Microstructure and mechanical behavior of neutron irradiated ultrafine grained ferritic steel

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ABSTRACT

Neutron irradiation effects on ultra-fine grain (UFG) low carbon steel prepared by equal channel angular pressing (ECAP) have been examined. Counterpart samples with conventional grain (CG) sizes have been irradiated alongside with the UFG ones for comparison. Samples were irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) to 1.37 dpa. Atom probe tomography revealed manganese and silicon-enriched clusters in both UFG and CG steel after neutron irradiation. Mechanical properties were characterized using microhardness and tensile tests, and irradiation of UFG carbon steel revealed minute radiation effects in contrast to the distinct radiation hardening and reduction of ductility in its CG counterpart. After irradiation, micro hardness indicated increases of around 9% for UFG versus 62% for CG steel. Similarly, tensile strength revealed increases of 8% and 94% respectively for UFG and CG steels while corresponding decreases in ductility were 56% versus 82%. X-ray quantitative analysis showed that dislocation density in CG increased after irradiation while no significant change was observed in UFG steel, revealing better radiation tolerance. Quantitative correlations between experimental results and modeling were demonstrated based on irradiation induced precipitate strengthening and dislocation forest hardening mechanisms.

1. Introduction

Advanced nuclear reactors employ higher temperature environments and require higher irradiation exposures for core structural components and fuel cladding compared to the current operating light water reactors (LWR) [1,2]. Thus, development of new materials with superior properties which can withstand such severe conditions is required to fill these needs [3–5]. Ferritic steels have been used widely as structural materials in light water reactors, but they suffer from irradiation hardening and embrittlement accompanied by increased ductile to brittle transition temperature (DBTT) and decreased upper shelf energy [6]. Previous studies have shown that major factors affecting low carbon steels under neutron irradiation are neutron fluence, irradiation temperature and chemical composition [7]. However, designing materials with tailored response that can sustain high amounts of radiation damage while maintaining their mechanical properties is a grand challenge in materials research [8]. During irradiation, point defects (vacancies and interstitials) are produced as a result of displacement cascade [9–11]. These point defects can cluster to form other types of defects that will alter the mechanical properties of irradiated materials. One method to suppress accumulation of these point defects is by annihilating them at interfaces such as grain boundaries. Ultra-fine grained materials are expected to be more radiation tolerant since grain refinement increases the area of grain boundaries which can act as sinks for radiation induced point defects.

It has been theorized that the large amount of grain boundary area will help to prevent accumulation of defects which can adversely affect mechanical properties [12–20]. Singh [13] showed that void swelling in electron irradiated helium doped stainless steel decreases as the grain size decreases. Rose et al. [21] illustrated using TEM that the defect density in ZrO2 irradiated by Kr ions reduces as the grain size decreases. Matsouka et al. [22] studied the effects of neutron irradiation on UFG SUS316L stainless steel and their TEM observations revealed defect-free zones along grain boundaries suggesting that the grain boundaries are acting as sinks for radiation induced defects. Kurishita et al. [23] showed that the density of voids in UFG W-0.5 wt %TiC is much lower than that in CG tungsten after neutron irradiation at 600 °C. Sun et al. [24] found that dislocation loops and He bubble densities in UFG Fe–Cr–Ni alloy after He ion irradiation are less than...
those in its CG counterpart. Computer simulation studies [14,25–27] demonstrated that materials with large surface area of interfaces or grain boundaries have a potential to increase irradiation resistance. The effect of a small grain size (large grain boundary area density) on radiation tolerance of a low-carbon ferritic steel is assessed in this study.

2. Materials and methods

2.1. Materials

Two material cases are considered in this study; ultra-fine grain low carbon steel with a composition of 0.1 C, 0.5 Mn, 0.27 Si with balance of Fe in wt% processed through ECAP [28–30] and their conventional coarse grain counterparts that were produced by annealing the UFG material at 800 °C for one hour [12]. UFG steel was made by ECAP using Bc route [31] with four passes where the material is rotated 90° in the same direction after each pass. This processing route ensures eventual restoration of the material cubic element [32]. More details about ECAP routes can be found elsewhere [33,34].

