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A Study on Thermal Behavior of Aluminum Cenosphere Powder Metallurgy Composites Sintered in Microwave

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Abstract

Aluminum Metal Matrix Composites (AMC) comprising of Aluminum powder and Cenospheres were fabricated through Powder Metallurgy (PM) route. Composites containing Cenospheres varying from 10 to 50 vol. % were prepared and dry pressed in hydraulic press. The densification of the composites was carried out in microwave sintering. The sintered composites have been measured for % Porosity, Thermal properties viz. Co-efficient of Thermal Expansion (CTE) and Thermal Shock Resistance (TSR) test. Compressive Yield Strength (MPa) was measured for the composites before and after thermal shock cycles. The measured properties were compared with composites sintered by conventional method in electric resistance furnace. Microwave sintered samples showed better properties compared to conventionally sintered ones.

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Keywords: Cenosphere; Micro-wave Sintering; Aluminium-Metal Matrix Composites; Powder Metallurgy

1. Introduction

Aluminium Metal matrix composites are known to possess higher specific strength, high stiffness and low thermal expansion co-efficient when reinforced with suitable particles compared to their base alloy matrices. There has been an increasing interest in composites containing low density and low cost reinforcements. Among various

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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. Cenospheres can be incorporated into metal as the dispersed phase to make low cost metal matrix composites with a range of useful properties (Sudharshan and Surappa 2008, Guo et al. 1998). One advantage of metal matrix composites is the ability to tailor the thermal properties, such as thermal conductivity and coefficient of thermal expansion through the proper control of reinforcement and matrix. In addition, the manufacturing flexibility of the metal matrix composite by various processes allows the fabrication of complicated shaped parts (Hyo et al. 2000).

Though considered as a waste, cenosphere is a useful by-product to be used to produce newer materials cheaply which otherwise poses major environmental and disposable problems. Fly-ash cenospheres have unique properties such as low density, non-toxic, non metallic hollow micro particles and are light in weight. The particle size of cenospheres varies from 1 to 20 µm and their mean particle diameter in the order of 8 µm. Cenosphere have a size range from 1 to 500 microns with an average compressive strength up to 7000 psi. Their color ranges from white to dark grey. They are also referred to as microspheres, hollow spheres, hollow ceramic microspheres, micro balloons, or glass beads (www.apitco.com). Mineralogical analysis revealed that fly ash cenosphere could be separated into three major matrices: such as mullite, quartz and magnetic spinel. The quartz (SiO_2), alumino silicate, gehlenite, ($Ca_2Al_2SiO_7$) and hematite (Fe_2O_3) are the predominant phase constituents, which influenced the concentration of alumina, silica and iron oxide (Wu et al. 2007)

Cenosphere can make an effective reinforcement material in the metal matrix composites. Metal matrix composites comprising of aluminium metal as matrix embedded with fly ash/ cenosphere particulates can be fabricated through the powder metallurgy route. Powder metallurgy (PM) is an attractive processing technique to produce near net shape products and is commonly used for the fabrication of engineering components and particulate reinforced metal matrix composites. The basic process involved in PM technology route is mixing and blending of powders, consolidation of the same and sintering the consolidated powders for densification. The densification process is generally carried out by conventional methods using a furnace, but other sintering methods such as microwave assisted sintering are gaining importance (Anklekar et al. 2005, Nawathe et al. 2009).

Microwaves can be defined as that part of the electromagnetic radiation spectrum having a wavelength ranging from about 1 mm to 1 metre in free space and the frequency ranging from 300 MHz to 300 GHz. However, only narrow frequency bands centered at 915 MHz and 2.45 GHz, 28-30 GHz and 80-81 GHz are actually permitted for research purposes. They are coherent and polarized waves obeying the laws of optics. Depending upon the type of material the microwave may get transmitted, absorbed or reflected by the materials. Microwave processing is quite significant and unique in recent times for material sintering because of its intrinsic advantages such as rapid heating rates, reduced processing times, uniform temperatures with minimal thermal gradients. Microwave sintering leads to substantial energy savings with higher efficiency, improved properties, finer microstructures, environmental friendly process and with less environmental hazards (Agrawal 1998, Oghabaei and Mirzaee 2010, Sutton 1989).

