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Synthesis, microstructure and mechanical properties of Al_2O_3 reinforced Ni_3Al matrix composite

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ABSTRACT

A new method to synthesize alumina reinforced Ni_3Al intermetallic matrix composites has been described. The powder mixture of nickel and aluminium was mechanically alloyed. The powder mixture was excessively heated during mechanical alloying and then exposed to atmosphere for oxidation. The oxidized powder mixture was transformed into alumina reinforced nickel aluminide matrix composite on subsequent pulse current processing. Alumina reinforcements were generated in the nickel aluminide matrix by in situ precipitation. The microstructure of the composite showed that the alumina reinforcements were 50–150 nm in size. The fine alumina reinforcements were homogeneously distributed in the matrix phase. The mechanical properties of the alumina reinforced nickel aluminide matrix composite fairly exceeded the nickel aluminide alloys. This novel synthesis approach allowed the rapid and facile production of high strength alumina reinforced Ni_3Al matrix composites.

1. Introduction

Ni_3Al alloys are important candidates for structural applications due to their strength at high temperature, low density, corrosion and oxidation resistance [1–3]. The brittleness at room temperature and low creep resistance of Ni_3Al limit its application. There are two methods to improve the mechanical properties of Ni_3Al (a) addition of alloying elements [4–6] and (b) dispersion of second phase particles [7–9]. Dispersion strengthening by second phase, such as carbides and oxides, into the Ni_3Al matrix has been employed to materialize their structural applications [7–11]. Mechanical properties of intermetallic matrix composites are strongly affected by the properties of interface, which are influenced by the interface structure and the chemical stability between the components during the production process. Al_2O_3/Ni_3Al couple exhibits very good chemical stability at elevated temperatures both in vacuum and protective atmospheres provided that the Al_2O_3 reinforcements are of high purity [9–13].

Oxide dispersion strengthened alloys exhibit superior mechanical properties at room and high temperature [14,15]. Recently, much work has been done on ODS Fe-base alloys, ODS FeAl alloys and ODS tungsten heavy alloys [14–18]. Mechanical alloying is one of the processes to synthesize ODS alloys [19–21]. It involves repeated cold welding, fracturing and rewelding of the powder particles. Intensive plastic deformation of the powder particles leads to the creation of variety of crystal defects, which act as short circuit diffusion paths for alloying to occur. Moreover, the rise in temperature due to ball to ball, ball to powder and ball to wall collisions during mechanical milling aids the alloying process [22,23]. Pulse current processing, a new consolidating technique of powder metallurgy, has advantages of low sintering temperature and short sintering time [24–26]. It provides rapid and facile approach to sinter bulk materials. It is feasible to synthesize ODS Ni_3Al alloys with fine oxide reinforcements and superior mechanical properties by combining mechanical alloying and pulse current processing.

So far, Al_2O_3 and Y_2O_3 particulate reinforced Ni_3Al matrix composites are prepared by ex situ method [27,28] whereas in this study mechanical alloying is used to excessively heat the Ni and Al powders to prepare oxidized powder mixture of Ni and Al. Subsequent pulse current processing of these oxidized powders produces in situ Al_2O_3 reinforcements in Ni_3Al matrix. In situ synthesis gives advantages of fine size, high purity and uniform dispersion of reinforcements in the matrix phase [29,30]. The microstructure and mechanical properties of oxide reinforced Ni_3Al composites fabricated by excessive heating of powders by mechanical alloying and pulse current processing are preliminary investigated.

2. Experimental

The raw materials were nickel powders (99.5% purity, <74 μm), aluminium powders (99.5% purity, <74 μm) and boron powders (98% purity, 3–5 μm). First Ni, Al and B were mixed in a molar ratio

