ELSEVIER
Journal of Nuclear Materials 271&272 (1999) 111–114
journal of nuclear materials

Microstructural examination of Ni-ion irradiated Fe–Ni–Cr alloys followed to micro-zone deformation

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Abstract

Examination of deformation microstructures induced by micro-indentation technique by means of transmission electron microscopy (TEM) following an MV range metallic ion irradiation was performed in order to study the influence of irradiation-produced microstructural defects on plastic deformation behavior of materials. In this work, specimens of an Fe–Cr–Ni austenitic model ternary alloy before and after irradiation with 4 MeV Ni ions at 723 K were indentation-tested at loads as low as 9.8 mN using an instrumented micro-indentation testing system. The dislocation microstructures of the specimens' regions beneath the indents were successfully examined with a TEM following a cross-sectional thin film processing with a focused ion beam (FIB) processing system. This paper describes the experimental procedure developed in this study and the results from the first series of the experiments.

1. Introduction

The hardening and the ductility degradation due to high energy neutron irradiation are among the crucial engineering issues for materials for both fusion structural and functional components. The ductility degradation of the austenitic stainless steels accompanied by the matrix hardening, which are neutron irradiated at about 473–673 K, is phenomenologically understood to a certain extent [1] but the underlying mechanism has not clearly been explained. Similar behavior might be a potential important issue for the advanced fusion materials such as reduced activation ferritic steels and vanadium alloys, for which the characterization of irradiation-induced phenomena is being extensively performed.

The charged particle irradiation using MV range accelerators is an effective experimental technique to study the irradiation-induced phenomena in the fusion materials, mainly for the advantages in controllability of the irradiation conditions that also can be well characterized. However, it is generally recognized not to be suitable for the studies on mechanical properties because of the limited penetration depth of the ions in that energy range, except for the combined use with the ultra-low load indentation technique or micro-indentation technique.

The micro-indentation on the ion-irradiated materials was successfully performed by Zinkle, and Oliver for relatively high energy ions [2] and more recently applied to various materials including the light water reactor pressure vessel steels [3] and reduced activation ferritic steels [4]. In this work, as a complement to the phenomenological studies on the irradiation-induced hardening, the microstructural examination of micro-indented materials was attempted for the ion-irradiated and unirradiated specimens. This paper covers the experimental technique that was developed in this work and the initial results on a model austenitic alloy.

2. Experimental procedure

The material used in this study was a polycrystalline Fe–15Cr–20Ni (mass%) austenitic model ternary alloy in

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PII: S0022-3115(98)00734-X

a solution annealed condition. The detailed description of the material has been published elsewhere [5]. A sheet of the model alloy was cut into specimens of the small coupon type using a high-speed precision cutter so-called dicing saw. The size of the specimens was $2 \times 3 \times 0.2$ mm and one of the 3×0.2 mm sides were irradiated after a diamond-polishing followed by a electrolytic surface finish.

The ion-beam irradiation experiment was carried out at the High-fluence Irradiation Facility, University of Tokyo (HIT Facility) [6]. The atomic displacement damage was introduced by 4 MeV Ni^{3+} ions accelerated with a Tandetron tandem accelerator operating at 1 MV. The irradiation temperature, displacement dose and displacement rate were 723 K, 4 displacement per atom (dpa) and 1×10^{-3} dpa/s, respectively. The nominal displacement dose and rate are the average over the 1 μm thick layer on the irradiated surface, as calculated by the TRIM-92 code [7] assuming a 40 eV of the average displacement threshold energy.

The irradiated specimens were then indentation tested at a load of 9.8 mN using an Akashi MZT-3 instrumented micro-indentation testing system. The direction of indentation was chosen to be in parallel to the ion beam axis or normal to the irradiated surface. The shape of indenter tip was triangular pyramidal with the semi-apex angle of 68 degrees. The unirradiated and irradiated specimens' regions right beneath the indents were made into thin films, with a JEOL/Micrion JFIB-2100 focused ion beam (FIB) processing system. The foils were made so that they include the indentation axis. The ion beam used in this process was Ga accelerated at 30 kV. The microstructural examination was carried out using JEOL JEM-2010EX transmission electron microscopy (TEM) operating at 200 kV.

3. Specimen processing with the FIB device

Fig. 1 illustrates a representing schematic procedure of micro-processing by means of the FIB. At first, the specimens were irradiated with 4 MeV Ni^{3+} ions and then micro-indented. These indents could not be observed by secondary electron or secondary ion images produced by the FIB system. However, they could be clearly observed by an optical microscopy. Secondly, following the initial specimen thickness reduction to about 5 μm by means of a coarse mode FIB processing, the irradiated and indented surfaces were protected by physically depositing tungsten as thick as typically about 1 μm micrometer. Finally, the thickness of thin films were further reduced to approximately 0.1 μm for the TEM examination. Fig. 2 shows the typical bright field TEM image of the thin film right after the FIB processing. As noticeable in the micrograph, the FIB processing introduced very fine dot-like defects in the specimens. These defects are not precisely characterized but supposed to be mostly small dislocation loops of the interstitial type produced by the Ga ion bombardments. These defects could totally be removed by an electrolytic polishing for a very short time. These efforts successfully enabled a TEM examination of both irra-