Microwave sintered aluminium cenosphere composites have good microstructure that helps to produce materials with better properties such as light weight, dense, hard, better engineering properties including effective EMI shielding and vibration damping properties. The composites have also been reported to give rise to significant improvements in mechanical properties such as high strength, high stiffness, better thermal properties like low coefficient of thermal expansion and lower thermal conductivity, better acoustic property and damping characteristics. In the conventional heat treatment, the heat penetrates the body from outside to inside creating a temperature gradient; therefore it is not possible to heat those samples at a very high heating rate. This results in long cycle time and makes the process energy intensive (Satapathy et al. 2012).

The purpose of the study is to fabricate aluminum matrix composite with cenospheres as reinforcement through powder metallurgy route and densification of the composite by sintering in microwave. The composite are studied for porosity, thermal behavior and compression strength.

2. Experimental Procedure

2.1 Raw Materials and Processing

Atomized Aluminum metal powder of 99.5% purity with a particle size of about ASTM 200 mesh (75 μm) obtained from M/s. NICE Chemicals make laboratory reagent powder was used for the matrix. Cenosphere was obtained from fly-ash received from M/s. NTPC Simhadri Thermal Power Station, India. 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 in this study and the SEM image of cenospheres is shown in Figure1.

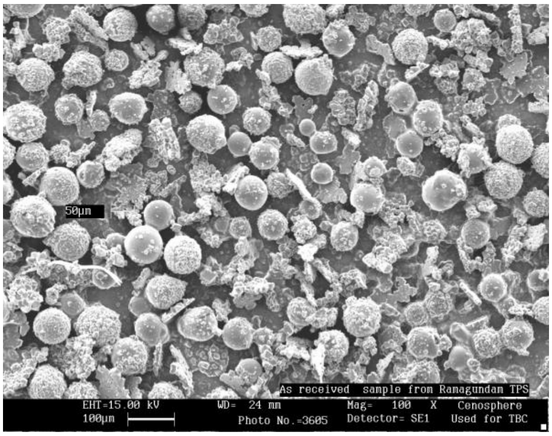


Fig.1 SEM Image of Cenospheres in Fly Ash

Six composite mixes of Aluminum powder and Cenospheres were prepared and the compositions are as follows:

Table No.1 Composition of the Aluminum and Cenosphere mix

Sample	Composition (Volume %)
1	Aluminum-100
2	90 Aluminum : 10 Cenospheres
3	80 Aluminum : 20 Cenospheres
4	70 Aluminum : 30 Cenospheres
5	60 Aluminum : 40 Cenospheres
6	50 Aluminum : 50 Cenospheres

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 25 kN in a hydraulic press. Two sets of composite pellets were prepared, one set of pellets to be sintered in Microwave and the other set through conventional resistance heating.

2.2 Sintering

One set of pellets was sintered at a temperature of 665 ±5^{0}C which is near about the liquidus temperature of Aluminum, in a BHEL make Microwave Sintering Facility operating at 1.1 kW power and microwave frequency of 2.45 GHz. 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 670^{0}C temperature which included soaking/ dwell time of 42 minutes. The rate of heating was 12^{0}C per minute for attaining the temperature of 665^{0}C and the pellets were soaked at this temperature for 42 minutes. Silicon Carbide crucible, a microwave susceptor, was used to hold the sample in the microwave sintering unit to aid sintering. The 2nd set of pellets was sintered conventionally in a muffle type resistance furnace heated with kanthal element, to a temperature of 670^{0}C and the sintering duration was 8 hours.

3. Results and Discussions

3.1 X-ray Diffraction and Scanning Electron Microscopy studies of the Composites:

The sintered pellets were characterized for its mineralogical phases by using PAN analytical make XPert PRO Model X-ray Diffractometer and morphology using Scanning Electron Microscope (SEM) Leica make Q500MC Model. The mineralogical phase analysis of conventionally sintered aluminum cenosphere pellets by XRD reveals that aluminum-cenosphere pellets have more crystallinity which is evident from sharp crystalline peaks. From the XRD peaks (Figure 2) it is evident that the predominant compounds present in composite are Aluminum and Mullite phases. The alumino silicate compound present as mullite phase in the cenosphere is depicted in the XRD pattern.

