An investigation on morphology and structure of Cu–Cr-Al_2O_3 powders prepared by mechanical milling

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Abastract:

In this study, the effect of the milling time on microstructure of Cu-0.5 wt.% Cr-1 wt.% $Al_2O_3$was investigated. The lattice parameter was evaluated by the X-ray diffraction technique. The microstructure was characterized by scanning electron microscopy. Also, size and distribution of Cr and Al_2O_3 were investigated by using X-rays energy dispersive (EDX) mapping. The result shows that the lattice parameter of the Cu phase increases with increasing milling time. The changing trend of Cu particle sizes as the alloying time increases, the particle sizes first increase and then decrease. When milling 1.5h, the powders coarsen than as-received Cu powder. Then the particle sizes of the samples decreased when the milling time increased to 3h and above. In addition, the size and distribution of Cr and Al_2O_3 decreased and dispersed much fine with increasing milling time.

Keywords: Cu- Cr- Al_2O_3 powder, microstructure, milling time, lattice parameter, particle size

1. Introduction

In engineering applications, the need for materials with high conductivity and high strength is increasing [1, 2]. Since Cu metal matrix materials are expected to have special physical and mechanical properties, studies on the synthesis and characterization of Cu metal matrix composites have attracted increasing interest. Al_2O_3 [3–6] particles and Cr precipitates reinforced Cu have been investigated for their excellent properties.

Cu–Cr alloys with high electrical and mechanical properties can be obtained when fine Cr precipitates present in the Cu matrix due to high dissolution of Cr in Cu, while coarse undissolved Cr particles may lead to deteriorated properties [7, 8]. Hence, attainment of high solid solubility of Cr in Cu is of interest. However, the Cu–Cr system shows a limited solubility range at equilibrium state according to the equilibrium phase diagram [9]. Mechanical alloying (MA), as a non-equilibrium processing method, can be used to extend the range of solubility in immiscible binary systems and produce alloys that are difficult or impossible to produce by conventional melting and casting techniques [10,11]. In addition, copper-based composites with nano-scaled grain structure and fine dispersion of Al_2O_3 particles can be produced by high-energy milling [12–14], which can attained better properties than pure copper and precipitation or solid-solution hardened copper.

Rencently, most of investigations have focused on Cu–Cr and Cu–Al_2O_3 powders by mechanical milling. Very limited studies have considered on Cu–Cr-Al_2O_3 powders. It is of great value to study to produce refined Cu–Cr-Al_2O_3 powders. In this study, refined Cu–Cr-Al_2O_3 powders were prepared by mechanical milling. The aim of present work is to study the effect of milling time on morphology and structure of Cu–Cr-Al_2O_3 powders.

2. Experimental

Commercially pure powders (≥99%) of Cu and Cr as well as Al_2O_3 with a particle size smaller than 75 μm were used as starting materials. Cu–1wt.% Cr–1wt.% Al_2O_3 alloy powders were prepared by mechanical milling was conducted in a high-energy ball mill (SPEX SamplePre 8000M Mixer/mill) at a milling speed of 875 rpm. More specifically, 10 g of Cu- Cr- Al_2O_3 and 100 g of steel milling balls (8 mm in dia.) were put into a cylindrical steel jar. The jar was evacuated and then filled with Ar gas. 80 wt.% ethanol was also used as the process control agent (PCA) in the milling.

The structural evolution of the milled powders was characterized by X-ray diffraction (XRD) using a PANalytical B.V X'Pert Pro PW3040/60 X-ray diffractometer with Cu Kα radiation (λ = 0.15418 nm). The true lattice parameter of the specimen was determined by a least square regression of the values calculated from each reflection against cosθcotθ, taking lattice parameter as the intercept of the regression line [15], and the instrumental broadening was eliminated by using the XRD pattern of strain free pure Si powders. The morphologies of the milled powders were characterized by scanning electron microscopy .The particle size of powders was obtained by measuring the dimensions of at least 100 particles in several micrographs for each sample.

