Journal of Nuclear Materials 386–388 (2009) 441–444

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Dislocation loops in Eurofer and a Fe–Cr alloy irradiated by ions at 350 and 550 °C at 3 dpa: Effect of dose rate

L. Boulanger *, Y. Serruys

Commissariat à l'Energie Atomique, DEN/DANS/DMN, Service de Recherches de Métallurgie Physique, Centre d'Etudes de Saclay, F91191 Gif- sur-Yvette, France

ABSTRACT

The purpose is to investigate the secondary defects distribution to test the influence of the irradiation mode on the microstructure in Eurofer97 and a model alloy (Fe–9 wt% Cr). Two modes are experimented at the same damage (3 dpa) at 350 and 550 °C. The first is a long-time continuous irradiation and the second is a short-time irradiation. The materials studied are irradiated by krypton ions. The first step consists in qualitative characterization of the secondary defects. Post irradiation in situ annealing experiments show that accumulated defects condense at temperature higher than 550 °C in Eurofer. In the investigated conditions, the irradiation mode does not induce a major difference: the morphologies, sizes and densities of loops are quite similar.

1. Introduction

The simulation of neutron irradiation is realized for the best by charged particles that create large collision cascades and makes available damages up to several dpa in less than an hour. The validity of such experiments requires knowledge of how the flux increase modifies the irradiation microstructure at a given fluence. The objective of this work is to observe the loops distribution of Eurofer and a model ferritic alloy when irradiated at 3 dpa under two flux conditions (Table 1). The first is a short time (18 min) and a second a long-time irradiation (6 h) corresponding to a factor 20 on the flux level. Two temperatures have been investigated: 350 and 550 °C.

In the following, we present first the morphologies of the dislocation loops formed by irradiation. In a case where no defect was present, a post-irradiation annealing has been performed to test the condensation of eventually accumulated defects.

2. Experimental method

The materials studied are Eurofer97 (Fe–9Cr–1W–0.4Mn–0.2V–0.15Ta–0.11C) and a model alloy (Fe–9 wt%Cr) irradiated as thin foils for electron microscopy. The Eurofer presents a martensitic structure with a dislocation network in the laths. The model alloy shows 50 μm equiaxed grains free of lattice defects.

The irradiations are performed in the Van de Graaff accelerator of the SRMP with 700 keV Kr^{2+} ions, at 350 and 550 °C at fluencies given in Table 1. The vacuum in the specimen chamber is lower than 10^{-7} Torr. With such setup used for several studies no contamination has been reported. The samples are thin foils (thickness lower than 200 nm) prepared by electro-polishing before irradiation using a mixture of perchloric acid (10%), butoxyethanol (20%) and ethanol (70%). The expected level damage was 3 dpa but we obtained a larger fluence after the long-time irradiations leading to conclusions to be confirmed in further experiments. The penetration range is about 200 nm and the ions mainly stay in the foil. The surface effect is considered as limited from the observation of reduced loop free zones.

The annealing has been performed inside the transmission electron microscope by use of a GATAN double tilt holder. After heating at 20 °C/min, we realized isochronal steps that consist in heating at various temperatures during 30 min and then come back to room temperature to make a fine observation of the eventual evolution.

3. Experimental results

3.1. Nature of loops

3.1.1. Irradiation at 350 °C

After the short-time mode, in Eurofer, the lath microstructure and the dislocation network does not show evolution after the irradiation. Between the lines, despite a fine investigation, no clusters have been detected. Conversely the model alloy show a homogeneous distribution of small loops visible as black dots.

After the long-time mode, inside the lath microstructure of Eurofer, dislocation loops are observed as large defects (max 22 nm) and black dots. Due to the bending of the foils, the observation is limited to a small region close to the extinction fringes and the better images are given by bright field conditions (Fig. 1). A

---

* Corresponding author.
E-mail address: loic.boulanger@cea.fr (L. Boulanger).

0022-3115/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.12.150

Table 1
Irradiation conditions.

