

Study of austenitic steel 16Cr15Ni microstructure after helium ion irradiation along ion trajectory

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Abstract

The paper investigates the microstructural evolution of 16Cr15Ni austenitic steel irradiated with 40 keV helium ions to a fluence of $5 \times 10^{20} \text{ m}^{-2}$. The effects of irradiation temperature and preliminary heat treatment on porosity and gas swelling were studied. Transmission electron microscopy (TEM) was used to examine steel samples irradiated at 50 °C and annealed at 750 °C for 1 h, as well as quenched samples and samples annealed after quenching and subsequently irradiated at an elevated temperature of 650 °C.

The highest gas swelling was observed in the sample annealed after quenching and subjected to subsequent high-temperature irradiation, whereas the lowest swelling was found in the quenched sample. The microstructure and porosity parameters of samples prepared for TEM by the focused ion beam (FIB) method were also compared with those of samples prepared by single-sided electrolytic thinning. The FIB-prepared samples showed somewhat higher porosity and swelling parameters, suggesting that this preparation method provides a more accurate characterization of the degree of radiation damage and irradiation-induced effects.

1 Introduction

Fast breeder reactors operate under intense fluxes of high-energy neutrons and are designed for more efficient utilization of nuclear fuel and natural uranium resources. The severe irradiation and thermal conditions inside the reactor place high demands on the structural materials used in the core. Austenitic ChS-68 steel is used for fuel cladding in the BN-600 and BN-800 fast neutron reactors [1–3]. The ability of cladding materials to retain their structural and mechanical properties under irradiation is particularly important because increasing fuel burn-up is one of the key factors determining the efficiency of fast reactor operation.

Under fast-neutron irradiation, energetic particles interact with the crystal lattice and generate large numbers of point defects, including vacancies and interstitials. At the same time, helium is produced through nuclear transmutation reactions. Because helium has very low solubility in metals, it tends to accumulate in vacancy clusters and form pressurized gas bubbles and gas-filled voids. The development of such defects contributes to radiation-induced swelling, hardening, and high-temperature embrittlement of structural materials [4–7].

Helium production is therefore an unavoidable process under reactor irradiation conditions. Its continuous accumulation and interaction with radiation-induced defects can significantly affect the stability of the microstructure and accelerate degradation of fuel-cladding materials. The formation and evolution of helium-vacancy complexes, bubbles, and voids depend on irradiation temperature, defect concentration, alloy composition, and the initial microstructural state of the material [4–16].

Improving the radiation resistance of structural materials is an important task in the development of advanced nuclear energy systems. The ability of the microstructure to absorb, redistribute, and annihilate radiation-induced vacancies and interstitial defects strongly influences the resistance of a material to radiation damage. ChS-68 steel has been developed and optimized as a structural material for fuel cladding in the BN-600 fast reactor [1–6, 14].

The radiation response of austenitic steels is strongly affected by their chemical composition, phase structure, and initial microstructure. These factors can influence helium diffusion, trapping, bubble nucleation, and subsequent bubble growth. Therefore, understanding the relationship between the initial structural state of ChS-68 steel and the development of helium-induced porosity is important for evaluating its behavior under irradiation. Transmission electron microscopy (TEM) is one of the most effective methods for direct characterization of radiation-induced defects and helium bubbles after ion irradiation.

Helium-induced porosity in ChS-68 steel has been investigated previously [4–6]. However, in these studies TEM specimens were mainly prepared by electrolytic thinning. Although this method is widely used for microstructural analysis, it does not allow the evolution of porosity to be studied continuously along the ion trajectory and through the irradiated layer. This limitation is particularly important for low-energy helium-ion irradiation, where helium concentration and radiation damage vary strongly with depth.

The focused ion beam (FIB) method makes it possible to prepare site-specific TEM lamellae from selected regions of the irradiated layer and therefore provides an opportunity to examine changes in the microstructure and helium-induced porosity along the ion trajectory. Comparison of FIB-prepared specimens with conventionally

Table 1: Chemical composition of ChS-68 steel (wt.%).

