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Preparation of Cu-Cr alloy powder by mechanical alloying

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ABSTRACT

A study of Cu–Cr alloy powder preparation by a newly developed mechanical alloying (MA) approach was conducted using a modified mill. Results showed that the preparation temperature was much lower in the tests employing modified mill than with traditional equipment, and that no distinct differences could be found in the products prepared by the vibratory and planetary mills. Active carbon powder was found to exhibit an excellent reducing capacity for metallic oxides, while graphite powder was found inappropriate in this process. Based on the experimental results, purified Cu–Cr alloy nano-scale grain can prepared by mechanical alloying in the modified mill at 325 °C for 3 h with a ball-to-powder mass ratio of 15:1. A crystal agglomeration phenomenon was also found in the final alloy powder, leading to an increase in particle size.

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1. Introduction

Cu–Cr alloys are an important electrical material that has been utilized in contact cables [1], lead frame materials in integrated circuits [2] and diverter target materials [3], etc. The presence of chromium improves the mechanical property of the material resulting in Cu–Cr alloys that possess extraordinary hardness and high electrical and thermal conductivity. Because of the extremely low solid solubility of chromium in copper even at eutectic temperature [4], many studies have been focused on the supersaturated solid solution preparation route [5–8], in which mechanical alloying (MA) has attracted a lot of attention as a competitive technique [9,10]. By this method, the solid solubility can be notably improved and the grain size of the alloy powder reduced to nano-scale [11], vital factors for the mechanical and electrical properties of Cu–Cr alloys [12].

MA involves repeated welding, fracturing and rewelding of powder by continuous milling and is a widely used technique for solid-state powder manufacturing. Copper and chromium powders can be composited on atomic scale using this approach. As reported in the literature, Cu–Cr alloy powder with outstanding performance can be produced by controlling the process parameters of MA, such as milling balls size [13], duration [14,15] and ball-to-powder mass ratio [16]. Nonetheless, some challenges of this process can be listed:

- (1) High purity (>99%) of fine metal powder leads to high costs [17].
- (2) Some substances are doped in the alloy powder during the milling process due to the abrasion of milling balls and lining, reducing the purity of product [18].
- (3) Long processing times (20–80 h) are needed for obtaining a fine grain size, limiting the production rate [14,19,20].

To solve the above problems, a procedure for MA for Cu–Cr alloy powder preparation was developed in this work employing a modified mill, based on the following innovations:

- (1) A precursor, Cu(OH)₂ and Cr(OH)₃, obtained by co-precipitation from copper- and chromium-bearing inorganic salts was used to substitute the high purity fine metal powder as raw material, lowering costs.
- (2) Milling balls and jar linings were made of copper, and a protective nitrogen atmosphere was applied during the whole process to guarantee the purity of Cu–Cr alloy powder.
- (3) The traditional vibratory and planetary mills with common structure were equipped with heating and automatic temperature-controlled elements.





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Aiming at the preparation of Cu–Cr alloy powder with high purity and fine size, a comparison of different types of equipment (muffle furnace, traditional mill, modified vibratory mill and modified planetary mill) and experimental conditions, including reducing agent (active carbon powder and graphite powder), temperature (275, 300, 325 and 350 °C), duration (1, 2, 3 and 4 h) and the ball-to-powder mass ratio (10:1, 15:1 and 20:1), were investigated in the present study.

2. Experimental

Among the various Cu–Cr alloys, 75 wt% of Cu and 25 wt% of Cr, often referred to as CuCr25, is a very popular electrical material with excellent properties [21]. Therefore, the CuCr25 alloy powder was the target product in this exploration study.

2.1. Preparation of precursor

Analytical grade reagents including copper nitrate, chromium nitrate, ammonium hydroxide and absolute alcohol were used to produce precursors by co-precipitation. According to the weight ratio 3:1 of copper and chromium in the finial alloy powder, copper nitrate and chromium nitrate were weighed and dissolved in deionized water, after which ammonium hydroxide was added droplet-wise into the solution which was agitated with a speed of 0.3 rad/s at room temperature, to synchronously precipitate Cu^{2+} and Cr^{3+} as hydroxide by controlling pH in the range of 7–7.5. By this process, a uniform mixing of the Cu(OH)₂ and Cr(OH)₃ can be achieved on the molecular scale. Hydroxide precipitate as the precursor was separated from the solutions by filtration and water washing and alcohol washing.

