MLF Experimental Report	提出日 Date of report
実験課題番号 Project No. 2013P0102 実験課題名 Title of experiment Strength of Materials and Related Engineering 実験責任者名 Name of principal investigator Yo Tomota 所属 Affiliation Ibaraki University	装置責任者 Name of responsible person Kazuya Aizawa 装置名 Name of Instrument/(BL No.) BL19 TAKUMI 利用期間 Dates of experiments 2013: 4/8-9; 5/6-9; 5/12-14 2014: 3/30-4/1; 4/2-5

1. 研究成果概要(試料の名称、組成、物理的・化学的性状を明記するとともに、実験方法、利用の結果得られた主なデータ、考察、結論、図表等を記述してください。

Outline of experimental results (experimental method and results should be reported including sample information such as composition, physical and/or chemical characteristics.

Main topics in this project include the following three groups; LPSO/Mg alloys, superconductor materials and advanced steel.

(1) LPSO Mg alloys

In situ neutron diffraction during compressive deformation was performed using a 18R $Mg_{85}Zn_6Y_9$ 1-directionally solidified specimen, aiming at obtaining the new insights on “kink deformation mechanism” proposed for Mb-based synchronized LPSO alloys. Here, a new technique with combination of macroscopic strain measurements and acoustic emission was employed, that was recently developed by BL19 group. Application of acoustic emission to materials science has been started in 1950 but it was rather difficult to use for quantitative evaluation on plastic deformation of metals and alloys; only qualitative discussion has been reported so far. In this project, the simultaneous measurements of neutron diffraction, strain/displacement, and acoustic emission was carried out. As a result, it was realized to distinguish the kink deformation from twinning by comparing lattice strain change with AE signals like energy, frequency and/or emission time interval. Here, macroscopic behavior can be interpreted based on atomic scaled information for plastic deformation mechanism. In addition, using an Euler cradle, texture evolution during plastic deformation with kink deformation was examined. It was found that kink bands were formed uniformly throughout a specimen with compression as shown in Fig. 1.

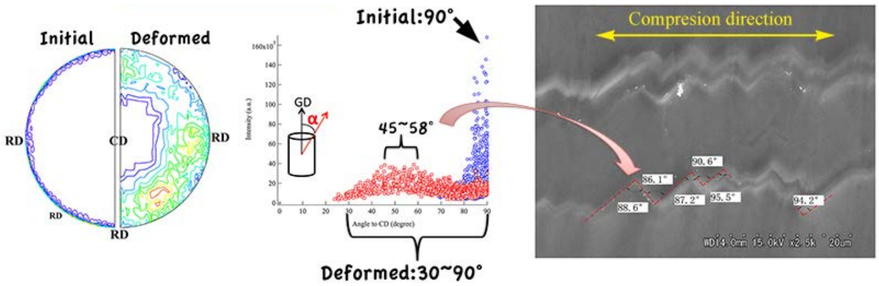

Fig. 1 Microstructural change with kink deformation for a bulk $Mg_{85}Zn_6Y_9$ specimen prepared by 1-direction solidification technique.

１．研究成果概要（つづき） Outline of experimental results (continued).

(2) Superconductor materials

Texture was measured under tension at an elevated temperature for a (Y, Gd)Ba2Cu3Oy tape wire was examined. This material has a strong texture; the c–axis was mostly normal to the substrate and a– and b–axes were oriented in both normal and longitudinal directions. It was confirmed by in situ neutron diffraction that the domains moved so as to make the b–axis became parallel to the longitudinal direction under high temperature tension. As results, two diffraction peaks for (200) and (020) merged to one peak as was presented in Fig. 2. The relationship between this finding and phase transition with oxygen depletion at high temperatures is now under investigation.