Element	Content	Element	Content
C	0.05–0.08	Mo	1.9–2.5
Mn	1.3–2.0	Ti	0.2–0.5
Si	0.5–0.8	V	0.1–0.3
S	≤ 0.012	Al	≤ 0.1
P	0.02	B	0.003–0.006
Cr	15.5–17.0	N	≤ 0.02
Ni	14.0–15.5	Co	≤ 0.02

Table 2: Heat-treatment and irradiation conditions for ChS-68 steel.

Sample	Pre-irradiation heat treatment	Irradiation temperature	Post-irradiation annealing
S1	1100°C for 1 h, WQ; 750°C for 1 h	$\sim 50^\circ\text{C}$	650°C for 1 h
S2	1100°C for 1 h, WQ; 750°C for 1 h	650°C	–
S3	1100°C for 1 h, WQ	650°C	–

prepared electrolytically thinned specimens can also provide information on the influence of specimen-preparation technique on the measured porosity parameters.

The aim of this work is to investigate the evolution of helium-induced porosity and microstructure in ChS-68 austenitic steel under different heat-treatment and irradiation conditions. Particular attention is given to the effect of irradiation temperature and initial microstructural state on helium-bubble formation and gas swelling. The microstructural parameters obtained from TEM specimens prepared by the FIB method are also compared with those obtained using single-sided electrolytic thinning.

2 Materials and methods

The material investigated in this study was ChS-68 austenitic steel. Its chemical composition is presented in Table 1.

Helium-ion irradiation was performed using 40 keV He^+ ions to a fluence of $5 \times 10^{20} \text{ m}^{-2}$. Irradiation was carried out at temperatures of approximately 50°C and 650°C . The initial heat-treatment conditions of the samples were varied, and one of the irradiated samples was additionally subjected to post-irradiation annealing. The heat-treatment and irradiation conditions are summarized in Table 2.

The microstructure of the samples after helium-ion irradiation was characterized by transmission electron microscopy (TEM). Focused ion beam (FIB) preparation was used to obtain site-specific TEM lamellae from the irradiated region and to study microstructural changes along the ion trajectory.

The projected range of helium ions and the corresponding depth distribution were estimated using SRIM calculations. The calculated helium depth profile is shown in Figure 1.

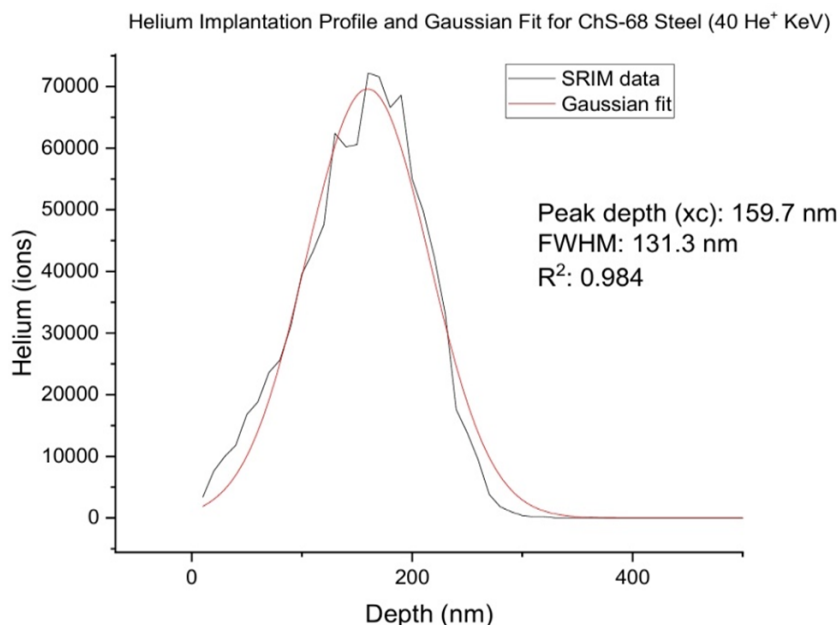


Figure 1: Calculated depth distribution of 40 keV He⁺ ions in ChS-68 steel obtained using SRIM.

Swelling was calculated as the ratio of the total volume of voids and bubbles within the analyzed area to the corresponding material volume. The thickness of the TEM lamellae was approximately 80 nm for all samples.

