Preparation and Characterization of Monodispersed and Nano-sized Cu Powders

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Abstract

Monodispersed and nano-sized Cu powders were synthesized from copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) inside a nonionic polymer matrix by using wet chemical reduction process. The sucrose was used as a nonionic polymer network source. The influences of a nonionic polymer matrix on the particle size of the prepared Cu powders were characterized by means of X-ray diffraction), scanning electron microscopy), and particle size analysis). The smallen Cu powders with size of approximately 100 ㎚ was obtained with adding of 0.04M sucrose at reaction temperature of 60 ℃. The particle size of the Cu powders prepared by the reduction inside polymer network was strongly dependent of the sucrose content and reaction temperature.

Keywords : Nano-sized Cu powder, polymer network, sucrose, wet chemical reduction

1. Introduction

Ultrafine metallic powders have many important industrial applications. They are commonly used in electrically conductive paste, solid oxide fuel cells and chemical catalysts, etc. For the formation of thick film conductors such as hybrid integrated circuits and multilayer ceramic capacitors (MLCC), the technology of making conductive thick films from metal powders is of considerable importance. Precious metals (such as Au, Ag, Pt and Pd), their mixtures or alloys, and cheaper metals (such as Cu and Ni) are used for making conductive films.

Among the various methods have been developed for the preparation of Cu powders [1-3], chemical solution methods facilitate atomic level control and also efficient scale up for processing and production. The chemical solution routes essentially involve precipitation of metallic copper through chemical reduction of a salt, oxide or hydroxide of copper in solution. Mild chemical reduction of these precursors inside the swollen polymer network produces metal atoms, which later agglomerate into metal nano particles. Consequently, the growth of the metal nano particles during reduction could become limited by steric restrictions imposed by the three dimensional polymer network, a possibility that would permit precise control of the metal nano particle size.

Present paper reports the preparation of Cu powders by the principles of chemical reduction synthesis with hydrazine by adding sucrose. In addition, the influences of the reaction temperature on the morphology and particle size distribution for the synthesized copper powders will be discussed.

2. Experimental and Results

The starting materials were used copper sulfate ($CuSO_4 \cdot 5H_2O$; Sigma-Aldrich Inc.), hydrazine hydrate ($N_2H_4 \cdot H_2O$; Junsei Chemical Co., Ltd 80%), sodium hydroxide (NaOH; Kanto Chemical Co., Ltd 97%) and commercial sucrose as a reactant, pH control agent and nonionic polymer network, respectively. An appropriate amount of copper sulfate (0.5M) was dissolved directly in distilled water with sucroses (0~0.04M). The resulting mixtures were heated at 60℃, 70℃, 80℃ and 90℃ for 2h, and then hydrazine hydrate (molar ratio of $N_2H_4 \cdot H_2O$/ $CuSO_4 \cdot 5H_2O$=7) slowly dropped in mixtures. After washing and filtration, as-prepared products were dried at 25℃ for 24 h in a desiccator.

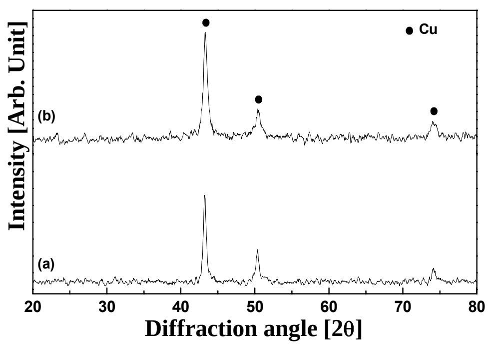

Fig. 1. XRD patterns of the samples prepared by wet chemical reduction without (a) and with (b) sucrose.

Fig. 1 shows the XRD patterns for metallic Cu powders prepared by wet chemical reduction without sucrose (a) and with sucrose (b). As can be seen in Fig. 1, the diffraction patterns exhibit mainly three characteristic peaks which can be indexed as fcc structure Cu (111), (200), and (222) no visible XRD peaks arising from the impurity phase such as CuO and Cu_2O can be found. The lattice constant calculated from its corresponding XRD pattern is a=3.6230 Å, which is well consistent with the literature value of a=3.6147Å given by JCPDS file No. 4-836 [4].

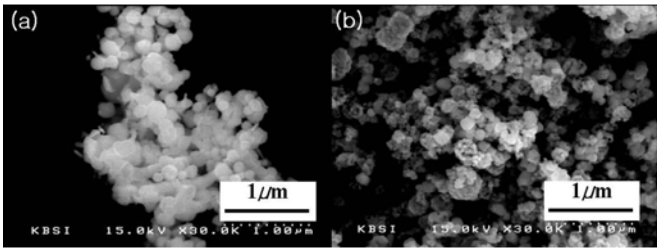

Fig. 2. SEM micrographs of the Cu powders prepared by wet chemical reduction without (a) and with (b) sucrose.

Fig. 2 shows the SEM images of Cu powders prepared without sucrose (a) and with sucrose (b). In the case of without sucrose, the larger Cu particles, which would be formed by the coalescence of primary particles, were agglomerated in irregular shape. On the contrary, the Cu powders obtained by adding of sucrose were slightly spherical in shape and not agglomerated. This would be due to the homogeneous metal distribution inside the swollen polymer network [1].

Fig. 3 shows the SEM images of the Cu powders synthesized by the addition of 0, 0.004, 0.02, and 0.04 M of sucrose. As the sucrose increased, the particle size of Cu powders steeply decreased and reached the minimum value of approximately 100 nm at the sucrose content of 0.04 M. The particle size increased again with a further increase of sucrose content. This is believed to be due to the pore size of the swollen polymer network.

As a result, the particle size of the Cu powders prepared by the wet reduction reaction inside polymer network was strongly dependent of the sucrose content.

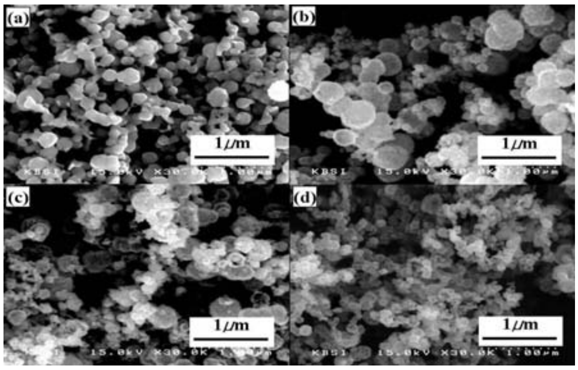

Fig. 3. SEM micrographs of the Cu powders prepared at different sucrose contents: (a) 0, (b) 0.004, (c) 0.02, and (d) 0.04M.

3. Summary

The nano-sized Cu particles have been successfully prepared by the wet-chemical reduction method of aqueous $CuSO_4$ using the hydrazine without and with sucrose as a polymer network. The influences of the sucrose on the morphology and particle size distribution for the synthesized Cu powders were investigated. The Cu powders essentially were monodispersed and irregular in shape regardless of reaction temperature and sucroses contents. The Cu powders prepared by adding of sucrose as a polymer network were nearly spherical in shape and seemed to be nano-sized, typically in the range of 100 nm with not being agglomerated. As a result, the particle size of the Cu powders prepared by the reduction inside polymer network was strongly dependent of the sucrose content and reaction temperature.

4. References

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