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Radiation-induced material changes and susceptibility to intergranular failure of light-water-reactor core internals

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Abstract

Current understanding of radiation-induced material changes that occur in light-water-reactor (LWR) core components is critically reviewed and linked to intergranular failure processes. Although the basic science of radiation damage processes in metals is reasonably well established, accurate prediction of microstructures, microchemistries and mechanical property changes in complex stainless alloys during irradiation at LWR temperatures is not possible at present. Mechanistic understanding of these radiation-induced changes in commercial alloys is considered to be of paramount importance for the mitigation of the intergranular environmental cracking that occurs in service. Fundamental research is needed to define defect–solute interactions and microstructural evolution at intermediate temperatures and dose rates pertinent to LWRs where transient effects often dominate behavior. In addition, it is essential that radiation effects on matrix microstructure and microchemistry and grain boundary microchemistry be understood. Finally, a stronger emphasis on accurately quantifying radiation effects on environmental cracking mechanisms and kinetics is needed. © 1999 Published by Elsevier Science B.V. All rights reserved.

1. Introduction

This review is based on a Research Assistance Task Force meeting jointly supported by the US Department of Energy, Office of Basic Energy Sciences, Division of Materials Science and the Electric Power Research Institute in March 1998. A primary goal of the task force was to identify basic radiation materials science needs driven by practical nuclear engineering concerns in electric power reactors. Mechanistic understanding of radiation damage processes in light-water-reactor (LWR) core-component alloys is a critical underpinning issue for the effective operation of existing commercial reactors as well as for the next generation of nuclear power systems. The task force included a diverse team of scientists and engineers from electric power utilities, reactor component vendors, industrial research organizations, universities and national laboratories. Members with complementary disciplines and expertise addressed key structural integrity issues concerning reactor core internals. It was clear from the meeting conclusions that sufficient fundamental understanding of radiation effects on materials behavior at LWR temperatures and dose rates is lacking for prediction of component properties and mitigation of service failures. Linking this knowledge to the mechanisms of environmentally induced, intergranular cracking was also recognized to be of vital importance. The current review assesses the critical radiation-induced material changes that are believed to influence LWR core component cracking and identifies basic research that is needed to elucidate failure mechanisms.

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2. Current understanding of reactor core component failures

A growing concern for electric power utilities worldwide has been core component degradation in nuclear power reactors which generate up ~17% of the world's electric power. Failures have occurred after many years of service in boiling water reactor (BWR) core components and, to a lesser extent, in pressurized water reactor (PWR) core components. These failures occur in stainless iron- and nickel-base alloys exposed to a significant flux of neutron radiation in the reactor coolant environment. The environment is typically oxygenated or hydrogenated water at about 290°C, but can range from 270°C to 370°C in specific locations. The chemistry of these environments can also be modified in crevice situations where component failures are often observed. Since cracking susceptibility requires the combination of radiation, stress and a corrosive environment, the failure mechanism has been termed irradiation-assisted stress corrosion cracking (IASCC). Until recently, the components affected have been either relatively small (bolts, springs, etc.) or designed for replacement (control blades, instrumentation tubes, etc.). Recent reviews [1–6] have been published which describe much of the current knowledge related to IASCC service experience and laboratory investigations. These reviews effectively reveal the limited amount of well-controlled experimentation that exists on well-characterized materials. Moreover, there are inherent difficulties to quantify SCC response that preempted making direct comparisons between radiation-induced changes and cracking behavior. This lack of critical experimentation makes it imperative that underpinning science be used to accelerate mechanistic understanding which is regarded as the only reliable approach to mitigate failures. Key aspects of the IASCC process are discussed below.

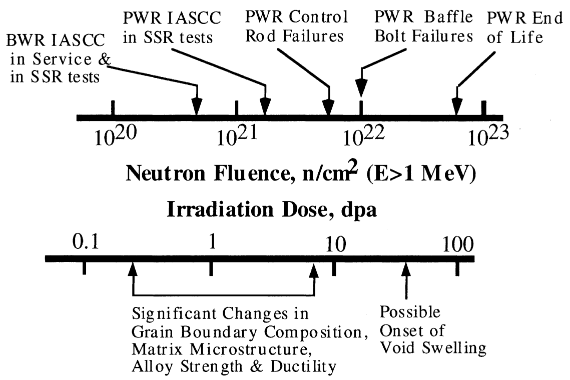

Fig. 1. Neutron fluence effects on irradiation-assisted stress corrosion cracking susceptibility of Type 304SS in LWR environments.

The importance of neutron fluence on IASCC has been well established. Intergranular (IG) SCC is promoted in austenitic stainless steels when a critical threshold fluence is reached although the threshold varies with stress, water chemistry, etc. The effect of neutron fluence on IG failure in high-temperature water environments is illustrated in Fig. 1. Cracking is observed in BWR oxygenated water at fluences above 5 x 10^{20} n/cm^2 ($E > 1$ MeV), which corresponds to about 0.7 dpa. A comparable threshold fluence for IASCC susceptibility has been reported for high-stress, in-service BWR component cracking and during ex-situ, slow-strain-rate SCC testing of irradiated stainless steels. This indicates that while in situ effects (e.g., radiolysis) are important, only 'persistent' radiation effects (material changes) are sufficient to produce high IASCC susceptibility.

As in classical SCC behavior [7], the aqueous environment chemistry and component stress/strain conditions also strongly influence observed cracking. Numerous attempts to reproduce IG cracking of highly irradiated stainless steels in inert environments have been unsuccessful. Water chemistry and electrochemical potential effects are apparent by a sharp increase in IASCC susceptibility with increasing dissolved oxygen. Cracking is typically not observed for high-stress components or during post-irradiation slow-strain-rate (SSR) SCC tests in hydrogenated water (BWR hydrogen water chemistry or WR water) until a fluence approximately 4 times greater than that observed in oxidizing water conditions.

Recent work has enabled many aspects of IASCC phenomenology to be explained (and predicted) based on the experience with IGSCC of non-irradiated stainless steel in BWR water environments. This approach has successfully accounted for radiation effects on water chemistry and its influence on electrochemical corrosion potential. However, the specific radiation-induced microstructural and microchemical changes that promote IASCC susceptibility are largely unknown. Well-controlled IASCC data from properly irradiated, and properly characterized, materials is sorely lacking due to the experimental difficulties and financial limitations related to working with highly activated materials. Many of the important metallurgical, mechanical and environmental aspects that are believed to play a role in the cracking process are illustrated in Fig. 2. Since only persistent material changes are required for IASCC to occur, in-core processes such as radiation creep and radiolysis influence cracking, but are not controlling mechanisms. The following sections examine the current understanding of persistent material changes that are produced in stainless alloys during LWR irradiation based on the fundamentals of radiation damage and existing experimental measurements.