The swelling values were determined from 3–4 TEM images, with 3–4 regions analyzed in each image. Since the helium concentration varies with depth, the swelling measured near the surface does not correspond to the swelling at the peak of the helium-ion distribution. Therefore, the values reported in this work are mainly averaged over the depth of the helium distribution. Where possible, the maximum swelling values corresponding to the peak of the helium-ion range are also presented.

3 Experimental results

Figure 2 shows the microstructure of sample S1 of ChS-68 steel after helium-ion irradiation. The TEM images were obtained along the helium-ion range. The sample surface is indicated by the dashed line.

Porosity becomes visible at a depth of approximately 30–40 nm from the surface. In the near-surface region, the pores are relatively coarse. The highest concentration of porosity is observed within a layer located approximately 100–200 nm from the surface, as indicated by the red lines in Figure 2. Helium-filled voids and bubbles are detected down to a depth of approximately 300 nm.

These observations are in good agreement with the calculated helium depth profile shown in Figure 1 and with the results reported in Refs. [17,18]. The maximum helium-ion range is located at a depth of approximately 150–160 nm.

Gas-filled voids and bubbles formed in the main porosity layer were predominantly polyhedral in shape. The maximum size of the voids and bubbles reached approximately 15 nm, while the average size over the entire irradiated layer was approximately 8 nm (Figure 2 and Table 3). The porosity parameters and swelling of

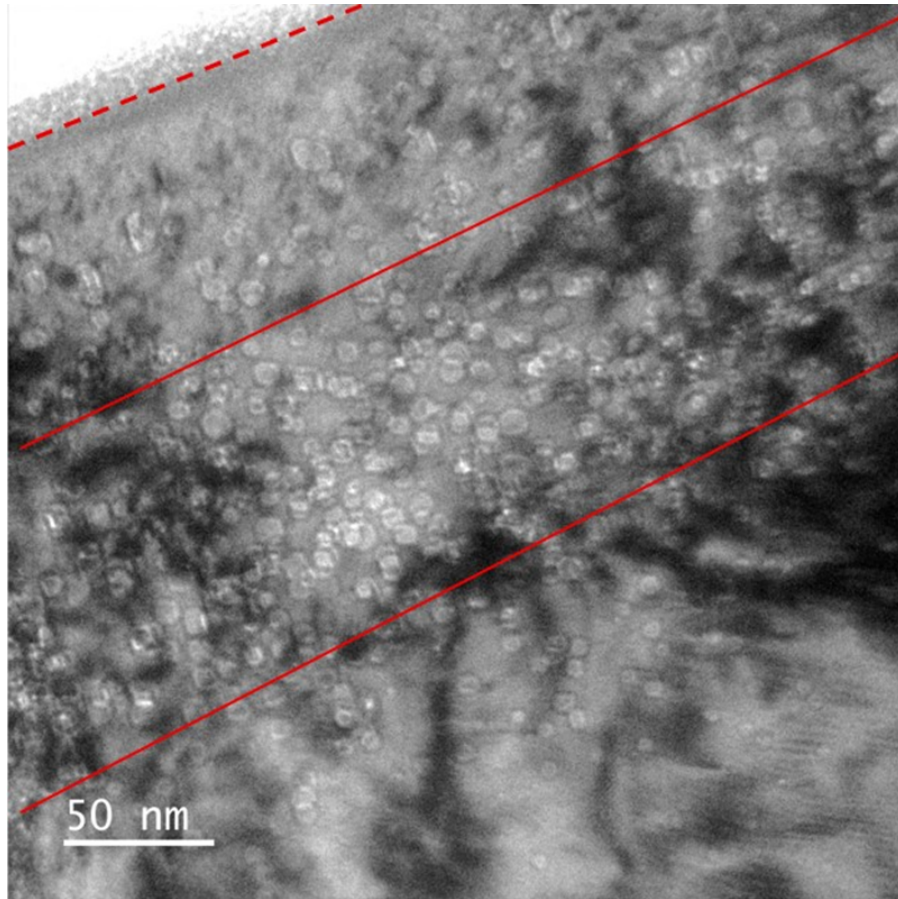


Figure 2: Microstructure of sample S1 after helium-ion irradiation.