2.2. Preparation of Cu-Cr alloy powder

To produce the Cu–Cr alloy powder, the precursor needs to be processed by roasting, reduction and MA for the transformation of hydroxide to metallic oxide and then to alloy powder. The whole process from precursor to Cu–Cr alloy powder was conducted in a modified vibratory mill or planetary mill, illustrated in Fig. 1(a) and (b), respectively. 100 g dry precursor powder, 1.2 times the theoretical mass for reducing agent and copper milling balls with three sizes of 1, 10 and 50 mm were poured into a copper jar followed by sealing of the mill and pure nitrogen (>99.99%)

injection with a very low speed. After 10 min, gas injection was stopped and two copper sticks were employed in planetary mill to plug the two holes of jar, giving the milling process an airtight environment to avoid some particles flow out with the gas leading a loss. When the temperature reached the set point, milling was started and maintained for the remaining process. Products were analyzed by X-ray diffraction (XRD), inductively coupled plasma (ICP), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and laser particle size analyzer for composition, crystallinity and grain size. A flow sheet of the process route is given in Fig. 2.

With the goal to study the effect of MA on the transformation temperature from precursor to metallic oxide, a series of roasting tests was carried out in the muffle furnace, using detection methods of XRD and thermogravimetric and differential thermal analysis (TG–DTA) to analyze the products.

3. Results and discussion

3.1. Effect of MA on the decomposition temperature of precursor

Cu(OH)₂ and Cr(OH)₃ can be decomposed into metallic oxide and water vapor by roasting. TG–DTA analysis of precursor obtained by co-precipitation showed no further mass loss after temperature reached about 470 °C, implying that the decomposition was completed at this temperature. Roasting experiments of precursor using the muffle furnace (at 400, 500 and 600 °C) and the modified vibratory mill (at 200, 300 and 400 °C) were carried out for 2 h respectively, followed by XRD analysis of the product. Results shown in Fig. 3(a) indicated that the principal products roasted in the muffle furnace were CuO and CuCr₂O₄. Moreover, the diffraction peaks of the two metallic oxides intensified with temperature while other diffraction peaks exhibited an opposite trend. Furthermore, the precursor decomposition was nearly completed if the process was conducted at 500 °C.

Fig. 3(b) shows the XRD patterns of the products roasted in the modified vibratory mill at different temperatures. A diffraction pattern analogous to the sample roasted in the muffle furnace at 500 °C was obtained when the precursor was roasted in the



Fig. 1. Illustration of the modified mills used in the study. (a) Vibratory mill: 1 – nitrogen inlet; 2 – gas outlet; 3 – sealed copper cap; 4 – slide track; 5 – insulating layer; 6 – heating elements; 7 – copper jar; 8 – buffer spring; 9 – crankshaft connecting rod; 10 – stainless stand; 11 – eccentic wheel; 12 – centrifugal machine. (b) Planetary mill: 1 – nitrogen inlet; 2 – sealing ring; 3 – bearing; 4 – chain wheel; 5 – flange; 6 – insulation layer; 7 – heating elements; 8 – sealed copper cap; 9 – copper jar; 10 – centrifugal machine; 11 – stainless stand; 12 – gas outlet.



Fig. 3. XRD patterns of the products obtained at different temperatures in the (a) muffle furnace, and (b) modified vibratory mill.

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modified vibratory mill at 200 °C for the same time, showing the strong effect of the milling. This influence on MA was also mentioned by Liu [22], who attributed the decrease of reaction temperature to a lower activation energy, while Nikaido et al. proposed that the structural change from crystalline state into amorphous state induced by grinding was the significant factor [23]. It was also found that the diffraction peaks of CuO and CuCr₂O₄ became high when temperature increased from 200 to 400 °C, but narrowed when temperature reached 400 °C. This reduction of peak width represented by full wave at half maximum (FWHM), which can be determined by XRD analysis, means that the grain size of the products increased while the lattice strain decreased [24].

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Fig. 4 shows SEM images of the products roasted in the modified vibratory mill at 300 and 400 °C. Uniform spherical metallic oxide powders without severe crystal agglomeration were obtained when MA was conducted at 300 °C, and a majority of the grain sizes of the powder observed in the image were smaller than 100 nm. At higher temperature, 400 °C, the grain size of the

product increased to nearly 500 nm with a morphological transformation from sphere into irregular shape. The particle size distribution of the product roasted in the modified vibratory mill at 300 and 400 °C determined by laser particle size analyzer is illustrated in Fig. 5, showing the particle size of product obtained at 300 °C to be mainly in the range of 60–100 nm coincident with the result from SEM analysis, while the size of particle increased to hundreds when tests were conducted at 400 °C. Therefore, it may be concluded that purified metallic oxides powder with high crystallinity and fine grain size can be prepared from precursor by roasting using the modified vibratory mill in the temperature range of 200–300 °C.

