In-situ high-energy X-ray characterization of neutron irradiated HT-UPS stainless steel under tensile deformation

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Abstract

The tensile deformation behavior of a high-temperature, ultrafine-precipitate strengthened (HT-UPS) stainless steel was characterized in-situ with high-energy X-ray diffraction at 20 and 400 °C. The HT-UPS samples were neutron irradiated to 3 dpa at 400 °C. Significant irradiation hardening and ductility loss were observed at both temperatures. Lattice strain evolutions of the irradiated samples showed a strong linear response up to near the onset of the macroscopic yield, in contrast to the unirradiated HT-UPS which showed a pronounced non-linear behavior well below the macroscopic yield. While the room-temperature diffraction elastic moduli in the longitudinal direction increased after irradiation, the 400 °C moduli were similar before and after irradiation. The evolution of the (200) lattice strain parallel to the loading axis ($\varepsilon_{L}^{(200)}$) showed unique characteristics: in the plastic regime, the evolution of $\varepsilon_{L}^{(200)}$ after yield is temperature-dependent in the unirradiated specimens but temperature-independent in the irradiated specimens; and the value of $\varepsilon_{L}^{(200)}$ at the yield is an irradiation-sensitive, temperature-independent parameter. The evolution of $\varepsilon_{L}^{(200)}$ corresponds well with the dislocation density evolution, and is an effective probe of the deformation-induced long-range internal stresses in the HT-UPS steel.

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1. Introduction

The high-temperature, ultrafine-precipitate strengthened (HT-UPS) 14Cr-16Ni austenitic stainless steel is a candidate material for structural and cladding applications in advanced nuclear reactors due to its improved creep, irradiation and corrosion resistances [1–8]. The HT-UPS steel was designed by controlling various alloying elements to produce nanosized MC (M: Nb, Ti, V) carbide dispersions and prevent the formation of embrittling intermetallic phases for enhanced long-term creep resistance and extended creep rupture life. The HT-UPS steel has been studied for its physical properties and mechanical properties of a material. The effect of radiation on bulk elastic constants has been investigated in a number of materials [10–15]. However, no measurements of plane-specific elastic moduli (so-called diffraction elastic constants) of neutron-irradiated polycrystalline materials have been reported in the literature. The recent development of in situ high-energy X-ray characterization of radioactive specimens [16] has made it possible to measure diffraction elastic constants of neutron-irradiated specimens by X-ray diffraction during in situ tensile tests, which is of vital importance to the understanding of internal stresses in neutron-irradiated materials.

Irradiation-induced defects can also cause significant hardening and embrittlement in irradiated metals. While there is a relatively good understanding of irradiation hardening mechanisms [17–22], a number of issues are still unresolved regarding the deformation and failure mechanisms in irradiated materials. Irradiation-induced ductility loss or embrittlement is often linked with localized deformation and early onset of plastic instability in...
irradiated materials. In the deformed microstructures of irradiated metals with low stack fault energy (SFE) such as austenitic stainless steels, a combination of mechanical twins and dislocation channels is often observed, where dislocation glides are evenly distributed and well confined in the narrow bands [23–28]. The formation of dislocation channels observed in the irradiated materials may be explained by the interactions of moving dislocations with sessile defect clusters such as Frank loops in austenitic stainless steels, which are unfauluted and removed through the prismatic glide [29]. The unfaulting of Frank loops can occur through stress-induced nucleation of Shockley partial inside a Frank loop, or though coplanar or non-coplanar glide of extended dislocations over the Frank loops [30]. Twinning is an alternative localized deformation mode formed by massive but confined dislocation glides [26].

Enabled by high-brilliance synchrotron sources, the in-situ high-energy X-ray characterization is a powerful tool for studying the deformation characteristics of steels used in various energy systems [31–35]. The evolution of lattice strains in different phases of a bulk material can be simultaneously measured through monitoring the positions of diffraction peaks, from which the load partitioning can be analyzed [36]. The plastic flow characteristics, such as the evolution of dislocation density, can also be investigated through the line-broadening analysis [37].

In this study, the in-situ high-energy X-ray characterization techniques were used to understand the tensile deformation behavior of neutron-irradiated HT-UPS steel. Irradiated and unirradiated HT-UPS specimens were characterized with wide- and small-angle X-ray scattering techniques while under in-situ tensile testing at 20 and 400 °C, respectively. Significant differences were found between the irradiated and unirradiated specimens through the analyses of lattice strain evolution and peak broadening under deformation. The irradiated and deformation microstructures were also characterized by transmission electron microscopy (TEM).

2. Experimental

2.1. Material and samples

The chemical composition of the HT-UPS austenitic stainless steel is listed in Table 1. The material was in a sheet form, and with a final heat treatment of solution annealing at 1200 °C for 1 h. The average grain size was 150 ± 7 μm. Tensile specimens were electrical-discharge-machined after the heat treatment of the alloy. The specimens were subbed sheet-type tensile specimens with a nominal gauge length of 5 mm, a gauge width of 1.2 mm and a gauge thickness of 0.7–1 mm.

