



Microstructural characterization of deformation localization at small strains in a neutron-irradiated 304 stainless steel



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ABSTRACT

A specific phenomenon – highly localized regions of deformation – was found and investigated at the free surface and near-surface layer of a neutron irradiated AISI 304 stainless steel bend specimen deformed to a maximum surface strain of 0.8%. It was shown that local plastic deformation near the surface might reach significant levels being localized at specific spots even when the maximum free surface strain remains below 1%. The effect was not observed in non-irradiated steel of the same composition at similar strain levels. Cross-sectional EBSD analysis demonstrated that the local misorientation level was highest near the free surface and diminished with increasing depth in these regions. (S)TEM indicated that the local density of dislocation channels might vary up to an order of magnitude. These channels may contain twins or may be twin free depending on grain orientation and local strain levels. BCC-phase (α -martensite) formation associated with channel-grain boundary intersection points was observed using EBSD and STEM in the near-surface layer.

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1. Introduction

It is well known that plastic deformation occurs in a localized fashion in neutron-irradiated polycrystalline metallic materials. Numerous defect-free channels (analogy of slip lines) form in the structure, providing accommodation of plastic strain. Deformation localization and channeling have been studied extensively, but the fundamental mechanisms are still not well understood [1]. The lack of understanding can be contributed to the complexity of the problem, which includes irradiation defect formation, changes in microchemistry, and influences from mechanical testing conditions. Investigations are further complicated due to difficulties in testing and characterizing irradiated materials. Localized deformation has been hypothesized as one of the causes for failure in austenitic steels for fission applications [1] and identified as a potential degradation mechanism for candidate materials in fusion applications [2].

Careful analysis of the literature shows overwhelming complexity for localized deformation in polycrystalline materials, especially when subjected to irradiation. One weakly explored area is structure evolution and localized strain distribution within grains and

grain colonies on the free surface of materials experiencing strains below 1%. The usual experimental procedure consists of straining at some degree (5%, 10% or so) and further microstructure analysis. In this case, processes incurred at strains below 1% are effectively masked by well-developed overall plastic deformation. At the same time, the area of strains below 1% is especially important for irradiated steels since small strains may be introduced in the material by occasional loading. Also, it has been shown that dislocation channels might form long before formal yield stress is reached [3,4]; however, the origin sites of such “earlier channels” were not analyzed. Fukuya et al. observed notable evolution of local misorientation caused by sub-yield stress loading, and it is interesting to take the next step and investigate structure evolution in the material in the small strain regime [5].

Here, a methodical, multi-scale approach to characterize localized deformation in a neutron-irradiated 304 austenitic stainless steel tested to small strain levels was employed. The multi-scale approach allows for detailed insights into the mechanical behavior of irradiated materials experiencing small strain levels at the free surface (<1.0%). This work couples several analytical microscopy techniques including optical microscopy coupled with digital image correlation (DIC), scanning electron microscopy–electron backscatter diffraction (SEM–EBSD), focused ion beam (FIB) site-specific transmission electron microscopy (TEM) sample preparation, and scanning transmission electron microscopy (STEM) based orientation mapping. A neutron-irradiated 304 austenitic stainless steel irradiated to 4.4 dpa at 320 °C was utilized for the study due

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to the range of information in literature on the irradiated microstructure.

2. Materials and methods

A high-purity commercial AISI 304 austenitic stainless steel annealed prior to irradiation was used for this study. Unirradiated archive material from the same heat of alloy was not available but a new heat with similar properties (composition, grain size, etc.) was fabricated to serve as an analog to the irradiated material. The compositions of the irradiated steel and unirradiated analog are provided in Table 1. The average grain size was 67 μm for the irradiated alloy and 57 μm for the unirradiated alloy. The irradiated specimens obtained for this study were part of a cooperative research program on irradiation-assisted stress corrosion cracking (IASCC) research [6]. This program utilized more than 10 modified alloys irradiated in the BOR-60 fast reactor and characterized across multiple institutions all using the same heats of alloys [6,7]. Irradiated tensile samples were not available for the current work resulting in specimens being fabricated from the end of irradiated non-deformed tensile bars, resulting in small plates with dimensions of 5.0 mm $\times$ 3.5 mm with a nominal thickness of 1.2 mm. The material was irradiated in the BOR-60 fast reactor to 4.4 dpa at 320 °C and an average dose rate of $\sim 8 \times 10^{-7}$ dpa/s [7]. Due to the larger source material of the unirradiated analog, SS-J3 tensile specimens were fabricated from the unirradiated material stock.

Samples were prepared for deformation experiments by mechanically polishing both sides to remove surface imperfections using standard metallographic procedures. Mechanical polishing was followed by electro-polishing using a Struers unit with standard A2 electrolyte and etched with 10% oxalic acid at 6 V DC to remove any deformation caused from mechanical polishing and to reveal grain structure. Final surface preparation was completed with a 2 s electropolish to remove any etching products. The final thickness of the investigated irradiated specimens was 0.8 mm. Analysis of prepared specimens showed clear austenitic structure without any signs of pre- or post-irradiation deformation in any sample. The surface of unirradiated specimens was prepared in exactly the same way.

