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EFFECT OF PROCESSING CONDITION AND COMPOSITION ON THE MICROHARDNESS OF Cu-(2.5-10)vol.%Al_2O_3 NANOCOMPOSITE POWDER PARTICLES PRODUCED BY HIGH ENERGY MECHANICAL MILLING

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Nanostructured Cu-(2.5-10vol.%)Al_2O_3 nanocomposites were produced using high energy mechanical milling. For the as-milled Cu-Al_2O_3 composite powder particles having Al_2O_3 volume fractions of 2.5% and 5%, the increase in average microhardness is significant with the increase of milling time from 12 hours to 24 hours. With the increase of the content of Al_2O_3 nanoparticles the microhardness increases and in the range of 255HV-270HV. The milled nanocomposite powders were heat treated at 150, 300, 400 and 500°C for 1 hour, respectively, to determine the thermal stability of the powder particles as a function of annealing temperature. The average microhardness increased/decreased for the Cu-Al_2O_3 composites after annealing at 150°C due to the dislocation density, while increasing the annealing temperature to 300°C and 400°C the average microhardness almost remained linear. Further increasing the annealing temperature to 500°C causes significant decrease in average microhardness due to reduction in dislocation density and coarsening of Cu grains of the Cu-Al_2O_3 composite powders produced after 24 hours of milling. This paper is to report and discuss the changes of the microhardness of the material, caused by the compositions and processing conditions, used to fabricate the Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powders.

Keywords: processing conditions, high energy mechanical milling, copper matrix composites.

1. Introduction

Dispersion-strengthened materials are being developed to meet the increasing demand for materials that perform well in severe environments, with enhancement of mechanical properties such as strength and hardness at elevated temperatures.[1-3] The aim of this work is to produce dispersion-strengthened copper by applying the method of mechanical milling. Mechanical milling is a complex process involving optimization of a number of process variables, such as milling time, milling atmosphere, nature and amount of processing control agent (PCA), etc, to achieve the desired product phase and properties. High energy mechanical milling (HEMM) has been widely used in producing nanostructured powders.[4-6] In using HEMM to produce ultrafine and nanostructured powders, one important material property parameter is the average microhardness of the

powder particles produced after milling and subsequent heat treatment. This parameter is easy to measure, and also shows very useful information in terms of the mechanical properties of the bulk materials produced from consolidation of the powder particles, the thermal stability of the powder particles and the compactability of the powder particles. Based on this consideration, we studied the effects of processing conditions and compositions on the microhardness of the Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced by HEMM.

2. Experimental Procedure

As starting materials, powders of Cu (99.7% pure; particle size<63μm) and Al_2O_3 (99.9% pure; average particle size~50nm) were used in producing the Cu-Al_2O_3 nanocomposites by HEMM. A hardened steel vial, stainless steel balls and a Restch PM 4000 planetary ball mill with a rotational speed of 400 rpm were used for the milling. The vial containing the balls and 100g of powder mixture was sealed in a glove box filled with high purity argon. Cu powder together with 0.6wt% steric acid as processing control agent (PCA) and Cu/Al_2O_3 powder mixtures with four nominal compositions: Cu-2.5vol.%Al_2O_3, Cu-5vol.%Al_2O_3, Cu-7.5vol.%Al_2O_3 and Cu-10vol.%Al_2O_3 were milled using two milling steps. In Step 1, the powder mixture was milled for 12 hours using 60 balls with a diameter of 12.5mm. In Step 2 follows Step 1 and was further milling for 12 hours using 12 balls with a diameter of 12.5mm and 6 balls with a diameter of 25mm. In both steps, the ball to powder weight ratio was 5:1. Between Step 1 and Step 2, the balls were changed in a glove box filled with high purity argon. In each step, the milling process was interrupted after milling for 6 hours to take a small amount of sample for analysis. The analyses and characterization of the samples were performed using standard materials characterisation techniques including x-ray diffractometry (XRD), transmission electron microscopy (TEM) and Vickers microhardness tester with a load of 25g and a loading duration of 20s.