Table 3: Porosity parameters of ChS-68 samples after helium-ion irradiation.

Sample	$d_{\max}$, nm	d_{med} , nm	S , %
S1	15.0 ± 3.0	8.0 ± 2.1	1.6 ± 0.5
S2	18.5 ± 4.0	8.9 ± 2.2	3.9 ± 1.1
S3	8.2 ± 2.5	4.4 ± 0.9	1.2 ± 0.3

the irradiated layer are summarized in Table 3. The gas swelling was approximately 1.6%.

Figure 3 shows the general microstructure of sample S2 of ChS-68 steel after helium-ion irradiation. As in sample S1, the gas-filled voids and bubbles were predominantly polyhedral in shape.

However, sample S2 showed several differences compared with sample S1. Porosity became visible at a depth of approximately 30–40 nm from the surface, while the main porosity layer started closer to the surface and extended over a broader depth range, as indicated by the red lines in Figure 3. Relatively large bubbles were observed within the layer from approximately 50 to 200 nm from the surface. The maximum size of the gas-filled voids and bubbles reached approximately 19 nm, while the average size was approximately 9 nm (Figures 3 and 4 and Table 3). Bubbles were also observed at depths of up to approximately 320 nm and were generally larger than those observed in sample S1.

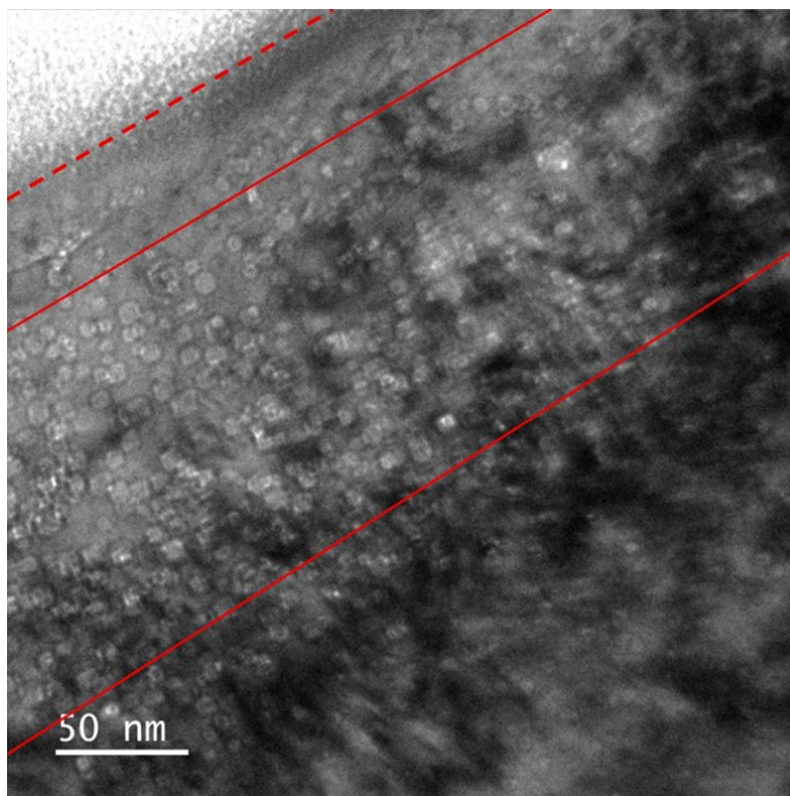


Figure 3: Microstructure of sample S2 after helium-ion irradiation.

