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

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ABSTRACT

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 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.
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 unveiled 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 C-200 nanoindenter with a Berkovich tip at room temperature. Four indentation matrices (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 matrices 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 nm³ 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 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 hardenability 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
stage of indentation, as the stress increases and the stress field expands in the material, the ease of continuous activation of slip/twinning systems plays an important role in determining the nanoindentation hardness.

As will be shown later, when the indentation direction deviates from the pole directions, the distributions of resolved stress on the deformation systems vary from the ideal case as provided in Table 1.

Table 1
Schmid factors for slip and twinning in fcc systems under uniaxial compressions along <001>, <101> and <111>.

<table>
<thead>
<tr>
<th>Compression</th>
<th>Slip</th>
<th>Twinning</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;001&gt;</td>
<td>0.41, 8 systems</td>
<td>0.47, 4 systems</td>
</tr>
<tr>
<td>&lt;101&gt;</td>
<td>0.41, 4 systems</td>
<td>0.24, 4 systems</td>
</tr>
<tr>
<td>&lt;111&gt;</td>
<td>0.27, 6 systems</td>
<td>0.16, 6 systems</td>
</tr>
</tbody>
</table>

3.2. Nanoindentation creep

Fig. 2a shows the load ($F$) vs. displacement ($h$) curves for six indentation tests on individual grains having the surface normal close to <111>, <101>, and <001>, denoted by dotted, solid and dashed lines, respectively. Under the same load, more displacement was observed in the (101) and (001) samples than in the (111) samples. This observation agrees with the anisotropy in hardness shown in Fig. 1. After the loading stage, the maximum load was held for 600 s; the additional displacement was monitored and is shown in Fig. 2b as a function of holding time ($t$). In Fig. 2b, the load-holding-induced additional displacement ($y$-axis) is denoted as $\Delta h$, which is the difference between the instantaneous displacement of the indenter ($h$) and its displacement at the beginning of the holding stage ($h_0$). The curves in Fig. 2b resemble general creep curves containing the primary and the secondary regimes. The deformation takes place more rapidly in the primary regime than the secondary regime. The displacement rate decreases with time in the primary regime and approximately reaches a constant in the secondary regime for each test. Overall, the constant strain rates were observed to be the highest in the indentations close to (111) grains and the lowest close to (101) grains.

To evaluate the tendency to creep deformation, a simple procedure was proposed to generate the parameter $P$ using Eq. (1) [18]:

$$P(t) = \frac{\Delta h}{dh}$$  \hspace{1cm} (1)

The $P$ values were averaged over 210 – 360 s of the load-holding stage and are presented in Table 2. The indentations on grains near (111) had the largest $P$ values, suggesting the highest tendency to creep, followed by the indentations on grains near (001) and then (101).

The instantaneous strain rate was deduced from $\dot{\epsilon} = \frac{1}{h} \frac{dh}{dt}$ [14] and is plotted as a function of stress ($\sigma$) in Fig. 2c–e for the six indentation tests during the holding stage, where $\sigma = F/(3\sqrt{3}h^2\tan^2 65.35)$ [29]. Correlating the data with the classic power-law creep model of

$$\dot{\epsilon} = K \exp \left( \frac{Q}{RT} \right) \sigma^n \, ,$$  \hspace{1cm} (2)

where $K$ is a material dependent constant, $Q$ the activation energy for creep, $R$ the gas constant, and $T$ the absolute temperature, the stress exponent $n$ can be obtained from the slopes of the $\ln \dot{\epsilon}$ vs. $\ln \sigma$ relationship as shown in Fig. 2c–e.

All the $\ln \dot{\epsilon}$ vs. $\ln \sigma$ data plotted in Fig. 2c–e show a transition between two linear fitting segments. Depending on the indentation orientation, the transition point corresponds to the time point of
60–120 s during the load-holding stage, which separates the primary and secondary regimes of the nanoindentation creep shown in Fig. 2b. The transitions in slope from the primary to the secondary regime depend on the indentation orientations. From a higher strain rate (primary regime) to a lower strain rate (secondary regime), the slopes of the {111} group data decrease while the slopes of the {110} group data increase. Table 2 lists the stress exponents $n$ fitted from the second regime, which mimics the steady-state regime of conventional creep. The values of $n$ are the lowest near {111} and the highest near {110}. As will be detailed in the following sections, the different indentation-induced microstructures lead to the difference in $n$. The data plotted in Fig. 2c–e are scattered, resulting to the significant standard deviations of the fitted $n$. That is because, at room temperature, the strain rate during nanoindentation creep is extremely low, leading to a very small span of stress of about 0.05 GPa during the secondary regime. Within such a small span of data points, any tiny disturbance, such as thermal drifting during nanoindentation, can lead to strong fluctuations in the data. Particularly for the indents near {110}, because the deformation during the secondary regime is so small, there are almost no changes in the stress during this regime. Nevertheless, the anisotropy in the nanoindentation creep behavior is strong enough to be distinguished by comparing Fig. 2c–e and the P and $n$ values in Table 2.

