# The radiation hardening and microstructural defect evolution in ion irradiated Fe-Cr alloys with irradiation temperature

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# 1. Introduction

Generally, neutron, ion and electron Irradiations cause a substantial amount of hardening and significantly alter the deformation behavior of metals and alloys at relatively low irradiation temperatures [1-3]. A radiation hardening is caused by the formation of microstructural defects such as dislocation loops, voids and precipitates under irradiation. Therefore, it is important to have a better knowledge of the irradiation induced microstructural defects under irradiation condition. As a part of the National mid- and long-term atomic energy R&D program, we are dealing with the radiation hardening behavior in Fe-Cr binary alloy. Fe-Cr binary alloy is a base alloy of Ferritic/Matensitic steel(F/M steel) planning to use for the Gen IV nuclear system. In this work, we investigated the radiation hardening and microstructural defect evolution in ion irradiated Fe-Cr alloys with irradiation temperature using nano-indentation measurement and transmission electron microscopy (TEM) observation.

#### 2. Experimental

Fe- Xwt% Cr(X=5, 9 and 15) model alloys were prepared by a vacuum induction melting using electrolytic metals. Specimens before an irradiation were polished electro-chemically for a removal of any damage formed on their surface. Atomic displacement damage was introduced by 8MeV Fe<sup>4+</sup> ions accelerated with an ion accelerator in the Korea Institute of Geoscience & Mineral Resources (KIGAM). The beam current was around 200nA. The specimens were irradiated at the fluence in the order of  $5.8 \times 10^{14}$  ion/cm<sup>2</sup> (1dpa at peak damage depth) up to 673K.

Nano-indentation measurement was performed on the irradiation hardening of Fe-Cr model alloy. The apparatus used in this work was MTS Nano Indenter<sup>™</sup> XP made by Nano Instruments, Inc. Depths of indents were 1.5 um. Each successive indent was displaced by more than 10 µm in order to avoid overlapping of plastic deformation zone onto another indent. Analysis for the calculation of nano hardness was conducted by the method used by Oliver and Pharr [4]. Thin foils for TEM observation were prepared with a focused ion beam (FIB) micro-processing device and low energy ion miller. TEM observation were performed by using a 200keV JEOL 2000FXII and JEOL 2100F microscope. Weak beam imaging by means of TEM has been very successful in studies of the geometry of dislocation loops and more complex clusters of size larger than

about 5nm which are largely complementary to blackwhite contrast analysis. It is sensitive to local strain fields and so is often useful for analyzing clusters of complex shape or successfully in studies of defects smaller than 5nm, but in these cases the variability of the contrast with the exact diffracting conditions has to be taken into account. So the application of weak beam microscopy to clusters smaller than 5nm is less well established. However, recent systematic simulation about weak beam images makes their characterization possible [4].

#### 3. Results and discussion

#### 3.1 Radiation hardening of Fe-Cr alloys

Nano-indentation measurement was performed to investigate the hardening behavior of irradiated Fe-Cr alloy. A nano hardness change is measured by subtracting the maximum hardness of the irradiated and the un-irradiated Fe-Cr alloys. Figure 1 shows the irradiation temperature dependence of the nano hardness change in irradiated Fe-Cr alloys. Nano indentation results indicate that the change of nano hardness of Fe-Cr alloys irradiated at 300K is linearly dependent on the Cr content. As irradiation temperature increases, the changes of nano hardness in Fe-5Cr alloy and Fe-9Cr alloy are decreased rapidly. Above 573K, there is little difference between Fe-5Cr alloy and Fe-15Cr alloy as shown in Figure 1. On the other hand, the nano hardness change in Fe-15Cr alloy remains steady up to 573K and then is decreased slightly at 673K.



Figure.1. The change of nano hardness in Fe-Cr alloys with irradiation temperature.

#### 3.2 Dislocation loop structure in Fe-Cr alloys

Figure 2(a) and 2(b) show weak beam TEM micrographs of the Fe-5Cr alloy and Fe-15Cr alloy irradiated at 300K. White spot contrasts in Fe-5Cr alloy were mainly observed whereas many coffee bean contrasts with larger size were observed by the reflection of g= 200 in Fe-15Cr alloy. It is published that the coffee bean contrast is identified as a dislocation loop with Burger vector of  $a_0 < 100 >$  by TEM image simulation [5] and a Burgers vectors analysis [6]. On the other hand, we observed the coffee bean contrast in both Fe-5Cr alloy and Fe-15Cr alloy irradiated at 573K. It is thought that the formation of a dislocation loop with Burger vector of  $a_0 < 100 >$  in Fe-Cr alloy is amplified irrespective of Cr content under high temperature irradiation as shown in Figure 2(c) and 2(d). Accordingly, the radiation hardening phenomena in high temperature irradiation could not be described in terms of a nature of irradiation induced dislocation loop. There are other factors that give rise to the radiation hardening at higher Cr content. Based on the phase diagram of Fe-Cr binary alloy, it is inferred that the existence of Cr rich precipitate( $\alpha'$ ) is feasible in Fe-15Cr alloy. It seems reasonable to suppose that the Cr rich precipitate act as the strong obstacles for the dislocation movement in Fe-15Cr alloy irradiated at high temperature.

(a)Fe5Cr 11dpa 300k (b)Fe15Cr 5dpa 300K



Figure 2 Dislocation loop structures in ion irradiated Fe-5Cr and Fe-15Cr alloys

## 4. Conclusions

Nano indentation examination and TEM observation were performed to investigate the radiation hardening and microstrucutral defect evolution in Fe-Cr alloys with irradiation temperature. Under a Fe ion irradiation, the radiation hardening in Fe-Cr alloy was lowered with increasing irradiation temperature. Hardening is much higher in Fe-15Cr alloy than in Fe-5Cr and Fe-9Cr alloy. Dislocation loops with Burger vector of  $a_0 < 100 >$ were observed in both Fe-5Cr alloy and Fe-15Cr alloy irradiated at 573K. It is expected that the hardening in Fe-15Cr alloy irradiated above 573K is due to the formation of Cr rich precipitate ( $\alpha'$ ).

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