The tensile specimens were irradiated in the Advanced Test Reactor (ATR), Idaho National Laboratory. Irradiation of the tensile specimens was conducted in gas-filled capsules in a cadmium shrouded basket in the East Flux Trap (EFT) of the ATR. The maximum fast-neutron flux in the EFT was \( \sim 9.7 \times 10^{13} \text{n/cm}^2\text{s} \) (E > 1 MeV). The neutron flux was \( \sim 2.1 \times 10^{21} \text{n/cm}^2\text{s} \) (E > 1 MeV), corresponding to a damage of \( \sim 3 \) dpa. The nominal irradiation temperature was 400 °C with the estimate uncertainty of ±25 °C. A SiC temperature monitor was inserted in the capsule to measure the irradiation temperature.

<table>
<thead>
<tr>
<th>Fe</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>Ti</th>
<th>Nb</th>
<th>V</th>
<th>C</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal.</td>
<td>13.91</td>
<td>16.00</td>
<td>2.59</td>
<td>2.00</td>
<td>0.11</td>
<td>0.04</td>
<td>0.28</td>
<td>0.11</td>
<td>0.53</td>
<td>0.06</td>
<td>0.02</td>
</tr>
</tbody>
</table>

2.2. In-situ X-ray experiment

The in-situ high-energy X-ray experiments were performed at 1-ID-E endstation of the Advanced Photon Source (APS) at Argonne National Laboratory. The experimental setup is illustrated in Fig. 1 and described in detail in Ref. [16]. Tensile tests were performed on a MTS® model 858 servo-hydraulic load frame under uniaxial tension. The in-situ irradiated materials (iRadMat) characterization apparatus was used for heating, as well as radiation shielding and containment for the radioactive samples. Four in-situ tensile tests were carried out: one irradiated specimen and an unirradiated control specimen were tested at room temperature in air, herein denoted by \( \text{Ir}, \text{T}_\text{test}=20^\circ\text{C} \) and \( \text{Unir}, \text{T}_\text{test}=20^\circ\text{C} \), respectively. Another irradiated specimen and an unirradiated control specimen were tested at 400 °C in vacuum (\( 10^{-5}\text{Torr} / 1.3 \times 10^{-2} \text{Pa} \)), herein denoted as \( \text{Ir}, \text{T}_\text{test}=400^\circ\text{C} \) and \( \text{Unir}, \text{T}_\text{test}=400^\circ\text{C} \), respectively. The tensile tests were conducted at varying strain rates, ranging from \( 10^{-5}/\text{s} \) up to \( 5 \times 10^{-3}/\text{s} \). A low strain rate (\( 10^{-3}/\text{s} \)) is used for the relative short elastic deformation stage to increase the periods of data collection, and higher strain rates are used at the plastic deformation stages.

To test a radioactive sample, a sliding tube was installed surrounding the specimen grips to encapsulate the specimen. The tube was made of Kapton for the room temperature tests, and quartz for the 400 °C tests. The specimens were heated under vacuum with the built-in tungsten heating elements. Two K-type thermocouples were embedded inside the top and bottom specimen grips, with their tips in close proximity to the specimen. During the 400 °C tests, the sample temperature was maintained within ±20 °C of the target temperature.

Wide- and small-angle X-ray scattering (WAXS/SAXS) measurements were conducted simultaneously with monochromatic X-rays in transmission geometry while the sample was under tensile deformation. Only the WAXS results are presented in this paper. The X-ray energy was 123 keV (wavelength 0.0101 nm) and the beam size was 0.2 mm × 0.2 mm. The sample-to-detector distance was 3135 mm and the maximum 20 (θ is the diffraction angle) was 9°, covering up to the 9th peak of the austenite phase (222). The X-ray signals were recorded with four GE amorphous Si area detectors (409.6 mm × 409.6 mm active area with 2048 × 2048 pixels) arranged such that SAXS signals can be captured unhindered with a detector placed approximately 6 m from the sample [16] (as shown in Fig. 1). The beam direction, the tensile loading direction and the transverse direction are denoted by \( \text{Z}_1, \text{Y}_1 \) and \( \text{X}_1 \), respectively. The specimen is oriented with the gauge length direction along \( \text{Y}_1 \), the gauge width along \( \text{X}_1 \), and the gauge thickness along \( \text{Z}_1 \). The number of grains in the volume illuminated by a single beam was relatively small (≤50), due to the large grain size (about 150 μm). To increase the number of grains contributing to diffraction signals, a two-dimensional mapping scan over the sample gauge area was implemented. The specimens were translated along the \( \text{X}_1 \) (gauge width) direction while the diffraction data were continuously recorded using the so-called ‘fastsweep’ data collection mode. The specimen was also translated along the \( \text{Y}_1 \) (gauge length) direction at multiple positions with a step size of 0.25 mm, and a “fastsweep” measurement was taken at each \( \text{Y}_1 \) position. During data analysis, diffraction patterns acquired at four steps in the \( \text{Y}_1 \) direction were summed together and analyzed as one data point, which covers approximately 500 grains.