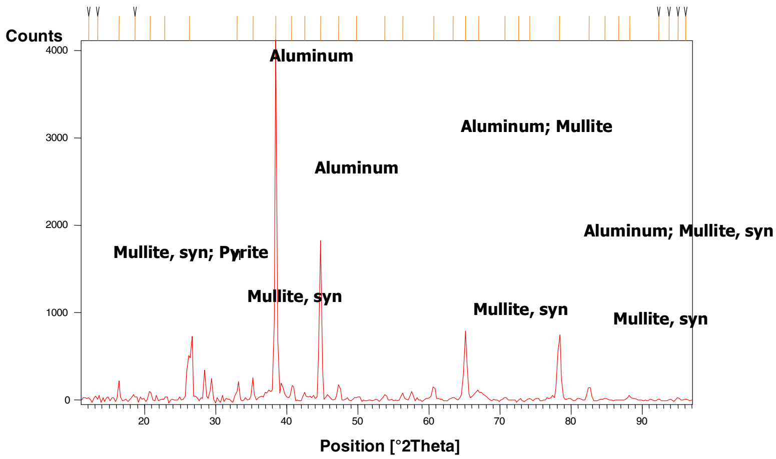


Fig. 2 X-ray pattern of the composite sintered conventionally at 670°C

The mineralogical phase analysis of microwave sintered aluminum cenosphere pellets by XRD also reveals that aluminum-cenosphere pellets have more crystallinity which is evident from sharp crystalline peaks. From the XRD peaks (Figure 3) it is evident that the predominant compounds present in composite are Aluminum and Aluminum Silicon phases. Sudharshan et al. 2008 has reported that there is a possibility of chemical reaction with aluminum melt and fly ash cenosphere particles. As cenosphere consists of predominantly silica, alumina and iron oxide, there is a chemical reduction taking place with these oxides when they come in contact with the melt, and the reactions are as follows:

$$2Al_{(1)} + 3/2\ SiO_{2(s)} = 3/2\ Si_{(s)} + Al_2O_{3(s)} \quad (1)$$

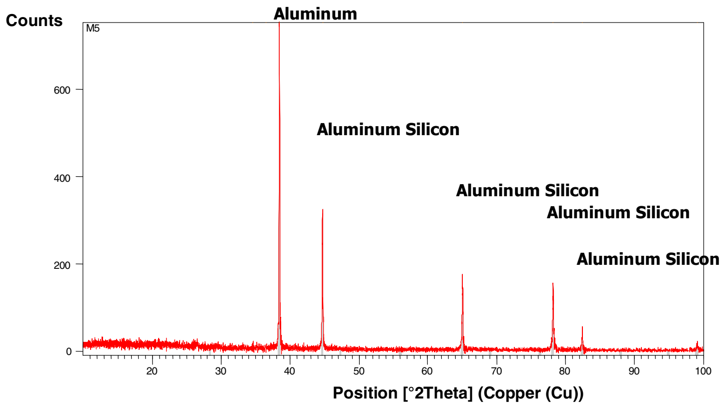
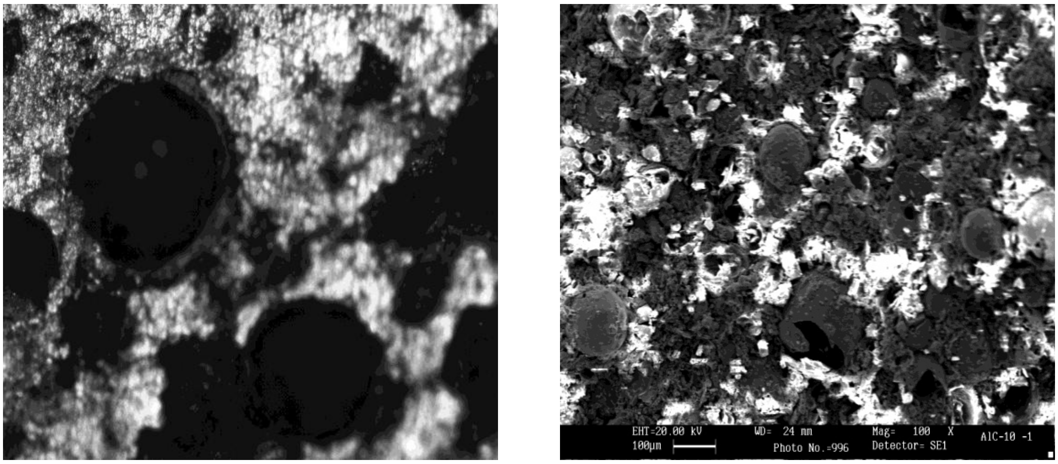