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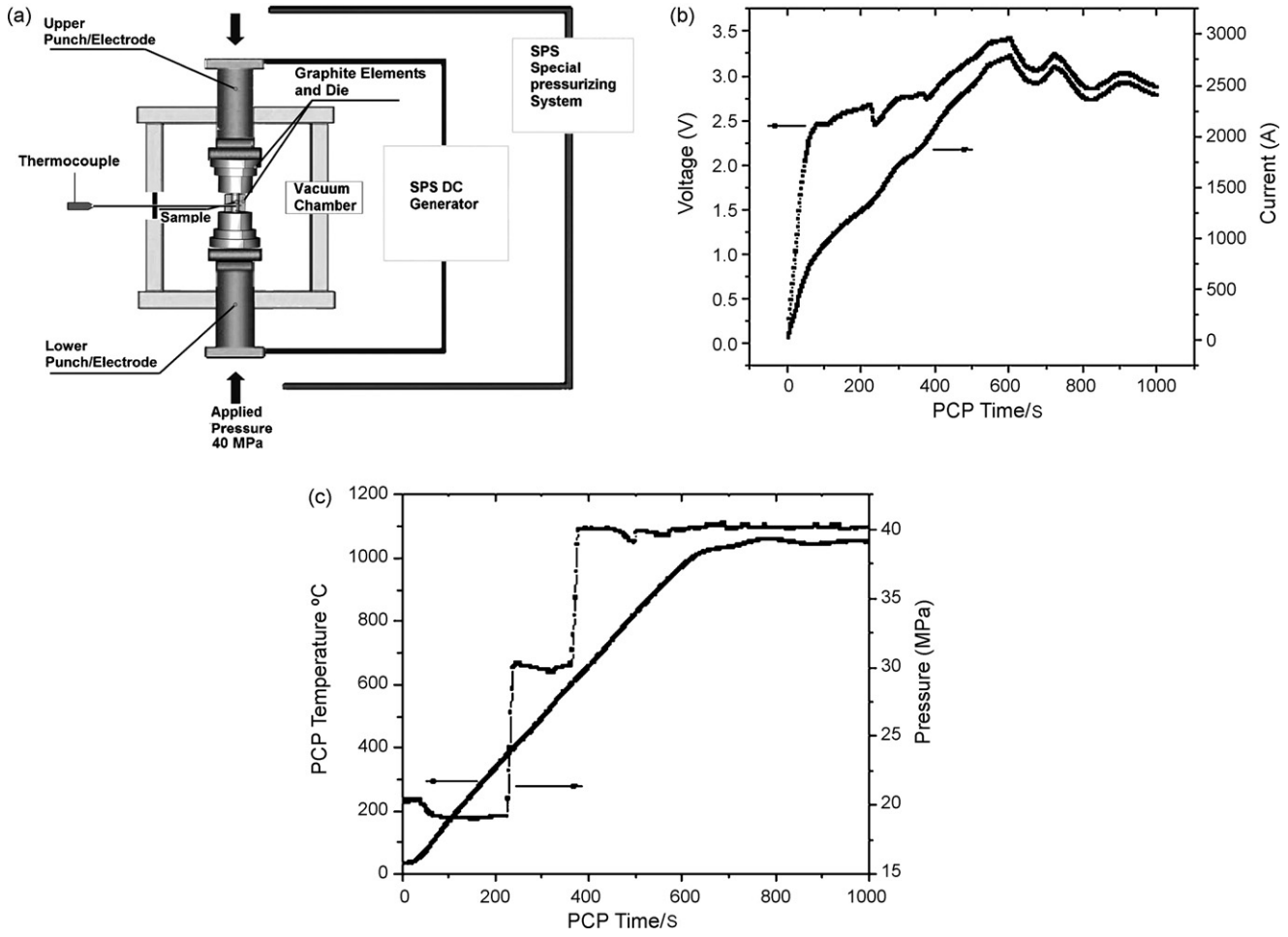

Fig. 1. (a) Schematic representation of Spark Plasma Sintering machine and (b, c) pulse current processing parameters to synthesize Al_2O_3 reinforced Ni_3Al matrix composites.

of 76.5:23.0:0.5, respectively to produce oxidized composite powders. The powder mixture was mechanically alloyed for 15–30 min. A high-energy vibratory type-milling machine with three vials was employed for the mechanical alloying experiments. The frequency of vibration of the machine was 1400 rpm. The vials were sealed with a rubber O-ring and thus the milling proceeded in a stationary atmosphere. For each milling run, powder mixture was canned into a stainless steel vials containing bearing steel balls in air. The weight ratio of the balls to the powder mixture was 20:1. The high ball to powder ratio was adopted in order to increase the stored energy in the powder mixture. The extent of filling the vial with the powder mixture and the balls was about 50%. The milling program was set for 15–30 min continuous milling for excessively heating the powder mixture. The maximum milling time was 30 min. The structure of the mechanical alloyed powders was analyzed by Rigaku Dmax-RB X-ray diffractometer (XRD) using Cu target (Kα, λ = 0.15406 nm) radiation at 40 kV and 200 mA settings.

As soon as mechanical alloying time elapsed, the powder mixture was exposed to air. The excessively heated powder mixture was ignited on exposure to air. Excess air was flown on the powder mixture for a homogeneous and complete ignition of the powder mixture. The powder mixture was burnt in the air to red-hot. After completion of the reaction it was cooled down to room temperature. Oxygen from the air was picked by the powder mixture. The powder samples were prepared several times following the identical processing to verify the reproducibility of the results. The oxygen content of raw powder mixture and oxidized powder mixture was measured by the wet chemical analysis. Oxidized powder mixture was poured into a graphite die of 30 mm diameter and sintered using SPS 1050 (Sumitomo Coal Mining Co., Japan) machine. Fig. 1 shows the schematic of Spark Plasma Sintering machine and summarizes the pulse current processing parameters. The sintering temperature was 1050 °C with a holding time of 5 min. The heating rate was 100 °C/min. The pressure applied during consolidation was 40 MPa. The hardness tests were performed on polished surfaces of the sintered compacts using a shimadzu vicker hardness tester. Bend strength tests were conducted on an Instron mechanical tester. Samples for bend strength measurement were machined from the sintered compacts in cuboids and polished on SiC emery papers up to 1000#. At least, four tests for each composition were conducted under the same conditions. Samples for scanning electron microscopy were cut from the sintered samples. The samples were polished to 0.5 μm diamond finish. The microstructure was characterized by Kevex Leo-1450 scanning electron microscope. For TEM study, 500 μm thin slices were spark cut from sintered compacts. Then, foils were prepared by mechanical grinding of slices to a final thickness of 50 μm. Double jet electro polishing, using a mixture of 90% CH_3OH and 10% H_2SO_4, was used for further thinning of samples. The bath temperature was 25 °C and the voltage was 15 V. The microstructure was characterized on a H800 transmission electron microscope.

3. Results and discussion

Fig. 2 presents the X-ray diffraction patterns of the powder mixture mechanically alloyed for different times. A broad diffuse