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Void swelling and microstructure evolution at very high damage level in self-ion irradiated ferritic-martensitic steels

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HIGHLIGHTS

- Void evolution was analyzed in self-ion irradiated HT9, T91 and T92.
- G phase and dislocation continued to evolve up to 650 dpa.
- M_2X precipitates inhibited void nucleation in T91 and T92.

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ABSTRACT

The void swelling and microstructure evolution of ferritic-martensitic alloys HT9, T91 and T92 were characterized following irradiation with Fe^{++} ions at 460 °C to damage levels of 75–650 displacements per atom with 10 atom parts per million pre-implanted helium. Steady state swelling rate of 0.033%/ dpa was determined for HT9, the least swelling resistant alloy, and 0.007%/ dpa in T91. In T91, resistance was due to suppression of void nucleation. Swelling resistance was greatest in T92, with a low density (~1×10^{20} m^{-3}) of small voids that had not grown appreciably, indicating suppression of nucleation and growth. Additional heats of T91 indicated that alloy composition was not the determining factor of swelling resistance. Carbon and chromium-rich M_2X precipitates formed at 250 dpa and were correlated with decreased nucleation in T91 and T92, but did not affect void growth in HT9. Dislocation and G-phase microstructure evolution was analyzed up to 650 dpa in HT9.

1. Introduction

Advanced reactor concepts are expected to reach damage levels greater than 200 displacements per atom (dpa) [1]. Understanding the effect of such high damage on cladding and duct is essential to maintaining safety and longevity of components. There are challenges to conducting reactor irradiations to such high damage levels, including the high cost and long irradiation times required and the high residual radioactivity making post-irradiation characterization difficult and expensive. These challenges to test reactor irradiation have led to increased interest in ion irradiation as a surrogate [2]. Self- or heavy-ion irradiations are unique in that they can reach damage rates of 10^{-3} dpa/s and above, allowing accelerated irradiation testing with well-controlled temperature, damage rate and total damage level. They have also been shown to successfully form many neutron-induced microstructure features [3–7]. In particular, ion irradiations are well suited to interrogating high damage phenomena, such as void swelling, which has a long incubation period for nucleation of cavities in swelling resistant materials such as ferritic-martensitic (F-M) alloys [3,7].

Due to the limited damage rates in test reactors, void behavior in swelling resistant materials in a neutron reactor environment has not been studied systematically at damage levels above 200 dpa where appreciable swelling (>1%) is expected. Prior to this work, nearly all comparisons between alloys have been performed in the nucleation regime of void evolution with little examination of void behavior in the so called steady state/linear swelling regime. However, some limited data [8–16] are available from neutron irradiations at the Fast Flux Test Facility (FFTF) and the High Flux Isotope Reactor (HFIR) and are provided in Fig. 1. Several key features are noted. There is an overall tendency to swell at a rate of

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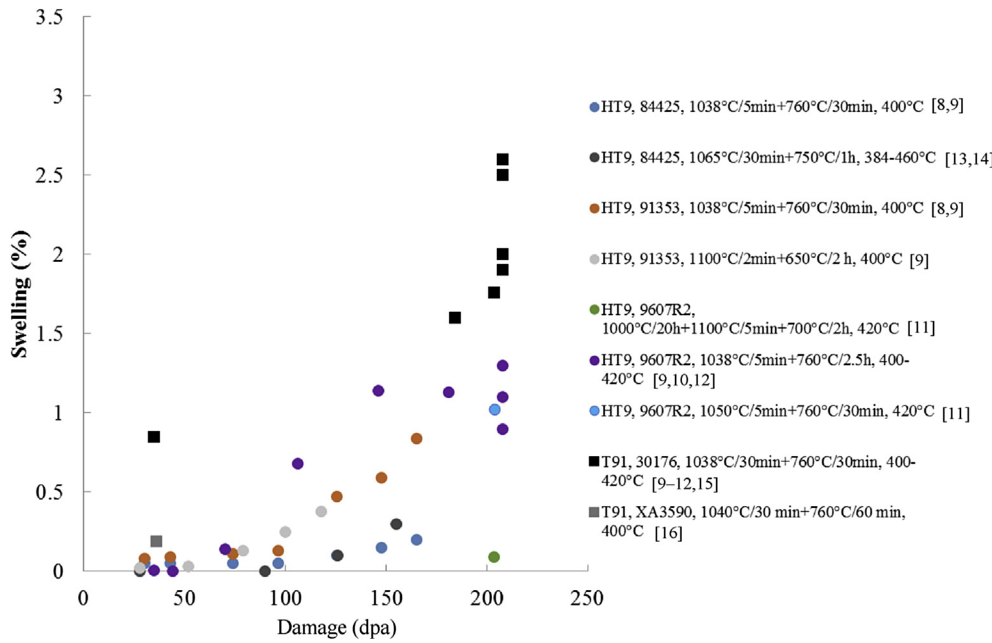

Fig. 1. Summary of HT9 irradiated in-reactor up to 208 dpa at temperatures varying from 400 to 443 °C.

