

# In-Situ EBSD Observations of Bending for Single-Crystalline Pure Copper

Takashi Sakai, Saori Yoshikawa, and Hideo Morimoto

**Abstract**—To understand the material characteristics of single- and poly-crystals of pure copper, the respective relationships between crystallographic orientations and microstructures, and the bending and mechanical properties were examined. And texture distribution is also analyzed. A bending test is performed in a SEM apparatus and while its behaviors are observed in situ. Furthermore, some analytical results related to crystal direction maps, inverse pole figures, and textures were obtained from EBSD analyses.

**Keywords**—Pure Copper, Bending, Single Crystal, SEM-EBSD Analysis, Texture, Microstructure

## I. INTRODUCTION

IN recent years, to attain higher precision in bending work, studies of microscopic material properties devoting attention to the crystal texture of materials have been promoted extensively [1]-[6]. Crystal texture means a distribution state of direction of a crystal lattice of each crystal grain present in polycrystalline metallic materials. Its close relation with mechanical properties of metallic materials is well known.

However, although various studies related to such changes in aggregate structure before and after bending process have been presented in the literature, exactly how each crystal changes during bending work remains unresolved. Behavior of changes during bending is an important factor for accurately forecasting changes that might occur after bending. Therefore, such behavior should be carefully clarified.

The object of this study is to clarify the behaviors of an aggregate structure accompanied by changes in bending angle through EBSD (Electron Backscatter Diffraction) analysis using a pure copper single-crystal material while a bending test is performed in a SEM (Scanning Electron Microscope) and while its behaviors are observed in situ. Observation of any point on the single-crystal material exhibits the same orientation. Therefore, the material is only slightly affected by a crystal grain boundary or crystal orientation. The method therefore enables observation of simple deformation behaviors. In light of these characteristics, this study identified processes of changes of aggregate structure during deformation at compression side, neutral area, and tension side

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of bending deformation. The results of this study were compared with observations of pure copper polycrystalline material performed under the same conditions. Finally, bending deformation characteristics can be discussed based on those results.

## II. SPECIMEN AND EXPERIMENTAL METHOD

### A. Specimen

Bending test specimen, a single-crystal material and polycrystalline material of pure copper, were produced for use as specimen in these experiments. The specimen dimensions were 30mm(B)×5mm(W)×1mm(H). The coordinate system presented in Fig. 1 is defined. The single-crystal material is a circular cylinder defined by  $\{111\}$  in the axial direction and  $\{110\}$  and  $\{112\}$  laterally. This material was cut using a wire cutter to produce materials of two types: a  $\{111\}$  single-crystal test piece having TD// $\{111\}$ , RD// $\{112\}$ , ND// $\{110\}$  and a  $\{122\}$  single-crystal test piece having TD// $\{122\}$ , RD// $\{110\}$ , ND// $\{113\}$ . Regarding polycrystalline materials, 99.96% pure copper (JIS-C1020) was cut similarly for use as the specimen. These specimens were subjected to wet polishing using emery paper and buffing polishing using  $Al_2O_3$  particles and colloidal silica solution as the abrasive compound.

### B. Measurement of Crystalline Orientation

The SEM-EBSD apparatus used in these experiments is a thermal field emission scanning electron microscope (JSM-7001F; JEOL). The EBSD images were taken using a CCD camera provided to SEM. Fingerprinting of the Kikuchi line was performed sequentially for every position of the incident electron beam from crystal structure data of a sample to ascertain the crystal orientation.

### C. Bending Test Machine

For in-situ observation in the present experiments, a bending testing machine (TSL Solutions) was used. Fig. 2 shows the bending testing machine, which can be mounted on SEM and which can perform bending tests with observations realized in the SEM lens barrel. Bending deformation by the bending testing machine is of three-point-bending. The bending load is applied while the fulcrum points at both ends are elevated. Because of this mechanism, the position coordinate at the bending part does not change. Observations before and after bending deformation are made at the fixed point without changing the sample platform coordinates.