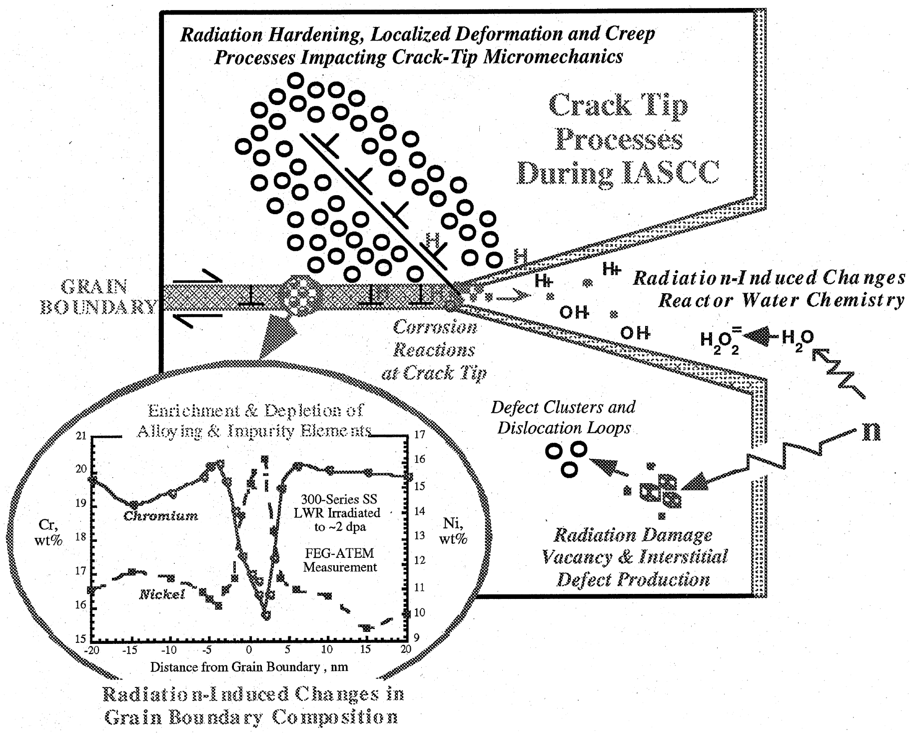

Fig. 2. Schematic illustrating mechanistic issues believed to influence crack advance during IASCC of austenitic stainless steels.

3. Basic mechanisms of radiation damage

Displacement of atoms during neutron irradiation is the basis for changes in the material condition of LWR core internals. The unit of displacements per atom (dpa) has been established as the preferred fundamental measure of material response to irradiation exposure and has been accepted for a wide variety of irradiation particle types including heavy ions, protons, electrons and neutrons. Each displaced atom produces one vacancy and one self-interstitial atom (SIA) known as a Frenkel pair. Frenkel pair production, migration, aggregation and annihilation result in changes in microchemistry and microstructure that depend on the irradiated alloy (bulk composition, microstructure and microchemistry) and the irradiation environment (temperature and dose rate). Radiation-induced microstructural evolution is controlled by the *partitioning* of vacancies and SIAs as shown in the flow chart in Fig. 3. This partitioning leads to the formation of clusters, dislocation loops and cavities. On the other hand, microchemical evolution is controlled by the *migration* of vacancies and SIAs to sinks (such as grain boundaries, dislocations, precipitates and surfaces) which induces local composition changes.

Considerable basic science insights have been obtained through atomistic simulations of displacement cascade events [8,9]. These simulations combined with point-defect rate theory models can elucidate many of the fundamental mechanisms impacting microstructural and microchemical evolution. However, there remains a significant disconnect between mechanistic studies conducted predominately on pure metals under simple irradiation conditions and the complex reality of a multi-component fabricated stainless steel structures in a commercial LWR.

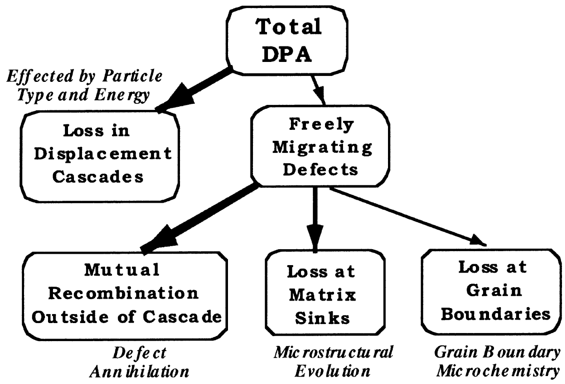

Fig. 3. Flow chart showing the fate of atoms displaced by the cascade event and subsequent creation of radiation-induced microstructure and microchemistry.

Although many problems associated with the quantitative application of these fundamental approaches to LWR component issues exist, they are perhaps the only tools available to assess radiation damage mechanisms and the dynamics of defect production, migration, aggregation and annihilation. Cascade models are well suited for describing the production of Frenkel pair defects based on the energy of incident bombarding particles. The process of tracking short-term annealing and clustering of these Frenkel pairs can be addressed by hybrid atomistic (Monte Carlo) and rate theory models, but they are unable to quantitatively predict LWR microstructural evolution in industrial alloys.

4. Radiation-induced microstructural evolution

4.1. Mechanistic issues under LWR conditions

Vacancy and interstitial clusters can form directly within a damage cascade or indirectly between cascades by nucleation and growth processes [10]. During cascade cooling, clusters form directly from the inhomogeniety of the cascade. On a longer time scale, cluster nucleation and growth occur by synergistic vacancy and interstitial annihilation processes. Long-term microstructural evolution requires a mechanism for partitioning vacancies and interstitials such that the vacancy and interstitial aggregates can coexist.

Differences in migration kinetics, sink strength and defect clustering control microstructural evolution at relatively low LWR irradiation temperatures and dose rates. At low temperature, slow vacancy migration and slow vacancy emission prevent development of large vacancy aggregates and allow interstitial loop growth to occur up to a few dpa. As dose increases, additional vacancies evaporating from their clusters can migrate sufficient distances to annihilate at interstitials and interstitial clusters. Interstitial loop growth is thus suppressed and loop size saturates with increasing dose. The interstitial loops dominate the defect sink strength such that both vacancies and interstitials annihilate at nearly equal rates at dislocation loops. At doses above a few dpa, cavities can develop in the matrix. Cavity growth occurs synergistically with interstitial loop growth by preferred absorption dictated by partitioning.

Microstructural evolution in LWRs is difficult to model because the controlling events are intermediate between rapid cascade events (atomic displacements and cluster dissolution) and persistent growth of partitioned defect aggregates (interstitial loop growth, loop unfaulting, network dislocation development and cavity growth). Cascade models track detailed atomic movement during primary damage production but are not capable of tracking long-term microstructural development. On the other hand, rate theory models are well suited for describing microstructural evolution when defect partitioning controls the sustained cluster growth after cluster nucleation has occurred. Unfortunately, microstructural evolution during LWR irradiation is influenced by the transient effects of nucleation, limited vacancy kinetics and local microchemical changes making the defect clustering processes difficult to predict both spatially and temporally. Consequentially, the most important insights into mechanisms for microstructural evolution have emerged from experimental measurements, not from independent model predictions.

4.2. Measurements of radiation-induced microstructural evolution

Several reviews have been published on microstructural evolution in irradiated stainless steels [11–14] with the majority of data for temperatures and doses much higher than those relevant to LWRs. The temperature regime that is important for LWR operation is 270°C to 370°C. Higher temperatures may be reached from gamma heating in certain thick components such as PWR baffle plates and formers. This temperature range represents a transition region between what is commonly termed as low-temperature (50–300°C) and high-temperature (300–700°C) behavior. Typical radiation-induced microstructural features in austenitic stainless steels are dislocation loops, network dislocations, cavities (bubbles and/or voids) and precipitates. Near 300°C, the radiation-induced microstructure changes from one dominated by small clusters and dislocation loops to one containing larger faulted loops plus network dislocations and cavities at higher doses, as illustrated in Fig. 4.

Small loops and clusters (<4 nm in diameter) along with larger dislocation loops are the most common low-temperature microstructural feature. It appears that the small features (often described as 'black spot' damage) are primarily vacancy clusters (created directly from the

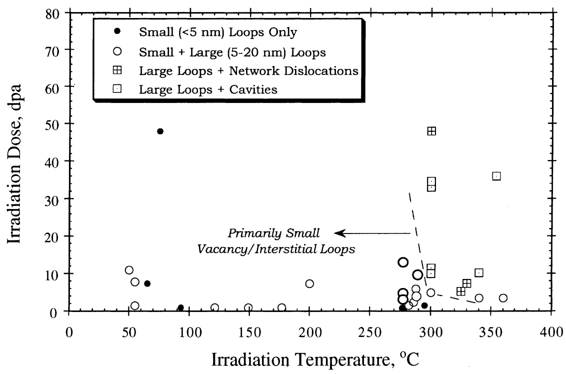

Fig. 4. Summary of reported defect structures in 300-series stainless steels as a function of irradiation dose and temperature [15–25].

