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IMPACT OF ADVANCED MICROSTRUCTURAL
CHARACTERIZATION TECHNIQUES ON MODELING
AND ANALYSIS OF RADIATION DAMAGE

F. A. Garner
Hanford Engineering Development Laboratory
G. R. Odette
U. of California, Santa Barbara

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IMPACT OF ADVANCED MICROSTRUCTURAL CHARACTERIZATION

TECHNIQUES ON MODELING AND ANALYSIS OF RADIATION DAMAGE

F. A. Garner
Hanford Engineering Development Laboratory
Richland, Washington

G. R. Odette
University of California
Santa Barbara, California

Design correlations based only on empirical analysis of macroscopic radiation-induced property change data are sometimes insufficient to meet all design needs of nuclear power generation devices. Theoretical modeling and analysis activities are therefore important to guide extrapolation of the limited data and insight into untested environments and operating histories. The appearance of unanticipated phenomena can also require design changes or modification of operational procedures prior to the accumulation of a relevant data field. Once again modeling and analysis activities are called upon to provide the needed direction.

The evolution of radiation-induced alterations of dimensional and mechanical properties has been shown to be a direct and often predictable consequence of radiation-induced microstructural changes. Recent advances in understanding of the nature and role of each microstructural component in determining the property of interest has led to a reappraisal of the type and priority of data needed for further model development.

This paper presents an overview of the types of modeling and analysis activities in progress, the insights that prompted these activities, and specific examples of successful and ongoing efforts. More importantly, however, a review is presented of some problem areas that in the authors' opinion are not yet receiving sufficient attention and which may benefit from the application of advanced techniques of microstructural characterization. Guidelines based on experience gained in previous studies are also provided for acquisition of data in a form most applicable to modeling needs.

Introduction

The majority of the modeling and analysis activities concerned with the evolution of radiation-induced microstructure in metals derive their financial support from organizations dedicated to the commercial application of nuclear technology. These organizations also support extensive experimental programs to develop a data base on macroscopic properties necessary to design and support the operation of power generation facilities. Design correlations based only on empirical analysis of macroscopic data are frequently insufficient, however, to meet all design needs. The constraints imposed by leadtime and dollar requirements usually ensure that the data base seldom covers the entire spectrum of possible application, requiring interpolation and extrapolation to untested environments and operating histories. Therefore the impact of radiation-induced microstructural alterations on dimensional and mechanical properties is of prime importance.

The appearance of unanticipated phenomena can also require design changes or modification of operating procedures prior to accumulation of a relevant data field. Examples of significant unanticipated phenomena are void swelling and the magnitude of irradiation creep in fast breeder reactors, the magnitude of irradiation growth in zirconium alloys in CANDU reactors, and the embrittlement of pressure vessel steels in thermal reactors. Even more recently, the radiation effects community has become aware of a substantial alteration of phase stability that occurs during irradiation of many structural alloys.

Modeling and analysis activities have been employed to address both the extrapolation of data to untested environments and the anticipated impact of unexpected phenomena. A major tool in this effort is the accumulation of knowledge of the identity and density of radiation-induced microstructural components and the role of each component in determining the property of interest. Such modeling efforts have recently gone beyond a relatively narrow focus on void growth to treat the entire time history and correlated evolution of microstructural and "microchemical" features. This has led to a rethinking of both the type and priority of data needed for further model development.

This paper presents an overview of the types of modeling and analysis activities in progress, the insights that prompted these activities, and specific examples of successful and ongoing efforts. More importantly, however, a review is presented of some problem areas that in the authors' opinion are not yet receiving sufficient attention and which may benefit from the application of advanced techniques of microstructural characterization. Guidelines based on experience gained in previous studies are also provided for acquisition of data in a form most applicable to modeling needs.

Types of Modeling and Analysis Activities

Modeling and analysis activities can be conveniently divided into four categories. The first of these is the identification and description of the various roles played by each microstructural component in response to in-reactor or ex-reactor environments. Not only has the action of some important components previously escaped detection, but components may evolve either late in the irradiation or exist at such small sizes that they are effectively invisible, beneath the resolution limit of previously employed characterization techniques. Other visible microstructural components such as precipitates were once assigned relatively minor roles, particularly with respect to the assumed larger roles of dislocations and voids. Recent insight, however, has led to an upgrading of the importance of precipitates (1).

The second category of analysis involves the search for data sets which contain microstructural evidence that allows the validation of various proposed mechanisms for the action of a given component. Where more than one mechanism has been proposed, such data sets are also analyzed for clues which allow the various mechanisms to be ranked in order of their relative contribution to the property of interest.