Long time: 6 h, low flux		Short time: 0.3 h, high flux	
Damage rate (dpa/s)	Flux (ions/cm^2 s)	Damage rate (dpa/s)	Flux (ions/cm^2 s)
1.4×10^{-4}	4.46×10^{10}	2.8×10^{-3}	8.9×10^{11}

histogram of the sizes distribution has been obtained, which shows two populations of loops (Fig. 2). One peaks around 7 nm and the second around 19 nm.

In the model alloy, a similar microstructure is present. Again, loops are present with a bi-modal distribution but the density is larger with a factor about 1.5. The high density of loops impedes a safe loop size histogram. Some loops are very large (>30 nm) and some dislocation lines are present.

3.1.2. Irradiation at 550 °C

After the short-time mode at 550 °C, in the Eurofer, the observation inside laths becomes difficult because the total number of dislocation lines has increased. This comes from two origins. First, it seems that the density of the dislocation network has increased (likely by climb), secondly, large clusters located in {1 0 0} plane are present (Fig. 3) as large dislocation loops (>80 nm) with ⟨1 0 0⟩ burger's vectors.

In the model alloy, the initial microstructure makes it easy to detect the loops that are homogeneously distributed. They are similar to the one present in the Eurofer (Fig. 4). The shape of the loops can be clearly observed and reveals indentations along the line limiting the loop (Fig. 5) showing a flower-like shape. Some smaller loops with straight dislocation lines are visible close to the foil edge.

After the long-time mode at this same temperature, large defects located in {1 0 0} are present as dislocation loops (>80 nm).

The model alloy shows less deformation of the grains, which allows the realization of better pictures than in the Eurofer. It presents a homogeneous distribution of loops with a density of varying about 1–2 × 10^{20} m^{-3}. The high density limits observations of other defects.

These loops are shown for the best when they are edge on. They are mainly located in {1 0 0} planes but some of them are shown in planes close to ~{1 2 0}. The diameter of these loops reaches the large value of 400 nm with a mean value around 200 nm.

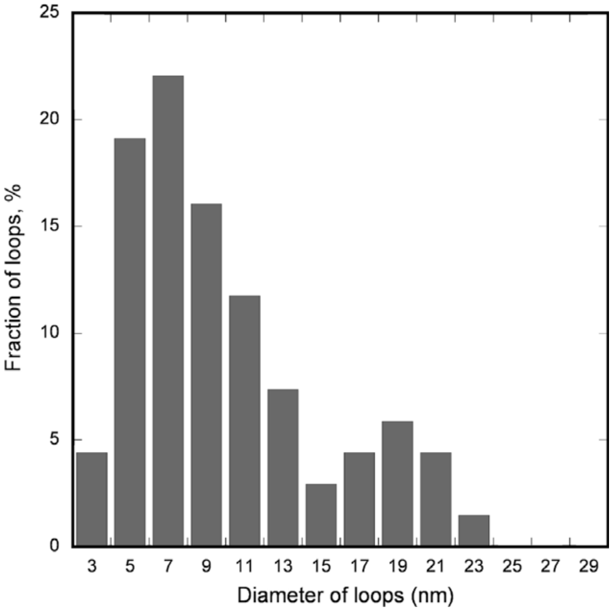

Fig. 2. Eurofer, distribution of the loops sizes after long-time irradiation at 350 °C, peaks at 7 and 19 nm.

4. Annealing effect in Eurofer and the model alloy

As the observations in Eurofer do not reveal any microstructure due to short-time irradiation at 350 °C conversely to the model alloy irradiated in the same conditions, which shows tiny loops (2 nm), post-irradiation annealing treatments have been performed. The purpose is to estimate if the produced defects have been totally eliminated by recombination or a large part of them is still present in the lattice.

