Composites: Part B 45 (2013) 785–791

Contents lists available at SciVerse ScienceDirect

Composites: Part B

journal homepage: www.elsevier.com/locate/compositesb

Investigation on NiAl–TiC–Al_2O_3 composite prepared by self-propagation high temperature synthesis with hot extrusion

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ARTICLE INFO

Article history:
Received 13 March 2012
Received in revised form 17 May 2012
Accepted 24 May 2012
Available online 4 June 2012

Keywords:
A. Intermetallics
A. Particle-reinforcement
B. Microstructure
B. Mechanical properties

ABSTRACT

The NiAl–TiC–Al_2O_3 in situ composite was fabricated by self-propagation high temperature synthesis and hot extrusion (SHS/HE) technique using element powders. Its microstructure and mechanical properties were investigated by OM, SEM, TEM and compression test. The results revealed that the NiAl–TiC–Al_2O_3 composite was densified by the SHS/HE process and had fine microstructure. In the composite, TiC particles along NiAl grain boundary agglomerated and grew, but the TiC particles in NiAl grain were fine. The TiC and Al_2O_3 particles exhibited an obvious trend to distribute along extrusion direction. Moreover, stacking fault and microtwins in TiC particles and thin amorphous layer along NiAl/TiC phase interface were also observed. In addition, Ti_2AlC particle with intergrowth TiC plate inside formed along the NiAl grain boundary. Generally the SHS/HE synthesized NiAl–TiC–Al_2O_3 composite possessed better mechanical properties, especially at room temperature, which should be ascribed to the fine microstructure and predeformation caused by hot extrusion.

1. Introduction

The NiAl intermetallic compound has been recognized as promising candidate for high temperature structural applications, due to its high melting point, low density and excellent oxidation resistance at high temperature [1–4]. However the use of single-phase NiAl as a structural material is limited because of its lack of ductility at room temperature and low creep resistance at high temperature. Fortunately, previous investigations [5–7] exhibit that the NiAl based materials possess good tribological resistance, especially at high temperature, which makes it promising to use as wear parts. Moreover, recent research [8] also reveals that high strength and good compressive ductility are beneficial to the tribological properties of NiAl. Therefore, it is necessary to improve its high temperature strength and room temperature compressive ductility in order to promote the application of NiAl-based alloy.

In recent decades, great efforts have been done to enhance the mechanical properties of NiAl by solid solution strengthening, precipitation strengthening, dispersion strengthening and second-phase reinforcement [9–14]. Among these methods, self-propagating high temperature synthesis (SHS) is considered as an economic technique to prepared net-shape products. Till now, many kinds of NiAl-based composites with boride, nitride, carbide or oxide inside have been fabricated. For instance, NiAl–TiB_2 [15], NiAl–Al_2O_3 [16] and NiAl–AlN [17] composites have been extensively investigated to incorporate the high strength and stiffness of these ceramics into the NiAl matrix. However, to prepare the densified composite by the common reaction synthesis method is still difficult. The porosity formed during synthesis can destroy the mechanical properties of the composite [18]. Though the subsequent hot isostatic pressing can partly solve the problem, but it results in the microstructure coarseness, which is detrimental to the room temperature mechanical properties. Therefore in the recent studies [19], the hot-press-aided exothermic synthesis (HPES) technique has been developed to fabricate NiAl–TiC intermetallic composites, which can well decrease the porosity. However, the high temperature, long heating time and high pressure limit its application. Morsi et al. [20] develops hot extrusion reaction synthesis (HERS) technique to prepare Ni_3Al intermetallic compound, which can well decrease the porosity and save the energy. Recently, authors' studies [21–23] on the self-propagation high temperature synthesis aided with hot extrusion (SHS/HE) technique finds that the subsequent extrusion procedure can densify the synthesized material without microstructure coarsening. Therefore, in the present paper, the NiAl–TiC composite with dispersed Al_2O_3 oxides is prepared by SHS/HE technique. Microstructure and mechanical properties of the NiAl–TiC–Al_2O_3 in situ composite have been investigated.

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http://dx.doi.org/10.1016/j.compositesb.2012.05.038

2. Experimental procedure

Elemental powders including Ni (98%, <1 μm), Al (98%, <13 μm), Ti (99%, <75 μm), TiO_2 (98%, <10 μm) and C (99.9%, <1 μm) were prepared for NiAl–TiC–Al_2O_3 composite. According to the former research [24], there are three reactions during the synthesis: Ni + Al → NiAl, Ti + C → TiC and $4Al + 3TiO_2 + 3C \rightarrow 2Al_2O_3 + 3TiC$, and the last one has the lowest Gibbs energy, so it will ensure the formation of TiC and Al_2O_3. Therefore, the Al, Ti, TiO_2 and C powders in the stoichiometric mole ratio 4:12:3:15 were mixed and marked A powders. Then Ni and Al powders in the stoichiometric mole ratio 1:1 were mixed together with 12 vol% of TiC (1:1 mol ratio) and 3 vol% A powders. These powders were blended with stainless steel ball in a plastic jar for 15 h to obtain a homogeneous mixture. After then the mixed powders were put into the SHS/HE system, as shown in Fig. 1a. Firstly, the powder mixtures were pressed into compact with diameter of 4 cm at a pressure of 40 MPa and then degassed by vacuum; subsequently a pressure of 120 MPa is exerted on the puncheon to densify the compact further. Secondly, the inductor coils heated reaction puncheon to 800 K to start the reaction synthesis. The temperature of the compact was measured by a thermocouple in the SHS/HE synthesis system. When the reaction synthesis began, the temperature of the powder would increase dramatically. Then, 2 s later, a force of 500 MPa was imposed on the reaction puncheon in order to extrude the synthesized alloy out from the reaction floor through a hole with diameter of 6 mm.

