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Fabrication of oxide-reinforced Ni_3Al composites by mechanical alloying and spark plasma sintering

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Abstract

Oxide-dispersion-strengthened Ni_3Al composites have been fabricated by mechanical alloying and subsequently spark plasma sintering with elemental powder. A solution Ni(Al) has been obtained during mechanical alloying. In addition, element O is introduced by mechanical alloying. Composites have a nearly full density after sintered at 1150 °C for 5 min under the pressure of 40 MPa. About 3 vol.% oxides disperse in the matrix uniformly. The average size of oxide is about 50 nm. Oxide-dispersion-strengthened Ni_3Al composites exhibit high bending strength (about 2450 MPa) due to results of uniformly dispersion of the oxide and the refinement of the grain size.

Keywords: Nickel aluminides; Dispersion strengthening; Mechanical alloying; Spark plasma sintering

1. Introduction

The intermetallic compound Ni_3Al has attractive characteristics of high strength at elevated temperature, low density, good corrosion resistance and oxidation resistance. But its brittleness at room temperature and low creep strength limit its application. The addition of alloying elements has been confirmed to be an effective method to improve the ductility of Ni_3Al [1–3]. And the method of dispersing second phase particles (such as oxides) into the matrix has been employed to enhance its properties to realize the structural application [4].

Particles reinforced Ni_3Al composites have been fabricated using variety of techniques including powder metallurgy [5], casting [6], metal infiltration [7], spray forming and coinjection [8]. From all these techniques, powder metallurgy such as hot isostatic pressing and hot extrusion has been deeply studied due to its advantages of precise control composition and easy realization of complex part shapes [9,10]. Mechanical alloying is one of the early successful processes to produce various oxide-dispersion-strengthened (ODS) alloys [11,12]. During this process, serve plastic deformation and interaction between powders make particles distribute uniformly in mixtures and enlarge the surface area and reaction activation of powder. Spark plasma sintering (SPS), as a new powder metallurgy technique, has advantages of short sintering time and low sintering temperature [13]. It is probable to realize fine ODS Ni_3Al with excellent microstructure and properties by combining mechanical alloying and spark plasma sintering.

So far, ODS Ni_3Al composites with refractory oxide (Al_2O_3, Y_2O_3) particles have been prepared [14], however, almost all of the processes need to add oxide as the raw reinforced material. Therefore, in this paper we use a new developed powder metallurgy process, which combines mechanical alloying with spark plasma sintering (SPS), to prepare oxide reinforced Ni_3Al composites by in situ reaction synthesis without the addition of original oxide. The microstructure and properties of oxide reinforced Ni_3Al composites fabricated by mechanical alloying and spark plasma sintering have been preliminarily investigated.

2. Experimental

Nickel powder (99.5%, <74 μm), aluminum powder (99.5%, <74 μm) and boron powder (98%, 3–5 μm) were mixed in the molar proportion of 76.6:22.9:0.5. The elemental powder mixtures were put into steel vial with bearing steel balls. Ball-to-powder weight ratio was 10:1. Mechanical alloying was performed by using vibratory mill for 1 h in air. The vibration frequency of ball mill was 1400 rpm. To prevent excessive welding in the bowl, petroleum ether was added to mixture as process

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control agent (PCA). The powders were put in a graphite die ($\Phi = 30$ mm) and sintered using SPS 1050 (Sumitomo Coal Mining Co., Japan). The sintering temperature was 1150 °C with a holding time of 5 min by heating rate of 100 °C/min. The sintered pressure was 40 MPa.

Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) were employed to investigate microstructure of the milled powder and the sintered body. After ultrasonic treatment, powder sample was fetched by micro grid for TEM observation. For TEM investigation thin slices (500 μm thickness) were spark cut from compacts. Then foils were prepared by mechanical grinding of slices to a final thickness of 50 μm and double jet electro polishing using a mixture of 90% CH_3OH and 10% H_2SO_4. The bath temperature was 25 °C and the voltage was 15 V. The X-ray diffraction patterns of the samples were carried out with a Rigaku Dmax-RC diffractometer in the θ–2θ geometry using Cu Kα radiation. The densities were determined by Archimedes method. Bending samples were spark cut from compacts into 3 mm × 3 mm × 18 mm and tested at a strain rate of 0.01 s^{-1} at room temperature to examine their mechanical properties.

