A Novel Alumina-Reduced Graphene Oxide Composite: Synthesis and Characterizations

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Abstract- A novel new alumina (Al_2O_3) matrix with reinforced of reduced graphene oxide (RGO) composites have been prepared by an in-house designed horizontal high energy planetary ball milling process. Al_2O_3-RGO composites can be considered as a novel material for many industrial applications because of favourable combination of low density, improved mechanical properties, retaining strength under higher temperatures and high thermal conductivity. The effect of RGO in the combination of Al_2O_3 have potential scope in several demanding fields like automobile, aerospace, defence, sports, electronics, bio-medical and other industrial purposes. The prepared composites have been studied by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), micro Raman spectroscopy and microhardness measurement. It is interestingly observed that microhardness and Young's modulus of the composites were increased with increasing content of RGO from 1 wt% to 3 wt% in Al_2O_3 matrix. The composite Al_2O_3-RGO with 3 wt% RGO shows microhardness and Young's modulus of 2008 ± 18 VHN and 520 ± 8 GPa respectively.

Keywords: Reduced Graphene Oxide, Composite, Microstructural, Mechanical Property

1. INTRODUCTION

Nature always insists us for developing advanced materials through intelligent way with superior properties. As the process of development, different materials in elemental, compound composites and their integrated forms are continuously developing to fulfill the material requirement of smart and advanced society. In this backdrop, the present authors have taken attempt to produce advance composites of alumina-reduced graphene oxide because of their superior properties. Alumina (Al_2O_3) is taken as an attractive ceramic for advanced high temperature structural applications and it is currently used in electronics, aerospace and automotive industries [1-5]. Though Al_2O_3 inspiring mechanical hardness and chemical inertness, moderate thermal conductivity, High corrosion and wear resistance, Low density (3.75 to 3.95 g/cm^3), but it has the poor fracture toughness which restricts Al_2O_3 use for advanced high temperature structural applications [6]. The most obvious tactics to improve the mechanical properties has been to add a reinforcing agent to formulate a nanostructured composite material. In this respect, graphene (reduced graphene oxide (RGO)) was evaluated as the reinforcing agent of Al_2O_3 ceramics using ball milling followed by sintering. The addition of graphene to Al_2O_3 simultaneously improved the fracture toughness and hardness of alumina–graphene composite. The graphene has shown exceptionally high mechanical (Young's modulus of 1.0 TPa), high electron mobility (15000 $cm^2 V^{-1}S^{-1}$), 100 times stronger than steel, high electrical (less resistivity than silver) and thermal properties, which make it one of the most promising reinforcements for ceramic matrix composites (CMCs) [7-10]. This 2-dimetional (2D) sheet of carbon has high surface areas compared to graphite, carbon black and CNTs, thus a small loading (less than 1.0 vol%) in a matrix may lead to large improvements in the composite properties.

In this work attempt has been taken to prepare Al_2O_3-RGO composite by ball milling followed by sintering technique. The morphology, structure, composition and hardness of composites were studied using various analytical techniques such as X-ray diffraction (XRD), Field emission, scanning electron microscopy (FESEM) and Nanoindentation.

2. EXPERIMENTAL

In this work alumina and RGO are taken as the starting material. The three different compositions of Al_2O_3-RGO by varying wt% of RGO i.e. 1, 2, 3% were ball milled by a horizontal planetary dry ball mill. The planetary ball mill was so constructed to generate both impact and shear forces to the samples. The planetary ball mill works to overcome the limitation of the gravitational field and continuously supplying strong acceleration field. The planetary ball mill consists of a gyratory shaft (580 mm) and two cylindrical steel jars of diameter 200 mm each, both are rotated simultaneously and separately at a speed of 345 rpm and 160 rpm respectively. The rotation of both jars and the shaft makes the balls (inside the chamber) to move very strongly and violently, leading to large impact energy of balls that improves the grinding kinetics and results ultra fine composites with reduced crystallinity. The sample to ball charge ratio of 1:7 was maintained in the planetary ball mill. The ball milling of the samples was carried out for 16 hr. The wt % of RGO in Al_2O_3 was varied as 1, 2 and 3%. After ball milled over, the samples were taken for sintering at 1500 ^{0} for time period of 6 hr.

