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Original Research Paper The synthesis and characterization of ultrafine grain NiAl intermetallic

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1. Introduction

The NiAl intermetallic is considered as a potential high-temperature structural material due to its attractive physical and mechanical properties, such as high melting point (1638 °C), low density (5.86 g/cm³), high thermal conductivity (2–4 times higher than conventional nickel-based alloys) and excellent oxidation resistance [1–3]. However, its practical application is restricted due to its inherent poor ductility at room temperature [4]. In order to overcome this drawback, considerable attempts have been carried out, such as thermomechanical processing, micro-alloying and macro-alloying [5–9]. Additionally, the preparation of fine-grained material is also a feasible approach to improve the room-temperature ductility, since brittle material may be transformed into ductile material by grain refinement [10,11].

MA is a viable method to prepare fine-grained materials. Especially, it has the advantages in fabricating compounds which are difficult to be prepared by conventional processes because of high vapor pressure and/or the large difference in melting points of components [12]. Fine-grained NiAl intermetallic has been previously prepared through MA route by several researchers [13]. However, the results of mechanical tests revealed that, due to the existence of Al_2O_3 and the lower relative density which were caused by the preparation process, there was a large deviation between experimental result and theoretical value.

In this study, in order to prevent potential oxidation, MA and hot-pressed sintering were conducted in a protective atmosphere

ABSTRACT

The nanocrystalline NiAl powders were synthesized by mechanical alloying (MA), and the ultrafine grain NiAl bulk materials were subsequently consolidated by vacuum hot-pressed sintering. The microstructure and mechanical properties of milled powders and bulk materials were characterized. The results reveal that the NiAl powders were synthesized after 1.67 h of milling and the grains of NiAl were refined to 18 nm after 22 h of milling. During milling, the temperature rise caused by MA led to the annealing effect and consequently resulted in the abnormal decrease in microstrain and microhardness. NiAl bulk material with a relative density of 99.4% was prepared after sintering at 1300 °C and its grain size was about 400 nm. Due to fine-grain strengthening, the compressive stress and compressive strain of NiAl bulk material were significantly improved at room temperature.

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and vacuum respectively. Dense and ultrafine-grained NiAl alloys were obtained by MA and subsequent sintering. The microstructures of milled powders and NiAl bulk materials were characterized. The mechanical behaviors of bulk materials were evaluated.

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2. Experimental details

The Ni (99.2 at.%, $25 \,\mu$ m) and Al (99.4 at.%, $15 \,\mu$ m) powders were used as original materials and their scanning electron microscopy (SEM) morphologies are shown in (Fig. 1).

MA was carried out in a homemade MI-1 stirring ball milling. Its schedule is presented in (Fig. 2). Reactant powders were mixed in a mole ratio of 1:1 (Ni:Al), then the powders and milling balls $(\Phi 8 \text{ mm})$ were sealed in the milling vial in a glove box under argon atmosphere. Prior to milling, the milling vial was vacuumed by a vacuum pump and then filled with argon gas. This process repeated for five times in order to prevent the powders from oxidation. Both the milling balls and milling vial are made of GCr15 steel. The filling fraction of milling vial was about one third. During milling, the ball-to-powder weight ratio and the rotation speed were maintained at 10:1 and 300 rpm, respectively. The milling vial was cooled by a circulating cooling system and the temperature of cooling water was about 22 °C. Throughout the process, milling and pause were carried out alternately every one third hour. The samples of milled powders were taken out periodically for morphology observations. After milling, the milled powders were canned and then kept in a glove box under argon atmosphere in order to minimize potential oxidation. Milled powders were consolidated in the ZRY55 hot-pressed sintering furnace at 60 MPa in vacuum ($\sim 1.3 \times 10^{-2}$ Pa). The sintering was conducted



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