2.3. X-ray data analysis

The data reduction and peak fittings were performed with a MATLAB® script provided by the Materials Physics and Engineering group of the APS. Instrument parameters (beam center, detector...
tilts, and sample to detector distance) were calibrated using a CeO$_2$
standard sample (NIST-SRM$^\text{®}$ 674b) [38]. Line profiles were
generated by integrating the diffraction data for a specific range of
azimuthal angle. The peaks were fit individually using the pseudo-
Voigt function and a linear function as background. No precipitates
were identified from the line profiles. Lattice strains from five
austenite peaks ([200], [220], [311], [222], [331]) were measured
along the loading and transverse directions. The lattice strains ($\varepsilon_{hkl}$)
were calculated by:

$$\varepsilon_{hkl}(\sigma) = \frac{d_{hkl}(\sigma) - d_{hkl}(0)}{d_{hkl}(0)}$$

where $d_{hkl}(\sigma)$ is the lattice spacing when the sample is under stress
$\sigma$, and $d_{hkl}(0)$ is the reference lattice spacing measured without
tensile loading. A 20° azimuthal angular range spanning around the
longitudinal and transverse axis directions was selected for data
integration and peak fitting, and the calculated lattice strains are
named as longitudinal ($\varepsilon_{hkl}^L$) and transverse ($\varepsilon_{hkl}^T$) lattice strains,
respectively.

Peak broadening for a particular diffraction peak was computed
using equation:

$$\Delta K = \cos(\Delta 2\theta)/\lambda$$

where $\Delta K$ is the peak broadening and $K$ is defined as $K = 2\sin(\theta)/\lambda = 1/d$; $d$ is the plane spacing for the family of crystallographic planes
contributing to the peak, $\lambda$ is the wavelength of the X-ray, and $2\theta$
is the measured peak broadening. The peak broadening was not
expected to be a function of the stress state in different azimuthal
angles, and therefore, the entire diffraction pattern was utilized to
improve the data statistics. To do this, the whole pattern was
divided into multiple 5° azimuthal angular cakes for data integra-
tion and peak fittings, and the resulted full-width half-maximum
(FWHM) values were averaged and used for the peak broadening
analysis. The instrumental broadening was measured with a LaB$_6$
standard sample (NIST-SRM$^\text{®}$ 660a [39]), and was subtracted
following $B_{\text{sub}}^2 = B_{\text{meas}}^2 - B_{\text{instr}}^2$, where $B_{\text{sub}}$, $B_{\text{meas}}$, $B_{\text{instr}}$
are the subtracted, as-measured, and instrumental broadening values ($B =
\Delta K$), respectively. Peak broadening analysis was performed using
the modified Williamson–Hall (W–H) method [40–42] as:

$$\Delta K = a + \beta \cdot \tilde{K} + O(\tilde{K}^2)$$

Where $\tilde{K} = K/\zeta^{1/2}$, and $\zeta$ is the average contrast factor to account
for the strain anisotropy. $O$ stands for a higher order dependency on
the $\tilde{K}$ variable, and is neglected in the analysis, which results in a
linear relationship between the peak broadening $\Delta K$ and $\tilde{K}$. The
modified W–H plot shows $\Delta K$ values against $\tilde{K}$ for the evaluated
diffraction peaks. A linear regression was used for fitting the plot and
determining the intercept $a$ and slope $\beta$. The intercept $a$ can be
correlated with the average domain size, while the slope $\beta$ is related
to the dislocation density as: $\beta = \tilde{M} \left( \frac{1}{\rho} \right)^{2/3}$, where $\rho$ is the
magnitude of Burger’s vector, $\rho$ is the dislocation density, and $\tilde{M}$ is a
constant depending on the outer cut-off radius of the dislocations
and the dislocation density. The average contrast factors $\zeta$ were
obtained using a linear summation of contrast factors for pure edge
($\zeta_E$) and pure screw dislocations ($\zeta_S$), as expressed below:

$$\zeta = f_E \cdot \zeta_E + (1 - f_E) \cdot \zeta_S$$

where $f_E$ is the fraction of edge dislocations over the total disloca-
tion population. $\zeta_E$ and $\zeta_S$ were calculated using a web-based
program: ANIZC [43,44], with inputs of the following elastic con-
stants: $c_{11} = 232$ GPa, $c_{12} = 154$ GPa, $c_{44} = 118$ GPa [45]. Assump-
tions were made that edge dislocations have a Burger’s vector of
$b = a\langle 2<011\rangle$, and a slip plane of $\{111\}$, and screw dislocations have
a Burger’s vector of $b = a\langle 2<011\rangle$. The calculated $\zeta_E$, $\zeta_S$ values are
given in Table 2. The W-H plots changes with the $f_E$ values which is a function of the $f_E$ values. The $f_E$ values are thus determined as the values that yield the best linearity of the W-H plots.

2.4. TEM characterization

Transmission electron microscopy (TEM) was conducted to characterize the irradiation and deformation induced microstructural changes. The specimens were prepared from the irradiated specimen after being tensile tested at 20°C, using the focused ion beam (FIB) TEM specimen preparation method [46]. The FIB damage was minimized by applying a Pt protective layer before the trench milling, and by using a 5 kV (low kV) final thinning process. Three TEM specimens with the lamella plane perpendicular to the tensile loading direction were lifted from the undeformed (“as-irradiated”), uniformly deformed, and necking regions, respectively. TEM was carried out on a FEl® Tecnai F30 microscope operated at 300 kV. FIB and TEM experiments were conducted at the Microscopy and Characterization Suite of the Center for Advanced Energy Studies, Idaho Falls, ID.

