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In situ Observation of the Defect Growth Behavior in Pure Fe under HVEM Irradiation

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1. Introduction

Irradiation experiments utilizing fission reactor need long days and years for obtaining the data at only one irradiation condition. This is a great handicap in making a systematic research of radiation damage with variables such as, irradiation temperature, dose and dose rate. However, the irradiation experiments with high voltage electron microscope(HVEM) solve these difficulties. The experimental parameters can be changed very easily in HVEM experiment. Especially the microstructure change can be observed and recorded in situ. The behavior of radiation damage cluster formation in pure Fe metal has been observed to make a reference which will be used at the evaluation of Cu impurity effect in-Fe-Cu alloy.

2. Method and Results

2.1 Experimental

The starting material for this study was Fe single crystal (99.98% Fe) supplied by Goodfellow Cambridge Inc.. It was sliced into 1 mm thickness plates. After a mechanical polish to ~70 μm, the plates were punched into 3 mm diameter discs. Thin areas were obtained by electropolishing in Struers twin-jet electropolisher using a solution of 5% perchloric acid and 95% methanol below 30 °C with an applied potential of 20 V. High energy electron irradiation and in situ observation was performed in a high voltage electron microscope operated at an acceleration voltage of 1,250 kV at room temperature in Korea Basic Science Institute. The electron beam flux was set at 2.6 x $10^{23}/m^2s$ which corresponds to a displacement per atom (dpa) rate of 4 x 10^{-4} dpa/s. For in situ TEM observation, bright-field image condition was used. The reflection adopted was g=110 with the deviation parameter, s of 0.03 nm^{-1}. The image of clusters formation was captured at a time interval of about 2 minutes. And the motion of clusters was recorded through CCD camera with a time interval of 1/30 s. The foil thickness was measured from the energy loss spectrum.

2.2 Defect Formation and Microstructure Evolution

Clusters were appeared after about 5 minutes. As the irradiation time increased, the density of clusters increased and the size of clusters grew. Fig. 1 shows a typical microstructure evolution in iron. It has been known that the defect clusters under electron irradiation condition were of interstitial type at room temperature [1].

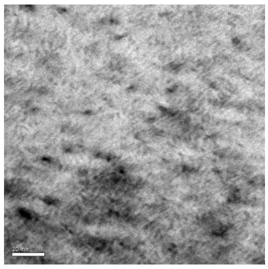

Fig.1.Microstructure of Fe irradiated with an electron fluence of 5.5 x 1025 e-/m2.

As the irradiation time increases, the focus of images became worse. Because the magnetization of specimen affected the focus and the shift of images, continuous focusing was needed.

2.3 Dependence of Cluster Density on Dose

Fig. 2 shows the cluster density as a function of electron irradiation fluence. The density increased with the electron dose almost linearly. This means the formation of interstitial defects build up continuously without any reaction with sinks. The cluster density was well consistent with the data of Arakawa's[2] under similar irradiation condition. When this result is compared with the data of Fe-1.0%Cu alloy[3], the cluster density of pure Fe is about 3 order less than that of Fe-Cu under the similar irradiation condition. This phenomenon can be explained as the effect of Cu atoms which trap the vacancies.

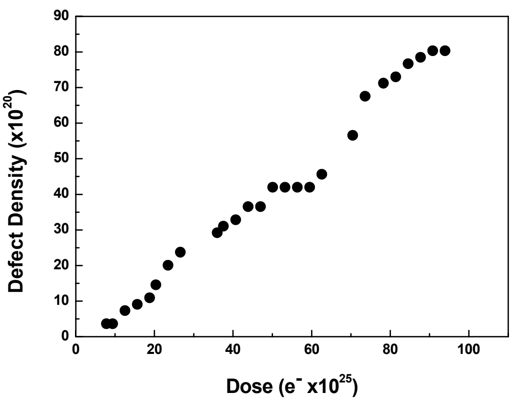

Fig. 2. Dose dependence of cluster number density in Fe irradiated with electrons.

2.4 One Dimensional Motion of Clusters

One dimensional motion of clusters could be observed at the CDD camera movie display. The clusters moved into the directions with the angle of 60° . From the diffraction pattern and the beam direction, the direction of motion could be analyzes as [111]. Generally the interstitial type loops are known to be sessile. But this result shows the clusters can be moved without any exterior stress. The dominant factor responsible for the driving force of motion is considered to be stress interaction between clusters themselves 4].
The radiation embrittlement such as sudden yield drop and initial plastic localization has been explained with the cascade induced source hardening (CISH) model [5]. Especially the dislocation channeling phenomena were explained with the dislocation glide under the Cottrell atmosphere of defects. However, such phenomena has not been proved clearly with experimental results until now. Our experimental results can solve the plastic localization with one dimensional motion of clusters. If the dislocations glide into the direction of one dimensional motion of clusters, [111] Burgers vector direction, the clusters can be moved with the dislocations.

2.5 Difference between Black Dot and Loop

When the size of clusters reached about 10 nm, the black image converted into round shape of loop suddenly as shown in Fig. 3. At the boundary of loops, a little of black shape remained after the formation of loop. This means the interior structure of loop is different from that of cluster. Generally the black dot has been explained as a small loop. Even though the additional structure analysis is needed, the difference is evident in our photograph.

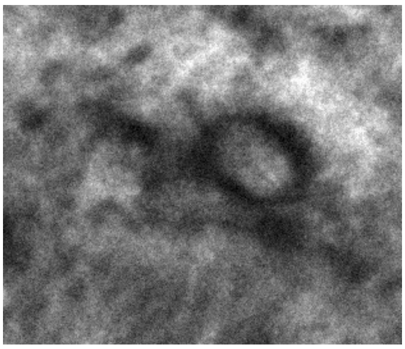

Fig. 3. The sudden change of cluster into dislocation loop.

3. Conclusion

The microstructural evolution and point defect behavior during irradiation in pure Fe have been studied by using HVEM.

1. The number density of clusters increases linearly with the increase of dose.
2. The clusters showed one dimensional motion into the [111] direction.
3. The black dots changed into dislocation loops suddenly when the size reached about 10 nm.

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