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The effect of grain orientation on nanoindentation behavior of model austenitic alloy Fe-20Cr-25Ni☆

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Instrumented nanoindentation was used to investigate the hardness, elastic modulus, and creep behavior of an austenitic Fe-20Cr-25Ni model alloy at room temperature, with the indented grain orientation being the variant. The samples indented close to the {111} surfaces exhibited the highest hardness and modulus. However, nanoindentation creep tests showed the greatest tendency for creep in the {111} indented samples, compared with the samples indented close to the {001} and {101} surfaces. Scanning electron microscopy and cross-sectional transmission electron microscopy revealed slip bands and dislocations in all samples. The slip band patterns on the indented surfaces were influenced by the grain orientations. Deformation twinning was observed only under the {001} indented surfaces. Microstructural analysis and molecular dynamics modeling correlated the anisotropic nanoindentation-creep behavior with the different dislocation substructures formed during indentation, which resulted from the dislocation reactions of certain active slip systems that are determined by the indented grain orientations.

1. Introduction

Nanoindentation is a robust technique for studying localized deformation behavior at the nanoscale and micron scale; it provides an efficient and economical approach to developing a fundamental understanding of the deformation mechanisms that can contribute to multi-scale modeling [1–7]. Small-volume deformation is often required to assess the mechanical properties of size-limited structures such as nanostructured materials, thin films, and ion irradiation–damaged regions [7–11]. Anisotropic hardness and elastic modulus have been observed in some materials at nanoindentation scales [2,3,8,12], which are related to the activation of different deformation mechanisms when the material is stressed along different crystallographic orientations [3–5,13]. Besides hardness and modulus, nanoindentation can probe creep properties from a very small volume of materials [7,8,14–18]. While holding the compression load during nanoindentation, materials experience continuous displacements, mimicking the conventional primary and secondary creep stages, from which similar creep parameters are extractable [14–16]. However, indentation-induced significant plastic deformation prior to indentation creep can be a problem and requires serious consideration in fitting nanoindentation-creep results to the power-law equation [7,17,18]. In addition, the indentation direction with respect to the indented grain orientation influences the activation of different slip systems, leading to possible variations in indentation creep behavior. To the authors' best knowledge, a systematic investigation of the anisotropy in nanoindentation creep, combining microstructural analysis and modeling efforts, has never been carried out.

Austenitic stainless steels (SSs) are widely used in different components of nuclear and fossil power plants. Newly developed and commercialized heat-resistant austenitic SSs, targeting

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promising thermal and mechanical properties and corrosion resistance in harsh environments, are likely to benefit life extension and improve the safety margin and thermal efficiency of power plants. Recently, the NF709 (20Cr-25Ni-1.5MoTiNb) class of alloy was selected for development as a candidate structural material for sodium-cooled fast reactors because of its superior creep and corrosion resistance compared with 300-series austenitic SS. Research efforts on NF709 have focused on creep behavior [19], thermal stability [20,21], and radiation resistance [22]. There is still no fundamental understanding of the deformation mechanisms of NF709, which would be of great value to the comprehension of strengthening-failure mechanisms, material development, and multi-scale modeling efforts.

In this study, we investigated the anisotropic nanoindentation hardness, elastic modulus, and creep behavior of a model alloy of NF709. The deformation mechanisms were revealed through electron microscopic observations and molecular dynamics (MD) simulation. The methodology explored in this study will be applicable to studies of similar materials. In particular, the results and understandings obtained from this study will serve as a critical baseline and guidance for further experimental and theoretical studies of the engineered alloy NF709 and alloys with radiation damage.

2. Materials and methods

A model austenitic alloy of NF709 with a measured composition of Fe-20Cr-24.5Ni-0.0013C in weight percentages was used in this study. The alloy was cast using vacuum arc melting and solution-annealed at 1050 °C for 1 h, followed by hot rolling at 900 °C with an 85% thickness reduction to 3.8 mm and water quenching. A sample of the alloy was mechanically polished to a mirror finish with colloidal silica as the final preparation step.

Nanoindentation tests were conducted on the polished sample using an Agilent G-200 nanoindenter with a Berkovich tip at room temperature. Four indentation matrixes (8 × 10) with 20 μm interspacing between the indents were performed on the sample using the continuous stiffness measurement with the hardness and elastic modulus calculated using Oliver and Pharr's method [23]. Each nanoindentation test ran at a constant loading rate of 0.053 mN/s to a maximum depth of 1000 nm into the indented surface. The hardness and modulus values were averaged over the indentation depth of 800–900 nm. After the maximum indentation depth was reached, the load was held for 600 s to probe the creep behavior of the material.