3. Results

3.1. Macroscopic stress-strain curves

The engineering and true stress-strain curves are plotted in Fig. 2, and the tensile properties are given in Table 3. The true stress ($\sigma$) and strain ($\varepsilon$) were calculated for the uniform deformation up to the maximum load using equations:

$$\sigma = s (e + 1)$$
$$\varepsilon = \ln(e + 1)$$

where $s$ and $e$ are the engineering stress and strain, respectively. Irradiation caused significant increases in the yield and tensile strength, and reductions in the uniform and total elongations. The increases in the macroscopic yield (0.2% offset) strength ($\Delta \sigma_{YS}$) were 528 and 358 MPa for the irradiated specimens tested at 20°C and 400°C, respectively. The strain hardening rates were much lower for the irradiated specimens than those of the unirradiated specimens.

3.2. Lattice strain evolution during tensile deformation

The evolutions of lattice strain versus true stress in the longitudinal and transverse directions are shown in Fig. 3 for the four tested specimens. The macroscopic yield stresses are marked with dotted horizontal lines in each of the plots. For both unirradiated and irradiated specimens, the lattice strains evolve anisotropically. The [200] grains (referring to the grains with the [200] plane-normal nominally parallel to the longitudinal or transverse directions) have the highest lattice strains, and the lowest diffraction elastic modulus (softest). The [200] grains remain the softest in the plastic regime. In contrast to the unirradiated specimens which show an evident non-linear behavior well below the macroscopic yield, the irradiated specimens exhibit a strong linear response up to near the onset of the macroscopic yield. The [200] lattice strain deviates from the linearity at a much lower stress than those in other grain orientations in the unirradiated specimens. It implies that yielding for grains in the unirradiated specimens is more heterogeneous than in the irradiated specimens.

Table 2

<table>
<thead>
<tr>
<th>$g$</th>
<th>(111)(222)(333)</th>
<th>(200)(400)</th>
<th>(220)</th>
<th>(311)</th>
<th>(331)</th>
<th>(420)</th>
<th>(422)</th>
<th>(511)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\tau_E$</td>
<td>0.138</td>
<td>0.288</td>
<td>0.175</td>
<td>0.217</td>
<td>0.165</td>
<td>0.216</td>
<td>0.175</td>
<td>0.257</td>
</tr>
<tr>
<td>$\tau_s$</td>
<td>0.064</td>
<td>0.290</td>
<td>0.120</td>
<td>0.183</td>
<td>0.104</td>
<td>0.181</td>
<td>0.120</td>
<td>0.242</td>
</tr>
</tbody>
</table>

Fig. 2. Macroscopic stress-strain curves for the unirradiated and irradiated HT-UPS specimens tested at room temperature and 400°C: (a) engineering stress-strain curves, and (b) true stress-strain curves.
Diffraction elastic constants were evaluated through the linear least-square fitting of the true stress vs. lattice strain in the elastic stage, following: 

$$E_{hlk} = \epsilon_{hlk}$$

where $E_{hlk}$ and $\epsilon_{hlk}$ are the elastic moduli and lattice strains for the $\{hkl\}$ reflections, respectively. The moduli for the longitudinal ($E_{lh}$) direction are given in Table 4, and plotted in Fig. 4. Values for the 20 °C tests are generally higher than the 400 °C tests, due to the temperature effect [47]. At room-temperature, the moduli of the irradiated specimens are higher than those of the unirradiated specimens, and yet at 400 °C the moduli are similar between the irradiated and unirradiated specimens.

Above the macroscopic yield, the lattice strains continued to increase with increasing stress in the unirradiated specimens, as well as in the irradiated specimen tested at 400 °C, as shown in Fig. 2. However, in the irradiated specimen tested at room temperature, the lattice strain responses are more complicated. The {200} and {311} lattice strains showed an initial increase with increasing stress and then saturated, while the lattice strains in the other three reflections remained nearly constant when the stress increased.

The characteristics of the lattice strain evolution in the plastic regime are further elaborated in Fig. 5, where the lattice strains in the longitudinal direction are plotted against the true strains.

### Table 4

<table>
<thead>
<tr>
<th></th>
<th>Unirr, T$_{test}$=20 °C</th>
<th>Unirr, T$_{test}$=400 °C</th>
<th>Irr, T$_{test}$=20 °C</th>
<th>Irr, T$_{test}$=400 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>{200}</td>
<td>103 ± 5</td>
<td>71 ± 4</td>
<td>120 ± 1</td>
<td>77 ± 1</td>
</tr>
<tr>
<td>{220}</td>
<td>187 ± 17</td>
<td>143 ± 24</td>
<td>244 ± 3</td>
<td>125 ± 2</td>
</tr>
<tr>
<td>{311}</td>
<td>155 ± 5</td>
<td>115 ± 10</td>
<td>181 ± 2</td>
<td>121 ± 1</td>
</tr>
<tr>
<td>{222}</td>
<td>215 ± 14</td>
<td>163 ± 12</td>
<td>232 ± 3</td>
<td>163 ± 2</td>
</tr>
<tr>
<td>{331}</td>
<td>182 ± 8</td>
<td>167 ± 9</td>
<td>233 ± 6</td>
<td>131 ± 2</td>
</tr>
</tbody>
</table>

Overall, the evolutions of lattice strain during the plastic deformation are highly anisotropic. The {200} planes have the largest lattice strains with a peak value of $-8 \times 10^{-3}$, and the {311} planes have the second highest lattice strains, while the other three lattice strains ({220}, {222}, {331}) are much lower. This result agrees with the observations in other austenitic stainless steels [48]. For the two unirradiated specimens all the lattice strains increase with the true strains at the two temperatures. In the two irradiated specimens, only the {200} lattice strains show significantly increases in the plastic deformation stage at two test temperatures. The behaviors of the {220}, {222} and {331} lattice strains are similar, and all experience pronounced drops beyond yielding in the irradiated specimens.

