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Quantitative analysis of toughening effect of crack deflection on Si_3N_4/Si_2N_2O composites through improved indentation method

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ABSTRACT

In this study, Si_3N_4/Si_2N_2O composite ceramics prepared by hot pressing were used as an example, and the material fracture morphology and fracture mechanism were analyzed. Based on the formula of fracture toughness measured by an indentation method, a quantitative computation method was proposed to determine the toughened effect of ceramic materials resulting from the crack deflection by the second phase. The grain size and sintering density are increased with the increase of sintering temperature. The toughening effects resulting from the crack deflection is increased, and the main mode of fracture is transformed into the transgranular fracture. The Si_2N_2O grains can play a role in the toughening process because these grains can hinder the cracks extending along the radial direction. However, when the cracks extend in the axial direction, the toughening effect of Si_2N_2O grains is not obvious because of the internal stacking faults in the axial direction. The improved indentation method can quantitatively analyze the toughening effect of the second phase of composite ceramics, and the validity of this method are verified by comparing the fracture toughness of Si_3N4/Si_2N_2O and fine grained β- Si_3N_4 ceramics.

1. Introduction

Fracture toughness is one of the main indexes of the mechanical properties of ceramic materials. Therefore, studying the fracture behavior, morphology, and toughening mechanism of ceramic materials is necessary to improve their fracture toughness, which is significant for designing ceramic materials and using products [1,2]. Numerous findings reported in recent years indicate that the bridging, pulling out, and fracture of the second phase in the process of ceramic fracture can hinder the crack propagation and enable the crack deflection to improve the fracture toughness, creep resistance, and fatigue resistance of the ceramic material [3–5].

The h-BN/Si_3N_4 composite ceramics were prepared by Vleugels [6] through hot pressing sintering method with α-Si_3N_4 and h-BN as matrices and Y_2O_3-Al_2O_3 as a sintering aid. The effect of h-BN content on the fracture toughness of h-BN/Si_3N_4 multiphase ceramics was studied. The effects of SiC content on the hardness and fracture toughness of SiC-Si_3N_4 multiphase ceramics were analyzed by Goto et al. [7]. Okazaki [8] prepared Si_3N_4 ceramics with high fracture toughness by adding rare earth as sintering aids into Si_3N_4 ceramic materials, and the effect of different additives on the fracture toughness of the composite ceramics was analyzed. Coppola [9] prepared Si_3N_4/TiC nanocomposite ceramic tool materials with excellent properties were prepared by adding polyethylene glycol, because Si_3N_4-based ceramic materials are difficult to sinter. In addition, the fracture morphology and mechanism of the composites were analyzed. Kingery [10] mixed TiC, TiN, WC, and other second-phase particles in Si_3N_4 powder to improve the fracture toughness and other mechanical properties of nano Si_3N_4 ceramics.

Most researches focus on improving the fracture toughness of ceramics by improving the sintering process of materials. Some scholars also conducted a qualitative analysis of the toughening effect, but there is no quantitative explanation of the effect of the crack deflection resulting from the bridging, pullout, and crack of the second phase on the toughening effect [11–13]. In this paper, Si_3N_4/Si_2N_2O composite ceramics prepared by hot pressing were considered an example, and the material fracture morphology and fracture mechanism were analyzed. A quantitative computation method based on the formula of fracture toughness measured through the indentation method was proposed. This computation method is aimed to determine the toughening effect of ceramic materials resulting from the crack deflection.

