Texture and mechanical properties of Cu alloy by cryogenic high-speed rolling

S Lee1, 2, R Muchime1, R Matsumoto1 and H Utsunomiya1
1) Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan
2) Microstructure Analysis Technology Group, Research Center for Structural Materials, National Institute for Materials Science (NIMS), 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

aitam0408@gmail.com

Abstract. Cryogenic high-speed rolling (CHSR), where the sheet cooled in liquid nitrogen is supplied to rolls rotating at 1500 m/min at room temperature, was applied to several Cu alloys. After the CHSR, mechanical strength was improved with texture transition from the 'copper-type' to the 'brass-type' components. This trend was clearly observed in the case of Cu alloys with intermediate SFE. It is considered that the deformation mode changed from the dislocation slip to deformation twinning and shear banding under high Z parameter conditions of CHSR. It is also found that CHSR can improve the strength of Cu alloys with less decrease in electrical conductivity.

1. Introduction
Copper and copper alloys have been studied to investigate the effects of stacking fault energy (SFE) on the deformation behavior of FCC metals because deformation twinning is known to be the dominant deformation mode when SFE is less than 25 mJ/m² [1]. SFE of Cu (=78 mJ/m²) decreases with the addition of alloy components, such as Zn and Al. The change in SFE significantly affects the microstructure change by plastic deformation, which is an important factor in determining the mechanical and electrical properties of copper and copper alloy.

Deformation mode is mainly determined by the SFE of material, but it also varies with deformation temperature and strain rate [2, 3]. The effects of these extrinsic factors are not significant, but if they have huge changes, deformation mode can be made enough to change from crystallographic slip to deformation twinning.

The authors reported researches rolling with a severe operating condition, named cryogenic high-speed rolling (CHSR). In CHSR, rolling specimen only cooled in liquid nitrogen with rolls at room temperature and supplied to high-speed rolling [4,5]. In the process, heat transfer from the rolls to the specimen is limited because of the short rolling duration. The low initial temperature (~100 K) and high strain rate (~1500 s⁻¹) could realize the high Zener-Hollomon parameter (Z), leading to the change deformation mode from crystallographic slip to twin. Evolution in microstructures and change in mechanical properties by CHSR were studied in Cu-Zn [4] and Cu-Al [5] alloys. However, the evaluation of CHSR in pure Cu and analysis of texture evolution has not been studied yet.
In this study, CHSR was applied to Cu and Cu alloys. After the rolling, texture evolution was observed. In addition, change in mechanical strength and electrical conductivity was measured to identify the improvement of balance of strength – conductivity after CHSR.

### Table 1. Chemical composition and stacking fault energy of the materials used.

|        | Al | Zn | Fe | Si | Cu     | SFE [1,6] |
|--------|----|----|----|----|--------|-----------|
| OFC    | -  | -  | -  | -  | bal.   | 78 mJ/m²  |
| Cu-5Zn | -  | 5.1| -  | -  | 0.02   | 50 mJ/m²  |
| Cu-3Al | 3.11| -  | 0.31| -  | 0.01   | 25 mJ/m²  |
| Cu-7Al | 6.79| -  | 0.03| -  | 0.006  | 7 mJ/m²   |

### Table 2. Rolling conditions employed in this study

|                            | Low speed cold rolling (LSR) | Cryogenic high-speed rolling (CHSR) |
|---------------------------|------------------------------|------------------------------------|
| Roll diameter             | ∅ 310                        | ∅ 530                              |
| Rolling speed             | 5 m/min                      | 1500 m/min                         |
| Temperature               | RT                           | 77 K                               |
| Reduction in thickness    | Up to 60–80 %                |                                    |

*Specimens for CHSR were soaked in liquid nitrogen for 900 s before the rolling.