The third category comprises the simulation, often computer-assisted, of the consequences of the competitive action of all microstructural components in each of their various roles. This type of modeling is particularly valuable for complex microstructures as well as for complicated operational histories.

The last category of modeling and analysis involves the application of the insight gained in the previous three categories toward the selection of the form of the empirical equation on which to condense the available data. This also provides a physically-based rationale for extrapolation of the equation beyond the boundaries of the current data base.

Radiation Damage in Metals: Major Insights

It is possible in a greatly simplified fashion to outline the major insights which in the authors' perception form the basis of most of the current modeling efforts on radiation-induced property changes relevant to various fission and fusion reactor programs. Some of these insights derive their inspiration primarily from theoretical efforts and others primarily from experimental programs.

The nature of the interaction of a microstructural component with radiation-induced point defects is largely based on theoretical grounds. The first major insight involves the concept of a selective bias of each microstructural component toward acceptance of various point defects, with interstitials thought to be accepted at dislocations somewhat more readily than vacancies (2,3). This results in a net partition of point defects between various microstructural components, some of which, such as voids, were originally thought to be neutral sinks. It was later postulated that both dislocations and voids possess strain fields capable of producing biases in favor of interstitials (4). The biases of each individual sink in this model are much larger than that previously ascribed to dislocations alone, and it is the competitive interaction of many sinks with different biases that determines whether a particular microstructural component grows as a net sink for vacancies or interstitials (5). Major advances have been made in the description of these biases with respect to their dependence on component size, spacing, applied stress, segregation at sink surfaces and other variables (5-10). The most exciting area of current interest is the concept that the bias of each sink changes strongly with the composition of the matrix in which it is embedded (1,8).

The initial development of experimentally-derived insight on problems relevant to fast reactors was hampered somewhat by the unrecognized interaction of the large number of variables which influence the development of microstructure during irradiation. Many of these variables were insufficiently controlled in early experiments. As each of these variables was recognized and brought under control, the reproducibility of the various phenomena was established and many trends became evident. One major recent insight is that many alloys evolve toward a saturation microstructure which appears to be independent of starting microstructure (11). Another is that gradients in point defect concentration that develop near microstructural components produce a selective flow of various elements along the gradients, leading to radiation-induced segregation of these elements (12). The consequences

of this phenomenon are sufficient to induce in some alloys a microchemical evolution of the alloy matrix involving significant changes in the concentrations of both solute and solvent atoms (1,13,14). This process causes a substantial alteration of phase stability and also appears in some alloys to proceed toward a saturation state that is independent of starting microstructure (13-15).

Another major experimentally-derived insight has been the concept that microstructural records are impressed on the post-irradiation microstructure by the action of physical mechanisms which cannot be observed directly while in progress. Whereas it was once accepted that, unlike void growth, irradiation creep left no record in the post-irradiation microstructure, the expectation that such a record might exist has led to the discovery of such records (16-18). Various radiation-induced diffusional processes have also been found to leave interpretable records in the matrix regions surrounding various sinks (15,19).

Examples of Current Modeling Activities

With the exception of irradiation of thin foils with electrons in a high voltage microscope, it is generally impossible to observe the action of microstructural components during irradiation. After the irradiation has ceased, processes such as dislocation climb and enhanced bulk and surface diffusivity decrease sharply or terminate. Some processes such as radiation-induced solute segregation may actually be reversed and some components such as vacancy loops may be annealed. In the most pessimistic sense, then, the use of post-irradiation microstructure in modeling efforts is best confined to the description of ex-reactor properties. In general, however, it appears that most microstructural components that develop during irradiation at low temperature and even in the range 300 to 700°C are relatively stable during cool-down, extraction from reactor, and during subsequent storage and handling. In the following sections, examples will be shown of the successful application of post-irradiation microstructural data to the modeling of both in-reactor and ex-reactor properties.

