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## Microstructure and mechanical properties of columnar-grained copper produced by the Ohno continuous casting technique



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#### ABSTRACT

The microstructure and mechanical properties of columnar-grained copper under casting and drawing conditions were investigated in the present work. It was found that the as-casted copper mainly consisted of continuous columnar grains with orientation preference along <100> and exhibited excellent plastic deformation ability. Moreover, the texture of columnar-grained copper transferred from <100> to <111> during the drawing process. A typical microstructural morphology as multangular-cup feature of as-casted copper as well as drawn copper was observed on the fracture surfaces. With increasing the drawn strain, the texture evolution and the grains broken occurred, leading to a significant increase in the work hardening rate and tensile strength.

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#### 1. Introduction

High quality copper has become a potential electrical and high-fidelity wire material for microelectronics and telecommunication applications due to excellent properties such as high extensibility and electrical conductivity at room temperature. However, internal defects in copper produced by tradition approaches, including cracks, inclusions and shrinkage porosity, lead to a reduction of the mechanical properties [1]. Furthermore, the transverse grain boundaries in copper also result in a relatively low elongation.

In recent years, much attention has been drawn to improve the plastic extensibility of copper [2,3]. Ohno continuous casting (OCC) process was introduced to fabricate metal alloys such as bismuth—tin binary alloys, aluminum alloys and copper with low defect rate and high-quality surface [4–7]. Additionally, OCC process could also be performed to generate elongated or single crystals [2]. Zhang et al. [8] prepared the pure copper rods with continuous columnar crystals by using the downward OCC equipment, and pointed out that the quality of the cast copper was attributed to several technological parameters including the mold temperature, cooling distance and casting speed. Wang et al. [9] fabricated copper rods by the continuous unidirectional solidification and found that the <100> columnar grains evolved into <111>, <110> and other fiber texture components during plastic deformation. Rittel et al. [10] studied the thermomechanical responses of single crystal and polycrystalline copper, and

reported that the scored energy of cold work for the polycrystalline copper was higher than that of single-crystal copper due to the presence of grain boundaries in the polycrystalline copper.

As mentioned above, the microstructure and texture evolution of different orientated copper has been studied [9,11,12]. However, most of the previous studies just focused on one side, i.e. the microstructure, properties or texture evolution, individually. There was rare report on their relationship and the deformation behavior of the columnar-grained copper fabricated by OCC technique. The questions remain whether the columnar-grained structure can improve the mechanical properties of copper and how the texture and work hardening rate change during cold drawing. The aim of this work is, therefore, to fabricate columnar-grained copper by the OCC technique, evaluate the microstructure and mechanical properties under casting and drawing conditions, and identify the underlying mechanisms.

#### 2. Experimental

#### 2.1. Preparation of copper bar

In the present study, the raw materials were copper ingots (produced by Yunnan Copper Co., Ltd., 99.99%). Initially, the columnar-grained copper bar (16 mm diameter) was fabricated by Ohno continuous casting. Firstly, the copper ingots were put into a graphite crucible and melted at 1150 °C for 30 min in argon atmosphere with a heating rate of 50 °C/min. Before the molten metal was introduced to the mold, the temperature of the molten metal was lowered to

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1100 °C, and the graphite mold was heated to 1100 °C. Then, the cooling water was turned on, a copper dummy bar was set at the mold exit, and then the initial copper bar (16 mm diameter) was casted. After that, the copper bar was drawn to one with a diameter of 6 mm through ten passes along the longitudinal direction at room temperature without annealing. Then, the specimens with diameters of 16 mm (as-casted), 12 mm (drawn strain  $\varepsilon$  0.58), 10 mm (drawn strain  $\varepsilon$  0.94), 8 mm (drawn strain  $\varepsilon$  1.39) and 6 mm (drawn strain  $\varepsilon$  1.96) were prepared.