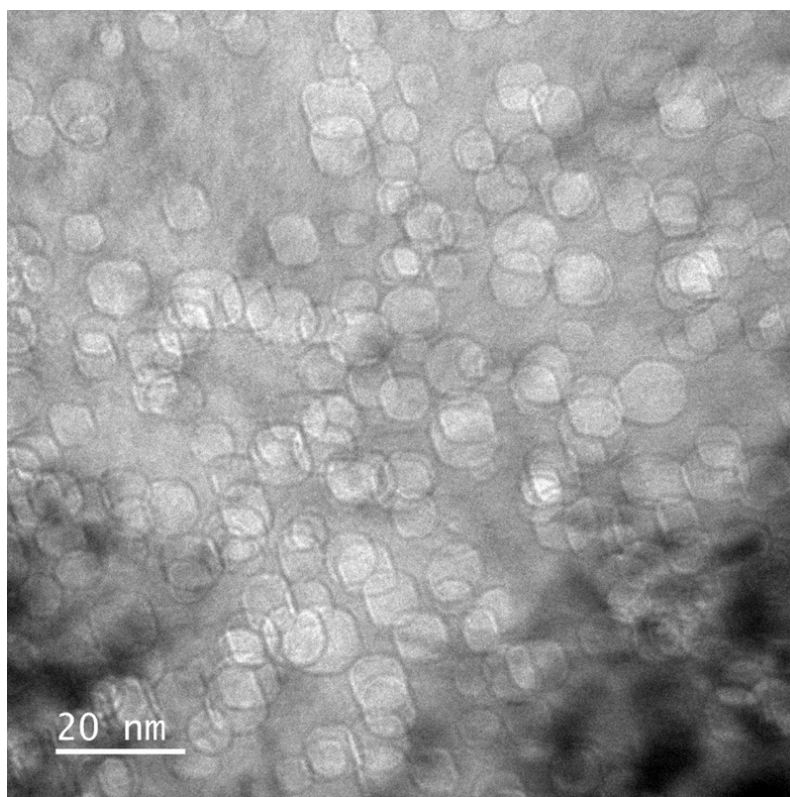


Figure 4: Porosity of sample S2 at higher magnification.

The microstructure of sample S2 is characterized by large, predominantly poly-

hedral voids and bubbles with a high number density. This microstructural state results in a relatively high average gas swelling of approximately 3.9% within the irradiated layer. In some local regions, the swelling reaches approximately 5.5%.

Figure 5 shows the general microstructure of sample S3 after helium-ion irradiation. The depth dependence of porosity is generally similar to that observed in samples S1 and S2. Porosity becomes visible at a depth of approximately 30–40 nm from the surface and extends to a depth of approximately 300 nm.

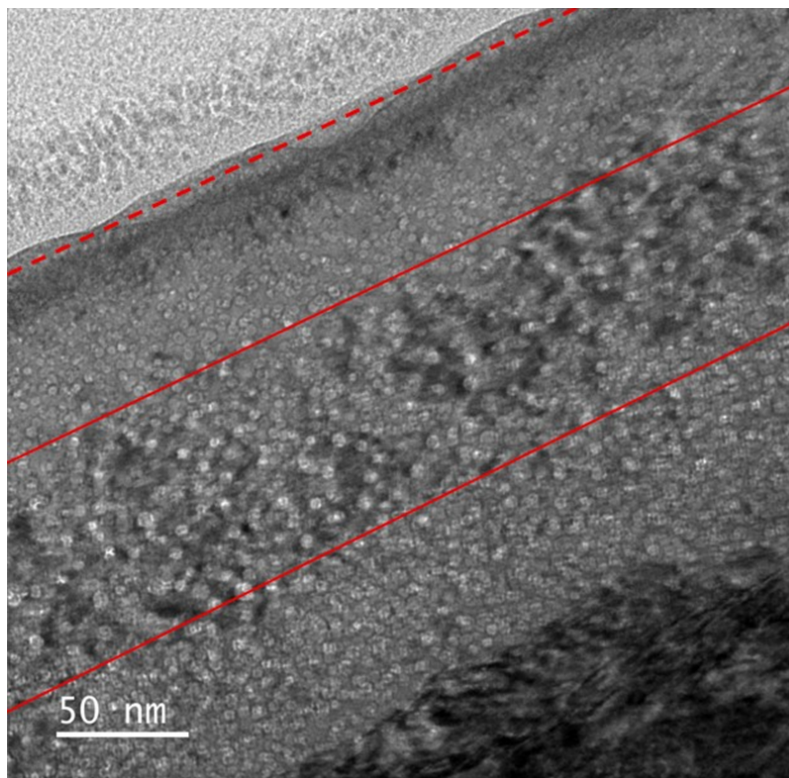


Figure 5: Microstructure of sample S3 after helium-ion irradiation.