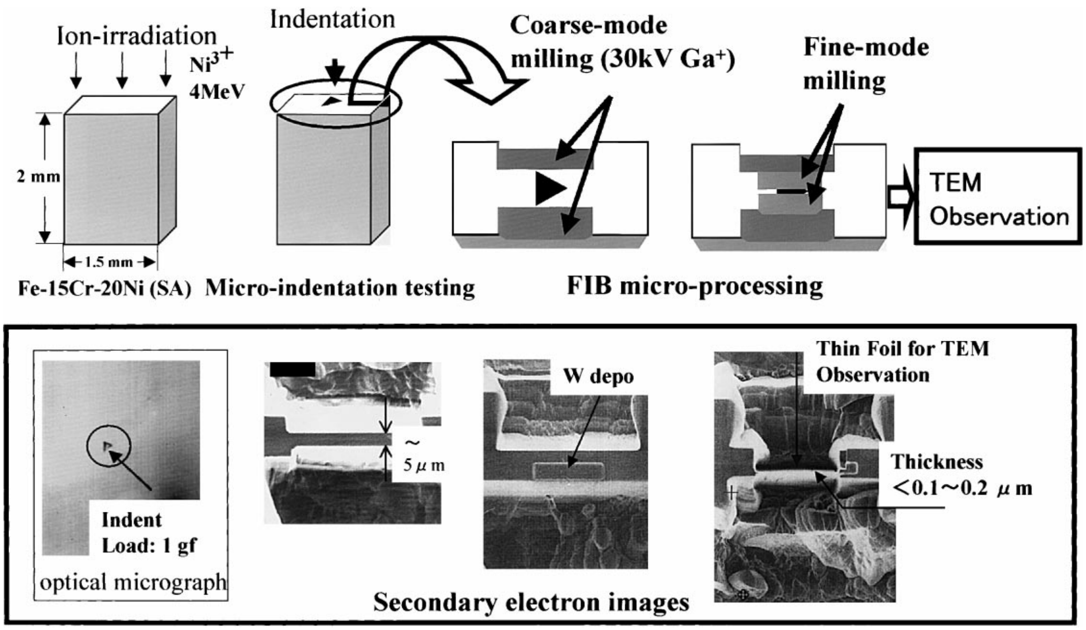

Fig. 1. A schematic illustration of the procedure of micro-processing developed in this study.

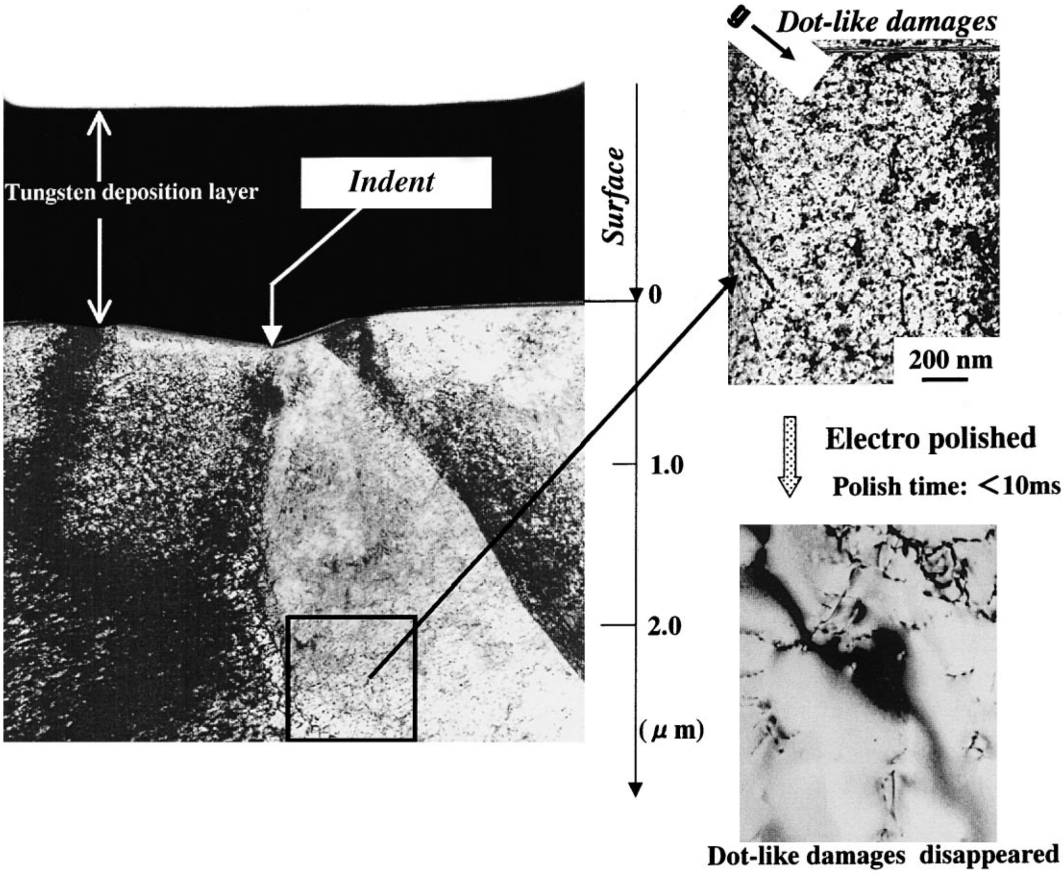

Fig. 2. Transmission electron micrograph of the FIB-processed thin film before the electrolytic polishing.

diation- and indentation-induced microstructural defects.