Eurofer presents some dislocation lines before annealing but during heating, no evolution can be detected up to 450 °C. A small

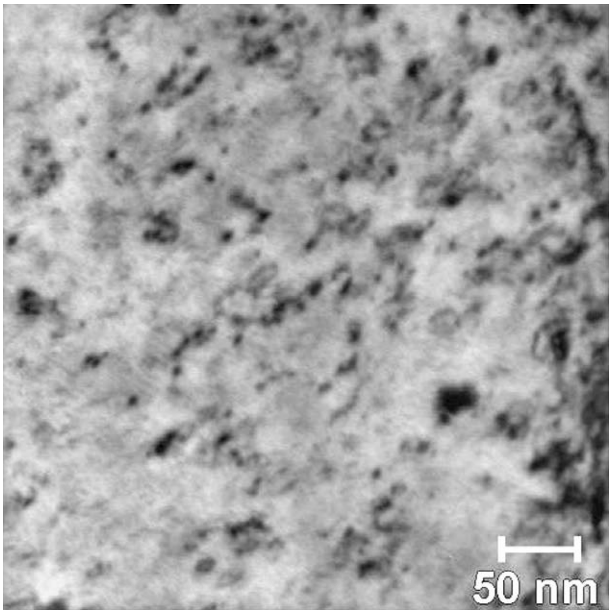

Fig. 1. Eurofer irradiated at 350 °C (long time: 1.4 × 10^{-4} dpa/s).

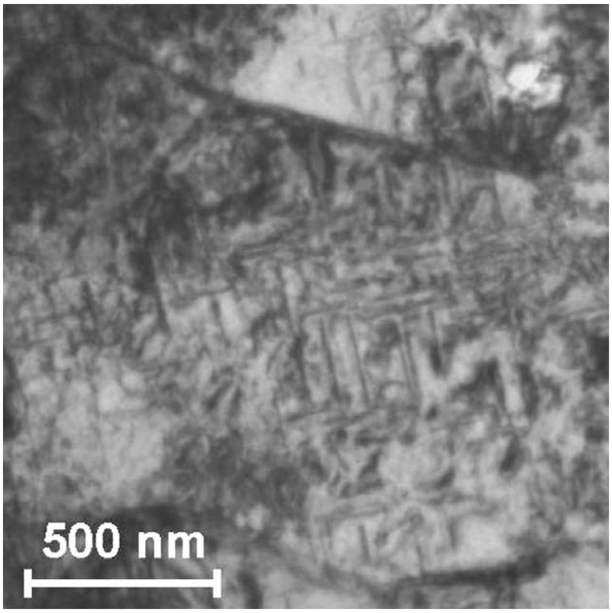

Fig. 3. Eurofer irradiated at 550 °C: loops edge on inside a martensitic lath.

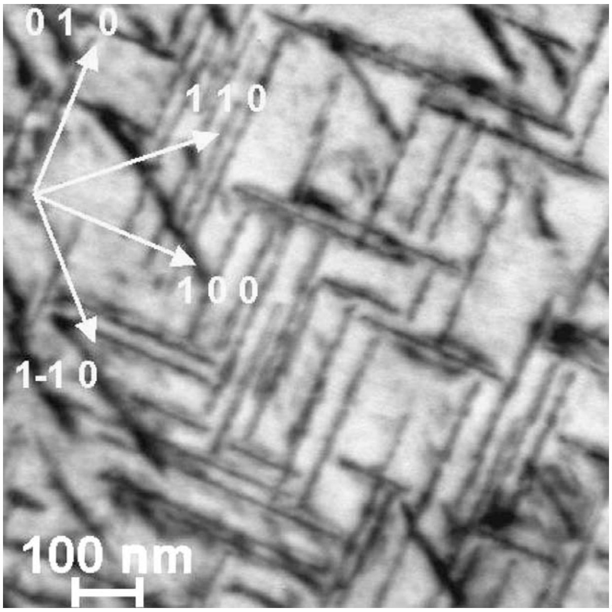

Fig. 4. Model alloy irradiated at 550 °C: loops in 100 and ~120 planes.

displacement seems to occur after heating at 500 °C. Beside the lines, no new defect appears up to heating at 550 °C. At this temperature, the evolution becomes important and small resolved loops appear.

