TEM analysis of Irradiation induced dislocation loop in ion irradiated Fe-Cr alloys

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1. Introduction

The Fe-Cr binary alloys are a base for low activated ferritic-martensitic steels which are now considered as one of the candidate structural materials for a fusion reactor steel because of its excellent resistance to a material degradation such as a swelling, helium embrittlement and irradiation creep [1]. But it has severe problems regarding an irradiation-induced embrittlement at a low temperature and an irradiation hardening with an increasing Cr content [2]. Extensive irradiation experiments by a neutron, a proton and an ion have shown that microstrucutral defects with nano scale features such as dislocation loops with Burgers vector (b), voids and precipitates formed in a matrix have a significant influence on a material degradation [3]. However, the effect of the chromium element on the irradiation hardening behavior of Fe-Cr alloys has not been elucidated clearly because of the resultant confusing and mixed microstrucutral defects under an irradiation condition. So it is one of the essential issues to analyze an exact evolution of a dislocation loop which is considered as a major factor affecting the hardening behavior of Fe-Cr alloy at a low irradiation temperature. In this paper, we aim to reveal the evolution of dislocation loops in ion irradiated Fe-Cr alloys through a transmission electron microscopy (TEM) examination.

2. Experimental

Fe-Xwt%Cr(X=5 and 15) model alloys used in this investigation were prepared by a vacuum induction melting using electrolytic metals. After the ingots were hot rolled and cold-rolled down to a thickness of 1mm, they were heat treated at a rate of 5K/s to a recrystallization temperature of 873K~973K for 3hr~5hr and then water quenched. Atomic displacement damage was introduced by 8MeV Fe^{4+} ions accelerated with an ion accelerator in the Korea Institute of Geoscience & Mineral Resources (KIGAM). The beam current was around 200nA. Specimens before an irradiation were polished electro-chemically for a removal of any damage formed on their surface and then irradiated at room temperature. The specimens were irradiated up to the fluence in the order of 6.16×10^{15} ion/cm^2 for the Fe-5Cr alloy and 2.8×10^{15} ion/cm^2 for the Fe-15Cr alloy, respectively.

3. Result and discussion

3.1 Nano indentation measurement

A nano indenter(MTS Nano Indenter™ XP) with a continuous stiffness measurement method (CSM) was used for a mechanical property measurement of the nanometer-thick layers of which the mechanical properties change with an indentation depth [4]. The Berkovich indenter is a three-sided triangular-based pyramidal diamond. Each successive indent was displaced by more than 10μm to avoid an overlapping of a plastic deformation zone onto other indents. When a nano indentation is progressed continuously, the nano hardness of the irradiated Fe-5Cr alloy is increased remarkably due to its irradiated damage layer. Nano hardness changes in Fe-Cr alloys determined by subtracting the maximum hardness of the irradiated and the unirradiated Fe-Cr alloys, are plotted as Figure 1. It is found that the nano hardness increase in the Fe-15Cr alloy is considerably larger than that in the Fe-5Cr alloy under ion irradiation at room temperature.

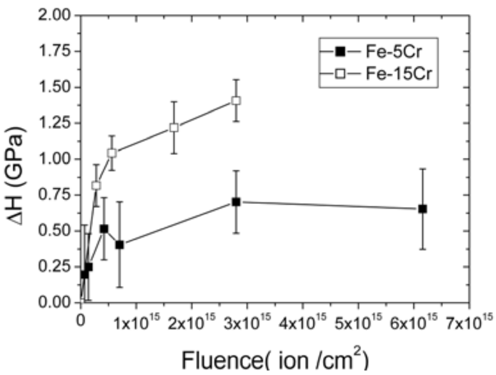

Figure 1 A change in the nano hardness of the irradiated Fe-5Cr alloy and the Fe-15Cr alloy with an irradiation fluence level.

3.2 TEM analysis

TEM examinations were carried out to observe the microstrucutral change of the Fe-Cr alloys irradiated at a maximum fluence level. Thin foils for the TEM observation were prepared with a focused ion beam micro-processing device (FIB) and a low energy ion milling.

