THINK INDIA JOURNAL

ISSN:0971-1260
Vol-22-Issue-37-December -2019

Experimental investigation to analyse the effect of graphene in ceramic matrix composite prepared by vacuum pressure sintering

Mandeep Singh Rayat[1*], Ranjit Singh[2], Hitesh Arora[3]
[1,2,3] *School of Mechanical Engineering, Lovely Professional University*
**Corresponding Author, email- mandeep.16445@lpu.co.in*

Abstract-

This paper presents the study on alumina ceramic reinforced with graphene nano powder and its influence on the mechanical properties of the composite. Initially, graphene nano powder was mixed in alumina powder by using planetary ball milling apparatus to get uniform dispersion. The sintering of the samples was performed in high vacuum pressure sintering with inert atmosphere at 1500^0C. The density of the composites was higher than that of the pure alumina with a maximum increase of + 2.22% for 2 wt% while the relative density has shown decrease with increase in graphene concentration. Similarly, the porosity of the composites also decreased with increase in graphene concentration with maximum percentage of 11.11% for 2wt. %. From the SEM analysis, it was found that the graphene was homogenously dispersed in the alumina matrix.

Keywords*- alumina, graphene, high vacuum sintering, density, porosity****.***

1. Introduction

In general, material is trivial term that is used for a mixture of substances that together constitute a single product [1]. While the combination of materials has two or more substances in it their nature may not remain the similar, i.e. the substances might be pure or impure, singular or complex, or either natural or artificial [3]. Materials are generally the raw materials used for production and manufacturing process in industries.

A composite material is defined as a material which is composed of two or more materials at a microscopic scale and has chemically distinct phases. A composite consists of two materials. The composites are generally classified into three types such as metal matrix composite, polymer matrix composite and ceramic matrix composite. Metal matrix composites constitute at least two parts- two different metals or combination of a metal and another material. The secondary material is provided as reinforcement for the metal matrix to improve its strength and its wear resistance [2]. Polymer matrix composite generally consist of a polymer matrix that is combined

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

with fiber reinforcement. Polymer is a large molecule composed of iterating units of monomers that are connected by covalent chemical bonds [2]. Ceramic matrix composites consist of ceramic fibers that are embedded in ceramic matrix, which results in the formation of a ceramic fiber reinforced ceramic. The matrix and fibers can consist of any ceramic material [1].

Padmavathi et al. [4] – In this research the effect of sintering temperature and graphite addition (up to 1.5%) on the corrosion response of austenitic and ferritic stainless steel has been investigated. Ferritic stainless steel 434L and austenitic stainless steel 316L were used in this research. The specimen was heated in conventional way and also with microwave furnace. When compared, the specimens that were heated in microwave furnace showed higher densification and better corrosion properties than the specimen that was heated using convention sintering method. Pietrzak et al. [5] – This paper is about sintered Al_2O_3 - Cr composites consisting of different chemical composition (from 25 to 75 vol.% of each component). The parameters taken into consideration while analysis is time (15 to 60 min), pressure (0.5 and 30MPa), temperature (1200 – 1600^0C) and powder grain for Al_2O_3 (80nm or 1µm). It was revealed that the nano-powder composites have higher densities than (by about 1-2%) micro-powder composites. By using 30MPa of pressure the sintering temperature obtained was 200^0C lower when compared to exposure to pressure of 0.5MPa. Zhou et al. [6] – This is a study based on the various particle sizes of filler alumina and the matrix that was used was silicone rubber to obtain a elasto-metric pad with 1.45 W/mK thermal conductivity that is required to dissipate the heat of the micro-electronics. The parameters such as the amount, size of the particles and the mixing ratio of the filler materials on the thermal conduction attribute and mechanical characteristics of silicone rubber were investigated. The results showed that the thermal conductivity of the larger grain size filler materials was the highest and the rubber with a mixture of hybrid particles with desirable mass ratio had high thermal conduction rather than rubber with filler of a single particle size. In addition, it is also stated that a surface treatment with 3-methacryloyloxypropyltrimethoxysilane of the hybrid filler can enhance the composite rubber's thermal conduction.

