Improving the mechanical properties and coefficient of thermal expansion of molybdenum-reinforced copper using powder metallurgy

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Abstract

This paper presents an experimental study of Cu-Mo alloys prepared by powder metallurgy (PM) method. Also, improving the dispersion and wettability of Mo in the Cu matrix was aimed. Mo particles were added by 0.24, 0.48, 0.73 and 0.97% volume fraction to Cu powder. The mixture was mechanically milled by planetary ball mill at a rotational speed of 140 rpm for 24 h under hydrogen atmosphere, with milling ball size of ~25 times the size of the metal powders. Liquid acetone was utilized as a process control agent (PCA). Paraffin wax (0.5 wt%) was used to decrease the friction with die during the compaction process. The mixture of the blended powder was compacted at ambient temperature under three different pressures (400, 600 and 800 MPa) and then sintered in a vacuum furnace at 1000 °C for 1 h by a heating rate of 5 °C min⁻¹. The microstructure examination showed a homogeneous dispersion of Mo particles within the Cu matrix with no evidence of new phases formation during the sintering process. Also, the relative density of samples has been increased by increasing both of Mo content and the compaction pressure. The results revealed that the compaction pressure of 600 MPa was the most suitable pressure as it gave the highest densification. Cu—0.97% volume fraction Mo alloy samples exhibited finer Mo particles with a homogenous distribution in the Cu matrix and well bonding with the Cu particles. The microhardness was increased gradually by increasing Mo wt%, while the compressive strength was decreased by increasing the Mo contents. Both the electrical and thermal conductivities were decreased gradually by the addition of Mo. While the coefficient of thermal expansion (CTE) was decreased by Mo addition.

1. Introduction

Metal matrix alloys (MMCs) have become of paramount importance in recent times as it possesses unique characteristics like specific strength and toughness, high impact property, wear and corrosion resistance, elastic modulus and possibility to control the thermal and electrical conductivity [1–4]. Copper is used extensively in industrial applications in which high thermal and electrical conductivity, corrosion resistance, and ease of fabrication with low cost are required [5, 6]. Copper is used in major part for many global industries on unalloyed form. It has high electrical properties and a low-cost, so it is used in many electrical and thermal management industries such as microwave and vacuum devices; heat sink components, and electronic packaging [7–10]. The dispersion of ceramic powders into copper matrix strengthens the produced alloy and enhances its high-temperature creep strength [11, 12]. Thermal behavior of the materials used in electronic
devices is a critical issue especially in highly-packed devices with complex patterns of components in a cramped size [13]. The use of different materials in electronic devices with of the disparate CTE results in inducing undesirable stresses which impairs lifetime and reliability of the device [14, 15]. Thus, it is highly desirable to develop new advanced materials with low CTE and a high thermal conductivity for heat spreaders and heat sink used in electronic devices [16–18]. Regrettably, copper, which is commonly used in electronic devices due to its high thermal and electrical conductivity, has poor mechanical properties and high CTE [19]. Some research’s in earlier works aimed at resolving these problems via enhancing copper properties through choosing the optimal metal or ceramic nano-additives that may improve its mechanical properties and wear resistance and reduce its CTE such as Tungsten [20, 21], Diamond [22, 23], Zirconia [24, 25], Alumina [26], SiC [27, 28], Graphite [29, 30], and stainless steel fiber [31].

Molybdenum is silvery grey metal which classified as hard transition metal with reasonable ductility. It has high modulus of elasticity and a relatively high melting temperature (2620 °C). Molybdenum has been proved as a valuable alloying element, as it contributes to the toughness and hardness of ductile materials [32–35]. The Molybdenum is also used in aircraft and missile components as well as nuclear energy applications. It has been used in many advanced alloys in which heat and corrosion resistances are highly required. It has been also used in fabrication of electrodes used in electrical glass furnaces as well as filament of electrical and electronic devices [36, 37]. Cu-Mo alloys are mixtures of the face-centered cubic (FCC) structure of Cu with Mo refractory metal. The alloys combine the high electrical/thermal conductivity of Cu and low CTE of Mo [38–40].