As a reference, the stress exponent $n$ for high-temperature creep of alloy 800 (Fe-21Cr-32Ni-TiAl) was fitted from the reported minimum creep rate and stress data [30]. Fig. 2f shows the fitted $n$ as a function of temperature. Except for the $n$ at 750 °C fitted with noticeably fewer data, $n$ increases approximately linearly with the decreasing temperature. The value of $n$ at room temperature for alloy 800 was estimated to be ~30 by linear extrapolation, which is within the range of the values of $n$, plus/minus the standard deviation.

Table 2

<table>
<thead>
<tr>
<th>Indented plane</th>
<th>P (nN\text{s}^{-1})</th>
<th>$n$, 2nd</th>
<th>SRS, 1st ($\times 10^{-5}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(-5 -6 6)</td>
<td>0.34 ± 0.13</td>
<td>18 ± 11</td>
<td>6.2 ± 0.9</td>
</tr>
<tr>
<td>(-5 6 6)</td>
<td>0.34 ± 0.13</td>
<td>34 ± 13</td>
<td>6.5 ± 0.7</td>
</tr>
<tr>
<td>(-3 -4 13)</td>
<td>0.20 ± 0.09</td>
<td>87 ± 63</td>
<td>4.8 ± 2.0</td>
</tr>
<tr>
<td>(-3 -2 19)</td>
<td>0.17 ± 0.08</td>
<td>81 ± 44</td>
<td>6.3 ± 0.7</td>
</tr>
<tr>
<td>(-3 -17 16)</td>
<td>0.03 ± 0.01</td>
<td>759 ± 488</td>
<td>5.9 ± 1.2</td>
</tr>
<tr>
<td>(3 -15 6)</td>
<td>0.06 ± 0.02</td>
<td>345 ± 301</td>
<td>5.4 ± 0.7</td>
</tr>
</tbody>
</table>

Fig. 2. (a) Load-displacement curves from loading to unloading; (b) load-holding-induced additional displacement ($\Delta h$) as a function of holding time; (c–e) strain rate-stress curves during the load-holding stage for six samples representing indentations on grains near {111}, {001}, and {101}, respectively; and (f) the M709 stress exponents from the nanoindentation creep tests as shown in (c) compared to high-temperature creep data for alloy 800 [30].

deviations, from the secondary regime of the near (111) nanoindentation creep tests. As will be discussed in the remainder of this paper, the indentation creep tests near (111) were less affected by the indentation-induced defect substructures and thus present a better simulation of conventional creep tests, despite the significant error bars introduced by the very small span of stress for the nanoindentation creep test.

Historically, the power-law model has been applied to several indentation-based tests, showing good agreement between the experimental results and literature [14–16]. However, the assumption of steady-state creep during indentation tests has been questioned by Goodall and Clyne [18]. Our results indicate that the value of \( n \) fitted from the primary stage of nanoindentation creep tests should not be treated as the steady-state stress exponent.

To understand the \( n \) values fitted from the primary regime, the Bailey-Norton model [17], which takes primary creep into account, was used:

\[
\varepsilon = A n^m t^m \tag{3}
\]

where \( t \) is the load-holding time; \( A, n', m' \) are temperature-dependent parameters. This formula can be derived into a strain-rate equation of

\[
\dot{\varepsilon} = m A n^m \sigma^n / \varepsilon^m \tag{4}
\]

Given that an ideal Berkovich indenter has an effective strain independent of the indentation depth [1], and that the additional displacement during the load-holding stage is much smaller than the displacement at the beginning of this stage, the parameter \( n \) fitted by the power law in Eq. (2) from the primary creep regime approximates the \( n'/m' \) in Eq. (4). The inverse of \( n'/m' \) in Eq. (4) refers to the strain rate sensitivity (SRS) when used in indentation-creep tests and tensile tests [31]. The SRS calculated from the first 60 s, i.e., the primary stage, was listed in Table 2. The values are in accordance with the results for 304 SS [31].

