A Comparison of Cavity Formation in Neutron Irradiated Nanostructured Ferritic Alloys and Tempered Martensitic Steels at High He/dpa Ratio – G.R. Odette, P. Miao, T. Yamamoto (Department of Mechanical Engineering, University of California Santa Barbara), D. J. Edwards, R Kurtz (Materials Science Division, Pacific Northwest National Laboratory) and H. Tanagawa (Japan Atomic Energy Agency)

OBJECTIVE

The objective of this work is to characterize the effects of high helium to dpa ratios (He/dpa) on irradiation induced microstructural evolutions in nanostructured ferritic alloys (NFA), in this case MA957, and to compare these results, to corresponding evolutions in normalized and tempered martensitic steels (TMS) at fusion relevant dpa rates.

SUMMARY

Microstructural evolutions in NFA under neutron-irradiation, at fusion relevant He/dpa ratios and dpa rates, were characterized using a novel in-situ ^{59}Ni(n,α) reaction He-implanter technique. MA957 was irradiated at 500°C in HFIR to a nominal 9 dpa and 380 appm He. A high number density of ≈ 1 He-bubbles were observed. Comparisons of these results to the cavity structures in TMS F82H, described in a companion report [1], provide additional evidence that TMS may be susceptible to both low (fast fracture) and high (creep rupture) temperature embrittlement as well as void swelling at fusion relevant He concentrations, while NFA are much more resistant to these degradation phenomena.

PROGRESS AND STATUS

Introduction

Fusion neutron spectra produce ≈ 2000 appm He at 200 dpa, and the He/dpa ratio is even higher in spallation proton targets. Helium precipitates gas bubbles in the matrix and on dislocations, precipitate interfaces and grain boundaries [2]. Helium bubbles can act as formation sites for growing voids at lower irradiation temperature and creep cavities on stressed grain boundaries at high temperatures. Both 14Cr nanostructured ferritic alloys (NFA) and 9Cr tempered martensitic steels (TMS) are resistant, but not immune, to irradiation effects. For example, TMS are embrittled by irradiation hardening and grain boundary He at lower irradiation temperatures [3], and undergo significant void swelling at high He/dpa ratios in dual ion irradiations [4]. NFA are more resistant to irradiation damage at all temperatures. The irradiation damage resistance of NFA derives from their high sink densities, including stabilized dislocation structures, and large number of nm-scale Y-Ti-O enriched nanofeatures (NF) which trap He in small bubbles [2]. However, their overriding advantage is that NFA can operate at temperatures, up to 800°C, above the displacement damage-swelling regime [2]. At $T > 0.5T_m$, the primary irradiation damage issue is creep embrittlement due to accumulation of He on grain boundaries. The objective of this research is to characterize the transport, fate and consequences of He at spallation proton and fusion neutron relevant He/dpa ratios and dpa rates.

Experiment

A novel in-situ neutron irradiation He-implanter technique was used to characterize the effect of the He/dpa ratio on microstructural evolution in MA957, Eurofer97 [5,6] and F82H for irradiations in the High Flux Isotope Reactor (HFIR). Helium is uniformly implanted to a depth of 5 to 8 mm from thin NiAl coatings deposited on TEM discs. The He is produced by a two-stage thermal neutron (n_{th}) reaction sequence: $^{58}Ni(n_{th},\gamma)^{59}Ni(n_{th}, \alpha)$. The fast neutrons generate dpa at fusion relevant rates, and the corresponding He/dpa ratio can be easily adjusted from values of less than 1 to more than 50 appm He/dpa. The irradiated microstructure of MA957 [5] and Eurofer97 [6] and F82H irradiated in the HFIR JP26 experiment at 500°C to about 9 dpa and 380 appm He were characterized transmission electron microscopy (TEM). Eurofer97 was also characterized previously at ≈ 4.3 dpa and ≈ 90 appm He at 300 and 400°C.

Results

Previously published results showed a high density of $N_b \approx 3x10^{23}/m^3$ of very small $r_b \leq 1$ nm bubbles in MA957, primarily located on the NF interfaces [5]. A lower density $N_b \approx 1.5x10^{22}/m^3$ of larger $r_b \approx 4.3$ nm bubbles were found in companion TMS Eurofer97, along with even larger (>10 nm) faceted cavities, that are likely voids [5]. The NF are stable after a 9 dpa irradiation at 500 ºC and large bubbles do not form in the He implanted region of MA957. The estimated He contained in equilibrium bubbles was ≈ 130 appm in MA957, suggesting that N_b is underestimated, and ≈ 380 appm in Eurofer97, in agreement with the nominal amount. The bubble sizes decrease and number densities increase in Eurofer97 for irradiations at 400 and 300°C. Bubbles preferentially formed on dislocations in the TMS at all irradiation temperatures. Preliminary evidence suggests that loop formation in Eurofer97 is suppressed in the He implanted layer, compared to regions with low He [6].