### 3.3. Peak broadening and the modified Williamson-Hall analysis

The peak widths for five reflections are shown in Fig. 6 as a function of the true strain. All peaks show broadening from...
deformation, and the evolution of peak widths varies in different lattice planes. The peak widths at zero strain are much larger in the irradiated specimens than in the unirradiated specimens, which may be due to the irradiation induced defects in the irradiated specimens [49]. While the peak widths increase monotonically in the unirradiated specimens, the peak widths of the Irr, $T_{test}=20^\circ C$
specimen show three characteristic stages marked as “I”, “II”, and “III” in Fig. 6 (c). After the initial transient stage, the peak widths increase rapidly in stage II, and saturates in stage III. For the Irr, T\text{test}=400 ^\circ C specimen, the peak widths increase but the rate of increase decrease with increasing strain.

There are mainly two factors contributing to the X-ray peak broadenings besides the instrumental contribution: the broadening due to finite domain sizes (size broadening) and the broadening due to lattice distortions (strain broadening) [50]. The size and strain broadening contributions were separated using the modified W-H method. The goodness-of-fit for the linear fittings in the W-H plots, measured by the $R^2$ value, was above 0.9 (1 for perfect linearity) for all four specimens. The estimated W-H slope values are plotted against true strains up to the uniform elongation in Fig. 7. The W-H slopes of the two irradiated specimens are similar, and both show a rapid increase in the initial stage and level out at higher strains (>10%). The W-H slopes for the unirradiated specimens are significantly lower than those for the irradiated specimens. Both of them increase at a relatively steady rate. The Unirr, T\text{test}=400 ^\circ C specimen has a similar slope value in the initial deformation stage (<10%), but a higher value at higher strains compared to Unirr, T\text{test}=20 ^\circ C specimen.

Fig. 8 shows the changes of the W-H slope along the gauge length at different strain levels for the Irr, T\text{test}=400 ^\circ C specimen. The gauge was scanned continuously during the deformation. The WAXS data from each measurement along the gauge length were collected. The gauge was scanned continuously during the deformation. The goodness-of-fit for the linear fittings in the W-H plots, measured by the $R^2$ value, was above 0.9 (1 for perfect linearity) for all four specimens. The estimated W-H slope values are plotted against true strains up to the uniform elongation in Fig. 7. The W-H slopes of the two irradiated specimens are similar, and both show a rapid increase in the initial stage and level out at higher strains (>10%). The W-H slopes for the unirradiated specimens are significantly lower than those for the irradiated specimens. Both of them increase at a relatively steady rate. The Unirr, T\text{test}=400 ^\circ C specimen has a similar slope value in the initial deformation stage (<10%), but a higher value at higher strains compared to Unirr, T\text{test}=20 ^\circ C specimen.

3.4. TEM characterization of microstructure

The as-irradiated microstructure was characterized in the undeformed region of the Irr, T\text{test}=20 ^\circ C sample by TEM. Frank dislocation loops were the dominant defects, and were quantified using the rel-rod dark field imaging technique [51] (Fig. 9 (a)), and the loop size distribution is plotted in Fig. 9 (b). The average loop size and density are $8.5 \pm 3.9 \text{ nm}$ and $1.2 \times 10^{23}/\text{m}^3$, respectively. The foil thickness for the density calculation was measured with the convergent-beam electron-diffraction technique under a two-beam diffraction condition.

The irradiated-and-deformed microstructure was characterized in the “uniformly-deformed” and “necking” regions of the Irr, T\text{test}=20 ^\circ C sample, respectively. Deformation twins were observed in both regions, as shown in Fig. 10. The twins are less dense in the “uniformly-deformed” region than in the “necking” region. A high density of Frank loops was observed between deformation twins.

4. Discussion

4.1. Irradiation hardening

Significant irradiation hardening (an increase in yield stress) was observed in the HT-UPS specimens, as shown in Fig. 2 and Table 3. Frank loops were the dominant defects in the as-irradiated specimens. The irradiation hardening of the HT-UPS specimens was evaluated using the dispersed barrier hardening (DBH) model [19,20,33]:

$$\Delta \sigma_{YS} = M \sigma b \sqrt{N d}$$

where $\Delta \sigma_{YS}$ is the increase in yield stress, $M$ is the average Taylor factor (3.06 for austenite [52]), $\sigma$ is the shear strength, $b$ is the Burger’s vector ($b = a/2<011>$), and $N$ and $d$ are the number density and mean size of the barrier (Frank loops), respectively. The average barrier strength of the dislocation loops, $\sigma$, was estimated using the measured $\Delta \sigma_{YS}$ (Table 3) and loop size ($d$) and number density ($N$) (Fig. 9). The barrier strengths are 0.28 and 0.19 for the 20 and 400 °C tests, respectively, which are similar to the literature values for neutron-irradiated austenitic stainless steels [19].

