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Texture Observation of Bending by XRD and EBSD Method

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***Abstract*—**The crystal orientation is a factor that affects the microscopic material properties. Crystal orientation determines the anisotropy of the polycrystalline material. And it is closely related to the mechanical properties of the material. In this paper, for pure copper polycrystalline material, two different methods; X-Ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD); and the crystal orientation were analyzed. In the latter method, it is possible that the X-ray beam diameter is thicker as compared to the former, to measure the crystal orientation macroscopically relatively. By measurement of the above, we investigated the change in crystal orientation and internal tissues of pure copper.

Keywords—Bending, electron backscatter diffraction, X-ray diffraction, microstructure, IPF map, orientation distribution function.

I. INTRODUCTION

MODERN bending is necessary to estimate spring back precisely and to process a target angle with high accuracy. Although conventional bending presumes spring back using mechanical characteristics such as a Young's modulus, precision processing further requires an understanding of texture [1].

This study measured the texture of pure copper subjected to bending by SEM-EBSD and XRD. Identical positions on a through-thickness plane after bending underwent texture measurement with EBSD and XRD [2]. The result was compared and examined quantitatively [3]. Moreover, crystal orientation distribution was measured quantitatively in the through-thickness direction and near the surfaces in regions subjected to bending using EBSD. An experimental study was conducted for the variation of crystal orientation, texture formation, and microstructure evolution.

II. SAMPLE MATERIAL AND EXPERIMENTAL

For use as a sample material, a 99.96% Cu polycrystalline plate was machined into bending test pieces of 10 mm width, 5 mm height, and 1 mm thickness. EBSD specimens were prepared by cutting the test pieces after a bending test. Mechanical polishing is carried out by using emery paper and Al_2O_3 abrasives. And then, electrolytic polishing was done using a solution of H_3PO_4 300 ml+H_2O 100 ml at 5 V and 0.2 A for etching duration of 5s.

The bending test was conducted using a 500 kN universal tester using an 88° V-bending punch and die at a bending radius of 0.6 mm. Spring back was measured using a high-precision angle sensor. The crosshead lowering speed was 1 mm/min.

Crystal orientation analysis was conducted using software (OIM ver. 6.0; TSL Solutions, Inc.) for EBSD measurement with step size of 1 μm. Fig. 1 shows EBSD measurement points on a bending test piece. Measurements were conducted at five points on the TD plane at the bent part of the test piece after the test from the upper to lower ends. For the five points of measurement, the uppermost point under a compressive stress, the point on the neutral axis, and the lowermost point under a tensile stress are hereinafter denoted respectively as Top, Center, and Bottom. Measurements were also conducted at five points on the TD plane at the unbent part in the same manner for comparison before and after bending testing. Furthermore, measurements were taken at seven points along the RD at the surface regions on the compression and tension sides by bending in this study.

Crystallographic orientation was investigated using XRD measurements (RINT-2000; Rigaku Corp.). The points of measurement are also presented in Fig. 1. A collimator for X-ray irradiation of a diameter of 1 mm was adopted for measurements so that measurements were conducted at one point each on the TD plane before and after bending.

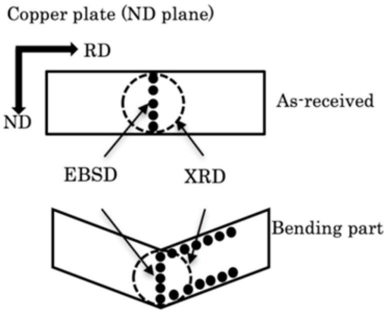

Fig. 1 Schematic illustrations of measured point of SEM-EBSD and XRD

III. QUANTITATIVE EVALUATION PROCEDURE OF TEXTURE

The properties and acquisition procedures of quantitative data from crystallographic orientation information measured using EBSD and XRD are described below.

Quantitative texture data were acquired from EBSD measurements by determining the crystallographic orientation through indexing orientation information according to the location (pixel) of incident electrons. Applying this procedure over the whole region of measurement at arbitrary measurement intervals provides an orientation map and the

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fraction of each orientation component in the range of this map. As an acquisition procedure of the quantitative data of texture by XRD in an ODF chart obtained by determining a crystallographic orientation distribution function by analysis code Standard ODF [4], the degree of integration of texture on a φ=45° section was determined quantitatively using a self-coded program at our laboratory. This program computes the pole density and orientation density not exceeding 5° from the ideal orientation of the main texture in the ODF chart for φ=45° [5].

