Cavity swelling of 15-15Ti steel at high doses by ion irradiation

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Abstract

The swelling behavior of titanium-modified austenitic stainless steel 15-15Ti was investigated by pre-implantation of He at room temperature followed by Ni irradiation at 580 °C to peak doses of 120, 240 and 400 dpa. Relatively smaller cavities were observed in the zone of helium implantation while large cavities appeared in the region near the damage peak. A correction formula of the dpa curve was proposed and applied for samples with large swelling. It is found that the steady-state swelling rate of 15-15Ti keeps at ~1% /dpa even to high doses. By comparing the swelling data of the helium-implanted and helium-free regions at same doses, 70 dpa and 122 dpa, the suppression of excessive helium on swelling can be deduced at such doses.

Full Text

Preamble

Cavity Swelling of 15-15Ti Steel at High Doses by Ion Irradiation

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Abstract

Objective: The swelling behavior of titanium-modified austenitic stainless steel 15-15Ti was investigated under high-dose irradiation conditions.

Methods: Helium was pre-implanted at room temperature, followed by nickel ion irradiation at 580 °C to peak doses of 120, 240, and 400 dpa.

Results: Relatively smaller cavities were observed in the helium implantation zone, while large cavities appeared in the region near the damage peak. A correction formula for the dpa curve was proposed and applied to samples exhibiting large swelling.

Conclusions: The steady-state swelling rate of 15-15Ti remains at approximately 1% per dpa even at high doses. By comparing swelling data from helium-implanted and helium-free regions at equivalent doses (70 dpa and 122 dpa), the suppression of swelling by excessive helium can be inferred at these dose levels.

Keywords: 15-15Ti; ion irradiation; irradiation swelling; helium effect; steady-state swelling

Introduction

Austenitic steels are selected as fast reactor fuel cladding materials due to their excellent high-temperature strength and good machinability [1,2]. To enhance the long-term economic performance of Sodium-cooled Fast Reactors (SFR), the damage dose of fuel assemblies could reach up to 200 dpa in future designs [3]. In the Generation IV SFR program, the integrity of fuel pins is highly dependent on whether the cladding can withstand high-temperature and high-burnup irradiation conditions. Swelling remains one of the primary challenges to the structural integrity of claddings in service [4].

Type 316 stainless steel (SS) was initially chosen as the cladding material for prototype fast reactors, but it exhibited excessive swelling at doses above 50 dpa. Studies have shown that swelling can be significantly reduced by adding stabilizing elements, adjusting chemical composition, and introducing cold work. The titanium-stabilized austenitic stainless steel 15-15Ti, derived from 316 SS, exhibits enhanced swelling resistance due to increased nickel content and the formation of intragranular nano-sized TiC precipitates [4,5]. Between 1982 and 1998, 15-15Ti cladding was used in the Phénix reactor, reaching a maximum damage dose of 130 dpa with reportedly acceptable deformation levels. Similar alloys have been selected as core materials for fast reactors worldwide, including ChS-68 (Russia), D9 (USA), D9I (India), Din 1.4907 (Germany), and JPCA (Japan) [6,7].

The swelling of austenitic steels as a function of damage dose is typically characterized by three regimes: an incubation period with negligible swelling at low doses, a transient regime, and finally a steady-state swelling regime. Cavity nucleation occurs primarily during the incubation period, leading to increased number density without significant size changes [2]. As the dose accumulates, cavity size continually increases because the generation and absorption rate of vacancies exceeds their emission rate. During the steady-state swelling stage, the swelling rate becomes nearly independent of dose rate, temperature, and alloying chemistry for a given material. Theoretical analysis suggests that when dislocations and cavities produced by irradiation have comparable sink strengths, the segregation of interstitials and vacancies is most effective, resulting in steady-state swelling with the maximum possible rate [8]. The steady-state swelling rate can thus be used to describe a material' s irradiation resistance [2]. Typical steady-state swelling rates are approximately 1% per dpa for austenitic steels and 0.2% per dpa for ferritic/martensitic steels [9]. In bcc steels, lower dislocation bias for preferential interstitial absorption results in a lower steady-state swelling rate [10].

In-pile irradiation and post-irradiation examination of materials are costly and time-consuming, and induced radioactivity complicates subsequent analysis. Heavy-ion irradiation can achieve high damage rates without residual radioactivity and at significantly lower cost [11], making it widely used as a surrogate for neutron irradiation [11-16]. Ion irradiations have been critical not only for understanding radiation effects but also for developing innovative reactor materials. Numerous studies have investigated irradiation-induced swelling mechanisms using ion irradiation. For example, Ref. [15] demonstrated that the steady-state swelling rate (~1% per dpa) of annealed AISI 304L SS could be reproduced using self-ion irradiation up to approximately 60 dpa. The swelling rates of two austenitic alloys—cold-worked 316 steel and alloy A709—both eventually reached ~1% per dpa between 200 and 300 dpa using Fe irradiation [16]. However, the swelling evolution for more irradiation-resistant materials such as 15-15Ti steel remains limited at very high doses.

