FABRICATION AND TESTING OF HYBRIDAL/SiC/FLYASH USING POWDER METALLURGY TECHNIQUE THROUGH MICROWAVE SINTERING

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ABSTRACT: Metal matrix composites (MMCs) possess significantly improved properties including high specific strength, specific modulus, damping capacity and good wear resistance compared to unreinforced alloys. MMCs could be produced by variety of methods such as Stir cast, Liquid Infiltration, Osprey and Powder metallurgy. Among this Powder Metallurgy processing is one of the effective methods to manufacture MMCs with high volume of reinforcement with fairly uniform distribution. The present investigation deals with the production of SiC particle reinforced Aluminium powder and Aluminium-flyash composite through powder metallurgy route with conventional sintering as well as microwave sintering. An attempt was made to optimize process parameter such as sintering time, sintering temperature and compacting load. The Aluminium powders (44 micron) and SiC particle (37 micron) and Flyash powder (20 micron) at a volume fraction of 5-20% of SiC and 20% of SiC-10% Flyash was processed. Compacting load was varied between 4000kg to 8000kg. Sintering was performed in Microwave furnace. At a load of 8000kg in Al-(20%)SiC-(10%)Flyash a maximum densification of (96%) was achieved. Hardness test was conducted in Rockwell testing machine. In Al-20%SiC-10%Flyash composition corresponding to the 8000 kg load, the Hardness 81HRB (Rockwell B-scale) was obtained.

I. INTRODUCTION

1.1 POWDER METALLURGY:
Powder metallurgy can be defined as the art of producing powders of metals, alloys, ceramics etc. mixing them in necessary quantities which are blended, pressed into a desired shape (compacted), and then heated (sintered) in a controlled atmosphere to bond the contacting surfaces of the particles and establish the desired properties. It is commonly designated as P/M.

1.2 HISTORY:
1. Early 3000 B.C. – a crude form of powder metallurgy appears to have existed in Egypt
2. Mid- or Late 19th Century – the mass production of P/M products begin
3. Early 20th Century – powder metallurgy was used to produce copper coins and medallions, and tungsten wires, the primary material for light bulb filaments
4. 1920s – tungsten carbide cutting-tool tips and nonferrous bushings were reproduced. Other early products were self-lubricating bearings and metallic filters.
5. After World War II – a period of rapid technological development occurred which are based primarily on automotive applications, and iron and steel replaced copper as a dominant P/M material.
6. Aerospace and nuclear developments accelerated demand for refractory and reactive materials, where P/M processing is quite attractive.
7. 1960s – full-density products emerged.
8. 1970s – high-performance superalloy components, such as aircraft turbine engine parts
10. From 1960-1990 – the consumption of iron powder increased tenfold making of fasteners (nuts and bolts) in large volumes.
1.3 MMC PRODUCTION BY POWDER METALLURGY:

Powder Metallurgy processing is one of the effective methods to manufacture MMCs with high volume of reinforcement with fairly uniform distribution. Powder metallurgy is a forming and fabrication technique consisting of three major processing stages. First, the primary material is physically divided into many small individual particles. Next, the powder is passed through a die to produce weakly cohesive structure (via cold welding) very near the dimensions of the object ultimately to be manufactured. Pressures of 1-10 tons are commonly used to compact the metal powder. Then subsequent heating or sintering of compacted objects in the mould with temperature below melting point under non oxidizing atmosphere.

1.4 NEED FOR THE STUDY:

The application of microwave sintering of metallic powder is successfully extended to metal matrix composite. Anklekar (2001) prepared a MMC of copper steel using microwave sintering. Microwave heating is recognized for its various advantages, such as: time and energy saving, very rapid heating rates, considerably reduced processing cycle time and fine microstructures and improved mechanical properties, better product performance. Bescher (1992) prepared a MMC of Al-SiC using Microwave Sintering. Aluminum, like most metals, cannot be considered a good microwave absorber. However, if it is under the form of a finely divided powder, the particles will slowly heat up and reach temperatures above 1473K. On the other hand, because of its lower electrical conductivity, silicon carbide is a highly absorbing material.

