



Emulation of reactor irradiation damage using ion beams

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Progress in understanding radiation damage in structural materials is hampered by the lack of test reactors, long irradiations and high cost. Here we show that through strict control of experimental parameters and accounting for He production and damage-rate differences, the microstructure of ion-irradiated ferritic–martensitic steel closely resembles that created in-reactor across the full range of microstructure features. The level of agreement establishes for the first time the capability to tailor ion irradiation to emulate in-reactor radiation damage.

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Fulfillment of the promise of advanced nuclear reactors with major improvements in safety, economics, waste generation and proliferation security, and life extension of existing light water nuclear reactors rest heavily on understanding how radiation degrades the materials that serve as the structural components in reactor cores [1,2]. In high-dose fission reactor concepts such as the sodium fast reactor, core internal components must survive up to 200 dpa¹ of damage at temperatures in excess of 400 °C, Figure 1. The traveling wave reactor pushes that limit to 600 dpa. At such high damage levels, the formation and growth of voids will affect the dimensional stability of components, the nucleation and growth (or dissolution) of precipitates will alter composition locally and can either embrittle or weaken the alloy, and both phenomena are affected by the evolving dislocation microstructure [3]. In some alloys these processes develop at low doses, but void swelling and radiation-induced precipitation may emerge only after high doses (100 dpa) [4,5]. While an understanding of the microstructural evolution of alloys under irradiation remains a major challenge to the integrity of reactor core components, a

more pressing problem is the diminishing capability to study these processes.

Radiation effects research is traditionally conducted by irradiating samples in test reactors, followed by comprehensive post-irradiation characterization. Predictive modeling of the radiation damage process helps to reduce the need for a full suite of experiments covering the entire parameter space. Several increasingly serious barriers are impeding the advancement of our understanding of radiation effects. The first is a paucity of worldwide test reactor capability, especially in the US, for addressing the unknowns in advanced reactor concepts. The US has only two test reactors capable of producing damage at a maximum rate of 8 dpa/year. Available reactors worldwide can only reach 20 dpa/year, making access to the required damage levels prohibitively time-consuming and expensive. Second, test reactors cannot create radiation damage significantly faster than that in commercial reactors, meaning that radiation damage research cannot “get ahead” of problems discovered during operation. Both of these factors conspire to create the third barrier: the extremely high cost of irradiation and post-irradiation characterization of highly radioactive samples.

A promising solution to the problem is the use of ion irradiation as a surrogate for neutron irradiation. Ion irradiation can yield high damage rates with negligible

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¹ A dpa (displacement per atom) is a unit of damage that represents the average number of times an atom is displaced from its lattice site.

(proton irradiation) or no (heavy ion irradiation) residual radioactivity and at very low cost. The use of ion beams for radiation damage study dates back to the 1960s and includes numerous significant contributions to our understanding of radiation effects (e.g. [6–9]). The advantages of ion irradiation are many: damage rates 10^4 times that of reactor irradiation can be attained, which means that 200 dpa can be reached in days instead of decades. Because there is little or no activation samples are easily handled, reducing the cost associated with post-irradiation characterization. Control of ion irradiation experiments (temperature, damage rate, damage level) is much better than irradiations in reactor and damage can even be observed in situ. However, the idea of using ion irradiation as a surrogate for neutron irradiation is relatively new and success requires both a high degree of experiment control and a systematic approach to accounting for the differences between reactor- and accelerator-based irradiations. Capturing the full extent of the entire irradiated microstructure created in-reactor has not yet been attempted. This paper presents a “formula” for emulating reactor irradiation with well-controlled ion irradiation.

