Synthesis of Nanocomposite Powder for Tungsten Heavy Alloy by Hydrogen Reduction of Ultrasonic-milled Oxide Nanopowders

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Abstract

Ultrasonic-milling of metal oxide nanopowders for the preparation of tungsten heavy alloys was investigated. Milling time was selected as a process variable. XRD results of metal oxide nanopowders ultrasonic-milled for 50 and 100h showed that mean crystallite size reduced with increasing milling time and there was no evidence of contamination or change of composition by impurities. It was found that nanocomposite powders reduced at 800°C in H_2 atmosphere had a composition of 93.1W-4.9Ni-2.0Fe by EDX analysis. Hardness of sintered samples of 50 and 100h was 390 and 463 Hv, respectively, which corresponds to the hardness of commercial products.

Keywords : Tungsten heavy alloy, Nanocomposite powder, Ultrasonic milling, Hydrogen reduction

1. Introduction

93W-4.9Ni-2.1Fe tungsten heavy alloys have been applied extensively to kinetic energy penetrator due to their high density, strength and ductility [1]. It is well known that the mechnical property of tungsten heavy alloys is deeply influenced by W/W and W/matrix interface charecteristics [2,3]. Thus, the design of interface structure is essential for enhanceing properties. In particular, it is necessary to reduce powder size, to homoginize powder mixture, and to maintain purity, in order to reinforce interface strength. Previous method has been represented by mechanical alloying (MA) using attritor. However, powder property was degraded by impurities formed during attrition.

In this respect, we applied ultrasonic-milling process for preparation of tungsten heavy alloy in this study. Ultrasonic-milling uses ultrasonic cavitation which leads to the violent collapse of macroscopic bubbles, producing extremely high energy densities. This can be a desirable source of energy which nanopowders free of impurities can be obtained. In this study, milling time was selected as the main process variable which can affect on the microstructure of powders and sintered parts. Based on the microstructural results, we also characterized mechanical properties of sintered parts and estimated their applications.

2. Experimental and Results

As source materials, WO_3, NiO and -Fe_2O_3 powders were mixed together to 93W-4.9Ni-2.1Fe composition using turbular mixer for 1h. The mixture was milled in ultrasonic bath for 50 and 100h, After drying at 80°C for 24h, the material was sieved down using 100 meshes. The powder mixture was then reduced at 800°C in H_2 atmosphere for 1h. The reduced powder was compacted at room temperature under pressure of 500MPa and then sintered at 1470°C in hydrogen atmosphere for 20 min.

The crystal phase of nanocomposite powders was identified by X-ray diffractometer (XRD). Microstructures and chemical composition of nanocomposite powders and sintered parts were analysed by scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). Hydrogen reduction condition of nanopowder was optimized by thermogravimetry (TG) measurement. Hardness of tungsten heavy alloy was measured by Vickers hardness tester.

Figure 1 shows microstructures of oxide mixture samples after ultrasonic milling for 50 and 100h. Mean particle size of sample of 50 and 100h was measured as 500 nm, and 400 nm, respectively. Compared with the sample of 50h (Fig 1(a)), the sample of 100h in Fig. 1(b) shows that oxide mixture includes more agglomerates. However, we can find that particle size distribution of sample of 100h is narrower and mean size of agglomerates is smaller. XRD analyses results revealed that the oxide mixtures after ultrasonic milling consisted of WO_3, $WO_{2.9}$, NiO, and -Fe_2O_3 phases.

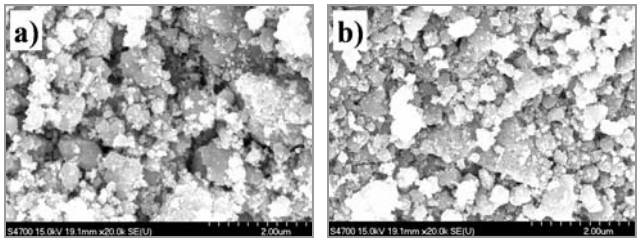

Fig. 1. SEM micrographs of oxide mixtures after ultrasonic-milling for (a) 50h and (b) 100h.