For ion-irradiated materials, extra interstitials introduced by ion implantation suppress swelling by reducing the cavity nucleation rate when defect recombination is significant [17]. This process has important consequences for more swelling-resistant steels. Ref. [18] showed that far fewer cavities were produced in solution-annealed JPCA irradiated to 50 dpa at 750 and 800 K by heavy ions compared to 316 SS. Cavity-denuded regions can be observed near the damage peak [19]. To compensate for swelling suppression by extra interstitials, pre-implantation of insoluble helium before heavy-ion irradiation is frequently used to enhance cavity nucleation [20-23]. However, the role of helium, particularly its concentration, is complex in swelling evolution [24-34]. The effect of helium on swelling depends on both the dose and the helium concentration [29]. Studies on both cold-worked and solution-annealed austenitic steels have shown that cavity density increases markedly while cavity size decreases gradually with increasing He/dpa ratio [20,29]. Cavity density tends to saturate while mean cavity size continues to decrease when the helium concentration exceeds an intermediate He/dpa ratio.

Within the dislocation bias model framework, swelling is driven by biased absorption of interstitials and vacancies [27]. Preferential absorption of interstitials by dislocations creates vacancy supersaturation in the matrix. Simultaneously, helium-induced cavity nucleation increases swelling. As helium concentration continues to increase, the growing number of cavities acting as neutral sinks promotes recombination of interstitials and vacancies, thereby hindering swelling.

In this paper, the swelling behavior of 15-15Ti austenitic steel under ion irradiation was investigated to very high doses. By pre-implanting helium, the irradiation damage region bombarded by heavy ions could be divided into helium-implanted and helium-free regions. Cavity morphology and swelling in both regions were examined by transmission electron microscopy (TEM), and the synergistic effect of helium with displacement damage on swelling was discussed.

2.1. Materials and Irradiation Experiment

The base material used in this study is a 20% cold-worked 15-15Ti steel. The nominal composition of this steel was given in Ref. [23]. The 15-15Ti steel in the form of cladding tube was sectioned into Φ 15 mm × 1.5 mm disks by electrical discharge machining. The specimens were then mechanically ground and fine-polished to a mirror-like surface prior to irradiation [23].

Irradiation experiments were conducted at the triple-beam irradiation facility at the China Institute of Atomic Energy (CIAE) [35]. The facility consists of an HI-13 tandem accelerator, a 300 kV helium implanter, and a 300 kV hydrogen implanter. In the irradiation chamber, samples were mounted on a copper base and connected to a PNC/PC heater, allowing heating from room temperature to 800 °C. A hole drilled in the middle of the copper base and brazed with a K-type thermocouple monitored the temperature. A thermostat connected to the heater and thermocouple enabled automatic adjustment of the target temperature to a preset value with an accuracy of $±$2 °C.

Specimens were pre-implanted with helium at room temperature before heavy-ion irradiation to promote cavity nucleation at low doses. A plateau of helium concentration in the depth zone of 350 to 700 nm beneath the irradiated surface was created by multiple-energy implantation. Rate theory calculations indicate that swelling of 15-15Ti increases with helium concentration and saturates at high helium levels [36]. The maximum helium concentration was set to 13,000 appm on the plateau. The implanted helium profile is shown in Figure 1 Figure 1: see original paper. Previous TEM observations revealed no helium bubbles or voids in similar specimens implanted with helium at room temperature [23].

The helium-pre-implanted specimens were irradiated using a 75 MeV defocused Ni beam through a Ta foil approximately 4 m thick. The defocused beam was preferred over raster scanning, as discussed in ASTM E521 standard [36,37]. The tantalum foil was fixed in front of the sample to reduce beam energy and further defocus the beam.