Ex-Reactor Mechanical Properties

Tensile test experiments yield a variety of data relevant to material performance, two of which are shown in Figure 1, but the yield stress is the

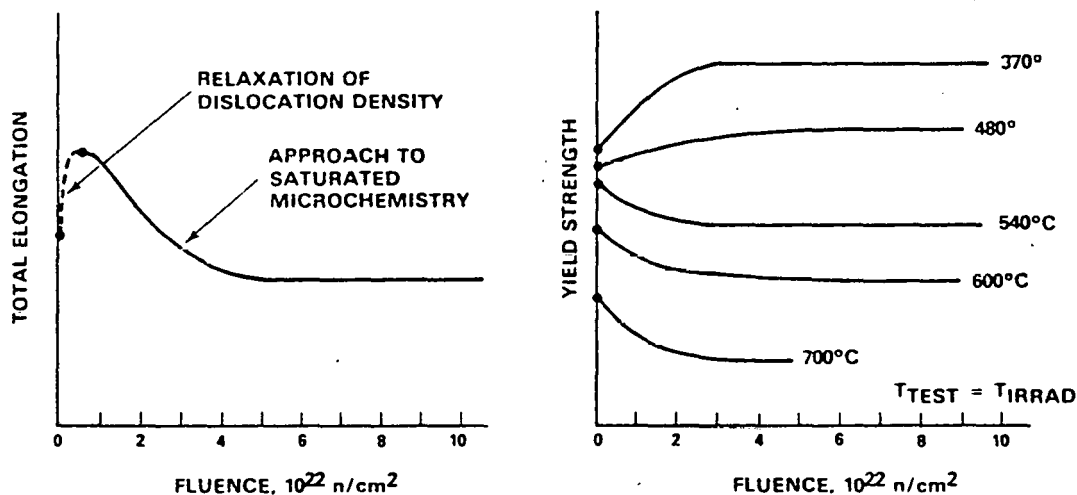


FIGURE 1. Schematic illustration of the fluence and temperature dependence of several mechanical properties of 20% cold-worked AISI 316 (23,24).

best measurement of the influence of radiation-induced rather than deformation-influenced microstructure. As our understanding of the microstructural evolution of 300 series stainless steels has evolved, a number of attempts (20-22) have been made to determine the relative contributions of each microstructural component to the hardening or softening of the material observed for a given set of irradiation conditions. These efforts have been hampered by incomplete or incorrect descriptions of the microstructure and some ambiguity concerning the exact nature of hardness models for each component. Much more complete characterizations now exist of the irradiation-modified microstructure of both annealed and cold worked AISI 316 stainless steel. These descriptions have been recently used to successfully construct a microstructurally-based yield stress correlation (23). In this effort those microstructural components sensitive to flux, stress, and temperature have been identified. An estimate has also been made of the cavity contribution to hardening.

The current empirically-derived design correlation for yield-stress of AISI 316 is based on a flow model/state variable approach derived only from cold work data obtained from irradiation in a fast breeder reactor (24). This equation contains no guidance on how to extrapolate the data to other environments, particularly those that involve different displacement rates and high helium/dpa ratios. It is anticipated however, that microstructural information developed from specimens irradiated in any new environment can be used with the microstructurally-based correlation to aid in the extrapolation of the empirical equation.

The extrapolation of microstructural information to other ex-reactor mechanical measurements such as total elongation is more complicated, since at grain boundaries other processes such as solute segregation, precipitate denuding, and cavity formation become important. In breeder reactors, helium generation and cavity formation are not as important as they would be in a fusion environment. Typical elongation curves developed from irradiation in breeder reactors display characteristics (Figure 1) which are consistent with previously described microchemical and microstructural considerations. There is an initial increase in elongation or "softening" due to the relaxation of the starting dislocation network to its saturation value, and a subsequent decline to a saturation value at a fluence consistent with the saturation fluence of the microchemical evolution (13,14). This suggests that if the grain boundary regions are important in determining this property then they may also evolve to a saturation state involving segregation of various elements. In a high helium-generating environment, the role of grain boundary cavities becomes much more important, and will strongly degrade the creep rupture and ductility properties. An example of the use of property-property correlation techniques to predict such behavior is shown in Figure 2 for fast reactor data on uniform elongation. The correlation is based on observed yield stress behavior (25).

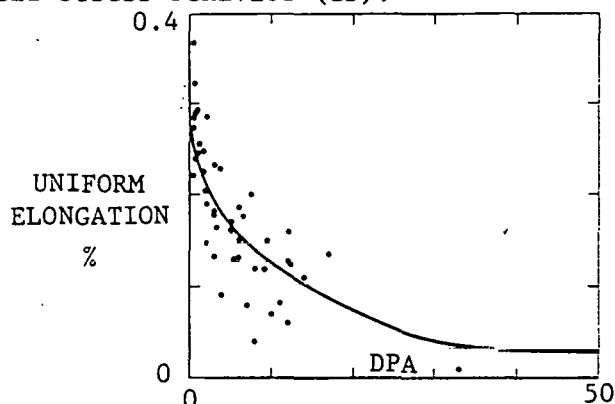


FIGURE 2. Comparison of calculated ductility and data of annealed AISI 316 based on a correlation with yield stress at 500°C (25).