Control of the bending testing machine in the SEM lens barrel was performed using software (Bend 4.54) installed on

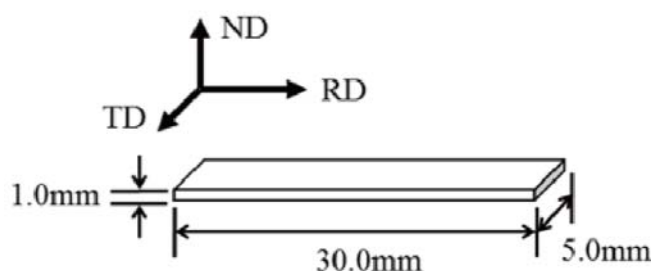


Fig. 1 Dimensions and directions of bending specimen

a personal computer. Controlled items are the fulcrum elevation displacement in  $0.001\mu\text{m}$ , bending load in  $0.01\text{N}$  and the loading speed. The maximum elevation displacement of the apparatus is  $10000.00\mu\text{m}$ ; the minimum load is  $100.00\text{N}$ . In this study, the degree of bending deformation amount is defined by the bending angle. Therefore, results obtained from preliminary angle measurement test are used for the input of elevation displacement corresponding to bending angle at observation. The maximum bending angle of  $\theta=90^\circ$  corresponds to the elevation displacement of  $9727.700\mu\text{m}$ .

#### D. Bending Test and In-situ Observations

A specimen subjected to polishing was set to the bending testing machine. The aggregate structure was observed in situ in the SEM-EBSD apparatus. Measurements were taken at four points from the upper compression side in the plate thickness direction at the bending part. Data obtained at four points were coupled in later analysis for use as data representing the whole bending part. This measurement was conducted seven times in all with bending angles of  $\theta=0^\circ, 15^\circ, 30^\circ, 45^\circ, 60^\circ, 75^\circ,$  and  $90^\circ$ . Experiments of  $\{111\}$  pure copper single-crystal material,  $\{122\}$  pure copper single-crystal material and pure copper polycrystalline material were conducted under identical conditions.

### III. EXPERIMENT RESULTS AND DISCUSSION

#### A. IPF Map

Fig. 3 portrays an IPF map of  $\{111\}$  pure copper single-crystal material,  $\{122\}$  pure copper single-crystal material, and pure copper polycrystalline material. The IPF map is an orientation mapping image in which scanned data are color-coded according to the inverse pole figure color key. Therefore, with pure copper single-crystal material in which the same orientation is aligned uniformly, a blue color map showing the  $\{111\}$  orientation was observed in  $\{111\}$  pure copper single-crystal material and light blue map showing  $\{122\}$  orientation was observed in  $\{122\}$  pure copper single-crystal material. With the  $\{111\}$  pure copper single-crystal material, although the initial orientation before deformation  $\theta=0^\circ$  was distributed in uniform fashion, changes in orientation at lower part were observed as the deformation made progress. Such changes were not observed with the

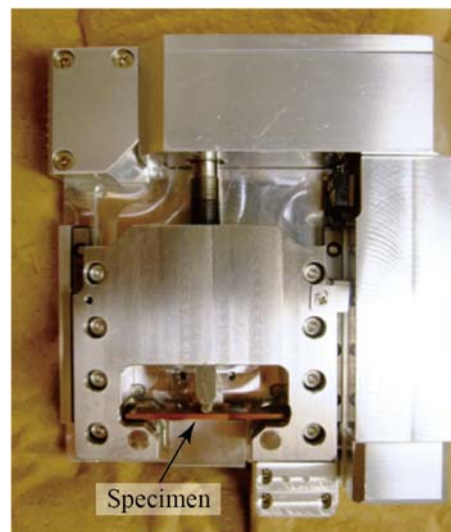


Fig. 2 Bending apparatus

$\{122\}$  pure copper single-crystal material. With pure copper polycrystalline material, the change in orientation started from the upper right part of the map as deformation progressed.

