Observation of behavior of radiation defect under deformation in ion-irradiated material

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1. Introduction

In order to understand material degradation of main structural material for nuclear reactor vessel and internals in nuclear reactor, we are investigating characterization of radiation induced defects and their behavior under deformation. To have a better knowledge of radiation induced hardening in highly neutron irradiated material, it stands in need of a TEM analysis method for direct observation of micro-structural defects under deformation. In this present work, this analysis method is described herein, from preparation of experiment specimen to analysis method, and a representative result of the analysis is presented.

2. Methods and results

2.1 Ion-irradiated material

The material used in this study was Fe-Cr model alloy having the chemical composition given in Table 1.

Table 1 Chemical composition (wt%) of material used in the present study

Cr	O	C	N	Fe
14.9	0.040	0.012	0.0003	balance

Disk specimens, 3mm in diameter, were punched out from a 100 ㎛ thick sheet. The specimens were mechanical-polished for ion irradiation experiment. Final polishing was carried out with a vibratory polisher using very fine Al_2O_3 suspension for the reduction of surface damage formed during the rough polishing.

Irradiations were performed using 8MeV Fe^{4+} up to a peak does of 5dpa. Irradiation temperature was 400℃. Matrix damage for depth from surface was calculated with SRIM [1] assuming 40 eV of average displacement threshold energy [2]. According to SRIM calculation, peak damage was expected to be formed at 1.7 ㎛ due to the Fe ion irradiation.

2.2 Preparation of TEM sample for in-situ experiment

For in-situ straining TEM examination, a special procedure of sample preparation was applied. In general, a TEM specimen for in-situ straining under TEM observation used to be thin bulk sheet with 1mm wide, 3mm long and 0.1mm thick [3]. However the Fe-Cr alloys used for this work have typical ferromagnetic effect in electromagnetic circumstance. The ferromagnetic effect can make it more difficult to observe very fine micro structural defects. Therefore, we had to fabricate TEM samples with very small volume because of positive tendency of ferromagnetic effect with volume. The best method for fabrication of TEM specimen with small volume is to use of focused ion beam system (FIB). Fig.1 shows procedure for fabrication of in-situ straining TEM sample. A grid with rectangular hole shown in fig. 1 was prepared using FIB for in-situ straining experiment. The grid must have a very thin wedge. A lamellae lifted off in fig 1 was welded on the hole in the grid. After welding on the grid, the TEM specimen must be clean carefully with low energy Ar ion miller for reduction of Ga ion damage formed during fine milling. The low energy ion milling with Gentle mill, which can operate at 200eV, was carried out after FIB milling.

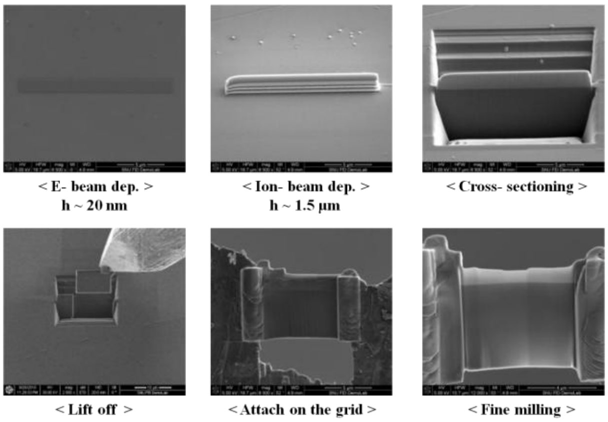

Fig.1 Preparation of TEM sample for in-situ straining experiment

2.3 in-situ TEM examination

Microstructures of the prepared Fe-Cr alloys were observed by a TEM (Jeol 2100F), operated at 200keV. We used a Gatan 641 in-situ straining holder for observation of deformation behavior of ion-irradiated specimen.

Fig. 2 presents microstructures of an in-situ sample during in-situ straining experiment. After strain up to

5%, cracking and failure of the TEM sample were observed as shown in fig. 2. The crack was introduced from edge of the sample. In addition, it was found that deformation slip was generated in the matrix due to straining.

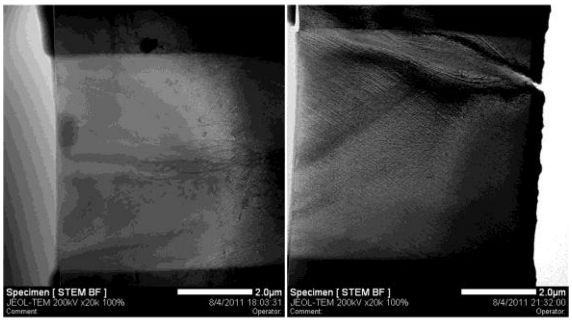

Fig.2 TEM micrographs of ion-irradiated Fe-Cr alloy before (left) and after (right) straining experiment

Fig. 3 shows examples regarding the interaction between microstructural defect and moving dislocation during straining in TEM. In general, slip system in iron has been known to be <111>{110}{112}{123}. Most of dislocations passed away on slip planes, which made slip traces. It was difficult to observe exact interaction behavior between a dislocation loop and a moving dislocation because of narrow gap between slip planes in this zone axis and fast movement of the moving dislocation. In fig 3, we can observe a lot of black dot contrasts, which were expected to be nanometer sized dislocation loops formed by the ion irradiation. Unfortunately, the Burgers vector of the dislocation loop was not able to be determined exactly because of limitation of titling analysis on the in-situ TEM experiment. However, we clearly observed pinning phenomena of moving dislocation at radiation induced defects.

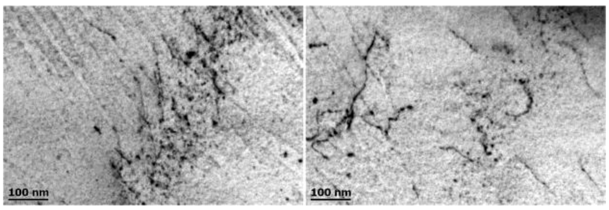

Fig.3 TEM micrographs showing pinning of moving dislocation at radiation defects in ion-irradiated Fe-Cr alloy during in-situ straining.

4. Conclusion

Method for direct observation of micro-structural defects under deformation was developed using FIB and in-situ straining TEM. Experimental sample with small volume was prepared with FIB and low energy ion miller. We observed interactions between micro-structural defects formed by ion irradiation and moving dislocation generated by in-situ straining.

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