International Journal of Engineering

Journal Homepage: www.ije.ir

Investigation on Mechanical and Electrical Properties of Cu-Ti Nanocomposite Produced by Mechanical Alloying

R. M. Babaheydari, S. O. Mirabootalebi*, G. H. Akbari

Department of Material Science and Engineering, Shahid Bahonar University of Kerman, Iran

P A P E R I N F O

Paper history:
Received 25 January 2020
Received in revised form 02 August 2020
Accepted 02 August 2020

Keywords:
Copper-Titanium Alloys
Mechanical Alloying
Electrical Resistance
Mechanical Properties

A B S T R A C T

In this paper, Cu-Ti nanocomposite synthesized via ball milling of copper-titanium powders in 1, 3, and 6 of weight percentage compounds. The vial speed was 350 rpm and ball to powder weight ratio kept at 15:1 under Argon atmosphere, and the time of milling was 90 h. Obtained powders were studied by scanning electron microscopy (SEM), X-ray diffraction (XRD), and dynamic light scattering (DLS). Crystallite size, lattice strain, and lattice constant were calculated by Rietveld refinement with Maud software. The results show a decrease in the crystallite size, and an increase in the internal strain and lattice parameter. Furthermore, the lattice parameter grew by increasing the percentage of titanium. Then, the powders compressed by the cold press and annealed at 650˚C. Finally, their micro-hardness and electrical resistance were measured. These analyses show that via increasing the proportion of titanium, Cu-6wt%Ti with 312 Vickers had the highest micro-hardness; due to the increasing the work hardening. Moreover, the results of the electrical resistance illustrate through increasing the amount of alloying material, the electrical resistance grew which the highest electrical conductivity was Cu-1wt%Ti with 0.36 Ω.

***doi**: 10.5829/ije.2020.33.09c.09*

1. INTRODUCTION

For the best performance of copper nanocomposites, it is necessary to make a good balance between its electrical and mechanical properties [1]. This means the alloy can be strengthened without a considerable reduction in the electrical properties of copper [2]. Increasing the mechanical properties through the formation of a saturated solid solution and the formation of fine nanometer-scale sediments is always a suitable way to increase the strength, toughness, thermal stability, creep resistance and at the same time, reduce changes in the electrical properties of copper [3-6]. A very good example here is Cu-Be alloys which have attracted very much attention by providing a strength of more than 1000 MPa and maintaining electrical conductivity of copper. But the main drawback of this alloy, which has limited its industrial use, is the high cost and toxicity of beryllium [7]. Cu-Ti alloys are the best alternative to Cu-Be and some similar alloys, because of the high strength, corrosion resistance, antibacterial property, high electrical conductivities and thermal stability [2, 8-11]. Some applications of Cu-Ti nanocomposite include relay controls [12], prosthetic dental applications [13], solar cells [14], anticorrosive applications [15], electrochemical denitrification [16], and, etc.

A variety of methods have been used to produce Cu-Ti alloys, for instance melting and casting processes or solid-state processing like powder metallurgy. In particular, new methods such as rapid solidification, severe plastic deformation (SPD), accumulative roll-bonding (ARB), electrolysis, high-energy milling and sol-gel have been proposed to prepare Cu-Ti alloys [17-22]. Among these approaches, mechanical alloying (MA) due to simplicity and cheapness of the process, being eco-friendly and homogenous dispersion of the second phase, has a special place for the production of Cu-Ti alloys.

Several studies have been carried out on Cu-Ti alloys produced by MA. Shkodich et al. and Sheibani et al. [23, 24], studied the formation and nanocrystalline phases of

*Corresponding Author Institutional Email: *oveis@eng.uk.ac.ir*
(S. O. Mirabootalebi)

Please cite this article as: R.M. Babaheydari, S.O. Mirabootalebi, G.H. Akbari, Investigation on Mechanical and Electrical Properties of Cu-Ti Nanocomposite Produced by Mechanical Alloying, International Journal of Engineering (IJE), IJE TRANSACTIONS C: Aspects Vol. 33, No. 9, (September 2020) 1759-1765

Cu-Ti by mechanical alloying. Preparing Cu-Ti via wet ball-milling was investigated by Eryomina et al. [25, 26]. Nagarjuna investigated the thermal conductivity of Cu-Ti alloy [27] and Guwer et al. discuss the micro-hardness of produced Cu-Ti alloys [28]. Likewise, Pourfereidouni and Akbari [29] analyzed the nano-structures of Cu-Ti Alloys in MA. Despite a wide range of studies on copper-titanium alloys in MA process, none of them focused on simultaneous changes in the crystal structure, micro-hardness, and electrical conductivity.

