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FABRICATION OF Ni NANOPOWDER USING WIRE EXPLOSION PROCESS AND ITS CHARACTERIZATION

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Abstract. We fabricated Ni nanopowders using wire explosion process and investigated an effect of the charging voltage on the particle size and the dispersibility. Ni nano particles with a few nm to 100 nm could be successfully fabricated without the clustering between particles. With increasing the charging voltage, the mean particle size slightly decreased from 40 to 27 nm.

1. INTRODUCTION

Metal nanopowders less than 1 mm in particle size have significantly high fraction of atoms located at their surfaces. These unique properties allow several remarkable advantages: (I) enhanced chemical reaction; (II) faster sintering kinetics; (III) higher electrical resistivity; (IV) increased magnetic property; and (V) microwave absorption, etc. [1]. Several methods for the nanopowders have been developed in the last year and can be classified into three major classes: (I) Mechanical process; (II) Physical process; (III) Chemical process. Among them, chemical process has been widely used in manufacturing of metal nanopowders. Using chemical process, however, unwanted products can be formed during chemical reaction. Recently, wire explosion technique has been attempted to physically produce metal nanopowders. A high power pulsed current, which passes through a thin metal wire, leads to the wire explosion and the large amount of joule heating causes wire to melt, and subsequent evaporation and ionization. The plasma formed during the process expands and cools when it interacts with the surrounding gas or liquid, and then nano-particles are formed through the nucleation process [2-3]. In general, it has been reported that the size and morphology of the particle fabricated by the wire explosion process are influenced by several conditions such as an amount of energy deposited to the exploding conductor, polarity of the charging voltage, the type of coolant, wire diameter, and feeding distance, *etc* [2]. Among the several metal powders, ultrafine Ni powders have been used in specific industrial fields, especially on the preparation of microwave absorbing materials, magnetic recording tape, commercial batteries, and catalysts for reforming of methane with carbon dioxide or steam for hydrogen production [4-6]. It is well known that the performance of metal powders for several applications depends on particle size, dispersibility, morphology, and surface modification, etc. Many studies have been carried out to improve the performance of metal powders using various techniques [7-9]. As mentioned above, wire explosion process would be one way to easily and physically make Ni nanopowders without unwanted products. In this work, we tried to produce Ni nanopowders using wire explosion process and investigate an effect of polarity of the charging voltage on particle size, morphology, dispersibility, and surface structure of Ni powders.

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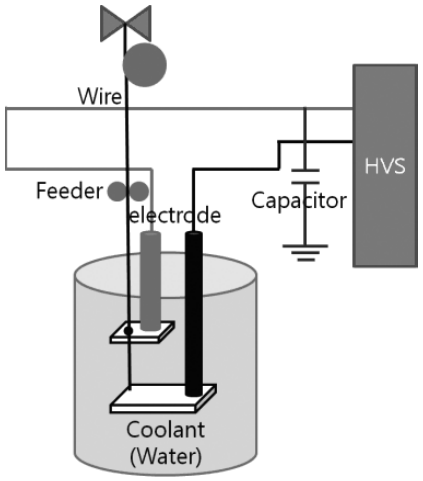

Fig. 1. Schematic illustration of wire explosion equipment.

2. EXPERIMENTAL PROCEDURE

The equipment of wire explosion system for Ni powders is schematically presented in Fig. 1. Diameter of wire used in this study was 0.25 mm and feeding length was 40 mm. Wire explosion was carried out at charging voltages of 200, 250, and 300 V. Pure water of 700 mmL was used as the liquid coolant. Total wire length exploded wire was 20 m. Table 1 shows wire explosion condition for this work. Transmission electron microscopy (TEM) study was carried out to evaluate particle size, morphology, and surface structure. Particle size was also measured by laser particle size analyzer. Zeta-potential analyzer was used to evaluate particle dispersibility. XRD was used to evaluate crystal structure of particles.