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. *EBSD Analysis*

Letting the radius of curvature of a neutral plane be *R* and the radius of curvature at a position separated by *Y* from the neutral plane be *r* in this bending test, then the strain at the bent part can be found using (1):

$$\varepsilon = \frac{r-R}{R} = \frac{Y}{R} \tag{1}$$

Fig. 2 presents the strain distribution over the thickness according to (1). The neutral plane was located at the upper part along the thickness in this experiment, so that strain was observed in the figure to turn from compressive to tensile at 0.3 mm from the top surface.

Fig. 3 depicts the inverse pole figure (IPF) map obtained from the EBSD measurement before and after the bending test. Grains uniformly elongated to the RD are distributed over thickness in as-received specimen before bending (a), in which the grain orientation was integrated into {111} and {001} by rolling and heat treatment. However, the specimen after bending (b) exhibited fine equiaxed grains under compressive stress, but exhibited elongated grains under tensile stress. This change in grains closely matched the strain distribution presented in Fig. 2. The grain morphology took an elongated shape bordering on around 0.3 mm in depth from the top surface.

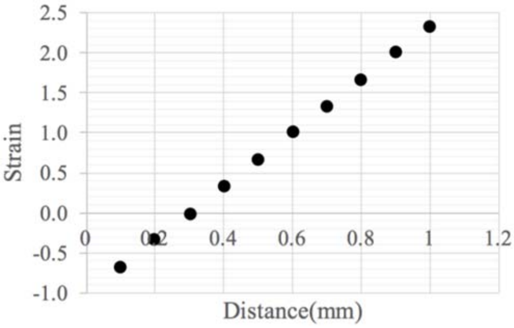


Fig. 2 Bending point strain of thickness

Fig. 4 presents an IPF map along the RD on the compression side of the specimen after bending. Fine grains observed in (b) were distributed to about 0.6 mm along the RD. An IPF map along the RD on the tension side of the specimen after bending is presented in Fig. 5, which suggests that elongated grains distributed along the RD. The orientation change was also remarkable. Crystal rotation from {111} to {011} was observed bordering on about 0.35 mm from the top surface, probably because {111} and {001} were developed strongly at the bent part as a result of a tensile stress state by bending, but change in a stress state to compression at a certain position triggered crystal rotation to {011}. The stress distribution states in the present measurement range indicated that this rotation took place under tensile stress in the bent part. Consequently, we presume the necessity of considering the effects not only of tensile stress but also of compressive stress on the tension side of the bent part in this measurement range.