4. Results and discussion

Fig. 3 shows the region right beneath the indentation in the unirradiated specimen. In this region, high density dislocations correspond to plastic deformation were observed. The radius of high dislocation density region was about 2.5 μm. The depth of indent after the indenter removal was 330 nm, whereas the maximum indenter displacement below the original surface during the indentation test was 345 nm. The shape of the deformed volume was rather anisotropic; the deformation reached as far as about 3.0 μm from the indent top in some directions though it stopped at less than 1.5 μm in other directions. This observation suggests the limited applicability of simple deformation models for the micro-indentation [8]. The deformation was also frequently observed to be terminated at the grain boundaries, although such function of the grain boundaries will substantially be influenced by their characteristics.

The microstructure around the indent in the irradiated specimen is presented in Fig. 4. In the region free from the indentation-induced deformation, the

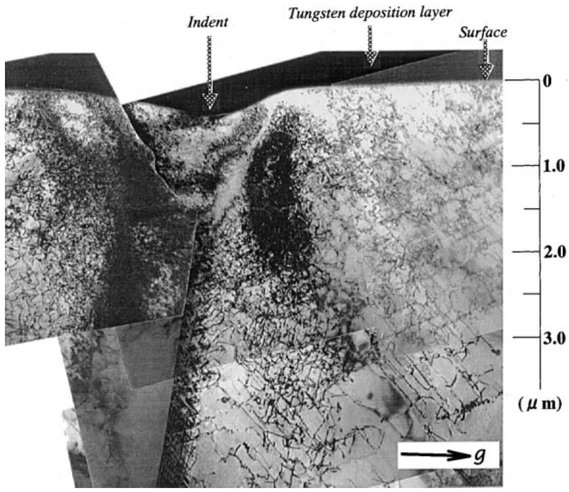

Fig. 3. Bright field TEM image of the region beneath the indent in the unirradiated specimen.

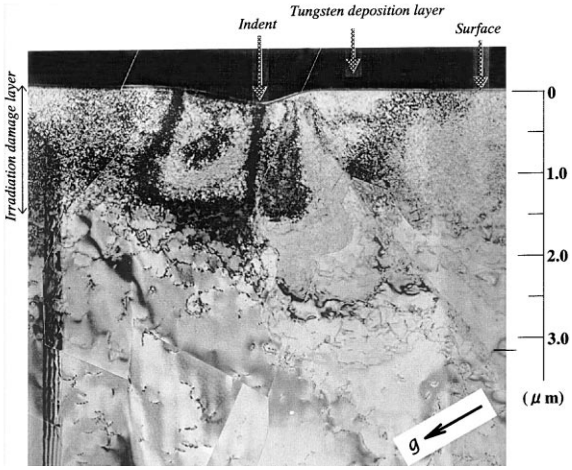

Fig. 4. Bright field TEM image of the region beneath the indent in the irradiated specimen.

distribution of damage microstructures due to the ion-irradiation was in good agreement with the damage distribution profile calculated by the TRIM code. In the case of irradiated specimen, the volume of indentation-induced high dislocation density region was significantly smaller than in the unirradiated specimen for the same indentation load, because of the increased plastic deformation resistance due to the irradiation-produced defects. The shape of the plastically deformed region seems like more hemispherical than that in the unirradiated specimen. This could be explained that the uniform distribution of the irradiation-produced high density small dislocation loops on four {1 1 1} family planes in the face centered cubic lattice worked as an isotropic barrier against the motion of *a*/2⟨1 1 0⟩ dislocations. The observation of the significant reduction in the dislocation loop number density within the highly deformed region indicates the Frank loop sweeping mechanism [9,10] by the mobile dislocations.

5. Summary

An experimental method, for the microstructural characterization of irradiated and deformed materials, by means of ion-irradiation, micro-indentation, FIB micro-processing technique and TEM was developed and applied to an Fe–Cr–Ni model austenitic alloy.

- The FIB processing introduced very fine dot-like defects in the specimens. The defects could easily be removed by electrolytic polishing. Tungsten deposition prior to the FIB processing was essential to protect the original irradiated surface.
- Both irradiation- and indentation-induced defects were clearly observed by TEM. The distribution of damage microstructures due to the ion-irradiation was in good agreement with the TRIM calculation.

In the region right beneath the indents, high density dislocations correspond to the plastic deformation were observed. The radius of indentation-induced high dislocation density region in the irradiated specimen was much smaller than in the unirradiated specimen. The evidence of the interaction of indentation-produced dislocations with the irradiation-produced dislocation loops was observed. Detailed investigation of defect interactions will be performed in future studies.

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