Deformation of the irradiated specimens was completed using a specialized four-point bend test assembly for sub-sized specimens. The experimental configuration allowed for small strains to be subjected to the sub-sized specimens with increased accuracy over previous configurations. The span distance was 3.77 mm with the top and bottom rods having a diameter of 0.40 and 1.23 mm, respectively. Bend tests were performed at room temperature (RT) with an estimated strain rate of $\sim 10^{-4}$ s $^{-1}$ on a MTS tensile screw-driven machine (model Insight 2–52; load capacity 2 kN). The design of the specialized load frame allowed for optical light microscopy observations of the surface during straining. A Keyence VHK-1000 long-focal optic microscope running at 15 frames per second (fps) was used to conduct in situ observation of the area of interest and to track channel appearance and propagation; the images were recorded at a magnification of 600 $\times$, corresponding to a $\sim 800 \mu\text{m} \times \sim 600 \mu\text{m}$ field of view.

To calculate the strain field from the four point bend specimens, VIC-2D commercial software and a custom program coupled to an

Allied Vision Technology GX3300 CCD were used; this method, called optic extensometry or digital image correlation (DIC), is described in detail elsewhere [8,9]. Using DIC, it was found that strain value at the center of the specimen was 0.008 ± 0.002 (or 0.8%). To calculate stress and strain fields in the deformed specimen, including stress distribution at the particular grain configuration, COMSOL 4.0 commercial finite-element analysis software was employed.

The unirradiated tensile specimens of SS-J3 geometry (gauge dimensions 5.0 $\times$ 1.2 $\times$ 0.7 mm) were strained at RT at strain rate of 10^{-3} s $^{-1}$. The same MTS load frame was used for the tensile test that was used for the four point bend tests. Specimens were deformed at 1%, 3%, 6%, and 9%. The surface of the specimens during and after the experiment was preserved from any occasional damage. It was assumed that at small strain level there is no principal difference in deformation behavior between the 4-point bend scheme and conventional tensile test used here.

SEM was performed using a JEOL JSM 6500F FEG-SEM equipped with EDAX's TSL/OIM analysis system. The surface of the specimens maintained good quality after deformation and therefore no subsequent polishing or sample preparation steps were needed for any specimen after deforming. SEM was performed with an accelerating voltage of 20 kV, a working distance between 12 and 15 mm, and a 70° tilt. EBSD maps were measured on a hexagonal grid with a step size of 0.1–2 μm with background subtraction corrections. The camera ran at ~ 50 fps in 1 $\times$ 1 binning mode.

Cross-sectional samples for EBSD and thinned samples for TEM and STEM were prepared from specific sites of interest using standard FIB lift-out procedures. A FEI Quanta 3D 200i DualBeam FIB was used to create the cross-sectional FIB lift-out specimens. A $\sim 20 \times 10 \times 2 \mu\text{m}$ lift-out was mounted in a chevron post. The chevron mount limited twisting and bending during subsequent thinning procedures. The lift-out was thinned and polished using a low-angle, low-keV ion beam to provide a defect-free surface for EBSD analysis. Specimens were left intentionally thick (>100 nm) to limit localized heating and increase the interaction volume available for EBSD analysis. After ion polishing, the specimen was transferred to the JEOL JSM 6500F FEG-SEM to conduct cross-sectional EBSD analysis. After analysis, the specimen was reloaded into the FEI Quanta and thinned to electron transparency for TEM and STEM investigations. Low-angle, low-keV ion polishing was performed on both sides to reduce artifacts from the FIB sample preparation.

Thinned FIB specimens were investigated using a Philips CM200 FEG S-TEM operated in both TEM and STEM mode with an accelerating voltage of 197 kV in both modes. TEM mode was used for general bright field (BF) imaging and defect contrast imaging. Due to the limited field of view of the TEM images, sequential images were taken and post-processed stitched to provide a full field of view of the specimen. STEM mode was used for further general imaging and STEM-based orientation mapping. STEM operation allows for scan coil control of the beam, providing precise placement of the beam across a feature of interest while simultaneously generating diffracted Kikuchi patterns needed for orientation mapping. Diffracted Kikuchi patterns were taken at a calibrated camera length of 119 mm at the CCD with the patterns indexed using software and procedures detailed in [10]. Pattern analysis was completed assuming both a face-center-cubic (FCC)

Table 1

Composition and condition of materials investigated. Irradiated alloy designation corresponds to the alloys described in Ref. [6].

Alloy	Fe	C	Mn	Si	Cr	Ni	Mo	N	Grain size (μm)	Condition	Irr. temp (°C)	Dose (dpa)
SW	Bal.	0.022	1.07	0.24	18.42	10.45	0.0	0.025	67	Annealed	320	4.4
SW'	Bal.	0.016	1.03	0.24	18.39	10.45	0.0013	0.056	57	Annealed	Unirr. analog	

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