

Influence of Various Sintering Processes on Cenosphere added Copper Composites

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Abstract:

Composites are lightweight materials widely used in automotive, aerospace, transportation and wind turbines. Different reinforcement materials can be used in composites of which fly ash attracts the researchers as an important reinforcement due to its low density, Fly ash cenosphere composites can be manufactured by different techniques like stir casting, compo casting and powder metallurgy method. The copper MMC is having high strength, high temperature sustainability with increased hardness with reduced wear loss, high thermal conductivity and electrical conductivity.

In this research work the copper metal matrix reinforced with different weight percentages of cenosphere fly ash particulates was produced through the Powder Metallurgy (PM) technique and compression strength and thermal shock test were evaluated. The present work was focused to optimize the effect of process parameters for the production of copper fly ash cenosphere composites produced by powder metallurgy method and study of its mechanical properties in particular compression strength. The strength of the microwave sintered composites and conventional sintered composites are in the range of 70MPa to 45MPa at 950°C.

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1. Introduction

The composite material can be defined as the system of material consisting of a mixture of combination of two or more micro constituents insoluble in each other and differing in the form and in material composition. These materials can be prepared by adding two or more dissimilar material in such a way that they act mechanically as a single unit. The properties of such materials differ from those of their constituents. These materials may have a hard phase embedded in a soft phase or vice versa. Normally in the composite material it has a hard phase in the soft ductile matrix. The hard phase acts as a reinforcing agent and increases the strength and

modulus similarly the soft phase acts as matrix material. Powder Metallurgy method in conjunction with deformation processing is used to fabricate particulate or short fiber reinforced composites. This typically involves cold pressing and sintering, or hot pressing to fabricate primarily particle- or whisker-reinforced MMCs. The matrix and the reinforcement powders are blended to produce a homogeneous distribution. The blending stage is followed by cold pressing to produce a pallet. This is known as a green samples, this is about 80% dense and can be easily handled. The cold pressed green body is canned in a sealed container and degassed to remove any absorbed moisture from the particle surfaces.

Copper and its alloys offer the advantage of superior electrical conductivity (5.96×10^7 S/m) as well as high thermal conductivity (401 W/m·K), high microstructural stability, high melting point (1083 °C), exceptional corrosion resistance and ease of fabrication. All these properties make Cu highly suitable for several applications in electronics as well as manufacturing industries. The modulus of Cu is 130 GPa and its yield strength is 117 MPa. Its ultimate tensile strength is 210 MPa. However, Cu and its alloys also suffer from several disadvantages such as poor mechanical properties and wear resistance. Due to its relatively high ductility as well as poor mechanical properties at high temperature, Cu often fails to meet the need in cutting-edge technological applications. Therefore, it has become essential to improve the properties of pure Cu for its use. Cu based metal matrix composites have been gaining vast importance. Cu matrix has been reinforced with various kinds of reinforcements like ceramic dispersoids and intermetallic compounds. It has been observed that Cu based metal matrix composites have a superior combination of thermal as well as electrical conductivity, high strength, and improved mechanical and tribological properties. Kalinin et al. reported that copper based composites have applications in making components like connectors, electrodes for spot welding processes, lead wires and other electronic components because of exclusive property combination of strength and conductivity at high temperatures [1]. Mahendra and Radhakrishna studied the Characterization of stir cast Al-Cu-(fly ash + SiC) hybrid metal matrix composites. They suggested that the addition of fly ash decreases the density and increases the tensile strength, compression strength, impact strength and wear resistance [2]. Sreekanth et al. showed that the effect of addition of fly ash on polymer based composite with varying particle size and reinforcement percentage. They reported that the