### 2. Experimental Procedure

Oxygen-free copper (OFC), Cu-5Zn [4], Cu-3Al [5], and Cu-7Al [5] alloy were used in this study. The chemical compositions and stacking fault energy [1,6] of the sheets are shown in table 1. The mean grain size of the alloys was 6.5 μm at OFC, 13.5 μm at Cu-5Zn, 19.9 μm at Cu-3Al, and 23.7 μm at Cu-7Al, respectively.

Rolling with two different conditions were carried out, as shown in table 2. CHSR was carried out on a 2-high, high-speed rolling mill with a roll diameter of 530 mm at 1500 m/min. Before the CHSR, specimens were soaked in liquid nitrogen for 900 s and then supplied to the rolls within 3 s. The sheet temperature before the CHSR was around 100 K, while that after the CHSR was still under room temperature. Conventional low-speed cold rolling (LSR) with a roll diameter of 310 mm at 5 m/min was also carried out at room temperature as the control group. The thickness was reduced up to 60-80%.

Microstructure and texture after the rolling were observed on the plane perpendicular to the transverse direction (TD) by a Scanning Electron Microscope with electron backscatter diffraction (SEM-EBSD) analysis in the cases of OFC and Cu-5Zn. The specimens were prepared by mechanical polishing until #2000 of SiC paper and 0.3 μm of Al2O3 powder, followed by electro-polishing in a solution (H3PO4 : C2H5OH : H2O = 50 ml : 50 ml : 100 ml), with a voltage of 5 V at 273 K. The operation voltage of the SEM was 15 kV. The step size in the Kikuchi-pattern scanning was 0.1-1 μm. The points with a confidence index (CI) less than 0.1 were excluded from the analysis. ND-IPF maps and (111) pole figures were plotted using TSL OIM Analysis 7 software.

In the cases of Cu-3Al and Cu-7Al, pole figure data were measured by the pseudo-Schulz reflection method using Rigaku SmartLab multipurpose X-ray diffractometer system equipped with Cu-Kα radiation. (111) pole figures were plotted using Smartlab Studio II software.

Vickers hardness test was conducted with an indentation load of 0.98 - 1.96 N (0.1 - 0.2 kgf) with a holding time of 15 s. The electrical conductivity of the specimen was measured using the four-probe method and expressed as a percent of International Annealed Copper Standard (%IACS), which is following,

\[
\%\text{IACS} = \frac{R_{\text{specimen}}}{R_{\text{annealed Cu}}} \times \frac{R_{\text{annealed Cu}}}{R_{\text{specimen}}}
\]
where $\kappa_{\text{specimen}}$ and $\kappa_{\text{annealed Cu}}$ is the conductivity of the specimen and that of the standard annealed copper, respectively. $R_{\text{specimen}}$ is the electrical resistivity of the specimen measured by the four-probe method, and $R_{\text{annealed Cu}}$ is the electrical resistivity of the annealed copper ($=1.7241 \times 10^{-8} \Omega \cdot m$, ASTM standard B193-02), respectively. The electric current was applied parallel to the rolling direction.

3. Results and discussions

3.1. Microstructure

Figure 1 shows the inverse pole figure (IPF) map showing ND orientation on the longitudinal section of the rolled sheets. As the reduction in thickness increased, elongated grains were observed parallel to the rolling direction. In the case of 30 % and 70 % LSR ed, ND//<111> orientation and ND//<112> orientation were mainly observed. On the other hand, in the case of CHSR, although the ND//<110> orientation was mainly observed, a little amount of ND//<111> orientation was observed. In addition, grains that were not elongated parallel to the rolling direction but diagonally developed were also observed, in the case of 70 % CHSR. It would be the twin deformation was locally occurred due to the high Zener-Hollomon parameter (Z) of CHSR.

Figure 2 shows the inverse pole figure (IPF) map on the longitudinal section of the rolled sheets showing ND orientation. Grains elongated in the rolling direction were observed. Deformation twins were formed in the inlined direction about 35 degrees to the rolling direction. Remnant grains are coarse and elongated in the rolling direction showing the ND//<110> orientation, while inlined and twinned grain showed ND//<111> orientation. CHSR showed a higher fraction of ND//<111> orientation than that of LSR.

