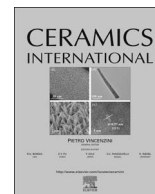




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Mechanical properties of graphene oxide reinforced alumina matrix composites

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ABSTRACT

Graphene oxide (GO) reinforced alumina matrix composites have been fabricated by using graphene oxide synthesized by a modified Hummer's method. Samples were prepared by powder metallurgy and consolidated by Spark Plasma Sintering (SPS). The influence of GO addition on the microstructure and mechanical properties of the composites was investigated. Results show a significant increase (almost 35%) of the fracture toughness for composites containing 0.5 wt% graphene oxide compared to sintered pure alumina. In order to find reasons for this improvement Scanning/Transmission Electron Microscopy (SEM/TEM) observations were carried out. They reveal a good interface between the reinforcement and the matrix as well as such mechanisms like branching, deflection and bridging of crack propagation.

1. Introduction

Advanced ceramic materials have attractive properties like high mechanical strength due to their hardness and elastic modulus, good chemical stability, high stiffness and stability at high temperatures. These attributes are superior to polymers or metals, allowing monolithic ceramics to be used in such applications as power generation, aerospace, military, transportation, biomedical sciences and electronics [1,2]. However, ceramic is brittle due to the ionic and covalent bonds. As such, low fracture toughness is its biggest disadvantage, which often limits the use of ceramic in structural applications. To overcome this restriction, most of the last decades' purpose was to improve ceramic toughness, either by the tailoring of the microstructure in micro/nanocomposites [3], or the addition of reinforcement [4], or both at the same time. Each route shows similar results, creating new intrinsic grain boundaries or extrinsic interfaces, thus making a longer path for crack propagation [5–7]. This caused the need to search for new materials able to fulfil the role of second phase in ceramic composites.

Since its rise [8], there is still an ongoing interest in graphene (Gn), due to its unique combination of thermal [9], electrical [10] and mechanical properties [11]. Additionally, its other characteristics like large specific surface area [12], small tendency to tangle and two-dimensional geometry seems to make it an ideal candidate for nanofiller in ceramic matrix composites [13]. This goal can also be obtained by graphene derivatives, e.g. graphene oxide; interest in

which has been increasing exponentially just a few years after the original emergence of graphene [14]. GO itself is the oxidized form of graphene, where graphene sheets have their sp^2 C basal plane decorated mostly with functional oxygen groups [15,16]. Those ceramic matrix composites containing graphitic materials have a wide potential application in such fields as lithium ion batteries [17], electrochemical capacitors [18], bio-sensors [19], or cutting tools [20].

Over the last few years numerous authors report a significant increase of fracture toughness with the incorporation of Gn/GO to an alumina matrix. In the work of Porwal et al. [21], graphene was prepared using liquid phase exfoliation and composites with up to 5 vol % of its content were consolidated using Spark Plasma Sintering. With the change of crack propagation mechanism from inter-granular to trans-granular, a 40% increase of fracture toughness (K_{IC}) was measured for composites with only 0.8 vol% of Gn. Similar results were reported by Liu et al. [22] where a 30% increase in both flexural strength and fracture toughness for samples with 0.78 vol% of Gn was observed. This specific work was related to crack deflection and crack bridging by the graphene platelets. Unlike previous authors, Wang et al. [23] used Gn instead of GO dispersion which was added to alumina/water and the mixture was reduced by hydrazine monohydrate and the final mixture was consolidated using the SPS technique. With the incorporation of 2 wt% of GO to the alumina matrix composite, the K_{IC} increased by 53% when compared to pure alumina. Slightly different results were achieved by Centeno et al. [24] where

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nearly a 50% enhancement of the mechanical properties was obtained for composites with 0.16 and 0.22 wt% Gn content. In this case, crack bridging was found to be the main mechanism. It was also observed that further increases to the content of Gn led to a decrease of the fracture strength. Similar measurements were done by Kim et al. [25] who reported a 75% increase of fracture toughness, though only for small amounts of Gn, between 0.25 and 0.5 vol%. As in the work of previous authors, further increases of reinforcing phase caused a decrease of K_{IC} . The expected reason for this was the creation of agglomerates and this was confirmed by the work of Kostecki et al. [26], who proposed a mathematical model for grain boundary coverage based on graphene platelet thickness and volume content which was subsequently confirmed by real composites measurements. It was proven that exceeding 1 vol% of Gn causes flakes agglomeration, forming additional porosity and reducing hardness as well as Young's modulus.

Even though there is so much well documented research done on Gn/GO-alumina matrix composites, there are still many obstacles in their fabrication such as obtaining high quality graphene/graphene oxide, degradation of reinforcement during sintering and its homogeneous dispersion in ceramic matrix. One interesting issue was also reported by Wozniak et al. [27] and related with the composite interface, where bonding between reinforcement-matrix can be too strong or too weak, consequently decreasing fracture toughness. An absolute lack of bonding is also possible, which results in the existence of voids. As a solution, Gn coated with a nickel-phosphorus layer was proposed.

In the presented work, the difficulties mentioned above were overcome with the use of specific processes. The Modified Hummer's method [28], was used to obtain high quality graphene oxide, which depends on excluding NaNO_3 from the process compared to the original path [29]. Instead, an increased amount of KMnO_4 is used and a reaction is performed in a mixture of $\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$. This modification results in eliminating the evolution of $\text{NO}_2/\text{N}_2\text{O}_4$ toxic gases and makes the process temperature easier to control. Incorporating synthesized GO in the alumina matrix also ensures a better interface than Gn due to the functional groups and dangling atoms so characteristic of graphene oxide. In addition, a powder processing route [30] was applied to guarantee a good dispersion of reinforcement in a alumina matrix. The SPS technique was used to carry out the final consolidation, allowing the process to complete faster in lower temperatures compared to traditional methods [31].