addition of fly ash improved the strength and thermal stability. On contrary elongation at break is decreased drastically. They also claimed that significant improvement in mechanical properties that was found in the composites is possible with small particle size of flyash [3]. Rahimian et al. reported the effect of alumina particle size, sintering temperature and sintering time on the properties of Al-Al₂O₃ composite. Studies were carried in the range of 500–600°C. sintering time was considered in the range of 30-90 min. There studies clearly indicated that prolonged sintering times had an adverse effect on the strength of the composite [4]. Sundaram et al. 2011 have observed that the Aluminum composite's density and strength decreases with increases vol. per cent of fly ash cenospheres in the composite.[5]. Zhi-qiu Huang et al. (2010) suggested the behavior of AZ91D Mg alloy/fly-ash cenospheres composites. They fabricated AZ91D Mg alloy/fly-ash cenospheres by metal stir technique. 100µm size cenosphere particles were used with mass fraction of 4%, 6%, 8% and 10%. The effects of mass fraction of cenospheres on the microstructure and compressive properties were characterized. They found that fly ash cenospheres are uniformly distributed in the matrix. The densities of composites are in the range of 1.858-1.92/g/cm³. It also reported that the hardness of AZ91D/FAC composites increases with increasing the content of the cenospheres. Finally it is concluded that the compressive strength of AZ91D/FAC composite is higher than that of as cast AZ91D Mg alloy. The composites containing 4% of cenosphere gives the higher compressive strength and higher yield strength is observed in composites containing 8% of cenosphere. [6]. Dou Z. Y. et al. (2007) revealed the high strain compression behaviour of cenosphere when fabricated with aluminium syntactic foams. The syntactic foam exhibits distinct strain rate of sensitivity and increase in peak strength. From this experiment cenosphere shows the greater

energy absorbing properties. The composites show 45-60% of higher dynamic compressive strength than that of quasi-static compression. [7].

R.Irandjezian et.al (2017) conducted a compression test and result analysis shows that the 5% cenosphere based Al-MMC gives better results than 0% based Al-MMC. It clearly shows that addition of cenosphere significantly improves the compressive strength nature of MMC [8]. Kiran Shahapurkaret al. (2018) investigated on Compressive properties of untreated and treated cenosphere/epoxy foams under room and arctic temperatures are analyzed. It is observed that the cenosphere/epoxy foams with untreated and treated fillers manifest lower strains to failure under compressive loading at room temperature conditions as compared to neat epoxy samples. All foam compositions show an increase in compressive modulus compared to that of the neat resin. However, an overall increase in compressive strength was observed when tested under in-situ arctic condition [9].

2. Sample preparation and test procedure

A different composition of copper and fly ash cenosphere were blended. Atomized pure copper metal powder of 99.5% purity with a particle size of about ASTM 200 mesh (75 μm) obtained from M/s. MASIL SCIENTIFIC PRODUCTS make laboratory reagent powder was used for the matrix. Among various dispersive reinforcements, fly ash and its derivative viz. 'cenosphere' are available in large quantities as solid waste by product in coal fired thermal power stations [Cenosphere was obtained from fly-ash received from M/s. NTPC Simhadri Thermal Power Station, India]. Cenosphere was obtained from fly-ash received from M/s. NTPC Simhadri Thermal Power Station, India fly ash cenospheres were harvested from the fly ash through a process which involved ash slurry preparation, stirring and

dispersion of slurry. The agitated slurry was then allowed to settle to a standstill. Later, the light weight floating material of the ash comprising mainly of cenospheres was removed and dried. The dried material was then sieved to remove cenospheres of various size fractions. Cenospheres with an average particle size of 10- 100 μm has been used. Their color ranges from white to dark grey. The composite powders were thoroughly mixed with dextrin solution, which was used as a binder to aid pressing the mix powder in the die and also to impart green strength. The mixing was carried out in a laboratory mechanical mixer for about 10 minutes to achieve homogeneity. The composite mixes were pressed into round pellets of diameter 40 mm x 7 mm height size, at a load of 25KN in a hydraulic press. Rapid sintering process through microwave sintering. The sintered composites were evaluated for compressive strength; thermal shock resistant. Copper and cenosphere were mixed with different proportion and grinded for several hours in a planetary ball mill.

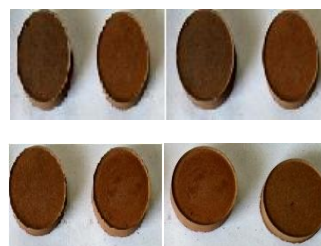


Fig.No.1. Pellets for compression strength

Two sets of composites samples were prepared with 6 mix compositions having 0,10,20,30,40,and 50 volume %cenosphere and the remaining portion of the mix being copper powder for conventional and microwave sintering.