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3.2. Effect of milling equipment

The motion mode of the mill is a significant factor for the milling behavior and the end products as it influences the collision energy. Two kinds of motion mode, vibration and rotation,



Fig. 4. SEM images of the products roasted in the modified vibratory mill at 300 and 400 °C.



Fig. 5. Particle size distribution of the product roasted in the modified vibratory mill at 300 and 400 $^\circ\text{C}.$

respectively, were investigated though the employment of vibratory and planetary mills. Additionally, to clarify the effects of reaction atmosphere on the product, traditional milling tests without nitrogen protection using a zirconia jar and milling balls were carried out in the modified planetary mill, and more reducing agent was employed in these tests considering the influence of oxygen. Because more carbon was used for an adequate reduction in the current study, the reaction occurred in milling jars can be given as

$$Cu(OH)_2 + Cr(OH)_3 + C \rightarrow Cu - Cr + CO_2 \uparrow + CO \uparrow + H_2O \uparrow$$
(1)

Results showed that the products processed in the modified vibratory and planetary mill give a similar trend when it comes to temperature and duration, but the products were quite different from those from traditional mill products.

Fig. 6 shows XRD patterns of the different products using different equipment at 325 °C for 3 h. MA was conducted during the whole period of roasting and reduction, enhancing the solid solubility of copper and chromium. For this reason, chromium (atomic radius 0.126 nm) dissolved in copper (atomic radius 0.128 nm): This can be observed as absence of chromium diffraction peaks, which should appear at about 44°, 65° and 82° in all the XRD patterns. A Cu-Cr alloy powder was produced in the modified vibratory mill without any oxygen found by EDS and ICP analysis. demonstrating the high reaction efficiency of the equipment. However, under the same experimental conditions, the reduction reactions had not reached completion in the traditional tests, as can be seen from the distinct diffraction peaks of metallic oxide in the XRD pattern. Hence, the modified vibratory mill was used further to investigate the effects of experimental parameters on the results.



Fig. 6. XRD patterns of the products prepared by different mills.

3.3. Effect of reducing agent

Carbon powder, pulverized coal, carbon monoxide, hydrogen and glucose can be used as reducing agent for metallic oxide reduction. For the MA process for Cu-Cr alloy powder manufacturing above 200 °C, carbon powder is an excellent choice considering costs, safety, product purity, etc. In particular, it is worth mentioning that some residual carbon can improve the welding resistance and compressive strength of Cu–Cr alloys [25]. Therefore, the use of activated (amorphous) carbon powder and (crystal) graphite powder was investigated. The precursor and reducing agent were reacted at 325 °C for 3 h using the modified vibratory mill. Fig. 7 shows the XRD patterns of the products reduced by different reducing agents. Purified Cu-Cr alloy powder, (Cu + Cr) wt% = 98.67%, C wt% = 1.02%, other wt% = 0.31% as determined by ICP analysis, was obtained using activated carbon powder. For the sample reduced using graphite powder, no diffraction peaks of Cu-Cr were found in the XRD pattern, demonstrating the ineffectiveness of this reducing agent under the present experimental condition. Therefore, activated carbon must be considered a good choice of reducing agent.

3.4. Effect of temperature

As is seen from the experimental results given in Fig. 8, the products prepared by the modified vibratory mill at different temperatures for 3 h show various phase compositions. Some CuO and CuCr₂O₄ was found in the end products when the processes were conducted at 275 and 300 °C, proving the reducing reaction to be incomplete. The diffraction peaks of metallic oxide have



Fig. 7. XRD patterns of the products reduced by different reducing agents.



Fig. 8. XRD patterns of the products prepared at different temperatures.

disappeared in the XRD patterns of the powder produced at 325 and 350 °C, while the intensity presented a declining trend with temperature implying decrease of crystallinity. In addition to this, the MA process for the Cu–Cr system is inhibited by high temperatures because of the positive mixing enthalpy, as reported by other researchers [26]. Therefore, the reduction should be conducted at 325 °C to guarantee a fast and complete reduction reaction within a reasonable duration time.

3.5. Effect of process duration

A series of tests was carried out using the modified vibratory mill at 325 °C for 1, 2, 3 and 4 h to clarify the effect of the duration of the process on the product. The resulting XRD patterns of the products are shown in Fig. 9, clearly indicating that the Cu–Cr diffraction peaks heighten and broaden with duration but no notable changes were seen after the time exceeded 3 h. This demonstrated the significant influence of milling duration on the alloy crystallinity and grain refinement by the continuous energy supply from collisions. Fig. 10 illustrates the particle size distributions of the products prepared after different durations, showing that particle size decreased from hundreds of nanometers to less than one hundred nanometers during the MA process. Milling longer than 3 h cannot give a further refinement effect of particle size of the alloy powder.