Fan et al. [7] – fabricated Al_2O_3 composites with homogeneously distributed graphene nano-sheets (GNS) of 2.5 to 20 nm thickness. For the homogeneous distribution of GNS they used ball mill apparatus followed by spark plasma sintering. The conductivity of the composite reaches

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

5709S/m when the composite has 15 vol% GNSs. The electrical conductivity of the prepared composites was temperature dependent and they behave like semimetal. This work paves a new way for the fabrication of GNS/ceramic composites with better electrical properties. Galusek et al. [8] – Al_2O_3-SiC nanocomposites with 3-8 wt. percent of SiC were manufactured in this research by pre-sintered in situ thermal decomposition of a preceramic polymer SiC in the Alumina matrix. The SiC volume fraction was controlled and adjusted by the polymer solution concentration as well as the condition of pyrolysis and sintering. The decomposition reactions in the composites were suppressed by the use of powder bed in producing SiO, CO and other volatile species proved to be vital in the successful densification of the composites. Analysis of the phases and structure morphology were performed with the help of SEM, XRD and TEM.

Karagedov et al. [9] – This research deals with the preparation and sintering of pure nanocrystalline α-alumina powder. Aluminum nitrate solution is being seeded with ≥ 0.5 wt.% of 25mm α-alumina powder. Depending on the amount of seeds and the level fusion of the contents in the aqueous form the entire transformation is done from 800 – 930^0C. the resulting sunder mild conditions, destroys the agglomerates and results in the production of Nano powder of which the dense bodies (95 – 99%) have been sintered at around 1300 – 1400 ^{0}C. Rashad et al. [10] – The effect of Graphene Nano-platelets (GNP) on the mechanical properties of magnesium – 10 wt.% titanium (Mg-10Ti) alloy was studied in this research. The Mg – 10Ti alloy with 0.18 wt.% GNP was synthesized by semi powder metallurgy preceded by hot extrusion method. The microstructure of the composite revealed that the matrix had uniform distribution of the reinforcement (Ti and GNP) particles. Tensile results that were done under room temperature revealed that there has been an increase of 0.2% in yield strength, failure strain and ultimate tensile strength. Scanning electron microscopy (SEM) was used to study the surface morphology while Energy – Dispersive X-ray spectroscopy (EDS) was used to investigate the elemental dispersion and X-ray diffraction (XRD) was used to perform the phase analysis. Rashad et al. [11] – In this study Aluminum – Graphene Nano-platelets composite was fabricated using semi powder metallurgy preceded by hot extrusion. The effect of GNP on the tensile, compressive and hardness properties of Al is investigated in this research. The addition of GNP in the weight ratio of 0.3% to the Al matrix resulted in 0.2% increase in yield strength, ultimate tensile strength, hardness and decrease in failure strain and ultimate compression strength (in comparison with monolithic aluminum).

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

Yang et al [12] prepared alumina- reduced graphene nano oxide composite with two step process. From the analysis it was found that thermal conductivity of composite increased with the increased concentration of graphene nano oxide particles.

In this study a ceramic composite was fabricated by using graphene in a different weight proportion in order to study its effect on mechanical properties of a composite.

2. Experimental Details:

The following steps were performed for the fabrication of alumina-graphene composite

2.1. Mixing: Mixing of matrix and reinforcement (having specified weight concentration) was done with the help of ball milling in order to blend the materials to get a uniform size and homogenous dispersion of reinforcement into matrix phase

2.2. Compaction: Once the mixing is done the next step is to make compacts of the powder of the desired proportions of the materials. For this purpose the hydraulic press of capacity up to 50 ton was used. For the compaction process a die of steel having internal diameter of 10 mm were used. And, then the compacts were sintered. The compacts of the materials are produced under high pressure.

2.3. Sintering: For the sintering purpose high vacuum pressure furnace was used and the compacts were sintered at 1500^0 C in an inert atmosphere as well as under high vacuum.

2.4. Properties Evaluation:

2.4.1. Density and porosity tests:

The samples density was tested using Archimedes principle. The samples were weighted in air using a digital weighing scale. Then their weight was measured by tying the sample to a thread and suspending them in water. The weight of the water in beaker was measured and then the sample was suspended in the beaker containing water and then the change in weight was noted. The difference between the weight of water with the suspended sample and the weight of water alone was noted.