Today traditional applications of the Cu–Mo alloy include heat sinks, electrical contacts, aeronautics and many other applications [41–43]. There is a large temperature difference between the melting points of molybdenum (2620 °C) and copper (1083 °C). Thus, conventional metal-casting processes cannot be used to produce Cu–Mo alloys but Powder Metallurgy (PM) technique is the best choice to produce this kind of alloys [44–46]. Cu–Mo alloy is widely used in vacuum and electronic devices as well as heat sink components. Furthermore, it has promising applications in portable apparatus and aerospace industry [16, 47]. Most of the conducted studies in literature investigated the effect of using high weight fraction of Mo on the properties of Cu–Mo alloy. Nevertheless, fewer experimental investigations were conducted on Cu–Mo alloy with low weight fraction of Mo. The fabrication of alloy material with high copper content is highly recommended to obtain higher thermal and electrical conductivities, however, the CTE will be increased [48, 49]. Cu–Mo alloy with high content of Mo can be obtained by conventional powder metallurgy processing due to excellent dissolution of Mo particles in Cu matrix [40, 50]. The common process for conventional powder metallurgy processing includes powders blending, pressing the produced blend, and sintering the pressed blend at a solid-state [51, 52]. For example, nano crystalline Cu–20 wt% Mo alloy synthesized using mechanical alloying technique as it can effectively decrease the crystallite size after 40 h of milling. With applying a load of 5 tons and sintering temperature of 1000 °C a high density of 93% could be obtained. CTE was lessened by about 25% compared to that of pure Cu [53].

Based on the aforementioned literature, the present work aims to develop a new Cu alloy with enhanced properties such as CTE, wear resistance and hardness by using Mo as reinforcing additive. Furthermore, preserving the electrical and thermal conductivities of Cu at reasonable levels is also achieved.

2. Experimental work

The raw Mo was supplied from Dop organic Kimya (Ankara, Turkey) with 99.96% purity and 0.5–2 μm average particle size. The Cu powder was supplied from Ecka Co., Germany with 99.96% purity and an average size of 20–70 μm. Both powders were mixed with Mo volume fractions of 0.24, 0.48, 0.73 and 0.97% by ball milling for 1 h with 140 rpm. The used ball size was 25 times greater than the powder. To minimize friction between the blend the die during the compaction process, liquid acetone and paraffin wax was used as a lubricant. Reduction of the mixtures were established at 450 °C for 1 h in hydrogen chamber. After drying, the powder mixtures were uniaxially pressed at ambient temperature under three different pressures; 400, 600 and 800 MPa. The obtained green specimens were subjected to sintering process in a vacuum furnace at 1000 °C for 1 h by heating a rate of 5 °C min $^{-1}$. The sintered samples were polished by emery paper with successive grades (400–2000 μm grit paper). The final polishing process is accomplished using 3 μm alumina paste on a velvet cloth. Then the surface of the samples is etching in nitric acid solution for 1 min. The composition of the prepared samples was examined using x-ray diffraction analysis. The microstructure of the prepared samples was investigated by both of the optical and scanning electron microscope (SEM). The density of the sintered samples was examined using Archimedes immersion method and the theoretical density was computed by applying the ‘rule of mixtures’. Vickers micro-hardness was measured by using ASTM E-384 Micro-hardness tester under a load of 500 g and a dwell time of 10 s and Vickers macro-hardness also was estimated under a load of 5 Kg and a dwell time of 10 s. Compression test specimens are prepared to be measured using a 500 KN universal Testing Machine (UH-50A)
at head speed of 1.0 mm min\(^{-1}\) and ambient temperature. The electrical and thermal conductivities were estimated by PCE-COM20 electric resistivity instrument. Thermal conductivity was verified via the equation:

\[
C = \sigma L (T + 273.15)
\]

where \(C\) and \(\sigma\) denote the thermal and electrical conductivities in W/m.K and \(\Omega^{-1}m^{-1}\), respectively, \(L\) denotes the Lorentz number which is a constant for alloy materials \((L = 2.45 \times 10^{-8} W \Omega K^{-2})\) and \(T\) denotes the temperature in °C. Finally, the CTE was measured using thermal diffusivity analyzer in a temperature range of 30 °C–600 °C.

