Effect of Mn on microstructure and properties of Cu-12Al powder metallurgy alloy

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Abstract

Cu-12Al-xMn (x = 0, 1, 2, 3, 4%, mass fraction) alloys were produced using powder metallurgy to investigate the effect of Mn content on the alloy’s microstructure and mechanical properties. The Cu-12Al (no Mn) and Cu-12Al-1Mn (1 wt% Mn) alloys consisted of the α + γ phases, where the size of the α grains in the Cu-12Al-1Mn alloy are much larger. Mn effectively inhibited the eutectoid transformation at a concentration above 1 wt%. Furthermore, the β phase was converted to the β′-martensite phase, which had a monoclinic structure after slow cooling. The phases of the alloy changed from the α + γ phases to the α + β′ + γ phases with the further addition of Mn, and the tensile strength of the 3 wt% Mn alloy was 380.59 MPa. The addition of Mn did not have a significant effect on the density of the alloy.

1. Introduction

Cu-Al alloys have excellent properties, including thermal conductivity, wear resistance, and corrosion resistance, and has been applied widely in bearings, bushings, and gears [1–6]. Al has a greater influence on the lattice constant of Cu than other alloy elements due to its high solid solubility in Cu [7], and its addition significantly improves the strength and hardness of the alloy [8]. Cu-Al alloys with an Al content between 9.6 and 15.6 wt% forms α + γ phase via eutectoid decomposition. γ-Al₄Cu₉ is similar to cementite in carbon steel and is distributed in a network. The proportion of the γ phase increases with higher Al content, which in turn leads to increased hardness and strength but lower plasticity [8–11]. Cu-12Al close to the eutectoid component has a eutectoid microstructure with high strength and hardness, but poor plasticity and toughness [12–14].

Mn improves the toughness and strength of the alloy by stabilizing the β-Cu₃Al phase, particularly at lower temperatures, and extends the composition range of the β phase [15, 16]. Mn decreases the eutectoid transformation temperature of Cu-Al alloys, thereby minimizing the formation of the brittle γ phase [17]. The β phase is formed by eutectoid transformation of the hindered β phase, and has favorable strength, elasticity, and wear resistance [18].

Powder metallurgy is a promising production process, and its near net shape, low cost, and segregation-free formation offer unique advantages in the manufacture of porous materials [19–23]. Improved composition control, finer grain size, and better mechanical properties can be expected using a powder metallurgy route [24–28]. A previous study by Kryachek et al. found that the a second phase (the intermetallic compound Cu₂Al) in the Cu-Al alloy improved wear resistance, but the study did not investigate this mechanism [29]. Masato Ohtsuki et al. produced an Cu-Al alloy using AlF₃-KF as a sintering aid and the eutectic reaction between aluminum and the sintering aid removed the oxide present on the surface of the powder [30]. Mitani et al. added
elemental Mn to the Cu-Al alloy which resulted in an abnormal exotherm at about 550 °C that was not observed when Al-Mn alloy powder was used. The use of elemental powder during the sintering process results in a much higher diffusion coefficient of Mn into Cu than that of a mixture of Cu and Al into Mn, causing expansion at the eutectic temperature during sintering [31, 32]. Gera et al. prepared a Cu-11.8Al-3.2Ni-3Mn-0.5Zr alloy using powder metallurgy and observed a martensite structure under air cooling conditions. However, the excessive sintering temperature led to increased grain size (>150 μm) after sintering for 1 h [33].

The use of metal powders for the preparation of CuAlMn alloys will lead to reduced cost, but few studies have investigated this method or the influence of Mn on the microstructure and properties of Cu-Al alloys. This study aimed to calculate the CuAlMn ternary phase diagram using Thermo-Calc software and establish suitable experimental parameters. A Cu-12Al-xMn alloy was prepared using powder metallurgy, and the mechanical properties and microstructure were evaluated.

2. Phase equilibrium

The isothermal section and vertical section phase diagrams of the Cu–Al–Mn system was calculated using the Thermo-Calc software (figure 1), where thermodynamic parameters from a previous study by J. Miettinen were used [34]. The phase transition process was evaluated using the phase diagram of the alloy. The Fcc_A1 (α) + Bcc_A2(β) + liquid phase was observed at 1000 °C (figure 1(a)). The BCC_A2 phase had a BCC structure and was present at high temperatures and the eutectoid reaction $\beta \rightarrow \alpha + \gamma + \tau$(Cu$_3$Mn$_2$Al) took place during cooling (figure 1(b)). Under equilibrium conditions, the $\tau$ phase was the stable phase at room temperature [35].

