in Tables 1. The steel balls with ball to powder weight ratio of ten were used as grinding media. In order to minimize the extreme tendency of aluminum to get itself welded during milling, 2 weight % of stearic acid was added as a process control agent. The milling was performed in Argon atmosphere to prevent contamination from atmospheric air. Argon was passed in the vials for 5 to 10 minutes to remove the air and create inert atmosphere in the vials to prevent contamination. The scanning electron micrographs of the powder samples were taken to study the change in powder particle morphology during mechanical alloying.

Fabrication of Al-SiCp composites through powder metallurgy process

Standard samples of Al-SiCp composites with 5, 10, 15, 20, 25 and 30 weight % of SiCp were fabricated through powder metallurgy route in the following stages:

1. **Sieve analysis of the powders**
   Aluminum powder of 5 to 30 μm size range and SiC particulates of about 400 to 600 μm were sieved separately and used in the present work. The equipment used for the purpose was horizontal vibratory sieving machine.

2. **Mixing of powders**
   The Aluminum and SiC powders of particular size range, obtained after sieve analysis were weighed individually and mixtures of six different compositions (viz. 5, 10, 15, 20, 25 and 30 weight percent of SiCp) were prepared. Figures 2 and 3 show the scanning electron micrograph of the
aluminum and SiC particulates respectively used in the present work. The micrographs show that the aluminum powders are of about 5 to 50 μm size range and the SiC particulates are of about 400-600 μm average size. 0.5 wt. % of Mg was also added to increase the wettability of SiC particulates with aluminum powders. The mixtures were put in plastic containers and mixing was done manually. After this, the mixing was continued in a pastel mortar for three hours to ensure proper mixing. The quality of mixing was inspected using optical microscope to ensure uniform distribution of aluminum and SiC powders.

Mechanical alloying
As discussed in literature [14] the simple blending and mixing provide a mechanical mixture of the powder constituents. Mechanical alloying results in mixing at molecular level. The SiC particles get embedded into Al powder particles and powder particles of Al-SiCp composite is obtained.

The powder mixtures were transferred to horizontal ball mill. The mill already contains the hardened steel balls as milling media. In order to minimize the extreme tendency of aluminum to get self welded during milling 2 wt. % of stearic acid was added as process control agent. The mill was run at 78 rpm for 12 to 15 hours. The powder becomes hot during milling therefore it is allowed to cool for 3-4 hours then it is taken out of the vials and stored in plastic containers.

**IV. METHODOLOGY**

**COLD ISOSTATIC COMPACTION**

Cold Isostatic compaction process results in better and more uniform properties as compared to die compaction because of uniform application of pressure from all directions and absence of die wall friction. The equipment used for this purpose was a pressure chamber, which is a compound cylinder designed for a pressure of 750 MPa [15]. Cold isostatic compaction was done using 2500 KN compression testing machine. The pressure chamber is provided with a neoprene 'O' ring along with a tellurium copper metal ring for perfect sealing. The chamber was filled with brake oil. The flexible mould was placed inside the chamber. The powder in the flexible mould was compacted to 600 MPa pressure. The compaction was done at a loading rate of 3.27 KN/second. The compact was extracted from the chamber and the mould was opened to remove the entrapped air. The rubber balloon was tied once again with a string and the above process of isostatic compaction was repeated. The green compacts were removed from the balloon.

**DIE COMPACTION**

For die compaction of Al-SiCp composites a die set-up (consisting of a die and punch has been used [2]. 1.5 weight % of die lubricant (zinc stearate) was mixed with powders for ease in compaction and ejection of powder compacts. The Al-SiCp composite powder of weighed amount was poured inside the die and the compaction was done on 500 KN press.

**SINTERING**

The green die compacts and cold isostatically pressed compacts were sintered in a muffle furnace by gradually raising the temperature to 580°C and the specimens were kept at this temperature for 30 minutes. The compacts were furnace cooled. Vacuum sintering of the Al-SiCp composites was also done which gave better properties. For this the Al-SiCp composite samples were placed in a quartz tube and the tube was evacuated using a vacuum system. After the high vacuum (10-6 mbar) was created in the quartz tube the tube was sealed by glass blowing using (LPG and oxygen) burners. The sealed tubes were placed in muffle furnace for sintering of Al-SiCp composites. The temperature was raised to 600°C and sintering was done for 45 minutes. A higher furnace temperature was used because the temperature inside the quartz tube is less then the outside temperature. After sintering the tube was furnace cooled and than the quartz tube was cut from one end and the sintered Al-SiCp composite
samples were taken out. Fig. 4 shows the photograph of the Al-SiCp composite specimens fabricated by PM process.