4.2. Effect of neutron irradiation on diffraction elastic moduli

The physical properties of a material can be drastically changed by a large population of irradiation-induced defects. Among the physical properties, elastic modulus or Young’s modulus is one of the key parameters critical for theoretical and modeling work concerning crystal plasticity and deformation. Our in-situ
experiment revealed for the first time, the effect of neutron irradiation on the plane-specific elastic constants in a polycrystalline material. As shown in Table 4 and Fig. 4, the diffraction elastic constants of the HT-UPS were slightly increased after irradiation for all reflections examined at room temperature, while at 400 °C the moduli were similar between the irradiated and unirradiated specimens. The measured diffraction elastic moduli for the unirradiated HT-UPS are smaller than the 316 stainless-steel (SS) reported in the literature [45], but both steels show the same plane and temperature dependence.

The effect of irradiation on elastic constants has been studied for a number of materials both experimentally and theoretically [10–14]. The experimental studies showed a complex dependence of elastic moduli on the type of irradiation (e.g. neutron, electron, and proton), irradiation temperature, and the nature of radiation defects. Straalsund and Day [10] measured the bulk elastic constants for Type 304 SS neutron-irradiated at 370–455 °C, and found a significant decrease in the elastic moduli with increasing

![Image](image1)

**Fig. 9.** (a) Rel-rod dark field image of the dislocation loops. The diffraction pattern is shown in the inset. (b) Size distribution of the Frank loops.

![Image](image2)

**Fig. 10.** TEM micrographs showing deformed microstructure observed in the Irr. *T*<sub>test</sub> = 20 °C sample: (a) and (b) the “uniformly-deformed” region and (c) the “necking” region. (b) is the bright field image taken with g(200) near the (011) zone axis, as indicated by the inset diffraction pattern.
irradiation dose. The changes in modulus showed a linear dependence of void swelling. A similar finding was reported by Shcherbakov et al. [11] where a decrease of the Young’s modulus with increasing irradiation dose was observed in a Fe-18Cr-9Ni steel neutron irradiated at 370–375°C to 1.5–21 dpa. Again, the decrease in Young’s modulus showed a good correlation with void swelling. However, in our study, the dominant defects observed under TEM were faulted dislocation loops rather than voids reported in the literature [10,11], and the diffraction moduli increased after irradiation. Therefore, the effects of neutron irradiation on the moduli in the irradiated HT-UPS specimens must be understood by different types of defects developed under irradiation.

The changes in elastic modulus under irradiation have been explained by a bulk effect, and/or a pinning effect. In close-packed metallic materials such as face-centered cubic (fcc) austenitic stainless steels, the presence of interstitials shortens the interaction distance and increases the elastic moduli, while the creation of vacancies decreases the modulus because of a lattice collapse. The influence of interstitials on the elastic modulus is expected to be more pronounced than that of vacancies. The theoretical calculations by Dienes [14] showed that vacancies alone decrease the elastic modulus by one order of magnitude corresponding to the bulk effect, while the presence of interstitials increases the elastic constants several orders of magnitude larger than the bulk effect. In contrast to the bulk effects of interstitials and vacancies on the elastic modulus, the dislocation theory suggests that the elastic moduli can be increased due to a pinning process of dislocations by interstitials and vacancies, and this effect can overshadow the direct effect of point defects [12]. A recent study by Hofmann et al. [15] reported lattice swelling (an increase in lattice parameter) and modulus changes in a He-implanted W-1%Re alloy due to the presence of small He-vacancy clusters and SIAs. The macroscopic lattice swelling was related to the average defect concentrations and their relaxation volumes. The neutron-irradiated HT-UPS specimens examined in this work showed a decrease in lattice parameter after irradiation with the average lattice parameter of 3.5995 Å for the unirradiated specimen and 3.5978 Å for the irradiated specimen. The lattice contraction was also observed in the HT-UPS neutron irradiated to 3 dpa at 500°C [49]. The lattice contraction in the irradiated HT-UPS specimens may be explained by the negative relaxation volumes of submicroscopic vacancy clusters. However, the increase in the room-temperature distortion moduli cannot be attributed solely to small vacancy clusters in the irradiated HT-UPS. The fact that a smaller lattice parameter was measured for the irradiated HT-UPS than that of the unirradiated HT-UPS at 400°C, while the distortion moduli were comparable between the unirradiated and irradiated HT-UPS specimens implies both the Schottky and Frenkel effects suggested by Hofmann et al. [15]. Stralsund and Day [10] suggested that irradiation-induced faulted loops made no major contribution to modulus changes. The theoretical study conducted by Panyukov and Rabin [13] also showed that dislocation loops have a relatively minor effect on the elastic moduli of a solid. The relative contributions of various types of defects in the irradiated HT-UPS specimens (dislocation loops, submicroscopic vacancy and interstitial clusters) to the lattice parameter and distortion moduli remain to be elucidated.

4.3. Effect of neutron irradiation on deformation behavior

The evolution of the lattice strain revealed the load partitioning and micro-plasticity of different grain orientations. In the plastic regime, the development of lattice strain deviates from the elastic response, accumulating load at a significantly lower rate and causing changes in load partitioning among different grain orientations. Among the five reflections examined, the (200) reflection is the softest orientation in austenitic stainless steels [53]. As shown in Fig. 11, several characteristics were observed in the development of the (200) lattice strain under tensile deformation: (1) the (200) lattice strain parallel to the loading axis ($\varepsilon_{(200)}^f$) at the yield ($\varepsilon_{(200)}^{yield}$) (which is defined as the first inflection point of the lattice strain–true strain curve) showed no temperature dependence at test temperatures of 20 and 400°C, in either unirradiated or irradiated HT-UPS specimens. Neutron irradiation significantly increased the $\varepsilon_{(200)}^f$, which implies that the $\varepsilon_{(200)}^{yield}$ is a temperature-independent, irradiation-sensitive parameter; (2) the evolution of $\varepsilon_{(200)}^f$ with strain after yield shows a strong temperature dependence in the unirradiated HT-UPS specimens, while no temperature dependence of $\varepsilon_{(200)}^f$ was observed in the irradiated HT-UPS specimens. It is interesting to note that while $\varepsilon_{(200)}^f$ is higher at 400°C than at 20°C in the unirradiated HT-UPS in the entire plastic regime, but the development of $\varepsilon_{(200)}^f$ follows the same trend after the true strain reaches ~24% (i.e., minimal temperature dependence in the later stage of plastic deformation).