2.2. Irradiation experiment

The irradiation experiment was carried out in the E-7 position of the East Flux Trap (Fig. 1b [35]) in the Advanced Test Reactor (ATR) at Idaho National Laboratory. The materials were irradiated to a neutron fluence of $1.78 \times 10^{23}$ neutrons/m² ($E > 0.1$ MeV) corresponding to a dose of 1.37 dpa. Fig. 1a is a schematic of the irradiation test assembly consisting of the experimental basket, support rod and capsule assemblies. The support rod was inserted at the bottom of the experimental basket to ensure that the test capsules are at the maximum flux location. The experimental basket of the test assembly is an aluminum tube designed for insertion in the capsule assembly in the ATR. The basket was designed such that there is adequate coolant circulation to prevent temperature distortions or mechanical effects, and that there is adequate mechanical support to secure the test capsule throughout the reactor insertion, irradiation, and removal from the reactor. Samples were cut from the bulk ECAP material and were prepared by grinding with a series of silicon carbide papers (600, 800 and 1200 grits) to optical flatness and then polished in colloidal silica resulting in deformation free surfaces. The prepared samples (UFG and CG steels) were loaded in sample holders (Fig. 2a) and a thin aluminum disc was tack-welded to the open end of each holder to position the samples inside it. Each group of sample holders was strung together using aluminum rods and was assembled into a sample train which was designed to hold the samples for easier removal following irradiation and also to help keeping the samples at the desired irradiation temperature (Fig. 2b, c and d). Finally, the sample trains were sealed in a stainless steel containment capsule that is essentially a sealed pressure vessel filled with helium (Fig. 2e). The integrity of the capsules was ensured using helium leak testing, dye penetrant testing and visual inspection.

Thermal analysis was performed using a detailed finite element model of the experiment using ABAQUS code [36]. MCNP code [37] was used to calculate the heat generation rate for each part of the experiment which was then used as an input to the finite element model (Fig. 3). The specimen temperature during irradiation was found to vary between 70 °C to less than and 100 °C depending on the sample position in the irradiation capsule. The low irradiation temperature is mainly due to the small helium gas gaps between specimens and holder, and between holder and capsule.
2.3. Post irradiation examination

The grain size distributions for UFG steel were measured using transmission electron microscopy (TEM) while electron back scattered diffraction (EBSD) technique was employed for CG steel; different techniques were used due to the differences in the mean grain sizes and an average of about 400 grains was used to estimate the average grain size. TEM studies were performed using a Tecnai TF30-FEG scanning transmission electron microscope operating at 300 keV. The EBSD measurements were conducted on a Quanta 3D FEG scanning electron microscope, and the crystallographic orientation mappings were made for the CG steel using a step size of 0.5 μm in an area of 100 μm x 100 μm. In addition, the microstructures of the low carbon steel for both grain sizes (CG and UFG) were characterized via X-ray diffraction (XRD) and atom probe tomography (APT) before and after irradiation. XRD was performed using PANalytical diffractometer with CuKα radiation (wavelength 0.15406 nm). All the diffraction profiles were obtained by varying 2θ from 40° to 120° with a scan step of 0.026° and the samples were rotated at 4 s/rev. APT was conducted using CAMECA LEAP 4000X HR instrument. The analyses were made using voltage pulsing (20% pulse fraction) at a specimen temperature of 50 K. The atom maps were analyzed employing IVAS v3.6.6 (CAMECA Instrument) software. The APT specimens were needle shaped with an apex radius of ~30 nm using focused ion beam (FIB), which was carried out using a Quanta 3D field emission gun (FEG) with a Ga ion source. Due to the irradiation embrittlement, some atom probe specimens fractured under the high stresses applied during the test. Thus, six tips were prepared from each sample and 3 tips from each sample were analyzed to maintain good statistics.

Irradiation induced hardening of the steel samples was quantified using both microhardness and tensile tests. Micro hardness measurements were carried out using Leco LM247AT Vickers micro hardness tester at a load of 500 g·f (~5 N). Twelve indents were made on each sample with a dwell time of 13 s and their average value is reported. The indents were 300 μm apart to avoid any influence from the previous indents on the hardness value. Tensile tests were performed on the unirradiated and irradiated samples using a closed loop Instron 5967 machine with 5 kN load cell. The tensile tests were carried out at a cross-head speed of 2 x 10^{-3} mm/s (strain rate of 1 x 10^{-3} s^{-1}) and three tests were performed for each condition. In order to minimize induced radioactivity, relatively small tensile specimens (2 mm gage length) were used in this study, and special tensile grips were designed for testing the miniature tensile samples (Fig. 4).