#### B. Comparison and Discussion

Fig. 4 (a) shows the  $\{111\}$  pure copper single-crystal material. Fig. 4 (b) shows the  $\{122\}$  pure copper single-crystal material. Fig. 4 (c) presents a graph showing the transition of the average GOS (Grain Orientation Spread) level at the compression side, neutral area, and tension side obtained from GOS map. Although the single-crystal material has no grain boundary, this material itself might be regarded as one greater crystal grain. Therefore, the three graphs shown above are used for comparison of changes in difference of orientation among the three test materials. First, the fact that changes in the difference of orientation at the tension side are the greatest is common to each graph. It might be said that particularly with a single-crystal material, a marked change occurred after  $\theta=30^\circ$ . With polycrystalline material, it occurred at  $\theta=15^\circ$  to  $75^\circ$ . It is therefore considered that a metallic mold condition in which changes are concentrated to the tension side in three-point bending test is confirmed by results of this study. If attention is devoted to the compression side, then changes in the initial stage of  $\theta=0^\circ-15^\circ$  exhibit different behaviors than those that occurred after  $\theta=15^\circ$ . This is regarded as being attributable to a small flaw caused by polishing. It can be recognized as a difference of orientation at  $\theta=0^\circ$  that is reduced because of minute stress load at the initial stage. Therefore, this trend is remarkable with single-crystal material that has no grain boundary, for which polishing flaws persist.

### IV. CONCLUSIONS

Bending tests were performed for this study in an SEM lens barrel using initial displacement  $\{111\}$  pure copper single-crystal material, initial displacement  $\{122\}$  pure