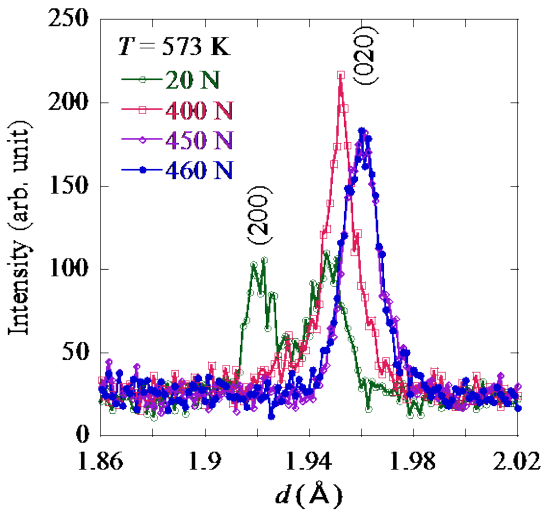

Fig. 2 Diffraction profiles for (Y,Gd)Ba2Cu3Oy high temperature superconductor tape along the longitudinal direction.
It is found that (200) diffraction peak (lower lattice spacing) disappear in (200)/(020) profile under tensile stress. It indicates that the texture changes by loading of 400N.

(3) Advanced steels

In this fiscal year, the 6 subjects were studied; (a) introduction of TMWP fitting for profile analysis that was common need of this project, (b) dynamic ferrite transformation, (c) tempering behavior of high Cr high C martensitic steel, (d) strain aging, (e)deformation mechanism of two–phase steel and (f) texture measurement (methodology) . The obtained results include the followings.

(a) Introduction of profile analysis technique to Takumi diffractometer

The instrumental profiles were measured using LaB_6 etc., with help by two experts on profile analyses. Then, the TMWP fittings were carried out for ferritic steels and martensite–retained austenite steel. An example of the fitting was presented in Fig. 3. This must become a powerful tool to evaluate dislocation density and dislocation structure for various kinds of plastically deformed materials. It seems to be possible to expand the application to multi–phase materials.

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1．研究成果概要（つづき） Outline of experimental results (continued).

(b) Dynamic ferrite transformation

In situ neutron diffraction during high temperature deformation was carried out to study TMCP and the details were described in the next page.

(c) Tempering behavior of high Cr high C martensitic steel

In situ neutron diffraction during heating was performed to make clear the change of retained austenite with tempering. Martensitic transformation during cooling from 950℃ was also tracked. The increase (or little change) of the retained austenite during tempering is a new finding.

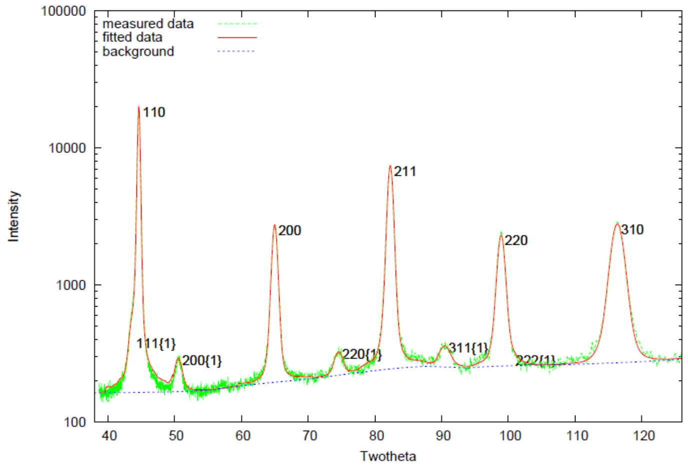

Fig. 3 TMWP fitting for 1.6Mn-0.9Si-0.2C martensitic steel containg the retained austenite.

(d) Strain aging

The dependence of deformation path of strain aging behavior was investigated by tension-compression with aging (heating at 150℃) were studied by in situ neutron diffraction. Here, the changes in intergrnular stresses were focused to clarify the influence of deformation history on strain aging.

(e) Deformation mechanism of two-phase steel

Ferritic steels containing Cu or VC particles were subjected to tensile deformation with in situ neutron diffraction and the effects of the second particles on work-hardening were studied. It was also studied by small angle neutron diffraction at BL15 (Taikan).

(f) Texture measurement (methodology development)

As a part of round robin test by Japan Light Metals Soc. , the standard Al and Cu alloys subjected to rolling deformation were employed for texture measurements at Takumi and iMATERIA. Although it takes longer time to obtain the data for ODF compared with iMATERIA, the quality of the data is better at TAKUMI. The obtained results will be compared with those by X-ray diffraction and EBSD analysis.