Figure 3 shows the microstructures of the rolled Cu-3Al obtained by the EBSD analysis. Twin boundaries appeared locally with a narrow width in elongated grains in the 30 % rolled 3Al. After the 67 % rolling, shear bands appeared near the grains where the deformation twinning has occurred. The CHSRed specimen showed a higher fraction of shear bands than that of the LSR, as shown in figure 3(c) and (e). The fragmentation of grains by shear banding was also observed after the 67 % rolling, which contributes to the grain refinement. In the case of CHSR, the intersections of shear bands turn into rhomboidal prisms observed in the microstructure.

Figure 1. Inverse pole figure (IPF) map (ND orientation) of oxygen free copper (OFC) after cryogenic high speed rolling (CHSR) and conventional cold low-speed rolling (LSR). (a) Initial specimen, (b) After 30 % LSR (c) After 30 % CHSR, (d) After 70 % LSR, and (e) After 70 % CHSR

Figure 2. Inverse pole figure (IPF) map (ND orientation) of Cu-5Zn after cryogenic high speed rolling (CHSR) and conventional cold low-speed rolling (LSR). (a) Initial specimen, (b) After 60 % LSR (c) After 60 % CHSR, (d) After 80 % LSR, and (e) After 80 % CHSR [4]
Figure 4 showed the EBSD maps of the Cu-7Al. In contrast to other Cu alloys, the LSR and the CHSR showed similar microstructures in the case of the Cu-7Al. After 60% rolling, the deformation twins and shear bands appeared within selected elongated grains, which were narrower than those of Cu-3Al.

3.2. Textures after the rolling

{111} pole figures were drawn based on EBSD data in the case of OFC and Cu-Zn, as shown in figure 5. The LSRed OFC and Cu-5Zn showed typical 'copper-type' texture, which is \{112\}<111> orientation developed with a very sharp peak. On the other hand, in the case of CHSR, the less significant \{112\}<111> orientation and the peak spread toward the \{110\}<112>. In addition, as shown in figure 5(d), 80% CHSRed Cu-5Zn showed evidence of a typical brass-type texture, where two spread poles at the top and bottom are connected at the center.

In the cases of Cu-3Al and Cu-7Al, the formation of shear bands leads to the low confidence index (CI) in EBSD analysis. \{111\} pole figures were obtained using XRD because of the limited observing area and low CI in EBSD. Figure 6 shows the incomplete \{111\} pole figures after 60–67% rolled Cu-Al alloys. Intensities in pole figures were normalized with each own X-ray diffraction raw data. In these cases, typical 'brass-type' texture was observed, which was shown widely spread peak around at \{110\}<112> and \{112\}<111> orientation, connected at the center, regardless of the rolling method. In contrast to OFC and Cu-5Zn, the maximum intensity of CHSR was higher than that of LSR. It is considered that the conflict between 'copper-type' deformation due to high SFE and 'brass-type' deformation due to high Z in CHSR induced to decrease the maximum intensity. On the other hand, twin deformation occurs at room temperature with a low strain rate in Cu-Al with intermediate and low SFE alloys. In these cases, the increased maximum intensity could be thought of as the promoted 'brass-type' deformation by CHSR.

