Toughening Mechanism of ZTA–TiC–Fe Ceramic Materials Produced by High-Gravity Combustion Synthesis

Hongwei Zhao, Shibin Guo, Jiangtao Li and Jinhong Li

Abstract Through coupling of high-gravity field and high temperature field of combustion synthesis, ZrO_2 toughened Al_2O_3 (ZTA) adding with TiC strengthened iron ductile (ZTA–TiC–Fe) composite materials were prepared by high-gravity combustion synthesis. The composite toughening of Al_2O_3–TiC ceramic materials was realized, including phase transformation toughening of tetragonal zirconia, zirconia fiber toughening, and metal ductile phase toughening. The results showed that the fracture toughness of ZTA–TiC–Fe composite materials reached 9–11 $MPa{\cdot}m^{1/2}$, which was 2–3 times higher than pure Al_2O_3–TiC commercial tool materials.

Keywords Al_2O_3–TiC · Combustion synthesis · Zirconia toughened alumina

Introduction

Ceramic materials have the advantages of high strength, high hardness, and corrosion resistance, so they have a great prospect in industrial applications. However, due to their poor fracture toughness, their application scope is limited. Materials researchers have tried a variety of methods to improve the toughness of ceramic materials [1–3]. In recent years, the accepted solution is to conduct composite

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toughening of ceramic materials, which can greatly improve the toughness of ceramic materials [4–7].

Consolidated bulk ceramics are generally produced by powder sintering, such as pressureless sintering (PS) [8], hot pressing (HP) [9], and spark plasma sintering (SPS) [10]. The sintering method requires long-time heat treatment at high temperatures by furnace, which means a long processing time and much energy consumption. A recently-developed method to prepare bulk cermets is combustion synthesis under a high gas pressure or in a high-gravity field [11–13]. Combustion synthesis utilizes the heat energy released during exothermic combustion reactions to synthesize the target materials and does not require any furnaces [14, 15]. The high gas pressure or high-gravity field is applied to realize simultaneous densification during the synthesis for directly preparing dense bulk products. As a fast and furnace-free method, combustion synthesis exhibits a reduced processing time and near-zero energy consumption, which may be competitive in comparison with the conventional sintering approach.

In this paper, multi-component like ZrO_2 toughened Al_2O_3 (ZTA) adding with TiC strengthened iron ductile (ZTA–TiC–Fe) ceramic materials were prepared by high-gravity combustion synthesis. This material consists of zirconia toughened alumina ceramic matrix and antioxidant metallic iron reinforced by TiC hard particles. The toughening mechanism of the ZTA–TiC–Fe ceramic materials were investigated.

Experiment

Commercial powders of Al, Fe_2O_3, ZrO_2, Y_2O_3, Ti, and graphite were used as raw materials. The raw materials were mixed according to the following chemical reactions:

$$Al + Fe_2O_3 \rightarrow Al_2O_3 + Fe \tag{1}$$

$$3Al_2O_3 + 2ZrO_2 + 0.06Y_2O_3 = ZTA \tag{2}$$

$$Ti + C = TiC \tag{3}$$

The thermite system was used to produce Al_2O_3 and Fe, ZrO_2 and Y_2O_3 were used to produce the ZTA phase, and Ti and C were used to produce TiC. The molar ratio of Al_2O_3 and ZrO_2 in the ZTA phase was Al_2O_3:ZrO_2 = 3:2. In order to obtain high temperature of the reaction system, the weight ratio between ZTA and Fe was fixed to be ZTA:Fe = 3:2, and the weight content of TiC reinforcement phase was 20%.

The powder mixture of the raw materials was well homogenized by rotatory ball milling for 2 h with a rotation speed of 60 r/min, and then dried at 110 °C for 2 h. Each batch of 200 g reactant powder was cold-pressed into a round compact with a

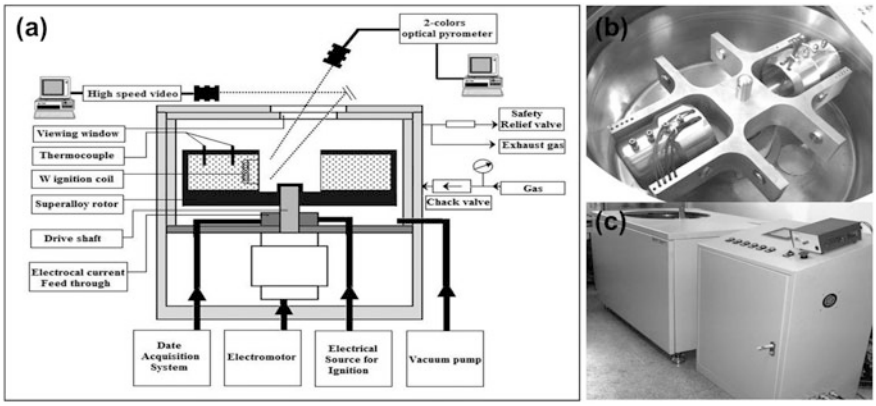

Fig. 1 A schematic diagram (**a**) and photos (**b**, **c**) of the facility for high-gravity combustion synthesis [11]