In-Reactor Dimensional Changes

As reviewed elsewhere, it is possible to correlate the onset and development of both swelling and irradiation creep to the details of the microstructural and microchemical evolution (1,13). It was, in fact, the inability of microstructure alone to account for the observed relative swelling behavior of cold-worked and annealed AISI 316 steel that led to the prediction and search for the microchemical evolution (14). Note in Figure 3 that the late

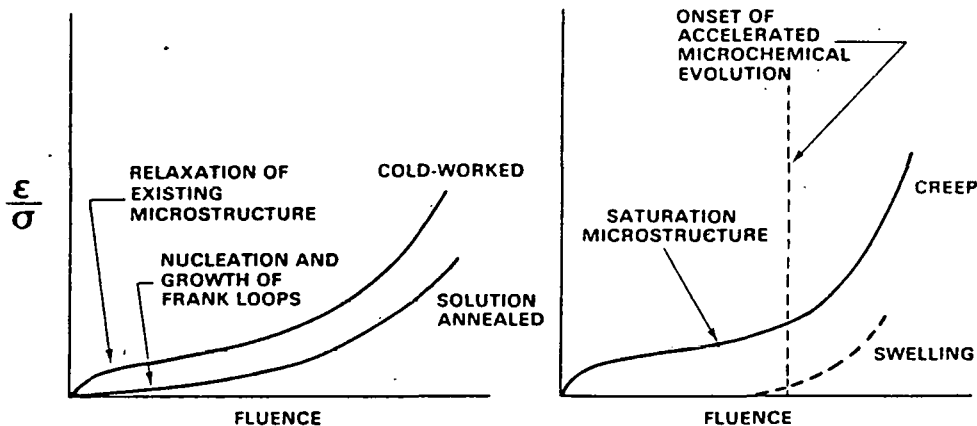


FIGURE 3. Schematic illustration of the evolution of swelling and irradiation creep in AISI 316 and the relationship of these to the evolution of microstructure and microchemistry.

term acceleration of irradiation creep and the concurrent onset of swelling were found to occur long after the attainment of a saturation network of Frank loops and dislocations. The acceleration of creep and swelling were found to coincide with the onset and acceleration of nickel removal from the alloy matrix, however, as shown in Figure 4.

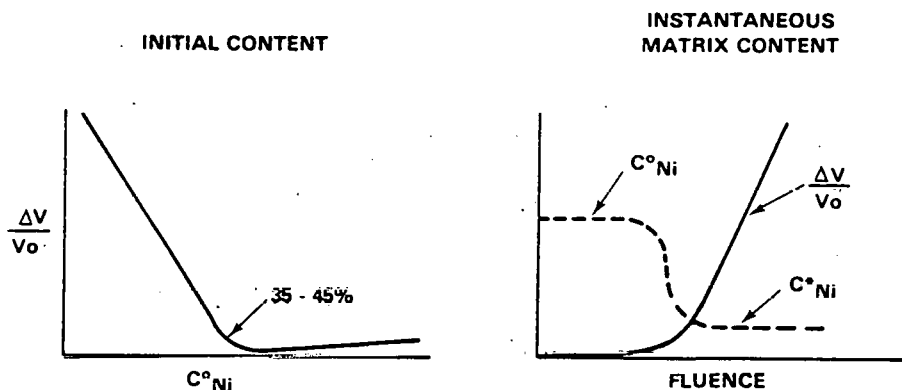


FIGURE 4. Schematic representation of the correlation observed between swelling $\Delta V/V_0$ versus the original alloy nickel content and swelling versus the average instantaneous nickel content in the matrix of AISI 316 (13).