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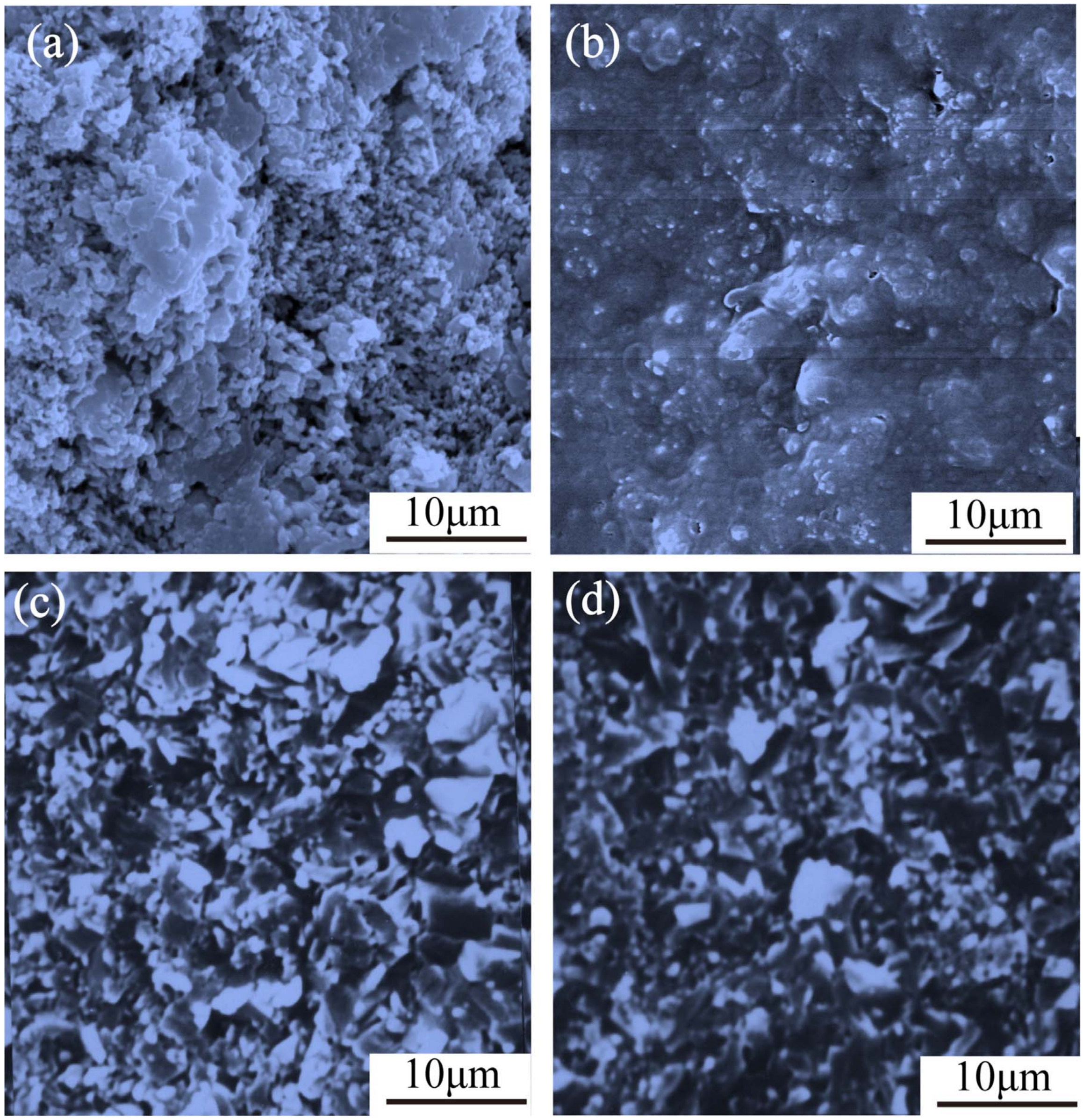

Fig. 1. Fracture morphology of Si_3N_4/Si_2N_2O multiphase ceramics sintered at different temperatures: (a) 1500 °C, (b) 1600 °C, (c) 1650 °C, and (d) 1700 °C.

2. Experiment

2.1. Material preparation

The Si_3N_4/Si_2N_2O multiphase ceramics were prepared by mixing amorphous Si_3N_4, Y_2O_3, and Al_2O_3 nanopowders with the mass ratio of 93:5:2 [14]. The grain size of the amorphous Si_3N_4 and Y_2O_3 powders [11] was 20 nm. The hot pressing sintering process was used in the experiment, and the sintering pressure was 25 MPa. Three sintering temperatures (1500, 1600, 1650 and 1700 °C) were used to prepare different samples, and the holding time was 1 h. After the sintering process, ceramic blocks with a thickness of 2 mm and a diameter of 30 mm were prepared. In preparing finegrained β-Si_3N_4, the amorphous Si_3N_4 powder was used to replace amorphous powder, and other experimental conditions were unchanged.

2.2. Analysis method

2.2.1. Microstructure and fracture morphology analysis method

The microstructure was observed and analyzed by using S-4800 scanning electron microscope and JOEL 2010 transmission electron microscope. The preparation method of the scanning samples is as follows: the samples are stuck on aluminum plates with the fracture surface to be observed facing upward, and the surface of the samples is sprayed with gold. Finally, the samples were treated by ion thinning and carbon spraying.

2.2.2. Quantitative calculation method for crack deflection toughening effect

In the research and application of fracture toughness testing technology for ceramic material, a common technique is measuring the fracture toughness of the material through the indentation method [15]. The general approach is to introduce an indentation crack on the surface of the specimen and then to measure the length of the crack and the hardness of the material to determine the fracture toughness of the material. The calculation formula is expressed in Formula (1) [16]. The study on crack propagation in the ceramic material indicates that the crack will be deflected, pulled out, and bridging during the crack propagation, which plays an important role in the toughening and strengthening of ceramic materials [17]. The microscopic actual length