### 3. Results and discussion

FE-SEM images of as-received Cu and Mo powders are shown in figure 1. The shown morphology of Cu and Mo powders reveals that their particles have spherical shape with an average particle size of 50 μm and 1 μm respectively.

Figure 2 displays the XRD pattern of the Cu–0.24, 0.48, 0.73 and 0.97% volume fraction Mo alloy powders obtained under compaction pressure of 600 MPa and sintering temperature of 1000 °C for 2 h. Diffraction peaks reveals the existence of Cu and Mo phases and no other peaks can be detected, which indicates that the powders were completely reduced, due to the sintering process in a vacuum furnace, so, oxide formation is inhibited and very distinct peaks of Cu can be observed. No other peaks corresponding to any intermetallic formed between Cu and Mo are recorded. It was also obvious that, the peaks intensity increases as the Mo content increases.

Figure 3 shows the optical micrographs of 0.97% volume fraction Cu–Mo alloy under 400, 600, 800 MPa pressure. As the microstructure is used as a key tool to figure out the interrelation of the physical and mechanical properties of the prepared samples, it is clear from figure 3 that 600 MPa is the most suitable pressure as it gives the more fine and homogenous structure.

Figure 4 presents the FE-SEM of Cu-Mo alloys after sintering stage. A homogeneous dispersion of Mo in the Cu matrix for the four the Cu-Mo alloys are observed. The gray area in all pictures represents the Cu matrix, while the white spots are belonging to the Mo particles. Finally, the black spots represent the pores which

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**Figure 1.** SEM images of as received powders (a) Cu powder, (b) Mo powder.

**Figure 2** XRD patterns for pure Cu and Cu–0.24, 0.48, 0.73 and 0.97 wt% Mo alloy.
decrease by increasing Mo percent, in which 0.97% volume fraction Mo sample has the lowest porosity percent. This may be due to the more grain refinement of Cu particles by the effect of Mo particles which has a ceramic material that acts an internal ball. The reduction in the particles size gives the smaller particles to fill the pores.

**Figure 3.** Optical micrograph for Cu–0.97% volume fraction Mo alloy compacted at (a) 400 MPa, (b) 600 MPa and (c) 800 MPa.

**Figure 4.** SEM micrographs of Cu–Mo alloy at Mo volume fraction of (a) 0.24, (b) 0.48, (c) 0.73 and (d) 0.97.
Generally, the mechanical, physical, and thermal properties of alloy materials are enhanced for those samples with homogeneous dispersion of the reinforcement phase into the matrix. Figure 5 shows the EDX analysis of the Cu-0.97% volume fraction Mo alloy at 600 MPa and 1000 °C, Spectra of both Cu and Mo are evident. This confirms the homogeneity of the Cu-0.97% volume fraction Mo alloy and the good dispersion of Mo all over the Cu matrix due to the good mechanical milling parameters.

Figure 6 displays the relationship between relative sintered density and used compaction pressures (400, 600 and 800 MPa) for various Mo contents which are 0.24, 0.48, 0.73 and 0.97 vo%. All samples were sintering at 1000 °C for 1 h. The relative density of the samples is increased by increasing Mo content. This is due to the higher density value of Mo than that of Cu (10.28 g cc−1 and 8.9 g cc−1) respectively. Along with the aforementioned observations, the density of the prepared samples is highly affected by the compaction pressure and Mo content. Density of the produced alloy is increased with increasing the compaction pressure up to 600 MPa due to the proper softening that occurs in Cu powders during of the compaction. This softening enhances motion of the Cu particles toward each other and consequently the pores are eliminated. Also, the grain refinement enhances the densification, that takes place by addition of more Mo to Cu.