In the field of materials science, the Scherrer formula is a simple way to estimate the grain size from the value of FWHM, here denoted by β (expressed in radians)

$$D = k\lambda/\beta\cos\theta$$



Fig. 9. XRD patterns of the products prepared after different durations.



Fig. 10. Particle size distribution of the products prepared after different durations.

where *D* is the average grain size (nm), k = 0.89 is the Scherrer constant, $\lambda = 0.154056$ is the wavelength of the X-ray and θ is the diffraction angle (rad). Fig. 11 shows the mean particle size and the average grain size of the powders prepared by different durations, which were obtained by Fig. 10 and Scherrer formula, respectively.

A similarly declining trend of the two sizes with duration can be found in Fig. 11, but the values of the particle size were always larger than the grain size. For example, the average grain size was only about 21 nm, nearly 1/7th of the measured particle size when the milling tests were conducted for 3 h at 325 °C in the modified mill. The reasons for this difference are the crystal agglomeration created by cold welding, the large surface free energy and the strong surface adsorption during the MA process [17]. Fig. 12



(2)

Fig. 11. Mean particle size and average grain size of the products prepared by different durations.



Fig. 12. SEM image of a Cu-Cr alloy grain aggregation.

shows the crystal agglomeration phenomenon in a SEM image of an aggregation constituted of many small Cu–Cr alloy grains. As for this aggregation, the Scherrer grain size represents the average size of the small alloy grains while particle size refers to the whole aggregate. This agglomeration usually appeared in MA process has a negative effect on material performance, thus a solution for this problem has yet to be found, such as using dispersing agent in the MA process.

The problem of impurity doping caused by the abrasion of milling balls and jar linings can be completely eliminated by the use of copper material in these MA parts, and nitrogen protection. Compared with the traditional MA processes conducted at room or lower temperature, experiments carried out at 325 °C using the modified mill drastically shortened the reaction time from dozens of hours to a few, giving a substantial increase in the production rate.

3.6. Effect of ball-to-powder mass ratio

The collision efficiency highly depends on the ball-to-powder mass ratio in the limited space of the mill. Three ratios 10:1, 15:1 and 20:1 were investigated at 325 °C for 3 h in the modified vibratory mill. The various sizes of balls can provide an adequate smash of different amplitudes and collision intensities to mix the two metals. XRD patterns of the three product samples shown in Fig. 13 reveal that a ball-to-powder mass ratio of 15:1 gives the highest diffraction peaks for Cu–Cr and the maximum FWHM compared with the results for lower and higher ratios. In the tests



Fig. 13. XRD patterns of the products prepared by different ball-to-powder mass ratios.

with the ratio 10:1, the precursor powders did not get enough energy from the collisions, and when the ratio was 20:1, the mean free path of the particles was decreased by the milling balls and this spatial restriction lowered the MA process efficiency. The weigh for milling balls was also conducted before and after milling process and no change could be found in all tests.

4. Conclusions

The preparation of Cu–Cr alloy powder by mechanical alloying (MA) in a modified mill was studied in this paper. For reasons of economy and efficiency, $Cu(OH)_2$ and $Cr(OH)_3$ obtained by co-precipitation from inorganic salts were used to substituting high purity fine metal powder as raw material, and the mill was innovatively modified in several ways to improve its action. Copper material was used in the milling balls and jar linings to prevent doping, a nitrogen atmosphere was applied to prevent oxidation and the mill was equipped with heating elements. The results showed that the preparation temperature of Cu–Cr alloy powder could be much lower in the modified mill compared with the conditions in the traditional equipment, while no distinct difference can be found in the products prepared by different equipment of vibratory and planetary mills.

The influence of several experimental conditions on the product was investigated. Activated carbon powder was found to be an effective reducing agent for copper- and chromium-bearing metallic oxides, while graphite powder did not exhibit desired reduction behavior in the current study. Purified Cu–Cr alloy nano-scale grains were prepared by MA in the modified mill and the optimal conditions were found to be a temperature of 325 °C, a duration of 3 h and a ball-to-powder mass ratio of 15:1. The duration of this process required was just about a tenth of the traditional approach, significantly enhancing the production rate. However, crystal agglomeration appeared in the product resulting in an increase of the powder size. This issue will be a problem to focus on in further studies.

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