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Graphene nanosheet/titanium carbide composites of a fine-grained structure and improved mechanical properties

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Abstract

Dense graphene nanosheets (GNSs)/titanium carbide (TiC) composites have been produced from graphene oxide (GO)/TiC composite powders by spark plasma sintering. It is unexpected to observe that an introduction of 1.0 vol% GNSs from GO completely stops TiC grain growth by pinning their grain boundaries and densification is completed under the confinement of the flexible GNSs. Such a mechanism assumedly comes from the ultra-thin structure of GNSs, which indicates a crucial role GNSs may play in ceramic processing and has not been reported previously. Compared with monolithic TiC, the flexural strength of GNSs/TiC composites is significantly improved as a result of the refinement of matrix grains and excellent strength of GNSs, while the fracture toughness is enhanced due mainly to crack deflection, GNSs bridging and pull-out.
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1. Introduction

Recently, the mono-layered 2 dimensional graphene has attracted enormous interest for its fascinating physical properties, given the Young′s modulus of $E = = 1.0$ TPa, third-order elastic stiffness of $D = = -2.0$ TPa and intrinsic strength of $\sigma_{int} = = 130$ GPa [1]. Compared with other materials, graphene or graphene nanosheets (GNSs) have a great specific surface area and they do not form agglomerates in a matrix when handled appropriately, suggesting they are a potential reinforcement for ceramic composites. Among those earliest reports on graphene/ceramic composites is our research of in situ produced C/TiC composites, which had a flexural strength and fracture toughness of 480 MPa and 6.5 MPa $m^{1/2}$, respectively [2]. Later we prepared fully dense GNSs/Al_2O_3 composites and observed that this composite had very high electrical conductivity with a low GNS content. More importantly, the charge carrier type could be manipulated by controlling the content of GNSs, enabling the graphene contained ceramic composites to be used as energy conversion material in high temperature environment [3,4]. We found that by adding only 1.2 vol% of GNSs, the average grain size of Al_2O_3 matrix is much finer than that of monolithic Al_2O_3 [5]. Besides, Centeno et al. [6] applied a simple, fast and scalable method to produce GNSs/Al_2O_3 composites by SPS.

More recently, we adopted an in situ strategy for fabrication of reduced graphene oxide/fused silica (rGO/FS) composites. Results showed that the addition of 1 wt% GO sheets to FS resulted in 72% increase in Vickers hardness, and 74% in the fracture toughness [7]. Dusza et al. [8] prepared GNSs/Si_3N_4 composites containing 1 wt% GNSs and reported an increase of about 43% in fracture toughness over the pure Si_3N_4. Liu et al. [9] fabricated GNS/ZTA composites with 0.81 vol% GNSs and found an increase of nearly 40% in fracture toughness.

In view of these achievements, graphene/ceramic composite has been a hot topic worthy further and detailed study. But to

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date, to our knowledge no research has been carried out on TiC composites reinforced by graphene.

Titanium carbide (TiC), as one of the most important high-temperature structural ceramics, has been used in various applications due to its high melting point, good strength and high hardness, good thermal stability, wear and erosion resistance [10–15]. However, like most other ceramics, TiC possesses a low toughness and poor fracture toughness, which hinder its applications as an advanced structural material. An effective way to overcome this problem is to fabricate TiC based composites. Metal bound TiC was expected to excel in these composites, however, due to the low melting point and being easy to soften at higher temperatures, such TiC materials suffered huge drawbacks at high temperature use. To obtain improved high temperature properties, intermetallic aluminides Ni_3Al and FeAl were used as the binder phase in TiC composites, but success is rather limited [16–18].

Another way is to fabricate ceramic particles (SiC, Al_2O_3, TiB_2, TiN) reinforced TiC composites [19,20]. Our previous work on TiC composites demonstrated Al_2O_3–TiC bulk composites by spark plasma sintering (SPS) had much improved mechanical and electrical conduction properties simultaneously [21]. Compared to particles, carbon nanotubes (CNTs) and carbon fibers (C_f) [22] are better to bear the load and prevent crack propagation in matrix for their large aspect ratios. The work of Song et al. [23] on TiC composites reinforced with 20 vol% short C_f showed that fibers could remarkably increased both the room-temperature and elevated-temperature strength and fracture toughness. Katsuyoshi et al. [24] reported that the mechanical properties of TiC matrix composites were remarkably improved with an additive of 0.35 wt% CNTs.

