実験報告書様式(一般利用課題·成果公開利用)

(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

| MLF Experimental Report | 提出日 Date of Report |
|--|-------------------------------------|
| 課題番号 Project No. | 装置責任者 Name of responsible person |
| 2015A0067 | Harjo Stefanus |
| 実験課題名 Title of experiment | 装置名 Name of Instrument/(BL No.) |
| In-situ study on the interplay between martensite formation, | TAKUMI (BL19) |
| texture and dislocation evolution of Co-Cr alloys | 実施日 Date of Experiment |
| 実験責任者名 Name of principal investigator | From 8, June. 2015 to 12, June 2015 |
| Shigeo Sato | |
| 所属 Affiliation | |
| Ibaraki University | |

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. | | |
|--|---|--|
| Tensile loading specimens of a Co-29Cr-6Mo-0.14N alloy were | 1200 | |
| prepared for the neutron diffraction measurements. The | 0.001 s ⁻¹ | |
| dimension of the gauge section of dog-bone type specimens was | 800 973 K | |
| 6 mm in diameter and 10 mm in length. The 0.2% proof stress | 1073 K | |
| and elongation of this specimen are about 500 MPa and 0.4, | <u>200</u> 1173 К | |
| respectively, at room temperature. The stress-strain curves | 0 0.5 1 | |
| obtained by the high-temperature compressive tests at a strain | True strain Fig. 1 True stress-strain curves of the Co alloy | |
| rate of 0.001 s ⁻¹ are shown in Fig. 1. | specimens. | |

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. **[Experimental method]** Neutron diffraction measurements were carried out at TAKUMI (BL19) beamline in MLF/J-PARC. The loading machine equipping with an infrared heating furnace was mounted on the sample positioning table. The load axis was aligned horizontally at 45 degree to the incident beam, so that simultaneous measurements of lattice strains in directions both parallel and perpendicular to the applied load. Because of the low scattering length density and high absorption coefficient of the Co alloy, tensile loading test for the line-profile analysis was performed at a small strain rate of 1.67×10^{-5} s⁻¹ for the room-temperature deformation, and was unloaded at every additional 5% strain. The Bauschinger effect was also investigated with the stress amplitude of 637 MPa. The phase transformation and texture evolution induced by high-temperatures deformation, whose strain rate was 0.001 s⁻¹, was observed at 973, 1073, and 1173 K. The instrumental profiles for the BCC and FCC steels were taken from the diffraction peaks of the annealed high-purity iron and SUS316L plates, respectively. The line-profile analysis was carried out with the convolutional multiple whole profile (CMWP) program.

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

[Results] Ultrafine grains with grain sizes smaller than 1 µm can be obtained by high-temperature deformation. To understand the mechanism of this phenomenon, microstructural evolution induced by high-temperature deformation was investigated by using neutron diffraction.

Figure 2 shows a tensile stress curve as a function of loading time and a variation in diffraction histogram with the tensile loading time at 1173 K. Only the face-centered cubic (fcc) phase was observed, and the diffraction peaks clearly broadened, indicating that crystallite refinement and an accumulation of dislocations proceeded with the tesile-loading regardless of the high temperature of 1173 K. The progress of dislocation accumulation may originate from the partial dislocations due to the low stacking fault energy of this alloy at this temperature.

Figures 3 and 4 show tensile-stress curves as a function of loading time and variations in diffraction histogram with the tensile loading time at 1073 and 973 K, respectively. Diffraction profiles of the fcc phase at 1073 and 973 K broadened with the loading time in similar manner to those at 1173 K, indicating microstructural refinement of the fcc phase. The martensitic phase with the hcp structure appeared at 1073 and 973 K, as denoted by arrows in diffraction histograms, suggesting that the strain-induced martensitic Fig. 3 (a) Stress as a function of loading time and transformation was prompted by the further decrease in the (b) a variation in diffraction histogram at 1073 K. stacking fault energy with the decrease in the temperatures. It should be noted that the peak position of the 200 reflection of the fcc phase at around 5.5 nm⁻¹ unchanged in the diffraction histogram at 973 K whereas the peak positions shifted to lower 1/d values, meaning the expansion of lattice, at 1073 and 1173 K with the loading time. This phenomenon may be related to the appearance of the serration in Fig. 4(a). The serration, which can be confirmed in Fig. 4(a), would be probably caused by the diffusion of nitrogen atoms, which can be trapped at stacking faults by the Suzuki effect. Consequently, the shrink and expansion of the fcc lattice occurred simultaneously.

Further analyses are to be done by using CMWP.



Fig. 2 (a) Stress as a function of loading time and (b) a variation in diffraction histogram at 1173 K.







Fig. 4 (a) Stress as a function of loading time and (b) a variation in diffraction histogram at 973 K.