Research Article

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Effects of ball milling on powder particle boundaries and properties of ODS copper

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Abstract: Al2O3 dispersion-strengthened (ODS) copper has an excellent comprehensive performance due to the strong hindrance of the high concentration nano-Al2O3 to the dislocations inside copper grains. However, the processability of ODS copper is seriously deteriorated, which is caused by the presence of unfavorable microlevel Al2O3 particles along powder particle boundaries. In this study, a strategy of ball-milling-induced impurity removal is adopted to surmount the dilemma. It was found that the ball milling process can significantly weaken the formation of large Al2O3 particles in the primary boundaries. However, due to the activation of the powder particle surface, the metallurgical bonding between the powder particles is strengthened. The results showed that the ball-milled samples exhibited the optimal properties, including the ultimate tensile strength of 488 ± 3 MPa, elongation of 18.7 ± 0.7%, reduction in the area of 46.8 ± 1.2%, 82.2 ± 0.3 Rockwell Hardness measured on the B scale (HRB), and electrical conductivity of 77.2 ± 0.1% International Association of Classification Societies (IACS).

Keywords: ODS copper, ball milling, Al2O3 particles, mechanical property, electrical conductivity

1 Introduction

Copper with face-centered cubic crystal texture exhibits good plasticity, excellent thermal conductivity, and electrical conductivity and thus has been widely used in power, electronics, energy, transportation, light industry, emerging industries, and other fields [1–3]. However, its significant weakness is low strength, which limits its scope of application [4]. Therefore, numerous investigations have been conducted to improve the strength of copper materials for application requirements [5–8]. Among these efforts, dispersion strengthening and age hardening are regarded as extensively and successfully applied technologies to enhance the mechanical properties of copper alloys [9–11]. Although the age-strengthened alloy has excellent mechanical properties and electrical and thermal conductivity at room temperature, there is a primary disadvantage of poor high-temperature stability because of the growth and redissolution of precipitated phases [12,13]. Since nano-Al2O3 particles are very stable at high temperatures, the dispersion-strengthened copper has outstanding elevated temperature properties [14].

In recent years, dispersion-strengthened copper alloys have attracted significant attention because of their application in welding electrode, lead frame, and electronic packaging [15]. The prevalent methods for preparing dispersion-strengthened copper alloys mainly involve mechanical alloying and internal oxidation. In particular, internal oxidation has been currently applied to industrial mass production [16,17]. As the initial raw material, the water-atomized Cu–Al alloy powder is oxidized to obtain nano-Al2O3 particles-strengthened copper powder, followed by densification treatment [18]. During the water atomization, most metals react with water or steam to form oxides, following the chemical reaction: \[ x\text{Me} + y\text{H}_2\text{O} = \text{Me}_x\text{O}_y + y\text{H}_2 \] [19]. The surface analysis of collected powder shows that the surface of the metal powder is usually covered with metallic oxides because the metal with high negative free energy and high diffusivity at elevated temperatures is liable to form oxides on the powder particle surface. The thickness of oxide film ranges from tens to thousands of nanometers [20–22]. Unfortunately, the resulting dispersion-strengthened copper has serious adverse effects on powder primary particle boundaries (PPBs) originating from aluminum oxidation, which roots in deoxidizing products in the Cu–Al alloy melting and aluminum segregation oxidation on the surface of powder particles during atomization [23].

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Generally, the PPB problem mainly exists in superalloys and high-speed steel [24,25]. Recently, increasing attention has been paid to nonferrous metal powder metallurgy materials, such as copper alloys and aluminum alloys [26,27]. In fact, the different types of ceramic particles manifested inside PPBs are the immediate source of adverse effects, including oxides, carbides, and oxy carbonoritrides. The weakness of the interpowder bonding, resulting from the particles located in PPBs, thereby significantly influences the powder consolidation and dramatically reduces the elongation-to-failure and fracture toughness of materials [28].

