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Microstructures and mechanical properties of Ti_3SiC_2/TiC–Al_2O_3 composites synthesized by reactive hot pressing

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Ti_3SiC_2/TiC–Al_2O_3 composites with different Al_2O_3 contents were fabricated by in-situ reaction and hot pressing sintering. Laminar Ti_3SiC_2 grains and granular Al_2O_3 grains were densely packed and tightly bonded, and cubic TiC grains presented in the surfaces of Ti_3SiC_2 grains. Al_2O_3 significantly restrained the grain growth of Ti_3SiC_2 matrix. The dispersed Al_2O_3 grains inclined to be pulled out, but Al_2O_3 aggregates inclined to be cut by the crack front at the crack surface. The flexural strength and fracture toughness first increased and then decreased with the increasing Al_2O_3 content. The composite with 20 wt% Al_2O_3 addition showed the highest flexural strength of 649 MPa and with 10 wt% Al_2O_3 addition showed the best fracture toughness of 7.15 MPa m$^{1/2}$. The mechanism responsible for improved mechanical properties for Ti_3SiC_2/TiC–Al_2O_3 composites were the synergistic action of particulate dispersion reinforcement, fine-grain toughening, grain pullout, microcrack deflection, and lamella bending and slipping from three different kinds of grains.

1. Introduction

Ti_3SiC_2 with lamellar structure is just like BN or graphite. However, it is stronger in mechanical properties and better in oxidation resistance than BN or graphite [1]. It is considered a potential structural/functional material for its combined metallic- and ceramic-like properties, such as low density, high modulus, good thermal and electrical conductivity, excellent thermal shock resistance and high damage tolerance and easy machinability [2–4]. However, the relatively low strength and hardness limit its application. Al_2O_3 has high hardness, melting point, mechanics strength and elastic modulus, and excellent adaptability in oxidation atmosphere at high-temperature. Al_2O_3 is a ceramic showing considerable promise for use in a number of engineering applications. TiC has high modulus, high melting point, high hardness and good erosion resistance. In fact, TiC phase usually coexists and shows special orientation relationship with Ti_3SiC_2 phase during the synthesis process of bulk Ti_3SiC_2 [4]. However, the potential of the ceramic materials has been limited by low toughness. Improving the fracture resistance of Al_2O_3 or TiC ceramic via microstructural design by introducing the second phase is a promise way.

Hard ceramic particles such as TiC [5–7], TiB_2 [5,8] and SiC [1,8,9] have been incorporated into Ti_3SiC_2 to improve the mechanical properties. Wang et al. [10,11] reported the increase by 50% in the hardness for Ti_3SiC_2/20 vol%Al_2O_3 composite but decrease in the other mechanical properties with Al_2O_3 addition higher than 5–10 vol%. Luo et al. [12,13] prepared Al_2O_3–Ti_3SiC_2 composites and its functionally graded materials, showing the hardness decreased but fracture toughness and strength increased with the increase of Ti_3SiC_2 content. Chin et al. [14] improved the strength and toughness of Al_2O_3 by adding Ti_3SiC_2 particles into Al_2O_3 matrix. Chen et al. [15] synthesized Ti_3AlC_2/TiC–Al_2O_3 composite in a $3TiO_2$–5Al–2C system which showed higher flexural strength and Vickers hardness than pure Ti_3AlC_2 ceramic.

By a synergy mechanism between two or more strengthening and toughening methods, the enhancement in mechanical properties can be brought by the combination of Ti_3SiC_2 with Al_2O_3 and TiC. Most importantly, compared to SiC, Al_2O_3 and TiC are more suitable candidate reinforcements for the Ti_3SiC_2 matrix due to the better thermal expansion match. The thermal expansion coefficients of both Al_2O_3 and TiC are lower than that of Ti_3SiC_2 but higher than that of SiC. Additionally, compared to TiB_2, Al_2O_3 has lower density and better oxidation resistance at high temperatures. However, previous studies rarely concentrated on the effects of the combination of Al_2O_3 and TiC on the microstructure and mechanical properties of Ti_3SiC_2-based composites. The present work focused on the microstructures and mechanical properties of Ti_3SiC_2/TiC–Al_2O_3 composites with different Al_2O_3 added amount from 5 wt% to 30 wt% fabricated by in situ reactive/hot pressing sintering. Ti_3SiC_2 matrix was synergistically reinforced by granular Al_2O_3 and equiaxed TiC grains in these composites.

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Too much content of reinforcements resulting in the inhomogeneous phase distribution and adversely affecting the mechanical properties of the composites was indicated in previous reported papers. However, different fracture modes between dispersed particles and particle agglomeration as reinforcements were not elaborated in detail. In this study, the detailed comparisons of different fracture modes between dispersed particles and particle agglomeration as well as between large particles and fine particles were drawn. The mechanism responsible for improved mechanical properties of Ti_3SiC_2/TiC–Al_2O_3 composites from three different grains was revealed.