The $\varepsilon_{(200)}^f$ evolution with strain in the plastic regime was in coincidence with the evolution of the W–H slopes, as shown in Fig. 7. The W–H slopes are correlated with the microstrains or lattice distortions caused by the plastic flow, and are proportional to the dislocation densities inside the material [51,52,54]. The dislocation activities under tensile deformation are thus reflected by the W–H slope evolution. The evolution of the W–H slope with true strain in the irradiated specimens was nearly indistinguishable when tested at 20 and 400°C, while the W–H slopes were higher at 400°C than at 20°C in the unirradiated HT-UPS specimens after the initial plastic stage (~7%). Consistent with the $\varepsilon_{(200)}^f$ evolution with strain in the irradiated specimens, the evolution of the W–H slope followed the same trend after ~24% true strain at the two test temperatures. This suggests that the evolution of both the $\varepsilon_{(200)}^f$ and the dislocation density have no temperature dependence in the irradiated specimens or in the later stage of plastic deformation in the unirradiated specimens.

The macroscopic stress–strain curves of the HT-UPS steel exhibit a different temperature and irradiation dependence from the lattice strain–macroscopic stress curves. As shown in Fig. 2 (b), the 20 and 400°C true stress–true strain curves can be superimposed by shifting the y (true stress) axis for both the unirradiated and irradiated HT-UPS. It implies that the macroscopic yield stress of the HT-UPS is sensitive to both irradiation and temperature, while the work hardening behavior is largely temperature-independent.

![Fig. 11. $\varepsilon_{(200)}^f$ plotted against true strain for the four tested specimens.](image-url)
but affected by irradiation, and irradiation significantly reduces the work hardening rate. According to Schoeck and Seeger [55], the macroscopic flow stress of a crystalline solid can be separated into two components: a thermal component, \( \sigma^* \), and an athermal component, \( \sigma_u \), i.e.,

\[
\sigma = \sigma^*(T, \varepsilon) + \sigma_u
\]

\( \sigma^* \) is dependent on temperature \( T \) and strain rate \( \varepsilon \), and associated with short-range obstacles. \( \sigma_u \) is associated with the long-range internal stress insensitive to temperature and strain rate. For the HT-UPS steel, the differences in thermal stresses estimated from the true stress—true strain curves were 80 MPa and 280 MPa for the unirradiated and irradiated specimens, respectively. The increase in thermal stress in the irradiated HT-UPS steel may be attributed to the thermally activated cutting of the moving dislocations through small obstacles, e.g., small radiation defect clusters, defect-solute complexes in the irradiated specimens [56].

The athermal component of the macroscopic flow stress in pure fcc materials arises from the elastic interactions of dislocations, and is often related to the dislocation density in the following form [57]:

\[
\sigma_u = \alpha \rho \sqrt{\rho}
\]

where \( \rho \) is the mean dislocation density, and \( \alpha \) is the constant. The spatial inhomogeneities of dislocation densities give rise to spatially varied internal stress field, which leads to spatial variations of the flow stress. To take into account of the heterogeneity of the dislocation distributions, Ungar and Mughribi et al. [58,59] described the local athermal stress, \( \sigma_{loc} \), and local dislocation densities, \( \rho_{loc} \), in the following equation:

\[
\sigma_{loc}(x) = \alpha_{loc} \mu \sqrt{\rho_{loc}} + \sigma_{1loc}(x)
\]

where \( \sigma_{loc} \) is the local internal stress. Because of the simple glide geometry and multiple slip of the [001]-oriented crystals, the [200] reflection is ideal for detecting the long-range internal stresses in fcc materials [58]. Ungar and Mughribi et al. measured the local internal stresses in the dislocation cell walls and the cell interiors in tensile-deformed [001]-oriented copper single crystals by the changes in the lattice spacing of [200] reflections. This measurement was able to reveal the deformation-induced long-range internal stresses resulted from the plastic strain mismatch between the “soft” (cell interior) and “hard” (cell wall) regions.

The long-range internal stresses have played an important role in the theory of work hardening, particularly in Stage II work hardening in fcc metals. Dislocation pile-ups, dislocation bundles and walls, and dislocation cells, dislocation glide through random obstacle array, etc. are considered as the microscopic origin of the long-range internal stresses [60]. The long-range internal stresses may increase from straining, precipitation, irradiation, etc. Because [001] crystals deformed under tension favor slip and not twinning in austenitic stainless steels [61], the evolution of the \( \dot{\epsilon}_{[200]} \) under tensile deformation can be used as the direct probe of internal stresses resulted from dislocation slip activities and to reveal the correlation between the local flow stress, long-range internal stress, and dislocation density in the HT-UPS steel.