3. RESULTS AND DISCUSSION

Fig. 2 shows TEM micrographs of Ni powders produced by wire explosion at 200, 250, and 300 V in the charging voltage. Prior TEM observation, it was found that pieces of wire are sunk to the bottom of the bottle, when the charging voltage is 200V. This result indicates that the charging voltage of 200 V is insufficient to perfectly explode the wire. However, no wire pieces are seen at bottom of the bottle at and above 250V in the charging voltage. The charging voltage for wire explosion can be written as

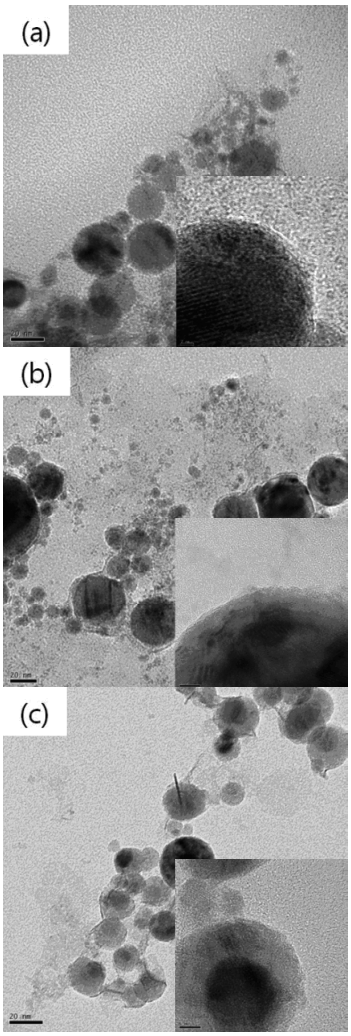

Fig. 2. TEM images of Ni powders produced by wire explosion process at the charging voltage of (a) 200 V, (b) 250 V, and (c) 200 V.

$$W_s = V_{volume} \times w_s = \frac{\pi \times d^2}{4} \times l \times w_s, \quad (1)$$

$$\text{If } K = 1, K = \frac{W}{W_s}, \quad (2)$$

Table 1. Wire explosion conditions in this experiment.

Sample No.	Wire diameter (mm)	Feeding distance (mm)	Charging voltage (V)	Coolant
A	0.25	40	200	Pure water
B			250	
C			300	

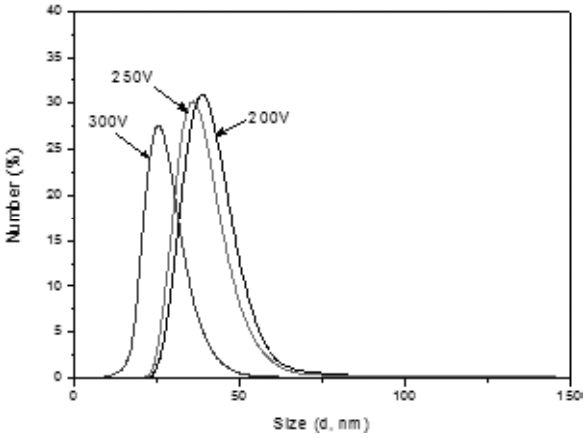

Fig. 3. Particle size distribution of Ni powders produced by wire explosion process at different charging voltage.

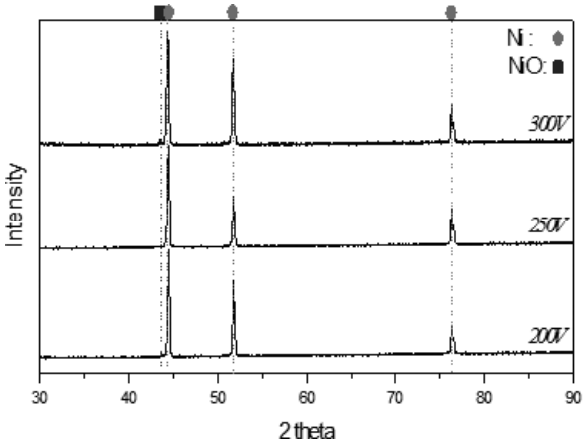

Fig. 4. X-ray diffraction (XRD) patterns of Ni powders produced by wire explosion process at different charging voltage.

