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Influence of process control agents on the performance of Ni₃Al by mechanical alloying

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Abstract

The influence of process control agents (PCAs) on the mechanical properties of Ni₃Al intermetallic compounds by mechanical alloying was investigated in order to develop oxide deposition reinforced intermetallics. The PCAs in mechanical alloying were pure ligroin, 75 vol.% ligroin + 25 vol.% alcohol, 50 vol.% ligroin + 50 vol.% alcohol, 25 vol.% ligroin + 75 vol.% alcohol, and pure alcohol. The normal composition is Ni-22.9at.%Al-0.5at.%B, the ball-to-powder weight ratio is 10:1, and the milling time is 30 min. Then, the powders were sintered by spark plasma sintering under 40 MPa for 5 min at 1000°C. The results show that a higher bending strength and a higher hardness were obtained when the PCAs were 75% ligroin + 25% alcohol in mechanical alloying. The bending strength is about 2700 MPa and the hardness (HV) is more than 6 GPa.

Keywords: intermetallic compounds; mechanical properties; mechanical alloying; process control agent; spark plasma sintering

1. Introduction

Ni-Al based intermetallic compounds possess advantageous properties: high tensile strength and yield point, low density, high-temperature creep resistance, good corrosion, and oxidation resistance at elevated temperatures [1-3]. One of the intermetallics for high-temperature applications is Ni₃Al with fcc order L1₂ structure [1, 4]. The yield strength of the Ni₃Al increases with increasing temperature up to 600-800°C [5]. However, there are many limitations in its application as engineering materials because of the extreme brittleness of polycrystalline Ni₃Al at room temperature, especially the fabrication of Ni₃Al by conventional cast is difficult and costly to process as structural components [6-7].

Mechanical alloying (MA) is an alternative technique for producing metallic powders in solid state [8-9]. Using MA for the preparation of Ni₃Al results in the occurrence of metastable states and dispersed microstructures that significantly change the properties of the alloy [10].

In this article, the microstructure and characteristics of mechanical alloying powder with a normal composition of Ni-22.9at.%Al-0.5at.%B has been investigated. The effect of process control agents (PCAs) on the performance of the powder has been studied. The properties include the density

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of sintered samples by spark plasma sintering (SPS), bending strength, and hardness.

2. Experimental

The powders of Ni, Al, and B were mixed to obtain the normal composition of Ni-22.9at.%Al-0.5at.%B. The particle size and purity of the powders are shown in Table 1. Mechanical alloying was performed at room temperature in air using vibratory milling. The powders and the hardened stainless steel milling balls were sealed in a hardened stainless steel vial. To decrease the agglomeration and adhering, pure ligroin, 75 vol.% ligroin + 25 vol.% alcohol, 50 vol.% ligroin + 50 vol.% alcohol, 25 vol.% ligroin + 75 vol.% alcohol, and pure alcohol were each added to the mixture as PCAs. The ball-to-powder weight ratio was 10:1. The milling time selected was 30 min. Then, the powders were sintered by SPS under 40 MPa for 5 min at 1000°C.

Table 1. Size and purity of powders as raw materials

Powder	Size / µm	Purity / %	
Ni	2	99.9	
Al	2	99.5	
В	3-5	98.0	

X-ray diffraction patterns of the powders and sintered samples were recorded using a Philips 330 diffractometer with Cu K_{α} radiation at 40 kV and 25 mA. Scanning electron microscopy with energy dispersive spectrometer (EDS) was used to investigate the microstructure of the samples. The densities of sintered samples were determined by the buoyancy method applying the Archimedes Principle. Bending specimens with 16 mm in gauge length, 3 mm in width, and 3 mm in thickness were prepared from the samples. Bending test was done at a constant strain rate of 0.5 s⁻¹ at room temperature. Micro hardness test was carried out

at a load of 20 N.

3. Results and discussion

The densities of the samples are shown in Table 2. It can be seen that the density decreases along with the increase in the alcohol content of the PCA, and the maximum density can reach 7.496 g/cm³, near to the theoretical density of Ni₃Al (7.5 g/cm³). The reason is that the oxygen content of the powders increases along with the alcohol content of the PCA, which results in the increase in the pore of the samples.

			Table 2.	Density of the samples		
Samples	PCA	<i>m</i> ₁ / g	<i>m</i> ₂ / g	Theoretical density / (g·cm ⁻³)	Density / $(g \cdot cm^{-3})$	Relative density / %
1#	100 vol.%L	16.5383	14.3319	7.5	7.496	99.90
2#	75vol.%L-25vol.%A	16.3623	14.1326	7.5	7.338	97.84
3#	50vol.%L-50vol.%A	14.9614	12.8475	7.5	7.078	94.40
4#	25vol.%L-75vol.%A	14.6544	12.5700	7.5	7.031	93.70
5#	100 vol.%A	14.7108	12.6246	7.5	7.051	94 .00

Note: L is ligroin, A is alcohol, m_1 is the mass of the sample in air, and m_2 is the mass of the sample in water.

The characterization of X-ray diffraction patterns of the sintered powders and bodies were shown in Figs. 1 and 2, respectively. In Fig. 1 it can be seen that some Al remains. It suggested that a supersaturated solution of Al in Ni was obtained after milling. While Fig. 2 shows that Ni₃Al was formed by spark plasma sintering (SPS) under 40 MPa for 5 min at 1000°C. During milling, Fe as a pollution was brought in, but it cannot be seen in the characterization of X-ray diffraction patterns because of very little.



Fig. 1. X-ray diffraction patterns of the as-milled powders.

Fig. 3 shows the microstructures of samples 1#, 2#, 3#, 4#, and 5#. More pores exist in sample 2# than in sample 1#. So the density of sample 2# is lower than that of 1#. But the dispersed Al₂O₃ increased the strength and hardness of samples. However, the pores can be seen in the intergranular from Fig. 3(c) to Fig. 3(e). This results in a lower strength and hardness of samples 3#, 4#, and 5#.

The relationship between the bending strength and the alcohol content, and the relationship between the hardness

and the alcohol content are shown in Figs. 4 and 5, respectively.



Fig. 2. X-ray diffraction patterns of bodies.

Fig. 4 shows that the maximum bending strength is 2768 MPa with 75 vol.% ligroin mixed with 25 vol.% alcohol as PCA. It can be observed that the maximum hardness (HV) is more than 6 GPa with 75 vol.% ligroin mixed with 25 vol.% alcohol as PCA (Fig. 5).

The oxygen of the sample with 100% ligroin is from milling, that of the other samples (sintered with 75 vol.% ligroin + 25 vol.% alcohol, 50 vol.% ligroin + 50 vol.% alcohol, 25 vol.% ligroin + 75 vol.% alcohol and pure alcohol as PCAs) are from milling and alcohol. The oxygen content of the sample with 100% ligroin is 0.20 wt.%, and that with 100% alcohol is 0.61 wt.%. The oxygen content increased with the increase in alcohol. Fig. 6 shows the composition of compacts with powder milled for 30 min by EDS. Fig. 6(a) shows the point scanning of second phase; Fig. 6(b) shows the area scanning of matrix. Obviously, the oxygen content

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