Fig. 3 X-ray pattern of the composite sintered in microwave at 675°C

The matrix continues to be Aluminum with eutectic Aluminum Silicon phase formed by reduction reaction alloying with the matrix.

The microstructure (Figure 4) of the sintered composite reveals uniform distribution on Cenospheres in the Aluminum matrix. However at some portions clusters of Aluminum melt with ruptured Cenospheres is observed with pores open to the surface, thereby contributing to increase in porosity.



(a) (b)

Fig. 4 Microstructures of the composite sintered a) conventionally b) microwave, at 675°C at 100X

3.2 Measurement of Apparent porosity (%)

The apparent porosity (Pa) of the samples was calculated by water immersion method using Archimedes's principle.

$$P_a\,\% = \frac{(m_3 - m_1)}{(m_3 - m_2)} \times 100 \qquad (2)$$

Where m_1 is the mass of a dried sample in air (g); m_2 is the mass of the saturated suspended weight of sample in water (g); m_3 is the mass of the sample saturated with water.

The variation in % porosity as a function of increase in cenosphere content for the Aluminum- Cenosphere composites in this study is shown in figure 5. The porosity of the pure Aluminum composites sintered in microwave appears to be on the lower side as compared to the conventionally sintered ones. But the porosity of the composites increased with the increase in cenospheres content from 10 to 50 volume % in both the sintered samples. Microwave sintered samples showed porosity increase of 13.2% with increase of cenospheres content from 10 vol. % to 50 vol. %, whereas conventionally sintered samples showed 16.8 % porosity increase. Microwave sintered samples showed less porosity compared to the conventionally sintered ones. Obviously, the porosity of the composites increases with the increase in volume % of Cenospheres.

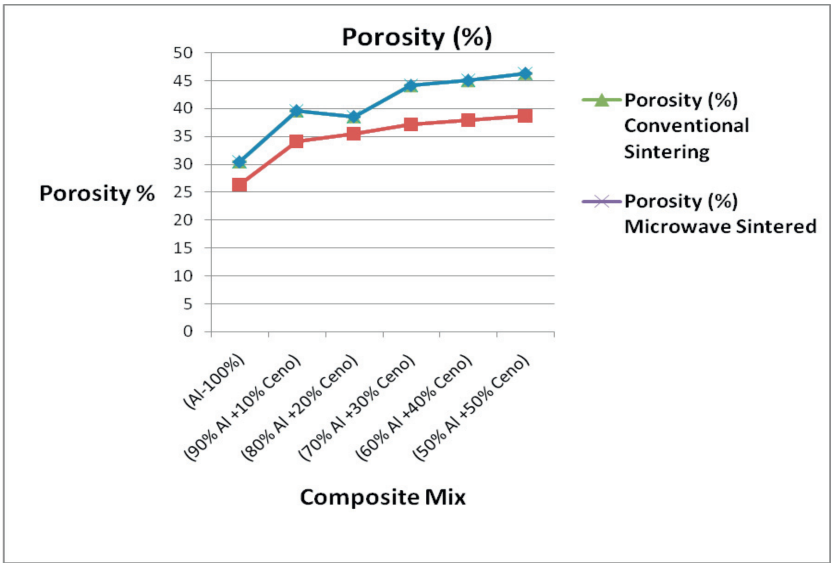


Fig. 5 Apparent Porosity (%) of composites sintered conventionally and in microwave