As a new interesting topic, the details of high temperature deformation, (b) will be described below.

1．研究成果概要（つづき） Outline of experimental results (continued).

< Dynamic transformation at elevated temperatures for Fe-6Ni-0.2C>

Dynamic ferrite transformation is a key technology to obtain fine grains by TMCP for steels. It has been known the transformation is accelerated with plastic deformation. A debating point is whether dynamic ferrite transformation takes place at a temperature above Ae3 (austenite stable region in the viewpoint of thermodynamic). To examine this issue, the specimen was cooled rapidly interruptedly during hot deformation and the microstructure was observed with microscopy at RT. It has been suspected that the intrusion of ferrite transformation during cooling. The austenite changes to martensite on cooling and hence the real situation at a high temperature cannot be confirmed. Hence in situ X-ray diffraction during TMCP was performed and reported ferrite transformation seemed to occur above Ae3. However, the observations of surface layer accompanies uncertainty such as decarburization. Thus, in situ neutron diffraction is expected to reveal the truth directly.

The steel used was 6Ni-0.1C (mass%). The Te3 temperature of this steel calculated by the software "Thermo-calc" was 728 ° C. As illustrated in Fig. 4(a), a specimen was heated up to 800 ° C for 3min (austenitization) and then cooled to 750 ˜ 700 ° C, where the compression of 40 % was given with a strain rate of 10^{-1} s^{-1} after isothermal holding for 3.6 ˜ 7.2 ks (in order to make temperature uniform in the specimen), followed by cooling to RT.

Fig. 4(b) - (d) show the results of in situ neutron diffraction at 700 ° C, (b) before compression (austenite single phase), (c) during deformation, and (d) after deformation (4.9ks). Ferrite peaks was confirmed during and after deformation indicating the occurrence of dynamic ferrite transformation. In case of deformation at 730 ˜ 750 ° C (above Ae3), no transformation was detected. Because this phenomenon depends on strain and strain rate (not only temperature), further examination must be needed to reach the true answer. This must be a powerful method which cannot be done by any other methods for TMCP development.

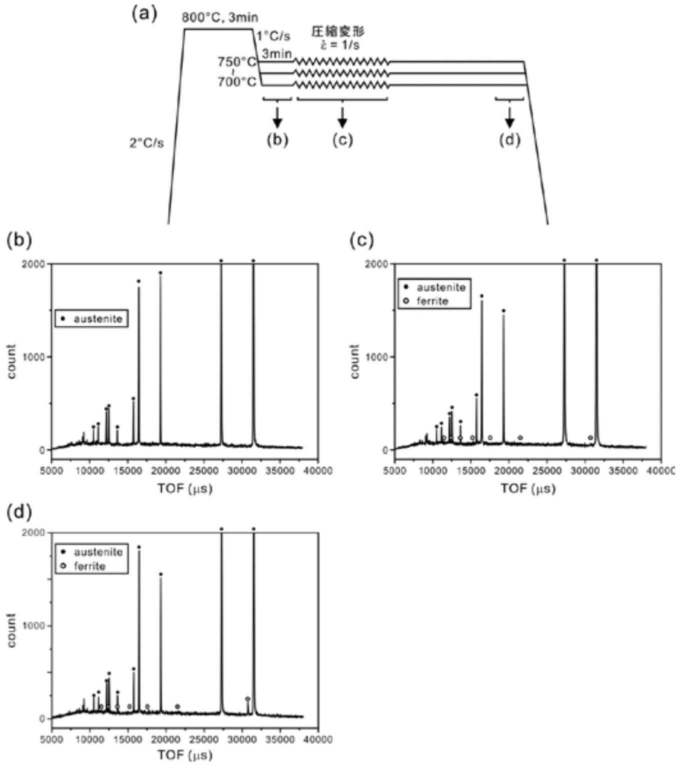

Fig. 4(a): Schematic illustration of thermo-mechanical process, (b)-(d): neutron diffraction profiles (b) before compression at 700 ° C, (c) during compression at 700 ° C, and (d) after subsequent holding at 700 ° C for 4.9 ks after compression.