The defects formed after heating consist in 3–15 nm resolved loops (Fig. 6). The larger ones show a square shape. The observations close to the foil edge and in the thicker region show the same distribution size despite a larger density (about 3) due to a larger thickness of the foil. This excludes a spurious effect as injection of vacancies from weak oxidation of the surfaces. More precisely, as the surface is similar for the two regions, an injection of defects (vacancies) from the surface would lead to larger clusters in the thin region.This means that the surface does not influence the loop growth.

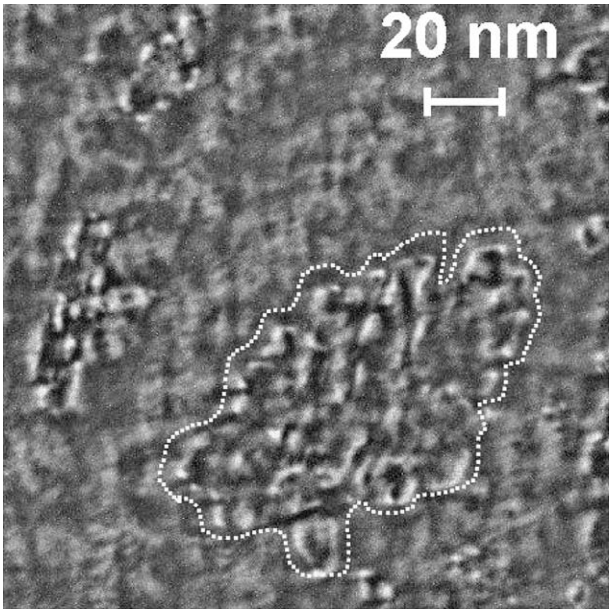

Fig. 5. Model alloy irradiated at 550 °C: loops tilted showing indentations.

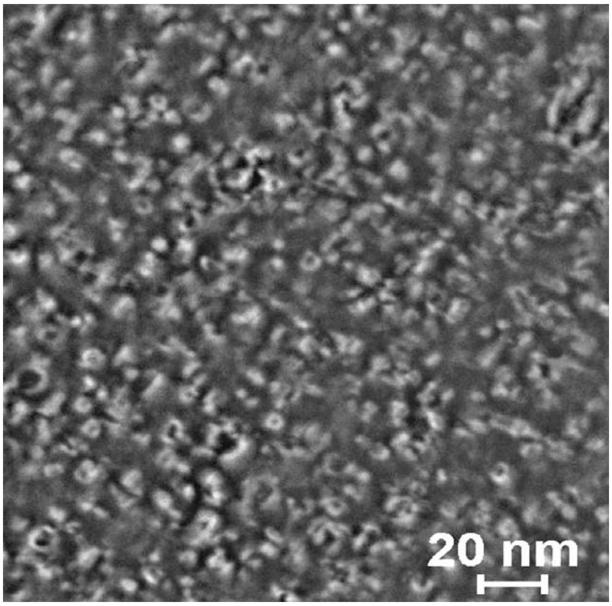

Fig. 6. Dislocation loops in the Eurofer after irradiation and annealing at 550 °C.

In the model alloy irradiated at 350 °C, a homogeneous distribution of tiny dislocations loops is present (<2 nm). During heating, the evolution begins at 456 °C and at the final temperature step (554 °C) loops are now resolved (5 nm). Many regions of the foil have been observed where the growth of initial tiny loops has been confirmed.

5. Irradiation mode effect

5.1. Discrepancies at 350 °C between short and long-time irradiations

One major difference is the lack of clusters in the Eurofer after short time relatively to the presence of clusters after long time (Table 2). This absence has been analyzed by heating the sample at higher temperature: 550 °C. The formation of defects has been observed, meaning that the clusters were too small to be observed at that fluence.

The larger fluence of the long-time irradiation is the main argument to explain the formation of loops in the Eurofer. The population of loops presents a bi-modal distribution with two peaks at 7 and 19 nm. Again, in the model alloy, as compared to short-time irradiations, the loop size increases and a bi-modal distribution is present.