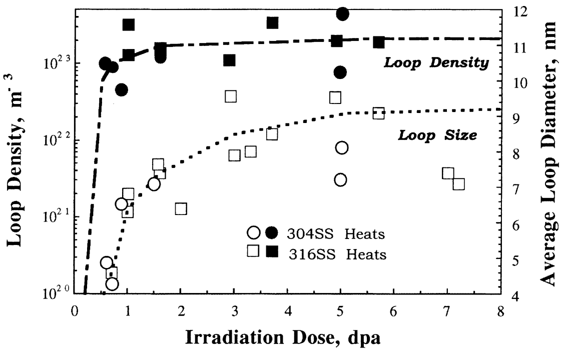

Fig. 5. Measured change in density and size of interstitial loops as a function of dose during LWR irradiation at 280°C [15].

collapse of a damage cascade during the irradiation process [12]) and interstitial loops. The larger dislocation loops (typically 4–20 nm in diameter) are faulted interstitial Frank loops that result from the clustering of SIAs. Interstitials are highly mobile and able to form complexes that can become loops, while vacancies are much less mobile. Interstitial loops increase in size with dose as a higher number of interstitials are absorbed than vacancies. An important transient behavior is the formation and stability of vacancy clusters with increasing dose. The irradiation-induced dislocation microstructure will tend to 'saturate' as interstitial loops become the dominant defect sink. If vacancies and interstitials annihilate at interstitial loops at the same rate, growth of existing loops will cease. The attainment of this saturation is reached at a relatively low dose (few dpa), as indicated by recent measurements on LWR-irradiated 300-series stainless steels [15] in Fig. 5. Radiation-produced cavities (bubbles and voids) and radiation-induced phases are not generally observed in the low-temperature regime for the low-dose conditions that have been reported.

The limited available data suggest that the saturation loop size is relatively insensitive to fluence at low temperatures. Loop sizes and densities become dynamically stable as a population when a balance is reached between new loop formation and the destruction of existing loops. As irradiation temperatures increase, the density of fine loops decreases and Frank loop size increases. Frank loops may unfault and form a dislocation network while new loops are continually being nucleated. The dislocation network can act as a sink for SIAs due to an elastic interaction between SIAs and dislocations.

Certain alloying or impurity elements can refine the dislocation microstructure during low-temperature irradiations. Phosphorus [26], as well as Ti and Nb [27], have been shown to increase interstitial loop density, while concurrently decreasing loop size. In conjunction with carbon, these effects are enhanced to an even greater extent. Undersize P (relative to the base metal atoms) is thought to bind with SIAs limiting mobility and growth kinetics, shifting network and cavity formation to higher temperatures [28,29]. Loop size appears to be stabilized and the growth and unfaulting of loops to form the dislocation network is inhibited. Adding Mo to an Fe–Cr–Ni alloy does not change the loop nucleation behavior, while Si promotes loop nucleation [30] in alloys without Mo. Silicon additions to Mo-containing alloys do not have the same effect. It is suggested that Mo traps Si atoms and prevents them from binding with SIAs. Available data show a complex synergistic behavior exhibited by various alloying additions that cannot be evaluated without systematic studies on high-purity alloys with individual elemental additions.

At typical LWR operating temperatures (275–290°C), the primary microstructural constituent can be summarized as a dense population of fine loops and clusters less than ~20 nm in diameter. Frank interstitial loops evolve in size to reach a 'saturation' diameter that depends on the specific irradiation conditions and alloy characteristics. Cavity (void or bubble) formation may be possible under these conditions, but few observations have been reported. However, this behavior appears to change at LWR-relevant dose rates when temperatures exceed 300°C. While data in this temperature range are very limited, a growing body of evidence shows that voids are likely to form at high dose levels in a variety of stainless steels [31–36].

The high density of radiation-induced defect clusters suppresses the supersaturation of vacancies at lower temperatures by enhancing the recombination of point defects at sinks. However at temperatures near 300°C, vacancy clusters in austenitic stainless steels become thermally unstable and begin to emit vacancies. This leads to a rapid evolution of the microstructure as the flux of vacancies to sinks increase. The fine loops that exist at lower temperatures disappear at higher temperatures. Over this same temperature range and at higher dose, bubbles and voids can begin to form and the dislocation structure evolves into a network structure as larger Frank loops unfault and become tangled. A high dose is generally required to reach a steady-state condition as there is considerable interaction between the microstructural components.

The formation of a fine dispersion of He bubbles (in reactors with high thermal neutron fluxes) is also an indication of the transition behavior near 300°C [37]. Bubbles have been observed following doses between 7 and 56 dpa at temperatures from 300°C to 330°C [26,28,38,39] depending on the He content in the stainless steel. Helium can be produced at low irradiation fluences by the ^{10}B reaction to form ^{4}He and ^{7}Li. This transmutation reaction decreases rapidly as the available

^{10}B is removed. The major He source thereafter is the high-energy threshold (n,α) reactions with all metallic elements, generally for energies above 6 MeV. A new He source is created by the production of ^{59}Ni from ^{58}Ni by thermal neutrons (via the two-step $^{58}Ni(n,\gamma)^{59}Ni(n,\alpha)^{56}Fe$ reaction sequence), which controls the total He at high doses. Nickel is also the major source of H via a threshold (∼1 MeV) reaction and a $^{59}Ni(n,p)$ reaction [40]. It has been recently suggested that H may promote cavity development synergistically with He [41].

The overall sensitivity of the microstructural evolution to minor variations in irradiation spectrum, dose rate and alloying composition increases with temperature. High doses are required to reach steady state as the microstructure goes from one dominated by dislocation structure to a bubble and void dominated structure. In general, increasing dose rate produces microstructural evolution at higher temperatures that is similar to samples irradiated at lower dose rate and at lower temperatures. The dislocation evolution will therefore occur more rapidly and bubble formation will be seen at lower fluence. Dislocation structures still appear to be the principal component of the microstructure during irradiations to moderate doses at temperatures below 350°C, while cavities become more important at higher fluences and temperatures.

Significant changes in defect microstructure evolution occurs over the LWR temperature range (270–370°C) as indicated by the transition from low- to high-temperature behavior described above. Unfortunately, there are few published measurements [15–25] of irradiation microstructures developed within this temperature range and several of these were on special alloys irradiated in test reactors. It is clear that much more data has been obtained at specific laboratories on LWR components, but is not accessible to the general community. It is important to note that the high-dose and higher temperature condition is of significant relevance to some PWR components where end of life conditions of isolated components may reach ∼80 dpa and temperatures up to 370°C.

4.3. Radiation-induced precipitation and phase instabilities

Radiation exposure of Fe- and Ni-base stainless alloys can accelerate or retard the precipitation of various second phases, modify existing precipitates, and produce new phases that do not form during thermal treatments [12,18,42]. A primary influence of radiation on second phase formation and stability will depend on solute segregation that will be described in the following section. For example, radiation-induced segregation (RIS) of Ni and Si to sinks in stainless alloys can lead to γ' or G phase precipitation and depletion of Cr can retard carbide precipitation at grain boundaries. These effects have been recently reviewed [11–13] and the analyses suggest that radiation-induced precipitation is unlikely below ∼400°C. Although characterization of materials after high-dose irradiations at pertinent temperatures is lacking, radiation-induced precipitation does not appear to be an issue for LWRs unless significant off-normal compositions are present in the stainless steel. The only second phase that is commonplace in 300-series stainless steels is metal carbides that are stable under LWR irradiation. However, high-strength Ni-base alloys such as alloy 718 have several second phases that dissolve (γ'), redistribute (γ'') or become amorphous (Laves) during LWR irradiation [43]. These changes can significantly influence the alloy properties.

