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Mechanical properties and toughening mechanisms of graphene platelets reinforced Al_2O_3/TiC composite ceramic tool materials by microwave sintering

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ABSTRACT

A type of Al_2O_3/TiC composite ceramic tool material reinforced with graphene platelets (GPLs) was fabricated by microwave sintering. The effects of GPLs content on the microstructure, mechanical properties and toughness mechanisms of Al_2O_3/TiC/GPLs composite ceramic tool materials were studied. The experimental results showed that the microstructure of the composite became finer with the incorporation of GPLs. The optimal mechanical properties were achieved with 0.2 wt% GPLs. The relative density, Vickers hardness and fracture toughness were 97.7 ± 0.2, 18.5 ± 0.5 GPa and 8.7 ± 0.4 MPa $m^{1/2}$, respectively. Compared to Al_2O_3/TiC composites, the Vickers hardness decreased by 12.7%, but the fracture toughness increased by 67.3%. Also, agglomerated GPLs and pores were observed in the new composite ceramic tool materials. The toughening mechanisms were crack deflection, crack bridging, crack branching and pull-out of GPLs.

1. Introduction

High-speed machining has become the main technology in our industrial production due to the high efficiency, high accuracy and simple process [1]. Al_2O_3-based ceramic tool materials possess great advantages in the field of high-speed machining for their high hardness, high melting point, corrosion resistance and low affinity with metal. Meanwhile, they show better cutting performance than traditional tools in hard turning, so they have been taken as suitable and competitive tool materials [2]. However, brittleness and fabrication have limited the application of Al_2O_3-based ceramic tools.

Up to now, some effective methods have been proposed to strengthen and toughen the Al_2O_3-based ceramic tool materials. One route is phase transformation of ZrO_2 in ceramic matrix. Volume expansion of the ZrO_2 particles can be caused for the transformation (from t-phase to m-phase) at 950 °C. The expansion particles would generate both dilatational and shear stresses, which can prohibit the propagation of crack [3]. Another way to overcome the brittleness is to incorporate submicron or nano particles, such as TiN, SiC, TiC and MgO [4–7], to fabricate Al_2O_3-based composites. These particles tend to pin in the grain boundaries, which refine the grain and enhance the strength. Yin [8] used hot-pressing (HP) sintering method to prepared Al_2O_3/TiC micro-nano-composite ceramic tool materials and reported a fracture toughness of 8.3 MPa $m^{1/2}$ with 6% nano TiC. Chakravarty [9] employed spark plasma sintering (SPS) to prepared Al_2O_3-based composites with the addition of 0.125% MgO, the fracture toughness of 4.5 MPa $m^{1/2}$ was obtained. Fibre or whisker reinforcing technology is also an effective way to improve the fracture toughness. Hansson et al. [10] fabricated alumina composites reinforced with SiC whisker by hot-pressed sintering, which reported that the optimal fracture toughness is 8 MPa $m^{1/2}$ and increased by 100% over the monolithic alumina. Bocanegra-Bernal [11] investigated the impact of carbon nanotubes (CNTs) on the mechanical properties of ZrO_2 toughened Al_2O_3 (ZTA) and the fracture toughness increased by 44%. However, one-dimensional fibers like CNTs or whisker as the reinforcement phase for the preparation of ceramic composite are easily to generate agglomeration in ceramic matrix. The aggregate degrades mechanical properties of the composites.

Graphene is one of the allotropes of elemental carbon, which has attracted significant attention in composites due to the unique properties and two-dimensional structure, since the discovery in 2004 [12–14]. Graphene platelets (GPLs) are made of several layers of graphene, which are also called as graphene nanosheets (GNS), graphene nanoplatelets (GNPs) or multilayer graphene nanosheets (MGN) in

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literature. GPLs possess large specific surface area and outstanding mechanical properties, which are taken as competitive nanofiller in composite materials [15,16]. Some researches have incorporated GPLs as nanofiller to strengthen and toughen pure ceramics. Chen [17] used HP to prepared GNS/Al_2O_3 composites and reported a 43.5% increase in fracture toughness with the incorporation of 0.2 wt% GNS. Walker et al. [16] used the SPS to prepared Si_3N_4/GPLs ceramic composites with addition of 1.5 vol% GPLs and the measured fracture toughness is 6.6 MPa $m^{1/2}$ which is 135% higher than that of monolithic Si_3N_4.

At present, HP and SPS are the main methods to prepared Al_2O_3-based ceramic tool materials [8,15]. Hot pressing sintering needs long sintering time in the densification process. Abnormal grain growth is inevitable, which is detrimental to mechanical properties of the ceramic tools. Although Spark plasma sintering (SPS) is a type of novel fast sintering technology, which is characterized by high heating rate and short sintering period, the high energy consuming make it difficult to prepared ceramic tools in economic viability [18].

In order to reduce the cost and increase the efficiency in fabricating ceramic composites, microwave sintering has been proposed [19–22]. Microwave sintering is a process in which heat is produced as a result of coupling reaction between the whole specimen and electromagnetic wave [23,24]. Therefore, the heating is more rapid and temperature distribution is homogeneous within the materials. Meanwhile, microwave electromagnetic can decrease sintering activation energy of the material and promote the grain-boundary diffusion [25]. The crystalline structure can also be refined in microwave file and much better properties of materials can be obtained.

