CARBON 90 (2015) 274–283

Available at www.sciencedirect.com

ScienceDirect

journal homepage: www.elsevier.com/locate/carbon

Highly strain tolerant and tough ceramic composite by incorporation of graphene

Yuchi Fan [a,*], Ginga Igarashi [a], Wan Jiang [b], Lianjun Wang [b], Akira Kawasaki [a,*]

[a] Department of Materials Processing, Graduate School of Engineering, Tohoku University, Sendai 980-8579, Japan

[b] State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, Donghua University, Shanghai 201620, PR China

ARTICLE INFO

Article history:
Received 8 February 2015
Accepted 14 April 2015
Available online 20 April 2015

ABSTRACT

Graphene is an ultra-thin, remarkably flexible and highly stiff 2D material that can profoundly change the microstructure of composite as filler phase, giving rise to mechanical properties greatly different from traditional composites. However, there are very few examples that demonstrate the exceptional properties in graphene based ceramic composite because of the tradeoff between small thickness of graphene platelet and dispersion uniformity in processing. Here, a fully dense Al_2O_3 composite with uniformly dispersed few-layer graphene (FLG) is prepared by heteroaggregation technique and spark plasma sintering. It is found that in comparison to monolithic Al_2O_3, drastically reduced Young's modulus (298 GPa), completely retained fracture strength (417 MPa) and enhanced fracture toughness (5.3 MPa m$^{1/2}$) are simultaneously realized in this composite, leading to an unprecedented increase of strain tolerance by ~40% at merely 2.18 vol.% of filler loading. It is believed that the unique highly wrinkled structure of FLG at triple junctions of ceramic matrix causes the inefficient load transfer before crack initiation and thus low stiffness in composite. Whereas after crack initiation, by the "stretched filler bridging" of FLG platelet behind crack tip, the toughness of composite is enhanced so that the high fracture strength can be retained.

© 2015 Elsevier Ltd. All rights reserved.

1. Introduction

Since the discovery of graphene, the effort of incorporating the fantastic 2D material with other material to achieve advanced performance has never ceased as encouraging results have been found successively. While tremendous works are concentrated on the transport properties of graphene based composite [1,2], the great potential for improving mechanical performance such as strength and Young's modulus of composite by additive of graphene has intrigued plenty of interest, though mainly in polymer matrix composites due to the apparently higher intrinsic modulus and ultimate strength of graphene in comparison to polymer [3].

For ceramic matrix composite (CMC), attention has intensively devoted to toughening effect of graphene so far [4–15], since brittleness is widely considered as the Achilles heel of ceramics. However, a fundamental but often ignored problem is that in most of the so-called graphene containing CMCs the fillers are mainly composed of thin graphite platelets (from ~10 to ~500 nm) rather than graphene. For instance, if a platelet on fracture surface can be identified by scanning electron microscopy, generally the thickness should be around 50 nm

* Corresponding authors.
E-mail addresses: fanyuchi@hotmail.com (Y. Fan), Kawasaki@material.tohoku.ac.jp (A. Kawasaki).
http://dx.doi.org/10.1016/j.carbon.2015.04.029
0008-6223/© 2015 Elsevier Ltd. All rights reserved.

[4,5,11,16–19]. Graphite platelet with this thickness represents more than 150 layers of graphene, whose discrepancy of property with respect to graphite is too ambiguous to clarify. It has to be emphasized that this is not only a problem of terminology, since albeit graphene and graphite possess identical Young's modulus and ultimate tensile strength in theory, graphene outperforms graphite in many aspects such as flexibility, aspect ratio and specific surface area, which deteriorate with increasing thickness and could deeply affect the mechanical performance of CMC. This situation in the community of graphene reinforced CMC reflects the great challenge in uniform dispersion of graphene in ceramic matrix. Note that even if single-layer graphene has been confirmed as starting material, agglomeration and restack can lead to severe rising of average thickness and alter the property of composite consequently [14].

In this study, fully dense few-layer graphene (FLG) reinforced Al_2O_3 composites with graphene content of 2.18 vol.% were prepared. The FLG is ultra-thin, remarkably flexible and homogeneously dispersed in matrix, giving rise to very unique 2D constructed 3D network in composite where FLG platelets appear to be highly wrinkled at triple junctions. Evaluation of the FLG/Al_2O_3 composite shows completely maintained fracture strength and drastically decreased Young's modulus by 25.7%, which represents an enhancement of strain tolerance by 39.2%. It is found that the load transfer in composite is very weak and the FLG platelet behaves like 2D pore in composite, owing to the special microstructure of FLG. Moreover, the enhanced fracture toughness is proved to be responsible for the reliability at large strain and the toughening mechanism is also discussed based on observation by transmission electron microscopy (TEM).