The volume of the composite samples was calculated using the following formula,

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

Volume (V) = $\prod * r^2 * h$

Formula to calculate the density of the composite:

Density (ρ_c)= average weight of the samples in air / volume of the composite

Formula to calculate relative densities of the composite:

Relative Density (ρ_{th}) = 1 / [(weight of graphene in the samples / density of graphene) + (weight of alumina in the samples / density of alumina)]

The porosity of the composite samples was calculated by the following formula,

Porosity = 1 – [density of the composite / relative density of the composite]

2.4.2. Microstructural Examination:

SEM is incorporated to analyze the morphology and microstructure of the materials of the composite after fabrication.

3. Results and Discussion

3.1. Density and porosity test results:

The density and porosity of the prepared composite samples were tested using Archimedes water displacement method. This test was performed on the circular samples with weight ratios of 0 wt%, 0.5wt. %, 1wt. %, 2wt. % graphene. The readings noted are given in the table 1.

Table 1: Weight of samples in air

Weight ratio	1st reading	2nd reading	3rd reading	Average	Weight of alumina in grams	Weight of graphene in grams
0 wt%	0.64 g	0.67 g	0.63 g	0.6466 g	0.6466 g	0.00 g
0.5 wt. %	0.64 g	0.66 g	0.65 g	0.65 g	0.6467 g	0.0032 g

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

1 wt. %	0.64 g	0.65 g	0.66 g	0.65 g	0.6435 g	0.0065 g
2 wt. %	0.669 g	0.671 g	0.672 g	0.6706 g	0.6572 g	0.0134 g

The volume was calculated to be 0.2356 cm^3. Then the densities of the composites were calculated and recorded in the table 2 below.

Table 2: Density of the samples

Weight ratio	Density of the sample
0 wt %	2.7444 g/cm^3
0.5 wt. %	2.7589 g/ cm^3
1 wt. %	2.7589 g/ cm^3
2 wt. %	2.8066 g/ cm^3

It is noted that the samples with highest content of graphene (i.e. 2 wt. % of graphene reinforcement) has the highest density, compared to the 0.5 wt. % and 1wt. %.

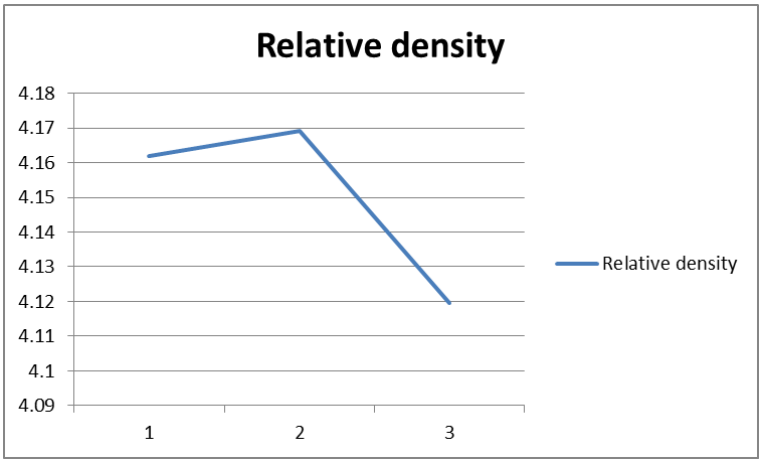


Fig. 1: Relative density of the samples

From the values relative density is noted to decrease with increase in the graphene content. 0.5 wt. % composite has the highest relative density (i.e. 4.1619 g/ cm^3), while 2 wt. % composite has the lowest (i.e. 4.1196 g/ cm^3) as shown in figure 1.

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

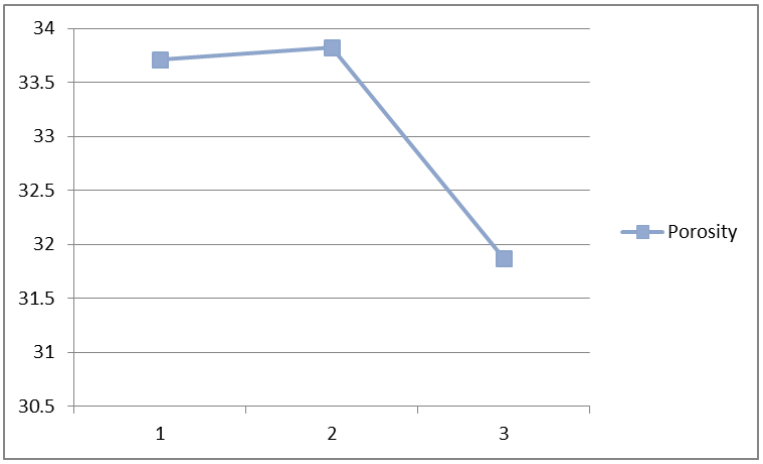


Fig. 2: Porosity of the samples

The porosity of the composite samples has shown decreasing trends with increase in graphene content of the composite. The porosity of 0.5 wt. % of graphene is higher than the 2 wt. % of graphene composite as shown in figure 2.