Swelling occurs only within a certain temperature range. At lower irradiation temperatures, defects are less mobile and less likely to form larger clusters. At higher temperatures, vacancies can emit from cavities, counterbalancing the net vacancy flow toward them and limiting growth [27,39]. Maximum swelling occurs at an intermediate temperature known as the peak swelling temperature, which depends not only on the material but also on the dose rate [27,40,41] and hydrogen/helium concentrations in multi-beam irradiations [30]. This temperature shifts to higher values with increasing dose rate. The peak swelling temperature in heavy-ion irradiation of austenitic steels has been measured using various characterization methods, such as positron annihilation techniques [35] and TEM examinations [30]. These studies suggest this temperature is around 580–590 °C [40] for heavy-ion irradiations with dose rates typically in the range of 10^2-10^3 dpa/s. In this study, heavy-ion irradiation was performed at 580 °C (the peak swelling temperature) to peak doses of 120, 240, and 400 dpa. Correspondingly, the doses in the helium implantation zone reached 30, 60, and 122 dpa, respectively. The damage was calculated using the SRIM code [42] with the Kinchin-Pease model, as shown in Figure 1(b). The displacement energies of Fe, Cr, and Ni were set to 40 eV [43].

2.2. Swelling Measurement

After irradiation, TEM lamellae were fabricated using the standard lift-out technique with a TESCAN Lyra3 focused ion beam (FIB). TEM observations were performed using a JEM-2100F microscope. Smaller cavities were observed under kinematical diffraction conditions using bright-field imaging and were measured for diameter and counted in selected regions.

In this study, electron energy-loss spectroscopy (EELS) was employed to measure the local thickness of TEM samples. EELS involves measuring the energy distribution of electrons that have interacted with the specimen and lost energy through inelastic scattering, enabling quick and reliable measurement of local thickness in transmission electron microscopy. Local thickness can be calculated using the formula where t is the sample thickness, λ the mean free path of electrons, I_t the integrated intensity of the total energy-loss peak, and I_0 the integrated intensity of the zero-loss peak. The λ values were measured to be 102, 104, and 98 nm for 200 keV electrons in Fe, Cr, and Ni, respectively [44]. The λ value for 15-15Ti was obtained by weighting its main compositions, yielding 102 nm. A typical TEM image and the selected area where EELS was performed are shown in Figure 2 Figure 2: see original paper and (b). The average thickness of the TEM lamella can be calculated for the same area as the cavity swelling measurement, as indicated in Figure 2(c).

For cavities smaller than the TEM specimen thickness, swelling can be calculated as follows, where V_D is the volume of cavities and V is the selected measurement region. According to the procedure in Refs. [23,38], the cavity volume fraction is corrected for intersection of cavities with the surface using the formula where A and t are the area and average thickness of the measurement region, D_i and n_i are the diameter and observed number of cavities in size class i. For multiple regions, the total cavity volume fraction can be written as where k indicates different regions. A typical image (helium implantation zone dose: 30 dpa) with multiple TEM recordings is shown in Figure 3 [Figure 3: see original paper]. The rectangle defines the area where voids are evaluated, with a width greater than 150 nm. The swelling of a specimen is given by the maximum value calculated using a band width of 150 nm, readily computed using Equation (1).

However, swelling calculated by this method will be seriously overestimated if cavity diameters are comparable to or larger than the TEM sample thickness. For cavities completely cutting through the TEM sample, swelling can be calculated using where V_A is the area of cavities and A is the selected measurement region.

2.3. Correction of the Damage Profile

Significant swelling induced by cavities distorts the distribution of displacement damage. Since swelling accumulates as cavities grow with time or dpa, the dpa profile should theoretically be corrected both spatially and temporally. However, the detailed evolution of swelling as a function of dpa is generally unknown, making accurate temporal correction impossible. To account for the effect of cavity swelling on the damage profile, various correction methods have been developed, typically using reduced mass density in SRIM calculations [16,45].

The fixed damage rate method and the fixed depth method were proposed in Ref. [45]. In the fixed damage rate method, the dpa profile is simply stretched according to the swelling profile. In the fixed depth method, the dpa profile is corrected by using reduced density in SRIM calculations. The mass density of each bin is calculated from the swelling profile at the final state (i.e., the worst case) or from the averaged swelling profile at the beginning and end of irradiation. In any case, the dpa peak remains the same in both methods. Authors have preferred the fixed depth method because inaccurate overlapping of dpa, injected interstitials, and pre-implanted helium could lead to unfair comparisons between different doses. Kim also proposed a correction procedure using a reduced mass density that evolves with swelling in SRIM calculations [16]. However, the density correction formulas used in Ref. [16] and Ref. [45] are approximations of the accurate formula. Small differences in dpa peaks before and after correction are more likely due to this approximation error at large swelling. It should be noted that not only the dpa profile but also the injected interstitial and helium profiles should be corrected, as they are pushed to deeper regions in the case of swelling.

In the following, we re-derive the dpa correction formula using an accurate reduced density. The basic principle is that cavities do not cause any energy loss. The formalism is essentially the same as the fixed damage rate method, as shown in Figure 4 [Figure 4: see original paper].