3. Results and Discussion

Fig. 1 shows the average microhardness of Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced by HEMM after Step 1 and Step 2, respectively. Several observations can be made from the microhardness measurement of the powder particles of different compositions and milled for different times and under different conditions. With Step 1 of the milling, a very small increase in average microhardness was seen with increasing the content of the Al_2O_3 nanoparticles from zero to 2.5vol.%. However, increasing the volume fraction of Al_2O_3 nanoparticles from 2.5% to 5% caused a much more drastic increase of the average microhardness from 185HV to 225HV. This trend continued with increasing the volume fraction of Al_2O_3 from 5% to 7.5vol.%, while the increase of the microhardness virtually stopped (with the increase being from 256HV to 263HV) with further increasing the volume fraction of Al_2O_3 nanoparticles from 7.5% and 10%. After Step 2 of the milling which involved milling for a longer time under a more severe plastic deformation condition, the average

microhardness of the powder particles increased significantly from 200HV to 255HV with increasing the volume fraction of Al_2O_3 nanoparticles from zero to 2.5vol.%, as shown in Fig. 1. With increasing the volume fraction of the Al_2O_3 nanoparticles from 2.5vol.% to 5vol.%, the increase of average microhardness was relatively smaller. Surprisingly, with further increasing the volume fraction of Al_2O_3 nanoparticles from 5% to 7.5% or 10%, the average microhardness of the nanocomposite particles even decreased slightly to 265HV or 255HV. In examining the effect of increasing milling time and milling severity on the microhardness of the powder particles, it can be seen that with increasing the milling time from 12 hours to 24 hours, the increase in average microhardness was significant for the Cu-2.5vol.%Al_2O_3 and Cu-5vol.%Al_2O_3 composite powder particles[7]. The increase of average microhardness with the increase in milling time and milling severity was much smaller for pure Cu and Cu-7.5vol.%Al_2O_3 composite powder particles. Surprisingly, a decrease in average microhardness with the increasing milling time and severity associated with Step 1 and Step 2 milling was observed for the Cu-10vol.%Al_2O_3 composite powder particles.

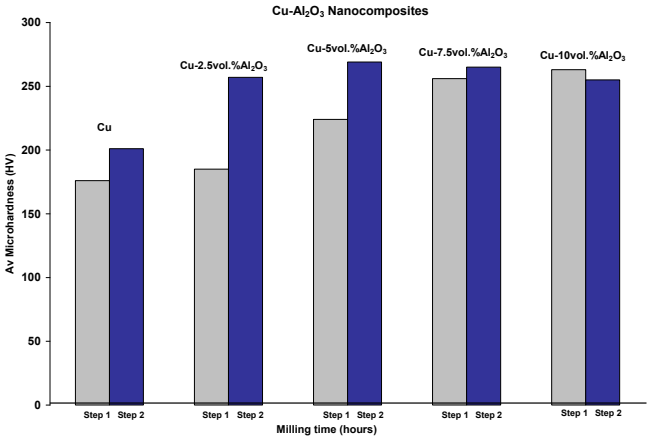

Fig. 1**:** The average microhardness of Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced by HEMM after Step 1 and Step 2 respectively.

Fig. 2 shows the change of the average microhardness of the Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced with Step 1 of HEMM as a function of annealing temperature. The average microhardness of the Cu powder particles produced with Step 1 decreased after annealing at 150°C, remained almost unchanged with increasing the annealing temperature from 150 to 300°C, and further decreased to 130HV with the increasing the annealing temperature from 300 to 400°C. Surprisingly, the average microhardness slightly increased with increasing the annealing temperature from 400 to 500°C. On the other hand, the average microhardness of the milled Cu-2.5vol.%Al_2O_3 composite powder particles first decreased slightly after annealing at 150°C, and then remained almost unchanged at 175HV with increasing the annealing temperatures from 150 to 500°C. This trend continues with milled Cu-5vol.%Al_2O_3 composite powder particles except that the average microhardness

decreased significantly with increasing the annealing temperature from 400 to 500°C. The average microhardness for the as-milled Cu-7.5vol.%Al_2O_3 composite powder particles remained almost unchanged at around 255HV after annealing at 150°C and with increasing the annealing temperatures from 150 to 400°C. While a sharp decrease in the average microhardness of the powder particles was observed with the increase of annealing temperature from 400 to 500°C. The change of the microhardness of the Cu-10vol.%Al_2O_3 composite powder particles followed a similar trend, except it decreased clearly from as-milled condition to being annealed at 150°C, as shown in Fig. 2.