2. Experimental

2.1. Preparation of graphene oxide (GO)/Al_2O_3 hybrid powder

GO was prepared by the modified hummers method reported elsewhere [20]. The commercially available α-Al_2O_3 powder (TM-DAR, Taimei Chemicals Co. Ltd., Tokyo, Japan), with a purity of 99.99% and an average particle size of 0.1 μm was used in this study. As-received α-Al_2O_3 (2.5 g) was directly poured into a beaker without any treatment. Then water (500 mL) was added and the pH was adjusted to around 3.33 using HCl solution (1 M). After 1 h sonication, an Al_2O_3 colloid was formed. 100 mL of GO colloid (as well as 150 mL and 200 mL for elastic modulus testing) was added to the Al_2O_3 colloid by titration while stirring. The mixture was separated by vacuum filtration followed by drying at 80 °C.

2.2. Sintering of FLG/Al_2O_3 composites, fully dense and porous monolithic Al_2O_3 ceramics

The bulk composites were prepared by using Spark Plasma Sintering (SPS) apparatus (Dr. Sinter 511-S, Sumitomo Coal Mining Co. Ltd., Japan). GO/Al_2O_3 hybrid powder was loaded into a 25 × 13 mm graphite die. Boron nitride was sprayed between the die and powder for easy removal. The powders were sintered in vacuum (residual cell pressure <6 Pa). The soaking time was 10 min and the heating rate was 100 K min^{-1}. The pressure was applied in two steps: the initial pressure of 32 MPa was applied, and consequently the pressure was increased to 80 MPa from 1000 °C upwards and maintained during the dwelling time at 1250 °C. Fully dense Al_2O_3 ceramics were sintered by using pure Al_2O_3 powder at the identical conditions of preparing composites, while porous Al_2O_3 ceramics were fabricated by sintering at initial pressure and lower holding temperature.

2.3. Preparation of burn-off sample

FLG was burned off from composite with filler fraction of 2.18 vol.% at 700 °C in air for different times. The density of sample was measured after each period of burn-off to determine the residual filler fraction. After 2.5, 5 and 10 h of burn-off the sample was taken out for measurement of Young's modulus without breaking the sample. After 20 h of burn-off, the sample was measured until failed to obtain fracture strength as well.

2.4. Characterization

The apparent density of the nanocomposites was measured by Archimedes' principle. The carbon content in composites was measured by combustion infrared detection technique (844 series, Leco, MI, US) and the filler fractions in composites were calculated accordingly. The morphology and microstructure of samples were characterized by TEM and high-resolution TEM (JEM 2100F). For observing the cracked sample, the composite was polished and ion-milled to match the basic standard of TEM observation, and then the sample was bent to create crack after sticked to a molybdem mesh. Micro-Raman measurements were conducted with a NRS-5100 (JASCO Inc., Tokyo, Japan) with 532 nm wavelength incident laser light and a 100× objective for obtaining the stiffness and fracture strength of FLG prepared in this study (see Supplementary material). A four-point bending test with a crosshead speed of 0.05 mm min^{-1} was performed to measure Young's modulus and fracture strength of the composite and pure ceramic specimens (2 mm × 2 mm × 20 mm with precisely parallel flat surfaces, 4 specimens each) after a strain gage (Kyowa, Japan) had been attached to the tension surfaces. A three-point bending test with a similar crosshead speed was performed to directly measure the K_{IC} of 4 notched specimens (Single Edge Notched Beam (SENB) method) with notch-root radius measured by SEM (∼35 μm).

3. Results and discussion

To guarantee the small thickness of graphene platelet while preserve scalability and cost efficiency in production, GO was chosen as starting material for the preparation of composite. The abundant hydrophilic groups on GO make it readily dispersed in water, enabling a colloidal processing to be exploited. In addition, nanoscale Al_2O_3 particles (∼100 nm) carrying opposite surface charge corresponding to GO also can form a uniform colloidal suspension if the pH value is properly adjusted. Following the strategy proposed in our

Download English Version:

https://daneshyari.com/en/article/1413351

Download Persian Version:

https://daneshyari.com/article/1413351

Daneshyari.com