In this work, we used expanded graphite as the starting material to fabricate GO colloid. Dense bulk composites were prepared from mixtures of TiC powder and GO colloid by SPS. During sintering, GO layers were reduced to GNSs at high temperatures. The aim of our research is to investigate the influence of GNSs on the structure and mechanical properties of the as-prepared GNSs/TiC bulk composites.

2. Experimental

2.1. Preparation of GO/TiC composite powders

In this work, the GO was prepared by the modified hummers method reported elsewhere [4,25]. Briefly, commercial expandable graphite (160–50 N, Grafguard, USA) (1 g) was added to a flask and filled with concentrated sulfuric acid (25 ml) at room temperature, followed by addition of potassium permanganate (3.5 g) slowly at 0 °C (ice bath). The as-prepared GO precipitated quickly because of the strong acid environment and the clear supernatant was decanted after a few hours. The precipitate mixture was washed with de-ionized water and centrifuged at 4000–11000 rpm for 30 min for several times to remove impurities. Finally, a GO aqueous solution was prepared from the gelatinous mixture by ultrasonic processing.

The commercially available TiC powder (Japan New Metals Co., Ltd.), with an average particle size of 1.3 μm was used in this study. As-received TiC (10 g) was directly poured into a beaker without treatment. Then water (500 ml) was added followed by ultrasonic stirring for one hour to ensure that the powder could be fully dispersed in water. The GO aqueous solution was added dropwise to the suspensions under ultrasonic stirring. The products were separated by the rotary evaporator followed by drying at 70 °C. The as-prepared mixture was blended on a planetary ball miller (Nanjing NanDa Instrument Plant Co, Ltd., QM-3SP2) with a rate of 200 r/min for 8 h before being dried at 80 °C for 12 h.

2.2. Sintering of GNSs/TiC bulk composites

Bulk composite samples were prepared using SPS apparatus (Dr. Sinter 725; Sumitomo Coal Mining Co, Tokyo, Japan). GO/TiC powders were loaded into a 15 mm inner diameter graphite die and sintered in a vacuum of 6 Pa. The heating rate was 100 °C/min and soaking time was 3 min. A uniaxial pressure of 60 MPa was applied from 1000 °C upwards and maintained during the dwelling at 1550 °C.

2.3. Characterization

The sintered bulk samples were grinded and polished by a polishing machine (UNIPOL-802, Shenyang Kejing Auto-instrument Co., Ltd.). Density measurements were conducted using the Archimedes' method. The morphology and microstructure of as-prepared samples were characterized by a field emission scanning electron microscopy (FESEM, Hitachi S-4800) and transmission electron microscopy (TEM, 2100F, Japan). Grain sizes were measured by the linear intercept method [26].

$$\overline{D} = 1.56\frac{C}{MN} \tag{1}$$

where $\overline{D}$ is the average grain size, C is the total length of test line used, N is the number of intercepts, and M is the magnification of the photomicrograph. About 400 intercepts were counted for each measurement.

The flexural strength was examined by three-point bending test. The testing was performed using a DS-II multifunctional desktop tester with a cross-head speed of 0.05 mm/min. Four samples were used for each run. It was calculated using the following equation Eq. (2) [27]:

$$\sigma = \frac{3PL}{2bh^2} \tag{2}$$

where P is the load at the fracture point, L is the span length, b is the sample breadth and h is the sample thickness.

The Vickers hardness and fracture toughness of the samples were determined by Vickers indentation technique (FV-700, Future-Tech Corporation) at a load of 3.0 Kg f (29.4 N) with a dwell of 5 s on carefully polished surfaces. Six measurements were conducted for each sample to calculate the average value.

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