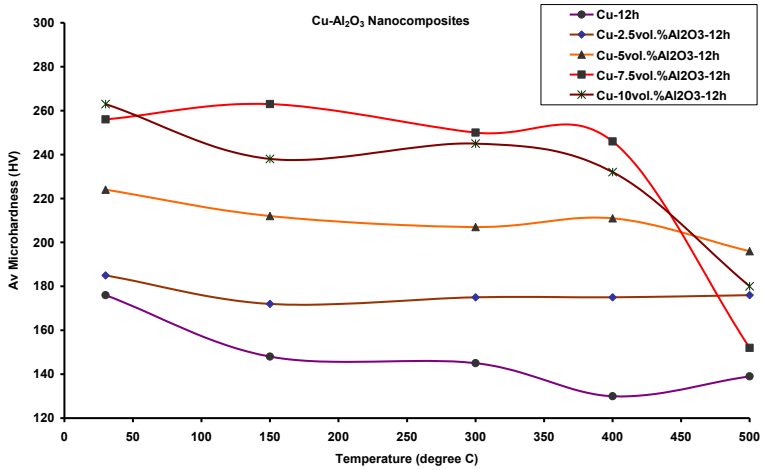

Fig. 2**:** Average microhardness of Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced with 12 hours of milling (Step 1) as a function of annealing temperature.

Fig. 3 shows the change of the average microhardness of the Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced after Step 2 of milling, as a function of annealing temperature. An increase in average microhardness was observed for the Cu powder particles produced with Step 2 after annealing at 150°C. Further increasing the annealing temperatures from 150 to 500°C, the average microhardness showed a decreasing trend. The average microhardness for the as-milled Cu-2.5vol.%Al_2O_3 composite powder particles produced with Step 2 of milling remained unchanged after annealing and with increasing the annealing temperatures from 150 to 300°C, but decreased sharply from 250HV to 225HV with further increasing the annealing temperature to 400°C.[8] The average microhardness remained unchanged with increasing the annealing temperature from 400 to 500°C which means that the trend of the microhardness change of Cu-2.5vol.%Al_2O_3 with increasing annealing temperature was almost the same as that of milled Cu powder particles. However, with 5vol.% of Al_2O_3 nanoparticles in the powder particles produced by Step 2 of milling, the average microhardness decreased almost linearly and remained unchanged with increasing the annealing temperatures up to 300°C and 400°C, respectively, but decreased sharply with increasing the annealing temperature from 400 to 500°C. The trend of the change of the average microhardness of the milled powder particles of Cu-7.5vol.%Al_2O_3 or Cu-10vol.%Al_2O_3 composites produced with Step 2 with annealing temperature was similar to that of the milled Cu-5vol.%Al_2O_3 composite powder particles, except that the

magnitude of the microhardness decrease with increasing annealing temperature from 400 to 500°C was much greater, as shown in Fig. 3.

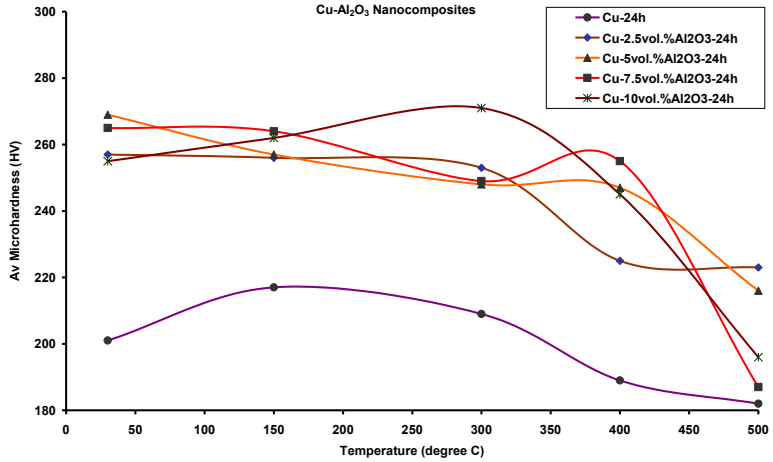

Fig. 3**:** The average microhardness of Cu powder particles and Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced with 24 hours of milling (Step 2) as a function of annealing temperature.

Fig. 4 shows the TEM bright field images of Cu-(2.5-7.5)vol.%Al_2O_3 nanocomposites produced with Step 1. With the increase of the volume fraction of Al_2O_3 nanoparticles from 2.5% to 5%, the grain size of the Cu grains had decreased from 100-500nm to 100-250nm. While further increasing the volume fraction of Al_2O_3 nanoparticles to 7.5%, majority of the Cu grains are less than 120nm. This reduction in grain size of Cu matrix increased the microhardness of the material, confirming the result as shown in Fig. 1.