The findings that the evolution of the \( \dot{\epsilon}_{[200]} \) and the W-H slope showed no temperature dependence in the irradiated HT-UPS specimens while was temperature-dependent in the unirradiated specimens may be understood by the effects of temperature on deformation modes and the interactions of radiation defects and dislocations. It is known that the deformation mode of austenitic stainless steels changes from mechanical twinning and planar slip at low temperatures to the cross-slip at higher temperatures [62,63]. As reported by Byun et al., a high density of thin twins was formed during early deformation at \( -150 \) °C in annealed 316LN, and a mixture of twins and dense tangled dislocations became evident with increasing strains to accommodate a large bulk strain. As the test temperature increased to 200 °C, dislocation tangles were the dominant microstructure under tensile deformation. These microstructural observations are consistent with the measurements of the (200) lattice strain and dislocation density evolution in the unirradiated HT-UPS specimens.

In contrast to the unirradiated HT-UPS, the evolution of the \( \dot{\epsilon}_{[200]} \) and the W-H slope followed the same paths with increasing macroscopic strain in the irradiated HT-UPS specimens tested at 20 and 400 °C. As shown in Fig. 9, Frank loops (with the mean size of 8.5 nm) were the dominant defects in the irradiated HT-UPS. The loop pinning of dislocations can contribute significantly to the long-range internal stresses as observed in irradiated copper by Essmann [64,65]. The rapid increase in the W-H slopes (dislocation density) in the early stage of deformation (<5% macroscopic strain) in the irradiated HT-UPS specimens is suggested to result from the passing of gliding dislocations through a high density of Frank loops that significantly enhances the multiplication of dislocations, and this process is largely athermal. Once the dislocation density reaches a high enough value, further multiplications of dislocations becomes more difficult due to the higher long-range strain-fields exerted by the dislocations, which is reflected by the slowing down of the W-H slopes at the later deformation stages. It is also possible that highly localized strain in dislocation channels made the radiation-defects transparent to the passage of glide dislocations, slowing down the dislocation multiplication process [23].

The evolutions of longitudinal lattice strains of other reflections showed different characteristics in the irradiated specimens. The (220), (222) and (331) lattice strains experienced pronounced drops after the yield. The internal stresses developed in these crystal orientations may make a negative contribution to the flow stress, in contrast to a positive effect on the flow stress of the (200) lattice strain. Though local stresses vary spatially and crystallographically, its spatial average should be equivalent to the external stress.

4.4. Plastic instability and necking

The modified W-H analysis has also shed a light on the initiation and development of necking. As seen in Fig. 8, the W-H slope peak position corresponded well with the fracture position for the Irr, \( T_{test}=400 \) °C specimen. The initiation of the W-H slope peak along the gauge represents the initiation of necking, when the sample deformation transfers from a uniform deformation along the whole gauge length to a localized deformation. The engineering strain of the initiation of the W-H slope peak was \(-15\%\), consistent with the uniform elongation determined from the macroscopic stress–strain curve, which was 15% for the Irr, \( T_{test}=400 \) °C specimen. After necking, the W-H slopes increased rapidly at the peak position, while the increases were minimal at other gauge positions. The results show that the strain hardening mechanism still operates in the necking region. Plastic flow is localized to create a large enough strain hardening in response to the increase in true stresses due to reductions in gauge cross-sections, which is reflected as the rise of the W-H slope peak. The sample fractures when the hardening rate in the “necking” area can no longer match the increase rate of true stresses.

5. Conclusions

Neutron irradiated (3 dpa/400 °C) and unirradiated HT-UPS steel samples were tensile tested at 20 and 400 °C, and in-situ
characterized by high-energy X-ray diffraction. It was found that:

1. The macroscopic stress-strain curves show significant irradiation hardening and ductility loss. Irradiation hardening was mainly caused by irradiation induced Frank loops and their barrier strengths were estimated using the dispersed barrier hardening model at the two temperatures.

2. The irradiated specimens exhibited a strong linear response of lattice strain evolution up to near the onset of the macroscopic yield, in contrast to the unirradiated specimens which showed an evident non-linear behavior well below the macroscopic yield. The room-temperature deformation elastic moduli in the longitudinal direction were increased after irradiation, however, the 400 °C differ-744-749. https://doi.org/10.1007/s11837-019-02422-3.

3. The lattice strain evolutions were anisotropic for all tested specimens. The evolution of the 201 lattice strain parallel to the loading axis (ε∥) showed unique characteristics: ε∥ was the micro-yield is an irradiation-sensitive, temperature-inde-347, https://doi.org/10.1126/science.1137771.

4. The evolution of the W-H slopes with strain in the irradiated specimens were nearly indistinguishable between tests at 20 and 400 °C. In contrast, the W-H slopes were higher at 400 °C than at 20 °C in the unirradiated specimens.

5. The evolution of ε∥ matches well with the W-H slope (dislocation density) evolution with macroscopic strain in both unirradiated and irradiated specimens tested at 20 and 400 °C. The evolution of ε∥ and the W-H slope revealed the long-range internal stresses developed during tensile deformation.

6. The irradiated specimens followed similar work hardening paths as the unirradiated specimens macroscopically. After necking, the microstrain increased rapidly in the necking region in the irradiated HT-UPS.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.actamat.2018.07.008.

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