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Characterization of Dispersion Strengthened Copper with 3wt%Al_2O_3 by Mechanical Alloying

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Abstract:
The copper matrix has been dispersion strengthened with 3wt.%Al_2O_3 by mechanical alloying. Commercial alumina powder with an average particle size of 0.75μm was used for alloying. The mechanical alloying process was performed in a planetary ball mill up to 20h in air. After milling all powders were treated in H_2 at 400^0C for 1h, and finally hot pressing was used for compaction (800^oC, 3h, Ar). Structure observations revealed a lamellar structure (Al_2O_3 particles largely restricted to interlamellar planes between adjacent copper lamellae) accompanied also by structure refinement. These structural changes were mostly completed in the early stage of milling, and retained after compaction. Micro hardness was found to progressively increase with milling time. So, after 5h of milling the micro hardness of the Cu+3twt%Al_2O_3 compact was 1540MPa, i.e. 2.5 times greater than for the as-received electrolytic copper powder (638MPa) compacted under identical conditions, while after 20h of milling it was 2370 MPa. However, after exposing the tested compact at 800^oC up to 5h, the achieved hardening effect vanished.
Keywords: *Dispersion strengthened copper, Mehanical alloying, Microhardness.*

1. Introduction

Dispersion strengthening is a well known procedure used for improving the hardness of metallic materials. Dispersoids can be introduced by a variety of methods but "Mechanical Alloying" was a breakthrough enabling obtaining of a homogeneous dispersoid distribution. The processing route involves high-energy ball milling of the starting powder mixed with a dispersoid. Attempts have been made to produce dispersion strengthened copper by mechanical alloying using alumina particles of various sizes [1, 2, 3]. The high-energy ball-milling powder process, through repeated fracture and welding of powder particles during ball-powder-ball and ball-powder-container collisions, enables achievement of a regular distribution of fine Al_2O_3 particles in the copper matrix. Such Al_2O_3 dispersoids contribute to the copper matrix strengthening providing resistance to dislocation motion according to Orowan's theory [4]. The oxide dispersion strengthened alloys, produced by mechanical alloying, are characterized by the presence of extremely small oxide dispersoids ranging from 30nm - 40nm, uniformly distributed in a very fine grain sized structure (<1μm in diameter) [4]. The aim of this work was to estimate whether commercial alumina particles with an average particle size of about 0.75μm could be broken down during milling and achieve the

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dispersion strengthening effect in Cu-Al_2O_3 alloys.

2. Experimental Procedure

Mixtures of electrolytic copper powder (average particle size: 30μm) with 3wt.% commercial alumina powder (average particle size: 0.75μm) were milled in air 3, 5, 10 and 20h in a planetary ball mill. The weight ratio of powders to steel balls was 1:35. After milling all powders were treated in hydrogen at 400^0 for 1h, in order to eliminate copper oxides. Compacts were obtained by hot pressing in an argon atmosphere at 800^0C for 3h under the pressure of 35MPa.

X-ray diffraction analysis, optical and scanning electron microscopy were used for characterization. X-ray diffraction was performed with a Siemens D-500 X-Ray Powder Diffractometer using CuK_α Ni-filtered radiation. The average lattice distortion and the area of coherent diffraction of X- rays were determined from the broadening of the first four X-ray lines (111, 200, 220 and 311) applying the Williamson and Hall approach [5, 6]. Copper matrix strengthening was assessed by micro hardness testing (50g).

3. Results and discussion

X-ray diffraction of Cu+3wt.%Al_2O_3 powder has shown progress in line broadening with milling time, as a result of severe crystal lattice distortion and grain structure refinement by fragmentation into domains determined by dislocations and possibly by stacking faults [5, 6]. Every domain basically represents the area of coherent diffraction of X-rays (ACD), while the crystal lattice distortion represents the average relative deviation of the lattice parameters from their mean value [5]. The effect of milling time on the ACD and lattice distortion of the examined powder is presented in Fig.1.