A sintering temperature of 1000 °C contributed to the densification of the solid phase during Cu-Al sintering and a reduction in pore size and total pore volume [36]. An increased Mn content caused the high temperature phase region of the alloy to occupy a solid-liquid two-phase region as well as a liquid phase region (figure 1(a)). Mn was added in varying quantities (0, 1, 2, 3 and 4 wt%) to the Cu-12Al alloy to investigate the effects on microstructure and properties.

3. Experimental procedure

The Cu-Al-Mn alloy was prepared using powder metallurgy with the following raw materials: electrolytic copper powder (99.9 wt%), water atomized aluminum powder (99.5 wt%) and mechanically crushed manganese powder (99.9 wt%). The morphology and the characteristics of each powder are given in figure 2 and table 1, respectively. CuAlMn alloys with the composition Cu-12Al-xMn ($x = 0, 1, 2, 3$ or $4$) were prepared.

A mixture of the above component powders was obtained by mixing and mechanically grinding for 3 h, and pressing the mixed powder in a 20 mm inner diameter mold at a pressure of 500 MPa. Sintering was performed in high-purity hydrogen gas, and the temperature was increased to 1000 °C at a rate of 5 °C min$^{-1}$ and maintained for 1 h. The sample was removed from the furnace after cooling to room temperature.

The structure of the samples was studied using optical microscopy (RX50 M, Sunny Corp., China), and scanning electron microscopy (JSM-7001F, Jeol Ltd, Japan). Etching was performed in a solution of 3 g FeCl$_3$/2 ml HCl/96 ml H$_2$O.
X-ray diffraction (SmartLab, Rigaku Corp., Japan) was used to analyze the phases and was carried out under the conditions of $\lambda = 0.15406$ nm, 45 Kv, 150 mA and a scanning speed of $20^\circ$ min$^{-1}$. The microstructure and crystal structure were analyzed by transmission electron microscopy (JEM2100F, Jeol Ltd, Japan), and selected area electron diffraction (SAED).

The density of the alloys was measured based on the Archimedes principle, as given in equation (1):

$$\rho_0 = \frac{m_0}{m_1} \times \rho$$  \hspace{1cm} (1)

where $\rho_0$ is the actual density of the sample after sintering (g cm$^{-3}$); $m_0$ is the mass of the sample in air before immersion in the paraffin solution (g); $m_1$ is the mass of the beaker and the sample after sealing the surface pores with paraffin and suspending in water (g); and $\rho$ is the density of water (g cm$^{-3}$).

The porosity ($\theta$) was calculated according to equations (2) and (3)

$$\rho_s = \frac{1}{x_1/\rho_1 + x_2/\rho_2 + \ldots + x_n/\rho_n}$$  \hspace{1cm} (2)

$$\theta = \rho_s / \rho_0$$  \hspace{1cm} (3)

where $\rho_s$ is the theoretical density of the alloy; $\rho_n$ is the density of the nth element; and $x_n$ is the mass fraction of the nth element.

The density of the pre-sintered alloy is listed in table 2.

The hardness of the alloy was measured using a Brinell hardness tester (HB3000) with a 187.5 kg loading stress and 2.5 mm indenter diameter. The average of 5 measurements for each sample was determined and the hardness value was calculated according to equation (4):

Table 1. Characteristics of the Cu, Al, and Mn powders.

| Powder particle size / μm | Cu            | Al            | Mn            |
|--------------------------|---------------|---------------|---------------|
| D10          | 12.806        | 13.111        | 5.131         |
| D50          | 23.857        | 21.701        | 16.950        |
| D90          | 42.701        | 37.707        | 37.129        |
| Purity/wt%  | 99.9          | 99.7          | 99.8          |
| Supplier     | Beijing Xing Rong Yuan Technology Co. |  |  |

Table 2. Characteristics of the raw material (a) copper powder, (b) aluminum powder, and (c) manganese powder.
where $F$ is the loading stress; $D$ is the diameter of the steel ball and $d$ is the diameter of the indentation.