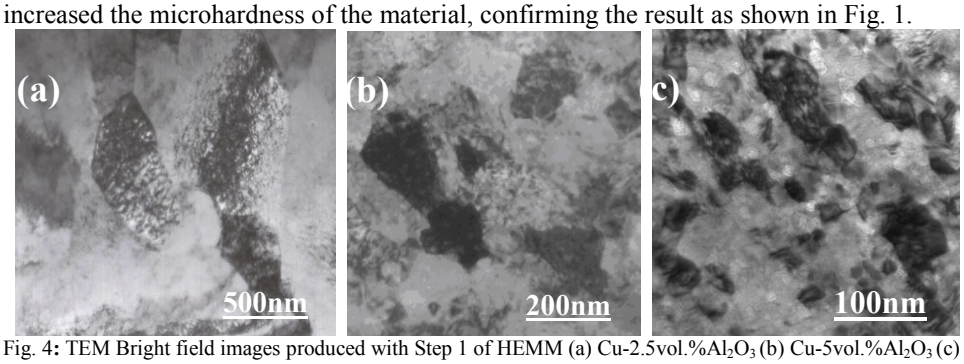

Fig. 4**:** TEM Bright field images produced with Step 1 of HEMM (a) Cu-2.5vol.%Al_2O_3 (b) Cu-5vol.%Al_2O_3 (c) Cu-7.5vol.%Al_2O_3.

TEM examination of as-milled Cu-5vol.%Al_2O_3 composite produced with Step 1 showed that with the increase of milling time from 12 to 24 hours, the grain sizes of the Cu matrix decreased,[7] which increases the average microhardness of the material, confirming the result as shown in Fig. 1. For the Cu-10vol.%Al_2O_3 composite produced with Step 1, TEM examination shows that grains of the Cu matrix decreased from the range of 50-120nm to 30-70nm due to recrystallisation after annealing at 150°C, resulting in formation of finer grains. However, this refinement of grains does not cause increase of the microhardness. Instead, the microhardness of the annealed sample become lower,

as shown in Fig. 2, suggesting that in the as-milled condition, a substantial fraction of the high hardness is due to high dislocation density in the heavily cold worked material as shown in Fig. 5(a). For the Cu-10vol.%Al_2O_3 composite produced with Step 2 and after heat treatment at 500°C, the average microhardness decreased sharply due to the reduction of dislocation density, as shown in Fig. 3, suggesting coarsening of the Al_2O_3 nanoparticles occurred. More details on this investigation can be found in Ref. 9. It appears that the associated microhardness decrease is not significant for the Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced after Step 2 and after annealing at temperatures up to 400°C. This suggests that the nanostructured powder can be consolidated at temperatures around 400°C without totally losing the nanostructure.

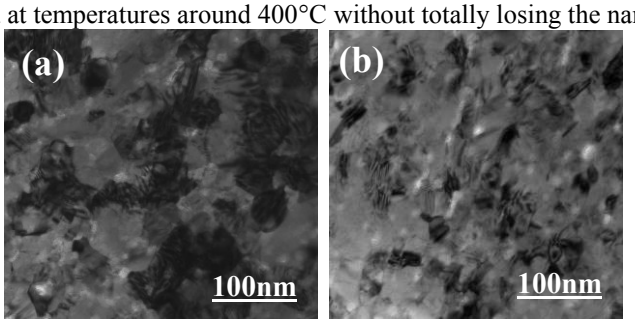

Fig. 5**:** TEM Bright field images of Cu-10vol.%Al_2O_3 produced with Step 1 of HEMM after annealing at 150°C.

4. Conclusions

The average microhardness of Cu-(2.5-10)vol.%Al_2O_3 nanocomposite powder particles produced by HEMM after Step 1 is significant with increasing the volume fraction of Al_2O_3 nanoparticles from 2.5% to 5% and 7.5%. Effect of increasing milling time on the microhardness of the powder particles, from 12 hours to 24 hours, the increase in average microhardness is significant for the volume fractions of 2.5% and 5% of Al_2O_3 nanoparticles. The average microhardness decreased sharply for the Cu-(7.5-10)vol.%Al_2O_3 composites produced with Step 2 and after heat treatment at 500°C, mainly due to the reduction of dislocation density. These changes can be reasonably well explained by examining the microstructure of the powder particles with different compositions and produced under different conditions.

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