The samples for microstructure observation and compression test were cut from the synthesized NiAl–TiC–Al_2O_3 composite by electro-discharge machining (EDM). The resultant phases in the composite were characterized by X-ray diffraction (XRD) with a Cu radiation at 40 kV and 40 mA. Microstructural characterization of sample was carried out on an OLYMPUS GX41 optical microscope (OM) and S-3400 scanning electron microscope (SEM). Samples for OM observations were prepared by conventional methods of mechanical polishing and chemical etching with an acidic mixture ($CH_3COOH/HNO_3/HCl$ = 8:4:1). The foils for transmission electron microscope (TEM) observation were cut from the center of the composite by EDM. The foils were mechanically ground from both sides to 30 μm and then thinned by ion milling. The thin foils were examined using a JEOL-2010 high-resolution transmission electron microscope with a point resolution of 0.19 nm and operated at 200 kV.

The surfaces of compressive specimens with size of $4 \times 4 \times 6\ mm^3$ were mechanically ground with 600-grit SiC abrasive paper prior to compression test. The compression was conducted on a Gleeble-1500 test machine at room temperature and 1273 K, with an initial strain rate of $2 \times 10^{-3}\ s^{-1}$.

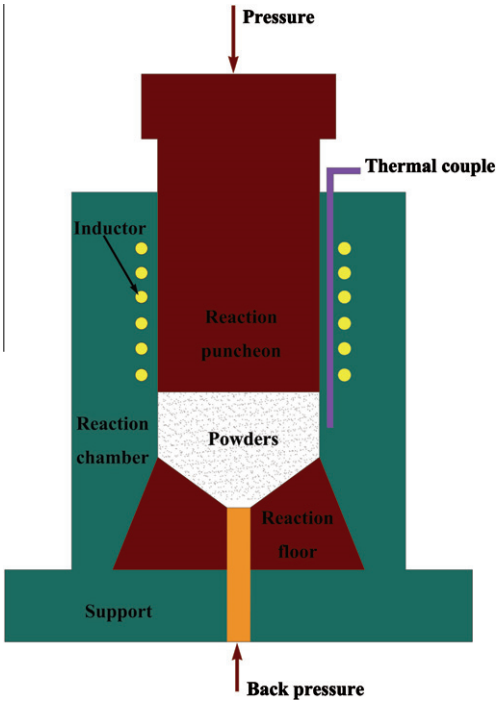

Fig. 1. Schematic diagram of the SHS/HE synthesis system.

3. Results and discussion

3.1. Microstructure characteristics

The NiAl–TiC composite with dispersed Al_2O_3 oxides was successfully fabricated by SHS/HE process. The X-ray diffraction analysis on the composite proves that the elemental powders have been transformed to the NiAl and TiC phases, as shown in Fig. 2a. The peaks of NiAl matrix and TiC phase are strong, but the peak of Al_2O_3 is weak, which should be attributed to its small amount. The typical microstructure of NiAl–TiC–Al_2O_3 composite is shown in Fig. 2b. From the OM micrograph, it can be seen that the composite is unsatisfied, and there are still some porosity inside. The enlarged image shows that the composite is mainly composed of NiAl Matrix and white TiC, as shown in Fig. 2c. The average grain size of the NiAl matrix is about 10 μm. Additionally, the NiAl phase is elongated along the extrusion direction, which indicates that the composite experiences deformation during hot extrusion. The TiC particles with several microns mainly distributed along the NiAl grain boundaries, which is beneficial to the grain refinement. The SEM observation on the cavity is shown in Fig. 2d. Obviously, the surface of cavity is full of fine particles, which forms the shell of the cavity. The EDS tests on the particles reveal that most of them are TiC and some are fine Al_2O_3. As is well known, the TiC and Al_2O_3 ceramic particles have high strength and high melting point. In the present research, the self-propagation high temperature synthesis procedure cannot generate enough heat to soften the ceramic particles. Therefore, if the synthesized TiC and Al_2O_3 particles are segregated, they may form the stiff shell of cavities. During the self-propagation high temperature synthesis, the synthesized NiAl is heated to be semisolid and the extrusion force the NiAl to deformation, which can promote the TiC and Al_2O_3 particles to redistribute and then reduce the amount of cavity. But for the small cavity, its shell constituted by TiC and Al_2O_3 particles can withstand great pressure, so it is very difficult to eliminate.

The TEM observation on the NiAl–TiC–Al_2O_3 composite is shown in Fig. 3. The NiAl matrix has dual-grain structure, as shown in Fig. 3a. Besides the large NiAl grain shown in Fig. 2c, there are much fine grains with hundreds of nanometers. The TiC particles exhibit two kinds of morphologies. The TiC particles along the NiAl grain boundary agglomerate into big ones, as shown in Fig. 2c. The TiC particle in NiAl grain exhibits fine and polyhedron characteristics, as shown in Fig. 3b. The inset selected area electron diffraction (SAED) pattern gives that TiC particle has an orientation relationship with NiAl matrix of $[100]_{TiC}//[100]_{NiAl}, (020)_{TiC}//(0\bar{1}1)_{NiAl}$, which was reported in the former research [14]. However, further observation on NiAl and TiC interface finds that an amorphous transition layer with several nanometers exists between NiAl and TiC, as shown in Fig. 3c. According to the previous researches [25,26], the formation of amorphous layer should be attributed to the interface stress between NiAl and TiC phases. Though NiAl and TiC phases are both have the cubic crystal structure, however the crystal parameter of NiAl is 0.2887 nm, while the crystal