The tensile strength was determined using a tensile tester (MTS-810, MTS Systems) and the average of 5 measurements for each sample was determined and calculated according to equation (5):

$$\sigma = \frac{F_b}{S_0}$$

where $\sigma$ is the tensile strength; $F_b$ is the maximum stress at which the sample failed; and $S_0$ is the original cross-sectional area of the specimen.

### 4. Results

#### 4.1. Microstructure and phases of the alloys

X-ray diffraction of the Cu-12Al-xMn alloys revealed that there was only two phases ($\alpha$ and $\gamma$) in the Cu-12Al and Cu-12Al-1Mn alloys, demonstrating that the eutectoid transformation was not affected when the Mn content was below 1 wt% (figure 3). At an Mn content above 1 wt%, three phases were observed, namely $\alpha$-Cu, $\gamma$-AlCu$_9$, and $\beta'$-Cu$_3$Al. As the matrix phase of the alloy was $\beta'$-Cu$_3$Al, its peak intensity decreased. According to the Debye–Scherer formula $[37]$, grain refinement leads to an increase in the half-peak width, and this was observed due to the refinement of Cu and Al$_4$Cu$_9$ upon addition of Mn (figures 4–6). However, no diffraction peak attributed to Mn was observed due to sensitivity limitations of the XRD technique $[35]$.

Secondary electron images (SEI) of the CuAlMn alloy microstructures revealed that alloys with Mn content in the range 0 to 1 wt% had both a network of $\alpha$ and $\gamma$ phases and $\alpha$ phase grains (figures 4(a) and (b)). The samples with an Mn content of 2 to 3 wt% were characterized by the absence of the $\alpha + \gamma$ network eutectoid. Furthermore, a needle-like $\beta'$ phase was distributed around the $\alpha$ phase, and a $\gamma$ phase surrounded the $\beta'$ phase. When the Mn content reached 4%, the $\alpha$ phase, $\beta'$-martensite, and $\gamma$ phases FCC, monoclinic and BCC, respectively.

Table 2. Densities of the pre-sintered alloy.

| Alloy          | Density (g cm$^{-3}$) |
|----------------|------------------------|
| Cu-12Al       | 6.47                   |
| Cu-12Al-1Mn   | 6.44                   |
| Cu-12Al-2Mn   | 6.39                   |
| Cu-12Al-3Mn   | 6.34                   |
| Cu-12Al-4Mn   | 6.31                   |

Table 2. Densities of the pre-sintered alloy.

Figure 3. The XRD patterns of the Cu-12Al-xMn alloys after sintering.

$$\text{HB} = \frac{2F}{\pi D (D - \sqrt{D^2 - d^2})}$$ (4)

where $F$ is the loading stress; $D$ is the diameter of the steel ball and $d$ is the diameter of the indentation.
Cu-12Al-2Mn (figure 6(c)), Cu-12Al-3Mn (figure 6(d)) and Cu-12Al-4Mn (figure 6(e)) were 89.9%, 88.5%, 88.3%, 88.9%, and 88%, respectively. The network structure of Cu-12Al and Cu-12Al-1Mn revealed that the grains distributed around the network in Cu-12Al-1Mn were larger due to grain growth. The network structure disappears in the other Mn containing alloys, and white crystal grains were observed in Cu-12Al-2Mn and Cu-12Al-3Mn.

4.2. Mechanical properties of the alloys

The stress-strain curve, tensile strength, density, and hardness of the sintered alloys (density = 88%–89%) were measured (figure 7).

The hardness value of the 1 wt% Mn alloy was than Cu-12Al, but the Brinell hardness value decreased as the Mn content increased from 1 to 4 wt%. The maximum tensile strength and strain of Cu-12Al were 352 MPa and 5.6%, respectively. Cu-12Al-2Mn exhibited the best toughness, with a tensile strength of 336.43 MPa and strain of 7.4%. Cu-12Al-3Mn has the highest strength of 380.59 MPa and a strain of 6.5%.