3.3 Compressive Yield Strength (MPa) before and After Thermal Shock Cycles

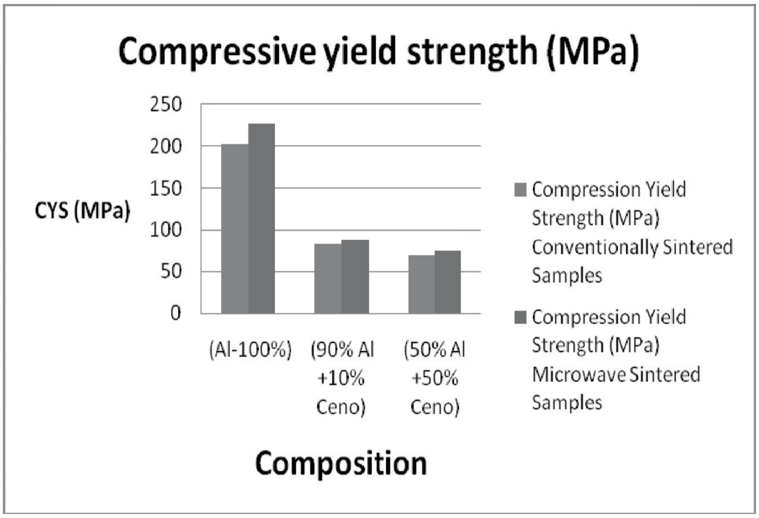
The conventionally and microwave sintered samples were subjected to compressive yield strength (CYS). The test was performed in the UTM machine Enkay make of capacity 100T. The compressive yield strength was evaluated for the sintered samples prior to and after the thermal shock resistance test.

The thermal shock resistance test comprised of heating the composite samples to a temperature of 500^0C and holding the samples at this temperature for 15 minutes. Immediately the heated samples are quenched in water bath held at ambient temperature. This constitutes 1 cycle composites containing 100 vol. % Aluminum and Aluminum-Cenosphere composite samples having 10 and 50 vol. % of Cenospheres only were studied for the test. The conventionally and microwave sintered samples were subjected to thermal shock resistance tests comprising of 5, 10 and 25 cycles.

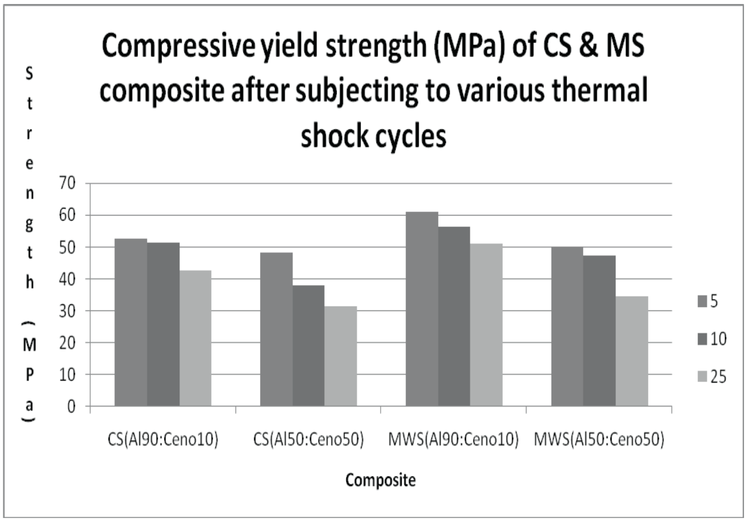
The compressive yield strength of the composites having 10 vol. % and 50 vol. % prior to thermal shock resistance test was found to be 83.4 and 69.4 MPa respectively for conventionally sintered samples (Figure 6a). For the microwave sintered samples of the same vol. % Cenospheres content the values were 88.5 and 74.2 MPa respectively. The compressive yield strength of 10 and 50 vol. % Cenosphere composite conventionally sintered samples decreased by 48.9% and 54.4 % respectively after 25 thermal shock resistance cycles and for composites of same vol. % microwave sintered samples the compressive yield strength decreased by 42.3 % and 53.6 %

respectively after 25 thermal shock resistance cycles. Microwave sintered samples showed higher compressive yield strengths for both composites (Figure 6b).

The development of physical and mechanical properties such as compressive strength is related to the phases formed due to reaction sintering between aluminum 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. As the density increases (and porosity decreases), the compressive strength increased and the porosity is decreased (ASM Handbook 1998). Sundaram et al. 2011 have observed that the Aluminum composite's density and strength decrease with increasing vol. per cent of fly ash cenospheres in the composite.