3. Results and discussion

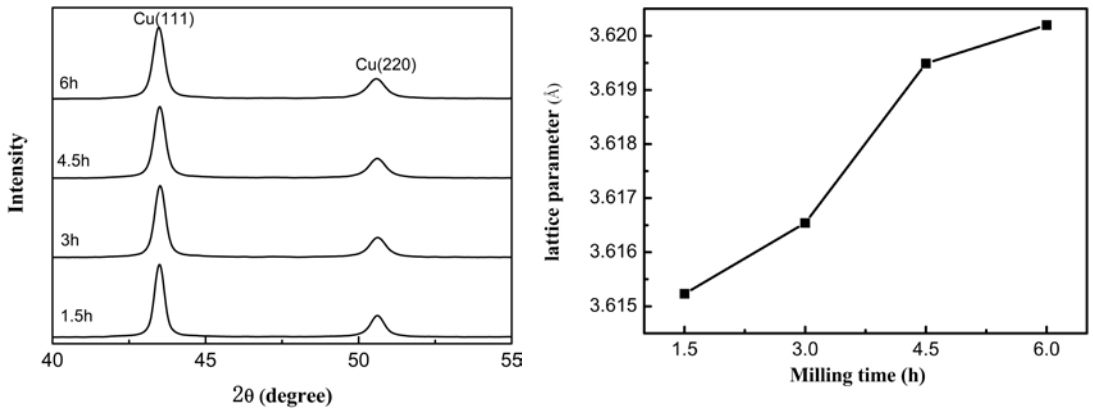

FIGURE 1. XRD patterns of Cu–1wt.% Cr–1wt.% Al_2O_3 and calculated values of lattice parameter milled for various times.

Fig. 1 shows XRD patterns of Cu–1wt.% Cr–1wt.% Al_2O_3 samples and calculated values for lattice parameter milled for various times. It can be noticed that the Cu peaks shifted to the lower-angle side and the full width half maximum (FWHM) increased by increasing the milling time. It should be noted that Al_2O_3 and Cr reflections could not be detected in the milled powder mixture, maybe due to its small quantity. This may also due to that Cu has been dissolved in Cr and a solid solution has formed. In order to understand if the Cu–Cr solid solution has been formed, the copper lattice parameter was determined.

The lattice parameter of the Cu phase increases with increasing milling time. The lattice parameters of the Cu-matrix were found to be 3.6202 ± 0.0001 Å for the samples of 6h milled, and it is 3.61523± 0.0001 Å for the 1.5h milled Samples. It should be noticed that the atomic radius of Cu is 0.1278 nm, the atomic radius of Cr 0.1249 nm [16]. The smaller Cr atoms are dissolved in the Cu phase and the inter-planar distance and the lattice parameter of Cu should decreases. While the lattice parameter change calculated from the XRD peaks is quite high and positive in the milled samples. Additional reasons for the change in the lattice parameter are manifold. In Ref. [17], an increase in the lattice parameter in nano-structure materials by various synthesis methods has been observed. It was related to the existence of rather large strains in nano-structure materials due to dislocations and severely distorted grain boundaries. The similar result has been obtained in other literatures [18].