XRD patterns of the samples were studied by PANalytical X'Pert Pro diffractometer equipped with CoKα radiation. The patterns were taken in the 2θ range 5–95 at a scan speed of 0.019 min^{-1}. The microstructures of samples were observed by FESEM (model: ZEISS SUPRA 55). Nanoindentation of the samples was determined by Nanoindenter—UMIS system (Fisher-Cripps, Australia) with diamond Berkovich indenter (tip diameter: 400 nm) at a maximum applied load of 50 mN,

3. RESULTS AND DISCUSSION

The X-ray diffraction was carried out to understand crystalline nature, phase purity and interlayer spacing of typical Al_2O_3-RGO (3 wt%) composite sample. The result was carried out and presented in Fig. 1. The composites having phases and planes are identified by comparing the observed *d*-values (experimentally determined) with the *d*-values of standard powder diffraction data file, C (graphite-2H): 00-041-1487 and Al_2O_3: 01-075-1862 supplied in JCPDS-ICDD PDF-2 (2004). The XRD results show patterns in 2θ range of 5^0-80^0. The composite nature of the sample is confirmed from the XRD analysis. The XRD analysis shows the peaks due to C and Al_2O_3. Al_2O_3 phase shows diffraction peaks of (104), (110), (113), (024), (116), (122) and (214). Al_2O_3 shows two major peaks at around 2θ = 41.0^0 and 2θ= 51.0^0. Similar kind of result was observed by literature [11]. A characteristic peak of C (002) is strongly appeared at 2θ = 30.0^0 is attributed due to the presence of RGO in the composite. The sharpness and FWHM of the peaks of the different phases were compared to their standard values which indicate the well ordered and better crystalline nature of the composite.

FESEM analysis of the typical composites of Al_2O_3-RGO with different content of RGO 2 wt% and 3 wt% are presented in Fig. 2 and 3 respectively. Uniform distribution of RGO within the Al_2O_3 grains is very important to prepare superior quality composite for various advanced applications. In Al_2O_3 with RGO 2 wt% shows irregular distribution graphene or RGO grains. Whereas as almost uniform distribution of graphene grains have been observed for $A_{l2}O_3$ with RGO of 3 wt%. The composites were also found to free from micro porosity and any kind of surface defects.