It was found impossible to describe in terms of microstructure only the response of both swelling (26-28) and irradiation creep (28) to changes in temperature during irradiation. Note in Figure 5 that small gradual decreases in temperature lead to decreased creep rates as expected from microstructural

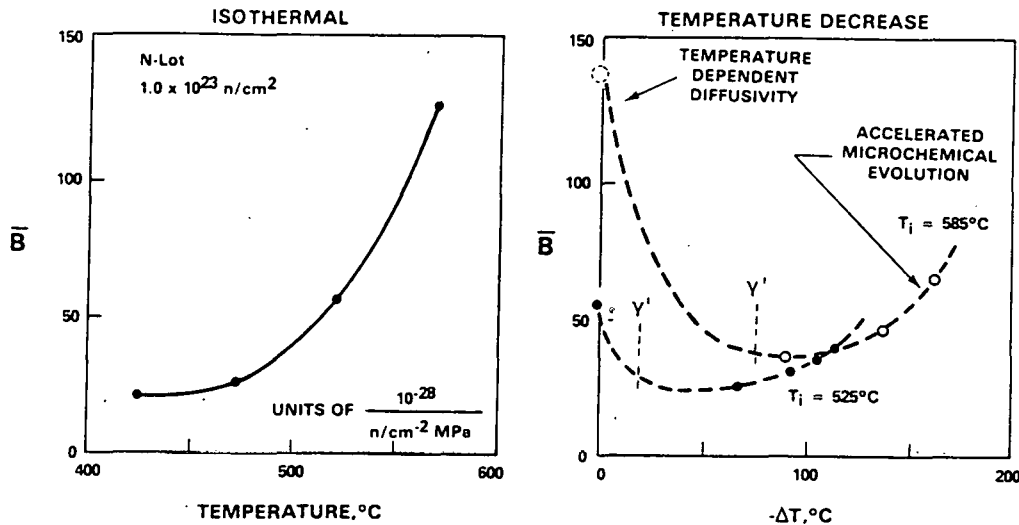


FIGURE 5. Comparison of average creep coefficients of 20% CW AISI 316 obtained in response to isothermal and gradually declining irradiation temperatures (28). The onset of γ' formation is shown.

and diffusivity considerations. This trend persists only so long as the microchemical evolution is not perturbed or accelerated. At some point, however, the further decline in temperature causes an accelerated formation of gamma prime precipitates, as confirmed by microstructural observations (28). An acceleration of both creep and swelling (Figure 6) was the direct result.

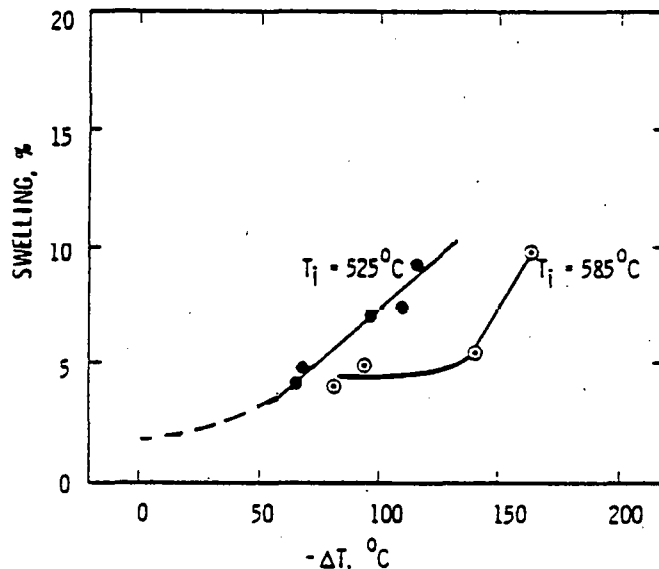


FIGURE 6. Enhancement of swelling by gradual temperature reductions during neutron irradiation of 20% cold-worked AISI 316 (replotted from Reference 26). T_i is the starting temperature.

It is therefore necessary for modeling purposes to expand the definition of relevant microstructural features required to describe the radiation-induced evolution. In addition to the usual descriptions of component identity, size distribution and density, the experimenter should include the time-dependent and spatial details of the matrix and precipitate compositions that evolve during irradiation. The spatial details are particularly impor-

tant as discussed in the following section.

The impact of the interaction between the microstructural and microchemical evolutions and other detailed examples of its consequences in alloy behavior have been discussed by Odette (29). In this latter paper a thorough review of the microstructural data base has been published.