V. RESULTS AND DISCUSSIONS

Study of changes in powder particle morphology during mechanical alloying of aluminum powders with 5 to 30 wt. % of SiC particulates. The changes in powder particle morphology of Al powders with 5, 10, 15, 20, 25 and 30 wt. % of SiC particulates at various stages of ball milling were studied. Fig. 5 shows the change in powder particle morphology of Al powders and 10 wt. % SiC particulates at magnification of 400X. Fig. 5 (a) shows irregular, morphology of Al powders with 10 wt. % SiC particulates before mechanical alloying. Fig. 5 (b) shows that after 4 hours of milling the equiaxed ductile aluminum particles are flattened due to micro-forging resulting in flat plate like structures while the SiC particulates were fragmented. Fig. 5 (c) shows a cluster type of powder particle morphology after 8 hours of milling. The increased surface area of aluminum particles results in cold welding of powder components while the SiC particulates were entrapped along the cold welded interfaces of Al powder particles. In the final stage of mechanical alloying, as shown in Fig 5 (d), a fine homogeneous equiaxed composite structure was obtained due to fracturing of composite lamella structure and their random welding orientation. Due to inert gas atmosphere the newly fractured surfaces were prevented from oxidation. The welding of powder particles took place. The individual lamellae were unresolved in an optical microscope. It has been observed that if the mechanical alloying is incomplete, it is very difficult to remove the powder from the balls and the inner walls of milling container while after completion of the mechanical alloying the composite powder can easily be taken off from the balls as well as the walls of the container. A similar type of change in powder particle morphology was observed by the SEM study of powder samples taken at four hour intervals during mechanical alloying of the mixture of aluminum and SiC particulates with 5, 15, 20, 25 and 30 weight % of SiCp, e.g. Figure 6 show the change in powder particle morphology during milling of Al-30 weight % of SiCp at X400 magnification.

ROCKWELL HARDNESS

The average Rockwell hardness values of Al-SiCp composites measured on the polished surfaces of the samples using C scale on Rockwell hardness tester are shown in Fig. 7. The Rockwell hardness of powder metal Al-SiCp composites increases with increase in weight % of SiCp from 5 to 30 wt. % of SiCp. Fig. 7 shows the Rockwell hardness for Al-SiCp composites fabricated using un-ball milled and ball milled powders. The Al-SiCp composites prepared using ball-milled powders show higher hardness values than those prepared using un-ball milled powders. This is because the mechanical alloying involves severe deformation of the aluminum powders and embedding of the SiC particles uniformly into the aluminum matrix. This gives a uniform equiaxed composite powder structure, which gives improved properties after compaction and sintering. The values shown in the graph are average of the four readings for each composition of the composite and the scatter of the actual hardness values about the average was limited to within $\pm 5\%$ of the average hardness values for the Al-SiCp composite samples. A relatively high variation in the hardness values measured at different positions on the samples made by PM process may be due to the presence of porosity.

POROSITY

The densities of isostatically pressed Al-SiCp compacts in green and sintered condition are shown in Fig. 8 and 9 respectively. Both the green and sintered densities were measured for 3-3 samples of each composition of the Al-SiCp composites and the average values were shown in the graph. The deviation in measured values about the average density was limited to $\pm 5\%$ of the average value under both the green and sintered condition. The deviation in measured values was attributed to the slight variations in a large number of processing parameters involved in the fabrication of composites and also to the errors encountered during measurements. The theoretical densities of Al-SiCp composite compacts increase with increase in weight % of SiCp from 5 to 30 weight percent because the SiC particulates have higher density than the aluminum. However the measured density of Al-SiCp composites does not increases with increase in weight percent of SiCp because of increase in porosity with
increasing weight percent of SiCp. The comparison of figures 8 and 9 reveals that sintering results in de-
densification, which is due to the removal of volatile material like stearic acid and ethyl acetate during sintering. The stearic acid was added during mechanical alloying as a process control agent and the ethyl acetate was added during manual compaction of powders. The de-densification is also due to the recovery of the compressed powder with passing of time as high compaction pressure was used in the green stage. Figure 10 shows the porosity of isostatically pressed Al-SiCp composites in green and sintered conditions. The porosity in green stage increases with increase in weight % of SiCp up to about 20 to 25 weight percent, which is due to the increase in percentage of coarser component (SiCp). However at higher weight % of SiCp the porosity of green compacts tend to become almost constant. The porosity of sintered compacts was more than the porosity of the green compacts.

**COMPRESSIVE STRENGTH**
The compressive strengths were also measured for three samples of each composition of the Al-SiCp composites and the average value of the compressive strength for PM samples were plotted in the graphs with weight % of SiCp. Figure 11 shows the compressive strength for powder metal Al-SiCp composites fabricated using ball milled and un-ball milled powders. The compressive strength of PM Al-
SiCp composites in both the cases increases with increase in weight % of SiCp from 5 to 30 wt. % of SiCp. The Al-SiCp composites fabricated using ball milled powders show higher values of the compressive strength than those fabricated using un-ball milled powders. This was attributed to the uniform dispersion and mechanical interlocking of SiC particles in the aluminum matrix obtained during mechanical alloying process, which strengthened the consolidated specimens. The scatter of the measured values of the compressive strength was limited to within □ 2.5 % of the average for the PM Al-SiCp composite samples. The variations are attributed to the experimental errors during fabrication and testing of properties of the composites. The compressive strengths of powder metal Al-SiCp composites are quite less, which is due to the inherent porosity of the powder metal compacts. Porosity is required for oil impregnation, which impart self-lubrication properties to the components.