diameter of 40 mm at a pressure of 40 MPa, and the porosity in the compact was about 50%. The compact was placed in a graphite crucible. The crucible was coated with carbon felt and placed into a steel vessel, which was mounted on a rotator in a reaction chamber (see Fig. 1). A high-gravity field with an acceleration of 800 g ($g = 9.8\ m/s^2$) was induced by the centrifugal effect. The combustion reaction was triggered by passing an electric current about 10 A through a tungsten coil closely above the reactant compact. When the reaction was over and the sample cooled down, the sample was taken out from the reaction chamber.

The synthesized samples were machined with an electroplated diamond wire saw and then well polished. The bulk density of samples was measured according to the Archimedes principle. The phase assemblage was identified by X-ray diffraction (XRD; D8 Focus, Bruker, Germany). The microstructure was examined by scanning electric microscopy (SEM; S4800, Hitachi, Japan). The Vickers hardness was measured by the indentation method with a load of 98 N and dwelling time of 15 s. The fracture toughness was tested by the single-edge-notched-beam (SENB) technique with bars of $25 \times 2 \times 4\ mm^3$. The notch was generated with a diamond saw, and had a width of 0.3 mm and depth of 2 mm.

Results and Discussion

Figure 2 shows the XRD pattern of the synthesized ZTA–TiC–Fe ceramic material. Four crystalline phases of TiC, Al_2O_3, tetragonal ZrO_2, and Fe are observed agreeing well with the designated phase compositions. From the experimental results, the combustion reactions are complete and the reactants are fully converted into the target products.

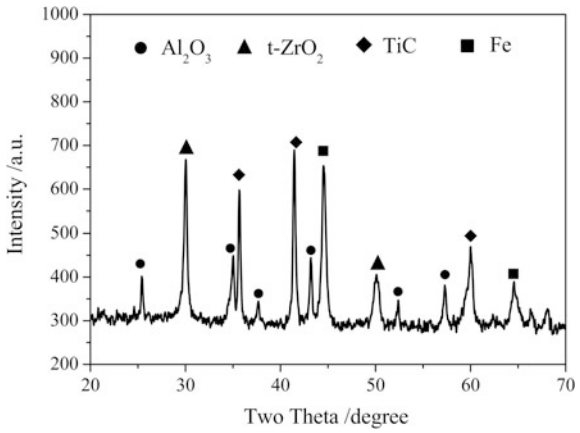

Fig. 2 XRD pattern of synthesized ZTA–TiC–Fe ceramic material

Figure 3 shows the microstructure features of the ZTA–TiC–Fe ceramic materials. The light Fe grains distribute uniformly in ZTA matrix. The TiC micro-grains locate at the interface between Fe grain and ZTA matrix. It forms a shell covering the surface of Fe particles, prevents the polymerization of Fe droplets, improves the wettability of metal Fe and ZTA ceramic matrix, and enhances the interfacial bonding strength of the two materials. Thus, this is conducive to improving the overall strength and toughness of the ZTA–TiC–Fe ceramic material.

The toughness of the ZTA–TiC–Fe ceramic material is 9–11 MPa·$m^{1/2}$, which is 2–3 times higher than pure Al_2O_3–TiC commercial tool materials. There are three toughening mechanisms that improved the toughness of the ZTA–TiC–Fe ceramic material. The first one is the ZrO_2 fibers toughened the Al_2O_3 matrix as show in Fig. 4a. The ZrO_2 fibers with 1–2 μm distributed uniformly in Al_2O_3 matrix, which has the same direction and equal interval. And that the ZrO_2 fibers are in situ