3. Results

Fig. 5 shows the microstructures of both UFG and CG steels before irradiation and the mean grain sizes for the UFG and CG steels are 0.35 ± 0.18 and 4.4 ± 1.8 μm, respectively. The XRD peaks (Fig. 6) show that there are no phase changes due to irradiation in both steels. All the X-ray diffraction peaks were fitted simultaneously using Modified Rietveld technique with suitable weightage by a pseudo-Voigt (pV) function using LS1 program [38]. This program uses the Modified Rietveld technique in its analysis and includes the simultaneous refinement of the crystal structure and the microstructural parameters like the domain size and the microstrain within the domain. The method involves Fourier analysis of the broadened peaks. Considering an isotropic model, the lattice parameters (a), surface weighted average domain size ($D_a$) and the average microstrain ($\epsilon_r^{1/2}$) were used simultaneously as the fitting parameters to obtain the best fit [39]. Fig. 7 represents a typical whole pattern fit using LS1 program for irradiated CG steel. Using the results obtained from the XRD analysis, the dislocation density, $\rho$, has been estimated from the following relations [40],

$$\rho = (\rho_D \rho_S)^{1/2}$$

$$\rho_D = \frac{3}{2} \frac{\rho_s}{k}$$

$$\rho_s = k(\epsilon_r^{1/2})/b^2$$

where $\rho_D$ and $\rho_S$ are the dislocation densities due to domain size and strain respectively, k is material constant equal to 14.4 for bcc metals [40]. X-ray analysis results are summarized in Table 1.

The reconstructed three dimensional atom maps of Si, Mn and C are shown in Figs. 8 and 9 for UFG and CG steels, respectively. In UFG steel, the grain boundaries were observed in both unirradiated and irradiated samples by solute enrichment that can only be attributed to segregation at grain boundaries. In CG steel, the
average total length of the matter analyzed is about 150 nm. Since the mean grain size for CG steel is about 4.4 μm, no grain boundary was observed in these volumes.

Solute atom analysis was performed using statistical methods to detect deviations from a random solid solution. A maximum separation method was used to identify the clusters in the examined materials [41]. This process defines a cluster based on a concept of nearest neighbor analysis. The solute atoms are identified as precipitate solute atoms (not solute atoms in a random solid solution) if they are within a maximum separation distance, $D_{\text{max}}$, from one another. Minimum size of clusters in terms of solute atoms that constitutes a significant cluster ($N_{\text{min}}$) is used to eliminate clusters of atoms that are not delineated as precipitates, so that any cluster with less number of solute atoms will not be considered. In order to include non-solute atoms in the defined cluster, all the matrix atoms within a distance ($L$) from the solute atoms are taken as being part of the same cluster. However, this process results in a shell of matrix atoms being included around each cluster. Thus, an erosion distance ($E$) is used to remove the shell of matrix atoms that lies within a distance less than $E$ from the nearest atom not defined as being part of the cluster [41–43].

Based on the deviation of the solute distribution in the tested specimens from random distribution, the maximum separation...
between solute atoms \( (D_{\text{max}}) \) was chosen to be 0.6–0.7 nm. Both the maximum separation of additional elements \( (L) \) and the erosion distance for removal of atoms near the cluster matrix interface \( (E) \) are equal to \( D_{\text{max}} \). The minimum size of the cluster in terms of solute atoms that constitutes a significant cluster \( (N_{\text{min}}) \) is taken as 11 atoms. APT characterizations of the irradiated steel revealed formation of Mn–Si-enriched nanoclusters (Fig. 10). However, the matrices of un-irradiated UFG and CG steel are found to be solid solutions with no solute clustering (the boxes in Fig. 10.

