Synthesis of Ultrafine Zr Based Alloy Powder by Plasma Arc Discharge Process

Gil-Geun Lee[1,a], Je-Shin Park[2,b], and Won-Baek Kim[2,c]

[1]Division of Materials Science and Engineering, Pukyong National University, San 100, Yongdang-Dong, Nam-Gu, Busan, 608-739, Korea
[2]Minerals and Materials Processing Division, Korea Institute of Geoscience and Mineral Resources, 30, Gajeong-Dong, Yuseong-Gu, Daejeon, 305-350, Korea
[a]gglee@pknu.ac.kr, [b]jspark@kigam.re.kr, [c]wbkim@kigam.re.kr

Abstract

In the present study, ultrafined Zr-V-Fe based alloy powder prepared by a plasma arc discharge process with changing process parameters. The chemical composition of synthesized powder was strongly influenced by the process parameters, especially the hydrogen volume fraction in the powder synthesis atmosphere. The synthesized powder had an average particle size of 50 nm. The synthesized Zr-V-Fe based particles had a shell-core structure composed of metal in the core and oxidse in the shell.

Keywords : zirconium, nano powder, plasma arc discharge, vapor condensation

1. Introduction

Metal ultrafine particles with a diameter between 10 nm and 100 nm have a wide range of potential applications, including magnetic recording media, catalysts, conductive or resistive pastes, ferrofluids, and others.[1)] Zirconium based alloy is one of the main elements for the application of metal ultrafine particles in various industrial fields. Ultrafined zirconium based alloy particles are of particular interest in the hydrogen storage and gas absorption. However, these properties of the ultrafined zirconium based alloy powder have been rarely studied exhaustively. The physical and chemical properties of metal ultrafine particles strongly depend on the particle and surface characteristics, including size, morphology, surface area, surface oxide, and others. In practical applications of metal ultra fine particles, the relationship between the preparation process parameters and their properties should be verified.

Ultrafine particles can be produced by several different methods, such as colloidal precipitation, mechanical attrition and a vapor condensation process.[2)] The latter is widely used method at present, based on its advantages of being relatively simple to scale up to high-rate production. Vapor condensation processes typically involve nucleation and growth of nanosized particles from a supersaturated vapor produced either by the evaporation of bulk materials or by the chemical reaction of gas-phase precursors. The plasma arc discharge process can be used for preparing metal, ceramic and their composite ultrafine particles by vaporization-condensation of metals or alloys in an active atmosphere using a DC arc-plasma.[3)] It has been recently synthesized that the pure metal powders, for example iron, alunium, tin and others by this process. However, it has been rarely reported that the synthesis of the metal alloy powders by the plasma arc discharge process.

In the present study, ultrafine zirconuim based alloy particles were prepared by the plasma arc discharge process under the various powder synthesis conditions, and then the morphology, phase structure and surface structure of the synthesized particles were studied using FE-TEM, XRD and XPS.

2. Experimental and Results

Figure 1 shows the apparatus for preparing the ultrafine particles by the plasma arc discharge method. This apparatus mainly consisted of a vacuum chamber, a tungsten cathode, a copper anode, a gas flow system and a DC power supply. $Zr_{57}V_{36}Fe_7$ bulk alloy was used as the raw material. The vacuum chamber was evacuated to 10^{-5} Torr and then backfilled with a hydrogen/argon mixture gas to 300~550 Torr. The arc plasma was then initiated between the tungsten cathode and the bulk alloy on the copper anode under an arc current of 140A and an arc voltage of 21~25V. The mixing ratio of the hydrogen:argon was changed from 4:6 to 5:5.

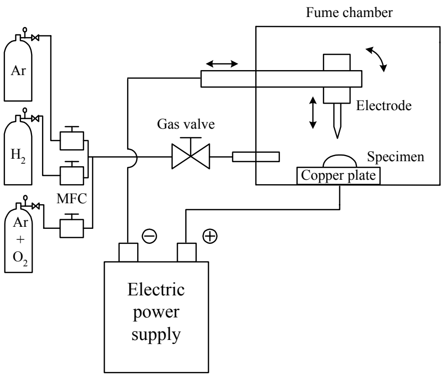

Fig. 1. Schematic diagram of apparatus for producing ultrafine particles by the plasma arc discharge process.

