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Effects of Nd_2O_3 on the mechanical properties and oxidation behavior of Ti/Al_2O_3 composites by vacuum hot pressing sintering

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ABSTRACT

A range of Ti/Al_2O_3 composites with different Nd_2O_3 content (0–5 vol.%) were fabricated by vacuum hot pressing sintering at 1420 °C for 1.5 h under the pressure of 30 MPa. The sintered samples with Nd_2O_3 additives exhibited more superior performances than those without additives. When 3.0 vol.% Nd_2O_3 was added, Ti/Al_2O_3 composite showed optimal density (relative density of 98.79%), hardness (Vickers hardness of 15.87 GPa), strength (flexural strength of 412.45 MPa), toughness (fracture toughness of 8.77 MPa $m^{1/2}$) and oxidation resistance (oxidation depth of ~244 μm). However, excessive additive (> 3.0 vol.%) would weaken these positive effects. SEM results revealed that the superior performances could be attributed to the grain refinement and microstructure densification. Moreover, plate-like grains were found at the interface and additional experiments demonstrated that their formation was caused by enrichment of Nd element, which is beneficial to the microstructure densification.

1. Introduction

Al_2O_3 is one of the most widely used ceramic because of its favorable mechanical properties, such as superior high hardness, excellent compressive strength, good wear resistance and high elastic modulus [1–3]. However, the application as structural materials has been limited by the low fracture toughness and weak thermal shock resistance [4,5], some researchers have been attempting to solve this problem by adding some reinforced phases [6]. So far, Al_2O_3-based cermet composites, such as Al_2O_3–Cu, Al_2O_3–Fe, Al_2O_3–Ni, have been widely used as service in high-temperature thermal or electrical conductors, iron catalyst and solar selective absorbing coatings [7–9], but the researches on improving the strength and toughness of the Al_2O_3-based cermet composites are not as ideal as expected. Mahmoud [10] reported that uniform distribution of nano-sized Cu reinforcement throughout the Al_2O_3 matrix was obtained after 20 h milling by Al_2O_3–20 wt.%–Cu nano-composites. Although the micro-hardness of the sintered composites was improved, the fracture toughness was reduced with the increasing of milling time for starting powder.

Ti and Ti alloy have gained widespread application in aerospace and automotive industries which are mainly ascribed to their desirable properties, such as light weight, high specific strength, good ductility and excellent corrosion resistance [11,12]. Moreover, Ti performs good physicochemical consistency with Al_2O_3 [13], which makes it ideal candidate for the ductile reinforcement. Hence, Ti/Al_2O_3 composites are expected to be promising materials for aerospace, cutter, biology and many other applications owing to the combination of the high toughness of metal and the high strength and hardness of ceramic [14–16]. However, relatively weak interfacial bonding between metal and ceramic leads to the unsatisfied hardness and strength, which limits the application in structural components. Recent investigations demonstrated that the interfacial bonding between Ti and Al_2O_3 was not firm enough, the mechanical properties of the composites were deteriorated due to the inherent brittleness of Ti–Al intermetallic compounds (TiAl and Ti_3Al) which were caused by the strong interfacial reactions between Ti and Al_2O_3 at high temperature [17,18].

Rare earth elements have excellent electronic characteristics and high chemical reactivity, which contributes to their reactions with nearly all the metal elements [19,20]. So far, they have been widely used in metallurgical, glass and ceramics industry, luminescence and laser materials [21–23]. Among them, light rare earth oxide Nd_2O_3 has great industrial value. Feng [24] found that Nd_2O_3 doped on $(BaSr)TiO_3$ ceramics could decrease the grain size and

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increase the density, additionally, lattice parameter was reduced from 0.397 nm to 0.3965 nm on account of lattice distortion.

At present, SiC, ZrB_2, and BN have been widely used to prepare high-temperature ceramics, but their mechanical properties are relatively poor [25,26]. Mehdi reported that ZrB_2–20 vol.% SiC composite sintered at 1850 °C for 60 min under 20 MPa by hot pressing reached the relative density of 99.6%, but the fracture toughness was only 3.8 MPa $m^{1/2}$, which was much lower than those of the cermet composites [27]. However, there are few researches on the oxidation behavior of Al_2O_3-based cermet composites at high temperature.

Generally, sintering method is closely related to the microstructures and properties of the sintered composites. In this experiment, hot pressing sintering with the distinct advantages of low sintering temperature and short sintering time requirement is used to fabricate the Ti/Al_2O_3 composites. Nd_2O_3 powders are used as additives to optimize the interfacial microstructures between Ti and Al_2O_3 and improve the mechanical and oxidation resistance properties of composites. The effects mechanism was also discussed.