However, the porosity parameters of sample S3 are lower than those of samples S1 and S2, as shown in Table 3. The maximum size of the voids and bubbles in the main irradiated layer is approximately 8 nm, while the average size does not exceed approximately 4.5 nm.

Figure 6 shows the porosity in the main irradiated layer of sample S3 at higher magnification. Comparison of the porosity in samples S2 and S3 (Figures 3–6) shows that sample S3 contains smaller voids and bubbles but with a higher number density. Despite the higher number density, their smaller size results in the lowest average swelling among the investigated samples, approximately 1.2%.

4 Discussion

One of the major irradiation-induced degradation mechanisms in austenitic steels used as reactor structural materials is vacancy swelling [2, 7, 8, 15]. Gas swelling is generally considered to be less critical in austenitic steels than in body-centered cubic (BCC) alloys. Nevertheless, the susceptibility of face-centered cubic (FCC) materials to helium-induced swelling depends strongly on irradiation conditions,

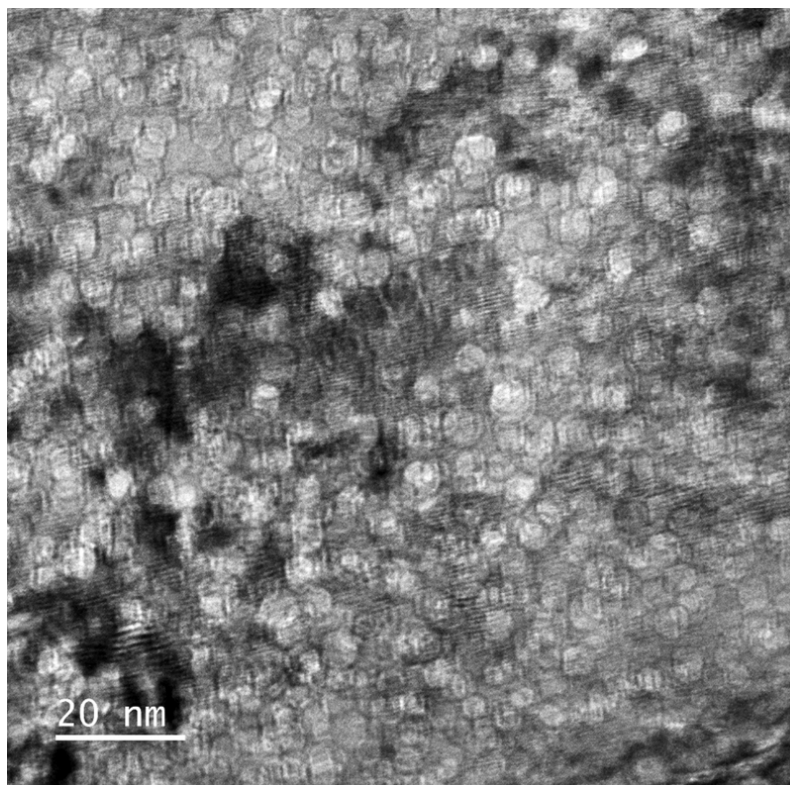


Figure 6: Porosity of sample S3 at higher magnification.

helium concentration, alloy composition, and the initial microstructural state [4–7]. At sufficiently high helium concentrations, substantial gas swelling may therefore develop in austenitic steels.

To interpret the differences observed between the investigated samples, both their initial structural state and the irradiation conditions must be considered. Two principal heat-treatment conditions were studied: quenching and quenching followed by annealing. In a complex-alloyed austenitic steel such as ChS-68, these treatments produce different structural and phase states, which can significantly affect the subsequent formation of helium-induced porosity.

After quenching, the austenitic matrix contains an increased concentration of quenched-in vacancies together with interstitial carbon and substitutional alloying elements such as Ni, Cr, and Mn in solid solution. The resulting defect structure contains numerous regions of local elastic strain that can act as nucleation sites for radiation-induced defect complexes. In samples annealed after quenching, the concentration of quenched-in vacancies is reduced, while part of the alloying elements may leave the solid solution and participate in the formation of secondary phases, including carbides and intermetallic phases [8,9]. Consequently, the density of potential defect-nucleation sites can be lower after quenching followed by annealing than after quenching alone [4–7].