### Table 1

Values of domain size \( (D_s) \), microstrain \( (\langle \epsilon^2 \rangle)^{1/2} \) and dislocation density \( (\rho) \) for different samples obtained by Modified Rietveld analysis.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( D_s ) (Å)</th>
<th>( (\langle \epsilon^2 \rangle)^{1/2} )</th>
<th>( \rho ) (m(^{-2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unir CG</td>
<td>1850</td>
<td>7.40 ( \times ) 10(^{-4})</td>
<td>1.06 (± 0.13) ( \times ) 10(^{13})</td>
</tr>
<tr>
<td>Irr CG</td>
<td>622</td>
<td>1.00 ( \times ) 10(^{-3})</td>
<td>4.26 (± 0.56) ( \times ) 10(^{13})</td>
</tr>
<tr>
<td>Unir UFG</td>
<td>385</td>
<td>1.38 ( \times ) 10(^{-3})</td>
<td>9.50 (± 1.24) ( \times ) 10(^{14})</td>
</tr>
<tr>
<td>Irr UFG</td>
<td>366</td>
<td>1.24 ( \times ) 10(^{-3})</td>
<td>8.98 (± 1.39) ( \times ) 10(^{14})</td>
</tr>
</tbody>
</table>

**Fig. 6.** XRD profiles for unirradiated and irradiated (1.37 dpa) CG (top) and UFG (bottom) steels.

**Fig. 7.** Whole XRD pattern fit for irradiated CG steel at dose 1.37 dpa using LS1 program.
were empty). The fact that these small clusters were not observed before irradiation confirms that their formation was radiation-induced. The cluster number density is estimated using:

\[ N_v = \frac{n_p \xi}{nV} \]  

(2)

where \( n_p \) and \( n \) are the number of clusters in the analyzed volume and the total number of atoms in the same volume, respectively. \( V \) is the atomic volume and \( \xi \) is the detection efficiency which is \( \sim 37\% \) for local electrode atom probe (LEAP) instruments [43].

The cluster size is estimated by finding the Guinier radius for spherical precipitates using [44,45]:

\[ r_G = \sqrt{\frac{5}{3} l_g} \]  

(3)

where \( l_g \) is the standard radius of gyration. The average size (radius) of gyration of the nanoclusters is found to be 0.75 and 0.7 nm for the UFG and CG steels, respectively. This yields an average Guinier radius of 0.97 ± 0.23 and 0.9 ± 0.16 nm, respectively (Fig. 11). The number density of clusters is found to be about 4 times higher than before irradiation. On the other hand, UFG steel did not show significant change in its dislocation densities (values are within the error bars). This can be attributed to the high density of grain boundaries in UFG steel that act as sinks for irradiation-induced defects.

Irradiation can cause local enrichment or depletion of solutes. If the local solute concentration exceeded the solubility limit, solute clusters or precipitates are formed. As shown earlier, after neutron irradiation and the appropriate results are tabulated in Table 2. Tensile test results revealed that CG yield strength increased after irradiation by 132% and its ductility decreased by 82%. On the other hand, UFG steel yield strength increased by 30% and the ductility reduced by 56%.

4. Discussion

Significant changes were observed in the values of the domain size and micro strain of the CG steel after irradiation to 1.37 dpa due to the increased defect concentration (Table 1). As shown in Eq. (1), dislocation density is proportional to the micro strain but inversely proportional to the domain size and thus the dislocation density for irradiated CG steel is found to be about 4 times higher than before irradiation. On the other hand, UFG steel did not show significant change in its dislocation densities (values are within the error bars). This can be attributed to the high density of grain boundaries in UFG steel that act as sinks for irradiation-induced defects.

Irradiation can cause local enrichment or depletion of solutes. If the local solute concentration exceeded the solubility limit, solute clusters or precipitates are formed. As shown earlier, while unirradiated steels show homogenous matrixes with no clusters detected, after irradiation both steels revealed formation of nano manganese–silicon enriched clusters. Although the sizes of clusters in both irradiated steels were very similar (mean cluster radii are 0.9 nm and 0.97 nm in CG and UFG steels, respectively), the irradiated UFG steel revealed higher number density of clusters compared to CG steel. The irradiation temperature was relatively
low (∼80 °C) and thus defect mobility will be relatively low thereby increasing the defect recombination rate and limiting the number of defects available to annihilate at sinks. However, the large grain boundary area per unit volume and the high dislocation density (resulting from the severe plastic deformation through ECAP processing) in UFG steel, increase the probability that the defects will migrate to the sink before being recombined. Thus, significant participation of solutes in the defect fluxes results in pronounced segregation at sinks, raising the local concentration above the solubility limit and thus having higher number density of clusters in UFG steel compared to CG steel that were irradiated at the same conditions.

