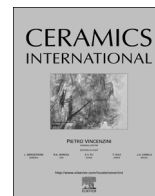




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## Preparation and mechanical properties of alumina composites reinforced with nickel-coated graphene

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### ARTICLE INFO

#### Article history:

Received 14 December 2015

Received in revised form

26 January 2016

Accepted 15 February 2016

#### Keywords:

A. Sintering

B. Composites

C. Mechanical properties

D. Al<sub>2</sub>O<sub>3</sub>

### ABSTRACT

This paper discusses the influence of nickel-phosphorus coated graphene (Gn–Ni–P) and uncoated graphene (Gn) addition to an alumina matrix and its impact on the mechanical properties of obtained composites. The composites are prepared via powder processing and consolidated using the Spark Plasma Sintering (SPS) method. The effects of the addition of coated graphene and coating thickness on mechanical properties were evaluated. Physical properties such as relative density, hardness and fracture toughness were analyzed. Significant improvement of the fracture toughness (60%) for the composites with 2 vol% Gn–Ni–P compared to reference sample was observed. Moreover, 35% higher  $K_{IC}$  was noticed for Gn–Ni–P reinforced composites than for Al<sub>2</sub>O<sub>3</sub>–Gn.

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### 1. Introduction

In the last few years, graphene has attracted great attention owing to its combination of mechanical, thermal and electrical properties [1,2]. Only small amount of graphene (Gn) should simultaneously improve mechanical, electrical and thermal properties of ceramic matrix composites [3–5]. Such composites can be applied in many fields including bio-sensors, transparent conductors and cutting tools [6–8].

Despite many advantages of graphene, preparation of composites reinforced with Gn still causes many problems, such as: production of high quality graphene, homogeneous dispersion of Gn in a ceramic matrix and degradation of graphene during sintering [9]. Those problems are solved by the selection of proper Gn synthesis methods (high energy milling [10] or Hummer's method [11]), application of powder processing [12] or colloidal processing [13] routes to disperse graphene in a ceramic matrix and using the sintering method to allow a decrease in temperature and time of consolidation process (Spark Plasma Sintering – SPS) [14]. Apart from the problems mentioned above the composites properties (not only reinforced with graphene) are critically influenced by the matrix-reinforcement bonding. Crack propagation in Gn-reinforced composites is similar to fiber-reinforced ceramic composites and may take place by three paths, i.e. singly deflected crack,

doubly deflected crack, and penetrating crack across the reinforcing platelets [15]. Crack is initiated and starts to propagate in the ceramic matrix and then the load is transferred to the fibers/graphene. If the interface is too strong, crack penetrates through the fiber and the composite becomes brittle. On the other hand, if the interface is weak enough, crack can be deflected along the interface, the fiber/graphene stays intact and the composite remains tough [16]. Additionally, there can be a lack of bonding between graphene and the matrix. In this case, composites have voids with graphene placed loosely inside. It will act as a stress concentrator and reduce fracture toughness of the composite. This situation may occur particularly in the manufacture of ceramic composites reinforced with multilayer graphene (MGn). MGn, when perpendicular to the carbon layer direction, indicates a much higher thermal expansion coefficient (CTE) compared to ceramics i.e. Al<sub>2</sub>O<sub>3</sub>. The big difference in CTE is that after sintering, the composites exhibit porosity, which significantly decrease the strength properties. Therefore, to minimize porosity of composites reinforced with MGn, the MGn surface can be modified. One idea is electroless coating of MGn with a nickel layer. It should improve bonding between multilayer graphene and the ceramic matrix and significantly increase the mechanical properties of composites. Such solution was successfully applied in the case of metal matrix composites reinforced with a carbon nanotube [17] as well as in Al<sub>2</sub>O<sub>3</sub>–Gn composites [18].

In this paper, nickel-phosphorus coated MGn reinforced alumina matrix composites were fabricated. Moreover, the three different thicknesses of Ni–P layers were compared and their

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influence on mechanical properties and microstructure of SPS sintered alumina matrix composites were analyzed.

## 2. Experiment

In the present study  $\text{Al}_2\text{O}_3$ -Gn/Gn-Ni-P composites were fabricated via powder metallurgy technique. The following substrates were used: commercial  $\alpha$ - $\text{Al}_2\text{O}_3$  powder (Taimei Chemicals CO., LTD., 99.99% chemical purity, 0.135  $\mu\text{m}$  average particle size) and commercial multilayer MGn powder (Graphene Supermarket, 99.2% chemical purity, 4.5  $\mu\text{m}$  average flakes size, 12 nm average flakes thickness). In the first technological step, the Gn powders were coated by a nickel-phosphorus layer. The coating of nickel-phosphorus alloy layers was carried out by a chemical reduction method [19]. The process was executed at elevated temperature (85–90 °C) in an electrolyte solution containing nickel salts, stabilizing compounds and a reducing agent. The process of coating was followed by autocatalytic chemical reduction reaction on the surface of graphene flakes. In order to ensure uniform coating thickness the mechanical stirring was applied. Detailed information on the coating process is given elsewhere [20]. In order to determine the effect of coating thickness on the properties of composites, three sets of MGn powders with different coating times were prepared: 130 s marked as S-I, 90 s marked as S-II and 60 s marked as S-III. Furthermore, to determine the effect of graphene coating on the mechanical properties, an additional set (S-0) of composites with uncoated graphene was prepared. At the next stage the powders were wet-blended at planetary type mill for 24 h in isopropyl alcohol suspension. Final consolidation of powder mixtures was carried out with the use of SPS (Spark Plasma Sintering) method:  $T=1450$  °C,  $t=4$  min,  $V_t=250$  °C/min,  $p=35$  MPa, vacuum. Produced alumina ceramic matrix composites were characterized with the following volume content of graphene:  $\text{Al}_2\text{O}_3-x$  Gn/Gn-Ni-P (where  $x=1; 2; 3$  vol% of MGn). The volume content of graphene for individual samples was the same regardless of the thickness of the coating of Ni-P. Moreover, pure alumina sample was sintered as a reference specimen.