3. Results and discussion

3.1. Analysis of milled powder

Fig. 1 shows SEM image of milled powder. Compared with original powder that has an average size of 67 μm, the milled powder is finer, which is about 25 μm. The particles in milled powder are observed to be multi-layer structures, and single particle is composed of Ni and Al from the results of the EDS. Fig. 2 shows TEM image of milled powder. The multi-layer structure can be observed clearly, as pointed by arrow in Fig. 2. Selected area diffraction (SAD) patterns reveal that the particles grain is fine due to the appearance of diffraction ring to the selected area. According to the diffraction ring its structure is approximately agreed with face-centered structure of Ni by calibration. The average lattice parameter of powder is 3.60 Å, which is larger than that of pure Ni. So it reveals that some Al atoms have dissolved in lattice of Ni. X-ray diffraction pattern of milled powder is also presented in Fig. 3. The peaks of Ni and Al appear, and the peaks of Ni shift to low angle slightly, which confirms that some Al atoms have dissolved in Ni. Since superlattice lines are

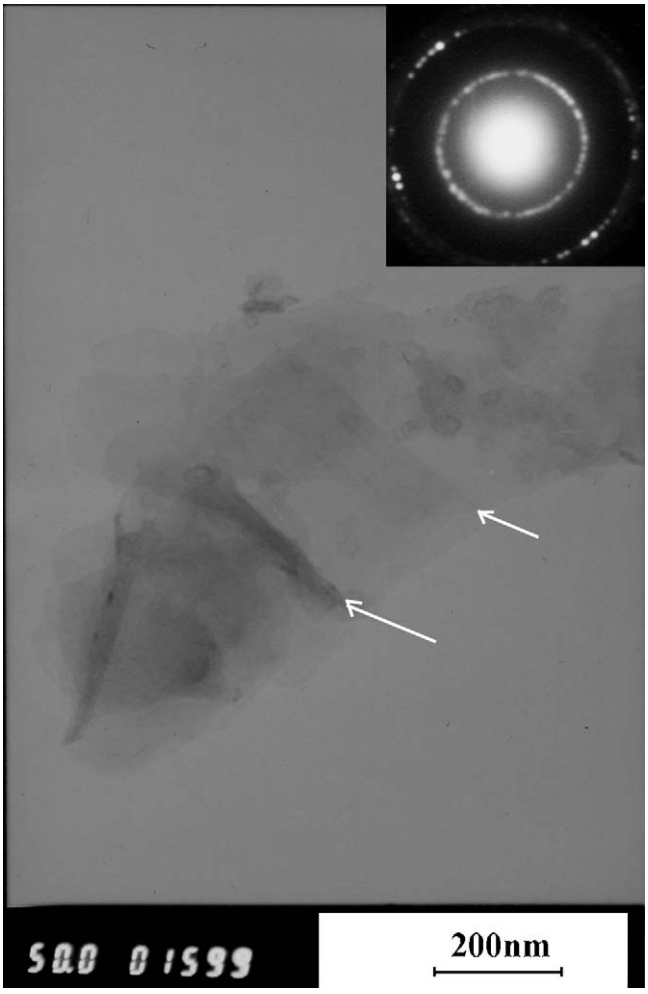

Fig. 2. TEM image of milled powder.

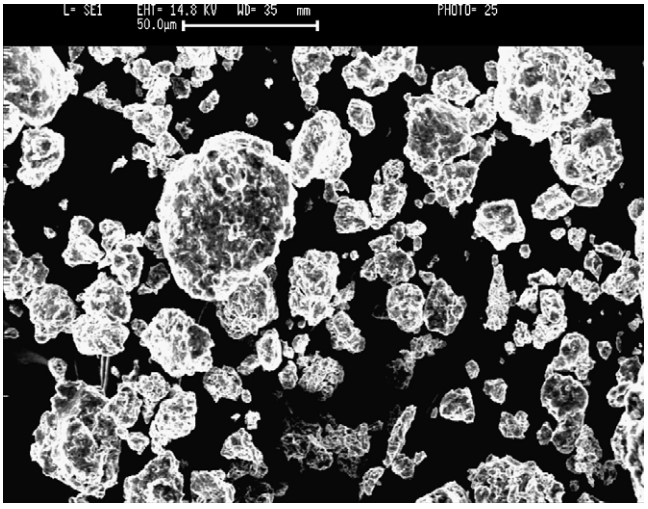

Fig. 1. SEM micrograph of milled powder.

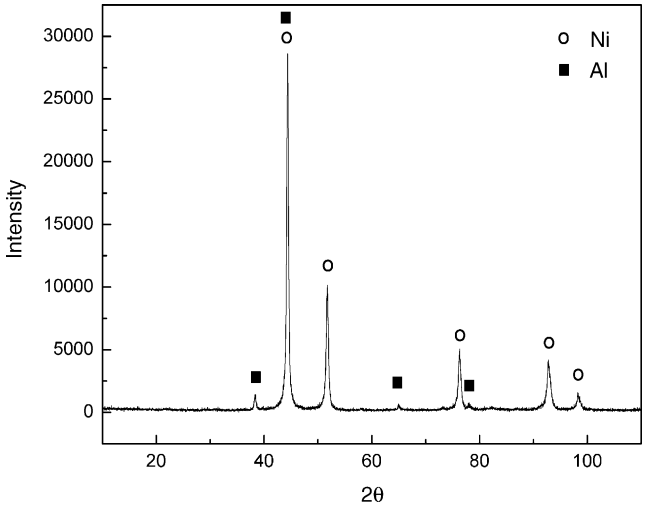

Fig. 3. X-ray diffraction pattern of milled powder.