Figure 2 shows the chemical composition of the synthesized Zr-V-Fe powders with the volume fraction of hydrogen gas in the powder synthesis atmosphere. The chemical composition of the synthesized powder drastically changed with the volume fraction of hydrogen in the synthesis atmosphere. The synthesized Zr-V-Fe powder under 40% H_2 atmosphere had $Zr_{14}V_{48}Fe_{36}$ composition, and the one under 50% H_2 atmosphere had $Zr_{55}V_{28}Fe_{16}$ composition. The chemical composition of the synthesized Zr-V-Fe powder changed to the chemical composition of the raw material, $Zr_{57}V_{36}Fe_{7}$, with the increasing hydrogen volume fraction. It knows that the mixing ratio of the hydrogen and argon in the synthesis atmosphere was very important for the control of chemical composition of the powder on synthesis of the ultrafine metal alloy powder by the plasma arc discharge process.

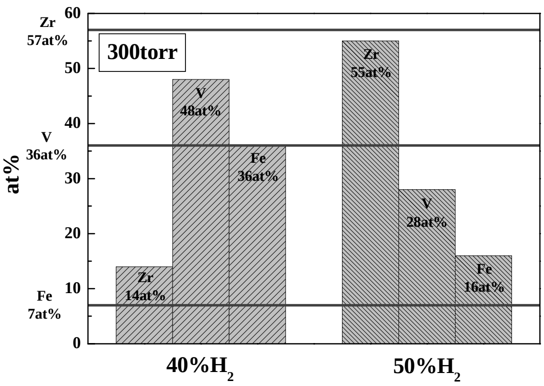

Fig. 2. Variation in the chemical composition of the synthesized powder with the volume fraction of hydrogen gas in the synthesis atmosphere.

Figure 3 shows the TEM micrograph of the synthesized Zr-V-Fe powder for hydrogen volume fraction of 50% H_2. The synthesized powder had an average particle size of 50 nm.

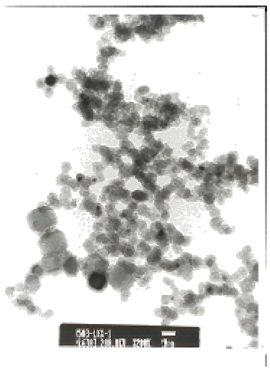

Fig. 3. TEM micrograph of the synthesized Zr-V-Fe powder.

Figure 4 shows the analysis results of the XPS peak of the synthesized Zr-V-Fe ultrafine particles. The analysis of the binding energy was carried out on O1s for the oxides based on the Gaussian function. The measured peak could be separated into three peaks; one was ZrO_2, Fe_3O_4, V_2O_5 and Fe_2O_3, other was FeO(OH) and Fe(OH)O, and the other was not indentified. The synthesized Zr-V-Fe ultrafine particles are known to have a shell-core structure compose of metal in the core and oxides in the shell.

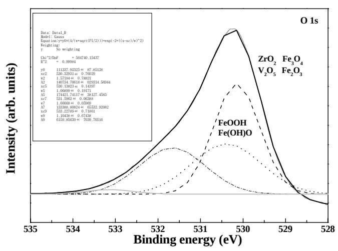

Fig. 4. Analysis results of the XPS peak of the synthesized Zr-V-Fe ultrafine particles.

3. Summary

The Zr-V-Fe alloy powder has an average particle size of 50 nm can be synthesized by the plasma arc discharge process. The hydrogen volume fraction in the synthesis atmosphere had an effect on the chemical composition of the powder. The synthesized Zr-V-Fe particles had a shell-core structure composed of metal in the core and oxides in the shell.

4. References

1. R. W. Siegel, Materials Science and Engineering, **A164**, 189 (1993).
2. B. K. Kim, G. G. Lee, G. H. Ha and D. W. Lee, Metals and Materials, **5**, 109 (1999).
3. Y. Sakka, and S. Ohno, Applied Surface Science, **100/101**, 232 (1996).