Graphene derivatives Influence on mechanical properties and microstructure of alumina based ceramic materials

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Abstract. In this study obtaining methods of matrix ceramic composites with different compositions by high temperature pressing technology have been discussed. Following ceramic composites have been obtained α-Al_2O_3, α-Al_2O_3-GO, α-Al_2O_3-RGO. Ceramic composites are obtained in high temperature vacuum furnace with different temperature and pressure conditions. Received ceramics do not have open pores and their density reaches 99.5 % of TD.

Introduction. Ceramic materials are widely used in many fields of science because they have excellent chemical, mechanical and thermal properties. Alumina (Al_2O_3) is remarkable example of ceramic materials. Due to its outstanding characteristics, alumina is used in autoindustry, aerospace and biomedical activities also as cutting materials. Alumina in its various levels of purity is used more often than any other ceramic materials. Alumina-based ceramic materials are the cheapest, because aliminum is one of most abundant elements on Earth and the methods of its production are well developed. Nevertheless, alumina-based ceramics have some disadvantages, such as low resilience and fracture toughness [1-3]. Improvement of the physical-mechanical properties of ceramics to a certain degree became possible by reducing the size of particles of sintered powders to nano sizes, using different dopants and reduction of sintering time, because during consolidation process the grain sizes dramatically increase, which negatively affects to final product properties. Recently special attention is paid to reinforced ceramic composites with carbonaceous materials. Considering the unique physical-mechanical properties of carbon nanostructures, they are widely used as reinforcement for alumina ceramic composites [4].

The aim of this work is to prepare and to study the mechanical properties and microstructure of alumina matrix composite material reinforced by (1.5 wt. %) graphene oxide (GO) and reduced graphene oxide (RGO) using hot pressing method.

Experimental procedure and sample preparation

Synthesis of graphene oxide and reduced graphene oxide. Synthesis of graphene oxide have conducted by intercalation method from graphite. Graphite flakes (2 g) mixed in 50 mL of H_2SO_4 (98%) and potassium permanganate (6 g) very slowly during 1 h. The flask kept under at ice bath (27-35°C) with continuous stirring. After 1 h 100 ml water was added in the mixture. Then continue stirring again about 1 h and 20 mL H_2O_2 was added. After washing and filtration, the mixture centrifugation has done. Stable graphene oxide suspensions have obtained which used as reinforcement materials in ceramic composite.

Synthesis of reduced graphene oxide. A weighed amount of GO and distilled water were placed in ultrasonic bath. The mixture was held for 3 h. The resultant black substance was then heated in a commercial microwave oven for 2 minutes at 800 watts. The solution was then filtered and washed with ethanol. The obtained residue was dried using an oven at 110°C for 6 hours. The obtained rGO solid were then characterized.

Preparation of pressing powdery composite. 65 g α-Al_2O_3 was mixed to graphene oxide (1.5% mas.) and in the other hand to reduced graphene oxide. 100 mL H_2O was added into mixture and homogenization carried out by nanomill during 24 h. Then the mixture dried and placed in press form.

SEM. Structural-morphologic investigation of ceramic composites has been performed by JEOL JSM-6510LV and Nikon ECLIPSE LV 150 microscope. Chemical content of these samples has been measured simultaneously with energy dispersive micro XRD analyzer.

Determination of microhardness. Microhardness and modulus of Al_2O_3 have been studied according to ISO-14577 international standard at dynamic ultra microhardness tester DUH-211S.

Results

Alumina is material which widely used for obtain of matrix ceramic composites. High temperature vacuum furnace (OXY-GON) has been used for obtaining of them [5-6]. Alumina based ceramic composites have obtained in the OXY-GON furnace: α-Al_2O_3, α-Al_2O_3-GO, α-Al_2O_3-RGO. Sintering temperature–1400-1600^{o}C, sintering time was 20-60 min at maximum temperature. Refrigeration of graphite pressure-shape has been in inert atmosphere. Obtained corundum product has black color, because there have been thermal dissociation and formation defect lattice of Al_2O_3 in vacuum. For purpose, relaxation (whiten) of product with defect structure have been annealing in high temperature furnace at air (1600˚C, 1 h). Physical-mechanical and structural-morphological study has been conducted for obtained materials.

As an electronic micrographs (Fig. 1) showed, average number of GO or RGO layers are 10-30 nm and graphene oxide particles sizes are approximate 50-70 nm in suspension.