~0.01%/dpa, though there is much scatter in the data. In general, there is a large variation in the amount of swelling between alloys (HT9 and T91), though T91 tends to swell more for a given damage level. There are also variations in terms of different heats of the same alloy. Finally, there is variation in single heats undergoing different heat treatments, which is best illustrated by a single study performed by Gelles [11] on commercial alloys (HT9 and T91) irradiated at FFTF up to approximately 200 dpa. A single heat of HT9 (9607R2) with two different heat treatments was examined: RFFL (Heat 9607R2, 1000 °C/20 h + 1100 °C/5 min + 700 °C/2 h) and RHFL (Heat 9607R2, 1050 °C/5 min + 760 °C/0.5 h) and compared to a third heat/heat treatment of T91 designated as PTFL (Heat 30176, 1040 °C/1 h + 760 °C/1 h). Overall, the T91 (PTFL) swelled the most when measured via the density change method with 1.76% average swelling. This is compared to the RHFL heat treatment of HT9, which swelled 1.02% and the RFFL heat treatment of HT9 that swelled by only 0.09%. This result indicates that heat treatment can be at least as important as alloy composition in terms of swelling resistance. Additional evidence is that the HT9-RHFL and T91-PTFL heat treatments, which swelled the most, also had the most similar heat treatments, with shorter tempering and austenization times. However, swelling in any of the 3 alloys/heats/heat treatments examined likely had not reached the steady state swelling regime, so this was a measurement of the resistance to void nucleation rather than an examination of the steady state swelling regime. However, the extent of that increase is not yet well understood.

Aside from neutron irradiation results, Smidt et al. [17] compared swelling behavior of HT9 and EM12 (nominally 9Cr-2MoVNb whereas T91 is nominally 9Cr-1MoVNb) under 2.8 MeV Fe^{+} ion irradiation. Heat treatments for both alloys varied only in tempering treatment: 780 °C/150min for HT9 vs. 755 °C/90 min for EM12. HT9 tended to swell more (peak swelling of 4.7% vs. 2.6% for EM-12 at 500 °C, 250 dpa) and had a broader temperature distribution where voids formed. The overall swelling rate was highly linear from 40 to 250 dpa for both alloys, and the swelling rates were very similar (0.011%/dpa for HT9, 0.017%/dpa for EM-12), which is in agreement with the results from neutron irradiations presented in Fig. 1. However, these results included the peak damage region, where the injected interstitial has been shown to have a suppressive effect on swelling [3,18–20].

A similar study performed by Toloczko et al. [7] compared the swelling behavior of HT9 to EP-450 under 1.8 MeV Cr^{3+}-irradiation up to 500 dpa. EP-450 is a duplex alloy consisting of ferrite and tempered martensite. Both alloys exhibited an extended nucleation regime (~150 dpa for EP-450 vs. ~400 dpa for HT9) and a final swelling rate of 0.2%/dpa, which is consistent with swelling rates predicted by Garner et al. based upon neutron irradiations of Fe-Cr binary alloys. [21], but is inconsistent with the neutron irradiation results presented in Fig. 1. Additionally, both Garner et al. [21] and Toloczko et al. [7] have hypothesized that the alloying affects only the length of the nucleation regime rather than steady state growth rate of void swelling.

With the exception of the last two ion irradiation studies [7,17], damage has been limited to 200 dpa or below, and as a consequence, the high damage and steady state swelling regime has not been examined systematically. Furthermore, previous analyses have focused on void swelling behavior only, with little examination of other microstructure features including the dislocation and precipitate microstructure, which are interconnected with the void microstructure and therefore must be understood and related to the response of such materials under neutron irradiation to better understand the observed swelling response under ion irradiation. Therefore, the objective of this paper is to present the microstructure evolution of HT9 and analyze the variations of swelling behavior between HT9, T91 and T92 irradiated with self-ions up to 650 dpa. A detailed study of the interaction of the various evolving microstructural features will be the subject of a future paper.

2. Experimental

Three different ferritic martensitic alloys were examined in this study. Alloy HT9 (Heat 84425 used in duct ACO-3 in FFTF) [13,14] was heat treated at 1065 °C/30 min followed by air cooling to room temperature followed by tempering at 750 °C/60 min and air cooling to room temperature, which resulted in a tempered martensitic structure, with some retained δ-ferrite [13,22]. This