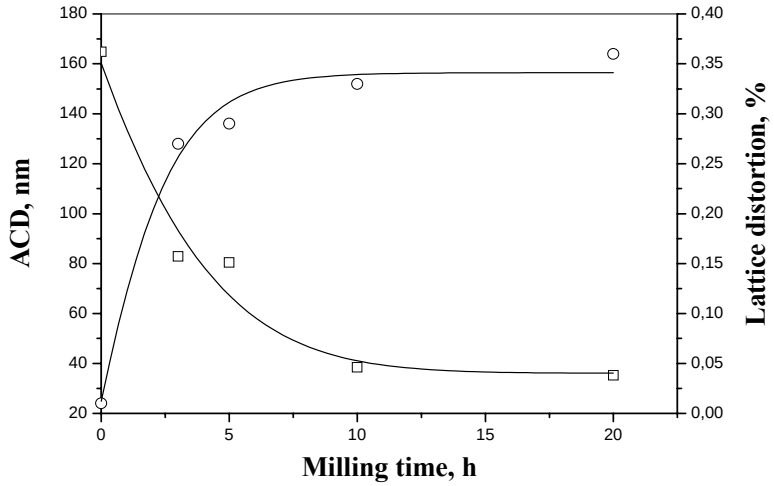


Fig. 1 The area of coherent diffraction of X-rays (ACD) and lattice distortion vs. milling time.O – ACD, □ - Lattice distortion.

As can be seen, the most intensive structure fragmentation occurs in the early stage of milling. In the period of 10 to 20h, the ACD remains practically constant ranging to 40nm. Since the increase of ACD during hot pressing is less than 60% [7], it is reasonable to expect

that the compacts are also characterized by expressed grain structure refinement. Fig.1 illustrates that lattice distortion strongly increases during 5h of milling, while after that it is less evident up to 20h that is quite in agreement with some earlier assumptions [5], that the deformation of particles occurs during the early stage of milling.

Fig.2 shows the mixture of Cu+3wt.%Al_2O_3 powder particles before milling (Fig.2a) and the development of Cu-2.5wt.%Al particle morphology with increasing milling time (Fig.2b-d). Fig.2 shows that 3h of milling time leads to particle coarsening, while 10 and 20h of milling followed with the formation of equiaxed particles. After 3h (Fig.2b) the milled powder particles are rather flattened as a result of strong plastic deformation occurring at the very beginning of milling.