Microstructural Records of Diffusional Processes

As discussed elsewhere, the precipitate phases that evolve in 300 series stainless steels become progressively richer in the elements nickel and silicon, and in some cases precipitates rich in these elements form which are only stable in the presence of irradiation (1,14,30,31). The study of this segregation process has been facilitated by microanalysis of the compositional gradients which develop in the vicinity of such precipitates. As shown schematically in Figure 7 and discussed in detail elsewhere, (15,31) the direction

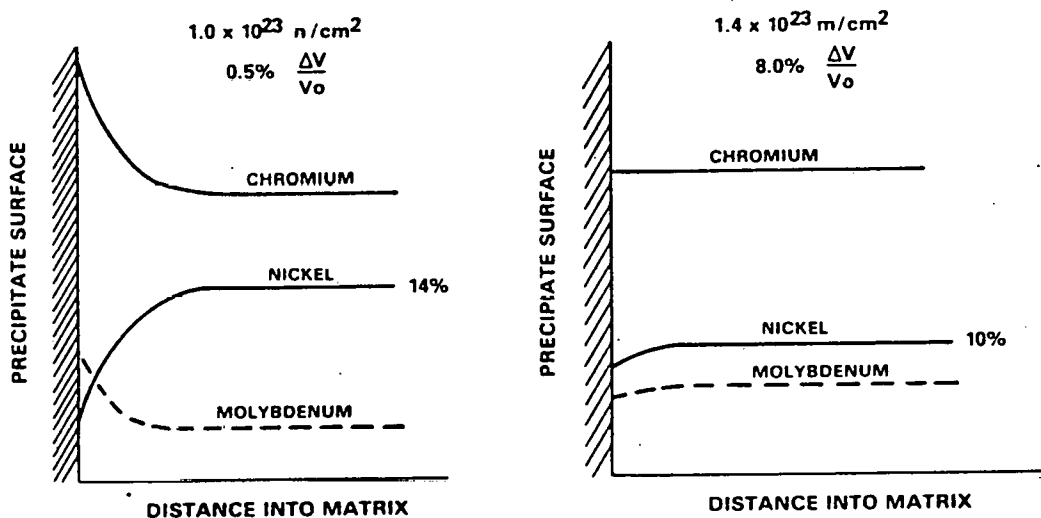


FIGURE 7. Schematic representation of compositional profiles measured near Laves precipitates in 20% cold-worked AISI 316 at 650°C (1,15). When the swelling level is low the gradients are much more pronounced than when observed at higher swelling levels. Note the changes in matrix composition which result from the infiltration-exchange process across the precipitate-matrix interface.

of solute flow can sometimes be inferred from the nature of the defect gradients near each precipitate, providing that the observation is made while the flow is occurring. While the onset of these segregation processes has been correlated with the onset of swelling and accelerated irradiation creep, the termination of the segregation process is signaled by a reduction in the gradients, and appears to correspond to the attainment of steady state deformation rates.

Elemental segregation also appears to occur at dislocation loops, grain boundaries and void surfaces (15,19,32,33). The segregation of nickel at void surfaces is primarily balanced by an outflow of chromium as would be expected from their respective diffusivities and the operation of the inverse-Kirkendall effect (34). As shown schematically in Figure 8, the effect is amplified in the region between two voids and demonstrates that at least at 650°C molybdenum and silicon are not directly visible as participants in the segregation process at voids (15,31). Data of this type are becoming very useful in the validation and ranking of various diffusional and solute-binding

mechanisms.

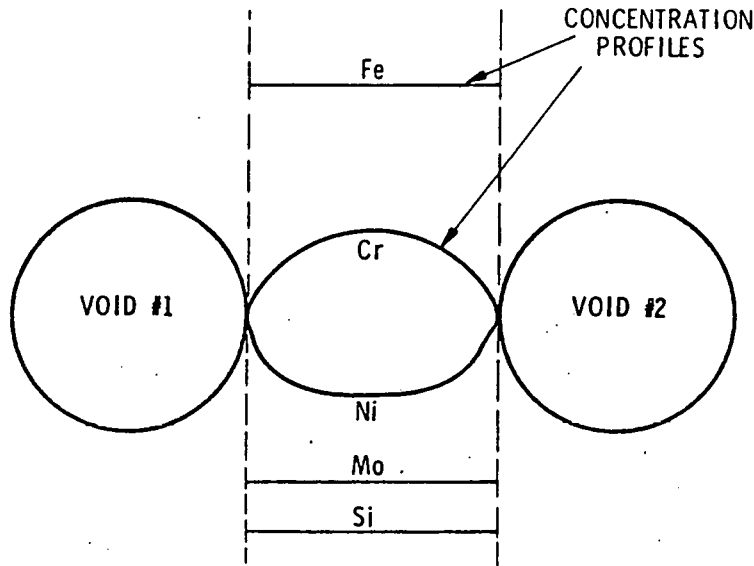


FIGURE 8. Schematic representation of the compositional profiles observed between two voids in 20% CW AISI 316 at 1.4×10^{23} n/cm² ($E > 0.1$ MeV) and 650°C (15,31).