Sintering is the process by which metal powder compacts (or loose metal powders) are transformed into coherent solids at temperatures below their melting point.

During sintering, the powder particles are bonded together by diffusion and other atomic transport mechanisms, and the resulting somewhat porous body acquires a certain mechanical strength.

Sintering process was carried out at different temperatures with the prepared sample to attain the desired properties. Sintering facility operating at 1.1 kW power and microwave frequency of 2.45 GHz as shown in Fig. No.1. This multimode microwave unit was operated at power level of 100 % with programmable controls. The sintering cycle time comprised of 90 minutes for sintering from room temperature to 900⁰ C temperature which included soaking/ dwell time of 60 minutes. The rate of heating was 30⁰c per minute for attaining the temperature of 900⁰ C and the pellets were soaked at this temperature for 60 minutes. Silicon Carbide crucible, a microwave susceptor, was used to hold the sample in the microwave sintering unit to aid sintering.

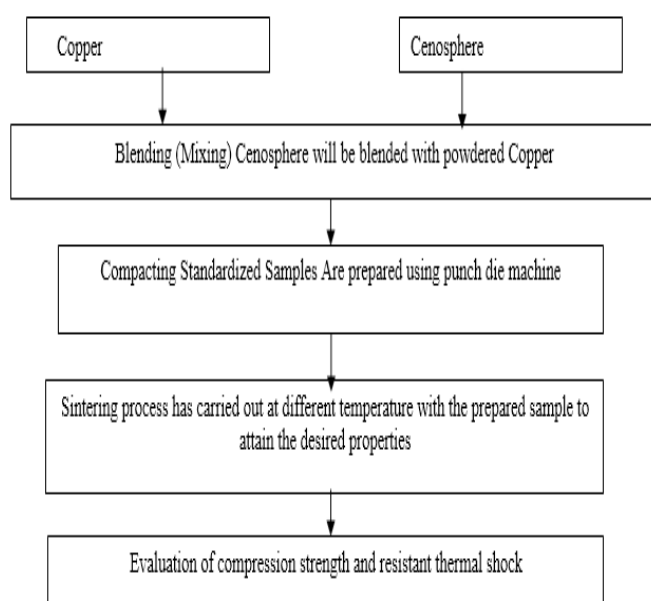


Fig.No.2. Microwave Sintering Facility

The conventionally and microwave sintered samples are subjected to compressive strength (CYS). The test was performed in the UTM machine Enkay make of capacity 100T. The compressive yield strength was evaluated for the sintered samples prior to and after the thermal shock resistance test.

The thermal shock resistance test comprised of heating the composite samples to a temperature of

800⁰C and holding the samples at this temperature for 20 minutes. Immediately the heated samples are quenched in water bath held at ambient temperature. This constitutes 1 cycle composites containing 100 vol. % Copper and Copper-Cenosphere composite samples having 10 and 50 vol. % of Cenospheres only considered for the test. The conventionally and microwave sintered samples were subjected to thermal shock resistance tests comprising of 5, 10 and 25 cycles.



Sintering Process: The powder metallurgy process is the blending of fine powdered materials and pressing these powders into a desired shape. After the desired shape has been achieved, the compressed part is heated in a controlled atmosphere, causing the material to bond. Sintering process, also commonly known as the powder metallurgy process, is divided into three main steps:

1. Blending (The process starts with the blending of [powdered metals](#))
2. [Compaction](#) (load being applied axially.)
3. [Sintering](#) 'green' part formed in the compaction step of the process to meet the required mechanical properties, parts must be sintered.

3. Results and Discussion

3.1 Compression Strength

Figure No3-6 shows the effect of processing parameter on compression strength of composite with varying percentage reinforcement of cenosphere. The results signify that as the sintering temperature increases the compression strength increases for all reinforcement combination of composites.