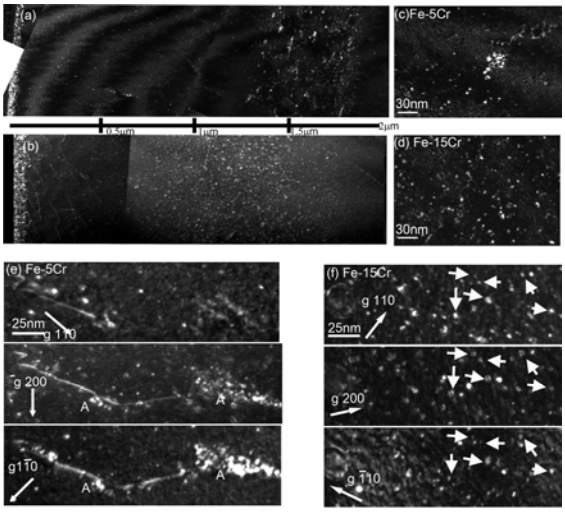

Fig.2 Cross-sectional view TEM micrographs of the irradiated Fe-5Cr alloy (a) and Fe-15Cr alloy (b), the evolution of irradiation induced dislocation loops in the Fe-5Cr alloy(c) and the Fe-15Cr alloy (d) at a depth of 1μm and weak beam dark-field micrographs of the irradiation induced dislocation loops with the reflections of g=110, g=200 and g=$\bar{1}10$ in the Fe-5Cr alloy (e) and the Fe-15Cr alloy(f)

Figure 2(a) and 2(b) present the cross sectional views of the Fe-5Cr alloy irradiated at a dose of 6.16×10^{15} ion/cm^2 and the Fe-15Cr alloy irradiated at a dose of 2.8×10^{15} ion/cm^2 respectively. We observed small white contrasts of a high density between 1μm and 2μm from the surface in both the irradiated specimens. The white contrasts were exhibited with a reflection (**g**) in weak beam dark field images if **g·b** = 1 or 2. They seem to be irradiation induced dislocation loops which have Burgers vectors of a_0<100> or 1/2 a_0<111> components. Figure 2(c) and 2(d) show that an agglomeration of the nanometer-sized dislocation loops was formed locally in the Fe-5Cr alloy and nanometer-sized dislocation loops were distributed uniformly in the Fe-15Cr alloy. The average size and the number density of the dislocation loops were measured to be approximately 2.5nm and 7.12×10^{22} /cm^3 in the Fe-5Cr alloy and 3.5nm and 5.54×10^{22} /cm^3 in the Fe-15Cr alloy respectively.

A Burgers vectors analysis by imaging the same defect with different diffraction vectors and observing the contrast was performed for a characterization of the dislocation loops. Figures 2(e) and 2(f) show weak beam TEM micrographs of the ion irradiated Fe-5Cr alloy and Fe-15Cr alloy for the same observation area under a series of reflections. White spot contrasts in the Fe-5Cr alloy were mainly observed with all the reflections whereas many coffee bean contrasts with a larger size were observed by the reflection of g=200 in the Fe-15Cr alloy. In the Fe-5Cr alloy, the dislocation loops marked A in Figure 2(e) were formed in the vicinity of the dislocation line. They are visible with the reflections of g=$\bar{1}10$ and g=200 and are invisible with another reflection of g=110. In the case of the Fe-15Cr alloy, the dislocation loops marked as arrows in Figure 2(f) exhibited a coffee bean contrast with the reflections of g=200 and were visible with the reflections of g=$\bar{1}10$ and g=110 as well. According to g·b extinction criterion, the white spot contrasts in the vicinity of a dislocation in the Fe-5Cr alloy are identified as 1/2 a_0<111> dislocation loops. Also, it is found that the coffee bean contrasts in the Fe-15Cr alloy are a_0<100> dislocation loops. The characterization of the coffee bean contrast is in keeping with Marian's work that a dislocation loop with a Burgers vector of a_0<100> has a coffee bean contrast at g= 200 by a TEM weak beam image simulation [5].

A population ratio of a_0<100> dislocation loops to 1/2 a_0<111>loops was measured quantitatively in Fe-Cr alloys. We assumed that the coffee bean contrast at g=200 is an a_0<100> dislocation loop and considered the visible condition of dislocation loops at g=200 for its measurement. The ratios in the Fe-5Cr alloy and Fe-15Cr alloy are measured to be about 0.9:1 and 1.9:1 respectively. So it is found that the formation of a_0<100> loops is more prevalent in the Fe-15Cr alloy than in the Fe-5Cr alloy. Moreover, the relatively large population of the 1/2a_0<111> dislocation loops in the Fe-5Cr alloy may have an effect on the agglomeration of the dislocation loops due to a high mobility of the 1/2a_0<111> dislocation loop.

4. Conclusions

Transmission electron microscopy observation were performed to investigate a microstrucutral evolution and in Fe-Cr alloys after an irradiation with 8MeV Fe^{4+} ions at room temperature. TEM observation reveals a significant population of the a_0<100> dislocation loops in the Fe-15Cr alloy and an agglomeration of the 1/2 a_0<111> dislocation loops in the Fe-5Cr alloy. The results indicate that the a_0<100> dislocation loops will act as stronger obstacles to a dislocation motion than 1/2a_0<111> dislocation loops.

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