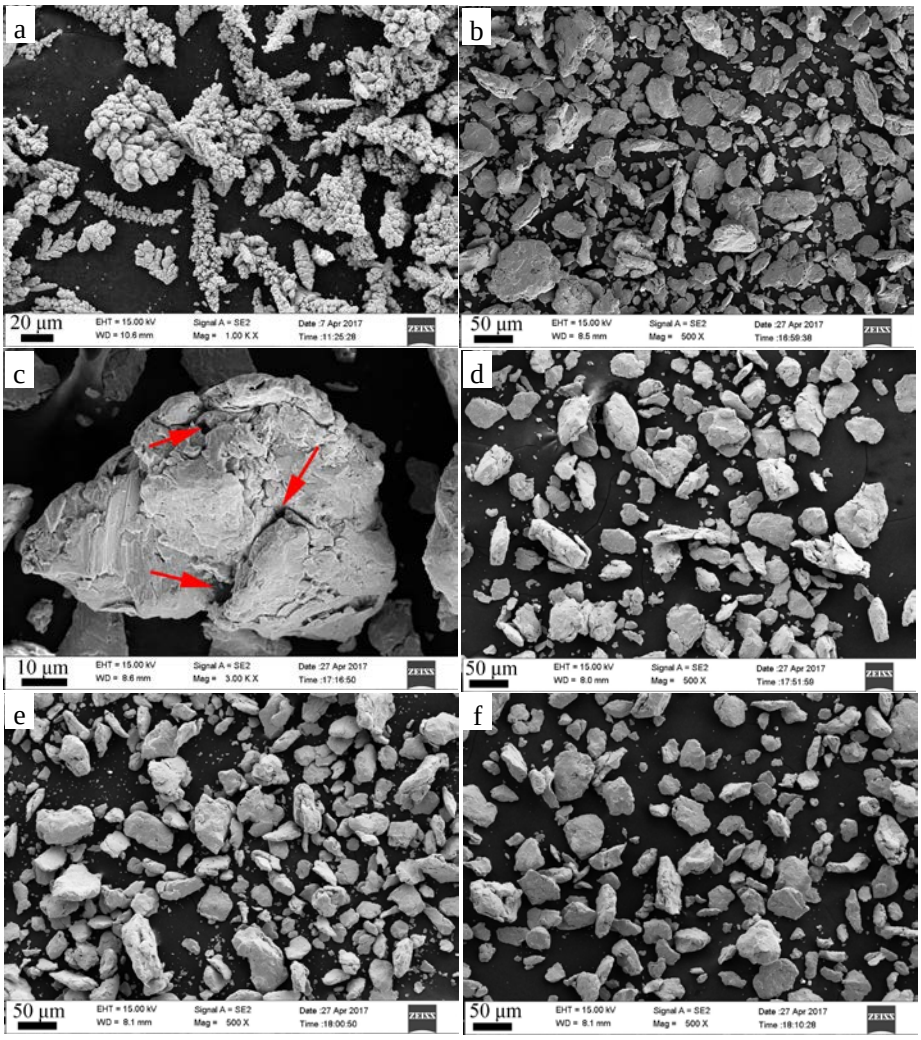

FIGURE 2. SEM micrographs of Cu-1 wt.% Cr-1 wt.% Al_2O_3 samples milling for various times (a) as-received mixed powder (b) (c) 1.5h (d)3h (e) 4.5h (f) 6h

Fig. 2 shows the SEM micrographs of Cu-1 wt.% Cr-1 wt.% Al_2O_3 samples milling for different times. It is clear that the as-received Cu powders have branch-like appearance with a rough surface, as is shown in Fig. 2(a), following with changing to flaky after milling 1.5h， as seen in Fig. 2(b), it is observed that the powders coarsen than as-received Cu powder, which may be caused by the aggregation of the soft Cu particles. When the milling time increased, the hardness of Cu is increased rapidly due to the smaller crystallite size and higher density of structural defects, therefor the effect of

cold welding be weaken. So, the particle sizes of the samples decreased with increase milling time when the milling time increased to 3h and above. In mechanical milling process, Cr, Al_2O_3 particles can act as grinding aids, a few are incorporated into each of the Cu particles and they during the alloying process. If the metal hardness are significantly different, the particles of the harder metals will tend to be encapsulated into the softer ones [19,20].So, Cr and Al_2O_3 as hard particles can gets further deformed, fractured and followed with embedding into the soft Cu particles as the process of milling continues. Then Al_2O_3 and Cr can be dispersed in between the Cu layers and located inside the Cu powders. In the meantime, the interlamellar spacing decreases, and the un-dissolved brittle particles get uniformly dispersed in the ductile matrix [21, 22]. Therefore, it will lead to a further increased in Cu particles and fracturing becomes the main process. The fragmentation has been characterized by a large number of cracks forming, which was indicated by red narrow in Fig. 2(c). These cracks can grow rapidly in the Cu particles, leading to fine particles with an irregular shape.