Density of specimens were examined with the use of a helium pycnometer (Ultrapycnometer 1000). Vickers hardness and fracture toughness were measured with a Vickers Hardness Tester (FV-700e.) with the use of the indentation method under the load of 49 N. All tests of mechanical properties were made on six samples. The microstructure observation was performed on an scanning electron microscope (SEM Hitachi 5500). Raman analysis was made using a Nicolet Almega XR-Dispersive Raman spectrometer (laser with an excitation wavelength of 532 nm) and for each sample at least 3 scans were performed.

## 3. Results

The morphology and the Raman spectra of uncoated graphene are presented in Fig. 1a and b respectively. The Raman spectrum is typical for multilayer graphene which suggests the 2D band shape. The spectra indicated a high quality  $\text{sp}^2$  carbon network with  $I_D/I_G$  ratio of 0.47. Comparing of  $I_D/I_G$  ratio with flakes size it can be said that D-band can be associated with plate edge effects rather than in-plane defects [21].

As mentioned above, to determine the effect of coating thickness on the properties of composites, three series of Gn-Ni-P powders with different coating times were prepared. Morphology of the powders are shown in Fig. 2a, b and c. When comparing powders after coating with uncoated graphene, the changes in morphology can be observed. The powder particles still indicate the shape of flakes but the thickness of flakes is greater. In order to

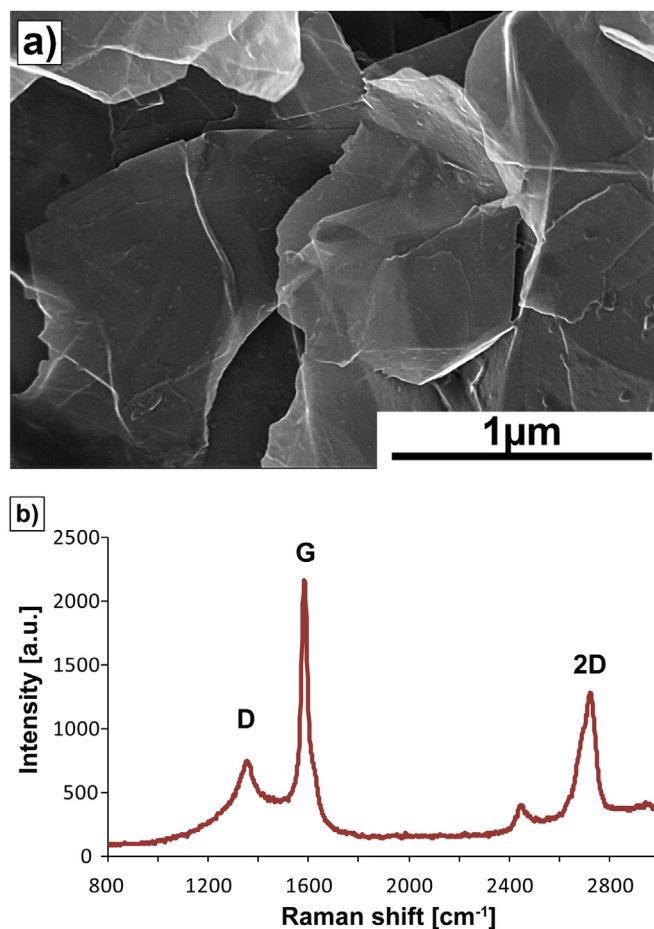


Fig. 1. Morphology (a) and the Raman spectra (b) of graphene flakes before coating.

precisely determine the thickness of Ni-P coatings the cross-sections through the coated graphene particles was executed. Exemplary image of Gn-Ni-P cross-section (S-III) is presented in Fig. 3a.

Graphene is visible on the cross-section of the S-III powder (dark areas) and lighter layer. It should be noted that the thickness of graphene is relatively high, which may indicate an agglomeration process. It seems necessary to better disperse graphene in an electrolyte. Analysis of the chemical composition made on the powder cross section (Fig. 3b) shows the presence of nickel and phosphorus, which confirm that the intended coating composition was achieved. The presence of silicon in the spectrum may be associated with residues of material after the sample preparation. Based on the Gn-Ni-P powder microstructures, the coating thickness measurements were executed. The average value of thickness of S-I, S-II and S-III coatings was 370, 220 and 170 nm respectively. Moreover, it should be noted that obtained coatings are characterized by relatively uniform thickness. The change in the average thickness correlates well with the time of coating. The highest layer thickness was achieved for the longest coating time, this indicates that the process can be easily controlled by changing coating time. Furthermore, to determine the effect of coating process on graphene, the Raman analysis was performed on flakes cross sections (Fig. 3c). The Raman spectra is almost identical as for uncoated flake which means that Gn structure was not changed during coating process.

Fig. 4 shows fracture surface of composite reinforced with 2 vol% of Gn-Ni-P (S-I). Bright particles on the microstructure are multilayer graphene particles coated by Ni-P film which confirm

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