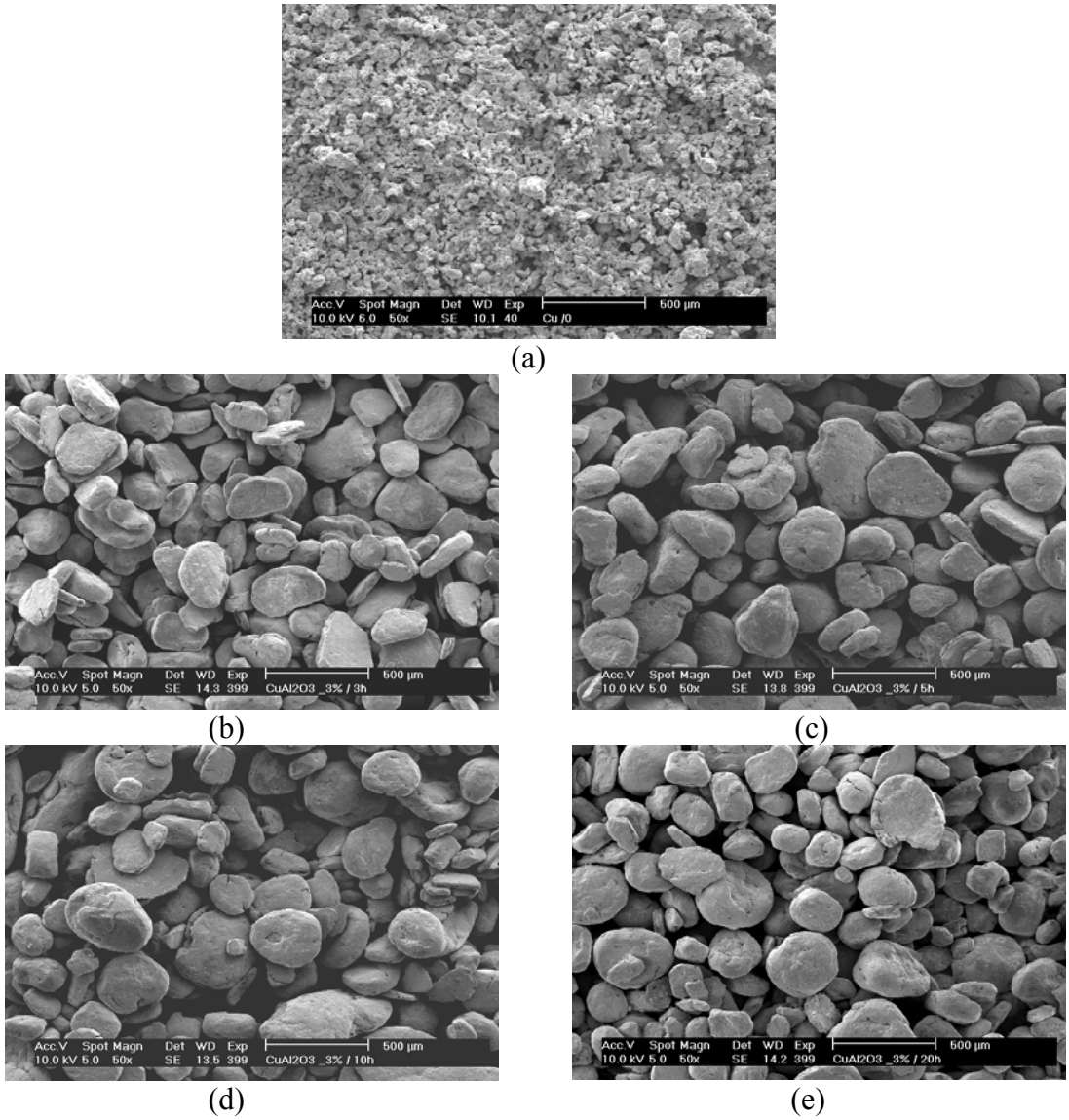
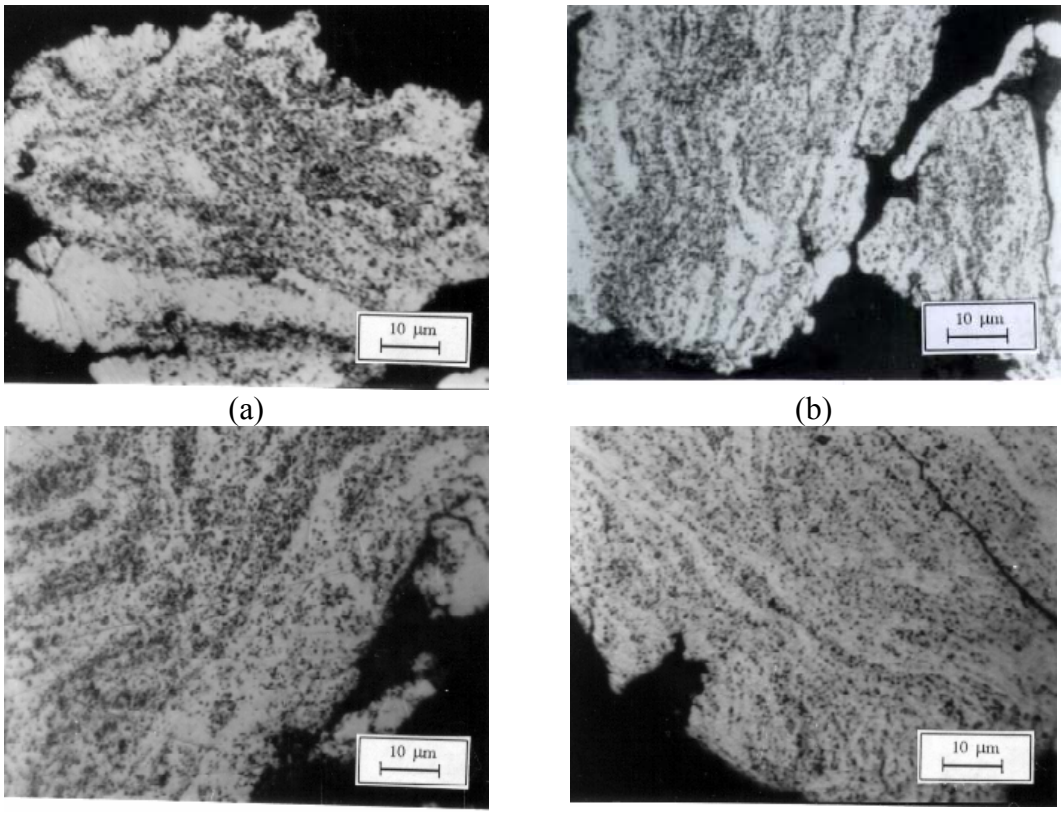


Fig. 2 Morphology of Cu+3wt%Al_2O_3 particles: a) before milling and after different milling times: b) 3h,c) 5h, d) 10h and e) 20h

The average particle size increases until the welding process dominates the milling process, and oppositely it decreases after the fracture process becomes dominant. In the case of balance of the fracture and welding processes the particles are rather uniform and equiaxed

[8]. Considering the particle morphology shown in Fig.2 it seems that a balance was not achieved after 20h of milling time, as particles of various sizes are present in the milled powder.