#### 2.2. Microstructure and mechanical properties

The tensile samples at different drawn strains were cut from the copper bars along the drawn direction. Tensile tests were performed on CSS-44100 electronic universal testing machine and the loading speed was set at 2 mm/min. The microstructures of copper before and after tensile were examined via optical microscope and scanning electron microscope (SEM). The specimens for texture evolution were mechanically cut from copper bars along transverse direction (perpendicular to drawn direction). The phase was analyzed via X-ray diffraction (XRD) using CuK $\alpha$  radiation, and the diffraction angle  $(2\theta)$  varied from  $10^{\circ}$  to  $80^{\circ}$ . The X-ray diffraction with the applied voltage of 45 kV and current of 40 mA was also used to measure the pole figures. Orientation distribution functions (ODFs) were computed from the pole figures with series expansion method.

#### 3. Results and discussion

#### 3.1. Microstructure

Fig. 1 shows the microstructure of the as-casted copper along transverse and longitudinal direction, respectively. It can be seen from Fig. 1(a) that the grain size of the as-casted copper along the transverse direction was 500  $\mu m$ . Furthermore, it is of interest to observe that the columnar grains in the longitudinal section were along the solidification direction as shown in Fig. 1(b), which was similar to that reported by Wang et al. [9]. This was attributed to the heated mold in the whole OCC process [1]. The external heat prevented the nucleation on the surface of the mold, and made a large axial temperature gradient at the liquid–solid interface together with the intense cooling system [13]. Thus, the grains grew along the axial temperature gradient, and the continuous longitudinal columnar crystal was obtained.

Fig. 2 shows the microstructure of columnar-grained copper at different drawn strains. It can be seen that both the fiber structure and deformation bands increased with the increase of drawn strain. When the copper was drawn to the one with diameters of 12 mm and 10 mm (drawn strain  $\varepsilon$  was 0.58 and 0.94, respectively), the slight grain splits occurred in the transverse section (Fig. 2(a) and (c)), and the continuous columnar grains remained in the longitudinal section as shown in Fig. 2(b) and (d). However, when the diameter of copper was 8 mm (drawn strain  $\varepsilon$  was 1.39), several grains were broken and more splits appeared inside the grains in the transverse section, as shown in Fig. 2(e). Meanwhile, it can be seen from Fig. 2(f) that a few deformation bands appeared inside the continuous columnar grains in the longitudinal section. When the copper was drawn to 6 mm (drawn strain  $\varepsilon$  was 1.96), the transverse section was occupied by fiber stripes, as shown in Fig. 2 (g). Deformation and transition bands arranged nearly parallel to the drawing direction as shown in Fig. 2(h). This can be attributed to the activation of different slip systems and the differences of stress in adjacent regions [14].

#### 3.2. Texture evolution

Figs. 3 and 4 show the XRD spectrum and ODF map of the ascasted copper produced by OCC. It is observed that the spectra of the as-casted copper exhibited two peaks including (111) and (200). Moreover, the intensity of (200) was much higher than that of (111), indicating that most of the grains in the copper grew parallel to (200) crystal plane. It can be seen that in the section map of  $\varphi$  2=0° (Fig. 4), the main texture was <100 > casting texture and its orientation density value was much higher than that of others. Therefore, the initial texture of the as-casted copper was nearly <100 > casting texture.

The casting texture during continuous unidirectional solidification process largely depends on the crystal structure of the material. It is known that the preferential growth orientation of face-centered cubic (fcc) copper is <100>[15]. In this work, the columnar-grained copper mainly grew along <100>, as shown in Figs. 3 and 4. However, Chen and Yan [16] reported that with the help of seed crystal, copper with the initial orientation of <111> could also be produced by the Bridgman method. The <111> orientation was mainly due to the use of the seed crystal, and the grains would normally keep the same orientation with the seed crystal. In this work, no seed crystal was used during the OCC process, and this resulted in the <100> orientated grains in the as-casted copper.

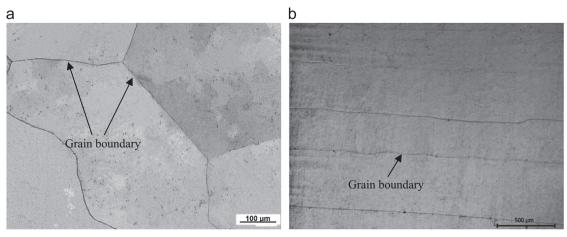


Fig. 1. Optical micrographs of the as-casted copper for (a) the transverse section and (b) the longitudinal section.

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