Al-SiC metal matrix composite prepared by microwave sintering produces better density, hardness, finer grain size, and the shape of the porosity is quite different than in a conventional part. Microwave absorption and the heating rate increase with decreasing SiC particle size (Susanne Leparoux2003). In microwave-processed powder metal components, it was observed that round-edged porosities producing higher ductility and toughness. Flyash particles are used as filler in aluminium alloy casting, it reduces cost and density and increases wear, seizure and abrasion resistance and stiffness (Guo1997) .

However detailed study on the role of microwave sintering parameters such as temperature, time, on the density, hardness and microstructure of Al-SiC, Al-flyash, and Al-SiC-flyash composite is not yet performed. Hence present study is undertaken.

A study on the role of microwave sintering parameters such as temperature, time, on the density, hardness and microstructure of Al- SiC composite with various volume of SiC is essential. Hence present study is undertaken.

II. PREVIOUS WORKS

Many researches have investigated on fabrication, microstructure and properties of Aluminium matrix composites. However most of them adopted only conventional furnace sintering(Guo et.al1997),Charles(2004), whereas few have utilised microwave sintering for Al matrix composites(Bescher et.al(1992) and Susanne leparoux,et.al(2003)).

Microwave sintering has been successfully used in ceramic systems and literatures are available on microwave sintering of tungsten carbide (VaradaRajan, et.al (2006) and Huanga, et.al (2009)

There are few literatures which describe the usage of microwave sintering in steels and copper –graphite ,Anklekar, et.al (2001) and Rajkumar&Aravindan (2009).

2.1 FURNACE SINTERED ALUMINIUM FLYASH COMPOSITE

Guo, et.al (1997) studied the Aluminium fly ash mixtures containing different weight percentages of fly ash were prepared and compacted at pressures from 138-414 MPa. The compacts prepared at 414MPa were sintered in nitrogen atmosphere at 600, 625 and 645 °C, respectively. The time of sintering ranged from 0.5-6 h.

Density, hardness and strength of the sintered compacts were determined as a function of weight per cent of fly ash particles. Volume changes during sintering of green compacts were also evaluated as a function of increasing fly ash weight per cent. Microscopic studies of green and sintered compacts were done to study the effectiveness of sintering. Green and sintered density of the compacts was found to decrease with increasing weight per cent 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 increasing weight per cent of fly ash however, the hardness was found to increase slightly up to 10 wt% fly ash, beyond which it decreased. The densification parameter increases with increasing value of the compacting pressure and approaches 0.85 and 0.95 at 414 MPa for 20 and 5 wt% fly ash, respectively.
The above values of densification parameter are close to 1, hence all the compacts subjected to sintering were made at 414 MPa. Scanning electron micrographs of green compacts of aluminium-precipitator fly ash shows that uniform distribution of fly ash particles in the aluminium matrix, whereas aluminium-cenosphere fly ash particles are broken into pieces during the compaction conditions. This adversely affects their capability of reducing the density of the composite by virtue of their hollow shape and low density. Because the cenosphere particles break during compaction and are not able to maintain their hollow shape, a detailed sintering study was carried out only on aluminium-precipitator fly ash compacts, because precipitator particles are mostly solid and do not break during compaction.

Charles (2004) studied the Aluminium hybrid composites reinforced with silicon carbide and fly ash particulates were fabricated by stir casting and powder metallurgy methods. Different volume fractions of SiC particles (10, 15 and 20%) and constant volume fraction of fly ash (10% vol) were used for the synthesis. Results of the investigations indicated that wear resistance and hardness were enhanced on increasing the volume% of SiC. The tensile strength was high at 10 vol% of SiC and it decreased as the vol% increased. Microstructure showed a fairly uniform distribution of the dispersoids. Electric discharge machining was done on the composite specimens, it was found that material removal rate and tool wear rate increased with increase in current and decreased with increase in pulse duration and vol% of SiC.