The number of nucleation sites for helium-filled voids and bubbles is an important factor controlling porosity evolution. A larger number of nucleation sites can promote the formation of a higher density of smaller bubbles, whereas a lower nucleation-site density can favor the growth of fewer but larger bubbles. Since swelling depends strongly on bubble size as well as number density, these differences in the initial microstructure can substantially influence the final swelling level.

The irradiation temperature and the presence or absence of post-irradiation annealing must also be taken into account. Sample S1 was irradiated with helium ions at a relatively low temperature of approximately 50 °C. Under these conditions, the mobility of radiation-induced defects and helium atoms is limited, which suppresses the formation and growth of large helium bubbles during irradiation. Instead, irradiation produces a highly defective structure containing vacancy clusters, dislocation loops, and helium-vacancy complexes [4–6, 19, 20].

During subsequent annealing, thermally activated migration and dissociation of helium-vacancy complexes promote helium redistribution and the formation and growth of gas-filled voids and bubbles. The resulting bubble size depends on the stability of the He_mV_n complexes as well as on the annealing temperature and duration [4–6]. In contrast, irradiation at 650 °C provides sufficient defect and helium mobility for bubble nucleation and growth to occur directly during irradiation.

Thus, low-temperature irradiation followed by annealing and direct high-temperature irradiation represent different pathways of porosity formation. In the first case, helium is initially accumulated predominantly in vacancy-related defect complexes, which subsequently evolve during annealing. In the second case, helium-filled voids and bubbles form and grow during irradiation itself. These differences are reflected in the microstructures shown in Figures 2–6 and in the porosity parameters summarized in Table 3.

The lowest void and bubble sizes were observed in sample S3, which was quenched and subsequently irradiated at 650 °C. The average swelling of this sample was approximately 1.2%. In comparison, annealing after quenching before irradiation, as in sample S2, and low-temperature irradiation followed by post-irradiation annealing, as in sample S1, resulted in the formation of larger voids and bubbles and consequently higher swelling.

The relatively low swelling of sample S3 can be associated with the large number of defect-nucleation sites retained after quenching. These sites promote the formation of a high number density of relatively small helium bubbles. In contrast, annealing after quenching reduces the concentration of quenched-in defects and can modify the distribution of alloying elements and secondary phases. Under the present irradiation conditions, the quenched state therefore appears to be more resistant to the development of helium-induced swelling than the annealed state.

Another important difference is the depth distribution of porosity. In sample S2, the region containing relatively large helium bubbles extends over a broader depth interval than in samples S1 and S3 (Figure 3). For irradiation with 40 keV He^+ ions, the main porosity region is typically observed at depths of approximately 100–200 nm [17–19], which is also consistent with the SRIM profile shown in Figure 1. This behavior is clearly observed in samples S1 and S3.

The broader porosity distribution in sample S2 may be related to structural changes produced by annealing after quenching. A more advanced redistribution of alloying elements and decomposition of the supersaturated solid solution during annealing may modify the trapping and migration behavior of implanted helium. As a result, bubble nucleation and growth may occur over a broader part of the implanted-ion range. A related tendency is also observed in sample S1, which was annealed after quenching. Although a broad high-density porosity layer is less pronounced in this sample, relatively large bubbles are still observed at depths below 50 nm (Figure 2).

The largest helium-filled voids and bubbles were observed in sample S2, which was quenched and subsequently annealed before irradiation. The swelling measured in different local regions of this sample ranged from approximately 3% to 5.5%, while the average swelling over the irradiated layer was approximately 3.9%. Sample S1, irradiated at approximately 50 °C and subsequently annealed, exhibited a lower average swelling of approximately 1.6%.

Comparison of the three samples indicates that the standard quenched condition provides the lowest swelling under the irradiation conditions investigated in this work. At the same time, the results obtained for samples S1 and S2 show that thermally activated redistribution of helium and radiation-induced defects can substantially increase porosity. The annealing treatments used in the present experiments activate diffusion-controlled processes that may also become important during prolonged operation at elevated temperatures. However, such annealing experiments should not be regarded as a complete reproduction of long-term reactor service conditions.