Both \( P \) and \( n \) suggest that nanoindentation along <111> leads to the strongest tendency to nanoindentation creep, whereas the <101> grains show the highest resistance to continuous deformation during the nanoindentation-creep tests. To understand this anisotropy in nanoindentation creep behavior, the following microstructure analysis and modeling efforts were carried out.

3.3. Microstructure analysis

Fig. 3a–c show the indents on grains with their surface normals close to [111], [001], and [101], respectively. In the vicinities of the indents, slip bands along certain directions were observed. The directions of the slip bands were found to be [-110], [-1-10] and [01-1] on the (111) surface, [±10] on the (001) surface, and [-1 ± 21] and [-101] on the (101) surface. The slip bands were all along the intersections of the (111) planes and the surface planes of each sample. The configurations of atoms in the surface planes are illustrated by the insets at the top right corners in Fig. 3a–c, with the dashed lines marking the intersections of (111) planes with the surface planes, which are parallel to the observed slip bands in all the samples. The patterns of the slip bands suggest that (111) planes are the active slip planes, and dislocation gliding is one of the deformation mechanisms for nanoindentation compression at room temperature. The three-fold, four-fold, and two-fold symmetries of the indentation-induced slip patterns on the (111), (001), and (101) surfaces were in accordance with previous studies [4,5,13].

Twinning was observed as an active deformation mechanism exclusively in the near [001] indented samples. The formation of twinning was revealed by the TEM characterization of cross-sectional lamellas lifted underneath the indents near [001]. Fig. 4a shows an example of the deformation twinning that took place within a radius of -2 \( \mu \)m beneath the indent tip, where the material was highly stressed and strained. Fig. 4b and c show dark-field images of the twins on the left and right sides of the indent tip, respectively, with the diffraction pattern insets indicating two of the (111) <112> twinning systems. Intersections of twinning were observed underneath the near [001] indents as the twins of different systems extended to cross each other.

Although indentation experiments generally apply multi-axial stresses [16], the presence of deformation twins in only the [001] sample rather than in the [101] or [111] samples was in agreement with the results of uniaxial experiments [27]. That is because in the region right underneath the indent, the direction of the compressive stress was close to the normal direction of the indented surface [16]. As shown in Table 1, the larger Schmid factor for twinning in the <001> compressed condition suggested that twinning is the predominant deformation mechanism over slip during compression along <001>. It is worth noting that we observed two [-111] [-1-12] and [1-11] [1-1-2] twinning systems on the left and right sides of the indent tip, respectively, as shown in Fig. 4b–c. The direction of the stress inclined toward the normal direction of the contacting surface between the sample and the indent tip. As a result, a particular twinning system, which was (-111)[-1-12] on the left or (1-11) [1-1-2] on the right of Fig. 4a, was activated by the received higher shear stress.

Deformation twinning was not observed in any of the near [101] or [111] indented samples, as shown in the STEM images in Fig. 5 obtained on the [011] zone-axis. High densities of dislocations were observed in both samples, with the densities gradually decreasing from the indented surface to the deeper regions of the samples. As pointed out by the white arrows, planar parallel dislocation arrays were observed in both samples on the (111) planes, suggesting that partial separation of dislocations was significant in these samples [35].

The dislocation arrays, as pointed out in Fig. 5, were further characterized using diffraction contrast imaging. Fig. 6a presents a bright-field image of the dislocation arrays in one of the near <111> indented samples, i.e., the (-5,6,6) sample shown in Fig. 2. Similar structures as presented in Fig. 6, referred as “dislocation tangles”, have been observed in tensile-strained SS 304 and 316LN [35,36]. Fig. 6b and c shows diffraction patterns of the [01-1] and [11-2]
zones, respectively. Fig. 6a was obtained by tilting the sample from the [112] zone to the [01-1] zone around the [111] axis. Comparing the direction of the dislocation arrays shown in Fig. 6a and the crystal orientations indicated by Fig. 6b, it can be seen that the dislocation arrays are parallel to the intersection of the (111) plane and the foil. Fig. 6d shows a higher-magnification bright-field image of the dislocation arrays, obtained at a two-beam condition of \(g = 111\), as indicated in Fig. 6b with the dashed circle. Fig. 6e–g shows the same area as Fig. 6d, imaged in weak-beam dark-field mode with \(g = 111\), \(g = 1-1-1\), and \(g = 2-20\), respectively. The operating \(g\) vectors of Fig. 6e–g are marked by the dashed and solid circles in Fig. 6b and the solid circle in Fig. 6c, respectively. The dominant parallel dislocation arrays are in good contrast at \(g = 111\) but out of contrast at \(g = 1-1-1\) and \(g = 2-20\), indicating that the dislocations in the arrays have a Burgers vector parallel to [110]. Based on the microstructure of the dislocations, which contains dissociated dislocations, as shown by the arrows in Fig. 6d, the tangled dislocations in the arrays are composed of \(\frac{1}{6}[110]\) stair-rods formed by reactions of partial dislocations [35].