2.2 MICROWAVE SINTERING ON CERAMICS:

VaradaRajan, et.al (2006) investigated the machining behavior of MMC. An attempt has been made to improve the performance of cemented tungsten carbide tools with post treatment by subjecting to microwave radiation. X-ray diffraction technique (XRD) and scanning electron microscopy (SEM) were used to evaluate the status of irradiated samples and worn out edges. Results showed that irradiated tool performs better in machining MMC suggesting that microwave radiation can be a potential post sintering technique for cemented carbide tools to improve machining ability. Irradiation of cemented carbide tool with microwave energy imparts densification and phase transformation that results in improved hardness. Magnitude of all the cutting force components with treated tool is generally less in comparison with untreated tool. This can be attributed to improved hardness of the treated tool imparted by microwave energy. Treated tool produces better surface of the MMC material machined compared to untreated tool. Tool life is improved by microwave irradiation, which imparts increased cutting wedge stability. Monitoring of AE signal and analyzing it in terms of rms value and peak frequency have illustrated that treated tool can perform better at relatively higher cutting speeds.

Huanga, et.al (2009) investigated that ceramics are sintered by hybrid-microwave sintering (HMS), which combines the characteristics of microwave heating and conventional heating. Microwave processing, as a new method for sintering ceramics, has key advantages such as increased heating rate, uniform heating and reduced cost compared to conventional methods. It is generally accepted that microwave sintering can improve the macroscopic mechanical performances of ceramics. To evaluate the homogeneous performance of the sintered ceramics, the behaviours of thermal residual stress distribution in the microwave-sintered and conventionally sintered ceramics were investigated by X-ray diffraction technique. The thermal residual stress investigation shows microwaves can sinter ceramics in entire volume while offering improved mechanical properties. The experiment results confirm that the sinterability of ceramics is homogenously improved by hybrid microwave sintering. The uniform heating property of two-directional hybrid microwave sintering is confirmed by the thermal residual stress and the pore ratio investigations on microscopic scale. The results of the thermal residual stress and the pore ratio investigations confirmed that sinterability of the ceramics is improved by hybrid microwave heating, and HV test on microwave-sintered specimen revealed superior mechanical properties compared to conventionally sintered specimen.

2.3 MICROWAVE SINTERING OF NON-AL MATRIX COMPOSITES

Anklekar, et.al (2001) investigated the modulus of rupture bar samples of coppersteel (MPIF FC-0208 composition) were successfully sintered by microwave technique to obtain higher sintered density, higher Rockwell hardness (HRB), and higher flexural strength as compared with conventional sintering. The improved mechanical properties of microwave sintered samples can be mainly attributed to the evolution of distinct porosity distribution, primarily consisting of small, rounded, and using uniformly distributed pores as against large, angular reporting the and non-uniformly distributed pores observed in the case of conventional sintering. The maximum sintered density obtained was 7.45±0.05 g cm⁻³ for the microwave sintered samples using the special carbon based coating material as a susceptor, and sintering at 1260°C for 20 min in forming gas.
atmosphere (5% H₂+95% N₂ mixture) with a dew point of −60°C. The highest Rockwell B hardness (HRB) of 82±2 was obtained for microwave processed samples using SiC rods as a susceptor, and sintered at 1260°C for 5 min soaking in flowing forming gas atmosphere. The microwave sintered samples using the special carbon based coating material as a susceptor showed minimum porosity, consisting of very small, rounded, and uniformly distributed pores as against conventional sintered samples, which showed maximum porosity, mainly consisting of large, angular, and non-uniformly distributed pores.

Dinesh Agrawal (2006) investigated the most recent application of microwaves which has been in the field of metallic materials for sintering, brazing/joining and melting. Several common steel compositions, pure metals and refractory metals have been sintered in microwaves to nearly full density with improved mechanical properties. Many commercial powder-metal components of various alloy compositions including iron and steel, copper, aluminum, nickel, Mo, Co, Ti, W, Sn, etc., and their alloys have also been sintered in microwaves producing better properties than their conventional counterparts by using a 2.45 GHz multimode microwave system. This work has been further extended to join and braze bulk metal pieces, especially super alloy based turbine blades. Further, in a specially designed microwave cavity, even the bulk metals can be made to couple with the microwave field and melted.