3.2. SEM and EDS analysis results:

Scanning electron microscopy (SEM) was performed at SAI labs at Thapar University. The purpose of performing SEM analysis was to check the dispersion and presence of graphene in alumina and to check the microstructure of the composite. 2 wt. % samples were used and was gold coated and then placed in the SEM apparatus. The sample was subjected to magnification of 1000x, 2500x, 7500x and 10000x as shown in figure 3. As the magnification was being increased the resolution of the image was being distorted.

The SEM analysis was followed by EDS (energy dispersive X-ray spectroscopy). EDS showed the presence of Carbon, Aluminum and Oxygen in the composite sample. The values for the EDS are tabulated in the table 3-7 below.

Table 3: EDS spectrum 1 analysis for the elements present in the sample

Element	Weight%
C	6.66%
O	38.38%

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

Al	54.96%

Table 4 : EDS spectrum 2 analysis for the elements present in the sample

Element	Weight%
C	5.50%
O	47.35%
Al	47.15%

Table 5: EDS spectrum 3 analysis for the elements present in the sample

Element	Weight%
C	3.34%
O	47.44%
Al	49.22%

Table 6: EDS spectrum 4 analysis for the elements present in the sample

Element	Weight%
C	3.07%
O	48.47%
Al	48.45%

Table 7: EDS spectrum 5 analysis for the elements present in the sample

Element	Weight%
C	23.66 %
O	37.45 %
Al	38.90 %

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

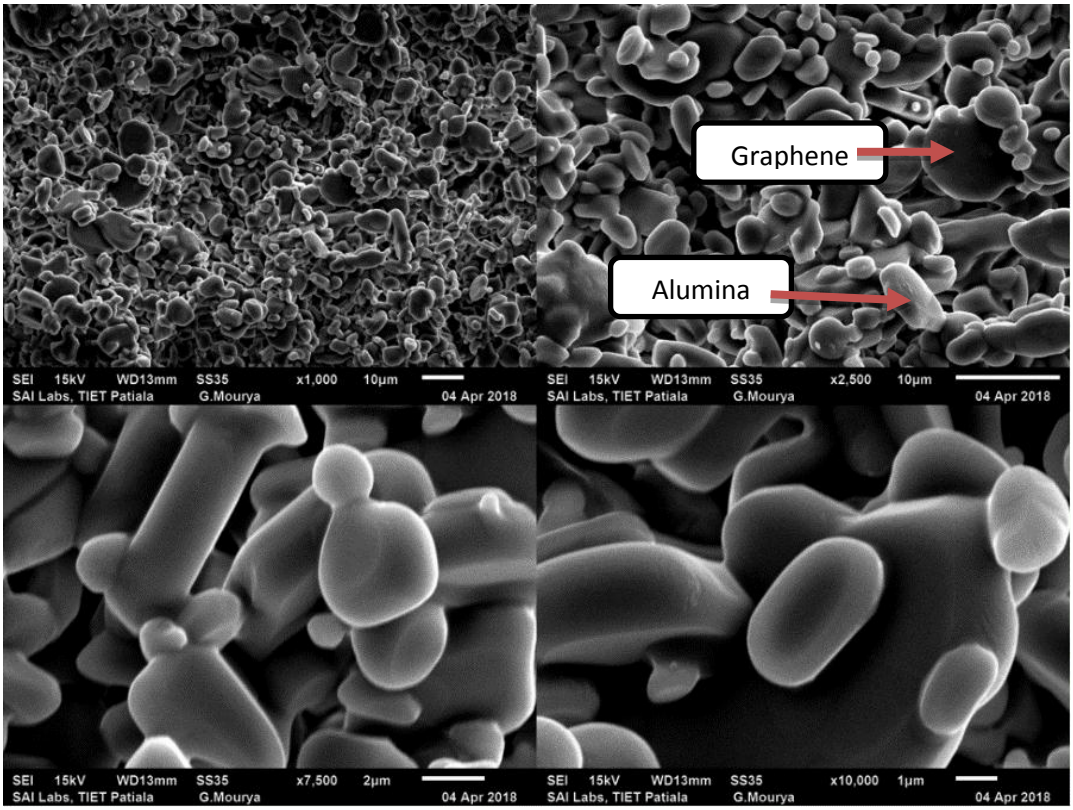


Fig 3.: SEM analysis on 2 wt. % sample with magnification 1000x (top left), 2500x (top right), 7500x (bottom left) and 10000x (bottom right)

4. Conclusion

From the result obtained from the tests, it is evident that,

- The addition of graphene has increased the density of the composite as compared to the pure alumina while there is a decrease in relative density of the composite with higher percentage of graphene.
- The porosity of the composite with higher weight percentage of graphene was decreased. This decrease in the porosity is because of the voids in the composite being filled by graphene, hence the decrease in porosity with increase in graphene content.
- From the microstructural evaluation it was found that the dispersion of graphene in alumina was uniform and from the EDS analysis confirmed the presence of carbon in the composite which is the main constituent of graphene.*

Copyright ⓒ 2019Authors

ISSN:0971-1260
Vol-22-Issue-37-December -2019