Assume the unirradiated material before depth h is divided into n bins. Considering a bin at depth h before irradiation, mass conservation after swelling gives where S(h') is the local swelling at the expanded depth h' . For small swelling, this equation can be approximated as , which is exactly the same as Equation (1) in Ref. [16]. However, this approximation becomes inaccurate for large swelling. Because there is no lateral expansion in ion irradiation, mass conservation can also be expressed as By summing all bins from the surface to h' , we obtain Assuming that swelling is caused by cavities that do not cause energy loss, the number of displaced atoms per injected ion remains the same in each bin regardless of swelling. The same holds for the dpa rate or total dpa, i.e., where ϕ(x) is the original dpa profile and ϕ '(x) is the corrected dpa profile. We are free to make the change h' → h, yielding a correction formula for dpa. As for the concentration of injected interstitials and helium in a bin, since they are calculated as the ratio of deposited atoms to material atoms in that bin (similar to displaced atoms), Equation (7) can also be used to correct the concentration profiles for injected interstitials and helium.

Equation (8) appears problematic because swelling changes during irradiation. In the following, we prove that time dependence need not be considered. Assuming the unirradiated material before depth h is divided into n bins and the irradiation period is divided into m time intervals, let S_{ij} be the swelling for the i-th bin during the j-th time interval. The bin at depth h will be at h' after irradiation, expressed by the sequential increment of depths in each time interval, where h_j is the depth of the bin at original position h after the j-th time interval, and Δh_{ij} is the thickness of the i-th bin after the j-th time interval. It can be easily proved that Equation (8) can be rewritten as Alternatively, Equation (9) can be written in differential form: dϕ '/dh = (1 + S(h)) dϕ/dh, where S(h) is the swelling of the bin at original position h. In practice, swelling is measured at the final state of an irradiated sample. We should treat swelling as a function of depth, then Equation (11) is actually the same as Equation (6). In other words, the dpa profile can be corrected by stretching the dpa profile using depth-dependent swelling data. This can be understood through the schematic drawing in Figure 4. Assuming cavities are uniformly distributed and cause no energy loss, the damage rate is actually the same at h and h' for both unirradiated and irradiated samples. This is also valid if reduced density for each layer is used in SRIM calculations.

3. Results and Discussion

TEM images of irradiated samples at different dpa levels are shown in Figure 5 [Figure 5: see original paper], overlapped with damage curves. Small cavities are visible in the helium-implanted zone around a depth of 1 m (Figure 5d-5f), while large cavities were observed in deeper regions beyond the helium implantation range (Figure 5a-5c). Particularly for the sample with a peak dose of 400 dpa, cavities coalesced into very large cavities near the damage peak [46]. No cavities were observed at the damage peak itself. Cavity size generally increases with dose in both helium-implanted and helium-free regions. The absence of swelling in the peak damage region indicates that injected Ni ions suppressed swelling at the damage peak during irradiation [47,48]; in other words, the local vacancy supersaturation was lower than the threshold for cavity swelling.

Swelling was not observed in 15-15Ti under single-beam nickel irradiation even at doses over 100 dpa [49]. However, large swelling was observed in the presumably helium-free region. Ref. [50] showed that helium could diffuse further into deeper regions due to vacancies created by ion bombardment. This helium could enhance cavity nucleation, leading to significant swelling when combined with high doses.

Based on the principle that cavities do not cause energy loss of injected ions, a dpa correction formula was re-derived in Section 2.3. The dpa profile is effectively stretched for an irradiated sample. To perform the correction, TEM images were divided into five bins of equal width and the swelling of each bin was measured. Depth-dependent swelling was then interpolated from the binned data. Using Equation (7) from Section 2.3, the damage curve was corrected and is shown overlapped with TEM images in Figure 5b and Figure 5c.

Compared to the helium-implanted zone and the region with large cavities, cavities in the helium-implanted zone are smaller and have higher number density than those in the displacement-damage-only region at the same dose. Note that the helium implantation zone in Figure 5b and the region just beyond the helium implantation range in Figure 5a receive a similar dose of approximately 70 dpa. However, cavities in the latter region are significantly larger than those in the helium zone, indicating that implanted helium may play a role in cavity growth. This anomaly is evident when comparing two regions in Figure 5c and Figure 5b where the irradiation dose is around 122 dpa.

Cavities in the helium-implanted zones were measured and counted at different doses, with their size distributions shown in Figure 6 [Figure 6: see original paper]. The tail of the size distribution increases markedly with increasing dpa, although the peak shifts only moderately to larger sizes, from approximately 5.5 nm to 7.5 nm as damage increases from ~30 dpa to ~122 dpa. Much larger cavities exceeding one hundred nanometers were observed in the helium implantation zone as damage reached ~122 dpa.