**INDIRECT TENSILE STRENGTH**
The indirect tensile strengths were also measured for the three samples of each composition of the Al-
SiCp composites and the average tensile strengths are shown in Fig. 12. The indirect tensile strength increases with increase in weight % of SiCp from 5 to 30 weight percent. The increase in the tensile strength of Al-SiCp composites with increasing wt. % of SiCp was reported to be due to the increase in the modulus of elasticity and the elastic limit of the material [2, 10]. The Figure 12 shows that a remarkable increase in the indirect tensile strength with increase in reinforcement content was observed only up to 20 wt. % of SiCp, however, a very small increase in tensile strength was observed above 20 wt. % of SiCp. This was due to the brittleness of the material at higher wt. % of SiCp [10]. The variation in measured values of the tensile strength about the average value was within □ 3.0 percent.

**7.6 SURFACE ROUGHNESS**
The surface roughness for isostatically pressed Al-
SiCp composites measured on Taly surf-6 surface roughness measuring instrument are shown in Fig. 13. The surface roughness has an important effect on the wear properties of the any components. The surface roughness of PM samples (Ra value ranges weight % of SiCp) is quite high, which is because of the coarseness of the powders used in the present work.

**MICROSTRUCTURAL ANALYSIS**
Figures 14, 15 and 16 show the scanning electron micrograph of un-sintered Al-SiCp composite samples with 10, 20 and 30 weight % of SiCp at magnification of 340X. The micrographs show that the aluminum and SiC particles are not properly bonded to each other in the green stage. Figures 17, 18 and 19 show the scanning electron micrograph of the vacuum sintered Al-SiCp composite with 10, 20 and 30 wt. % of SiCp respectively. SiC particles are visible in the micrograph. The micrograph shows that the bonding has taken place between aluminum and SiC particles after vacuum sintering. Some amount of porosity is also visible in the micrographs.
INTELLIGENT SYSTEM FOR PREDICTION OF MECHANICAL PROPERTIES OF MATERIAL BASED ON METALLOGRAPHIC IMAGES

This article presents developed intelligent system for prediction of mechanical properties of material based on metallographic images. The system is composed of two modules. The first module of the system is an algorithm for features extraction from metallographic images. The first algorithm reads metallographic image, which was obtained by microscope, followed by image features extraction with developed algorithm and in the end algorithm calculates proportions of the material microstructure. In this research we need to determine proportions of graphite, ferrite and ausferrite from metallographic images as accurately as possible. The second module of the developed system is a system for prediction of mechanical properties of material. Prediction of mechanical properties of material was performed by feed-forward artificial neural network. As inputs into artificial neural network calculated proportions of graphite, ferrite and ausferrite were used, as targets for training mechanical properties of material were used. Training of artificial neural network was performed on quite small database, but with parameters changing we succeeded. Artificial neural network learned to such extent that the error was acceptable. With the oriented neural network we successfully predicted mechanical properties for excluded sample.

VI. CONCLUSION

1. Mechanical alloying of aluminum and silicon carbide powders for 12 hours of milling results in fine homogeneous equiaxed composite powder structure. SEM studies of ball milled powders at intermediate stages reveal that due to impact of steel balls, the repeated cold welding, fracturing and
2. Re-welding of powder particles takes place and SiC particulates get embedded in the aluminum matrix. Finally the Al-SiCp composite powders are obtained.

3. During isostatic compaction of powders, the quality of final product depends upon the quality of initial manual compact; therefore the manual compact should be prepared carefully and should be given proper allowances in dimensions to get the desired final product.

4. Cold isostatic compaction at 600 MPa followed by vacuum sintering at 600°C has been successfully used to produce Al-SiCp composites.

5. Rockwell hardness, Density, porosity, compressive strength and indirect tensile strength of powder metal Al-SiCp composites increases with increase in reinforcement content from 5 to 30 weight percent of SiCp.

6. Sintering of Al-SiCp composites result in densification due to higher compaction pressure used in the green stage and also due to the removal of volatile materials during sintering and thus improving the oil retention properties.

7. Mechanical alloying of powders result in improvement in hardness, compressive strength and indirect tensile strength of Al-SiCp composites with 5 to 30 weight percent of SiC particulates.

8. Scanning electron micrographs of powder metal Al-SiCp composites reveals that the vacuum sintering results in bonding between aluminum and SiC particles. The micrographs also show some amount of porosity and uniform distribution of SiC particulates in aluminum matrix.

REFERENCE