In the present study, the supersaturated solid solution copper with different percentages of titanium synthesized by high energy ball milling. The fabricated Cu-Ti nanocomposites were investigated by XRD, SEM and DLS analyses and the changing in the crystallite size, internal strain, the lattice constant, structure of the particles and particle-size distribution at different amount of Ti were investigated. Subsequently, heat treatment was performed and the hardness of the nanocomposite alloys was studied by the Vickers Micro-hardness test. Finally, the specimens were placed in an electrical circuit and their electrical resistance was evaluated.

2. MATERIALS AND METHODS

Precursors were pure Cu (99.7%,<6μm) and Ti (97%,10 μm). Samples with three compounds of 1, 3 and 6 weight percentages of Ti were milled for 90 h in a planetary ball mill at Ar atmosphere. The initial amount of powders was 15 g, the balls were 10 and 15 mm in diameter and the ball-to-powder weight ratio was kept at 15:1, and the vial speed was 350 rpm.

The structural changes in obtained samples were studied by an X-ray diffractometer (Philips X'Pert, Cu-Kα, λ=0.1542 nm). Rietveld refinement was used to calculate the copper crystallographic parameters including lattice parameter, crystal size, and internal strain. Morphology and size of ball-milled samples were analyzed by using scanning electron microscopy (Cam Scan mv2300) and the size distribution of samples was analyzed by zeta-seizer (ZEN3600). For sintering the mixed powders, the specimens were first molded to a diameter of 1 cm and thickness of 1 mm. Hence, 1.4 grams of the synthesized Cu-Ti powders were under pressure a coaxial cold press machine (12 ton). Sintering process applied in a tube furnace for a half-hour at 650˚C in Ar. The Vickers microscopy test was performed according to ASTM E 348-89 standard (Duramin20 Strues microprocessor) and micro-hardness was performed with a force of 98.7 mN at 400 magnification for 5 s. The electrical resistance of the samples was measured by using an electrical circuit. In this approach, the produced samples were placed in the circuit and by applying a voltage, the passing current through the tablets calculated and then by the Ohm's law, the resistance of the samples was obtained.

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of Cu-1, 3, 6 wt% Ti powders after 90 hours of ball milling. By comparing the peaks, the highest and the lowest peaks were observed in Cu-1wt%Ti and Cu-6wt%Ti, respectively. The low amount of the alloying element makes it difficult to detect the peaks of alloying elements in the XRD pattern.

During the ball milling of Cu-Ti, powder particles were severely deformed by the impact of the balls. This process leads to an increase in the local temperature and as a result, atomic diffusion occurs. Furthermore, density of crystalline defects such as vacancies, dislocations and stacking faults are greatly increased. Therefore, the particles get work hardening over time and as the effects of work hardening expanded, the internal strain and the width of the peaks were increased. The crystal defects are diffusion pathways for Ti atoms to dissolve in the copper matrix. On the other hand, after long milling time and increasing internal strains, the dislocations were regularly incorporated into the copper lattice and create subgrain boundaries. By continuing the milling process and increasing the density of the dislocations at the subgrain boundaries, they provide the basis for rotating these boundaries and converting them to the original boundaries.

