MLF Experimental Report

提出日 Date of Report August 7, 2012	
課題番号 Project No. 2012A0094 実験課題名 Title of experiment In situ neutron diffraction study for Fe-based shape memory materials 実験責任者名 Name of principal investigator Hiroyuki Y. Yasuda 所属 Affiliation Osaka University	装置責任者 Name of responsible person Kazuya Aizawa, Stefanus Harjo 装置名 Name of Instrument/（BL No.） TAKUMI/ (BL19) 実施日 Date of Experiment June 28-30, 2012

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.

(A) Fe-Al single crystals

Fe-20at%Al, Fe-23at%Al, Fe-28at%Al (tensile axis, <149> orientation)

(B) Fe-Ga single crystals and polycrystals subjected to different heat treatments

Fe-23at%Ga, Fe-24at%Ga, Fe-25at%Ga

(C) Fe-Al-Ga polycrystals

Fe-20at%Ga-5at%Al

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.

In situ neutron diffraction study was carried out using TAKUMI diffractometer (BL19) to examine pseudoelasticity in binary Fe-Al, Fe-Ga and ternary Fe-Al-Ga alloys with the $D0_3$ structure.

(A) in situ neutron diffraction study for Fe-Al single crystals

Fe-Al single crystals containing 20~28Al (at%) were tensile deformed and in situ study was performed using TAKUMI diffractometer. Fig. 1 shows a typical stress-strain (S-S) curve of Fe-23Al single crystals pulled to a plastic strain (ε_p) of 7% with <149> orientation at room temperature. During loading, the flow stress remains almost constant up to ε_p = 7% after yielding. On the other hand, the applied strain is completely recovered during unloading resulting in large pseudoelasticity. During loading and unloading, neutron diffraction study was carried out for the deforming samples. The cross-head of the tensile machine on the sample stage was stopped during the neutron diffraction measurement. The measuring time at each step was about 15 min. Stress drop in the S-S curve in Fig. 1 corresponds to the measurement.

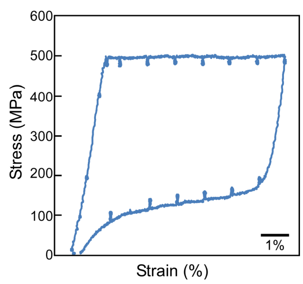

Fig. 1 A S-S curve for a Fe-23Al single crystal.

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

Fig. 2 shows neutron diffraction profiles for Fe-23Al single crystal. During loading, each peak shift to higher flight time corresponding to an increase in lattice spacing due to tensile loading. Moreover, the peak becomes broad due to an introduction of plastic strain, especially at ε_p = 7%. In contrast, during unloading, the peaks shift to the starting position and are sharpened again. The pseudoelasticity of Fe-23Al single crystals is considered to be based on to-and-fro motion of dislocations dragging anti-phase boundaries (APB). The shift and broadening of the diffraction peaks are in good agreement with the dislocation-based pseudoelasticity. It is also interesting to note that the shape of the diffraction peaks in TAKUMI detectors varied with strain as shown in Fig. 3. In the figure, 420 peak moves forth and back during loading and unloading, which corresponds to the crystal rotation due to slip deformation. This is also consistent with the model for the pseudoelasticity. In contrast to Fe-23Al single crystals, Fe-20Al and Fe-28Al crystals never showed pseudoelasticity; peak broadening was not recovered even after unloading.

(B) in situ neutron diffraction study for Fe-Ga polycrystals

Some Fe-Ga polycrystals showed pseudoelasticity based on a martensitic transformation as shown in Fig. 4. In the diffraction profiles of Fe-23, 24Ga polycrystals solutionized at 800 °C, some peaks which may correspond to the martensitic transformation appear during loading. The structural analysis of the martensites is being undertaken, On the other hand, no such peak can be observed in solutionized Fe-25Ga polycrystals. This means that the stress-induced transformation leading to the pseudoelasticity takes place at a limited Ga concentration. It is also noted that the martensitic transformation never occurred in Fe-Ga polycrystals annealed at 600 °C for $D0_3$ ordering, suggesting that the transformation depends strongly on the degree of order.

(C) in situ neutron diffraction study for Fe-Al-Ga polycrystals

Pseudoelasticity of Fe-20Ga-5Al pseudo-binary polycrystal was also studied by in situ neutron diffraction. No peak resulting from the stress-induced martensitic transformation could be seen in the polycrystals. This suggests that the pseudo-binary alloys show pseudoelasticity based on the dislocation motion.

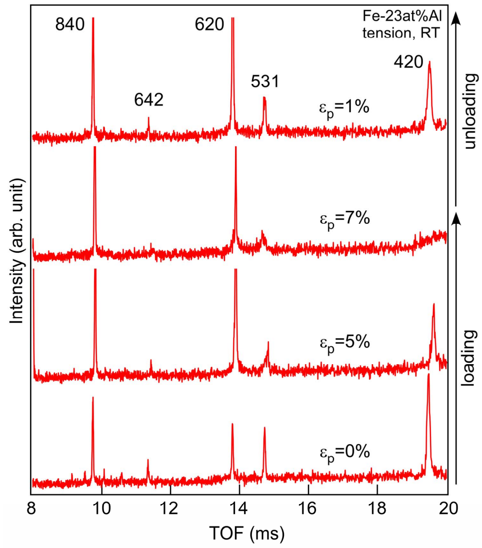

Fig. 2 Neutron diffraction profiles of the Fe-23Al single crystal.

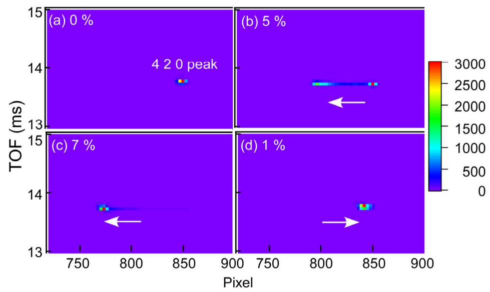

Fig. 3 Shift of 420 peak in the Fe-23Al single crystal.

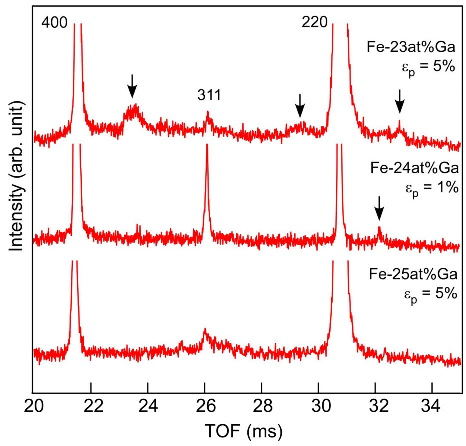

Fig. 4 Neutron diffraction profiles of solutionized Fe-23~25Ga polycrystals. Arrows indicate peaks corresponding to the phase transition during loading.