Figure 7 shows the microhardness values for Cu–Mo alloy compacted at 600 MPa. It is affected not only by composition, but also by the level of porosity and densification. From this figure Micro hardness values increase with increasing the molybdenum content and densification. These results can be attributed to the high hardness for pure Mo with its ceramic nature, and the higher density and lower porosity of Cu–Mo alloys. Also, the grain refinement of Cu by addition of Mo which is established from microstructure that is a good reason for the improvement of hardness. The presence of a hard ceramic metal such as Mo dispersed in a ductile metal like Cu helps in increasing the strength of the overall alloy. In which, Mo micro-particles resist matrix deformation and material slipping, leading to improvement of the alloy hardness [54].

Figure 8 shows the experimental results of the compressive strength. It is clear that the compressive strength increases by increasing the Mo content. This may be explained by the strength effect of Mo particles which is a hard metal that increases the hardness of Cu and retards the dislocation motion. Reinforcing Cu with Mo particles creates dispersion hardening effects which prevents the dislocation of particle, consequently, increases...
the compressive strength. Also, by Mo additions the densification increased which indicates low porosity. So, Mo centers of crack are presented in the Cu matrix, consequently the compressive strength increases \[55\].

From figure 9, it is obvious that the electrical conductivity of Cu decreases by increasing the Mo percent. In which for pure Cu, it recorded 50 M\(\text{s m}^{-1}\), while for 0.97\% volume fraction Mo it is fall down to \(~33\text{M}\text{s m}^{-1}\). This may be due to the lower electrical conductivity value of Mo compared with that of Cu. Also, the presence of Mo at the grain boundaries of Cu matrix retards the electron motion in the Cu matrix which increases the resistivity, so the overall conductivity decreases. Presence of some pores inside the Cu–Mo alloy also, decreases the conductivity as its conductivity is zero which prevents the motion of the charge carriers.

Figure 10 displays the change in the thermal conductivity Cu with Mo additions. The thermal conductivity decreases gradually by increasing the Mo percent. This due to the lower value of thermal conductivity of Mo than
that for Cu, also the presence of pores which have a zero conductance. But generally, both the electrical and the thermal conductivity of Cu–Mo alloys still in the working area of Cu application.

Figure 11 shows the variation of the CTE versus temperature for Cu–Mo alloys with different Mo contents. It is noticed that CTE decreases when the Mo content increases. These results can be attributed to low CTE for pure Molybdenum. Also, the good distribution of Mo particles into Cu matrix that acts as an internal network restricts the expansion under the heat effect [56].

4. Conclusion

The present study investigates the fabrication, morphological changes, microstructure and characterization of copper-molybdenum alloys successfully synthesized by powder metallurgy. The results can be summarized as follows.

1. The best compaction pressure for the Cu/Mo alloy was 600 MPa.
2. The density of the samples increased by raising Mo content.
3. The alloy structure was analyzed using EDS, which confirmed the homogeneity of the Cu–Mo alloy.
4. The sintering process did not produce any new phases or intermetallic between Cu and Mo alloy.
5. The microhardness values were increased with increasing of Molybdenum content and densification.
6. The compressive strength was increased by increasing the Mo content.
7. Electrical and thermal conductivities were decreased gradually by the addition of Mo in the Cu matrix. But their values were still in Cu range applications.
8. The coefficient of thermal expansion (CTE) was decreased with the increasing of Molybdenum content.
9. The manufactured Cu–Mo alloy were suitable for mechanical application as well as heat sink materials.
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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