Figure 2 provides information about lattice constant of Cu-1, 3, 6 wt% Ti after 90 h of milling. We can see with the increasing the amount of titanium, the lattice parameter increased. As well as, by increasing the percentage of Ti to 6 weight percentages, the lattice constant increases at a higher rate. The presence of large titanium atoms (0.147 nm radius) in the copper matrix increased the copper lattice parameter and is a sign of the formation of a solid solution. The high concentration gradient in the presence of large quantities of the reinforcement element in the primary powder mixture will help the titanium atoms to diffuse in copper lattice, therefore, a more saturated solid solution is formed. In fact, the possibility of collisions increased by raising

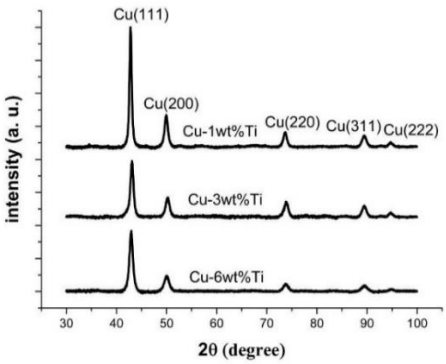

Figure 1. X-ray diffraction pattern of Cu-1, 3, 6 wt% Ti samples after ball milling for 90 hours

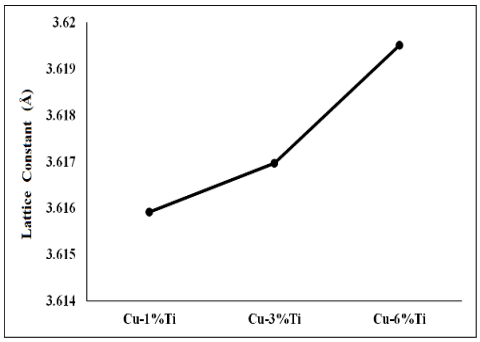

Figure 2. Diagram of changes in the copper lattice constant of 1, 3, 6wt% Ti after 40 h of ball milling

in the proportion of titanium, thereby the lattice parameter increased.

The crystallite size of copper for the Cu-1, 3, 6wt% Ti samples after 90 h of ball milling is shown in Figure 3. The crystallite size of all samples was in the range of 9-32 nm, and Cu-6wt% Ti has the largest reduction in crystal size (9 nm), while the Cu-1wt% Ti powder has the highest crystal crystallite size (17 nm).

Generally, the crystallite size is determined by the competition between plastic deformation through the motion of dislocations and the recovery and recrystallization [30]. Indeed, recovery and recrystallization increased by reducing the plastic deformation, conversely, the crystallite size decreases.

In this study, due to the low speed (350 rpm) and 15 min stop after 30 minutes of ball milling, the vial temperature did not increase and so the recovery and recrystallization phenomenon did not occur and the crystallite size decreased uniformly with increasing plastic deformation. The higher initial proportion of titanium accelerates work hardening, fraction, diffusion and segmentation of crystallite. Thus, Ti is dissolved and the hardness of the matrix increase. The reason for the more reduction of the crystal size in the compositions with more fraction of titanium is the aggregation of the effect of cold working and saturation of the copper matrix. Moreover, by increasing the percentage of Ti, the effects of the cold working become more apparent, and as a result, subgrain boundaries and dislocations create more and more.

Figure 4 shows the strain of the copper lattice for Cu-1, 3, 6wt%Ti samples. The internal strain and its rate grow by increasing the proportion of Ti. Initial powders are strain-free, but by starting the milling process, there is a rapid increase in the number of dislocations and other crystallographic defects. Gradually, with the formation of dislocations and reaching the crystallite size to a few nanometers, the number of dislocations has reached the saturated extent and under this situation, no new dislocations are created by increasing milling time. On the other hand, Ti atoms have also been entered into the Cu lattice during this period, these two phenomena impose strains on the lattice of copper. As shown in Figure 4, by increasing Ti percentage, the lattice strain of all samples is grown. In copper composites due to combination with the higher amount of Ti, the effects of cold working were much more severe, and the dissolutions of alloying elements increased. Consequently, matrix structure more affected due to the cold working and more defects, and eventually a much higher strain on the lattice is produced. Indeed, titanium by dissolving in the copper matrix and forming a solid solution, create more strain on the copper lattice.