Higher strength of the UFG steel compared to the CG steel before irradiation is due to the grain refinement (Hall–Petch relation). After irradiation, the CG steel exhibits increased hardness and strength accompanied by decreased ductility as per the commonly observed radiation hardening and embrittlement. However, the UFG steel clearly indicates less significant changes. Tensile strength in CG steel increased after irradiation by 94% compared to 8% in UFG steel (Table 2). Fig. 13 shows that yield point phenomena is observed only in the unirradiated CG steel due to non-negligible source hardening attributable to the pinning of dislocations by impurity atoms (principally C) [46]. Before a Frank-Read (FR) source can be operated by the applied stress, dislocations have to be unpinned from the impurity atoms. However, during irradiation the impurity atoms get attracted to radiation produced defects thereby decreasing source hardening [47] resulting in reduced yield points which disappear following higher radiation dose (1.37 dpa). On the contrary, no yield point phenomena were observed in the UFG steel presumably because impurity atoms (principally carbon) tend to migrate to the grain boundaries thereby not being available for pinning the dislocations.

Although irradiation hardening was minute in the UFG steel compared to the CG counterparts as delineated by Fig. 13, the irradiation induced embrittlement is clear in the UFG steel after irradiation. However, the % decrease in the ductility of UFG steel is quite less than that of CG counterparts. According to Odette and Lucas [48], the primary mechanism of embrittlement in ferritic steels is the hardening produced by nanometer size features that
develop as a consequence of radiation exposure. However, since results showed that there is no significant change in dislocation density in UFG steel after irradiation, the high density of irradiation induced Mn–Si-enriched clusters found in UFG steel is considered to be responsible for the observed embrittlement in UFG steel.

To achieve better understanding of the effect of irradiation microstructural changes on the steel's mechanical properties, the increase in yield stress is related to different strengthening mechanisms. After irradiation, four major mechanisms may be responsible for increased strength as shown in the following:

\[ \Delta \sigma_{\text{irr}} = \Delta \sigma_{\text{GS}} + \Delta \sigma_{\text{SS}} + \Delta \sigma_{\text{Clu}} + \Delta \sigma_{\text{Dis}}, \]

where the subscripts GS, SS, Clu and Dis correspond to strengthening due to grain size, solid solution, clusters and dislocations, respectively. However, no significant change in grain size is observed for both UFG and CG steels after irradiation (Table 3). Thus, strengthening due to Hall–Petch effect is neglected.

Fig. 10. Si–Mn-enriched cluster distribution post neutron irradiation for (a) CG and (b) UFG steel. The dimension of the interior (colored) boxes is 20 × 20 × 100 nm3.
(Δσ_{CG}=0). APT results showed that the percentage of solute atoms that left the matrix to produce clusters is very small (mean atomic concentration of solutes reduces by 0.05% after irradiation). Therefore, the change in the concentration of solutes before and after irradiation does not cause any significant change in the hardening due to solid solution and the solid solution strengthening is ignored. As a result, the remaining two strengthening mechanisms dominate and they cause the increase in the strength after irradiation: cluster strengthening (Orowan precipitate hardening) and strengthening by dislocations (forest strengthening). Thus, the change in the yield strength due to irradiation can be represented as:

\[ \Delta \sigma_y = \sigma_{after\ irr} - \sigma_{before\ irr} = \Delta \sigma_{Clu} + \Delta \sigma_{Dis}. \]  

(5)

As discussed earlier, APT analyses of the samples revealed presence of Mn–Si-enriched clusters in the irradiated UFG and CG steels. However, no clusters were observed in both the unirradiated steels. Hence, the strength increase due to irradiation-induced clusters can be described using Orowan–Ashby model [49,50] shown in Eq. (6). It is important to note that Orowan–Ashby model is commonly used for incoherent precipitates; however, previous studies [51,52] on different alloys showed that hardening due to nano clusters modeled by Orowan model is capable of rendering explanation for observed yield stress. Thus,

\[ \Delta \sigma_{Or-Ashy} = M \frac{0.83 G b}{2 (1-\nu) \pi^2} \frac{1}{2} \ln \left( \frac{2 r_s}{Gb} \right) \]  

(6)

where \( G \) is the shear modulus of the \( \alpha \)-Fe matrix (78 GPa), \( b \) is the magnitude of the matrix Burgers vector (0.248 nm), \( \nu \) is the Poisson’s ratio (0.33) [53], \( M \) is Taylor factor used for converting shear strength to an equivalent uniaxial yield strength (3.06) [54], \( r_s \) is the average radius of cross section of clusters on the slip planes, \( r \) is the mean radius of the clusters and \( \lambda \) is inter-particle spacing which describes the basic characteristics of the clusters inside the irradiated material in terms of their volume fraction and the average cluster radius [49,55],

\[ \lambda = \left( \frac{\pi}{4} - 2 \right) r_s, \]  

(7)

where \( f \) is the volume fraction of the clusters.