2. Experimental procedures

2.1. Sample preparation

Ti_3SiC_2-based composites were prepared by in situ reaction combined with hot-pressure sintering. The Al_2O_3 powder was added to improve the mechanical properties of Ti_3SiC_2-based composites. The addition amount of Al_2O_3 powder was 5–30 wt% with an interval of 5 wt%. For ease of reference, the composite samples with the different Al_2O_3 powder addition amount would be named TA5, TA10, TA15, TA20, TA25 and TA30 respectively. The sample without Al_2O_3 addition named as T was also synthesized for comparison.

The starting mixtures of Ti (average particle size: 40.0 μm, > 99.5% purity), Si (average particle size: 40.0 μm, > 99.5% purity), carbon (average particle size: 6.27 μm, > 99.5% purity) in molar ratio of 3:1:2 combined with Al_2O_3 (average particle size: 6.23 μm, > 99.5% purity) at different mass contents were prepared by wet ball milling in ethanol for 6 h and dried. Zhang et al. [16] reported Ti_3SiC_2 was formed through the reaction between $Ti_5Si_3C_x$, TiC_x and carbon mainly at 1400–1500 °C based on the starting materials of Ti, Si and graphite by hot pressing sintering. Song et al. [8] reported the decomposition of Ti_3SiC_2 at 1550–1600 °C in reactive hot-pressed (TiB_2+SiC)/Ti_3SiC_2 composites. Therefore 1500 °C was chosen as the sintering temperature in this experiment. The final mixture with the desired composition was then reactive hot-pressed under a pressure of 25 MPa in a graphite die coated with BN under a vacuum atmosphere at 1500 °C for 3 h to obtain a dense composite sample. A heating rate of 10 °C/min was used to heat up to 1500 °C following with cooling to room temperature in the furnace. The samples with cylinders of 50 mm in diameter and about 10 mm in height were obtained.

2.2. Characterization

The open porosities and bulk densities were measured by Archimedes' method according to ASTM C-20 standard. The phase compositions and relative content of several phases in the

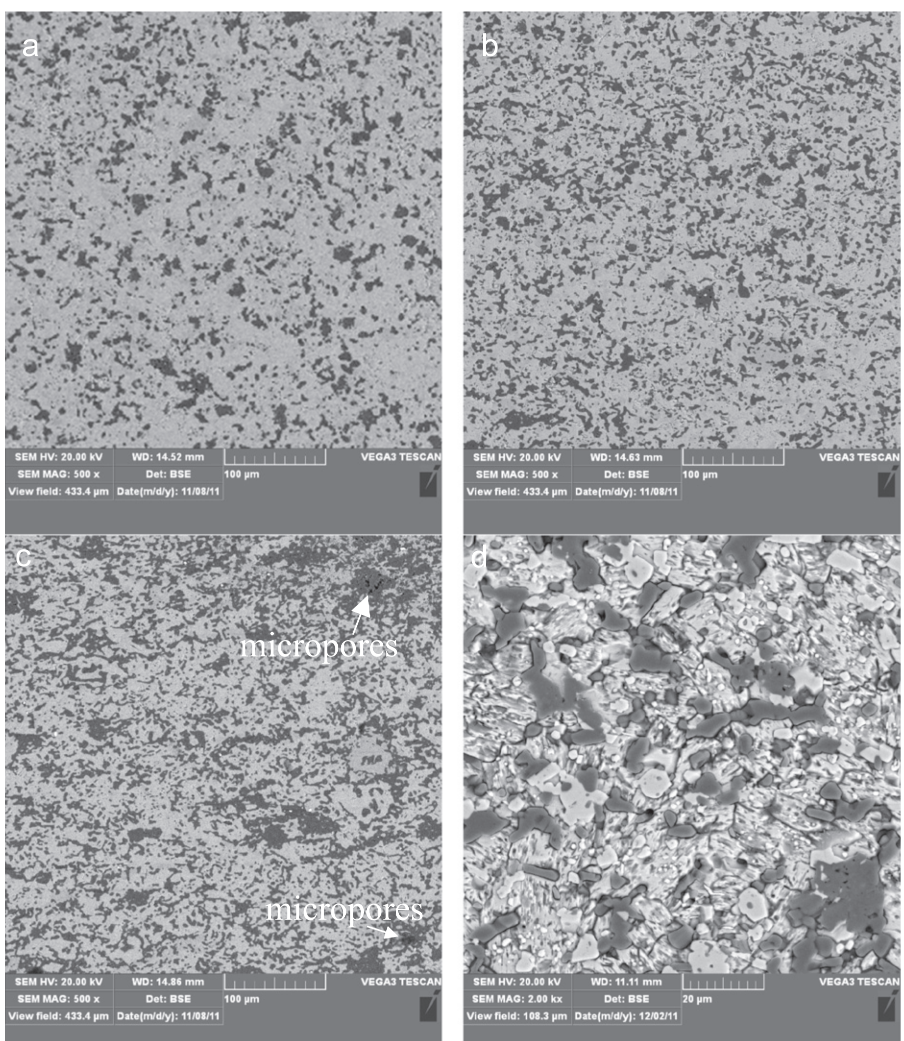

Fig. 1. Microscopic morphology of sintered samples: (A) TA10; (b) TA20; (c) TA30 and (d) etched SEM microstructure of TA15.