$$U = \frac{1}{2} \times C \times V_{voltage}^{2} \text{ or } U = \frac{W}{0.85}, \quad (3)$$

$$V_{voltage} = \sqrt{\frac{2 \times U}{C}}. \quad (4)$$

Here, W_s is the energy for sublimation of the wire per volume unit, w_s is the sublimation energy of the wire (Ni: 53.9 J/mm^3), V_{volume} is wire volume, U is the charging energy for explosion, C is capacitance, K is the super heating factor, and $V_{voltage}$ is the charging voltage for wire explosion. From these equations, the super heating factor (K) could be calculated and the result is presented in Table 2. It is also found that the large sized powders sank to bottom of the bottle in all conditions. However their volume fraction decrease with increasing the charging voltage, indicating that the recovery rate of nanopowders increases with increasing the charging voltage. TEM observation was carried out using Ni powders excepting powders sunk to the bottom of the bottle. Entirely, the particles were found to be nearly spherical in powder shape. TEM images show the absence of dislocations in Ni particles and the particle size lies in the range of a few nm to ~100 nm in all conditions. In particular, it is found that NiO layer is rarely seen at surface of particles by HR-TEM work even that Ni powders were surface passivated by liquid coolant (pure water). Using TEM images, it was not easy to exactly measure the particle size because the volume fraction of the Ni particles detected in TEM images is relatively small. Beside, no discernable difference in particle size between Ni powders produced at 200, 250, and 300 V was visible. In order to investigate an effect of the charging voltage on particle size, laser particle analyzer was used. Fig. 3 shows the particle size distribution plotted as a function of the charging voltage for Ni powders. With increasing the charging voltage the particle size distribution range becomes to be narrow and small. The mean particle size obtained from particle size distribution diagram was estimated to be 39.95 nm for 200 V, 38.31 nm for 250 V, and 26.83 nm for 300 V in the charging voltage. This result is well corresponding to the previous result reported by Kwon [10-11]. They reported that one of

Table 2. Relation between the charging voltage and the super heating factor in this experiment.

Sample No.	Energy for sublimation of the Ni wire per volume unit (J)	Sublimation energy of Ni wire (w_s, J/mm^3)	Capacity (μF)	Charging voltage (V)	Charging energy for explosion (J)	Super heating factor (K)
A				200		0.35
B	105.8	53.9	2,200	250	79.7	0.55
C				300		0.8

the main parameters influencing the properties of the produced powders including particle size, morphology, and surface modification is the super heating factor or the specific energy input into the wire. They also revealed that the produced particle size decreases with increasing the super heating factor and the specific energy input into the wire. In general, the dispersibility of nano particles has been considered as an important property in application of nanopowders because the clustering between nano particles deteriorates the catalytic activity. Zeta-potential analysis is a method to identify the dispersibility between particles [12]. From this experiment, zeta-potential of Ni powders was estimated to about 26 mV for 200 V, 29.6 mV for 250 V, and 30.7 mV for 300 V in the charging voltage. Entirely, zeta-potential of Ni powders produced in this work lies in the range of 25 to 31 mV and slightly increases with increasing the charging voltage. Here, the absolute value larger than ±30 mV in zeta-potential means that particles are well dispersed without the clustering. Although zeta-potential of Ni powders produced at 200 V in the charging voltage is slightly lower than ±30 mV, it can be revealed that Ni powders produced by wire explosion process are relatively well dispersed without the clustering between particles. The crystal structure along with the particle size, the surface morphology and the dispersibility is also considered as an important property in application of nanopowders. The crystal structure of Ni powders produced by wire explosion process was confirmed by the XRD using Ni powders dried at room temperature for 48 hr and the result is presented in Fig. 4. The strong peaks corresponding to Ni phase and the very weak peaks corresponding to NiO phase are detected. Considering that NiO layer was rarely observed at particle surface in TEM images, NiO phase may be formed during drying stage. Also, since the surface energy of particle increases with decreasing particle size, NiO phase may be partially formed at an extremely fine particle. Further studies on oxidation behavior are needed to confirm this result.

4. CONCLUSION

We could successfully produce Ni nanopowders using wire explosion process in pure water atmosphere. Well dispersed Ni nanoparticles without the unwanted products such as NiO phase could be obtained. Most of Ni powders exhibited near spherical type in shape and very small size less than 100 nm in particle size. The mean particle size decreased with increasing the charging voltage affecting to the specific energy input into the wire (W_s/W) or the super heating factor (K).

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