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Effect of Chromium Content on Mechanical Properties and Microstructural Evolution of Ion-Irradiated Fe-Cr Model Alloys

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1. Introduction

Fe-Cr alloy of a ferritic martensitic microstrucuture is one of the candidate structural materials for fusion reactor steels. It is well known that Fe-Cr alloys have a high resistance to a swelling and an excellent resistance to a helium embrittlement and to irradiation creep[1]. But it has a severe problem about low temperature irradiation-induced embrittlement below 673K [2]. The mechanical behavior of an irradiated material is mostly affected by the formation of irradiation induced defects called black dots, a dislocation loop and a void in the matrix. The main objective of this present study is to investigate the effect of chromium content on the irradiated hardening behavior of a polycrystalline Fe-Cr alloy. We measured a hardness change with a Nano indenter for irradiated Fe-Cr alloys and analyzed the size and fraction of the irradiation induced defects with a transmission electron microscopy.

2. Experimental procedure

2.1. Materials and heat treatment

Fe-Cr model alloys were prepared by a vacuum induction melting using electrolytic metals. We used samples of Fe-Xwt%Cr (X=5,9,12 and 15) model alloys. After the Fe-Cr model alloys were hot rolled, they were machined into cubic specimens with a size of 10mm and then they were cold-rolled down to a thickness of 1mm. The specimens were heat treated at a rate of 5 K/s to a recrystallization temperature for 3hr~5hr and then finally water quenched

2.2. Ion irradiation

An ion irradiation test was performed with a multipurpose ion accelerator in the Korea Institute of Geoscience & Mineral Resources (KIGAM). The atomic displacement damage was introduced by 8MeV Fe^{4+} ions accelerated with an ion accelerator operating at 2MeV. The prepared specimens were irradiated at about room temperature up to a level of around 12dpa. The damage rate was approximately 4.15×10^{-4} dpa/s.

2.3. Nano-indentation test

To measure the mechanical properties of the ion irradiated specimen, a nano-indentation test was carried out. In the case of the ion-irradiated specimen, as the layer of the damage is below around 2μm in depth, we can not easily obtain the exact mechanical properties. CSM (continuous stiffness measurement) method as a nanoindentor is ideal for a mechanical property measurement of nanometer-thick layers of which the microstructure and mechanical properties change with an indentation depth[3]. The load resolution of the instrument is 50 nN and the displacement resolution is less than 0.02 nm. A Berkovich indenter, a three-sided diamond pyramid with an area-to-depth function that is the same as that of a Vickers indenter, was used in all the experiments. A total of nine indents were made on each sample and the results presented are an average of these indentations. The spacing between indents was 50 μm.

2.4. TEM observation

The microstructure of the prepared Fe-Cr model alloys was observed by a transmission electron microscopy (TEM). We mainly observed the evolution of the microstructural defects under weak beam dark-field imaging conditions using diffraction vectors(g) of the <110> and <002> types. The characteristics of the microstructural defects were classified by a morphology of a weak beam dark field image[4]. And a thickness of the thin foils was determined by a convergent beam electron diffraction technique.

3. Results and discussion

3.1. Nano indentation measurement and TEM observation

Nano-indentation measurement was performed to investigate the hardening behavior of irradiated Fe-Cr alloys. Figure 1 shows a hardness increase of the Fe-Cr alloys with the level of the irradiation damage. A hardness increase of the Fe-Cr alloys roughly increases with the dose. But the hardening behavior is different with the Cr content. A value of a hardness increase above 1 dpa in Fe-9wt%Cr is almost constant with the dose, whereas in Fe-5wt%Cr it is slightly increased and in Fe-12wt%Cr and Fe-15wt%Cr it is largely increased as shown Figure 1.

A hardness increases is due to an evolution of irradiated defects. Figure 2 shows the evolution of the defects in the ion irradiated Fe-9wt%Cr alloy up to 12dpa. Ion irradiation caused the formation of defects which look like white dots and a double bean as shown in the dark field images of Figure 2. We easily measured an average size of the irradiation induced defects from an image analysis. The number density of the dislocation loops can be evaluated from the quantified analysis using a pair of g=<110> and g=<002> dark field images taking from near the {001} pole. Figure 3 shows the dose dependence of a size and a number density of the dislocation loops of the ion irradiated Fe-9wt%Cr. In this analysis, it was assumed that the irradiation defects contain only a<100> and a/2<111>.

