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Pool Volume Influence on Carbon Nanotube Dispersion in Cu-Cr Matrix by Wet Ball Milling

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1- Introduction

Copper matrix hybrid composites reinforced with ceramic and metallic precipitates, have received considerable interest in researches due to their favorable properties, namely good mechanical properties, high electrical and thermal conductivity. Among copper matrix composites, Cu-Cr is an in-situ composite. Chromium has a small solid solubility in copper matrix because of its positive heat of mixing which its formation results from decomposition of supersaturated Cu-Cr solid solution. On the other hand, carbon nanotubes (CNTs) are good candidates to be used as reinforcements in polymer, ceramic and metallic composites due to their valuable properties. So far, most of the investigations on CNT composites have been focused on polymer matrix composites and ceramic matrix composites compared to metal matrix composites because of difficulty in homogeneous dispersion of CNTs in the metal matrix due to their agglomeration, density mismatch and weak interface bonding with metallic matrices. Among different approaches for fabricating hybrid nano-composites, ball milling is a simple, low cost and high yield method. Also, it is an efficient way for producing CNT strengthened alloys due to the possibility of dispersing carbon nano-tubes as reinforcement elements in metal matrix composites. Despite these advantages, agglomeration and defects introduced on CNTs during ball milling have negative effects on their morphology, mechanical and physical properties. Wet milling in presence of a liquid phase is an efficient method that can be used to enhance the dispersion of CNTs in metal matrix composites. In this study, the preparation of Cu-1wt.%Cr-5wt.%CNT nano-composite by wet milling process at three different levels of milling energy and two different pool volumes was investigated and the effect of pool volume on the CNTs dispersion and mechanical property have been investigated.

2- Experimental

Starting materials used in this research were commercially pure Cu, Cr and multi-walled CNTs. It should be mentioned that CNTs were sonicated in ethanol for 90 minutes, to break up the CNT agglomerates. Powder mixtures were mechanically milled in a planetary high energy ball mill (PM2400) with hardened steel vial and balls under argon atmosphere. In the first step, Cu-1wt.%Cr solid solution was prepared as the matrix phase. The ball-to-powder weight ratio (BPR) and milling speed were 30:1 and 300 rpm, respectively. 1wt.% of toluene was used as a process control agent. In the second step, Cu-Cr solid solution alloy together with 5wt.% of CNTs was wet milled for 5 h. The milled samples were dried on hotplate for about an hour at 80^{o}C to evaporate the ethanol. Table 1 summarizes milling conditions of different samples. The structural evolution and solid solution formation were evaluated by X-ray diffraction technique. The microstructure was characterized by scanning electron microscopy. Also, the mechanical property was investigated by microhardness test.

Table 1 Milling conditions of different samples

Sample	Rotation speed (rpm)	BPR	Pool volume(ml)
WM1	200	10	10
WM2	200	10	25
WM3	300	10	10
WM4	300	10	25
WM5	300	30	10
WM6	300	30	25

3- Results and Discussion

According to the XRD patterns and the calculation of lattice parameter of Cu, it was concluded that Cu-1wt.%Cr solid solution was formed after 20 h. Based on the results of first milling step, 5wt.% of CNTs were added to 20 h milled Cu-Cr solid solution sample and milling was continued for 5 h in wet milling medium. To have a detailed study on the effect of milling conditions and pool volume on the Cu structure at the same time, the Cu mean crystallite size changes as a function of milling energy for different samples were calculated and are shown in Fig. 1. It can be seen that the mean crystallite size of samples decreases as the milling energy increases which is due to the formation of defects such as grain boundaries and dislocations in the structure. Also, the presence of well-dispersed CNTs can be another reason for this decrease. However, in wet milled samples at higher pool volumes, less energy is attributed to the changing in crystallite size which is

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weakened in the highest level of milling energy. FESEM images showed that wet milling at high levels of milling energy and less pool volume can be considered as the most efficient way to reach perfectly embedded CNTs. This result can be attributed to the fact that this level of milling energy is sufficient for dispersing of CNTs. Indeed, at low and medium levels of milling energy, higher pool volume can guarantee the dispersion of CNTs, but at higher levels of milling energy the effect of pool volume is weakened and it can be more effective on having less damaged imbedded CNTs in the composite matrix.

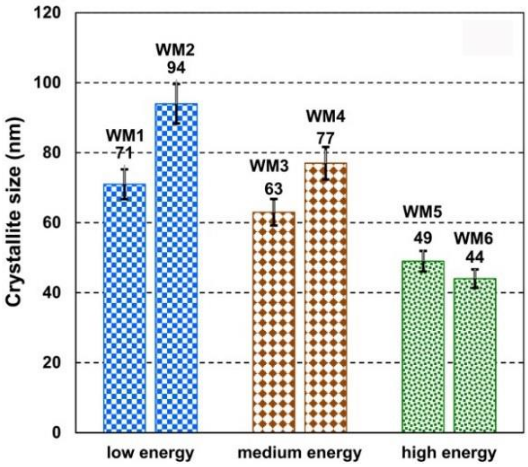

Fig. 1 Changes of crystallite size as function of milling energy for different samples

Fig. 2 shows the FESEM image of WM5 sample with the best dispersion of minimum damaged CNTs in comparison to other samples with different milling conditions.

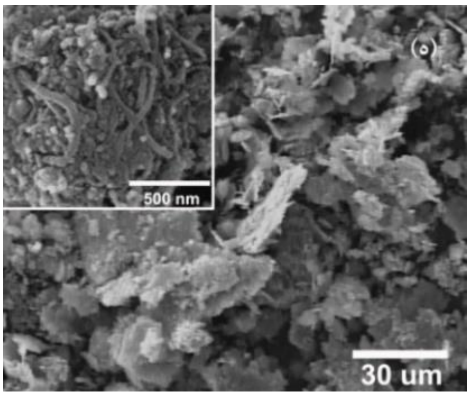

Fig 2 FESEM image of WM5 sample

The results of microhardness test (Fig. 3) corresponds to other results and the microhardness result verifies the best dispersion of CNTs in WM5 sample since this sample has the highest hardness.

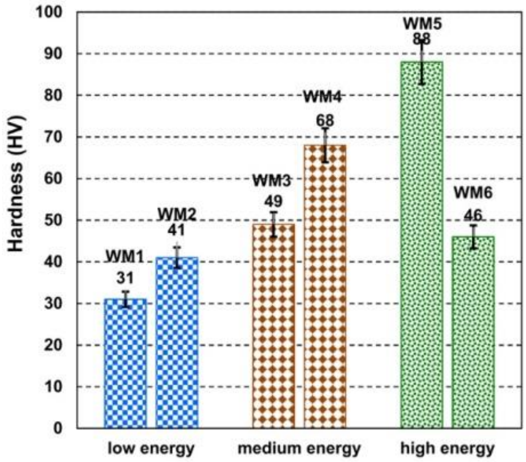

Fig 3 Microhardness values of different samples as a function of milling energy

4- Conclusions

Cu-1wt.%Cr-5wt.%CNT nano-composites by wet milling process at three different levels of milling energy and 10 ml and 25 ml of ethanol were fabricated. According to FESEM images which were in agreement with microhardness test results, wet milling at high level of milling energy and smaller pool volumes is an efficient way for dispersing CNTs while introducing less damage on them. Finally, it can be concluded that wet milling with controlled milling parameters can be an effective way for dispersing CNTs in Cu matrix composites.