To evaluate the role of dislocations to strengthening in the CG and UFG steels, yield strength increment due to dislocation forest strengthening is given by [56]:

\[ \Delta \sigma_{dis} = a M G b (\sqrt{\rho_{irr}} - \sqrt{\rho_{unirr}}), \]  

(8)

where \( \rho_{irr} \) and \( \rho_{unirr} \) are dislocation densities in irradiated and unirradiated materials respectively, and \( a \) is the corresponding strengthening coefficient; for body-centered cubic metals, \( a = 0.4 \) [57]. The change in the material yield strength due to irradiation can then be found through the following equation:

\[ \Delta \sigma_y = \Delta \sigma_{Or-Ashy} + \Delta \sigma_{Dis} = M \frac{0.83 G b}{2 (1-\nu) \pi^2} \lambda \ln \left( \frac{2 r_s}{Gb} \right) + a M G b (\sqrt{\rho_{irr}} - \sqrt{\rho_{unirr}}). \]  

(9)

Using the results obtained from both X-ray and APT analyses, the increase in yield strength due to neutron irradiation (\( \Delta \sigma_y \)) is calculated using Eq. (9). Error calculations were made using error propagation method (Eq. (10)) starting from the standard deviation of the cluster size distribution (Fig. 11) and propagating forward by finding the error in each equation leading to Eq. (9). Calculated results for different strengthening mechanisms and their contributions to yield strength with their corresponding errors (uncertainty) are tabulated in Table 4.

\[ \sigma_{err(x,y)} = \sqrt{ \left( \frac{df(x,y)}{dx} \sigma_x \right)^2 + \left( \frac{df(x,y)}{dy} \sigma_y \right)^2 }, \]  

(10)

where \( \sigma_{err(x,y)} \) is the standard deviation or error in the function \( f(x,y) \). Both tensile yield stress measurements and strengthening mechanisms calculations illustrate similar results. Agreement between the two methods indicates that the Orowan–Ashby along with Taylor strengthening model is sufficient and can be useful for explaining the contribution of nano-cluster strengthening and dislocation forest hardening to the overall strength of the ECAP ultra-fine grained and the CG steels. The results show that while
Irradiation induced dislocation density has a high influence on the total yield stress increase in neutron irradiated CG steel, the irradiation hardening in the UFG steel was mainly due to the irradiation induced clusters.

5. Summary and conclusions

Neutron irradiation effects on ECAP steel were investigated after irradiation to 1.37 dpa dose in the ATR reactor at Idaho National Laboratory. Changes in both microstructural and mechanical properties due to irradiation were analyzed using atom probe tomography, X-ray diffraction, micro-hardness and tensile testing. The following conclusions are drawn:

- High number densities of nano Mn–Si-enriched precipitates were observed in both CG and UFG steels after irradiation. However, the number density and the radius of the clusters were larger in the case of the UFG steels due to the shorter path that the defects need to diffuse before they reach the grain boundary and hence less defect recombination probability in the matrix. The fact that these small clusters were not observed before irradiation confirms that their formation was radiation-induced.
- Both micro hardness and tensile tests revealed radiation hardening in the conventional grain sized steel as expected. However, UFG steel hardness/strength showed minute changes after irradiation indicating less radiation hardening effect.
- Irradiation induced precipitate strengthening and dislocation forest strengthening were evaluated and correlated to the experimental measurements. Irradiation induced dislocation density in the UFG steel is found to be negligible and thus the change in UFG steel strength after irradiation is considered to be due mainly to the cluster hardening.
- As the area of grain boundaries (which act as sinks for radiation-induced defects) is significantly increased by grain refinement, UFG steel revealed better irradiation tolerance. However, irradiation induced solute clustering in UFG alloys needs to be carefully considered.

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References