Microstructural Records of Irradiation Creep

There is currently considerable controversy as to the parametric dependence of various proposed irradiation creep mechanisms, particularly that of stress-induced preferential absorption (SIPA) of interstitials (35). Indeed, the very existence of this creep mechanism has been questioned. The description of this and other creep mechanisms has been aided recently by the development of detailed studies of Frank loop and dislocation populations which evolve in response to anisotropic stress distributions. Okamoto and Harkness (36) first provided the evidence shown in Figure 9 that indicates

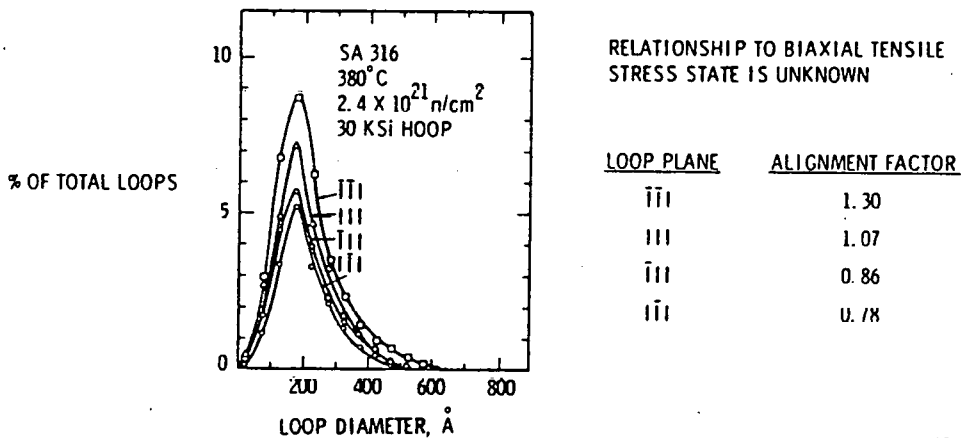


FIGURE 9. Frank loop distributions observed by Okamoto and Harkness at low fluence in annealed SA 316 irradiated under a biaxial tensile stress in a pressurized tube (36). The alignment factor is defined by the ratio of the loop density on a specific plane to the average density on all planes.

that Frank loop populations might respond in a nonuniform way to the biaxial tensile stress state found in the walls of pressurized tubes irradiated to

low fluence. Fluctuations of as much as $\pm 30\%$ from the mean value were found, but unfortunately it was not possible in that experiment to relate the planar distributions to the stress state. Wolfer later showed that the most optimistic assessment of loop alignment models (based on rotation of di-interstitial clusters) could not account for this magnitude of fluctuation (37).

Brager and co-workers (38) showed (Figure 10) that at higher fluence and stress levels, more pronounced fluctuations about the mean planar density were possible and related these to the stresses acting on each plane. Subsequent reinterpretation of these data by Garner and co-workers (16,17) indicated that this evidence indeed appeared to validate the existence of the SIPA mechanism. It was also asserted that a relative ranking in importance of the SIPA mechanism over the stress-induced alignment mechanism was supported by these data (17). Later data for AISI 316 at higher fluence and in other steels has provided even more insight on the nature of and competition between various irradiation creep mechanisms (18). Validation of the existence of SIPA is important not only to fast reactor studies but also the extrapolation of low-fluence creep data on zirconium alloys to higher fluence in the CANDU reactors (39).

