



Tribological properties of Ni₃Al produced by pressure-assisted volume combustion synthesis

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ABSTRACT

The production of Ni₃Al was performed under an uniaxial pressure of 150 MPa at 1050 °C for 1 h. The formation temperature of Ni₃Al was determined to be 655 °C. The presence of Ni₃Al was confirmed by XRD analysis. SEM analysis revealed that the Ni₃Al phase has very low porosity. The relative density and microhardness of test materials were 97.8% and about 359 ± 31 HV_{1.0}, respectively. The specific wear rate of Ni₃Al was 0.029 mm³/N m for 2 N, 0.017 mm³/N m for 5 N and 0.011 mm³/N m for 10 N, respectively. The distribution of alloying elements was determined by energy-dispersive spectroscopy (EDS).

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1. Introduction

The Ni–Al binary phase diagram contains five intermetallic compounds (Al₃Ni, Al₃Ni₂, Al₃Ni₅, NiAl, and Ni₃Al). Of these intermetallics NiAl and Ni₃Al have by far received the majority of scientific attention as they are considered candidate materials for high temperature structural and coating applications because of their high melting points and relatively low densities high strength, as well as good corrosion and oxidation resistance [1–4]. Ni₃Al or the γ' -phase is also a strengthening constituent in nickel-base superalloys used in high temperature applications. The difficulty with the brittleness of polycrystalline Ni₃Al is generally overcome by microalloying small contents of boron [5]. The major advantages from the use of Ni₃Al include resistance to oxidation and carburization up to 1100 °C, good tensile and compression yield strengths at 650–1100 °C, superior creep strength and fatigue resistance, excellent wear resistance at high temperature, etc. [3]. In general, conventional processing techniques such as melting and casting methods are inapplicable to the fabrication of many intermetallic alloys. Combustion synthesis with the advantages of time and energy savings has been recognized as an attractive alternative to the conventional method of producing advanced materials [3,6]. This synthesis is concerned with the ignition of a compressed powder mixture, producing a chemical reaction with sufficient heat release (exothermic reaction) that it becomes self-sustaining. There are two basic modes of

combustion reactions: “propagating” and “bulk” reactions [7,8]. In this method, the samples are heated by an external source (e.g., Tungsten coil, laser) either locally (self propagating high temperature synthesis, SHS) or uniformly (bulk way) (volume combustion synthesis, VCS) to initiate an exothermic reaction. The VCS is more appropriate for weakly exothermic reactions that require preheating prior to ignition [9].

The main aim of the present study is to investigate tribological properties of single Ni₃Al intermetallic compound manufactured by pressure assisted volume combustion synthesis in one step without using any inert atmosphere.

2. Materials and methods

2.1. Material processing and characterization

In this study, the pressure assisted volume combustion synthesis method was utilized in order to produce the Ni₃Al intermetallic compound. Nickel (99.8% purity, particle size 3–7 μ m) and Al (99% purity, particle size < 15 μ m) powders at required stoichiometry (3:1 Ni to Al molar ratio) were mixed using a small ball mill containing Ni balls at a speed of 150 cycle/min for 10 min under Ar+3%H₂ gas medium with the addition of a small amount of ethanol (0.1%). The powder mixture was cold pressed into a cylindrical compact in a metal mold which was coated with a thin layer of boron nitride by applying a uniaxial pressure of 150 MPa. The pressed compact was inserted in an open air furnace and was heated to 1050 °C at a heating rate of 20 °C min⁻¹ and was kept at a particular temperature for 60 min under a uniaxial pressure of

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150 MPa without using vacuum or inert gas. Then, the samples were removed from the furnace at 900 °C in normal atmosphere and were cooled to room temperature. The diameter and height of the sintered samples were 15 mm and 5 mm, respectively.

Thermal analysis was performed by differential scanning calorimeter (DSC) in nitrogen atmosphere and at a heating rate of 20 °C min⁻¹ in order to determine the reaction sequence and formation temperature. X-ray diffraction analysis (XRD, D-2200 Rigaku and Cu K α radiation) was used to determine the Ni₃Al phase. For estimating the relative density together with the amount of closed and open pores, Archimedes' method with balance sensitivity of 0.0001 g was employed. The produced samples were polished and etched using HNO₃ (33.3 vol%), CH₃COOH (33.3 vol%) and HCl (33.3 vol%). The morphologies of the samples were studied by using scanning electron microscopy (SEM, JOEL JSM-6600). Micro-hardness measurement was performed on synthesized sample using a Vickers indentation technique (FUTURE TECH. FM 700) at an applied load of 10 N with a dwelling time of 15 s.