The ceramic particles have been experimentally proved to be derived from the master alloy, the crucible lining, the leaking package, and the flushed nozzle [20]. The PPB problem and inclusions lead to a sharp decline in plasticity. Furthermore, some large oxide particle inclusions directly result in an abrupt fracture during the deformation process. Therefore, many approaches have been introduced to solve the PPB problem, including selecting clean raw materials, vacuum gas atomization, and powder purification [29,30]. However, these methods are hard to implement in the industries owing to their high cost.

In this study, a simple and effective method of ball milling was carried out to eliminate or weaken the PPB problem of the dispersion-strengthened copper alloy. The effects of ball milling on the mechanical and physical properties of dispersion-strengthened copper were studied, and the related mechanism was also interpreted.

## 2 Experimental

### 2.1 Material preparation

Figure 1 shows the schematic of the experimental procedure in this study. The Cu-0.25 wt% Al powder was prepared by water atomization, followed by internal oxidation, diffusion, and reduction. Dispersion-strengthened copper powders with internal nano-Al₂O₃ were obtained as starting materials. The initial powders were ground via a high-energy planetary ball mill with a ball-to-powder weight ratio of 3:1 for 1 h. The rotating speed was kept at 200 rpm. The stainless steel vial with the capacity of 1 L and the stainless steel balls (10 mm in diameter) were matched with the planetary ball mill. During the ball milling process, the ethanol was added as the process-control agent to prevent excessive cold welding. Subsequently, the powder reduction was carried out at 450°C for 2 h in hydrogen. Then the powders were cold isostatic pressed at room temperature for 5 min with a pressure of 200 MPa, obtaining green bodies with a diameter of 45 and 40 mm in height. Next, the green bodies were sintered at 950°C for 1 h under a decomposing ammonia atmosphere and immediately extruded with a ratio of 36:1.

### 2.2 Characterization

The morphology of powder particles before and after ball milling was investigated and analyzed using a field-emission scanning electron microscope (FE-SEM; SUPER55, ZEISS, Germany) equipped with an energy-dispersive X-ray spectrometer (EDS; Oxford Instruments, Abingdon, UK). The microstructure of specimens for mechanical testing was also identified using FE-SEM after mechanically grounding and polishing. The Rockwell hardness (HRB) was measured with 980 N load using hardmeter (HBRV-187.5, Hua Yin, China). The Archimedes method was applied to measure the density for each sample (ASTM B962-13). Tensile tests were conducted on an MTS810 testing machine with a constant speed of 0.5 mm-min⁻¹ at ambient temperature. At least three tensile samples were prepared to test, and the average values were taken according to the standard of ASTM E1004-09. The inclusion analysis was carried out by using the ASPLEX inclusion analysis system (Aspex Explorer, FEI, USA). The analyzed area was 3 mm × 3 mm. In this study, only the inclusions with a maximum diameter of more than 0.5 μm were studied.
3 Results and discussion

3.1 Characteristics of powders

Figure 2(a) shows the SEM morphology of the water-atomized raw powders after reduction. It can be observed that the powders are displayed in an irregular shape with slightly sintered agglomeration. As shown in Figure 2(b), SEM observations with higher magnification reveal some spherical and white particles on the rough surface of the dispersion-strengthened powders. Further EDS analysis shows that these ~500 nm particles in size are basically spherical oxide composites mainly containing Al and O elements, as shown in Figure 3(a) and (b). These results are probably attributed to the aluminum segregation on the powder particle surface and oxidation during the water atomization process. According to previous research, the oxide particles are the fracture origin, which negatively influence the plasticity and fatigue properties. Therefore, it is essential to remove the oxide particles.

The SEM image of the cross-section of non-milling Al2O3 dispersion-strengthened (ODS) Cu powder is shown in Figure 3(c). There are white particles and lines on the grain gap. The concentration of Al and O on the grain boundary according to the EDS line-scan, as shown in Figure 3(d), is consistent with the findings obtained by Sabard and Low [31,32]. The concentration of Al and O on the grain boundary causes the PPB problem and results in poor plasticity, which is hard to be well coordinated with the matrix during the deformation process, so these large-sized Al2O3 particles would become a potential crack source and induce crack propagation. There are extraordinary concerns that the continuous distribution of Al2O3 particles along the grain boundaries and particle boundaries weakens the interface bonding in the ODS Cu alloy, which dramatically affects the plastic deformation capacity [33].