5.2. Discrepancies at 550 °C between short and long-time irradiations

The same type of large loops is present after short and long-time irradiations in the two alloys (Table 3). After long-time

Table 2
Microstructure at 350 °C after short and long-time irradiation.

		Eurofer	Model alloy
Short time	Loops density	No observable defects	Uni-modal distribution: ~5×10^{21} m^{-3}
	Loops size	–	Small loops: <5 nm
Long time	Loops density	About twice than in the model alloy ~3×10^{21} m^{-3}	Bi-modal distribution: ~5×10^{21} m^{-3}
	Loops size	Two sizes: 7 and 19 nm	Larger loops, two sizes: 7 and 28 nm

Table 3
Microstructure at 550 °C after short and long-time irradiation.

		Eurofer	Model alloy
Short time	Loops density	Heterogeneous, $<10^7$ m^{-1}	$<10^{20}$ m^{-3}
	Loops size	Max 150 nm	Max. 150 nm
Long time	Loops density	At least twice larger than after short time	1–2 × 10^{20} m^{-3}
	Loops size	Max. 300 nm	Max. 400 nm

irradiation, the clusters are very much larger with a higher density. Too many defects are present to give a good observation.

In the Eurofer, the original dislocations network is still visible between loops after short-time irradiation in Eurofer. It is no more possible to observe it after the long-time irradiation.

6. Discussion and conclusions

After irradiation at 550 °C, the loop free zone close to surfaces is small and the microstructure of the two alloys consists mainly of large edge loops located in {1 0 0} planes. They are present in Eurofer, meaning that the initial glide dislocations are not good sinks. These loops do not have a tendency to glide and interact to form a dislocation network in comparison to what occurs in fcc alloys. The occurrence of indentations on loops has been reported after electron irradiation in pure iron [1,2] and Fe–Cr (about 9–10%) [3,4]. It is the first report of their occurrence after ion irradiation. It is correlated to a flux of vacancies towards the interstitial loops.

After irradiation at the lower temperature (350 °C), smaller loops are almost always present. They do not show the same morphology than after high temperature irradiation (550 °C), and two populations are present whose size are, respectively, centered at 7 and 19 nm. In the case where no loops were observed in Eurofer, a post-irradiation heating at 550 °C reveals resolvable loops. They originate from the defects accumulated during the low-temperature irradiation that migrate and create large loops. In the model alloy, loops begin to grow at a lower temperature. This means that at the highest flux, the recombination rate does overcome the accumulation of defects.

We observed that the mean diameter of loops increases with a large factor (8–10) from 350 to 550 °C. This evolution is attributed to the number of nucleus that decreases with temperature. The evolution intensity is larger than in stainless steels and the discrepancies need to be analyzed in terms of point defects behaviour.

About the flux effect, the lack of defects in short-time irradiation at low temperature in Eurofer requires to be reconsidered by comparison to long-time irradiations at the very same fluence. Nevertheless, it seems that the flux variation does not modify qualitatively the morphologies of loops and the microstructure is similar. Some observed discrepancies on size of loops, at high temperature are also attributed to fluctuations of the ion beam leading to an unexpected higher fluence.

Despite a dose rate thousand times lower than after in reactor neutrons irradiations, the microstructure is similar to the one of a small damage neutron irradiation. Consequently the simulation by heavy ions appears as a reliable way to predict neutrons effects.

Acknowledgments

This work was supported by European communities within the European Fusion Technology course.

References

[1] N. Yoshida, M. Kiritani, F. Eiichi Fujita, J. Phys. Soc. Jpn. 39 (1975) 170.
[2] M. Kiritani, H. Takata, K. Moriyana, F. Eiichi Fujita, Philos. Mag. A 40 (1979) 779.
[3] D.S. Gelles, S. Ohnuki, H. Takahashi, H. Matsui, Y. Kohno, J. Nucl. Mater. 191–194 (1992) 1336.
[4] T. Muroga, Y. Nonaka, N. Yoshida, J. Nucl. Mater. 233–237 (1996) 1035.