From the TGA curves in Fig. 2, we can find that reduction of tungsten oxide of powder sample of 100h is separated into 2 steps at around 700°C. Considering the fact that the two reduction steps of tungsten oxide is duplicated in the coarsed and densified microstructure, this results implies that powder is so porous that H_2 gas diffuses out easily and powder size is very fine.

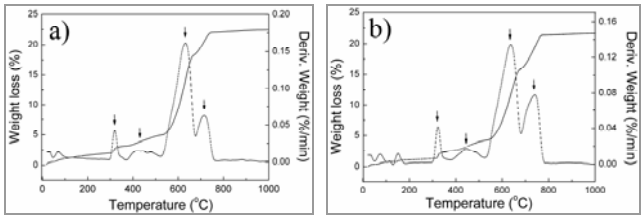

Fig. 2. TGA plots for oxide mixtures ultrasonic milled for (a) 50h and (b) 100h upto 1000°C in H_2 atmosphere at a heating rate of 10°C/min.

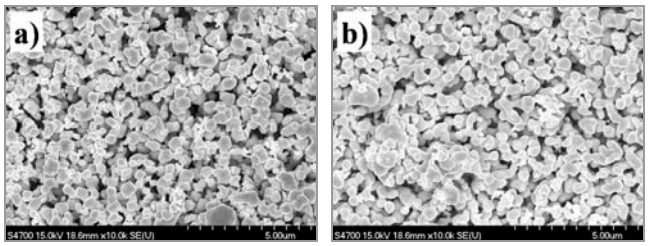

Fig. 3. SEM micrographs of nanocomposite powders reduced from oxide mixtures milled for (a) 50h and (b) 100h in ultrasonic bath.

From the microstructures in Fig. 3, it was found that both samples of 50h and 100h consist of primary particles of 400–500 nm in diameter. However, it was also found that particles in the sample of 100h were sintered more. This reveals that particle size in the sample of 100h was very fine by the ultrasonic milling and sintering between particles occurred vigorously. XRD analyses for these powder samples showed that these nanocomposite powders consist of tungsten and -FeNi phases. Also, EDX results showed that the composition of nanocomposite powder was 93.05W-4.91Ni-2.04Fe and no evidence of impurities for all powder samples.

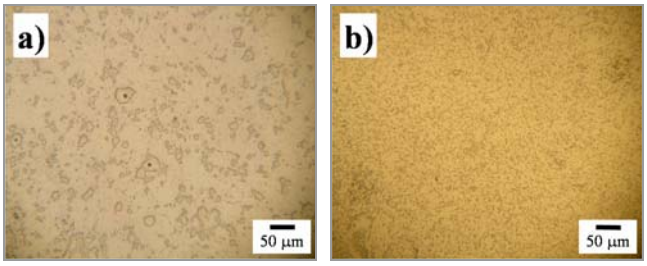

Fig. 4. Micrographs of surface of sintered parts. Powder samples of ultrasonic milling for (a) 50h and (b) 100h.

These reduced powders were sintered at 1470°C for 20 min after compaction at pressure of 500 MPa. Though the both samples had a density of 95% of theoretical density, we can find that the liquid phase of sample of 100h is distributed homogeneously from Fig. 4.

In order to analyze mechnical property of sintered parts of tungsten heavy alloys, we measured hardness using Vickers hardness testing machine at applied load of 200g. Hardness of sample of 100h was 463 Hv, whereas sample of 50h was 390 Hv. Especially, samples prepared by ultrasonic-milling showed sufficiently high values of hardness because homogeneously mixed nanocomposite powders are distributed between tungsten and matrix. Also, the hardness of samples in this study was similar to that of commercial tungsten heavy alloy of same composition (Rockwell hardeness (c scale): 34–48, Vickers hardness: 336–484 Hv) [4].

3. Summary

In this study, ultrasonic-milling was applied to refinement of nanocomposite powders for heavy alloy. Tungsten heavy alloy samples of 50 and 100h showed an adqueate hardness corresponding to that of commercial products, due to the refinement and high purity of powder.

4. References

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