2.4 MICROWAVE SINTERING OF Al MATRIX COMPOSITES

Bescher, et.al (1992) investigated the Electromagnetic radiation in the microwave range has been utilized for the fabrication of SiC particulates-reinforced aluminum matrix composite. The Aluminum powder particle size was 20 micro meters, whereas the SiC was 1 micrometer, both were manually mixed, placed in a crucible, insulated with a 3-inch thick alumino-silicate fiber. Powder mixtures with compositions ranging from 70 wt% Al - 30wt% SiC were placed and processed under protective nitrogen atmosphere in a 750 W multimode cavity operating at 2.45 GHz. Temperatures larger than 800 °C have been achieved in a time varying with the mixture composition. When 10, 20 or 30 weight percent of silicon carbide are added to a poorly absorbing aluminum powder, the heating curve is changed. Upon four minutes of microwave exposure, the mixtures initially reach very high temperatures for a very short time. The penetration depth at 2.45 GHz for most metals is usually less than 3 micrometer. The heat is propagated to the rest of the bulk by conventional heat transfer. Microstructures and some physical properties of the composites are compared.

Jiping Cheng, et.al (2000) investigated the large batch of commercial alumina abrasive grit was successfully microwave sintered using a continuous microwave sintering technique. The properties of the microwave sintered grit product can be easily controlled by adjusting the sintering temperature and the feed rate. In comparison of the conventionally sintered product, the microwave sintered material exhibited better abrasive and mechanical properties. Microscopic examination revealed a much finer microstructure of the microwave processed material than for the conventional product. The quality of the microwave sintered products depends on the sintering temperature and sintering time. During the continuous microwave sintering processing, the sintering time can be controlled by the feeding speed of the sample. The results show that the microwave sintered product exhibits much a higher abrasive index and Vickers’s hardness when compared with the conventionally sintered products.

Susanne leparoux, et.al (2003) investigated the sintering of SiC reinforced Al-matrix composite in-situ synthesis of TiC in a powder mixture of Ti and Carbon. In the first case, Al and SiC powders of various volume ratios were prepared by powder mixing in a Turbola mixer. Mixing was done without any additive in a time range of 1-3 hour. For each mixture 10 pellets were produced by uniaxial pressing with 5mm thickness and a diameter of 50mm. Microwave energy is absorbed by SiC grains, heating the metal matrix composite to sintering and even melting temperature. The composite is processed at 1kW microwave power. Microwave absorption and heating rate increase with decreasing SiC particle size. Composites with high SiC content (70% volume) are processed at 650 °C /1h in the microwave furnace whereas conventional resistive heating at the same temperature did allow sintering of the sample. The applied pressure was limited to 100 MPa, so a densification of only 70 % (relative to the theoretical density) was obtained for the different green bodies. For the reduction of silicon carbide particle size there is no significance difference in microstructure has been revealed but the processing time is quite different. Initial green density (74%) did not increase during sintering. Due to the volumetric heating and the radiative heat loss at the surface the effective bulk temperature of the sample is higher than the surface value indicated by the pyrometer. Optical microscope revealed that in both cases the melting temperature of Al was reached in the material and sometimes liquid Al was expelled to the
sample surface, thus leading to the loss of the sample beneath the surface. A Maximum of 10k/s is achieved leading to a gain in overall process time of 60% compared with conventional sintering without damaging the sample and the density of the sintered parts can be improved.

III. METHODOLOGY

3.1. METALPOWDER SELECTION

The powders selected are as follows

1. Aluminium powder (commercial pure)
2. Silicon carbide powder (commercial pure)
3. Flyash

Aluminium Powder

Purity Aluminium : min 99%

Maximum limits of impurities

| Iron (Fe) | 0.5% |
| Heavy metals (Pb) | 0.03% |
| Size | 44 micron (325 mesh) |

1. Aluminum properties include good appearance, ease of fabrication, good corrosion resistance, low density, high strength-to-weight ratio and high fracture toughness.

2. Aluminum powder is a light, silvery-white to gray, odourless powder. It is a reactive flammable material. Aluminum powder is a fine granular powder made from Aluminium.