5. Radiation-induced microchemical evolution

5.1. Mechanistic issues under LWR conditions

In contrast to microstructural evolution, microchemical evolution is a continuous process that does not progress through an ill-defined nucleation stage. The strength of defect–solute interaction and the kinetics of back diffusion control RIS. Back diffusion is rapid at distances greater than a few nm from the sink where radiation enhanced diffusion is dominant. Therefore, RIS-induced concentration gradients are confined to short distances from the sink. Near a sink, back diffusion is inhibited because of the locally depleted defect concentration and consequently steep concentration gradients result at grain boundaries. Rate theory calculations for RIS are based on knowledge of defect production, diffusion and defect/solute interactions. RIS models have been able to rationalize measured segregation in a wide range of alloys and irradiation environments.

Complex irradiation and thermal processes control grain boundary composition in austenitic stainless steels. RIS is a non-equilibrium process that is created by the flow of defects to sinks as shown in Fig. 6. Two important solute-defect interactions affect RIS: inverse Kirkendall and interstitial association [44]. Both occur concurrently, but one mechanism will likely dominate for a specific alloy composition. In the first case, vacancy exchange rates with solute atoms during its migration to sinks will control RIS. Slow diffusing elements enrich and fast diffusing elements deplete at grain boundaries. In the second case, elastic interactions between a solute atom and an interstitial or vacancy control RIS. Solutes that have significant under-size misfit enrich at grain boundaries at the expense of oversized solutes.

Because vacancies and interstitials are produced as Frenkel pairs, the flux of each to grain boundaries is similar in magnitude. Irradiation variables will affect

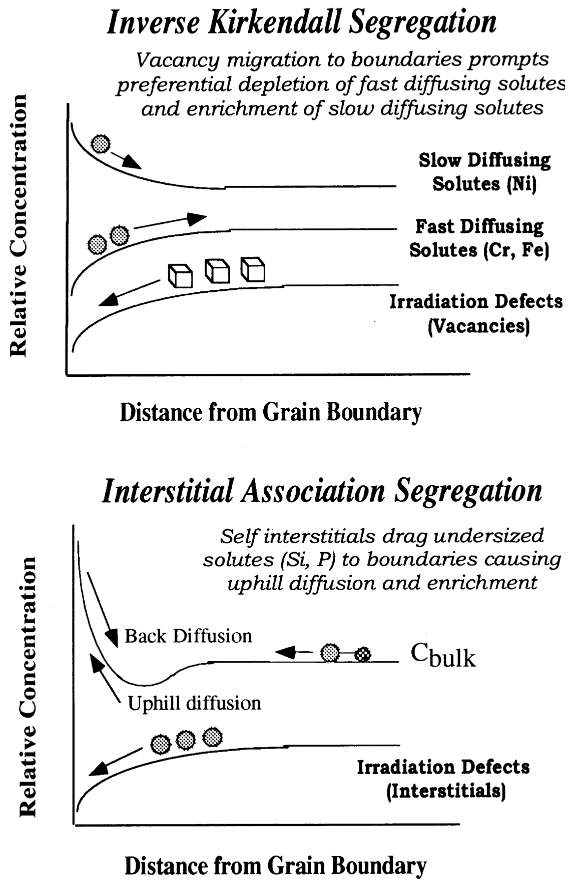

Fig. 6. Representations showing defect flows and sink composition changes for mechanisms of radiation-induced segregation.

each mechanism in a similar fashion. Temperature and irradiation flux are the primary factors controlling RIS. A map of the influence of temperature and flux is shown in Fig. 7 for particle irradiation types used in studies of IASCC. The need to use higher irradiation temperatures for higher flux experiments (protons and heavy ions) is evident. This 'temperature shift' with increasing flux is necessary to balance the production rate of defects with the annihilation rate of defects. Understanding of the temperature shift has allowed the use of laboratory particle irradiation for establishing quantitative RIS models for prediction of LWR RIS behavior.

A systematic review of alloy systems and RIS mechanisms reveals that the interstitial association mechanism qualitatively rationalizes observed segregation in most cases. However for alloy systems with minor solute size misfit and significantly different solute-vacancy exchange rates, RIS appears to be controlled by relative diffusivities in contrast to relative size misfit. In the case of ternary Fe–Cr–Ni alloys of interest to IASCC, it has been shown that the vacancy exchange mechanism effectively explains the observed major alloying element segregation [45–47]. It is important to note that other solutes in stainless alloys with significance size misfits (e.g., Si) do segregate by the interstitial association mechanism.

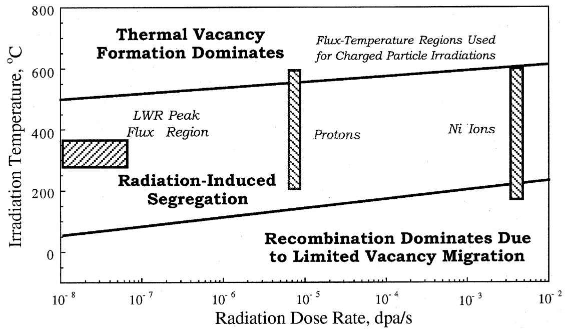

Fig. 7. Predicted radiation temperature and flux effects on segregation behavior in austenitic stainless steels. Typical flux-temperature regimes for LWR neutrons and charged particle irradiations are shown.

5.2. Measurements of radiation-induced microchemical evolution

Although basic mechanisms have been proposed, little quantitative information has been available to evaluate RIS to grain boundaries in neutron-irradiated stainless steels. Difficulties in working with irradiated materials and limitations in characterization techniques have produced data that can only suggest trends in segregation behavior. Composition profiles at grain boundaries are extremely narrow even after relatively high dose. Most measurements of these nm-scale profiles have been performed using analytical transmission electron microscopy (ATEM). The current generation of high-resolution field-emission-gun (FEG) TEMs has been shown to effectively measure these concentration gradients using energy dispersive X-ray spectroscopy as illustrated in Fig. 8 for RIS profiles in a 300-series stainless steel. Information on the primary alloying and impurity elements that segregate to stainless steel grain boundaries during LWR irradiations is summarized in the following sections.

Major alloying elements. Radiation-induced Cr depletion has been the focus of many IASCC studies because of its well-documented effects in promoting IGSCC in sensitized stainless steels under oxygenated BWR conditions. RIS results in a sharp change in interfacial composition at low-to-moderate fluences (0.1–5 dpa) with a much smaller rate of change seen at higher dose. ATEM measurements of neutron fluence effects on grain boundary Cr profiles are illustrated in Fig. 9. General Cr segregation in 300-series stainless steels is

