Synthesis of CuO nanometer powder by one step solid state reaction at room temperature

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Abstract Copper oxide nanometer powder was synthesized by one step solid state reaction of $CuCl_2 \cdot 2H_2O$ with NaOH at room temperature. The solid product CuO was characterized by X-ray diffraction and a transmission electron microscope. It has been found that the particles of prepared copper oxide are spherical with average diameter of 20 nm. This new method has the advantages of convenience and high yield compared with literature methods.

Keywords: **solid state reaction, nanometer powder, copper oxide.**

SOLID state chemistry is of fundamental importance in material science. Recently, the solid state reactions of coordination compounds have been developed and a lot of coordination compounds[1] as well as sulfur-containing cluster compounds[2], isomers of complexes[3], and solid-coordination compounds[4] have been synthesized at low heating temperature.

Because of the very fine grain size, ultrafine grains exhibit a variety of properties that are different and often considerably improved in comparison with those of conventional coarse grained materials. There has been a continued increase in the number of investigations in recent years on the synthesis, characteristic properties and potential application of these novel materials. The methods which have been commonly employed to synthesize these materials include inter gas condensation, mechanical alloying, spray conversion processing, physical vapour deposition, sol-gel process, laser ablation, etc. In this note, we report the preparation of nanometer CuO by one step solid state reaction at room temperature. This new method has the advantages of convenience and high yield compared with literature methods.

1 Experimental

Synthesis of CuO nanometer powder was as follows: Accurately weighed $CuCl_2 \cdot 2H_2O$ with NaOH in a 1:2 molar ratio were mixed and ground in an agate mortar in order to get the best possible homogeneity. The color of the mixture immediately transferred from sky-blue to dark at room temperature after 20 min. The products were washed by distilled water and 95% ethanol several times and air-dried. The CuO powder sample was measured by a Y-4Q Model X-ray diffractometer with copper target. The product CuO was ground in an agate mortar and was dispersed in 95% ethanol by ultra-sonic dispersion. The particle size and shape were characterized by a JEM-1000 Model transmission electron miscroscope.

2 Results and discussion

The experiment shows that $CuCl_2 \cdot 2H_2O$ with NaOH in a 1:2 molar ratio are mixed and ground to produce a dark product CuO immediately, which is different from the results of $Cu(OH)_2$ in solution. Considering that $Cu(OH)_2$ was disposed of by strong alkali to produce CuO[6], we speculate that the reaction of $CuCl_2 \cdot 2H_2O$ with NaOH to produce CuO is divided into two steps. The first step is to produce $Cu(OH)_2$ and the seond one is to dehydrate $Cu(OH)_2$ to produce CuO.

Obviously, in the X ray diffraction patterns of products (fig. 1), the diffraction peaks of the starting materials disappear and new ones appear instead, which are the peaks of CuO and NaCl. The diffraction pattern of the solid product CuO is the same as that of the analytical grade CuO, except that its diffraction peaks are wider than those of analytical grade CuO, which may be attributable to the solid product CuO with small size and bad crystalline form. Based on the X-ray diffraction data, the average particle size of the CuO powder can be estimated using Scherrer equation. The calculated data are about 23 nm. The transmission electron microscope photograph of the nanometer grains CuO is presented in fig. 2. TEM results show that the particle of prepared sample is spherical with average diameter of 20 nm. It presents a narrow particle size distribution. For these reasons, we draw the conclusion that $CuCl_2 \cdot 2H_2O$ reacts with NaOH in 1:2 molar ratio to produce nanometer powder CuO.

It is known that the crystal size is determined by the relative velocity of crystal growth and crystal

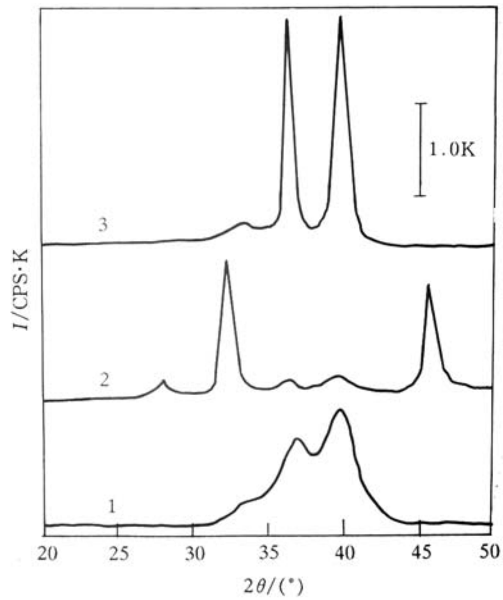

Fig. 1. XRD patterns of CuO nanometer powder. 1, nanometer CuO; 2, $CuCl_2 \cdot 2H_2O$ + NaOH reacting for 20 min at room temperature; 3, analytical grade CuO.

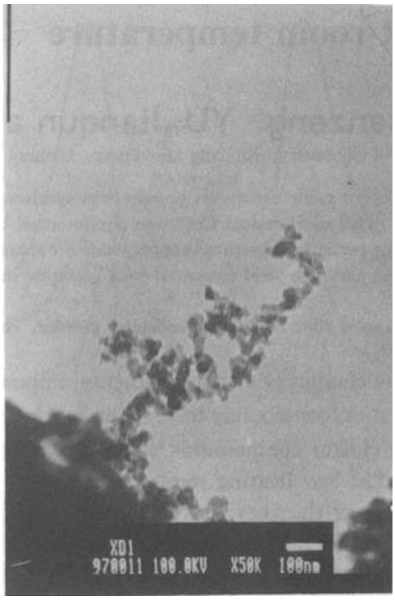

Fig. 2. TEM picture of CuO nanometer powder.

formation. In this work, $CuCl_2 \cdot 2H_2O$ with NaOH in a 1:2 molar ratio could produce a dark product CuO immediately after they were mixed and ground, which shows that the velocity of this reaction system is fast and the velocity of crystal formation is by far faster than that of crystal growth , so crystal grains of CuO nanometer powder with small size can be obtained.

In addition, we also studied other reaction systems of $CuSO_4 \cdot 5H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $Cu(OAc)_2 \cdot H_2O$, $Cu(gly)_2 \cdot H_2O$, $[Cu(NH_3)_4] \cdot SO_4$ etc. with NaOH separately. The solid products CuO of every reaction system were measured by X-ray diffraction (table 1) and the transmission electron miscroscope, the results of which were all similar to that of $CuCl_2 \cdot 2H_2O$ with NaOH, and nanometer CuO was obtained by every reaction system.

Table 1 XRD data of CuO

Reaction system	1			2			3			4			5		
I/I_0	8	90	100	10	80	100	12	85	100	9	80	100	10	90	100
d	2.75	2.52	2.31	2.75	2.51	2.34	2.73	2.51	2.33	2.78	2.52	2.32	2.79	2.52	2.33
$2\theta(°)$	32.5	35.5	38.9	32.5	35.7	38.5	32.8	35.7	38.9	32.2	35.6	38.8	32.1	35.6	38.7

1, Analytical grade CuO; 2, $CuSO_4 \cdot 5H_2O + 2NaOH \rightarrow CuO$; 3, $Cu(NO_3)_2 \cdot 3H_2O + 2NaOH \rightarrow CuO$; 4, $Cu(gly)_2 \cdot H_2O + 2NaOH \rightarrow CuO$; 5, $[Cu(NH_3)_4]SO_4 + 2NaOH \rightarrow CuO$.

Acknowledgement This work was supported by the National Natural Science Foundation of China (Grant No. 59462001).

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(*Received September* 22, 1997)