実験報告書様式(一般利用課題・成果公開利用)

<b>MLF</b> Experimental Report	提出日 Date of Report
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課題番号 Project No.	装置責任者 Name of responsible person
2013P0006	T. ISHIGAKI, A. HOSHIKAWA
実験課題名 Title of experiment	装置名 Name of Instrument/(BL No.)
Measurement of Texture and Dislocation Density by	iMATERIA
using iMATERIA	実施日 Date of Experiment
実験責任者名 Name of principal investigator	2014.3.7-8
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試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと) Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.
(1) Cold rolled low carbon steel, Fe-0.1mass%C, 10mm × 10mm × 10mm
(2) Annealed low carbon steel, Fe-0.1mass%C, $10mm \times 10mm \times 10mm$
(3) Multiphase steel, Fe-18mass%Ni-0.1mass%C, 10mm × 10mm × 10mm
(4) Magnesium alloy AZ31, Mg-3.0mass%Al-1.0mass%Zn, 10mm × 10mm × 10mm
(5) Cold rolled aluminum alloy, Al-0.20Si, 10mm ×10mm ×10mm

## 2. 実験方法及び結果(実験がうまくいかなかった場合、その理由を記述してください。)

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

For modern automobile, civil engineering and other industries, the high value-added structural metallic plates and sheets with lower energy consumption and longer service life expectancy have been emphasized than ever, and the simultaneous bulk microstructure and texture control is thought as an important way to realize advanced materials sheets with high strength and high formability. Recently, the rapid bulk texture measurement using neutron diffraction has been required for new materials R&D activities. At J-PARC center, iMATERIA time-of-flight neutron diffractometer was being employed to establish a reliable technical environment for rapid bulk texture measurements. Moreover, iMATERIA was also being employed to evaluate the microstructure characteristics of non-textured and textured multiphase materials, including dislocation density distribution and phase volume fraction. In order to confirm the technical environments of iMATERIA texture measurement/analysis, the full pole figures of some reference samples were measured through 120 times of  $\chi/\phi$  rotations using the Euler cradle at TAKUMI neutron diffraction.

## 2. 実験方法及び結果(つづき) Experimental method and results (continued)

The back-scattering detector banks, the sample-environmental detector banks and the low-angle detector banks were primarily divided into 82, 96, 180 detector groups, respectively, and the neutron event-type neutron diffraction information from each detector group was utilized to compose an independent neutron diffraction pattern. An  $\omega$ -axis rotatable sample exchanger with 30 sample holders was employed to control the sample rotations along its normal direction. As a fundamental measurement,  $10 \times 10 \times 10$  mm<sup>3</sup> cubes of a cold rolled aluminum alloy and a multiphase steel as reference samples were rotated to different orientations with  $\omega$ =0, 20, 90, 110 degrees to collect the crystallographic orientation information. Rietveld texture analysis was carried out using the MAUD (materials analysis using diffraction) software technique.

Fig.1 showed the neutron diffraction patterns of the interstitial free steel collected from the BS bank, together with the relative change in full width at half maximum (FWHM) with the tensile pre-strain before unloading. The changes in peak intensity revealed a strong texture effect. The relative changes in FWHM evaluated with FWHM/FWHM<sub>0</sub> suggested that the dislocation density has evident orientation-dependent distribution characteristics, and the further investigation is being carried out using the CMWP software (convolutional multiple whole profile fitting, Fig.1b).



Fig.1 Relative change in FWHM values (a) and CMWP analysis for dislocation density (b).

Fig.2 showed the sample exchanger employed at iMATERIA, recalculated (111) complete pole figure from iMATERIA and the relevant result from TAKUMI for reference. The recalculated (111) complete pole figure of an aluminum alloy sample from iMATERIA shows a cold rolled texture comparable with the result from TAKUMI neutron diffraction texture measurement, suggesting that iMATERIA is possible to be applied to the general bulk texture measurements.



Fig.2 Batch texture/dislocation density measurement using sample exchanger (a), recalculated (111) pole figure of cold rolled aluminum alloy from (b) iMATERIA (4 time of  $\omega$ -axis sample rotations in 20 minutes) and (c) TAKUMI (120 times of  $\chi/\phi$  2-axis sample rotations in 120 minutes).