2.2. Wear test

In order to determine friction and wear performance of test materials a ball-on-disk type tribometer according to the G66 ASTM standard test method was used. The counterface material was made of alumina ball, 9.5 mm in diameter, with a highly polished surface finish of better than 0.04 μ m CLA roughness. Ni₃Al intermetallic disks were run against the ball under the loads of 2, 5 and 10 N, with sliding velocity of 0.1 m/s. The ball was firmly fixed to a stationary holder for the ball-on-disk configuration. The disk was attached to a horizontal chuck driven by a variable-speed electric motor. The frictional force, monitored by a load cell attached to the ball holder, was recorded continuously. The friction coefficient was measured when it reached steady-state. The maximum compressive contact pressure at central point of the contact area was calculated from the Hertzian equation [10]. According to the Hertzian equation, maximum contact pressures of 0.68, 0.93 and 1.17 GPa (for disk Ni₃Al $E=178$ GPa, $\nu=0.35$, for Al₂O₃ ball $E=420$ GPa, $\nu=0.24$ [11,12]) were obtained at normal loads of 2, 5 and 10 N, respectively. Wear rate was measured primarily by volume loss. Volumetric wear of specimens was determined by measuring the diameter of the wear track and calculated by means of volume loss of the specimens. Wear rates of samples were calculated using Eq. (1). Width of wear scar was measured by optical microscopy.

$$W = \frac{2\pi(R+(L/2))(r^2/2)(\theta - \sin \theta)}{\text{sliding distance}} \quad (1)$$

where W is the wear rate (mm³/m), L is wear track width (mm), R is radius of the wear scar (mm), r is radius of ball (mm) and $\theta=2\arcsin(L/2r)$ (rad).

3. Results

3.1. Material characterization

Differential scanning calorimeter analysis revealed that the Ni₃Al intermetallic compound formed at temperature 655 °C which corresponds to an exothermic reaction (Fig. 1) confirmed by means of the x-ray diffraction (XRD) technique (Fig. 2). As it can be seen in Fig. 1, there is an exothermic peak at the temperature of 655 °C and an endothermic peak at the temperature of 659 °C. The formation mechanism of intermetallic nickel aluminides strongly depends on heating rate. Several exothermic and endothermic peaks were observed at lower heating rates (5–20 °C/min⁻¹) that result in a

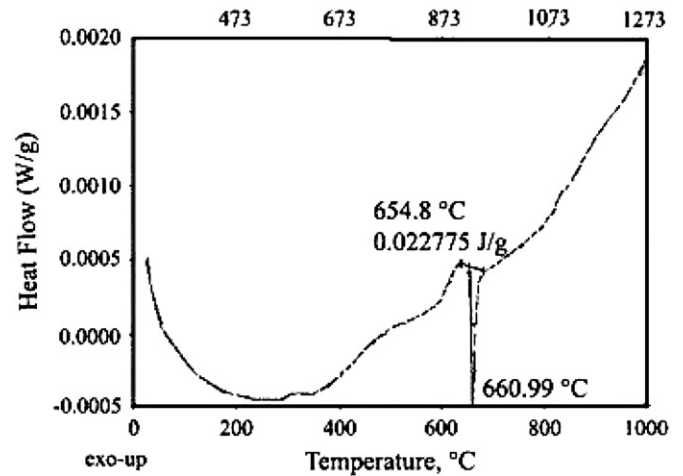


Fig. 1. DSC analysis of Ni–Al powder mixed in a molar proportion of 3:1.

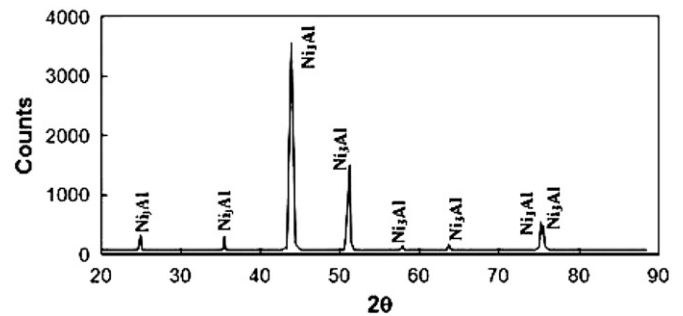


Fig. 2. X-ray diffraction patterns of Ni₃Al processed at 1050 °C for 1 h.

solid-state pre-combustion reaction (two-step reaction) between Ni and Al as well as intermediate reactions in the process of formation of nickel aluminides such as NiAl₃ and Ni₂Al₃ forming prior to the final products. However, both slow heating and effective heat transfer from the sample to the massive metal mold prohibit the reaction from becoming self-sustaining and allowed the reaction to remain solid-state diffusion-controlled. The heat loss resulting from the heat exchange between the sample and metal mold may suppress the combustion reaction [12]. The reaction is completed at 1050 °C in 1 h and samples having a uniform Ni₃Al structure with small amount of porosity were produced by the pressure assisted combustion synthesis method (Fig. 3).

The hardness of test materials was measured by using the Vickers indentation technique with a load of 10 N. The hardness of Ni₃Al intermetallic was about 359 ± 31 HV_{1.0}; the data are an average of at least five points for each sample.

3.2. Friction and wear

Wear tests applied on the test materials produced by pressure-assisted combustion synthesis revealed that the friction coefficient varied between 0.5 and 0.7 for long-duration testing. Fig. 4(a)–(c) shows the variation of friction coefficient of Ni₃Al intermetallic as a function of cycle number for a load of 2 N, 5 N and 10 N. Friction coefficient initially increases and then reaches a steady-state level. Fig. 5 reveals the variation of friction coefficients as a function of applied load. As can be seen, initially friction coefficient of Ni₃Al increases and then decreases with increase in load.

The wear rate of Ni₃Al sliding against alumina ball ranged from 0.056 mm³/m to 0.114 mm³/m, depending on applied load as shown in Fig. 5. It is clear that wear rate of Ni₃Al increases with

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