The microstructural analysis indicates that slip took place in all three different indentation orientations near \{001\}, \{101\}, and \{111\}, while deformation twinning was activated only by indentations on near \{001\} grains. The operations of the slip and twinning mechanisms identified in this work are consistent with literature reports on fcc systems for deformation at low temperatures [18,27,32–34]. TEM micrographs suggest the formation of stair-rod dislocations, which can serve as obstacles to dislocation glide and thus influence the creep behavior of the material. In the

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**Fig. 5.** Bright-field STEM micrographs, imaged on [011] zone axis, of near (101) (a) and (111) (b) indented samples.

**Fig. 6.** (a) TEM micrographs showing the parallel dislocation arrays in one of the near <111> indented samples. (b–c) Electron diffraction patterns at the [01-1] and [112] zone axis, respectively. (d) Bright-field image obtained at the (111) two-beam condition schematically marked by the dashed circle in (b). (e–g) Weak-beam dark-field images of the same area shown in (d), with the operating \(g\) vectors marked by the dashed circle (111) and solid circle (1-1-1) in (b), and the solid circle (2-20) in (c), respectively.
following section, we will confirm the formation of stair-rod dislocations by MD simulation, which also correlates the dislocation structures with the resistance to nanoindentation creep. That discussion is followed by a discussion regarding the formation mechanisms of the stair-rod dislocations.

3.4. Modeling

MD simulations of nanoindentation were conducted on the (001), (011), and (111) surfaces of Fe-20Cr-25Ni at 300 K. The maximum indentation depth was set to 1.4 nm with the displacement rate controlled at 0.01 nm/ps. Fig. 7 shows the evolutions of the nanoindentation hardness and densities of three types of dislocations as a function of indentation depth. The densities of dislocations were calculated using the Dislocation Extraction Algorithm [43]. In Fig. 7a, the hardness is the contact pressure, defined as the ratio of the indentation force and the projected contact area. The fluctuation in hardness decreased as the indentation depth increased. The hardness values between the indentation depths of 0.7 and 1.4 nm were 18.18 ± 0.36 GPa, 16.06 ± 0.22 GPa, and 16.26 ± 0.31 GPa for (111), (011), and (001) indentations, respectively. Compared with the experimental values shown in Fig. 1, the hardness values from the MD simulations were almost one order of magnitude larger. The overestimation of hardness from MD simulations is generally observed in different fcc systems [37-39], primarily because of the higher strain rate and indentation size effect [37,38]. Nevertheless, the calculated highest hardness on the (111) surface and comparable hardness on the (011) and (001) surfaces are qualitatively in agreement with the experimental results in this study.

It is worth noting that the highest hardness was observed on the (111) surface of the Fe-20Cr-25Ni alloy in this study, whereas MD simulations of pure aluminum and nickel showed the highest hardness on the (001) surface and the lowest hardness on the (111) surface [3,37]. Ju et al. showed that when a material is deformed through the formation of dislocation defects, the ease of formation of which depends on the number of slip angles of the (111) planes [3]. In this study, the stacking fault energy of the alloy was much lower than those of pure aluminum and nickel [40]; therefore, dislocations tended to glide in the form of Shockley partials, as evidenced by the observations of dissociated dislocations in Fig. 6 and the high-density Shockley partials in the MD results in Fig. 7b. Because of the high mobility of Shockley partials, deformation, even on the scale of MD simulations, was shown to occur by means of the slip. Consequently, the nanoindentation hardness is related to the ease of activation of slip systems through the resolved shear stresses on the slip systems.

Fig. 7c and d plot the densities of Lomer-Cottrell (L-C, 1/6<110>) and Hirth 1/3<100> dislocations as functions of indentation depth, respectively. There is an obvious difference in the populations of L-C dislocations among the three indentation orientations, while the evolutions of Hirth dislocations in the three cases are similar. On the experimental scales, the formation of L-C dislocations was more significant, given that the formation of L-C dislocations was more energetically favorable than that of the Hirth dislocations [41]. In fact, 1/6<110>-type dislocations are much frequently observed in experiments than 1/3<100>-type dislocations, as shown in this and previous studies [42].