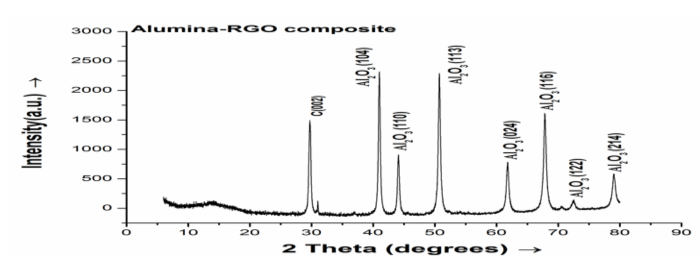

Fig. 1. XRD pattern observed for typical Al_2O_3-RGO (3 wt%) composite sample

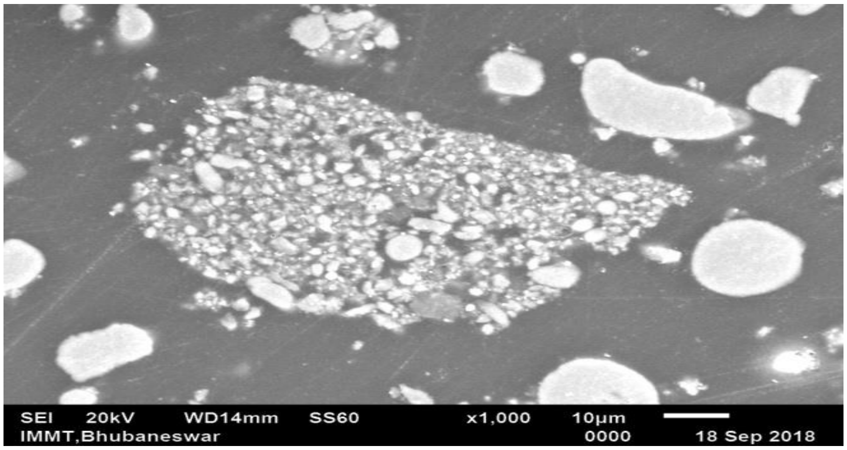

Fig. 2. FESEM micrograph of Al_2O_3-RGO (2 wt%) composite sample

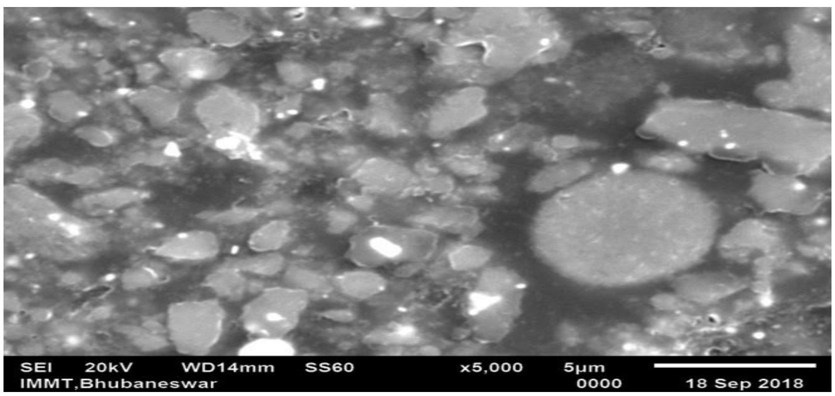

Fig. 3. FESEM micrograph of Al_2O_3-RGO (3 wt%) composite sample

Microhardness of Al_2O_3 and Al_2O_3-RGO composites with different compositions of RGO (1, 2, 3 wt%) determined by nanoindentation method. It is found from Fig. 4 that the microhardness of only Al_2O_3 phase is 1480 ± 10 VHN. Before graphene/RGO is reinforced to Al_2O_3 phase, it was thought that it may increase the mechanical property of the composites. This is interestingly matching to our expectation that the value of microhardness increases with increase in RGO content. In this work the value of microhardness of Al_2O_3-RGO (3 wt%) is found to be higher i.e. 2008 ± 18 VHN. The Young's modulus of the composites were determined and compared with Al_2O_3. It was found that while only Al_2O_3 shows Young's modulus of 395 ± 43 GPa, whereas Al_2O_3-RGO composite with 3% RGO shows 520 ± 8 GPa. The enhanced mechanical properties of composites may be attributed to presence of graphene/RGO in Al_2O_3. Porosity free nature of the composite along with optimized sintering has some contribution for the development of mechanical property.

The above improved microhardness and Young's modulus of 26 % and 22 % respectively inspires to use the composite for advanced structural applications in aerospace and automotive industries, etc.

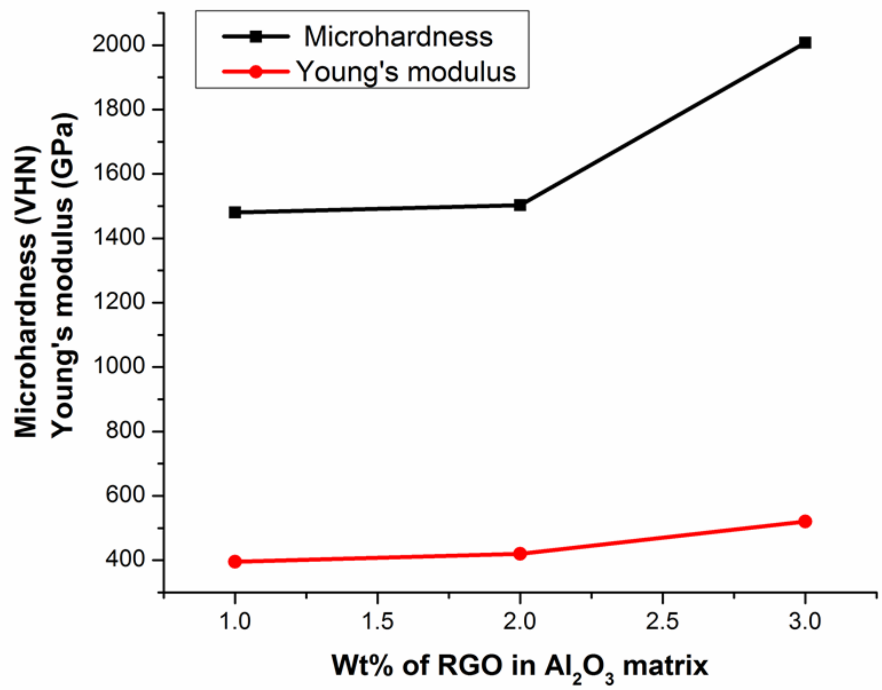

Fig. 4. Graph showing plot of microhardness and Young's Modulus vs. RGO content in Al_2O_3-RGO composites

4. CONCLUSION

In this work, it has been possible to prepare three different compositions of Al_2O_3-RGO by varying wt% of RGO from 1-3 wt%. All Al_2O_3-RGO composites were ball milled by using a dry planetary ball milling technique for 16 hr. After ball milling, the samples were sintered at 1500 ^{0}C for 6 hr. XRD analysis of the composites show the peaks of Al_2O_3 and C (graphene/RGO). The composite nature further confirmed from FESEM analysis. The composites are found to micro free from porosity and any kind of surface defects. The uniformity of distribution of graphene/RGO is found to increase when the wt% of RGO is increased from 1 wt% to 3 wt%. In this work, it is evident that the maicrohardness and Young's modulus of Al_2O_3-RGO composites were found to increase when RGO content is increased from 1 to 3 wt%. The composite Al_2O_3-RGO with 3 wt% RGO has microhardness and Young's modulus of 2008 ± 18 VHN and 520 ± 8 GPa respectively. Such values of microhardness with Young's modulus indicate reduced brittleness of the composites. Overall attempt has been taken in the work to correlate RGO content with various microstructural properties and hardness. The composite with improved mechanical property has great potential scope for various advanced ceramic and structural applications.

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