The particle size distribution of Cu-1, 3, 6wt% Ti samples is shown in Figure 5. The average particle sizes for Cu-1, 3, 6wt% Ti were 188, 165 and 141 nm, respectively. According to Figure 5, sizes of particles were decreased by increasing the proportion of second phase and creation the richer solid solution, which leads to an expand the work hardening of the samples. Therefore, the samples with higher amounts of Ti were more fractured and the powder particles due to the more work hardening of the solid solution, have the smaller size. It should be noted that in all the samples some large particles of powder in the solution are deposited and the results illustrate the smaller particles. Although, the important point in this analysis is proving the achievement of the nano-sized particles in Cu-Ti composites by the MA method.

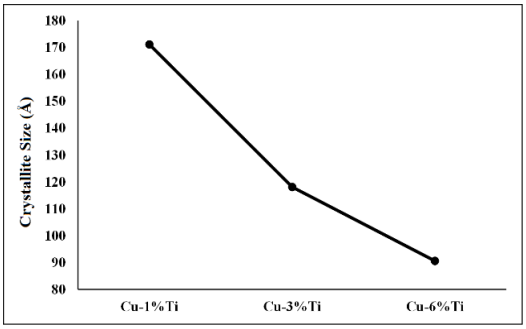

Figure 3. Crystallite size of Cu-1, 3, 6wt%Ti samples after 90 h of milling

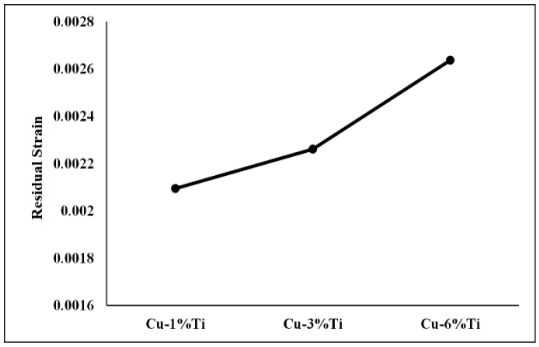

Figure 4. Strain changes of Cu-1, 3, 6wt%Ti samples

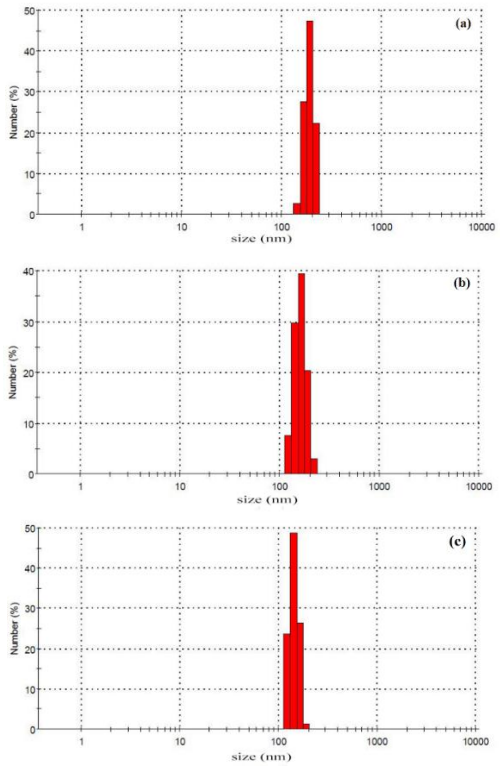

Figure 5. The particle size distribution Cu-1, 3, 6wt% Ti in a, b and c, respectively

The morphology and particle size of the powder mixtures are shown in Figure 6. Cu-1wt% Ti has the largest particle size and the size of particles decreased with increasing the amount of Ti. Due to the long milling time (90 h) of copper-titanium powder and making a balance between cold welding and fracture, the particle size distribution is reduced and have a uniform shape and dimensions, they also became agglomerated.

Titanium particles have an HCP structure and trapped between copper particles with FCC structure, which have been faster work hardening than copper. At this stage, the brittle titanium particles are distributed among the softer copper particles and create tiny cracks in their edges. As these cracks grow and spread into the powder grains, failure would occur more rapidly. On the other hand, in the compounds with a high initial titanium percentage, a rich solid solution was obtained. That is a reason for the higher hardness of these compounds. Hence, the samples with higher titanium content were more fractured and the powder particles, due to the higher hardness by the formation of the richer solid solution, had the smaller sizes.

