



Microstructure evolution and mechanical properties of Ni₃Al/Al₂O₃ composite during self-propagation high-temperature synthesis and hot extrusion

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ABSTRACT

The Ni₃Al/Al₂O₃ composite was fabricated by self-propagation high-temperature synthesis with and without hot extrusion methods. Its microstructure and mechanical properties were investigated by using combination of optical microscope, transmission electron microscope and compression test. The results show that the Ni₃Al/Al₂O₃ composite without hot extrusion has relative coarse microstructure, which contains γ -Ni and Ni₄Al₃ phases along the Ni₃Al phase boundary. In addition, κ -Al₂O₃, θ -Al₂O₃, α -Al₂O₃ and cavities are observed in the composite without hot extrusion, which segregate greatly in original powder boundary. The hot extrusion process densifies the composite; eliminates the element segregation and redistributes Al₂O₃ particles homogeneously. Moreover fine Ni₃Al crystalline with high density of dislocations and twinned Ni₃Al crystals are observed in the extruded part. The hot extrusion improves the mechanical properties of the Ni₃Al/Al₂O₃ composite significantly, especially its ductility.

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1. Introduction

Ni₃Al intermetallic compounds has received considerable attentions for high-temperature structural and coating applications, such as heat shields for combustion chambers and as first-row vanes in industrial gas turbines, due to its high melting point, low densities, high strength, good corrosion and oxidation resistance [1–4]. In spite of these attractive properties, however, low ductility, brittle fracture, and processing problems were the major disadvantages of this kind of nickel aluminides [3–5]. In order to conquer these problems, many kinds of methods have been adopted and numerous alloys based on Ni₃Al have been developed with broad utilizations ranging from furnace rolls and radiant burner tubes for steel production, and corrosion-resistant parts for chemical industries [3,4]. Former researches [6–8] reveal that the brittleness of Ni₃Al partly stems from an environmental effect caused by hydrogen generated through the reduction of moisture in air by aluminum in the aluminides. A major breakthrough to resolve this issue is the discovery of the dramatic effects of boron addition on ductility improvement for Ni₃Al at ambient and high temperatures. Liu et al. [9] found that 40–50% tensile ductility can be achieved in the Ni₃Al alloy with the addition of small amounts of boron up to

0.4 wt.%. In addition, more investigations [10,11] find that the grains refinement and dispersoid particles are beneficial to the mechanical properties of the Ni₃Al alloy.

Recently, Morsi [1] reviewed a number of novel processes applied to the reaction synthesis of Ni–Al intermetallics. Among them, combustion synthesis with the advantages of time and energy savings has been recognized as a promising alternative to the conventional methods of producing advanced materials, including carbides, borides, nitrides, and intermetallics [12–15]. Lebrat and Varma [16] finds that higher green density and pre-heating temperature can prepared fully reacted product with a well-developed microstructure. Though the synthesis of Ni₃Al has been extensively studied, how to densify the synthesized Ni₃Al without coarsening its microstructure is still a problem. The recent studies [17–19] reveal that the combustion synthesis and hot extrusion can get well-densified materials. Therefore in the present paper, the Ni₃Al/Al₂O₃ composites with trace B addition were fabricated by self-propagating high-temperature synthesis (SHS) with and without hot extrusion, and their microstructure evolution and mechanical properties were investigated.

2. Experimental procedure

The NiO (99.9% purity and particle size of <1.2 μ m), Al (99.5% purity and particle size of 1.45 μ m), Ni (99.5% purity and particle size of 0.98 μ m) and boron powders (99.5% purity and particle size

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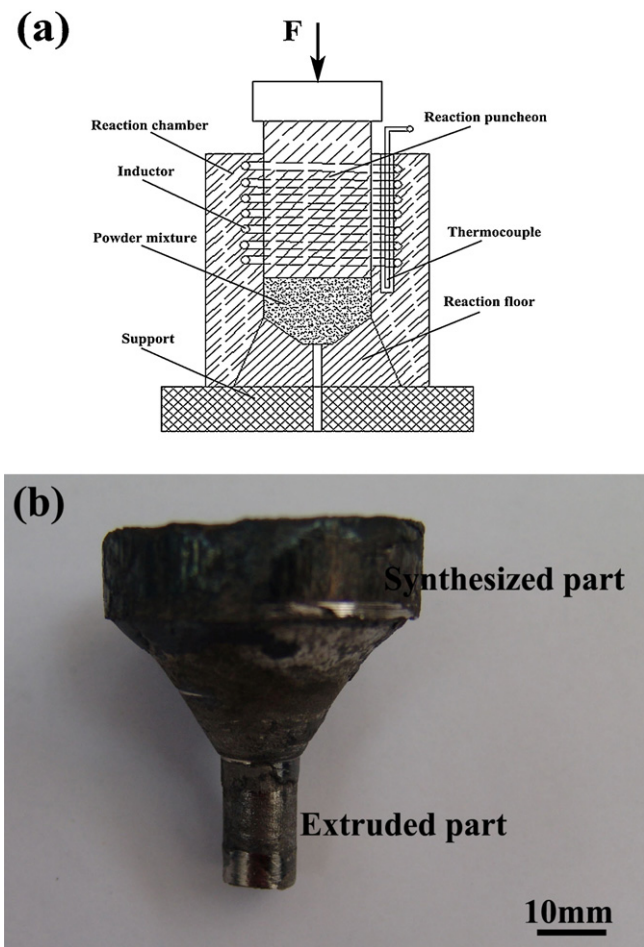