Physical Properties

| Density | 2.7g/cc |

Mechanical Properties:

1. Modulus of Elasticity | 68.0 GPa |
2. Poisson Ratio | 0.360 |

Silicon Carbide Powder

Size : 37 micron (400 mesh)

Silicon carbide (SiC), also known as carborundum, is a compound of silicon and carbon with a chemical formula SiC. Grains of silicon carbide can be bonded together by sintering to form very hot ceramics which are widely used in applications requiring high endurance, such as car brakes and ceramic plates in bullet proof vests.

It was originally produced by high temperature electro-chemical reaction of sand and carbon. It is used in abrasives, refractories, ceramics and numerous high-performance applications.

Mechanical Properties:

1. Density | 3.21 g/cc |
2. Hardness | 2800 knoop hardness number |
3. Flexural strength | 550 MPa |
4. Poisson ratio | 0.14 |
5. Compressive strength | 3900MPa |
6. Fracture toughness - 4.6 MPa m^{1/2}

**Thermal properties**

1. Coefficient of thermal expansion - 4.0 \times 10^{-6} \degree C
2. Thermal conductivity - 120 W / m \degree K
3. Maximum working temperature - 1650 \degree C
4. Specific heat - 750 J/Kg

**FLYASH POWDER**

Fly ash is one of the residues generated in the combustion of coal. It is an industrial by product recovered from the flue gas of coal burning electric power plants. The specific gravity of fly ash vary in the range of 0.6-2.8 gm/cc.

Size: 20microns (1000mesh)

**Physical Properties:**

1. Density - 2.25g/cc

**3.2 DIE PREPARATION**

The die material selected was EN24 which is a medium carbon steel with Cr and Mo.

**Properties and Composition of EN24**

It is a high quality, high tensile alloy steel easily machineable, giving good ductility and shock resisting properties combined with resistance to wear.

**3.3 DESIGN CONSIDERATIONS IN POWDER METALLURGY**

Design requirements are essential for P/M parts:

1. The shape of the parts must be as simple as possible. PM parts should be made with the widest tolerances. The PM process is capable of achieving tolerances of bigger than 0.1 mm.
2. In the present study, cylindrical specimen of dia 15mm and thickness of about 5mm was selected for the study.
3. Hole and grooves must be parallel to the direction of ejection.
4. Sharp corners, radii, thin section must be avoided. Minimum wall thickness is 1.5 mm.

**3.4 DIE DESIGN**

To facilitate the easy removal of compacted specimen, tappered section has been used. Dimension of tappered section used is 16 to 15 mm for overall length of 50mm in punch and (40mm, 10mm) in die.

The front view and top view of die parts and the assembly view are shown in Figure 3.1 to 3.
Figure 3.1 Base plate

Figure 3.2 Punch
3.5 POWDER MIXING
Al-5% volume of SiC powders are mixed by Magnetic Mixer. The calculation for mass of aluminium and silicon carbide powder needed for a sample of 10mm thickness and 15mm diameter are as follows.

3.6 COMPACTING
The purpose of the compacting is to consolidate the powder into the desired shape and as closely as possible to final dimensions, it is designed to impart the desired level and type of porosity and to provide adequate strength for hardening. Compacting was done in UTM (Universal Testing Machine) as shown in the figure 3.6. The various compacting load from 4000kg to 8000kg were used for compaction.

3.7 SINTERING
In the sintering operation, the pressed- powder compacts are heated in a controlled – atmosphere environment to a temperature below the melting point but high enough to permit the solid-state diffusion and held for sufficient time to permit bonding of the particles.

IV. RESULTS AND DISCUSSION
The Al-30%and 40% SiC composites were prepared. The Effect of compacting load on green density and sintered density as shown in Table 4.1.