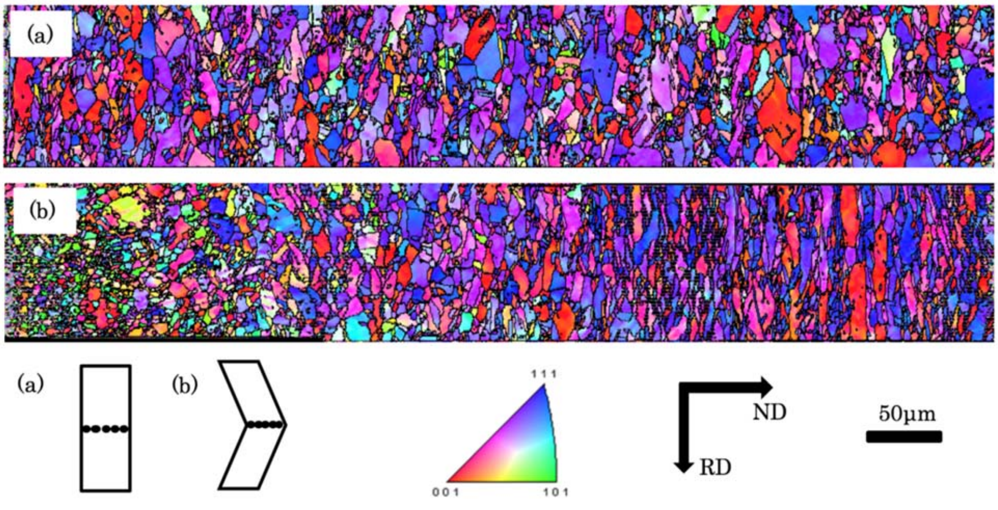


Fig. 3 IPF map of (a) as-received and (b) bending part

TABLE I
COMPARISON OF THE MEASUREMENT RESULTS OF THE XRD AND EBSD OF AS-RECEIVED AND AFTER BENDING

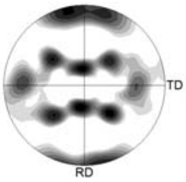
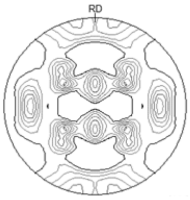
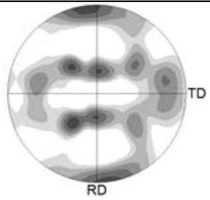
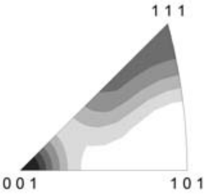
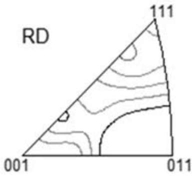
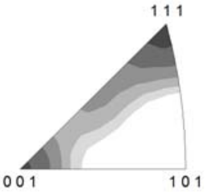
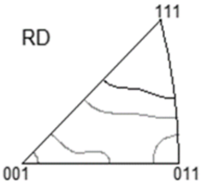
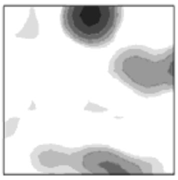
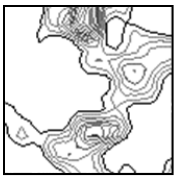
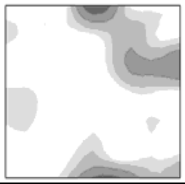
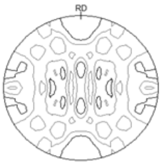
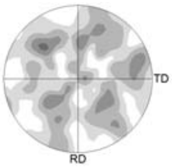
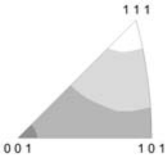
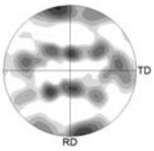
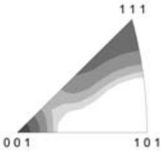
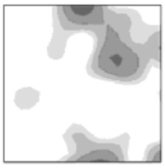
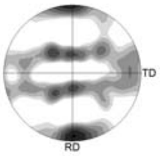
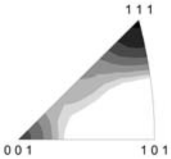
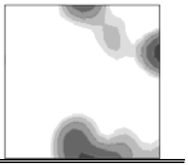
	As-received			Bending	
	EBSD	XRD		EBSD	XRD
PF(111)			PF(111)		
IPF(RD)			IPF(RD)		
ODF(45°)			ODF(45°)		

TABLE II
COMPARISON WITH THE RESULTS OF THE XRD AND THE RESULTS OF EACH MEASUREMENT POINT OF THE EBSD

PF(111)		IPF(RD)		ODF(45°)	
XRD	EBSD	XRD	EBSD	XRD	EBSD
	Top 		Top 		Top 
max = 5.0 5.000 3.624 2.627 1.904 1.380 1.000 0.725	Center 	max = 2.8 4.500 3.331 2.466 1.825 1.351 1.000 0.740	Center 	max = 5.0 15.000 8.727 5.078 2.954 1.719 1.000 0.582	Center 
	Bottom 		Bottom 		Bottom 

B. Comparison of Analysis Results by EBSD and XRD

Table I presents a comparison of the pole figures (PF), the IPFs, and the orientation distribution functions (ODF) of specimens before and after the bending test computed based on measurement data by EBSD and XRD. The PFs of the specimen before bending by both measurement methods suggest formation of a structure very similar to the pure metal type structure that is the rolling texture of f.c.c. metal. The IPF along the RD indicated prominent integration to {001} and {111} to the rolling direction. The ODFs by both measurement methods showed development of the Cube orientation as a recrystallization texture and the Copper orientation as a rolling texture. Accordingly, the measurement results of the specimen before bending were very similar irrespective of measurable ranges and measurement areas. Regarding the specimen after bending, the PF by EBSD shows an identical structure to that before bending, whereas the PF by XRD shows no rolling texture but very random orientation. The IPF by EBSD suggests integration to {001} and {111}, but to {001} and {011} by XRD. The ODF by EBSD still presents formation of the Cube orientation and Copper orientation, whereas development of {112}<110> and {221}<110> components were remarkable in the ODF by XRD.

Table II compares analysis results obtained separately for three measurement points Top, Center, and Bottom at the bent part by EBSD with overall results by XRD. The PFs show a random orientation at Top by EBSD, but no similarity to the results obtained by XRD. The IPFs indicate weak development of {011} at Top, which is a similar integration mode to the result of XRD. The ODF results demonstrate similarity between the result at Top by EBSD with integration to {112}<110> and an integration mode in ODF charts by the XRD results. Consequently, the results described above suggest that difference in analysis results by EBSD and XRD arose from differences in the measurable ranges. However, further local EBSD analysis of crystal orientation at the bent part revealed significant similarity between the results of macroscopic orientation and those at the compression side. Accordingly, this study suggests that the orientation change is prominent at the compression side in the upper part of the bent part.