Detailed structure observations have shown that in most mechanically alloyed copper particles a very homogenous distribution of alumina is achieved after 3h of milling, and with increasing milling time it further improved. Distribution of alumina particles in the copper matrix is illustrated in Fig.3. Most alumina particles in Fig.3 were out of the range required for achieving the dispersion strengthening effect. Alumina particles within the range required for dispersion hardening, finer than 100nm [9], are hardly metallographically visible, but their presence was confirmed in an earlier study [10]. Jung-Ho Ahn *et al*. [11] also found fragmented alumina particles (initial size: 2μm) of 20-500nm embedded in the copper matrix after mechanical alloying.



(a) (b)

(c) (d)

Fig. 3 Distribution of alumina patricles in the copper matrix after different milling times: a) 3h, b)5h, c)10h and d)20h.

The powder particles undergoing milling not only changed in morphology, but particles coarsening occurred and their structure became typically lamellar with lamellae presenting individual plastically deformed particles. Such a lamellar structure is retained after compaction. The lamellar particles and compact structure after 5h of milling is illustrated in Fig.4.

The compact micro hardness measurement by a 50g load (Fig.5) confirms that mechanical alloying of copper with commercial alumina particle causes an increase in hardness. Mechanically alloyed copper shows considerably higher values of micro hardness ranging up to 2370MPa. The greatest increase in micro hardness occurred at the beginning of the milling process. After 5h of milling micro hardness of the compact (1540MPa) is 2.5

times greater than for the as-received electrolytic copper powder (638MPa) compacted under identical conditions.

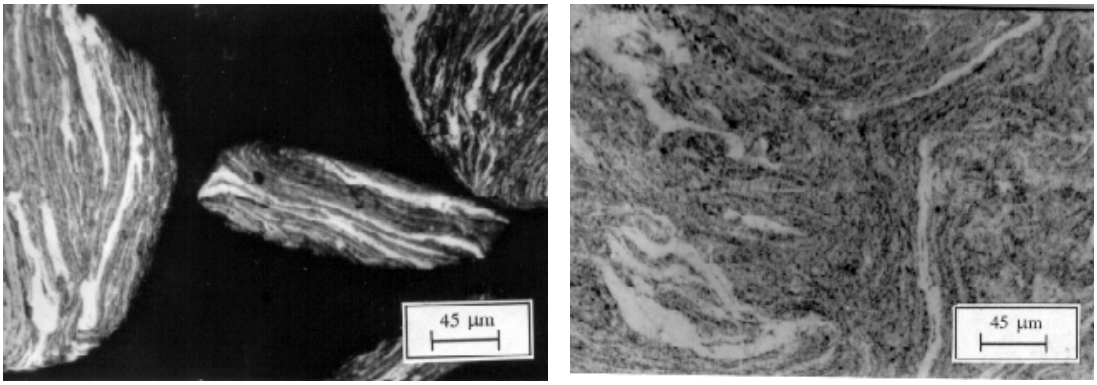


Fig. 4 Structure of Cu+3wt%Al_2O_3 particles (a) and compact (b) after 5h milling.

The results obtained (Fig.1 and Fig, 3) indirectly suggest that micro hardness increase of mechanically alloyed copper (Fig.5), is a consequence of achieving a regular distribution of alumina particles into the copper matrix during milling accompanied by grain structure refinement.

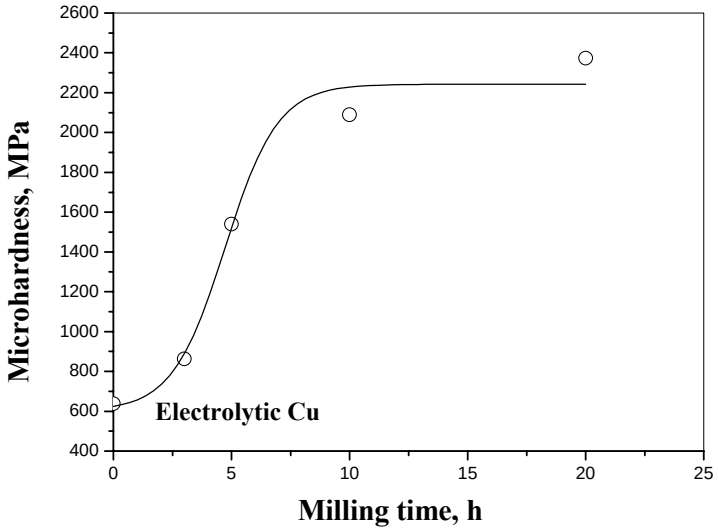


Fig. 5 Microhardness of Cu+3wt%Al_2O_3 compacts vs. milling time

The present results revealed that the alumina content of 3wt.% has a strong effect on strengthening of the copper matrix. This observation is similar to that made by Anish Upadhayaya and G.S. Upadhyaya [3] and J.G.S. Schroth and V.Franetovic [1]. They reported that increasing the content of alumina, up to 3wt% in the case of average particle size of 0.3 μm [3] or up to 9wt% in the case of particles ranged to 50 nm [1] effectively increased the hardness.