Figure 7 gives information about the hardness changes of Cu-1, 3, 6wt% Ti after annealing at 650˚C. As can be seen, the micro-hardness increases by increasing the percentage of Ti. Cu-6wt% Ti with a hardness of 312

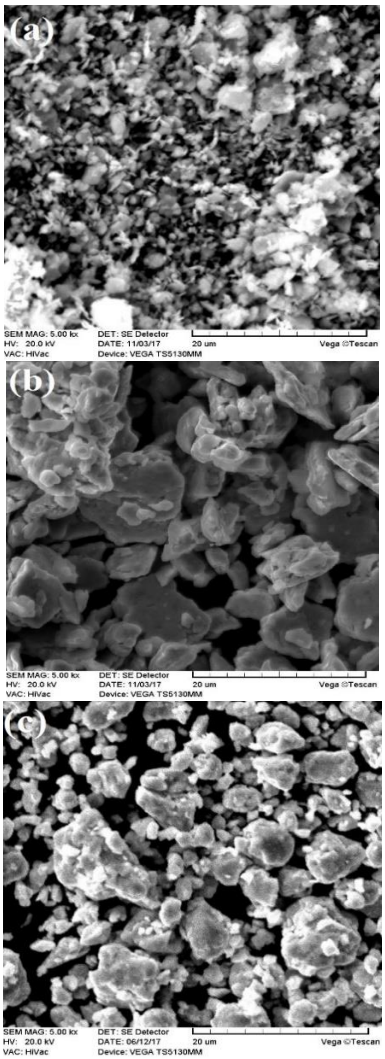

Figure 6. SEM images of Cu-1, 3, 6wt% Ti samples (a, b and c, respectively) after 90 hours of milling

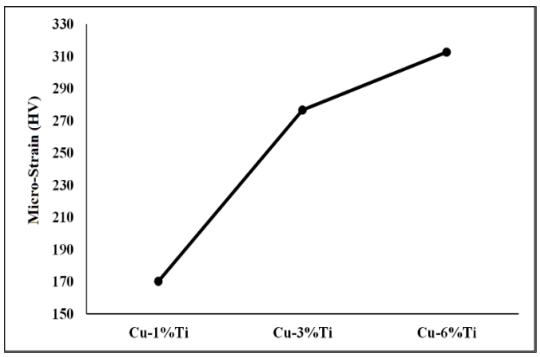

Figure 7. Micro-hardness changes of Cu-1, 3, 6wt% Ti after 90 hours of milling

Vickers had the highest hardness and the rate of increasing hardness decreased with an increasing amount of titanium. Due to the high work hardening of the

powders at the ball-milling process, the applied force during compression does not cause significant changes in micro-hardness [30].

The solubility of titanium in copper is very low at room temperature (<0.1 atomic percentage). In the mechanical alloying of copper-titanium, the concentration of titanium in the copper matrix is increased and reached to more value of the steady-state condition. This over-dissolution of titanium in the field of copper by MA and annealing at 650˚C provides a suitable situation for the deposition of titanium atoms in nanometer-sized particles. Therefore, the formation of titanium-rich nanoparticles in the matrix of copper in the sintering step, simultaneous with recovery and recrystallization of the processes will not only delay the softening process but also halt in many cases.

In general, the Cu4Ti sediments in the early stages of sintering are in the form of a coherent and fine nanometer. These sediments which were created inside the grains are obstacles to dislocation movement. Thus, delays recovery and recrystallization increases the micro-hardness.

Figure 8 shows the electrical resistance changes of the Cu-1, 3, 6wt% Ti. The electrical resistance increased by increasing the percentage of titanium. Cu-6wt% Ti with 0.44 Ω and Cu-1wt%Ti with 0.36 Ω, had the highest and lowest electrical resistance, respectively. Furthermore, the electrical resistance rate grows by increasing the proportion of Ti.