In this investigation synthesized graphene oxide was characterized to confirm its nature, followed by the production of alumina matrix composites reinforced with different weight contents of GO. Microstructures of the obtained samples were studied and correlated with measured mechanical properties. All results were compared to this with pure alumina as a reference specimen.

2. Materials and methods

2.1. Substrates

The components used in the presented work were commercial $\alpha\text{-Al}_2\text{O}_3$ powder (Taimei Chemicals CO., LTD.) with a chemical purity of 99.99% and the average particle size of 135 nm, and GO prepared by a modified Hummer's method using commercial graphite flakes (Graphene Supermarket) with a chemical purity of 99.9% and 150 μm or more in lateral size.

2.2. Graphene oxide synthesis

Graphite flakes (5g) were added into 110 mL of 98% solution of H_2SO_4 (Sigma-Aldrich) and 11.5 mL of 85% solution of H_3PO_4 (Sigma-Aldrich). The mixture was then mechanically stirred for 1 h at a speed of 180 rpm. Next, the solution was cooled by transferring it into a

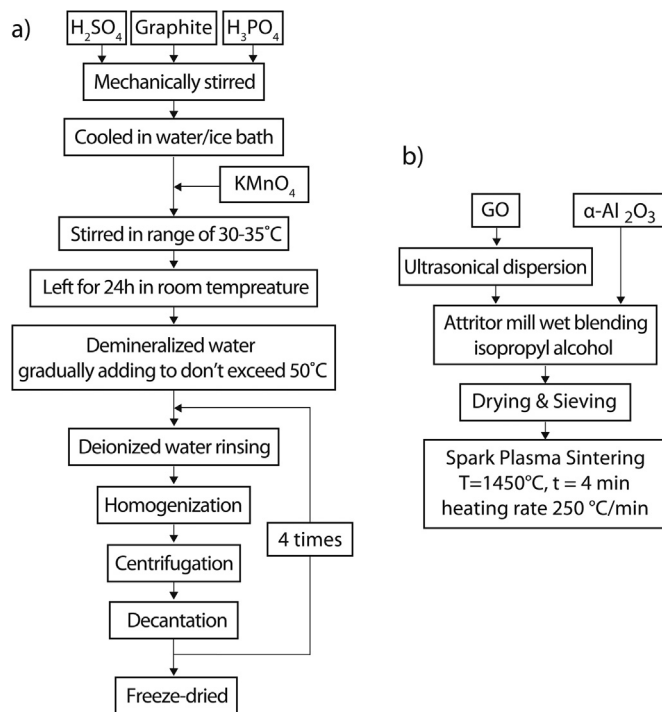


Fig. 1. Technological steps for GO synthesis using modified Hummer's method (a) and producing Al_2O_3 -GO composites (b).

water/ice bath where its temperature was kept below 5 °C, and 15g of KMnO_4 (Sigma-Aldrich) was then gradually added. The obtained mixture was taken out of the ice bath and kept in range of 30–35 °C whilst being stirred for 1 h at a speed of 250 rpm. Following that, the solution was left at room temperature for 24 h without any further stirring. After that time, 400 mL of demineralized water was added to the stirred mixture to prevent its temperature from exceeding 50 °C. The obtained solution was then used in a cycle consisting of: rinsing in 3 L of deionized water, ultrasound homogenization (VCX 750 W, Sonics & Materials Inc.), centrifugation (Rotina 420, Hettich Zentrifugen) with a speed of 4500 rpm for 20 min, and finally decantation. The entire series was repeated 4 times in total. The resulting suspension of concentrated graphene oxide flakes was freeze-dried using a lyophilizer (Epsilon 2-6D LSCplus, Christ) over 26 h at a temperature of -26.3 °C and a vacuum of 0.33 mbar. The simplified production scheme for GO using this method is presented at Fig. 1a.

2.3. Al_2O_3 -GO powders preparation and sintering

Composites Al_2O_3 - x GO ($x=0.2, 0.5, 0.7, 1, 1.5, 2$ wt%) were fabricated by powder metallurgy. Firstly, graphene oxide was ultrasonically dispersed in isopropyl alcohol (Sigma-Aldrich) for 30 min in periodical working mode (1 s work / 2 s brake) with power of 300 W. Following that, GO solution was wet blended with Al_2O_3 with the use of an attritor type mill in isopropyl alcohol for 8 h. Alumina grinding media were used to exclude other admixtures from the attrition process. After drying, the obtained powder mixtures were sieved ($\phi=0.5$ mm) to ensure even better homogenization. The Spark Plasma Sintering method was used to carry out final consolidation. In order to achieve composites with high mechanical properties, optimisation of sintering conditions for pure Al_2O_3 and Al_2O_3 -GO composites was carried out, with the following variables: sintering temperature, dwell time and heating rate. The influence of changes to those parameters on the mechanical properties were analyzed and optimal conditions were chosen. The parameters of the sintering process presented in this work were as follows: temperature 1450 °C, heating rate 250 °C/min, dwell time 4 min, applied pressure 35 MPa and vacuum atmosphere. In addition, two unreinforced alumina samples were sintered as reference specimens: one

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