Accelerated irradiation has several challenges, the most significant being the effect of an accelerated damage rate on the resulting microstructure, and the need to account for important transmutation reactions that occur in-reactor. Gas production by transmutation can be emulated by pre-injection, or better still, by simultaneous gas implantation with damage creation. The damage rate is the larger issue and is resolved using the invariance theory derived by Mansur [10–12]. According to this theory, a change in the value of an irradiation variable from reactor conditions is accommodated by a shift in other variables with the goal of preserving the aggregate behavior of defects during irradiation. Such shifts were constructed for specific microstructure processes such as radiation-induced segregation or void growth. In both cases, the higher dose rate in an accelerator relative to a reactor requires a corresponding increase in temperature to achieve the same result [6]. The analysis assumes a steady state and that the irradiation is in either the recombination-dominant or sink-dominant regime. Damage rate differences may also result in a change in the dose to nucleation of defect clusters, but there is little theoretical or computational modeling guidance for the nucleation process. Nevertheless, a growing body of evidence for low-dose (<10 dpa) proton irradiation [13–18] has shown that the microstructural features (dislocation loops, precipitates, voids, radiation-induced segregation) and properties (hardness, stress corrosion cracking susceptibility) are in excellent agreement with reactor irradiation when the damage-rate difference is of the order of 100. We report here on the application of the theory to the high-dose and high-dose-rate regime in experiments conducted on a specific heat of ferritic–martensitic alloy HT9 that was used both in-reactor and in controlled ion irradiation experiments to determine the ion irradiation conditions that emulate the in-reactor irradiation. It is important to emphasize that both the alloy heat and heat treatments were identical for both the reactor irradiation and the ion irradiation, removing heat-to-heat variability as a potential source of disagreement (see below).

Ferritic–martensitic alloy HT9 (Fe–12Cr–1Mo) heat 84425 was used in the hexagonal fuel bundle duct labeled ACO-3, in the Fast Flux Test Facility, Hanford, WA (FFTF). It was heat treated at 1065 °C/30 min/air cooling followed by 750 °C/60 min/air cooling. The fuel bundle was irradiated in several locations in the FFTF over the time period 1985–1992 during which it accumulated a total damage of about 155 dpa at an average temperature of 443 °C [5]. Pieces from an archival section of this same duct were irradiated in a 1.7 MV tandem accelerator with self-ions (Fe^{2+}) at 5 MeV at a temperature of 460 °C and to a dose of 188 dpa determined using SRIM [19] in the Kinchin–Pease mode [20]. At this energy, Fe^{2+} ions come to rest at a depth of $\sim 1.6 \mu\text{m}$ below the surface. The ion irradiation temperature represents a 17 °C increment above the neutron irradiation temperature, as predicted by invariance theory [12]. Temperature was closely monitored throughout the irradiation using a 2-D infrared thermal imager and ion beam current was monitored before and during the experiment. To emulate in-reactor transmutation, He was implanted prior to ion irradiation to a concentration of 1 at.ppm over a depth range of 300–1000 nm by varying the implantation energy over five different values. This amount of He was set below that generated in-reactor to compensate for the initially high He/dpa ratio in the ion irradiation experiment. Figure 2 shows the damage profile, implanted He distribution and the injected interstitial concentration as a function of depth. Note that the variation of He content is $\sim 10\%$ of the average and is due to the different energies used for implantation.

Principal features of the irradiated microstructure consisted of dislocation loops, precipitates and voids, which were characterized using transmission electron microscopy (TEM) with a variety of techniques: two-beam bright-field imaging (dislocation loops), dark-field imaging (precipitates), and bright-field through-focus or high-angle annular dark-field imaging (voids). Atom probe tomography (APT) was used to determine precipitate composition. Following ion irradiation, samples for characterization were made by focused ion beam (FIB) milling in cross-section to span the full depth of the irradiation. The region of the damaged zone selected for microstructure analysis (500–700 nm from the surface) was determined after considering the factors that can affect the validity of measurements. Analysis of void data in this study and MD simulations by Stoller et al. [21] have indicated that the surface effect on defects can

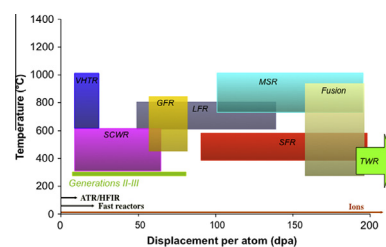


Figure 1. Schematic of the temperature-dpa requirements for various reactor concepts and the achievable annual damage rates in different reactors and with ion irradiation. VHTR = very high temperature reactor, SCWR = supercritical water reactor, GFR = gas fast reactor, LFR = lead fast reactor, MSR = molten salt reactor, SFR = sodium fast reactor, TWR = traveling wave reactor, Generations II-III = present day light water reactors, ATR/HFIR = advanced test reactor, high flux isotope reactor.

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