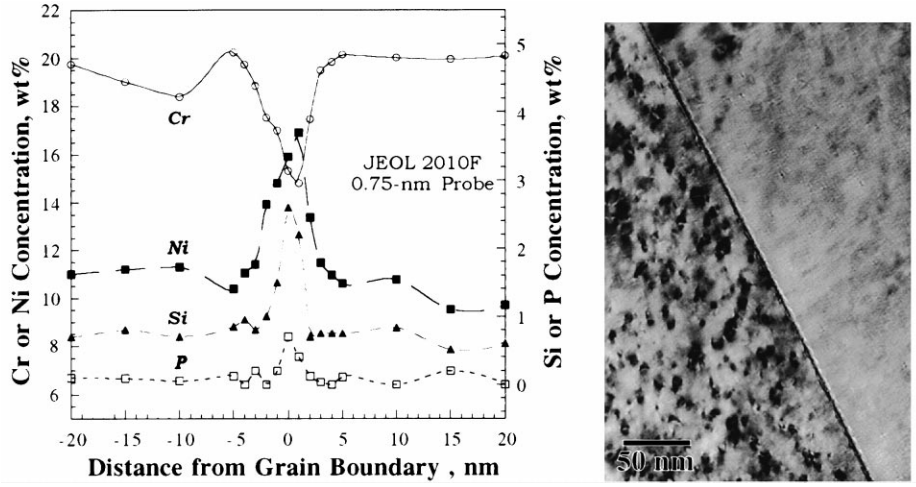

Fig. 8. Analytical transmission electron microscopy measurement of composition profiles across a grain boundary in a neutron-irradiated 300-series stainless steel.

summarized in Fig. 10 [50] showing considerable scatter for low-dose conditions due primarily to differences in the initial grain boundary composition. Taking into account the wide range of materials and starting conditions (typically not characterized), most data show a consistent exponential decrease in Cr content to ∼13 wt% with increasing fluence. Presegregation of Cr in mill-annealed stainless steels promotes the formation of W-shaped profiles (narrow depleted regions adjacent to an enriched boundary) at low dose and delays the development of a steady-state depletion profile [15,48–50]. The formation of a W-shaped profile also suggests that some type of binding for Cr and Mo within the boundary plane and interactions with cosegregated elements such as B and P [48].

Nickel enrichment and Fe depletion occurs concurrently with the Cr depletion described above. The slowest diffusing element Ni becomes enriched, while much faster Cr and somewhat faster Fe deplete. As a result, radiation-induced Ni levels can reach relatively high measured levels at boundaries (up to ∼30 wt%). Measured grain boundary Fe concentrations drop with increasing dose by an amount slightly less than that for Cr, consistent with their relative diffusivities.

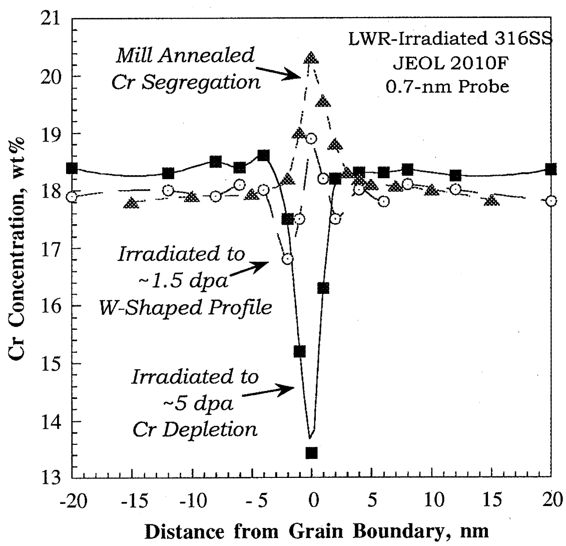

Fig. 9. Irradiation effects of Cr composition profiles across high-energy grain boundary in a commercial Type 316SS.

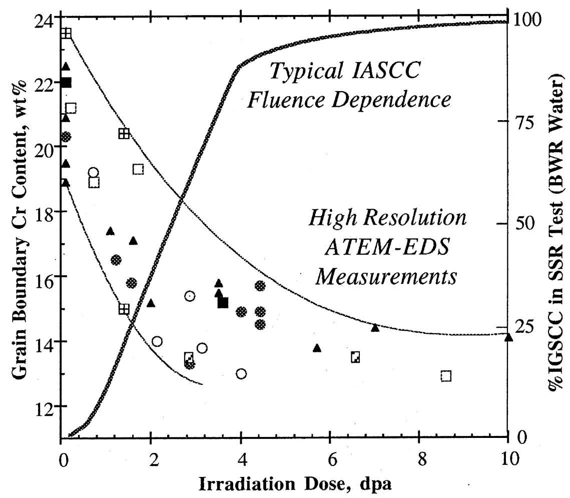

Fig. 10. RIS of Cr as a function of neutron irradiation dose in comparison to typical IG cracking for 304SS in SSR (high O_2) tests.

The inverse Kirkendall segregation mechanism based on vacancy exchange rates has been used to model nanoscale segregation profiles of major alloying elements in irradiated stainless steels. During irradiation of austenitic stainless steels, vacancy flow to grain boundaries causes enrichment of Ni and depletion of Cr and Fe at grain boundaries. These predicted and measured composition changes are consistent with known fast diffusion of Cr and slow diffusion of Ni in austenitic stainless alloys. Heavy ion [46], proton [47] and neutron [45] irradiation experiments have demonstrated that the inverse Kirkendall segregation mechanism is quantitatively consistent with several hundred measurements of grain boundary composition in irradiated austenitic alloys. Measurements of Fe, Cr and Ni RIS were made for a wide range of temperatures, doses, and alloy composition. The quantitative determination of relative diffusivities for Fe, Cr and Ni from RIS are consistent with the relative diffusivities determined independently from thermal annealing experiments. The agreement between the inverse Kirkendall model (without including interstitial association contribution) and grain boundary measurements suggests that the interstitial contribution to major alloy element RIS is not significant.

Transition element additions. Alloying elements such as Mn and Mo both show significant depletion at grain boundaries after irradiation. Manganese levels decrease both due to transmutation (at high doses) and due to RIS, prompting boundary levels to drop to very low levels. Unfortunately, Mn is a difficult element to quantitatively measure in a neutron-irradiated stainless steel by ATEM and composition profiles are not routinely reported. Strong inverse-Kirkendall RIS of Mn is expected due to its rapid diffusivity compared to Fe, Ni and Cr. Fast reactor irradiations have demonstrated that Mn is depleted at grain boundaries and void surfaces. This can lead to phase instability and property degradation in Fe–Cr–Mn austenitic stainless steels [51–53]. Measurements of RIS in a series of high-purity and commercial purity Type 316 stainless steel heats has shown strong Mo depletion with many showing boundary Mo depletion of more than 50% after irradiation to ~3 dpa [15,54]. This behavior is also consistent with the increased diffusivity of Mo and its large size misfit versus the base metal elements.

Impurity and other minor element segregation. Silicon and P are the most common minor elements found to be enriched at grain boundaries in neutron-irradiated stainless steels [55]. Silicon observations are more common because of its high bulk content (0.7–2.0 at.%) compared to P (0.01–0.08 at.%). Measured grain boundary concentrations for Si appear to increase with fluence (Fig. 11) and reach levels of about 6–8 at.% at a moderate dose of $\sim 2.2 \times 10^{21}$ n/cm^2; i.e., ~3 dpa. Very few ATEM measurements are available at higher doses to determine if segregation levels continue to increase. However, Ni-silicide precipitation has often been observed at dislocation loops and void surfaces in stainless steels irradiated at higher temperatures (>380°C) and to higher doses (>20 dpa).

The few measurements of grain boundary P in neutron-irradiated stainless steels do not show a consistent evidence of RIS. Interfacial compositions range from not detectable (<0.4 at.%) to ~3 at.% [55]. Such variability may result from differences in P segregation before irradiation or a complex segregation response with neutron dose. P strongly segregates to grain boundaries in both Fe- and Ni-base stainless alloys during thermal treatments approaching levels of several atomic percent in the solution-annealed condition. Unfortunately, unirradiated archive materials are rarely characterized and detailed examinations of the neutron dose dependence have not been reported.