2. Experimental procedures

Al_2O_3 (99.8% pure, an average grain size of 1.5 μm, Henan Changxing Co., Ltd, China), Ti (99.6% pure, an average grain size of 27.5 μm, Shanghai ST-Nano Science and Technology, China) and Nd_2O_3 (99.5% pure, an average grain size of 5.6 μm, Sinopharm Chemical Reagent Co., Ltd, China) were used as raw materials in this experiment. Firstly, different volume percentages of Nd_2O_3 (0–5.0 vol.%) powders were added into the Ti/Al_2O_3 mixed powders, then the raw materials were ball milled with absolute ethanol (99.7% pure) in planetary ball mill (XQM-2, China) for 2 h at rotational speed of 200 r/min and ball-to-powder ratio of 2:1, then the slurry was dried in the drying oven (DF102, China) at 50 °C for 3 h. After that, the sintering process was performed in vacuum hot pressing device (VVPgr-80-2300, China) with heating rates of 10 °C/min (0–1200 °C) and 5 °C/min (1200–1420 °C), then temperature was hold at 1420 °C for 1.5 h under pressure of 30 MPa. Previous work from our laboratory [28] has shown that 1420 °C can be the appropriate sintering temperature for the Ti/Al_2O_3 composites and their mechanical properties may reach the optimal level. Lastly, the sintered composites were taken out when cooled naturally in the stove to about 100 °C. Six kinds of samples were sintered, which were labeled as N0, N1, N2, N3, N4 and N5, respectively, their detailed components are listed in Table 1.

The relative densities of the samples were determined by the Archimedes method. The Vickers hardness measurements were conducted on Vickers Hardness Tester (HV-10001S, China) at room temperature with a load of 9.8 N and a dwell time of 15 s. The flexural strength test of the composites was measured using 3 mm × 4 mm × 35 mm specimens by electromechanical universal testing machine (CMT5504, MTS SYSTEMS, China) with a crosshead speed of 0.5 mm/min and span of 30 mm at room temperature. The fracture toughness test was carried by the single edge notched beam (SENB) method using 3 mm × 4 mm × 35 mm specimens with notch depth of 2 mm and notch width of 0.2 mm, similarly, the load was applied with a crosshead speed of 0.5 mm/min and span of 30 mm. According to the previous test [29], oxidation resistance test in this experiment was conducted through static oxidation in the air at 1300 °C for 1 h in a muffle furnace (TCW-32B, China). The phase compositions of the composites before and after oxidation were characterized by X-ray diffraction (D8-ADVANCE, Germany), the microstructure and elements distribution were analyzed by scanning electron microscopy (SEM, FEI QUANTA FEG 250, United States) equipped with an energy dispersive spectroscopy (EDS).

Table 1
The compositions of the samples.

Specimen	Nd_2O_3 (vol%)	Al_2O_3 (vol%)	Ti (vol%)
N0	0	60	40
N1	1	59	40
N2	2	58	40
N3	3	57	40
N4	4	56	40
N5	5	55	40

3. Results and discussions

3.1. Analysis of the phase compositions of the sintered composites before oxidation

The phase compositions of the Ti/Al_2O_3 composites with different volume percentages of Nd_2O_3 additives are characterized by XRD. As revealed in Fig. 1(a), four kinds of phases (Al_2O_3, Ti, TiAl and Ti_3Al) can be identified in the sintered composites without additives, which is in accordance with the report of Wu [30]. According to this report, loose interfacial microstructures caused by the Ti–Al intermetallic compounds (TiAl and Ti_3Al) can be the dominant reason why the mechanical properties of the Ti/Al_2O_3 composites deteriorate. However, when Nd_2O_3 is added, solid solutions of Nd-Al-O ($NdAlO_3$) and Nd-Ti-O ($Nd_2Ti_4O_{11}$) can be detected in the samples (Fig. 1(b)–(e)), which suggests that Nd_2O_3 additives react with Ti and Al_2O_3 to form new phases. Moreover, with the incorporation of the Nd_2O_3, the intensity of the Al_2O_3 peaks decreases obviously, this change is caused by the solid solutions of Nd-Al-O. According to the previous report [31], the formation of the solid solutions could increase the crystal defects and then change the lattice parameter. These phenomena confirm the feasibility that the Nd_2O_3 additives may influence the microstructures and properties of the Ti/Al_2O_3 composites.

3.2. Microstructures of the sintered composites

Typical fracture surfaces of the Ti/Al_2O_3 composites with different volume percentages of Nd_2O_3 additives are observed by SEM and the results are shown in Fig. 2. The interfaces between metal and ceramic are magnified and highlighted in the yellow

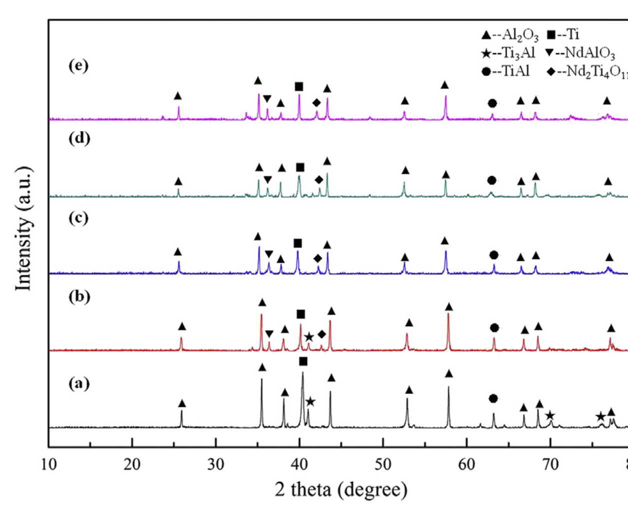

Fig. 1. XRD patterns of Ti/Al_2O_3 composites with different Nd_2O_3 content: (a) 0 vol.%; (b) 1.0 vol.%; (c) 2 vol.%; (d) 3 vol.%; (e) 4 vol.%.