As above mentioned, Obtained ceramic samples with various thickness and cylindrical diameter forms (Fig. 2) their microhardness was established at dynamic and static conditions. Microhardness is relatively high at small depth (load) and its value falls when depth (load) increases and goes at stationary value. Reason may be is obtaining condition of ceramics. Also some physical-mechanical parameters of obtained composite materials are given in table 1.

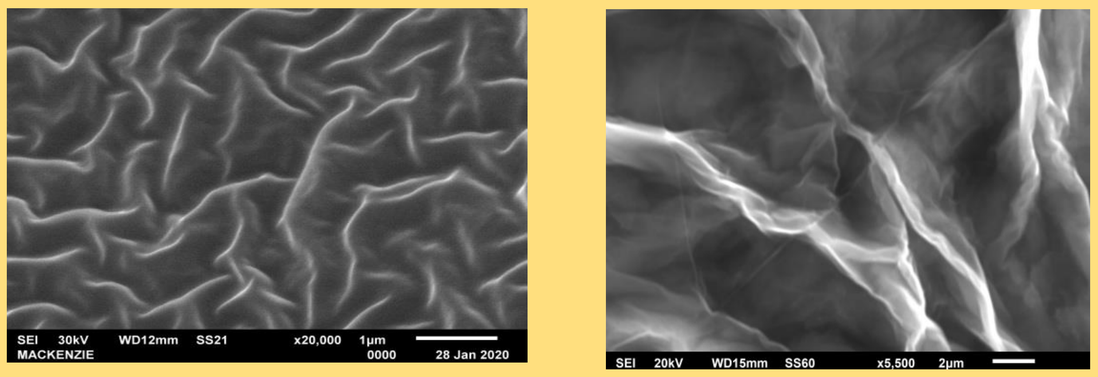

Fig. 1. SEM of GO and RGO

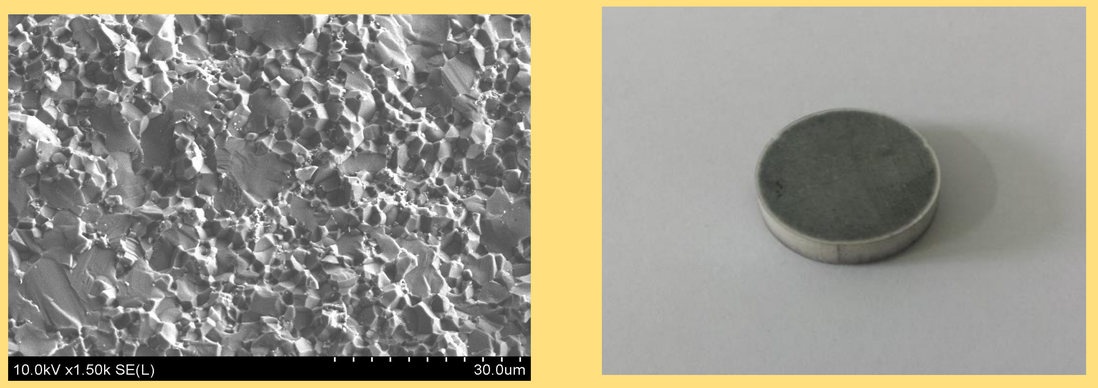

Fig. 2. SEM of alumina based ceramic composite

Table 1. Physical-mechanical properties of composite ceramics which is reinforced with graphene oxide and reduced graphene oxide

Sample	density g/cm^3	Open porosity , %	water absorption %	Microhardness , GPa (200 g)	σ-bending strength GPa
α-Al_2O_3	3,94-3,96	0,08-0,11	0,03-0,05	12	300
α-Al_2O_3-GO (1.5% mas.)	3,98-4,00	0,02-0,04	0,03-0,05	16.9	385-410
α-Al_2O_3- RGO (1.5% mas.)	3,95-4,00	0,01-0,03	0,03-0,05	18,4	380-400

Conclusions

In this works, obtaining ceramic matrix composites was investigated, which is based on consolidation of powdery composites in high temperature vacuum furnace OXY-GON. Following pressing powdery composites α-Al_2O_3, α-Al_2O_3-GO, α-Al_2O_3-RGO have been obtained by ball milling. Ceramic products were obtained by sintering at 1400-1600^{o}C (1 hr, 50 MPa), which is characterized by high flexural strength (380-450 MPa), microhardness and fracture toughness and without of open porosity. Their relative density achieves 99.5% of the TD.

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