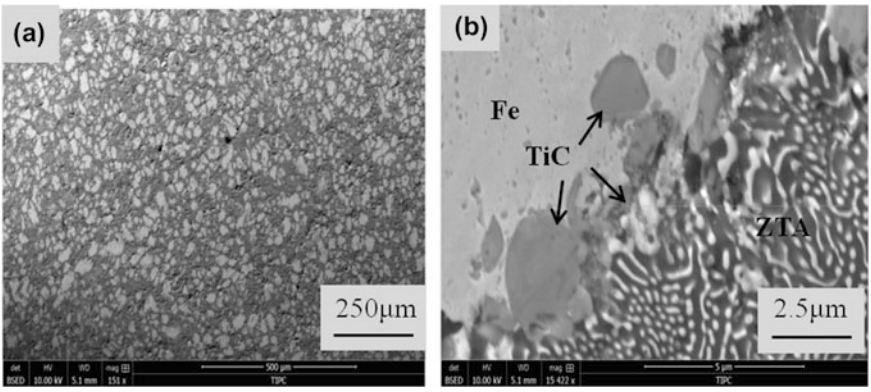

Fig. 3 Microstructure features of ZTA–TiC–Fe ceramic materials: **a** Fe uniform distribution in ZTA matrix, **b** TiC micro-grain at the interface between Fe and ZTA

formed in Al_2O_3 matrix with few defects at interface. It is better than the traditional method of extra addition. The second one is the phase transformation toughening of the tetragonal ZrO_2 as shown in Fig. 4b. There is obvious volume expansion at the fracture of the ZrO_2 fiber, this is due to tetragonal phase transformed monoclinic phase in ZrO_2 fiber by stress driving. The third one is ductile metal phase

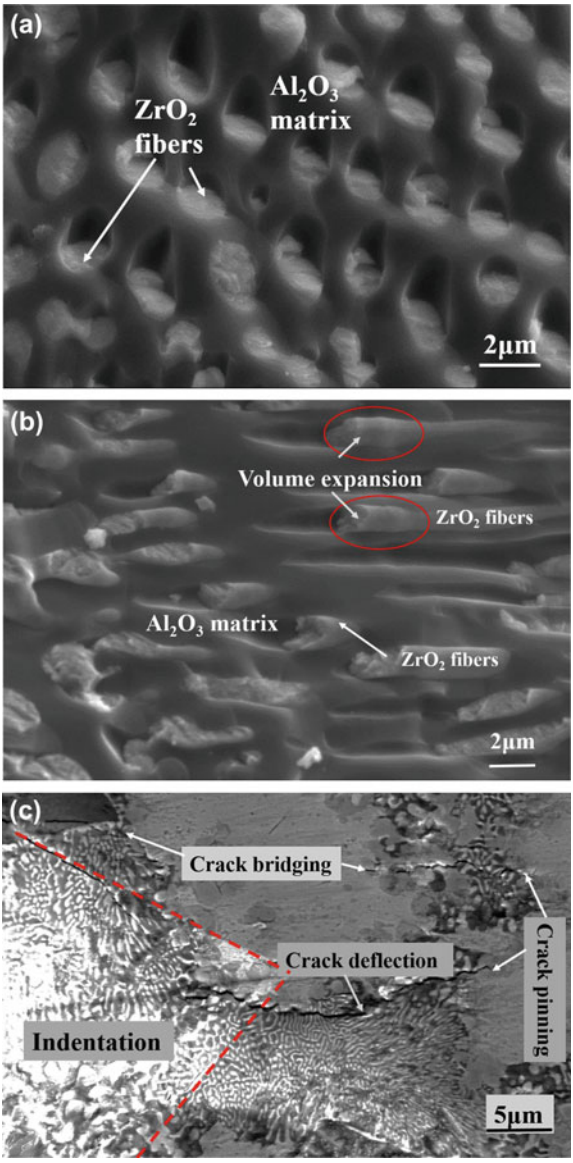

Fig. 4 Toughening mechanisms in ZTA–TiC–Fe ceramic material: **a** ZrO_2 fibers toughening; **b** ZrO_2 transformation toughening; **c** toughening by ductile metal phase

toughening as shown in Fig. 4c. The Fe grains cause the crack deflection for adding the crack propagation distance, and absorb some or whole stress of the crack tip appearing the crack bridging or crack pinning phenomenon.

Conclusion

ZTA–TiC–Fe ceramic material has been prepared by a fast and furnace-free way called high-gravity combustion synthesis. The Fe–TiC core-shell structure is conducive to distributing Fe grain in ZTA matrix and improving the interface strength. For the combination of three toughening mechanisms, such as fiber toughening, phase transformation toughening, and ductile phase toughening, the toughness of the ZTA–TiC–Fe ceramic material reaches 9–11 $MPa \cdot m^{1/2}$, which is 2–3 times higher than pure Al_2O_3–TiC commercial tool materials.

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