Swelling behavior of Fe-21Cr-32Ni Model Alloy

Muhammet AYANOGLU[1] and Arthur T. MOTTA

Department of Mechanical and Nuclear Engineering, Pennsylvania State University

Introduction

Irradiation induced swelling is a concern for some of the candidate materials planned to be used in advanced reactors where radiation doses can reach up to 200 dpa (displacement per atom) with operating temperatures of 400°C or above [1]. Therefore, it is crucial to understand the swelling behavior of these materials under irradiation.

Ion irradiation has been widely used to deliver high doses of irradiation since neutron irradiation requires very long exposure times due to its low damage rates, and the usage of hot cells to handle activated samples. One challenge to correlating the two is the absence of helium during the ion irradiation because no transmutation reaction takes place during ion irradiation. In nuclear reactors, swelling behavior can be significantly affected by the presence of helium generated from (n, α) reactions. This is because helium is insoluble at low temperatures (unlike other residual gases) and therefore, it can stabilize cavities in the matrix. To address this question helium can be implanted simultaneously to emulate reactor irradiation.

In this study, the swelling behavior of 21Cr32Ni type austenitic alloy (a simple model alloy analogue to the commercial alloy 800H) was investigated after dual beam irradiation of 5 MeV Fe and simultaneous injection of helium using transmission electron microscopy (TEM). Further discussion is presented.

Experimental Methods

Bulk 21Cr32Ni samples were irradiated to doses of 1 dpa, 10 dpa and 20 dpa using single beam of 5 MeV Fe^{++} ions and 16.6 dpa using dual beam where helium (1 appm/dpa) was simultaneously injected to emulate reactor irradiation conditions. The irradiation temperature used in the experiments was ~440°C and the dose rate was 5×10^{-4} dpa/s. The target dose for each irradiation was achieved at around ~0.6 µm depth from the irradiated surface. The damage profile and ion range of 5 MeV Fe ions in bulk 21Cr32Ni austenitic alloy were determined using the Stopping Range of Ion in Matter (SRIM) program with the Kinchin-Pease-Quick calculation mode and a displacement energy of 40 eV.

Focused ion beam (FIB) was then used to prepare TEM specimens. The irradiated microstructure was studied using bright field/dark field (BF/DF) micrographs. The overfocus/underfocus technique was used to image voids.

The size of the voids was measured by the inner most diameter using the dark fringes visible in underfocus condition and voids were counted using ImageJ software and their densities calculated using a thickness measurement of each sample using both Convergent Electron Beam Diffraction (CBED) and Energy Filtered TEM (EFTEM). The irradiated region was binned using 100nm bin width from the surface to the maximum Fe ion range to obtain the depth-dependent swelling profile.

Results

The damage profile and ion range of 5 MeV Fe ions as well as helium implantation in 21Cr32Ni model alloy is shown in **Fig. 1** for the dual beam irradiated alloy. The damage profile in this figure shows that the maximum range of Fe ions is ~2 µm with the peak dose located at ~1.3 µm.

Fig. 2a shows a bright field image of a sample irradiated to 16.6 dpa with dual beam at 446°C. Cavities are observed which are highlighted in

[1] mua214@psu.edu

yellow for better visibility in **Fig. 2b**. **Fig. 2c** shows a high magnification image recorded from the region indicated with the red box shown in **Fig. 2a** and shows a high density of small cavities with the average diameter of ~2±1.5 nm. The cavities were observed to be homogenously distributed over a depth range of ~300-1200 nm as shown in **Fig. 2a**, whereas less voids were observed in the vicinity of the irradiated surface than in the shallow regions, as shown in **Fig. 2**, suggesting that the surface is an effective sink for point defects at the irradiation temperature. Also, no voids were observed beyond a depth of ~1300 nm which corresponds to the ion implantation peak shown in **Fig. 1**.