5. Discussion

5.1. Microstructure of the alloys

The binary phase diagram of the Cu–Al systems with an Al content ranging 8.1%–15.9% underwent a $\beta \rightarrow \alpha + \gamma$ eutectoid transformation when slowly cooled to 565 °C (figure 8), where thermodynamic
parameters from a previous study by J. Miettinen were used [34]. The addition of Mn to the Cu-Al alloy led to stabilization of the $\beta$ phase and hindered the eutectoid transformation [8].

According to the Cu-Al-Mn phase diagrams (figures 1(a) and (b)), the $\beta$-Cu$_3$Al phase was only observed in the Cu-12Al-xMn alloys at 1000 $^\circ$C, despite the presence of Mn. The vertical cross-section of the Cu-12Al-xMn ternary phase diagram indicated that the equilibrium phase at room temperature in the alloys with a Mn content above 1 wt% was $\alpha + \gamma + \tau$. However, the eutectoid reaction $\beta \rightarrow \alpha + \gamma$ was controlled by the cooling rate [38]. In this study, the samples were cooled in the furnace, and the microstructure of the samples indicated that only part of the $\beta$ phase had undergone eutectoid transformation (figure 9).

The microstructure, phase analysis and phase diagrams of the CuAlMn alloys (figures 1, 3, 4 and 5) indicated that the addition of Mn reduced the eutectoid transformation temperature and improved the stability of the $\beta$ phase. The matrix phase of the alloys with 0 and 1 wt% Mn was $\alpha + \gamma$, and the addition of 1 wt% of Mn did not affect the eutectoid transformation of the $\beta$ phase during sintering (figures 3(a) and (b)). The majority of the Mn was dissolved in the matrix at 1 wt% and had a minimal effect on the eutectoid transformation. The large $\alpha$ phase grains were formed in both of these alloys as there was two phases of $\alpha + \beta$ at high temperature, of which the $\alpha$ phase formed $\alpha$ phase grains after cooling and the eutectoid transformation of the $\beta$ phase formed a eutectoid ($\alpha + \gamma$) network during cooling. A portion of the supercooled $\beta$ phase was converted to the $\beta'$ phase and an acicular microstructure structure was observed.

When the mass fraction of Mn was increased above 1%, the alloy morphology changed and a martensite structure was observed. This was attributed to suppression of the $\beta \rightarrow \alpha + \gamma$ eutectoid transformation that converted the $\beta$ phase to the $\beta'$ martensite phase. The addition of Mn increased bonding between the atoms, which resulted in a higher diffusion activation energy, reduced atomic diffusion rate and reordering of the alloy [40]. Martensite transformation is a non-diffusion transformation and involves a reversible phase change that does not affect the composition or degree of ordering. Reordering of the martensite phase results in orthogonal distortion [41]. When stabilized, the martensite structure changes from the monoclinic system to the orthorhombic system, and this transition can be inhibited if the monoclinic angle of martensite is equal to or close to 90° [42].

The $\alpha$ phase was distributed around the $\beta'$ phase in the Cu-12Al-2Mn, Cu-12Al-3Mn and Cu-12Al-4Mn alloys and was more refined compared to Cu-12Al and Cu-12Al-1Mn (figure 4). Furthermore, the $\gamma$ phase was distributed around the martensite in the microstructure of the Cu-12Al-2Mn, Cu-12Al-3Mn and Cu-12Al-4Mn alloys. Although Mn was added to the alloy, a fraction of the $\beta'$ phase underwent eutectoid decomposition during the cooling process of sintering. According to the thermodynamic calculations (figure 1), the alloy contained $\tau_3$
Figure 6. Optical micrographs showing the microstructure of the (a) Cu-12Al, (b) Cu-12Al-1Mn, (c) Cu-12Al-2Mn, (d) Cu-12Al-3Mn, and (e) Cu-12Al-4Mn alloys.

Figure 7. Hardness and tensile strength of CuAlMn alloys given as (a) the stress-strain curve and (b) the Brinell hardness, tensile strength, and density.
(Cu₃Mn₂Al) after the addition of Mn and the \( \tau_3 \) phase was observed in the phase analysis at an Mn content of 4 wt%.