Hardness values of mechanically alloyed copper after heat treatment at 800^0C in argon for varying times are given in Tab. I. It is obvious that dispersion-strengthened copper, produced by mechanical alloying loses the relatively high hardness after high temperature

exposure. This is assumed to be due to the presence of mainly non-fragmented alumina particles in the copper matrix (Fig.4). Namely, having in mind their sizes, they can't drag the recrystallization and boundary migration [12] at elevated temperature. The number of fragmented alumina particles, found in mechanically alloyed Cu+3wt.%Al_2O_3 [9], is not enough for obtaining this effect. So, mechanically alloyed copper with 3wt.% commercial alumina particles, can't retain its properties after exposure to high temperatures.

Tab. I Microhardness of dispersion strengthened copper before and after high temperature exposure

Milling time	Microhardness, MPa		
	Before heat treatment	After heat treatment	
	Room Temperature, ^{0}C	800^{0}C/1h	800^{o}C/5h
5h	1540	716	549
20h	2440	942	726

4. Conclusion

During milling the powder mixture of Cu+3wt.%Al_2O_3 powder undergoes severe changes. Grain structure refinement takes place and fine Al_2O_3 particles achieve a regular distribution in the copper matrix. As a consequence, the micro hardness of mechanically alloyed copper increases. After 5h of milling microhardness of the compact (1540MPa) is 2.5 times greater than for the as-received electrolytic copper powder (638MPa) compacted under identical conditions. After exposing the mechanically alloyed copper at 800^{o}C up to 5h, the achieved hardening effect vanishes.

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***Резюме**: Основу меди дисперсионно упрочняли механическим легированием с 3об.% Al_2O_3. Для упрочнения использован технический порошок Al_2O_3 со средним размером частиц 0,75 мкм. Механическое легирование проводили в планетарной мельнице со стальными шариками в течение до 20 часов. Восстановление проводили в водороде при температуре 400 ^oC в течение 1ч. Порошки потом подвергали горячему прессованию (800^oC, 3ч, 34 МПа, Ar). Уже в начале измельчениа порошков произошло уменьшение зерен и образование слоистой структуры; частицы Al_2O_3 находились на поверхностя соседних слоев. Такая же структура частиц сохранилась и после прессованния. С продолжением времени измельчения микротвердость прессовок возрастает до 2370 МПа. После измельчения в течение 5 часов микротвердость прессовок Cu+3об.% Al_2O_3 (1540 МПа) увеличилась на 2,5 раз в отношении микротвердости прессовок из порошка электролитической меди, спрессованого при одинаковых условиах (638 HV). Однако, после нагрева прессовок при 800^oC эффект упрочнения теряется.*
***Ключевые слова**: Дисперсионно упрочненная медь, механическое легирование, микротвердость.*

***Садржај**: Основа бакра дисперзно је ојачана механичким легирањем са 3теж.,%Al_2O_3. За ојачавање је коришћен комерцијални прах Al_2O_3 просечне величине честица 0,75μт. Механичко легирање извршено је у планетарном млину са челичним куглама у трајању до 20h. После редукције (400^0C/1h, H_2), прахови су компактирани топлим пресовањем (800^0C/3h, Ar, 34MPa). Већ на почетку млевења прахова дошло је до уситњавања зрна и стварања ламеларне структуре, са честицама Al_2O_3 распоређеним по површинама суседних ламела. Ова структура честица праха задржала се и после компактирања. Са временом млевења микротврдоћа компаката расте до 2370MPa. После 5h млевења микротврдоћа компаката Cu+3теж.%Al_2O_3 (1540MPa) је 2,5 пута већа у односу на микротврдоћу компакта од праха електролитичког бакра комапктираног под истим условима (638HV). Међутим, ефекат ојачавања губи се након жарења на 800^oC.*
***Кључне речи**: Дисперзно ојачан бакар, механичко легирање, микротврдоћа.*