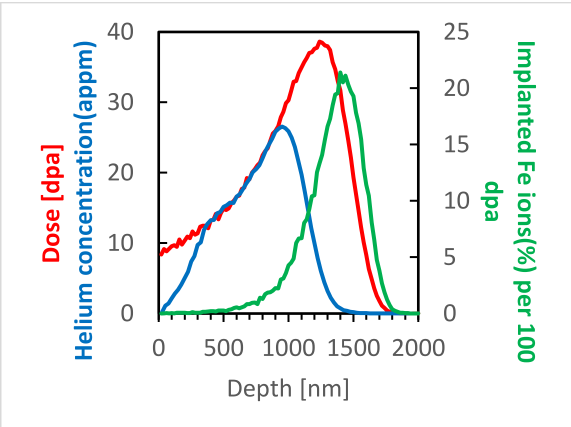

Fig.1 Damage profile (red) and ion implantation (green) as calculated by SRIM for 5 MeV Fe atoms into Fe21Cr32Ni model alloy. Helium implantation is shown with blue line.

Fig. 3 shows a bright field image of cavities along the grain boundary in the 21Cr32Ni sample irradiated to 16.6 dpa at 446ºC. It is clear that cavities formed preferentially in the vicinity of the grain boundary where cavity nucleation is favored due to the existence of more atomic space.

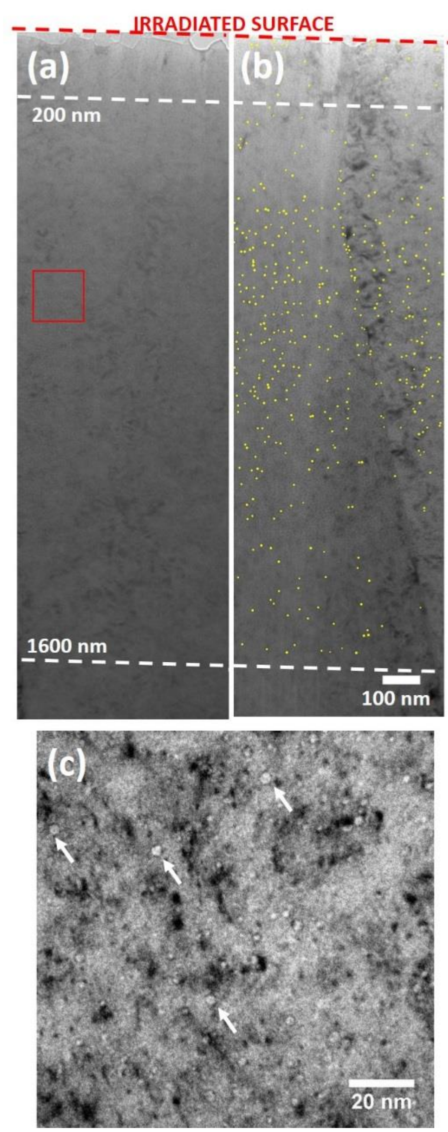

Fig. 2 (a) Cavity distribution in 21Cr32Ni model alloy after dual beam irradiation at ~440ºC dose in dpa, **(b)** cavities in (a) are highlighted for better visibility, **(c)** High magnification image of the cavities observed at ~600nm target depth indicated with red box in (a)

The percent swelling was calculated for each 100 nm bin from the irradiated surface to the maximum ion depth (~2 μm) using the following equation:

$$S(\%) = \frac{\frac{\pi}{6}\sum_{i=1}^{N} D_i^3}{A_i \times \delta - \frac{\pi}{6}\sum_{i=1}^{N} D_i^3} \times 100$$

