Synthesis of Spherical Micrometer-size Copper Particles

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Abstract: Spherical micrometer-size copper particles were synthesized by chemical reduction of cuprammonia sulfate solution with ascorbic acid as reducing agent. The sample was perfectly sphere and it was characterized by X-ray diffraction and scanning electron microscopy (SEM).

Key words: Metals and alloys; Microstructure; Micrometer-size; Copper; Reduction
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1 Introduction

Copper powders were extensively applied in powder metallurgy, ceramic materials, catalyst, and electronics industry. It can be obtained by technique of mechanical milling, electrochemical deposition, water atomization, flow-levitation[1] and chemical reduction in liquid polyols[2]. Using traditional method, often get coarse-micro-crystalline material that the grain size exceeds 100 nm. NaH_2PO_2[3], $N_2H_4 \cdot H_2O$[4], KBH_4[5], ascorbic acid[6] and formaldehyde[7] can be used as reducer in chemical reduction to prepare micrometer size copper powder. F. Fievet[2] showed that ethylene glycol or diethylene glycol was both solvent and reducing agent in reduction system. N. Srikanth[8] investigated nano-copper that was finer compared to coarse-micro-crystalline material. Their research showed that nano-copper exhibited unconventional behavior. In this paper, we report a convenient and environment-friendly route to synthesize perfectly spherical micrometre-size copper particles.

2 Experimental

2.1 Materials and processes

30 mL ammonia was added into 200 mL 0.5 mol·L^{-1} $CuSO_4 5H_2O$ solution, then 100 mL 1.25 mol·L^{-1} ascorbic acid was slowly added into above mixed solution. The temperature of the process was kept at boiling point and the solution was stirred to be homogeneous. The color of the solution would change from blue to green, yellow, brick-red and brown gradually. Finally, the obtained copper solution was centrifuged, washed several times with distilled water and absolute ethanol, and then dried in a vacuum at 333 K.

2.2 X-ray diffraction and scanning electron microscopy

The X-ray diffraction analysis (XRD) was recorded to identify the powders with Fe Ka radiation on a D/MAX-IIIB X-ray diffractometer (Rigaku Ltd, Japan). The morphologies of the synthesized

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materials were studied by JSM-5600LV scanning electron microscopy (SEM) (JEOL Ltd, Japan).

3 Results and discussion

Fig. 1 showed XRD pattern of spherical copper and the product is a single phase and well crystallized. This indicated that ascorbic acid acted as a reducing reagent and reduced copper compound to elemental copper.

SEM images were shown in Fig. 2, which illustrated copper spherical particle with a diameter ranging from 1 to 2 μm. In this experiment, we successfully synthesized fine, highly pure, monodisperse and non-agglomerated copper particles in ammonia aqueous solution.

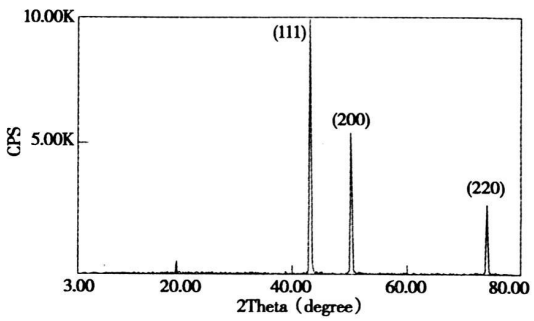

Fig.1 X-ray diffraction pattern of spherical copper

The reaction of the synthesis is $Cu^{2+} + NH_4OH \longrightarrow [Cu(NH_3)_4]^{2+} + [Cu_n(OH)_{2n-2}]^{2+}$, $[Cu(NH_3)_4]^{2+} + C_6H_6O_4(OH)_2$(ascorbic acid) $\longrightarrow [C_6H_8O_6 \cdot Cu]^{2+}$, $[C_6H_8O_6 \cdot Cu]^{2+}$ (heated) $\longrightarrow Cu_2O$, $Cu_2O + C_6H_6O_4(OH)_2$(ascorbic acid) $\longrightarrow$ Cu

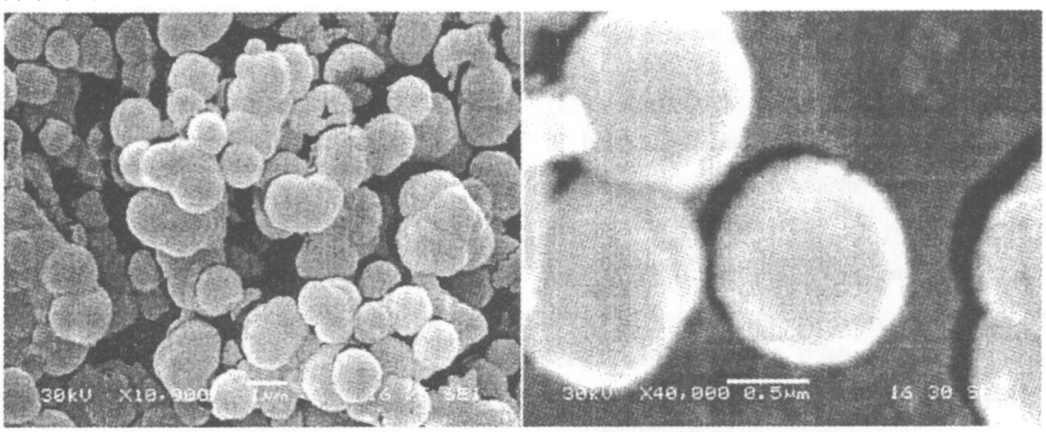

Fig.2 SEM of spherical copper

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微米级球形铜粉的制备

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摘　要: 以抗坏血酸为还原剂, 一定条件下还原硫酸铜氨溶液可以得到微米级球形铜粉。样品经 X－射线衍射和扫描电子显微镜测定, 并探讨了反应机理。

关键词: 金属合金; 微观结构; 微米; 铜; 还原