For the above samples the sintering temperature was maintained at 600°C. The sintered density value increases with increases in load. In this Microwave sintering, a better densification of 96% was achieved.
corresponding to compacting load of 8000kg respectively.
The role of compacting load on density and hardness was studied and the results are shown in the Table 4.2

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compacting load(kg)</th>
<th>Theoretical density (g/cc)</th>
<th>Green density (g/cc)</th>
<th>Sintered density (g/cc)</th>
<th>Densification (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AL-5%,10%,15%,20%SiC</td>
<td>1 4000</td>
<td>2.73</td>
<td>2.26</td>
<td>2.37(Microwave)</td>
<td>86</td>
</tr>
<tr>
<td></td>
<td>2 4500</td>
<td>2.76</td>
<td>2.46</td>
<td>2.56(Microwave)</td>
<td>89</td>
</tr>
<tr>
<td></td>
<td>3 6000</td>
<td>2.78</td>
<td>2.50</td>
<td>2.51(Microwave)</td>
<td>90</td>
</tr>
<tr>
<td></td>
<td>4 7000</td>
<td>2.81</td>
<td>2.51</td>
<td>2.68(Microwave)</td>
<td>95</td>
</tr>
<tr>
<td>AL-20% SiC-10%FLYASH</td>
<td>5 8000</td>
<td>2.76</td>
<td>2.66</td>
<td>2.67(Microwave)</td>
<td>96</td>
</tr>
</tbody>
</table>

Table 4.1 Lists various compacting load and density of Al-5-20% and 20% SiC-10%Flyash

<table>
<thead>
<tr>
<th>Composition</th>
<th>Compacting load(kg)</th>
<th>Rockwell Hardness (B Scale)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-5%SiC</td>
<td>4000</td>
<td>58HRB</td>
</tr>
<tr>
<td>Al-10%SiC</td>
<td>4500</td>
<td>61HRB</td>
</tr>
<tr>
<td>Al-15%SiC</td>
<td>6000</td>
<td>70HRB</td>
</tr>
<tr>
<td>Al-20%SiC</td>
<td>7000</td>
<td>75HRB</td>
</tr>
<tr>
<td>Al-20%SiC-10%Flyash</td>
<td>8000</td>
<td>81HRB</td>
</tr>
</tbody>
</table>

Table 4.2 Hardness value for Al-40%SiC corresponding to the load of 4500kg.
A microwave sintered sample of Al-5%SiC was seen in the Scanning electron Microscope. Here the particle size are not uniform in shape. Some of them are spherical and few of them are Flakey in nature.
Fig. 1 Al-5% SiC

Fig. 2 Al-10% SiC
A uniform distribution of SiC seen in the Aluminium Matrix.

Fig. 3 Al-15% SiC
In the above Al-15%SiC a clustering of SiC particles are Matrix particles are seen.

![Image](https://via.placeholder.com/150)

**Fig.4. Al-20%SiC**

In the above Fig. Al-20%SiC, SiC particles are rounded in nature.

![Image](https://via.placeholder.com/150)

**Fig.5. Al-20%SiC-10%Flyash**

In the above fig clustering of SiC Particles are seen. Flyash particles are spongy in nature.
IV. CONCLUSION

1. Preparation of Al-SiC,Al-SiC-Flyash composites by powder metallurgy technique is attempted during this project work. Composites are prepared by varying different compacting load.

2. The composition of composite prepared are 70% Al + 5,10,15,20% SiC, 70% Al + 20% SiC + 10% Flyash. This composite was compacted using UTM under different compacting load 4000kg to 8000kg were used for compaction.

3. Sintering was done by Microwave Furnace. Sintering was done at the temperature of 600°C for duration of 1 hour.

4. It was found that a maximum densification of (96%) was achieved for the compacting load of 8000 kg using Universal testing machine for the Microwave sintered sample. Higher the compacting load, correspondingly higher density was obtained.

5. Hardness test was conducted in Rockwell testing Machine. In Al-20% SiC-10% Flyash composition at the compacting load of 8000 kg, the hardness 81HRB (Rockwell B-scale) was obtained. Higher the Percentage of Silicon carbide, correspondingly higher hardness was obtained.

6. Compare with manual mixing of Powders, Magnetic Mixing of powders yields better hardness of 81HRB.

7. It was found that a uniform distribution of Silicon carbide and flyash in the AluminiumMatrix.

REFERENCES