Electron backscatter diffraction (EBSD: EDAX OIM 6.0) was performed on the indentation matrixes to analyze the grain sizes of the material and to identify the grain orientation under each indent. Results for the indentations, measured near the centers of the grains larger than 30 μm, were included in the statistical analyses. Presumably, there was negligible influence from grain boundaries because of the small indentation size compared with the grain size. Indentations exerted on sample surfaces close to {001}, {101}, and {111} were selected for detailed microstructural characterization. Scanning electron microscopy (SEM: JEOL 6500 F at 5 kV) investigated the indentation-induced surface morphology changes; cross-sectional transmission electron microscopy (TEM: JEOL 2100 F at 200 kV) in either conventional TEM or scanning TEM (STEM) modes investigated the microstructural evolution underneath the indents. A focused-ion-beam (FIB: FEI Versa 3D) instrument was used to lift out the lamella underneath the selected indents for TEM characterization. Progressive final cleaning steps using a 5 to 2 kV ion beam were employed to minimize possible artifacts on the lamellas from FIB processing. The localized deformation mechanisms were understood by correlating the mechanical properties with the microstructures.

In addition to the experimental efforts, the large-scale atomic/molecular massively parallel simulator (LAMMPS, http://lammps.sandia.gov) [24] was used for MD simulations of the indentations in three Fe-20Cr-25Ni systems with surface orientations of (001), (101), and (111). An embedded atom method potential developed by Bonny et al. [25] was used. The simulation cells had dimensions of ~40 × ~40 × ~40 nm^3 with a periodic boundary condition applied in the *x*- and *y*-axes. In the *z*-axis, six layers of atoms at the bottom were fixed. The top surface in the *z*-axis was a free surface that was indented. The radius of the rigid spherical indenter was 5 nm. The interactions between the indenter and the sample were described by the Ziegler-Biersack-Littmark (ZBL) part of the potential from Bonny. Before the nanoindentation simulation, the systems were equilibrated at 300 K using a constant-temperature, constant-volume ensemble (NVT). The OVITO software was used to visualize and analyze the simulation results [26].

3. Results and discussion

3.1. Hardness and elastic modulus

The EBSD characterization indicated equiaxed grains with some twins in the material, suggesting complete dynamic recrystallization occurred during the hot rolling. The grain size, analyzed by area, averaged 40 ± 20 μm in diameter, disregarding the twins. Among the 320 nanoindentation tests, 70 indents were found near the central areas of the 70 large grains. The elastic modulus and hardness results of the 70 indentation tests are plotted in the standard stereographic triangles (SST) with color-coded values in Fig. 1a–b. Each data point represents the indented grain crystallographic orientation referring to the surface normal, i.e., the direction of the indentation. The statistic modulus and hardness distributions of the indentations close to {001}, {101}, and {111} are plotted in Fig. 1c–d, with the indented grain orientations within a solid angle of 0.14 steradian from the pole directions. To better visualize the distributions, the Gaussian fitting curves were superimposed on the statistical data. The modulus results in Fig. 1a and c show a strong anisotropy depending on the crystallographic orientation of each grain. The grains close to {111} have the highest moduli on average, followed by the grains close to {101} and then {001}. The elastic moduli range from 197 to 234 GPa, slightly higher than the values for 316L SS (155–190 GPa) and for the model austenitic alloy Fe-15Cr-15Ni (171 GPa) [12,13]. The dependence of hardness on grain orientation in Fig. 1b and d is not strong; yet the grains near {111} have the highest hardness, whereas the hardness near {001} and {101} is comparably lower. The hardness ranges from 1.85 to 2.21 GPa, which is close to the nanoindentation hardness range of 316L (2.0–2.2 GPa) but higher than that of alloy Fe-15Cr-15Ni (1.46 ± 0.02 GPa) [12,13]. The anisotropy in elastic modulus and hardness is in agreement with values for 316L and Hadfield steels reported earlier [12,27].

The anisotropy in moduli was generally observed in uniaxial experiments on single crystals and is attributed to the anisotropy in elastic constants. The anisotropy in hardness can be qualitatively correlated with the resolved shear stresses on slip and twinning systems estimated from Schmid's law for uniaxial compression. Table 1 lists the maximum Schmid factors for face-centered cubic (fcc) slip and twinning systems under compressions along the <001>, <101>, and <111> directions and the numbers of systems having the maximum Schmid factors. The maximum Schmid factor for the <111> compression is considerably lower than those for the <001> and <101> compressions; that difference could be the reason for the higher measured hardness near the <111 > pole in the SST [8,28]. This correlation suggests that during the loading