

액상환원법에 의한 $\text{Cu}(\text{OH})_2$ 슬러리로부터 미세구리분말 제조 및 분산화 처리
Synthesis and disperse treatment of Cu powder from $\text{Cu}(\text{OH})_2$ slurry by wet
reduction methods

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Abstract

Ultra-fine copper powders with particle size about 150 nm were synthesized from copper hydroxide slurry by wet method using hydrazine as reduction agent and several surfactants at below 80°C. The particle size distribution and dispersion of synthesized powders as function of temperature, feeding rate of reduction and especially, surfactants were characterized by XRD, BET, PSA and SEM by this process.

Introduction

One of the successful methods for preparation of metal powders is so-called heterogeneous reaction process [1]. In our study, copper powders were synthesized by hydrazine hydrate as reaction agent from $\text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$ slurry. The objectives of this study are the preparation of ultra-fine copper powders from $\text{Cu}(\text{OH})_2$ slurry and to control the shape and particle size distribution associating with stabilizing powders by polymer coating to obtain monodisperse copper powders.

Experimental

The suspension with copper hydroxide in reaction vessel was heated, while stirring. Hydrazine was introduced drop by drop into reaction cell. Several surfactants as gelatin, tannic acid, $\text{Na}_4\text{O}_7\text{P}_2 \cdot 10\text{H}_2\text{O}$ and PVP(K-30) were used for dispersion and anti-oxidant agents.

Results and discussion

When the feeding rate of reductants was increased, the particle size of copper powders obtained was decreased because nucleation rate was higher. For stabilizing and inhibiting the agglomeration, PVP and $\text{Na}_4\text{O}_7\text{P}_2$ were used. The mean particle size was decreased with increase of PVP/ $\text{Cu}(\text{OH})_2$ ratio because of diffusion barrier on the surface of copper powder by PVP and protection against copper grain growth. The mean particle size decrease with increase of stirring speed. That mean particle size varies from 1.4 μm to 0.150 μm as stirring speed varies from 200 to 600 rpm(Fig. 1).

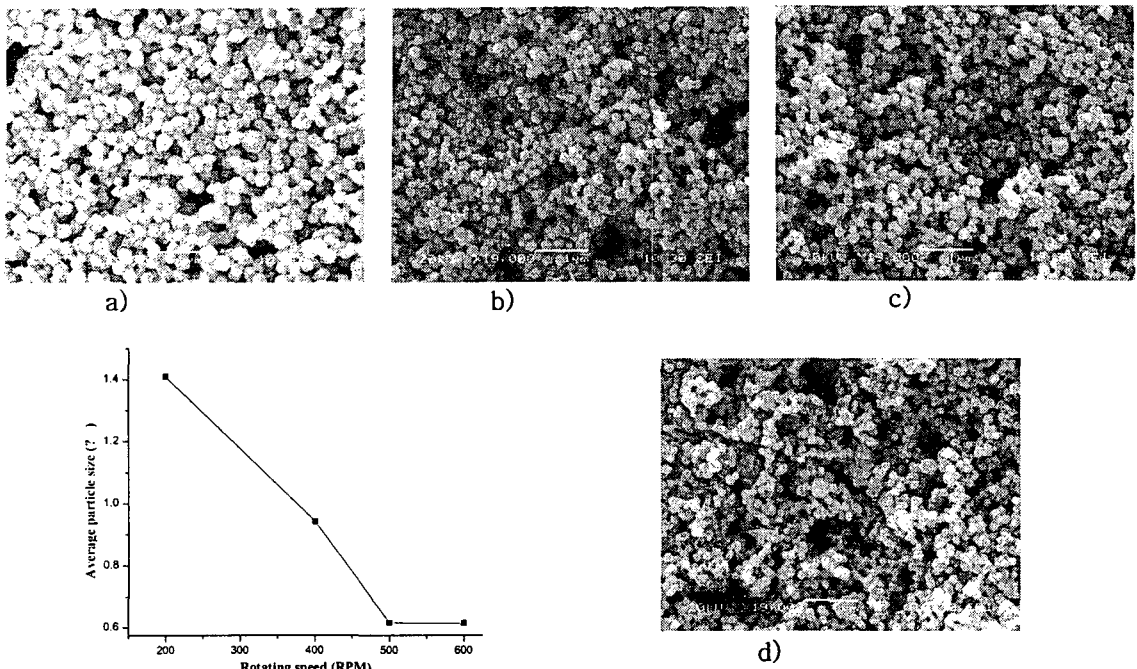


Figure 1: Effect of stirring speed on synthesized copper powders: a) 200RPM, b) 400RPM, c) 500RPM, d) 600RPM ($\text{Cu}(\text{OH})_2$: 10g/l, Temp: 60°C, Feeding rate: 1.75ml/min, $\text{Na}_4\text{O}_7\text{P}_2$: 0.994g/l)

Conclusions

Ultra fine copper powders with specific particle size around 150nm were synthesized by chemical reaction from $\text{Cu}(\text{OH})_2\text{-H}_2\text{O}$ slurry with hydrazine monohydrate as a reduction agent. The parameters such as reaction temperature, feeding rate of reduction, stirring speed, $\text{Cu}(\text{OH})_2$ concentration, and surfactants were investigated. It was found that the feeding rate of the reduction agent and the addition of surfactant are the main factors affecting the rapid changes in shape and size of the prepared copper powders. In addition, the decrease of their particle sizes lead to the increase of the agglomeration degree, resulting in the increase of the average particle size.