(a)



(b)

Fig. 6 Compressive Yield Strength (MPa) of composites a) prior to thermal shock, b) after thermal shock cycles

3.4 Co-efficient of Thermal Expansion

The Co-efficient of Thermal Expansion (CTE) test was conducted on the composite samples using Orton Dilatometer, USA make.The samples were cut into cylindrical shape of size 10 mm dia and length 25 mm. The samples were heated to a temperature of 500^{o}C with rate of heating at 3^{o}C/min in oxidizing atmosphere. The change in length Δl of the samples were measured at 500^{o}C for all the composites.

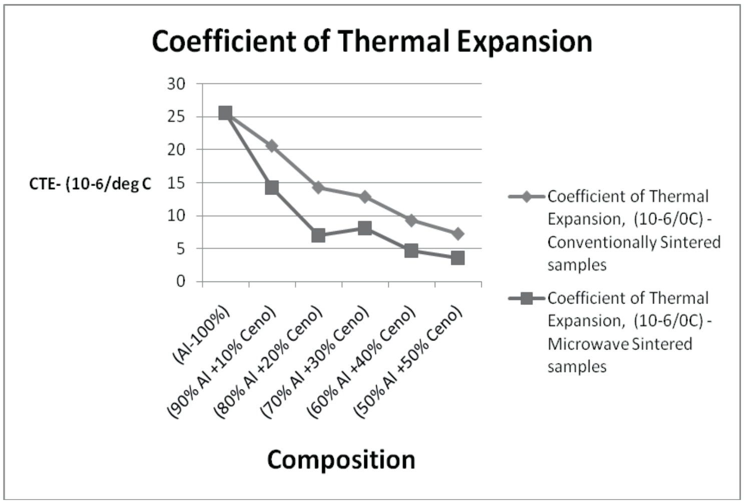


Fig. 7 Co-efficient of Thermal Expansion of composites sintered conventionally and in microwave

The Coefficient of Thermal Expansion (CTE) of the composites for pure aluminum powder samples showed value of 25.6 x $10^{-6}/^{0}$C which is near the theoretical value of pure aluminum. The CTE of the composites decreased as the cenospheres content increased from 10 vol. % to 50 vol. % i.e. from 20.7 to 7.3 x $10^{-6}/^{0}$C for the conventionally sintered samples (Figure 7). For the microwave sintered samples, the CTE of the composites decreased as the cenospheres content from 14.3 to 3.6 x $10^{-6}/^{0}$C which is much lower than the conventionally sintered samples.

Rohatgi et al. 2006 has reported that Cenosphere is ceramic in nature, and that the ceramic particles will have low coefficient of thermal expansion when compared to metallic samples and therefore the incorporation of cenospheres which is a ceramic material, as filler in the metal matrix composite, will reduce the CTE of the resulting composite. Since cenosphere is also an alumino-silicate, this also reduces the CTE in aluminum matrix. This is evident from the CTE values which shows that as the cenospheres content is increased from 10 to 50 %, the CTE is also reduced in both the conventionally and microwave sintered samples. Microwave sintered samples still showed lower CTE compared to conventional sintered samples.

Guo et al. 1998 have also reported that a ceramic phase is formed at the interface between cenospheres and Aluminum due to reaction of Al with Si present in Cenospheres. This ceramic phase formation is also one of the reasons for reduction in CTE of the composite containing cenosphere.

4. Conclusions

The following are concluded based on the study of the Aluminum Cenospheres composites' for thermal properties.

- It is possible to produce Aluminum Cenosphere metal matrix composites through powder metallurgy route with sintering in microwave for applications in products desired for thermal insulation properties.
- Microwave sintering is rapid, economical and fast. The microwave sintered samples show better thermal properties like co-efficient of thermal expansion, thermal shock resistance than the conventionally sintered ones. This is attributed to the fine microstructure and the relative phases that are developed in the microwave sintered product which enhances the properties of the composite including thermal properties.
- Microwave sintering also aids in achieving properties such as high compressive yield strength and reduced porosity.

- The composite mix and the amount of cenospheres that can be loaded into the matrix needs to be optimized to design product with desired thermal and mechanical properties, which are cheap, economical, through microwave sintering.

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