The interstitial association mechanism [56–58] was established to describe enrichment of undersized solutes (e.g., Si in Ni) at sinks. Interstitial flow and binding between undersized Si and self-interstitials in Ni rationalizes RIS measurements as a function of dose and temperature. Binding strengths greater than ~0.4 eV were used to explain the segregation data and are ex-

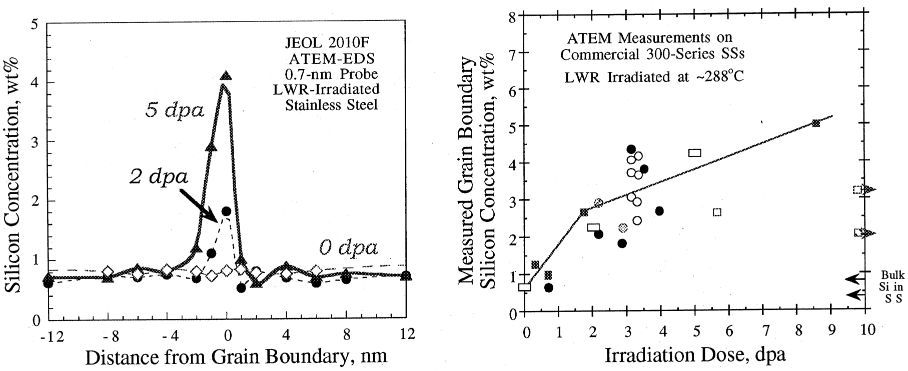

Fig. 11. Summary of reported radiation-induced grain boundary Si concentrations versus neutron irradiation dose [55].

pected based on elastic energy calculations and annealing experiments. Unlike the inverse Kirkendall mechanism, there have been no fundamental studies or quantification of the interstitial association mechanism for austenitic stainless steels. Preliminary attempts [59] to predict LWR RIS behavior indicate that measured enrichments of Si and P are much less than expected based on the binding energies established for binary alloys [60,61].

Although other undersized solutes such as S, C, N, and B should also segregate, the existence of RIS for these elements has not been established. Sulfur is occasionally mentioned, but rarely reported. It is important to note in this context that nearly all RIS measurements are on BWR-relevant low-dose (<10 dpa) samples. Sulfur may become more important in high-dose PWR-relevant conditions (>10 dpa) due to the transmutation of Mn (to Fe) and potential release of S (from MnS inclusions) [62]. Carbon, N and B will enrich at grain boundaries due to equilibrium and non-equilibrium mechanisms, but cannot be effectively measured using ATEM-EDS techniques. Scanning Auger microscopy measurements of LWR-irradiated stainless steels have not shown a significant segregation of these elements, but observations are limited [15].

Another element that may enrich at or near the grain boundary is He produced by transmutation. Helium as mentioned earlier can be produced at low irradiation fluences by the ^{10}B reaction or at higher dose by the ^{59}Ni two-step reaction that controls the total He content at high dose. The redistribution of this He is uncertain at low temperatures, but it does rapidly segregate to grain boundaries at much higher temperatures. The B source is particularly interesting, because it may be enriched at grain boundaries in the as-received stainless steel. A highly energetic He atom is produced by the transmutation reaction which will probably end up a distance of >2 μm from the original grain boundary position. This is a very large distance considering He diffusion kinetics, but due to the three-dimensional nature of the recoil a finite fraction (a few %) will end up within nanometers of the boundary. If B is strongly segregated to grain boundaries before irradiation, it is possible that He could be slightly enriched in the near-boundary region after relatively low dose with potential consequences for IG embrittlement.

6. Influence of radiation-induced microstructure and microchemistry on IASCC

6.1. Radiation-induced segregation

Radiation-induced Cr depletion has been the focus of many IASCC studies because of the well-documented effects of sensitization on IGSCC of non-irradiated stainless steels in oxidizing BWR water environments. Data where radiation-induced Cr depletion and IGSCC in post-irradiation SSR tests have been measured are summarized in Fig. 12 [63,64]. The SSR tests are limited, but indicate that some level of depletion exists in all stainless steels that fail by IGSCC in BWR-type (moderate-to-high electrochemical potentials) water environments. As the grain boundary Cr concentration drops by 1–2 wt% (below ~17 wt% for 304SS), irradiated stainless steels become susceptible to intergranular cracking under the specific conditions of the SSR test. The data points for 316 SS suggest a slightly lower minimum, corresponding to an interfacial Cr depletion of ~2 wt% below the matrix, consistent with the 304SS results.

The relationship between thermally induced Cr depletion and IGSCC reported by Bruemmer [64,65] has been plotted with the irradiated materials data in Fig. 12. All irradiated specimens that show IG cracking have sufficient grain boundary Cr depletion for IGSCC susceptibility in the SSR tests. Thus, Cr depletion can explain the observations of IGSCC under oxidizing BWR water conditions without considering other radiation effects on microstructure and microchemistry. However, questions still remain as to why many LWR-irradiated stainless steels show resistance to IGSCC in similar tests even with significant radiation-induced Cr depletion (e.g., down to ~12 wt%). Proton-irradiated high-purity 304-type stainless steels have also shown cracking resistance in SSR tests for certain alloys exhibiting severe Cr depletion (although differences in inclusion content and other factors that influence crack nucleation play a role) [66]. While it is recognized that the SSR tests do not give a quantitative assessment of SCC susceptibility, these results suggest that other microstructural (e.g., radiation hardening) and/or microchemical aspects influence cracking susceptibility. Such a conclusion is reinforced by the observation of a high-dose fluence threshold for IASCC in non-oxidizing (BWR-HWC or PWR) water. Under these environ-

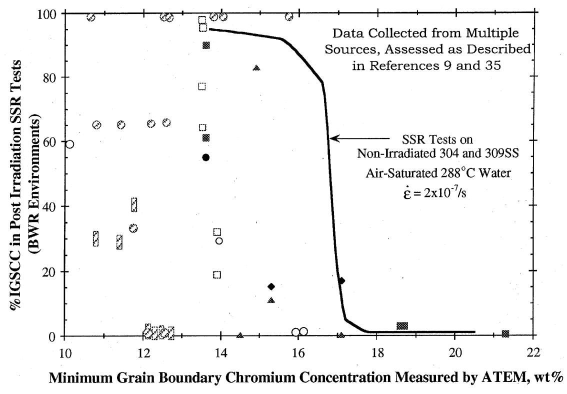

Fig. 12. Comparison of radiation-induced Cr depletion to the observed IG cracking in a post-irradiation SSR test [9,35].

mental conditions in the absence of significant aqueous impurities, Cr depletion does not promote IGSCC in non-irradiated stainless steels.

The other significant changes that occur in grain boundary compositions during LWR irradiation are for Ni, Fe and Si. Since the measured enrichment of Ni and depletion of Fe is nearly always correlated with the depletion of Cr, similar empirical relationships to IGSCC result. However, no corresponding linkage to IGSCC susceptibility in non-irradiated stainless steels can be made. Silicon does not appear to play a critical role since high-purity stainless steels (where Si or other impurities are not available to segregate) show cracking in a similar fashion as for commercial alloys with significant impurity levels at the grain boundary. There is no clear evidence that other impurity elements both segregate during irradiation and impact IASCC susceptibility. As a result, current information would indicate the only direct effect of RIS on IASCC susceptibility is due to Cr depletion, and then only on cracking in oxidizing water environments which produce pH-shifts at the crack tip.

6.2. Radiation-induced microstructure and hardening

The other material change that likely promotes IASCC susceptibility is radiation hardening. The evolution of the defect microstructure during LWR irradiation strongly affects the strength and mechanical properties of the alloy through interactions with dislocations. Post-irradiation data for neutron irradiation of 300-series stainless steels at about 300°C show that with increasing dose, the yield strength increases, and ductility and fracture toughness decrease. Relative to the unirradiated value the yield strength can increase by a factor of 4 at moderate dose, as illustrated in Fig. 13. The increase in yield strength can be seen to follow a square root dose dependence reaching saturation by ~10 dpa. Homogeneous deformation that is seen at low doses is shifted to heterogeneous deformation at higher doses when the defect microstructure evolves to the point where dislocation motion is effectively impeded. Subsequent plasticity is localized to channels created when a dislocation clears a slip plane of radiation damage debris. This provides an easier path for subsequent dislocation motion and slip becomes confined to a narrow band of slip planes that are free of defects. This 'dislocation channeling' results in intense shear bands and can cause localized necking and a sharp reduction in uniform elongation.