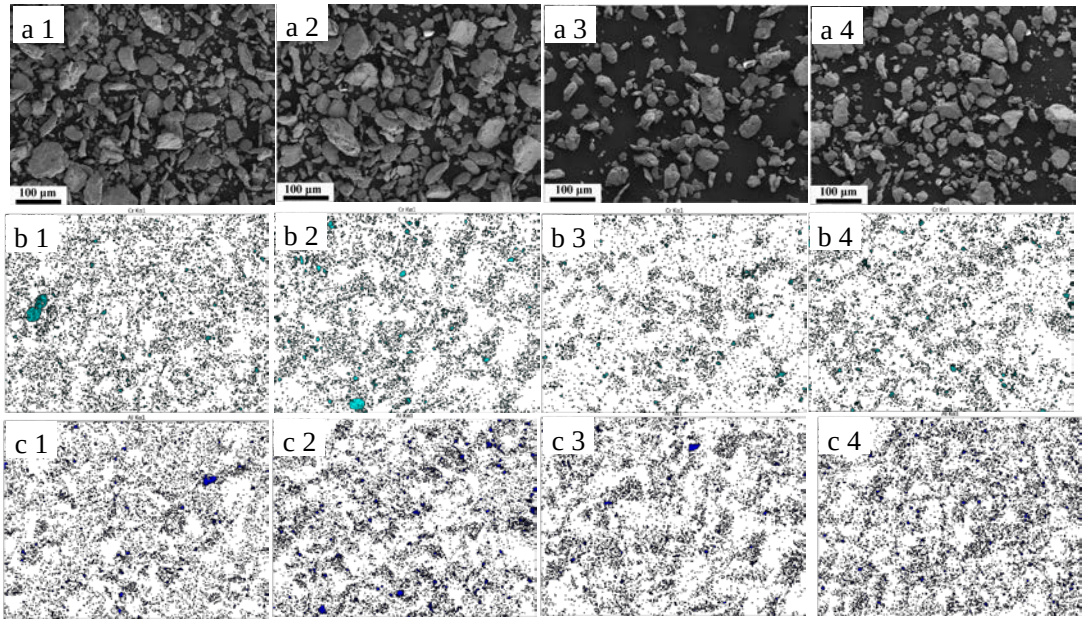

FIGURE 3. Typical SEM images of Cu-1 wt.% Cr-1 wt.% Al_2O_3 under different milling time (a 1)1.5h (b)3h （c）4.5h (d) 6h and the corresponding SEM–EDX mapping of (b) Cr and (c)Al

Using X-rays energy dispersive (EDX) mapping confirmed that the size of Cr and Al_2O_3 decreased and dispersed much fine with increasing milling time, as is shown in Fig 3. It should be noted that the distribution of oxygen element and aluminium element were highly identical. We use the Al element to characterize the distribution of Al_2O_3.

4. Conclusion

Cu–1wt.% Cr–1wt.% Al_2O_3 alloy powders were prepared by mechanical milling in a high-energy ball mill for various times. The effect of milling time on morphology and structure of Cu–Cr-Al_2O_3 powders were investigated. The following result could be obtained.

(1) The lattice parameter of the Cu phase increases in Cu-1 wt.% Cr-1 wt.% Al_2O_3 with increasing milling time.

(2) Because of aggregation of the soft Cu particles, the size of powders increased when milled 1.5 h. When the milling time increased to 3h and above, the particle sizes of the samples decreased with increase milling time.

(3) The size of Cr and Al_2O_3 decreased and dispersed much fine with increasing milling time.

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