Fig. 1. (a) Schematic diagram of the self-propagation high-temperature synthesis and hot extrusion synthesis system and (b) appearance of the SHS/HE synthesized sample.

of 3 μm) elemental powders were used as starting materials. The main powders (Ni:Al:NiO = 7:3:1 in atomic ratio) and trace B boron powder were dry mixed in a ball mill for 10 h. The mixed powders were compacted into a cylinder with a size of $\text{Ø}40 \text{ mm} \times 40 \text{ mm}$, and then the powder compact was put into the self-propagating high-temperature synthesis with hot extrusion (SHS/HE) synthesis system, as shown in Fig. 1 (a). Firstly, the induction coil heated the reaction puncheon rapidly to 760 K to start the reaction synthesis. A thermocouple in the SHS/HE synthesis system was used to measure the temperature of the powder mixtures. When the temperature increased dramatically, it signaled the start of reaction synthesis. Then, 2 s later, a force of 400 MPa would load on the reaction puncheon in order to extrude the synthesized Ni_3Al out of the reaction floor through a hole with diameter of 6 mm. The as fabricate sample is shown in Fig. 1(b). In order to investigate the effect of the hot extrusion, a sample without hot extrusion is fabricated in the present paper.

The samples for microstructure observation and compression test were cut from $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$ composite prepared by SHS without hot extrusion and different position in the one with hot extrusion. Microstructural characterizations of all samples were carried out on an Axiovert 200 MAT optical microscope (OM). Samples for OM observations were prepared by conventional methods of mechanical polishing and chemical etching with an acidic mixture ($\text{CH}_3\text{COOH}/\text{HNO}_3/\text{HCl} = 8:4:1$). The slices for transmission electron microscope (TEM) observation were cut from all samples by electro-discharge machining (EDM). The slices were mechanically

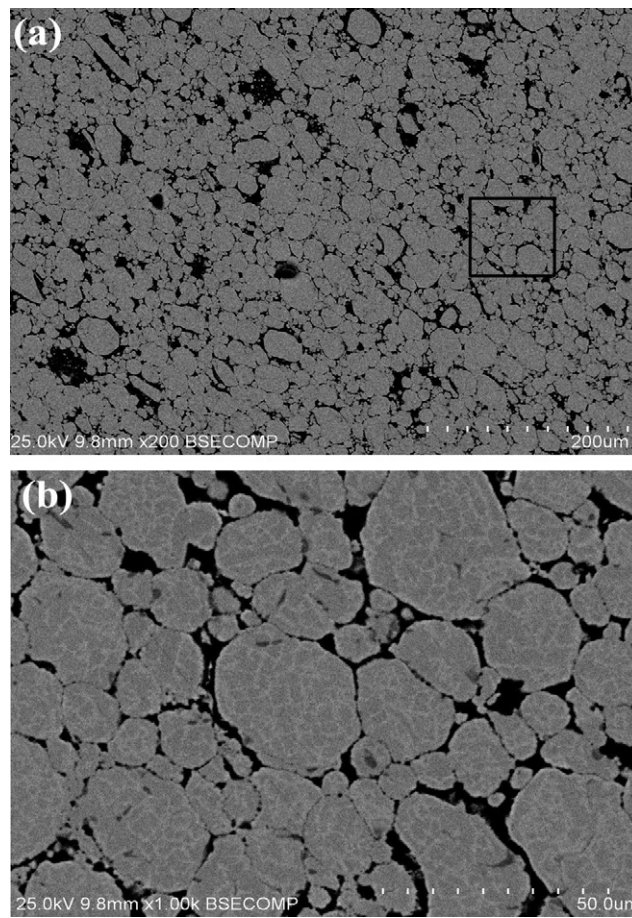


Fig. 2. (a) Backscatter SEM of the synthesized $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$ composite without hot extrusion and (b) microstructure of the square area drawn in line in (a).

grounded from both sides to 30 μm and then thinned by ion milling. The thin foils were observed on a JEOL-2010 high-resolution transmission electron microscope with a point resolution of 0.19 nm and operated at 200 kV.

Due to the small size of the synthesized sample, microhardness and compression test were adopted to evaluate its mechanical properties. Microhardness measurement was carried out on a Vickers microhardness tester (AMH43) using a load of 25 g and a dwell time of 15 s. Seven measurements were performed to obtain an average value. The compressive specimens with size of $\text{Ø}4 \times 6 \text{ mm}^3$ were cut from the different samples and all surfaces were mechanically ground with 600-grit SiC abrasive paper prior to compression test. The compression test was conducted on a Gleeble-1500 test machine at room temperature (RT), with an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$.

3. Results and discussion

3.1. Microstructure characteristic

The typical microstructure of the $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$ composite without hot extrusion is shown in Fig. 2. It can be seen that the elemental powders have been transformed to the $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$ composite, but it still has some original powder characteristics, as shown in Fig. 2(a). The powders gather into big sphere during the milling and the shell of the sphere changes into the boundary during the synthesis. Among the boundaries the black particles segregate greatly, and inside the boundary the gray phase is separated by the white film, as shown in Fig. 2(b). SEM observation shows that the black

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