The main reason for increasing the electrical resistance by increasing the proportion of titanium is the lower conductivity of titanium than copper, which has a great effect on electrical conductivity. The higher density of defects and the richer solid solution lead to work hardening along the increasing amount of titanium and provide a high volume fraction of the sediments. Consequently, movements of electrons are prevented and the electrical resistance has increased, by these crystallographic defects and the sediments.

Regarding the percentage of the second phase and micro-hardness of the samples, electrical resistance in all fabrication of Cu-Ti alloys, such as the aging and severe drawing, and laser welding [31, 32]. It seems high capability of the ball-milling to mono-dispersion of the second phase particles cause to strengthen the grain boundaries and better precipitate of the titanium. On the other hand, the low content solution of Ti in the copper lattice has a minor effect on the electrical properties of the copper composites. The comparison of the micro-hardness and the electrical resistivity results represents the maintaining of mechanical strength simultaneous with the proper electrical conductivity of the produced Cu-alloys.

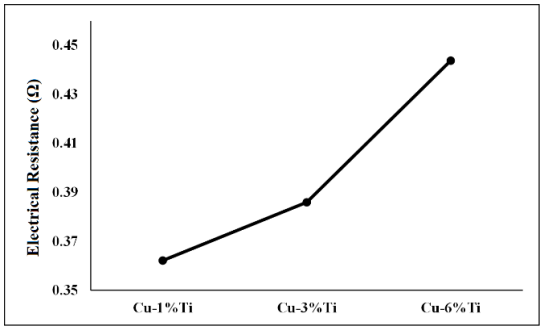

Figure 8. Electrical resistance of Cu-1, 3, 6wt% Ti compounds after 90 hours of milling

4. CONCLUSION

1. By mechanical alloying of copper and titanium with different percentages, it is possible to produce nanostructured Cu-Ti with nanometer-scale particle size.
2. The lattice constant increased by increasing the proportion of titanium.
3. The crystallite size is decreased and the internal strain is increased as the proportion of the titanium was increased.
4. Higher percentage of titanium lead to smaller particle size. Cu-6wt%Ti with average size of 141 nm has the smallest particle size.
5. Micro-hardness is increased by increasing the proportion of titanium.
6. Addition of titanium to the copper leads to homogeneous morphology.
7. The electrical resistance grew by increasing the amount of titanium.

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Persian Abstract

چکیده

در این مقاله ، نانوکامپوزیت مس-Ti از طریق آسیاکاری گلوله‌ای مخلوط پودرهای مس - تیتانیوم در ترکیبات ۱ ، ۳ و ٦ درصد وزنی سنتز گردید. سرعت کاپ ۳۵۰ دور در دقیقه و نسبت وزن گلوله به پودر ۱۵: ۱ بود. فرآیند در اتمسفر آرگون انجام شد و همچنین زمان آسیاب ۹۰ ساعت بود. پودرهای به دست آمده توسط (SEM)، (XRD) و (DLS) مورد بررسی قرار گرفت. اندازه کریستالی ، کرنش شبکه و ثابت شبکه با آنالیز Rietveld و نرم افزار Maud محاسبه شد. نتایج حاکی از کاهش در اندازه کریستالیت و افزایش کرنش و پارامتر شبکه است. علاوه بر این ، پارامتر شبکه با افزایش درصد تیتانیوم افزایش می‌یابد. سپس پودرها توسط پرس سرد فشرده شده و در دمای ٦۵۰ درجه سانتیگراد آنیل می شود و در نهایت میکرو سختی و مقاومت الکتریکی آنها اندازه گیری شد. تجزیه و تحلیل این تست ها نشان می دهد که Cu-6wt%Tiبا ۳۱۲ ویکرز بالاترین میزان میکرو سختی را دارد و با افازیش تیتانیوم سختی افزایش می‌یابد. نتایج مقاومت الکتریکی نشان می دهد که با افزایش مقدار عنصر آلیاژی تیتانیوم، مقاومت الکتریکی بیشتر می‌شود که بیشترین هدایت الکتریکی مربوط به Cu-1wt%Ti با $0.36\ \Omega$. است.