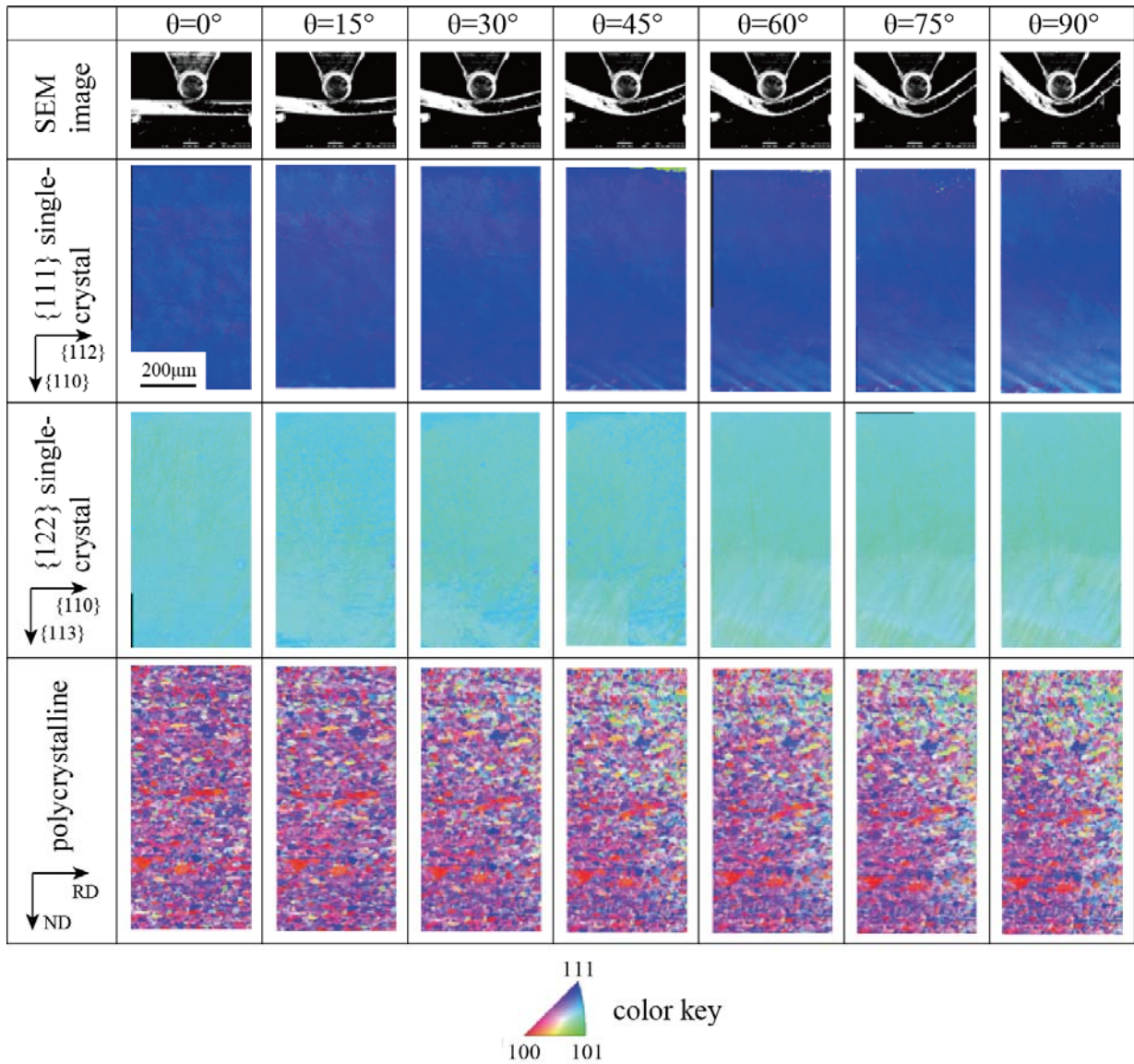


Fig. 3 IPF map of {111} and {122} pure copper single-crystal material, and polycrystalline material

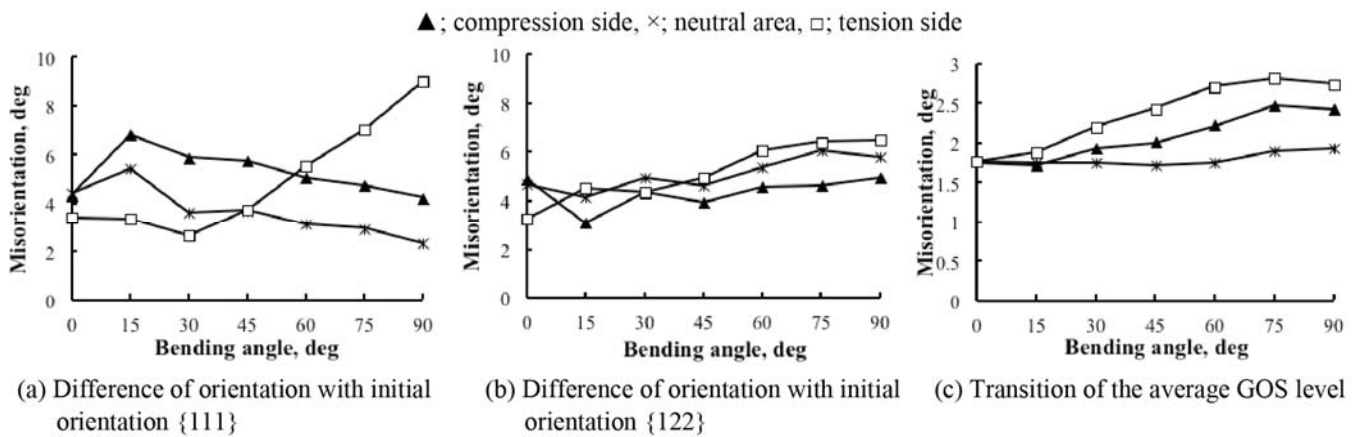


Fig. 4 Transition of average difference of orientation

copper single-crystal material, and pure copper polycrystalline material. The changes in aggregate structure accompanied by bending deformation were investigated in situ. Furthermore, deformation characteristics were investigated through comparison of results obtained from three materials, which revealed the following findings.

- (1) Changes in aggregate structure accompanying bending deformation of pure copper single-crystal material depend on a sliding system.
- (2) For three-point bending tests used in this study, a metallic mold condition prevails in which changes are concentrated to the tension side.

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