Cavity density in the helium-implanted zones is compared in Figure 7 [Figure 7: see original paper]. Cavity density decreased significantly with increasing dose. Previous studies have shown that voids rather than bubbles predominate in specimens after helium implantation followed by high-dose heavy-ion irradiation [23]. The increase in cavity size and decrease in density are mainly due to cavity growth and coalescence with increasing dose.

Cavity swelling in the helium-implanted zone and the helium-free region near the damage peak as a function of dpa is plotted in Figure 8 [Figure 8: see original paper]. Swelling exceeds 100% at 243 dpa and increases up to 142% at 278 dpa. As discussed previously, swelling increases with dose in both regions. In the helium-free region, a steady-state swelling rate of ~1% per dpa is observed at high doses. It is also clear that swelling in the helium-implanted zone is lower than that near the damage peak region in the 70–122 dpa range. In the dislocation bias model, swelling is caused by bias-driven vacancy growth, which requires excess vacancy flux [51]. Cavities such as helium bubbles become the main point defect sinks due to increased density in the presence of excessive helium. Abundant cavities as neutral sinks result in nearly equal absorption of interstitials and vacancies, thereby inhibiting cavity growth and suppressing swelling [27].

4. Conclusion

In summary, the swelling behavior of 15-15Ti stainless steel was investigated by pre-implanting helium at room temperature followed by Ni ion irradiation at 580 °C to peak doses of 120, 240, and 400 dpa. TEM lamellae were prepared and examined for the irradiated samples, and electron energy-loss spectroscopy was used to measure the thickness of cavity-containing regions in the TEM lamellae.

Swelling induced by smaller cavities was calculated based on cavity and thickness measurements in the same region. Large cavities were observed in the displacement-damage-only region and coexisted with smaller cavities in the helium-implanted zone. Swelling in the region of large cavities was determined by measuring the porous region. A correction formula for the dpa curve was proposed using more accurate reduced densities based on the assumption that cavities do not cause energy loss.

Cavity size increases but density decreases with dose in both helium-implanted and helium-free regions, primarily attributed to cavity growth and coalescence under irradiation. The 15-15Ti steel shows severe swelling at high doses, exceeding 100% at 243 dpa. A steady-state swelling rate of ~1% per dpa was observed in 15-15Ti at high doses.

Comparing swelling in helium-implanted and helium-free regions, cavities are much smaller in the helium-implanted zone than in the damage region at the same doses (70 and 122 dpa), although number density is higher. Lower swelling is observed in the helium-implanted zone. It appears that excessive helium suppresses swelling at these doses, likely because higher cavity density in the helium-implanted zone promotes recombination of interstitials and vacancies, thus inhibiting cavity growth.

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Figure Legends

Figure 1 (a) Distribution profile of implanted helium; (b) Profiles of displacement damage (solid) and implanted Ni (dotted). The He implantation profile is shown as a dashed line.

Figure 2 TEM images and EELS measurement of 15-15Ti after irradiation to a peak dose of 120 dpa. (a) Overview image in bright field; (b) Dark field image in STEM mode in the helium-implanted zone (dose ~30 dpa). The rectangle indicates the area measured by EELS; (c) Actual thickness of the EELS-measured area, obtained by multiplying the relative thickness from EELS measurement with the electron mean free path. The 150-nm-wide rectangle indicates the selected region for cavity measurements.

Figure 3 Combined TEM image for a specimen with a helium implantation zone dose of 30 dpa. The red rectangle defines the zone where cavities were measured. TEM images were rotated so that the horizontal axis is parallel to the projected surface of the specimen.

Figure 4 Schematic drawing of ion injection into a material with normal and reduced densities.

Figure 5 Microstructure of 15-15Ti after irradiation at peak doses of (a) 120 dpa; (b) 240 dpa; (c) 400 dpa. Images are overlapped with profiles of displacement damage (solid), implanted Ni (dotted), and He (dashed). Damage curves as a function of depth in (b) and (c) were corrected due to the presence of extraordinarily large cavities. Microstructure in the helium-implanted zone is shown in (d-f) at doses of 30 dpa, 60 dpa, and 122 dpa, respectively.

Figure 6 Cavity size distribution in the helium-implanted zone.

Figure 7 Cavity densities in the helium-implanted zone.

Figure 8 Cavity swelling of the He-implanted zone and the region near the damage peak.

Note: Figure translations are in progress. See original paper for figures.

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