The SEM morphology of powders after ball milling is shown in Figure 2(c) and (d). Obviously, the powder surface morphology exhibits a significant difference before and after ball milling. In Figure 2(c), the powder surface is rugged, whereas all the particles present a more uniform shape with a smooth surface after ball milling shown in Figure 2(d). The morphology of the powders after ball milling has been ground into a flake shape.

3.2 Microstructure and density

Figure 4 shows the back scattering diffraction (BSD) result of sintered ODS Cu at 950°C for 1 h under a decomposing ammonia atmosphere. During sintering, Al2O3 cluster on particle boundaries weakens the interface between the two Al2O3 particles because Al2O3 can hardly

![Figure 2: Low-magnification (a and c) and high-magnification (b and d) SEM images of raw powder and milled powder.](image-url)
be sintered at this temperature. The sintering process also has been seriously hindered [34].

The black regions in Figure 4 present such residual pores in sintered bulks. Because of these defects, many pores are observed in the microstructure of sintered bulks of ODS Cu with poor sintered properties. The powder particles shown in Figure 4(b) are smaller than those shown in Figure 4(a) because ball milling process has opened the original agglomerated particles. Figure 5 shows the relative density of sintered samples. The density of non-milled and milled samples is 83.2 ± 0.3 and 80.5 ± 0.6%, respectively. The hardness and yield strength of powder particles were improved by ball milling, so the relative density of milled powder compact was lower than non-milled compact. Therefore, the sintering density of milled samples is lower. The density of non-milled and milled samples is 99.5 ± 0.4 and 99.7 ± 0.3% after extrusion. The main reason for this phenomenon is that the densification was strongly inhibited by Al2O3 in the copper matrix [35].

Figure 6 is the BSD microstructure of the extruded specimens. As observed from the cross-section of Figure 6(a) and (b), Al2O3 appears in the shape of a continuous net in the copper matrix. In the longitudinal section of the non-milled and milled samples shown in Figure 6(c) and (d), there were elongated oxide particles. In the cross-section and longitudinal sections, it could be found that the ball milling

![Figure 3](image3.png)

**Figure 3**: (a) SEM image of the ODS Cu powder surface, (b) the EDS of white spot shown in (a), (c) cross-section of ODS powder, and (d) EDS scan over the powder boundaries in (c).

![Figure 4](image4.png)

**Figure 4**: BSD morphology of sintered samples by (a) raw powder and (b) milled powder.
process reduced the number of inclusions and made them evenly distributed.

Besides, there was a significant change in the total inclusions and inclusions per unit area for each sample (shown in Table 1). The total number of non-milled inclusions is 7,151, whereas the number of inclusions after 1 h milling is 2,956. The number of inclusions per unit area decreases with ball milling, and the values of non-milled and milled are 58 and 36 mm$^{-2}$, respectively. The total number of inclusions decreases by 38% through ball milling.

Based on the rating criteria for PPBs reported by other researchers [36], the rating for PPBs of the extruded specimen without ball milling is Grade 3. With ball milling, the fine Al$_2$O$_3$ particles become discontinuous, indicating that the PPB problem decreases accordingly. After ball milling, the distribution of Al$_2$O$_3$ particles changes from the previously continuous network structure to a discontinuous condition along the boundary of the powders.

It is noticeable that the existence of a continuous network of Al$_2$O$_3$ particles along the boundary would cause a decrease in mechanical properties, especially in plastic-processing property. There are many factors affecting the PPB problem on the Cu–Al powder surface. However, water is highly in contact with Cu–Al alloy powders at high pressure during water atomization, and since the affinity of H$_2$O and Al is greater than Cu during water atomization, the elemental segregation of Al and O would occur on the powder surface [23].

| Milling time (h) | Numbers of total inclusions | Number of inclusions per unit area (mm$^{-2}$) |
|-----------------|-----------------------------|-----------------------------------------------|
| 0               | 7,151                       | 58                                            |
| 1               | 2,956                       | 36                                            |

Figure 5: Relative density changes after sintering and extrusion.