3.3. Mechanical and electrical properties after the rolling

Figure 7 shows the Vickers hardness of Cu and Cu alloys before and after the rolling. All of the initial specimens of Cu and Cu alloys were shown the Vickers hardness less than 100 HV. OFC showed the highest hardness (89 HV), which is the lowest initial mean grain size. However, the increase in hardness changes depending on the SFE of the alloys after the rolling. The tendency with the lower SFE, the higher hardness was observed. The 60% CHSRed Cu-7Al showed the highest hardness, which was 256 HV.
The CHSRed specimens showed higher Vickers hardness than those of LSRed specimens, regardless of the alloy system. However, the increased amount of hardness after CHSR was dependent on the alloy system, compared to LSR. For example, in the case of 30 % CHSRed OFC, only 7 HV improved, which was 5 % increasing. On the other hand, the 30 % CHSRed Cu-3Al showed 11 % improved hardness (217 HV). As the reduction in thickness increases, the effect of CHSR appears to be less effective. The 60 % CHSRed Cu-5Zn and Cu-7Al showed 6 % and 3 % hardness improvement, respectively. As the SFE decreases, the accumulation of dislocation density at each strain and accumulation limit of the dislocation density increases [6]. The higher Z parameter in CHSR affected a similar effect with the decrease in SFE [7]. It is considered that CHSR inhibited recovery and lead to rapid work hardening at the early stage of rolling, and reached the accumulation limit of dislocation density faster. However, Cu-7Al with 60 % CHSR showed a similar hardness compare to that of LSR. This is thought to be because too low SFE is dominant to induce insensitive to the changes in temperature and strain rate.

The electrical conductivity decreased with an increasing reduction in thickness, as shown in Figure 8. Decreasing SFE decreased conductivity remarkably. The CHSRed Cu-5Zn and Cu-3Al, which were intermediate SFE alloys, showed a similar (or slightly higher) conductivity than those of the LSRed specimens. Meanwhile, the conductivities after CHSR were less than those after LSR, in the cases of OFC and Cu-7Al. However, it is notable that CHSR improved to the high strength compare to LSR; the electrical conductivities of both specimens are comparable.

3.4. Effect of cryogenic high-speed rolling on Cu alloys
The balance between Vickers hardness and electrical conductivity is shown in Figure 9. It was found that the balance of CHSR improved compare to that of LSR; the details are as followed.

In the case of OFC with high SFE, it was confirmed that the electrical conductivity also decreased with the improvement of the hardness. Therefore, the balance has improved but is less effective than other alloy systems. Although CHSR induced twin deformation and development of brass-type texture, slip deformation and development of copper-type texture was found to be dominant.

![Figure 5. {111} pole figures after cryogenic high speed rolling (CHSR) and conventional cold low-speed rolling (LSR). (a) 70 % LSR, OFC, (b) 70 % CHSR, OFC (c) 80 % LSR, Cu-5Zn, and (d) 80 % CHSR, Cu-5Zn](image1)

![Figure 6. Incomplete {111} pole figures after cryogenic high speed rolling (CHSR) and conventional cold low-speed rolling (LSR). (a) 67 % LSR, Cu-3Al, (b) 67 % CHSR, Cu-3Al (c) 60 % LSR, Cu-7Al, and (d) 60 % CHSR, Cu-7Al](image2)
On the other hand, in the case of Cu-7Al with very low SFE, the effect of improving the hardness and electrical conductivity of CHSR was not significant compared to LSR. In this case, the twin deformation and the development of brass-type texture are too dominant, and CHSR is less effective.

It is found in the cases of Cu-5Zn and Cu-3Al, the balance was significantly shifted to the right, which promoted twin deformation, leading to improvement in properties and development of the brass-type texture. Therefore, it was confirmed that CHSR improves the strength-conductivity balance in the copper alloy, and it was more clearly observed when SFE was intermediate range 25 – 50 mJ/m².

4. Conclusion

Cryogenic high-speed rolling (CHSR), where the specimen is cooled in liquid nitrogen and supplied to high speed rolling with rolls at room temperature, of the Cu and Cu alloys (OFC, Cu-5Zn, Cu-3Al, and Cu-7Al) was carried out to investigate the mechanical and electrical property and microstructure and macrotexture evolution. The remarks obtained are as follows,

1. CHSR promoted twin deformation and the development of brass-type texture.
2. CHSR is more effective to Cu-5Zn and Cu-3Al with intermediate SFE in the balance of Vickers hardness and electrical conductivity. In other words, it is less sensitive in the case of OFC with high SFE and Cu-7Al with extremely low SFE.

Acknowledgments

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