According to Savitskii [43], a liquid phase begins to form at 500 °C during the sintering of the Cu-Al alloy. The liquid phase extends over the entire volume due to the capillary effect, leaving voids in the positions previously occupied by Al particles. As a result, liquid phase diffusion was recorded during the macroscopic growth of the specimen volume. The sintered Cu-Al alloy had a level of porosity due to the Kirkendall effect and volumetric contraction took place [44] (figure 6). The pore morphology, distribution and size of the alloys of the same composition of the various alloys after sintering were similar, indicating that Mn content had little effect on the pore structure. The atmosphere used during sintering can be effective in controlling the high temperature chemical reactions used to remove lubricants and contaminants, which can cause sample size changes [45].

**Figure 8.** The binary phase diagram of Cu-Al.

**Figure 9.** C-curve of the Cu-12Al alloy [39].
Sintered metal with a degree of porosity was impregnated with lubricating oil in the range of 10%–40% (volume fraction), which can be used in a self-supplying state [46].

5.2. Hardness and strength of the alloys

The \(\alpha\) phase is a Cu-based solid solution with a microhardness (HV) of 200 to 270. The \(\beta\) phase is a supercooled \(\beta\) phase produced by hindered eutectoid transformation and is a solid solution based on a Cu13Al electronic compound with a microhardness of 290 to 407 [47]. These two phases are isomers, with the \(\alpha\) phase having a stable orthorhombic lattice structure below 325 °C, while the \(\beta\) phase has a body-centered cubic lattice. The \(\gamma\) phase is a solid solution based on Al4Cu9 with a microhardness of 549. After slow and maintained heating or cooling to a temperature below 559 °C, the \(\gamma\) phase can coarsen and become hard and brittle, which results in a loss of plasticity [48–50].

Hardness is not affected by pore shape, but rather the porosity of the material, while strength is affected by the shape, distribution and porosity of the pores [51]. However, the small amounts of Mn caused a significant difference between the alloys, and thus porosity had no significant effect on the material properties under the conditions of this study.

The Cu–12Al–1Mn was harder value than the Cu–12Al alloy with no Mn (figure 7(a)). Mn dissolved in the Cu–Al matrix and allowed for solid solution strengthening. As the Mn content was increased in the range 1 to 4%, the hardness of the alloy decreased because the matrix phase was converted from the \(\alpha + \gamma\) eutectoid phases to the \(\alpha + \beta\) phases. The microhardness of the \(\beta\) phase is lower than that of the \(\gamma\) phase. Within this range, the hardness contribution of the \(\gamma\) phase was low and the overall hardness of the alloy was lowered due to this matrix phase change.

The tensile strength and tensile curve indicated that the addition of Mn to the Cu–12Al alloy can effectively improve the strength and toughness of the alloy. The tensile strength of the Cu–12Al–1Mn was lower than the Cu–12Al binary alloy due to the addition of Mn, which increased the \(\alpha\) phase and triggered grain growth. When the Mn content exceeded 1 wt%, the matrix phase of the alloy was \(\beta\)-Cu3Al, which led to improved toughness. A small amount of \(\gamma\)-Al4Cu9 was distributed around the \(\beta\)-Cu3Al to strengthen the matrix, and the Cu–12Al–3Mn alloy exhibited the highest strength. At a Mn content of 4 wt%, the strength began to decrease due to the depletion of the \(\gamma\) phase.

6. Conclusion

Powder metallurgy was used to prepare CuAlMn alloys with a density controlled within the range of 88%–89%.

The phase distribution of the alloys was altered by adding varying amounts of Mn to Cu–12Al. Higher Mn content led to a gradual decrease in the \(\gamma\) phase of the reticulated eutectoid, which precipitated from the network as a nubby precipitate. The structure of Cu–12Al–1Mn (1 wt% Mn) was similar to Cu–12Al, but the \(\alpha\) phase grains are larger and the tensile strength was lower.

At a Mn content above 2 wt%, a needle-like \(\beta\) phase with a monoclinic structure was observed, and the alloy consisted of a needle-like \(\beta\) phase and bulk \(\alpha\) and \(\gamma\) phases. Mn was mainly distributed in the \(\alpha\), \(\gamma\) and \(\beta\) phases, and the distribution ratio was equivalent in each phase. The hardness and tensile strength of the alloy decreased with the addition of Mn content. The highest tensile strength was observed in the alloy with of 3 wt% Mn content (380.59 MPa), while the highest strain was observed in the 2 wt% Mn alloy (7.4%).

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