Figure 6: BSD morphology of as-extruded samples: (a and b) cross-section and (c and d) longitudinal section of (a and c) non-milled sample and (b and d) milled sample.
However, during the smelting of Cu–Al alloys, Al readily reacts with oxygen to form alumina. The atomizing nozzle is generally made of alumina. Under the repeated scour action of the high-temperature liquid flow, the inner hole of the nozzle gradually becomes bigger so that the alumina flushed away from the nozzle is mixed with the powders. In addition, the raw powders and impurities, which dropped from furnace lining during smelting, would lead to the presence of micron-sized Al$_2$O$_3$ particles [36].

### 3.3 Mechanical properties

The values of mechanical and physical properties are shown in Table 2 and Figure 7. The tensile strength is $435 \pm 5$ MPa and increases to $488 \pm 3$ MPa with an enhancement of 12% after ball milling. The yield strength of the milled sample is $417 \pm 4$ MPa, with 15% increase compared to the non-milled sample. As shown in Table 2, the elongation and section shrinkage of ball milling specimen are much higher than that without ball milling, an increase of 30 and 88%, respectively, indicating the beneficial effect of ball milling on enhancing the plasticity of ODS Cu alloy. These promising results may be attributed to the fact that ball milling weakens the impact of PPBs and improves the interface bonding. Figure 8 shows the schematic of ball milling process about the variation of oxides on the powder particle surface. Raw powders with PPBs were ball-milled in alcohol. Due to the violent collision during ball milling, part of the ceramic inclusions on the surface of the powder particles fell from the surface of the powder particles and entered into the alcohol. After ball milling, they were filtered out together with the alcohol. Under the interaction between the copper powder and the steel ball, the soft powder particles were squashed to produce layered composite powder, so that part of the ceramic particles originally on the surface of the powder was incorporated inside the powder [37]. Through the ball-milling treatment, the total content of ceramic inclusions is reduced, and at the same time, the related PPBs effect is weakened.

As the strength of the copper alloy is greatly affected by inclusions in the matrix, the larger the size and number of inclusions, the lower the yield strength and tensile strength of the material. Numerous reports have shown that cracks are apt to initiate around inclusions [39–42].

Herein, the number of inclusions has a greater influence on the section shrinkage than the elongation. Inclusions in copper reduce the transverse ductility of copper. Therefore, the tensile strength, yield strength, and elongation of copper material have been significantly improved by ball milling [38,39].

As shown in Figures 7 and 9(a) and (b), it is noted that the fracture surface contains typical dimples considered as the ductile fracture at low magnification. However, the substantial macroscopic difference in the fracture aspect is observed between non-milled and milled...
samples. In Figure 9(c), the mixed fracture surface consisted of fine dimples, and a few particle boundary facets can be found. These PPBs mostly contain fine powders with severe surface oxidation, which are difficult to be sintered and easily maintain the original state even after the extrusion process [24,43]. The oxide on the surface of the powder along PPBs can inhibit boundary migration and lead to interparticle fracture. In Figure 9(d), the phenomenon of PPBs could hardly be observed, and the fracture surface consists of fine dimples and some secondary cracks. Figure 9(e) and (f) in the high magnification of non-milled and milled samples shows small dimples due to grain tearing.

As seen in Table 2, the variation trend of the HRB is the same as the tensile strength. The hardness value of the sample without ball milling is 72.0 ± 0.9 HRB. The hardness reaches 82.2 ± 0.3 HRB after ball milling. Moreover, the electrical conductivity of the fabricated dispersion-strengthened copper remains nearly unchanged after ball milling. The electrical conductivity is unaffected before and after ball milling.

4 Conclusion

In this study, the effect of ball milling of dispersion-strengthened copper on its microstructure and properties was investigated. Conclusions are as follows:

1. The ball milling process significantly weakens the PPB problem of ODS Cu alloy compared with the non-milling samples.
2. After ball milling, the elongation increases by 30%, and the section shrinkage sharply increases by 88% compared with non-milling specimens.
3. The ultimate tensile strength and hardness of specimens reached to 488 ± 3 MPa and 82.2 ± 0.3 HRB with an enhancement of 12 and 14%, respectively.
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