The copper reinforced with 10% of fly ash cenosphere, has the high ductility than copper content when compared to other material composites prepared. As the % of cenosphere particles increases in the fly ash cenosphere composite the ductility of the material decreases. This increases its proneness to crack formation under loading ultimately results in low compression strength. The above fact can be explained with the results of the composite prepared and shown in Figure No.3-6 with different reinforcements under the same processing parameters. After a detailed literature review, different composites prepared at the compaction load of 25KN, conventional sintering and microwave sintering was conducted on a temperature of 900°C and 950°C respectively for 60min. The compression strength values 54, 51, 49, 46 and 42 MPa obtained for the composites with 10 vol. %, 20 vol. %, 30 vol. %, 40 vol. % and 50 vol. %, cenosphere reinforcement respectively for microwave sintered at 950°C.

The compression strength values are 49, 47, 45, 43 and 40 MPa obtained for the copper fly ash cenosphere composites with 10 vol. %, 20 vol. %, 30 vol. %, 40 vol. %, and 50 vol. %, cenosphere reinforcement respectively for conventional sintered at 950°C as shown in Figure No.3. The compression strength values obtained for the composites with different reinforcement for conventional sintered and microwave sintered

at 900°C shows less strength when compared with 950°C sintered composites.

From the above results it is clearly evident that the compression strength of the copper fly ash cenosphere composite gradually decreases with the increase in cenosphere reinforcement as shown in Figure No.4. This is due to the agglomeration of the cenosphere reinforcement in the copper composite, increases as the percentage reinforcement increases, which leads to weakening of areas leading to crack initiation. As the agglomeration increases, the ability of the matrix to hold the reinforcement decreases which results in sinking of compression strength as the percentage reinforcement increases.

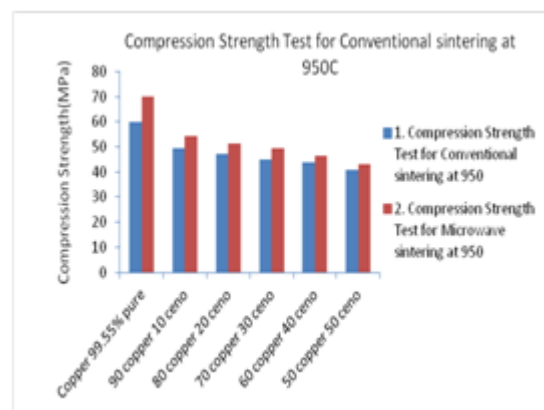


Figure No.3 Compression Strength Test for Microwave sintering at 950°C Vs Compression Strength Test for Conventional sintering at 950°C

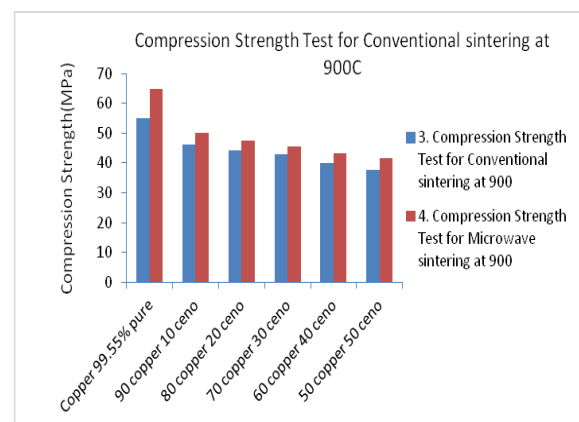


Figure No.4 Compression Strength Test for Microwave sintering at 900°C Vs Compression Strength Test for Conventional sintering at 900°C

3.2 Compressive Strength (MPa) before and After Thermal ShockCycles

The compressive yield strength of the conventionally sintered composites having 10 vol. % and 50 vol. % prior to thermal shock resistance test is 49.36 and 40.96 MPa respectively Figure No.3. For the microwave sintered samples containing 100 vol. % Copper and Copper-Cenosphere composite Cenospheres content the values are 54.31 and 42.91MPa respectively. The compressive yield strength of 10 and 50 vol. % Cenosphere composite conventionally sintered samples decreased after 25 thermal shock resistance cycles. 100 vol. % .Copper- Cenosphere microwave sintered samples the compressive strength is decreased after 25 thermal shock resistance cycles. Microwave sintered samples showed higher compressive yield strength for Copper- Cenosphere composites of temperature 900⁰c and 950⁰c compared to conventional sintered composites samples Copper- Cenosphere (Figure No.5&6).