References:

1. Groover, M. P. (2010). Welding Processes. *Fundamentals of Modern Manufacturing, 4th ed. Hoboken: Wiley*, 713.2. Callister, W. D., & Rethwisch, D. G. (2007). *Materials science and engineering: an introduction* (Vol. 7, pp. 665-715). New York: John wiley & sons..

3. Martin, J. W. (2006). *Concise encyclopedia of the structure of materials*. Elsevier.

4. Padmavathi, C., Joshi, G., Upadhyaya, A., & Agrawal, D. (2008). Effect of sintering temperature, heating mode and graphite addition on the corrosion response of austenitic and ferritic stainless steels. *Transactions of the Indian Institute of Metals*, *61*(2-3), 239-243.

5. Pietrzak, K., Chmielewski, M., & Włosiński, W. (2004). Sintering Al2O3–Cr Composites Made From Micro-and Nanopowders. *Science of Sintering*, *36*(3), 171-177.

6. Zhou, W., Qi, S., Tu, C., Zhao, H., Wang, C., & Kou, J. (2007). Effect of the particle size of Al2O3 on the properties of filled heat-conductive silicone rubber. *Journal of Applied Polymer Science*, *104*(2), 1312-1318.

7. Fan, Y., Wang, L., Li, J., Li, J., Sun, S., Chen, F., ... & Jiang, W. (2010). Preparation and electrical properties of graphene nanosheet/Al2O3 composites. *Carbon*, *48*(6), 1743-1749.

8. Galusek, D., Klement, R., Sedláček, J., Balog, M., Fasel, C., Zhang, J., ... & Riedel, R. (2011). Al2O3–SiC composites prepared by infiltration of pre-sintered alumina with a poly (allyl) carbosilane. *Journal of the European Ceramic Society*, *31*(1-2), 111-119.

9. Karagedov, G. R., & Myz, A. L. (2012). Preparation and sintering pure nanocrystalline α-alumina powder. *Journal of the European Ceramic Society*, *32*(1), 219-225.

10. Rashad, M., Pan, F., Tang, A., Lu, Y., Asif, M., Hussain, S., ... & Mao, J. (2013). Effect of graphene nanoplatelets (GNPs) addition on strength and ductility of magnesium-titanium alloys. *Journal of Magnesium and alloys*, *1*(3), 242-248.

11. Rashad, M., Pan, F., Tang, A., & Asif, M. (2014). Effect of graphene nanoplatelets addition on mechanical properties of pure aluminum using a semi-powder method. *Progress in Natural Science: Materials International*, *24*(2), 101-108.

12. Yang, G., Yu, X., Wu, Q., Zhang, H., Li, W., & Qian, Y. (2018, July). Preparation and Thermal Conductivity of Alumina/Reduced Graphene Oxide Composite Dispersed Aqueous Nanofluids. In *IOP Conference Series: Materials Science and Engineering* (Vol. 381, No. 1, p. 012071). IOP Publishing.

Copyright ⓒ 2019Authors