The presence of a high number density of small bubbles in MA957 was recently confirmed, as shown in Figure 1a. These data are currently being quantified. As shown in Figure 1b, as in the case of Eurofer97, TMS F82H also show fewer (≈ 1 $x10^{23}/m^3$) and somewhat larger bubbles (2.0 nm) compared to MA957, along with what are likely polyhedral voids. Cold worked F82H had a slightly higher density of slightly smaller bubbles. The He bubbles are generally smaller and more numerous in F82H than in Eurofer97. The bubbles in F82H are estimated to contain about 340 appm He and are predominantly associated with dislocations and interfaces. Notably, Figure 1d shows that a boundary in F82H is highly decorated with small He bubbles, while a boundary in MA957, shown in Figure 1c, is relatively bubble free.

These preliminary observations strongly support the hypothesis that very high He concentrations can be managed in NFA. These studies are continuing, including comprehensive quantification of the irradiated alloy microstructures, as well as examination of additional alloys and irradiation conditions.

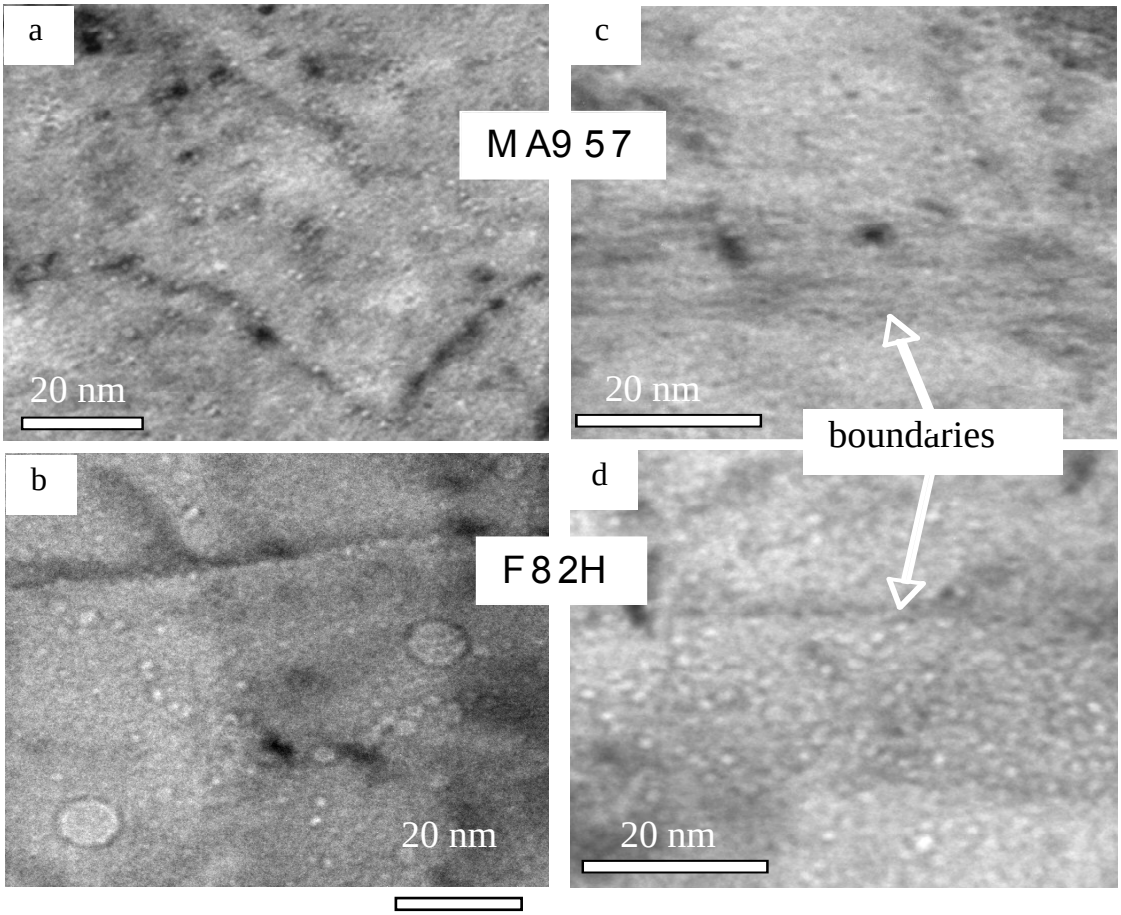

Figure 1 Cavity structures in NFA MA957 and TMS F82H: a and b) The (mostly) matrix cavities with smaller and more numerous bubbles in MA957 (a) compared to F82H (b); c and d) The boundary in F82H (d) has a high concentration of bubbles while the boundary in MA957 (c) is much cleaner.

Future Work

These results barely scratch the surface of what can be learned about the transport, fate and consequences of He using the in situ injection technique. Thus in addition to further characterization studies of the alloys and irradiation conditions described in this and previous publications, a major future effort will be directed at characterizing the large matrix of other alloys and irradiation conditions from the JP26 and JP27 experiments, that contained a total of ≈ 360 He injected TEM discs.

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