Fabrication of Al Nano Powders by Pulsed Wire Evaporation (PWE) Method

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Abstract: Nano-sized Al powders were synthesized by the Pulsed Wire Evaporation (PWE) method. The wire, which was fed continuously into a chamber filled with Ar gas, was exploded into nano powders by high pulsed current and voltage. After the nano powders were synthesized, they were classified by sizes using cyclone and various kinds of filters. We passivated the powder surface with a thin oxide layer to stabilize at room temperature. The size and shape of the nano powders were investigated by TEM. XRD was conducted for phase analysis and grain size determination. XPS and BET analyses were conducted for the investigation of the oxide layer on the Al powder and specific surface area, respectively. From the TEM observation, it was found that the Al nano powder was spherical shape of 80 ~ 120 nm diameter with the thickness of the oxide layer of 2 ~ 3 nm. The Al phase was crystalline with FCC structure, on the other hand the oxide layer was an amorphous state.

Keywords: pulsed wire evaporation, Al nano powder, specific surface area, crystalline Al, amorphous oxide

Introduction

Synthesis of nano metallic powders from atomic or molecular sources depends on the control of a variety of nanoscale attributes desired in the final product. In general, there are basically two broad areas of synthetic techniques for nano metallic powders, namely, physical methods [1] and chemical methods [2]. Several different physical methods are currently in use for the synthesis and commercial production of nano powders. The most widely used technique involves the synthesis of single-phase metals by the inert-gas evaporation technique [3]. The generation of atom clusters by gas phase condensation proceeds by evaporating a precursor material, either a single metal or a compound, in a gas maintained at a low pressure. The other physical method involves generation of nano powders via severe mechanical deformation [4]. The nanometer-sized grains nucleate within the shear bands of the deformed materials converting a coarse-grained structure to an ultrafine powder. Although this method is very useful in generating commercial quantities of the material, it suffers from the disadvantage of contamination problems resulting from the sources of the grinding media. Among the chemical methods, solution chemistry is used sometimes to prepare the precursor, which is subsequently converted to nanophase particles by nonliquid phase chemical reactions. Precipitation of a solid from a solution is a common technique for the synthesis of fine particles [5,6]. The primary advantage that chemical processes offer over other methods is good chemical homogeneity, as chemical synthesis offers mixing at the molecular level. However, there are certain difficulties in chemical processing, such as complexity, hazard, contamination from the byproducts, agglomeration, etc. Finally, although many chemical processes are scalable for economical production, it is not always straightforward for all systems.

In this study, we used the pulsed wire evaporation (PWE) method for the synthesis of metallic Al nanopowder. The PWE method, which is a kind of inert gas phase condensation (IGC) method, is one of the most promising methods for the production of low-cost nanopowders. This method consists of dispensing a thin metallic wire as a high-power current pulse is passed through it [7]. When the high current passed through the metal wire, it was heated up, over its boiling temperature,
in a very short time of several micro-seconds and exploded. After it vaporized and met the surrounding gases like Ar, N₂, etc, the metal vapors condensed and formed powders with nano size. The brief sequence of this process is drawn in Figure 1. In ref. [8] it was recognized that a major factor determining the particle size after the PWE is superheating of the evaporated material. When an energy in the order of or higher than two binding energies is injected into the metal, all particles are formed from the vapor phase. It is possible to synthesize the nano metals [9], oxides, nitrides, carbides and even alloy powders by controlling the atmosphere in the reaction chamber. It is also possible to synthesize a nano-powder with high melting point metals such as W and Mo by the PWE method. The typical particle size of metal powders produced by the PWE method reduces substantially with increasing superheat of the metal, K=W/W₀ (where W is the energy injected into the evaporating wire and W₀ is the wire sublimation energy) [10], diminishes insconsiderable with decreasing wire diameter [10], and decreases as the surrounding gas pressure is raised [11].

After synthesis of Al nanopowder by the PWE method, we characterize the size, shape of nanopowders with several methods.

Experiments

Synthesis of Al Nano Powders

The experimental setup shown in Figure 2 consists of a pulse power generator, reaction chamber, wire feeding system, electrical filter, gas supply system, blower, and...
where $S_s$ is the specific surface area of powder resulted from the BET measurement, and $\rho$ is the density of powder. And we used the laser particle size analyzer (LPSA, BIC 90Plus) to determine the size distribution of nanopowders and compared each results.

**Results and Discussion**

**Synthesis and Characterization of Al Nano Powders**

The sublimation energy of Al is 33 J/mm$^2$. The length of Al wire was 88 mm, and input voltage was 26 kV. According to this experimental condition, the $K (=W/W_s)$ value of our experiment was 2. The shape of Al powders synthesized by the PWE method in a given condition was spherical with size distribution of 80 to 120 nm as shown in Figure 3. It was easily thought that as the Al wire was exploded, it was dispersed to a vapor phase. The Al vapors met the Ar gas in chamber and condensed to sphere with nano size to minimize the surface energy. Whether a liquid phase or semi solid phase was condensed, it became a relatively larger particle like A or B than others in Figure 3 [13,14].

The result of the phase analysis of passivated Al nano powder by XRD is shown in Figure 4. It shows that the only pure Al peaks were observed without any peak of the passivated oxide layer. However, from Figure 5 we can see the oxide layer on the surface of Al powder. It was considered amorphous state from the high resolution TEM view in Figure 5(c). Because the oxide phase was very thin and not a crystalline state, it was hardly detected by X-ray diffraction method. The size of the Al

![Figure 3. SEM view of Al nanopowders synthesized by the PWE method.](image)

![Figure 4. XRD pattern of Al nanopowders synthesized by the PWE method.](image)

![Figure 5. TEM view shows particle size and thickness of passivated oxide layer on the surface of Al nanopowder.](image)
powder was about 100 nm having passivated oxide of 2.5 nm thickness. The crystalline Al phase showed the FCC structure.

By XPS for the measurement of binding energy between elements, it is possible to verify the phase as shown in Figure 6. The Al\(^{3+}\) at 75.00 eV means passivated Al\(^{3+}\) oxide on the surface. Al\(^{0}\) at 77.73 eV shows the pure metallic Al. The Pt is from the specimen holder. Because the peak was broad and the thickness of the passivated layer was so thin, it was hard to observe the oxide layer in Figure 4 as same explanation from the TEM analysis [15].

Comparison of Grain and Powder Size by Different Methods.

From Figure 3, the average powder size was 80 ~ 120 nm by direct observation. The relative surface area was 20.4 m\(^2\)/g from the results of the BET measurement. It can convert to particle size, \(d_{BET}\), of 109 nm by the equation (2). By the laser particle size analysis (Figure 7) the average particle size was about 87.7 nm, which was similar with the result from direct observation. On the other hand the average grain size was obtained as about 28 nm from XRD analysis. Therefore each particle may not be single crystal but poly crystal, which have several grains in the particle. Particle sizes measured by each analysis were summarized in Table 1.

**Conclusions**

From the synthesis of Al nanopowders by the PWE method, the following conclusions were obtained:

1) The Al nano powder was polycrystalline with an average grain size of 28 nm. The particle was about 80 to 120 nm in size from the LPSA and SEM results.

2) The layer on the surface for the passivation and stabilization of Al powder was Al\(_2\)O\(_3\) with 2 to 3 nm of thickness. It was very stable in air. This oxide layer was an amorphous state as observed by TEM and XPS analysis but it was hard to detect by XRD method. On the other hand the Al was crystalline with FCC structure.

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