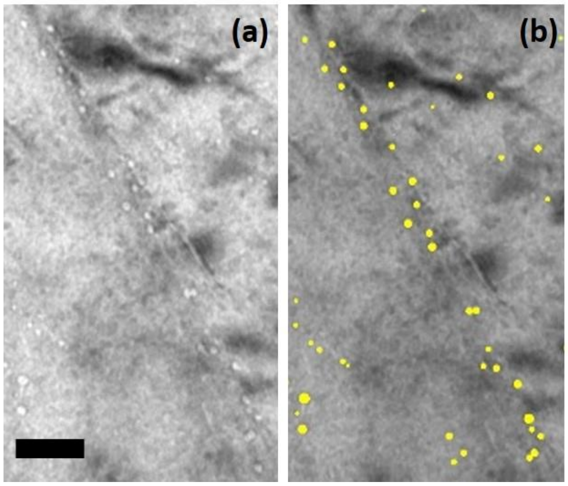

Fig. 3 (a) Dual beam irradiated 21Cr32Ni alloy microstructure showing cavities preferentially formed along the grain boundary, **(b)** voids in (a) are highlighted for better visibility.

The result of this calculation of the percent swelling is shown in **Fig. 4** calculated for the 21Cr32Ni austenitic alloy after dual beam irradiation to 16.6 dpa at 446ºC. The swelling increases with depth following the damage profile shown in **Fig. 2** and reaches a maximum value at a depth around ~500-600nm where helium concentration is higher. The swelling at the targeted dose (~600nm) is calculated as ~0.06%. The corresponding number density of the cavities is calculated as ~1.6×10^{21} m^{-3} which is similar to the data reported for 304, 316 type austenitic stainless steels irradiated with neutrons at ~375ºC [2, 3].

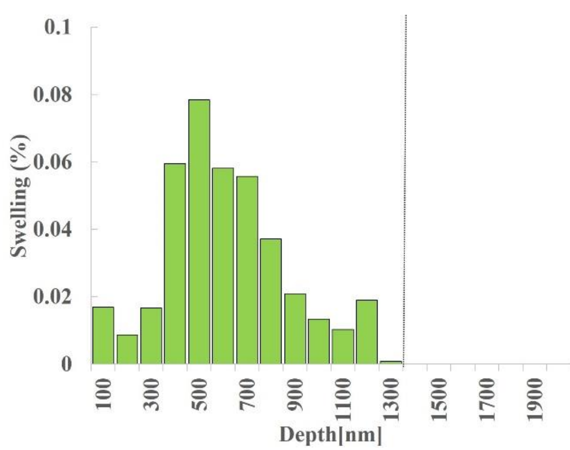

Fig. 4 Percent swelling vs. depth profile in dual beam irradiated 21Cr32Ni austenitic alloy.

Dashed line indicates the location of the maximum iron implantation. **Fig. 4** also shows that the swelling is suppressed near the ion implanted region due to the enhance recombination.

Conclusion

The microstructure of the 21Cr32Ni model alloy was investigated after irradiation to 16.6 dpa with 5 MeV Fe ions and co-injected helium ions at ~440ºC.

Dual beam irradiation of the alloy results in the formation of a high density of small cavities (~2 nm in size) e. Cavity formation was found to be preferential along the grain boundaries as well as dislocations.

The average void diameter and void number density calculated for the dual beam irradiated 21Cr32Ni model alloy at 446ºC is found to be similar to the data reported in the literature for the austenitic stainless steels (304, 316 type) irradiated with neutrons at ~375ºC.

Reference

[1] B. Sencer, G. Was, M. Sagisaka, Y. Isobe, G. M. Bond, F. Garner, J. Nucl. Mater., 414, (2011), 237.

[2] D. Edwards, F. Garner, E. Simonen, S. Bruemmer, Characterization of Neutron-Irradiated 300-Series Stainless Steels to Assess Mechanisms of Irradiation-Assisted Stress Corrosion Cracking: Volume 2: Core Components, EPRI, Palo Alto, CA, 2001. pp. 1001497.

[3] C. Pokor, Y. Brechet, P. Dubuisson, J. Massoud, A. Barbu, J. Nucl. Mater., 325, (2004), 19-29.

[4] M. Ayanoglu and A. T. Motta, 'Microstructural Evolution of the 21Cr32Ni model alloy under irradiation', J. Nucl. Mater., in press, *2018*