As we know, Al_2O_3/TiC ceramic tools are widely used in machining hardened materials in high speed [2,26]. In our previous work, Al_2O_3/TiC ceramic tool materials have been prepared by microwave sintering, and the cutting performance was also discussed [20,27]. In this paper, we attempt to fabricate Al_2O_3/TiC/GPLs composite ceramic tool materials reinforced with GPLs using microwave sintering and evaluate the potential of the new ceramic tools. The effects of GPLs content on microstructure and mechanical properties were studied. The toughening mechanisms were also investigated.

2. Experimental procedure

2.1. Preparation of materials

The Al_2O_3/TiC/GPLs composite ceramic tool materials (ATG) were composed of 60 wt% α-Al_2O_3 (purity: 99.9%, d_{50}=0.5 μm, Shanghai, China), 30 wt% TiC (purity:99.9%, d_{50}=0.5 μm, Shanghai, China), 6 wt% Mo and Ni (purity:99.9%, d_{50}=2.0 μm, Shanghai, China), 4 wt% MgO and Y_2O_3 (purity:99.9%, d_{50}=1.0 μm, Shanghai, China), and GPLs were purchased from Nanjing XFNANO Materials Tech Co Ltd. The quality and morphologies of the purchased GPLs are shown in Fig. 1. The GPLs are stacks of graphene about 3–10 nm in thickness and 5–10 μm in level dimensional. Additionally, different GPLs contents were added into the composites (0.1, 0.2, 0.4, 0.6 and 0.8 wt%). MgO and Y_2O_3 [28] were added as sintering aids to improve the process of densification for ATG. The GPLs were dispersed in the N-Methyl-2-pyrrolidone (NMP) and sonicated for one hour with concentration of 2 mg/ml. Then, the α-Al_2O_3, TiC, Mo, Ni and sintering aids were added into the suspensions and further sonicated with mechanical agitation for 20 min. The mixed powders were ball milled with alumina grinding media in planetary ball mill (Model QM-3SP2, Nanjing, China) for 48 h, 3 wt% Poly Vinyl Alcohol (PVA) with a concentration of 5 wt% was added to the mixed powders as binder for 2 h before the end of ball mill. Subsequently, the mixed slurries were dried in a vacuum drying oven (Model DZF-1, Shanghai, China) at 120 °C, and then sieved through a 100-mesh sieve for further use. A certain value of dried powders were put into a cold steel die, and pressed uniaxially at 200 MPa for 2 min. Rectangular samples with 13 mm length and about 7 mm height were prepared for further characterization and sintering.

2.2. Microwave sintering

The green samples were put into a powder bed with silicon carbide and graphite powder, which can heat samples in low temperature and prevent samples from oxidation [20]. The experiment was made in a 2.45 GHz microwave furnace (Model NJZ-1, Nanjing, China) with power output in the range of 0–6 kW. The temperatures was measured by an infrared pyrometer which was installed in the furnace chamber with the start temperature from 350 °C. The samples sintering was performed at 1700 °C with the holding time of 10 min using the heating rate of 30–40 °C/min in high pure argon atmosphere. After that, the sintered samples were cooled down to room temperature.

2.3. Characterization

Before the test, all sintered samples were ground and polished to 1 μm roughness. The relative density of the samples were measured by Archimedes' method with the distilled water as immersion medium using density of 2.1 g cm^{-3} for GPLs. Vickers hardness tests were taken on the polished surface using a Vickers diamond pyramid indenter (Model HV50, China) with a load of 196N and a loading duration of 15 s. The fracture toughness was determined by the Vickers indentation method proposed by Evans and Charles [29]. At least five available dates were collected for each property test. The fractured surfaces and crack extensions on the polished surfaces were observed by scanning electron microscopy (SEM, Quant 250FEG, USA). X-ray diffraction (XRD, D8 Advance, Germany) with copper Kα radiation was used to analyze phase identification. The liner intercept method has been used to measure the grain size. The morphologies used to evaluated grain size were magnified 4000 times by SEM. No less than three images were chosen and more than 200 grains were covered.

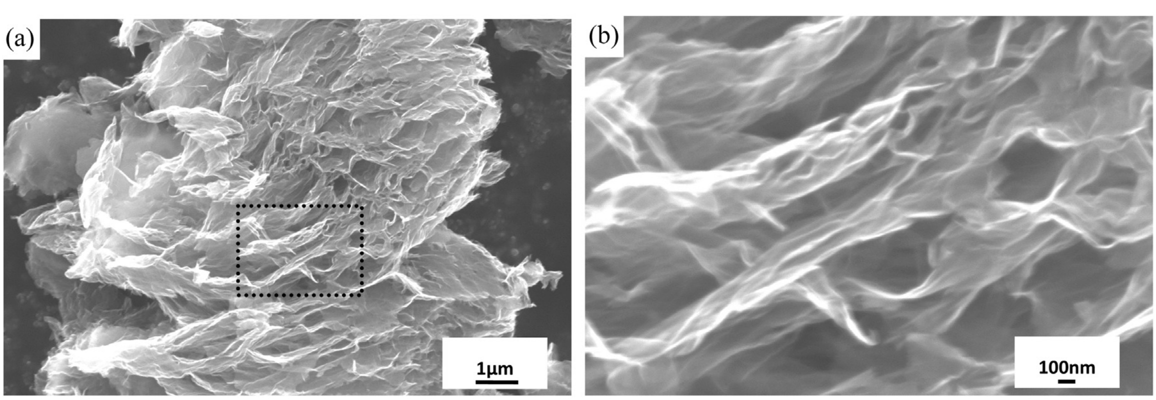

Fig. 1. (a) SEM morphology of GPLs and (b) the GPLs with large magnification.