An additional aspect of this work concerns the influence of TEM specimen-preparation technique on the measured porosity parameters. Under helium-ion irradiation, porosity is distributed non-uniformly through the irradiated layer. According to the SRIM calculations, the maximum helium concentration for 40 keV He^+ irradiation is located at a depth of approximately 150–160 nm. The FIB technique makes it possible to prepare a cross-sectional lamella containing the entire irradiated region and therefore to characterize the variation of porosity along the ion trajectory.

A comparison between FIB preparation and electrolytic thinning was previously performed for irradiated vanadium alloys [17]. One of the objectives of the present study was to perform a similar comparison for ChS-68 steel, because samples irradiated under comparable conditions had previously been investigated using conventional electrolytic thinning [4–6].

In particular, Ref. [5] reported porosity in ChS-68 steel in two initial structural states: after quenching and after quenching followed by annealing, corresponding to the initial conditions of samples S3 and S2, respectively. Comparison with the present results shows that the porosity parameters obtained from the FIB-prepared specimens are generally higher. For the quenched sample, the maximum void/bubble size determined in the present work is approximately 1.5 times larger, while the average size is approximately twice as large. For sample S2, the average sizes agree within the experimental uncertainty, whereas the maximum size obtained from the FIB specimen is approximately 1.5 times larger.

An even larger difference is observed for the calculated swelling. For sample S2, the present value is approximately 3.9%, compared with approximately 1.6% obtained previously, corresponding to a difference of about 2.5 times. For sample S3, the difference is substantially greater: approximately 1.2% in the present work compared with approximately 0.14% in the previous study.

These differences can be associated with the geometry of TEM specimen preparation. Single-sided electrolytic thinning does not necessarily provide access to the complete irradiated depth range. Depending on the remaining specimen thickness and the location of the electron-transparent region, only part of the helium-implanted layer may contribute to the TEM observations. This is particularly important for 40 keV helium-ion irradiation, for which the implanted helium and

irradiation-induced defects extend over a depth substantially larger than a typical electron-transparent TEM region. Consequently, measurements based on electrolytically thinned specimens may underestimate the contribution of regions containing the highest bubble density or the largest bubbles.

In contrast, cross-sectional FIB preparation allows direct observation of the complete irradiated layer, including the near-surface region, the helium-concentration maximum, and the deeper part of the ion range. The present results therefore indicate that FIB-prepared specimens provide a more complete characterization of the depth-dependent porosity and can yield higher integrated swelling values than conventional electrolytic thinning. This conclusion is consistent with the earlier comparison reported in Ref. [17].

5 Conclusions

This study investigated helium-induced porosity and swelling in ChS-68 austenitic steel as a function of the initial heat-treatment condition and irradiation parameters. The results obtained from FIB-prepared TEM specimens were also compared with previously reported data for specimens prepared by single-sided electrolytic thinning.

1. Irradiation of 16Cr15Ni-type austenitic steel with 40 keV He^+ ions leads to the formation of pronounced gas-filled porosity. Depending on the initial heat-treatment condition, the average swelling of the irradiated layer can reach approximately 4%, while local swelling near the maximum of the helium distribution can exceed 5%.
2. The lowest porosity parameters and swelling were observed for the quenched sample irradiated at 650 °C. The average swelling in this condition was approximately 1.2%. This indicates that the quenched structural state provides the highest resistance to helium-induced swelling among the conditions investigated in this work.
3. Annealing after quenching resulted in increased void and bubble sizes and higher swelling. This behavior is likely associated with changes in the structural and phase state of the steel during annealing, including partial decomposition of the solid solution and a reduction in the number of effective nucleation sites for helium-filled voids and bubbles. This tendency was observed under the irradiation and heat-treatment conditions investigated in this work.
4. Comparison with previously reported results obtained using single-sided electrolytic thinning shows that the FIB-prepared specimens generally exhibit higher measured porosity parameters and swelling. For several characteristic porosity parameters, differences of approximately 1.5–2 times were observed, while the difference in calculated swelling was considerably larger in some cases. Cross-sectional FIB preparation provides access to the entire irradiated layer and therefore allows a more complete characterization of the depth-dependent porosity formed along the helium-ion trajectory.

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