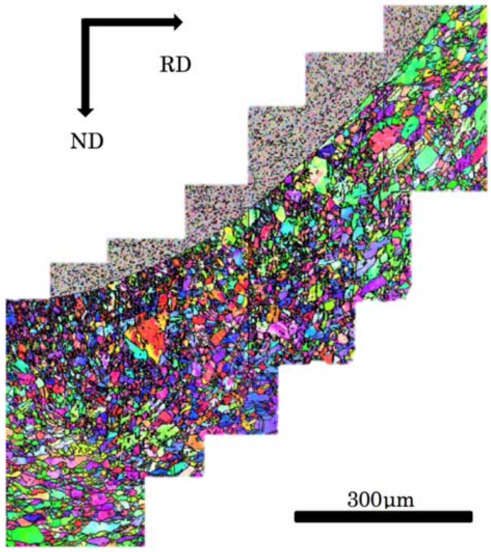


Fig. 4 IPF map of compression side

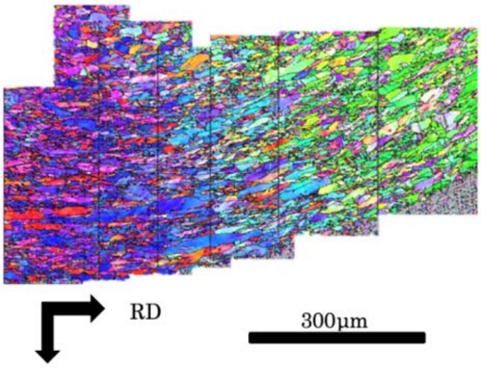


Fig. 5 IPF map of tension side

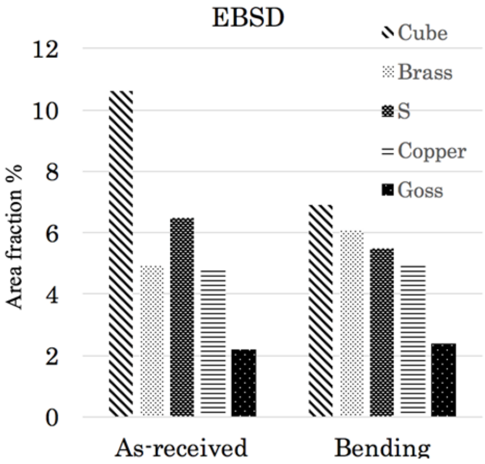


Fig. 6 Quantitative results of texture by EBSD

C. Quantitative Evaluation of Texture

Figs. 6 and 7 respectively present charts expressing texture, quantitatively computed based on the orientation information obtained by EBSD and XRD. The Cube orientation declined after bending, but little change was observed for other orientations. Fig. 6 depicts that the S orientation as a rolling texture had the highest value, followed by the Cube orientation as a recrystallization texture before bending, although the S orientation and the Cube orientation declined sharply after bending.

It is difficult to evaluate the rolling texture accurately using EBSD. Decline in the S orientation as a rolling texture can be evaluated using XRD in this measurement.

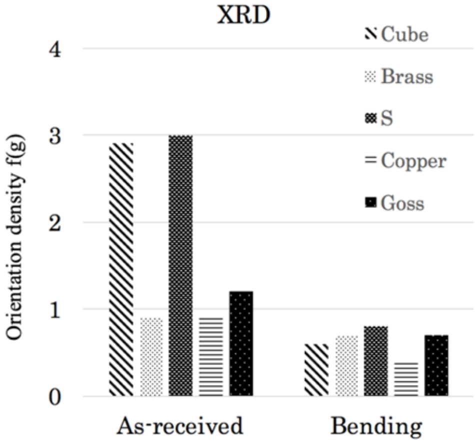


Fig. 7 Quantitative results of texture by XRD

V. CONCLUSIONS

Change of crystallographic orientation and texture accompanying bending of polycrystalline Cu was determined by EBSD as a microscopic measurement and XRD as macroscopic measurement in this study. The following results were obtained:

1. EBSD measurement results indicate that bending deformation brought about change in grain morphology

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and orientation in the microscopic structure according to a stress state at each point of measurement of the bent part.

2. Change of the grain morphology and orientation along the RD took place within about 0.6 mm at the surface regions on the compression side at the bent part because of compressive stress by bending.
3. Change of grain morphology and orientation occurred at the surface regions on the tension side at the bent part because of tensile stress. However, the stress state changed bordering on about 0.35 mm in depth; rotation took place to {011} by compression.
4. Comparison of measurement results by EBSD and XRD before bending indicated marked similarity, with a certain difference after bending. This difference is attributable to the change of stress in the upper part of the bent part under compressive stress. Results of this study suggest that orientational change is prominent at the compression side in the upper part of the bent part.
5. Quantitative analysis results of texture detected persistence of the S orientation as a rolling texture by orientation observation in a macroscopic range.

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