The development of physical and mechanical properties such as compressive strength is related to the phases formed due to reaction sintering between copper and silica from cenosphere forming alumino silicates and formation of compact microstructure due to microwave sintering. The properties of the composites are better for the microwave sintered samples.

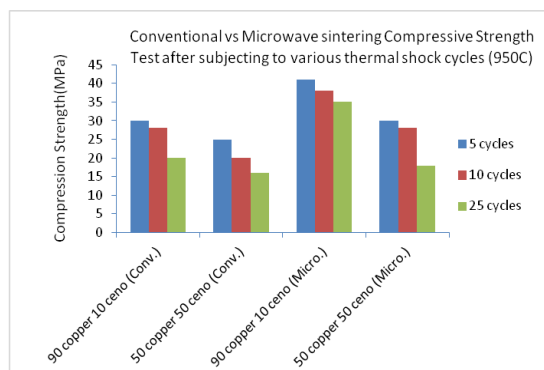


Figure No.5 Conventional sintering Vs Microwave Sintering Compressive Strength Test after subjecting to various thermal shock cycles (5 cycles, 10 cycles and 25 cycles) at 950⁰c

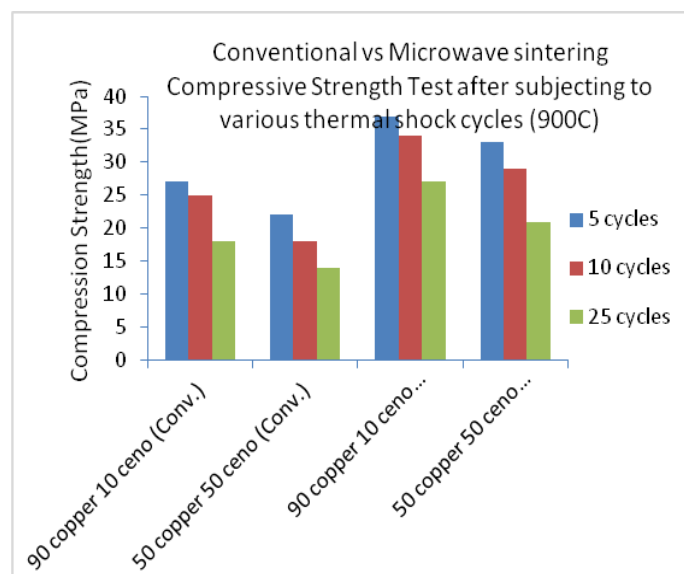


Figure No.6 Conventional sintering Vs Microwave Sintering Compressive Strength Test after subjecting to various thermal shock cycles (5 cycles, 10 cycles and 25 cycles) at 900⁰c

4. Conclusion

1. The copper fly ash cenosphere metal matrix composites were successfully produced by Powder Metallurgy route. The compressive strength is measured for the microwave sintered copper –fly ash cenosphere composites and conventionally sintered composites. The compressive strength is in the range of 70 to 45MPa for microwave sintered composites at 950⁰c.

2. Copper Fly ash cenosphere composites were processed by conventional and microwave sintering technique at 900°C and 950°C respectively. From the above results it is clearly evident that the compression strength of the composite gradually decreases with the increase in cenosphere reinforcement. This is due to the agglomeration of the reinforcement in the composite increases as the percentage reinforcement increases, which leads to weakening of areas leading to crack initiation. As the agglomeration increases the ability of the matrix to hold the reinforcement decreases which

results in sinking of compression strength as the percentage reinforcement increases.

3. The compression strength of copper fly ash cenosphere composites produced by microwave sintering exhibits higher strength values as compared to conventionally sintered copper fly ash cenosphere composites samples.

4. As the volume fraction of fly ash cenosphere is weighted, reduced compression strength values were observed because density of the copper fly ash cenosphere composite was found to decrease with increasing cenosphere content.

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