Although direct comparisons have not been made between radiation-induced microstructural evolution and IGSCC, the rapid increase in the density and size of faulted interstitial loops is consistent with the onset IASCC fluence thresholds. The radiation-induced microstructure reaches apparent saturation at a dose of several dpa. This is similar to where IASCC begins to be observed in non-oxidizing water environments. Yield strength changes with dose are related to the evolving defect microstructure and show this same general dependence (compare Figs. 5 and 13). The relationship between tensile yield strengths and IGSCC susceptibility from SSR tests in oxidizing water environments is examined in Fig. 14. Initial IG cracking is seen only after the yield strength has increased to about 500 MPa (more than double the typical value for annealed stainless steel). Insufficient data exist for a similar comparison for tests in non-oxidizing environments. However since the onset of IASCC susceptibility in BWR-HWC or PWR environments is typically at doses near 5 dpa, corresponding yield strengths tend to reach values between 700 and 800 MPa.

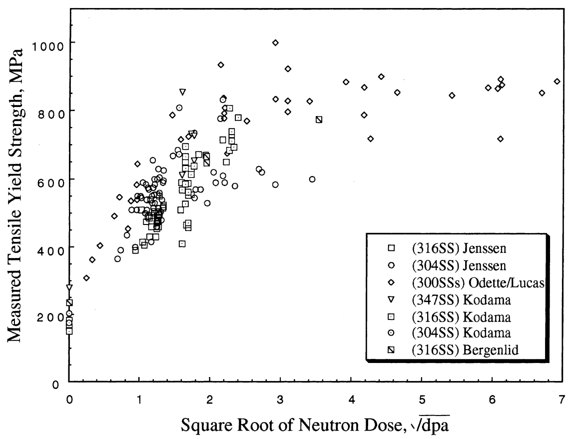

Fig. 13. Irradiation dose effects on measured tensile yield strength for several 300-series stainless steels [19,67–70].

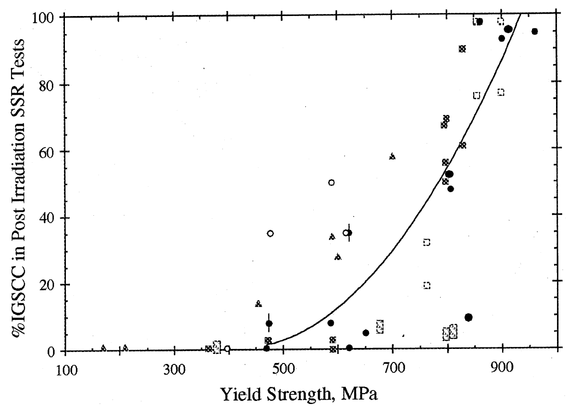

Fig. 14. Comparison of measured tensile yield strengths to IGSCC response in oxygenated high-temperature water [6,63].

Current results suggest that the radiation-induced microstructure, hardening and the change in deformation characteristics play a role in IASCC. The combination of very high stresses at channel-grain boundary intersections and the weakening of grain boundaries relative to the hardened matrix could promote IG creep cracking even at temperatures below 400°C [71]. Intergranular cracking may also be promoted by localized plasticity within or near the boundary plane. Since the grain boundary is a sink for radiation-induced defects, a narrow region (on the order of nanometers) surrounding the interface may contain a much lower density of dislocation loops. Grain boundary sliding and active slip on ⟨1 1 1⟩ planes within this narrow zone may occur. This process is more likely in-core where continuous migration of vacancies and interstitials to grain boundaries will increase local diffusivities and dislocation mobilities. Localized plasticity of this type aided by localized dissolution/oxidation at the crack tip or by the action of hydrogen produced from corrosion reactions may act in concert with a decohesion mechanism to produce IG cracking.

Manahan et al. [72] reported IG cracking in highly irradiated stainless steels without an environmental component if the test strain rate is slow enough. However, these observations have not been reproduced in a number of similar tests on LWR-irradiated materials and it is clear that the aqueous environment is required for IASCC observed at low to moderate doses. Nevertheless, IASCC susceptibility may be a precursor to a more general IG embrittlement susceptibility at very high doses. This discussion points out the need for an improved understanding of deformation mechanisms in irradiated stainless steels and improved data to evaluate critical hypotheses. It is necessary to elucidate dynamic irradiation and deformation effects on matrix, near boundary and grain boundary dislocation activity.

7. Research needs to improve IASCC understanding

Advances in IASCC understanding require research focused in radiation materials science and more generally on the fundamentals of grain boundary behavior, corrosion, localized deformation and fracture. Radiation materials science underscores the atomic displacement processes that drive change in material condition. However, the ability to link these changes to environmental cracking necessitates that underlying principals be elucidated for both irradiated and non-irradiated conditions. Critical research needs are identified below to improve the mechanistic understanding of radiation-induced material changes and IASCC of LWR core internals. This underpinning knowledge is essential for the continued effective operation of current LWRs and to enable design of optimized nuclear power systems.

7.1. Radiation Materials Science Issues

Important progress has been made over the last decade to isolate specific parameters that promote IASCC susceptibility. It is now clear that persistent radiation-induced changes to the stainless alloy control behavior. Application of high-resolution characterization techniques to the examination of the material contribution to IASCC in LWR-irradiated materials has clarified many issues related to microstructural and microchemical evolution during LWR irradiation. However, nearly all measurements have been performed on uncontrolled commercial stainless steel heats without any systematic variation of irradiation or material parameters, especially composition.

There is a paramount need for mechanistically driven single-variable experiments to elucidate radiation-induced material changes and their effects on IASCC. The use of alloy compositions and irradiation conditions much broader in scope than the standard LWR component experience is crucial to understanding IASCC behavior and defining opportunities for improved materials. These compositions and conditions must be selected based on the fundamental understanding of radiation-induced changes in order to isolate the basic processes controlling IASCC. Prior experience [42] in establishing the mechanisms and material variables influencing void swelling (and in the development of swelling-resistant materials) is an excellent example demonstrating why such single-variable experiments are imperative.

Radiation-induced changes proceed from atomistic events, to their accumulated effects on microstructure and microchemistry. This review has documented these accumulated effects for LWR irradiation conditions. From a radiation materials science point of view, critical research areas are: (1) multi-component cascades, (2) multi-scale microstructural modeling, (3) transient evolution of microstructure, and (4) defect/solute interactions at grain boundaries. Progress addressing these issues would enable mechanistic insight and prediction of specific alloy responses to radiation.

Multi-component cascade mechanisms may influence the efficiency of point defect production and the onset of material changes with increasing radiation dose. Empirically, certain solutes are known to decrease RIS and certain other solutes are known to affect cluster development. There is a need to understand and measure how minor additions of species can have significant effects on microstructural and microchemical evolution. Atomistic molecular dynamics modeling offers the ability to interrogate cascade events, cluster formation and solute effects. Delay in the threshold for IASCC could be achieved by retarding the efficiency of damage by tailoring alloys for low damage efficiency, enhanced in-cascade recombination and inhibited cluster evolution.