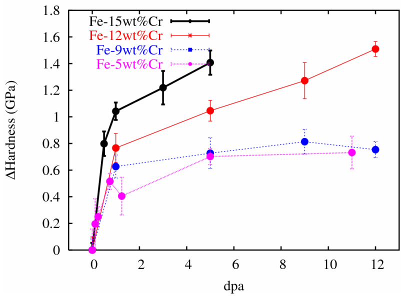

Figure 1. A hardness increase of Fe-Cr alloys with the level of irradiation damage.

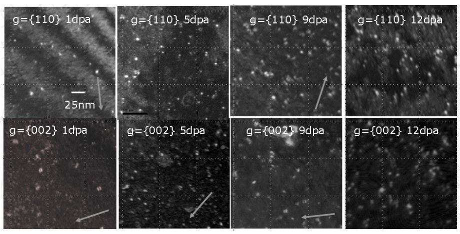

Figure 2. TEM micrographs of Fe-9wt%Cr with the level of irradiation damage.

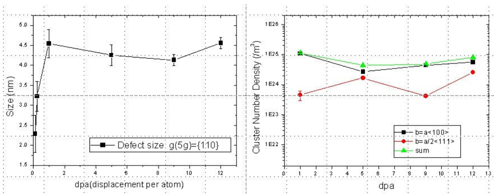

Figure 3. an average size and a number density of defects of Fe-9wt%Cr with the level of irradiation damage

An average size of the defects of the Fe-9wt%Cr alloy was measured to around 4.54, 4.25, 4.13 and 4.56nm at 1, 5, 9 and 12dpa respectively. A number density was about in the range of $4.4E^{24}$ [/m^3] and $1E^{25}$ [/m^3] as shown in Figure 3.

A character of the defects is not changed largely when compared to a value at a dose of 1 dpa. Thus we found that the magnitude of the size and density of the defects are saturated, which is similar with a hardness increase of the Fe-9wt%Cr alloy with a dose in Figure 1. In the previous section about a nano indentation measurement, we knew that adding a Cr element elevated the hardness increase which is induced by an irradiation. To investigate the effect of the Cr content on increasing of the hardness, we analyzed characters of the irradiation defects of Fe-5wt%Cr and Fe-15wt%Cr at doses of 5dpa and 11dpa respectively, which are the maximum values of the doses we performed for the ion irradiation experiment. Figure 4 presents weak beam micrographs obtained with g={110} and g={002} showing the irradiation induced damage in Fe-5wt%Cr at 11dpa and Fe-15wt%Cr at 5dpa. Generally the damage microstructure consists of small defects which look like white spots and a double bean as shown in Figure 2. But defects a like double bean are not observed in Fe-5wt%Cr at 11dpa as shown in Figure 4.

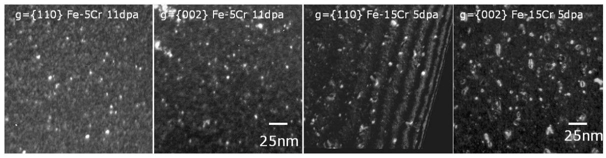

Figure 4. TEM micrographs of Fe-59Cr at 11dpa and Fe15Cr at 5dpa.

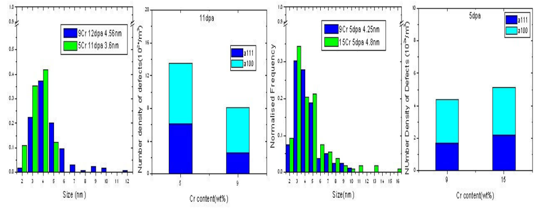

Figure 5. A size distribution and a population of defects of Fe-Cr alloys

Figure 5 shows a size distribution and a fraction of the a<100> and 1/2a<111> defects of Fe-5wt%Cr at 11dpa and Fe-15wt%Cr at 5dpa. We found that a size of a loop and a fraction of the a<100> loop is proportional to the chromium content in the range of 5wt%Cr and 15wt%Cr and the number density of the defects is the minimum at Fe-9wt%Cr and a maximum at Fe-5wt%Cr. From the analysis of the TEM and nano indentation, we can deduce that an irradiation hardening is associated with the size and the fraction of the a<100> loop due to a Cr addition as well as the number density of the irradiation defects.

4. Conclusion

Irradiation hardening in ion irradiated Fe-Cr model alloys at room temperature was investigated with a nano indentation test and TEM observation. Adding a Cr element to the Fe-Cr model alloy causes an increase of the size and the fraction of a<100> dislocation loop which can be considered as a prominent effect on an irradiation hardening at room temperature.

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