The observation of L-C dislocations from the MD simulation agrees with the TEM observations in Figs. 5 and 6. In addition, MD simulation also showed that the population of stair-rod dislocations was higher in (011) indented samples than in (111) indented samples. In fact, this difference was qualitatively observed in the STEM images of the indented samples, as the interspacing of the dislocation arrays is much smaller in Fig. 5a than in Fig. 5b. The networks formed with L-C locks were obstacles for the gliding dislocations. As shown earlier, dislocation gliding was the operating deformation mechanisms for (011) and (111) indented samples. It is believed that the difference in the population of the L-C dislocations was the primary reason for the different nanoindentation-creep resistances observed in the (011) versus (111) indentations shown in Fig. 2.

To understand the formation of the L-C dislocations, we reconsidered the activation of slip systems based on Schmid’s law under compression. The four active slip systems under indented (011) are (1-1-1)[110], (1-1-1)[101], (111)[-101], and (111)[-110]. They can react through the following L-C reactions to form 1/6[011] stair-rods:

![Fig. 7. MD simulation calculated evolutions of nanoindentation hardness (a) and densities of 1/6<112> (b), 1/6<110> (c), and 1/3<100> (d) dislocations as a function of the indentation depth exerted on the (001), (101), and (111) surfaces.](image-url)
shown in Fig. 8a, when the material is indented, the region right to the multi-axial stresses applied by the nanoindentation. As stress deviates from [111], for instance, with stress acting in the direction of [111] when the six active slip systems are either perpendicular or at angles of 60° from each other. As a result, no L-C reactions could happen among the six active slip systems. The active formation reactions of L-C locks due to the stress along the indentation direction in (110) samples explains the higher population of L-C locks in (110) indented samples.

On the other hand, the six active slip systems under indented (111) are (-1-1-1)[0-1-1], (-1-1-1)[10-1], (-11-1)[1-1-1], (-11-1)[0-1-1], (-11-1)[0-1-1], and (-11-1)[10-1]. The slip directions of the six systems are either perpendicular or at angles of 60° from each other. No L-C reaction is possible among these systems. Moreover, when observing a cross-sectional lamella patterns on indented (111), as observed in Fig. 3a and in previous studies [6].

Taking account of the three-fold symmetry of the atomic arrangement on the (111) plane, Fig. 8c schematically shows the distribution of L-C locks on the (111) indented sample looking along the indentation direction. This pattern agrees with the slip band patterns on indented (111), as observed in Fig. 3a and in previous studies [6]. Moreover, when observing a cross-sectional lamella lifted out along the dashed line in Fig. 8c, as the sample shown in Fig. 5b was prepared, one should see L-C locks skewed toward one side of the indent, which is confirmed by Fig. 5b.

It is thought that most of the dislocations and twins were induced by the plastic deformation during the nanoindentation loading period. The continuous deformation under the load-holding creep period relied on the continuous operation of the active slip or twinning systems. Comparing the cases of <111> and <011> indentations, the ease of L-C lock formation under <011> compression led to the stronger resistance to creep of [011] samples that is observed in Fig. 2. In the case of <001> indentation, the intersections of the twins could prevent the growth of twins under the held load. Together with twin-slip interactions, they suppressed the deformation of the material during the nanoindentation creep tests [33].

4. Conclusion

Systematic nanoindentation-based tests, together with microstructural characterization using SEM and TEM, were conducted on a model 709 alloy (Fe–20Cr–25Ni) at room temperature. Anisotropies in elastic modulus, hardness, and nanoindentation creep were investigated. A distinct grain-orientation dependency of the elastic modulus was observed for [001], [101] and (111) grains in ascending order in the range of 197–234 GPa. Similarly, (111) grains
exhibited the highest hardness, up to 2.21 GPa, but the hardness discrepancy between [011] and [001] grains was not significant.

During the indentation creep tests, the time-dependent displacement showed a similarity to general creep curves containing primary and secondary creep stages. Following the power-law equation, the indentation stress exponent (n) in the secondary stage was fitted to be from -18 to 34 for the near [111] grains. The stress exponent values for the grains near [111] were comparable to the stress exponent extrapolated from the literature creep data for alloy 800. Microstructural characterization revealed slip and twinning as the two deformation mechanisms of the alloy. Unlike slip, which was present underneath surfaces indented near [001], [011], and [111], twinning was observed only underneath the surfaces indented near [001]. This finding is explainable by the orientation-dependent Schmid factors for slip and twinning under [011] and [111].

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References