Multi-scale microstructural models integrating atomistic molecular dynamics, Monte Carlo, and rate theory approaches have the potential to establish the basic mechanisms of extended defect structure evolution (clusters, loops and cavities). Models need to evolve to the point that commercial alloy complexities (such as alloying and impurity elements, dislocations and grain boundaries) can be incorporated into the simulations and their influence on fine-scale defect microstructure evolution interrogated. Structural characterization of fine-scale defects ($\leqslant 2$ nm in diameter) irradiated at LWR temperatures and doses is needed. It is essential that the dose, temperature and material (composition and condition) dependence of defect microstructure evolution be better established both computationally and experimentally. This understanding is necessary for control of fine-scale microstructural features that promote significant hardening at low doses ($\leqslant 1$ dpa).

Transient evolution of microstructures at higher LWR temperatures and doses consists primarily of nucleation and growth of faulted loops (and cavities). This assessment is established from a remarkably small series of observations at intermediate doses (1–10 dpa) where saturation of the interstitial loop size and density occurs commensurate with the maximum radiation hardening. Rate theories for change in cluster size distributions should be developed and applied to the transient (as opposed to steady-state) evolution of extended interstitial and vacancy microstructural damage. Detailed microstructural characterization is again needed to guide mechanism development and quantitative modelling of faulted loop evolution. Similar issues of transient evolution exist for cavities that must be understood and mitigated to ensure high-dose structural performance (e.g., ductility and fracture toughness) of core internals apart from IASCC concerns. The possibility must be addressed that void swelling and the associated acceleration of irradiation creep may interact with other radiation-induced changes to promote cracking under high-temperature and high-dose conditions.

In contrast to microstructural evolution, radiation-induced segregation in LWR core component alloys has been better examined using high-resolution measurement and modeling techniques, thereby demonstrating the consequences of *defect–solute interactions*. The major and minor elements that enrich or deplete grain boundaries are reasonably well known. Qualitatively, the mechanisms that control RIS have been established and changes in grain boundary composition with dose can be modeled. Somewhat surprisingly, the initial grain boundary (Cr and Mo enrichment) in mill-annealed stainless steels has been more difficult to explain along with its effect on subsequent RIS. From a basic science point of view, these phenomena both result from defect–solute interactions and represent a fundamental need to improve mechanistic understanding of grain boundary microchemical evolution from equilibrium and non-equilibrium processes.

Similar to the LWR temperature/dose regimes noted for microstructural evolution studies, many uncertainties still exist for the evolution of grain boundary composition with irradiation. Measurements reported to date have almost all been for 275–290°C irradiation and doses below 15 dpa. Thus, LWR microchemical evolution at higher temperature and doses are unknown. A critical research need at lower doses is to understand the complex changes in Cr and Mo composition profiles from one of narrow enrichment in the unirradiated condition, to a W-shaped profile and finally to depletion with increasing radiation dose. An important aspect for this understanding is the quantitative assessment of light-element (e.g., B, C and N) segregation behavior. In addition, the unexpected small degree of grain boundary segregation for undersized elements such as Si and P needs to be explained. As with any IG process, mechanistic understanding of local composition evolution is essential to assess behavior.

8. Materials and corrosion science issues

It must be recognized that advances in radiation materials science alone will not lead to IASCC mechanistic understanding. The fundamentals of this complex environmentally induced IG cracking process can only be elucidated by linking radiation-induced material changes to known (and perhaps not-yet-known) structure-property relationships for core component alloys. A continuum must exist between known non-irradiated material behavior (grain boundary properties, corrosion reactions, deformation and fracture) and behavior of irradiated alloys. The continuum approach emphasizes that the irradiated alloy properties are not unique. Radiation simply perturbs the material microstructure and microchemistry, and thus changes the threshold conditions for IG cracking. The crystal structure, base alloy composition and exposure to stress and environment are the same for irradiated and non-irradiated alloys although critical details in material condition differ. A consistent interpretation of material response must be developed that satisfies mechanistic understanding of both irradiated and non-irradiated behaviors.

Advances in selected subfields of materials and corrosion science are critical for understanding IASCC. These subfields include: (1) deformation and fracture, (2) grain boundary structure and properties, and (3) corrosion/electrochemistry in high-temperature water environments including properties of the material/environment oxide interface at the crack tip. Radiation impacts each of these areas and the resultant core component cracking, but underlying mechanisms are common to irradiated and non-irradiated materials. A detailed review of these areas was beyond the scope of this paper, but focused research on these issues is imperative to address IASCC.

Austenitic stainless steels deform by planar slip, strengthen by addition of obstacles for dislocation glide, and fracture by IG and transgranular mechanisms that are influenced by exposure to water. It is obvious that mechanistic understanding of *deformation and fracture* is essential to interpret IASCC failures. Specifically, there is a need to model and measure elastic and plastic interactions between dislocations and the radiation-induced defect microstructure. The influence of fine-scale defects and faulted interstitial loops on flow localization and strengthening must be determined and linked to IG environmental cracking. Single-variable experiments on irradiated materials with tailored microstructural characteristics are required to delineate individual effects. Experiments should range in scale from high-resolution, in-situ TEM straining to bulk tensile behavior and crack-growth tests that are relevant to IASCC.

Grain boundary science is an underpinning discipline that helps define critical material change for IASCC. Specifically, changes in grain boundary microchemistry and structure are important. Irradiated grain boundaries are special cases of grain boundaries found in materials. Chemistries are perturbed; large numbers of point defects are absorbed; and the consequences of localized matrix deformation must be accommodated. It is possible that in some extreme non-equilibrium chemistry cases, phase changes may be possible at or near the grain boundary.

There is a need for complementary theories and experiments to accurately define the structure and chemistry of irradiated boundaries on the atomic scale. Critical needs include quantification of light elements at low concentrations and structural characterization of grain boundaries including possible two-dimensional phase formation. Dynamic temperature and stress effects on the interfacial structure in LWR-irradiated materials are particularly important. A mechanistic understanding of radiation-induced changes in the grain boundary structure and composition may allow detrimental effects to be mitigated through initial processing.

Since IASCC does not occur in inert environments, it is imperative to identify environmental mechanisms as revealed by understanding the underlying *corrosion science*; the third underpinning discipline. The reaction of water with the alloy surface results in metal oxidation, film formation and possible solid-state absorption of oxygen and hydrogen as illustrated in Fig. 2. The alloy surface of interest is not a general location but rather a special location (crack tip) affected by local grain boundary structure and chemistry. The mechanical and chemical character of the corrosion film that forms may control the atomistics of crack advance. Measurement and modeling of IASCC crack-tip characteristics are needed to interrogate detailed reactions between the material and the environment driving cracking. The unique grain boundary crack-tip electrochemical reactions that occur in oxidizing and non-oxidizing LWR environments must be delineated. Many aspects of these reactions are understood from research on non-irradiated materials, but specifics have not been established for IASCC. Effective prediction and mitigation of cracking in LWR environments requires that this continuum knowledge be established.

9. Conclusions

Radiation-induced microstructural and microchemical evolution in LWR core internals is shown to be remarkably complex and dependent on a wide range of irradiation and material variables. The microstructural defects that form and the changes in grain boundary composition that occur are now qualitatively well understood. However, accurate prediction of microstructures, microchemistries and mechanical property changes in complex stainless alloys during irradiation at LWR temperatures is not possible at this time. Mechanistic understanding of these radiation-induced changes in commercial alloys is of paramount importance to predict and mitigate IG cracking that occurs in service. Fundamental research is needed to define microstructural and microchemical evolution at intermediate temperatures and dose rates pertinent to LWRs where transient effects often dominate behavior. In addition, it is essential that both radiation-induced changes in the matrix and at internal grain boundaries be understood and integrated with environmental cracking mechanisms. These advances in radiation materials science must be accompanied by related advances in deformation and fracture, grain boundary science, corrosion science and environmental cracking.

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