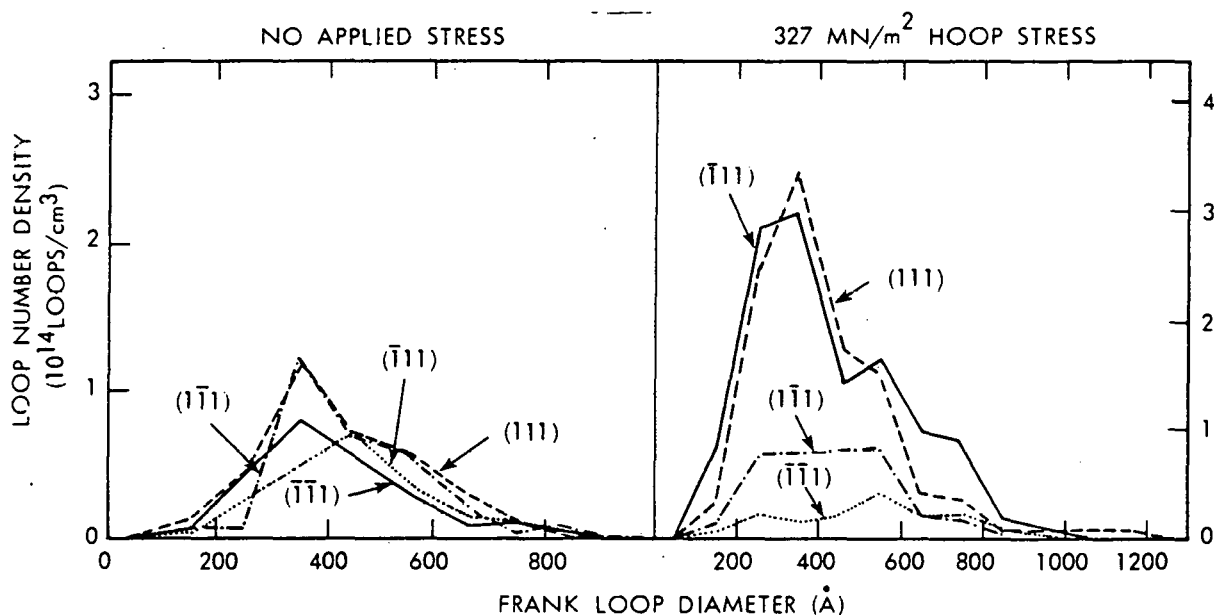


FIGURE 10. Comparison of Frank loop size distributions on each $\{111\}$ plane in two specimens of 20% cold-worked AISI 316 irradiated at different stress levels at 500°C to 3×10^{22} n/cm² ($E > 0.1$ MeV) (38).

Problem Areas Requiring Attention

There are a number of areas in which the application of advanced microstructural characterization techniques might possibly aid in the modeling of material response to irradiation. Several of these problem areas are discussed here in the hope that advanced techniques will be brought to bear on each problem and provide the needed insight.

Superfine Microstructure

Irradiation-induced increases occur in the yield stress and the ductile-brittle transition temperature in pressure vessel ferritic steels during irradiation in light water reactors. These have been correlated primarily to the copper and phosphorous content of these alloys (40). In a simple Fe-0.3%Cu alloy irradiated to about 3×10^{19} n/cm² ($E > 0.1$ MeV) at 290°C ,

the possible role of copper in the degradation of mechanical properties has been identified. Brenner and co-workers (41) have demonstrated by the use of field-ion microscope and atom probe techniques that 0.6 nm copper-stabilized microvoids formed during irradiation at densities of $8 \times 10^{17} \text{ cm}^{-3}$. This exceptionally high density of potential pinning centers could not be resolved in transmission microscopy studies, in which only a much smaller density of 3-5 nm dislocation loops was found. Gelles and co-workers (42) later demonstrated in this same material that a dislocation pinning point analysis conducted on specimens strained at room temperature confirmed the existence and density of these clusters. The alloys actually employed in pressure vessels are much more complex, i.e., tempered bainite which is generally stable under light-water reactor irradiation conditions. Illumination of the mechanisms of embrittlement in such alloys will require a better understanding of the governing microstructures, both visible and unresolvable.

Several researchers have noted that in 300 series stainless steels large scale radiation-induced segregation of nickel into precipitate phases leads to a transformation of portions of the austenite matrix to ferrite (31,43). There is a possibility that these ferrite phases may develop from superfine ferromagnetic nuclei formed at very low fluences. Using magnetic measurements, Stanley (44) observed in AISI 316 $\sim 3 \times 10^{17} \text{ cm}^{-3}$ of small magnetic particles below $3 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) in the range of 450 to 620°C. Microscopy did not detect resolvable ferrite regions however. Although Stanley noted that nickel segregation to void surfaces could be a possible source of the magnetization increases, the magnetic particle density was about three orders of magnitude greater than that of the voids. Baron and co-workers also found ferromagnetism in AISI 316L in structural components irradiated in the Rapsodie fast reactor (45). The relationship of the magnetic permeability profile relative to that of the temperature, flux and swelling profiles led these researchers to hypothesize that ferrite formation and trapping of transmutation-produced hydrogen in voids might account for the magnetism. It is significant to note that the yield-stress modeling effort discussed earlier failed to find any hardening contribution that such clusters might induce.

Other types of superfine microstructural components that might not be resolvable with currently applied techniques are small point defect cascade remnants in low temperature irradiations and very small helium aggregations that probably function as void embryos. Positron annihilation studies have demonstrated the existence of such microvoids and microbubbles (46).

Distribution and Influence of Helium

The large levels of helium that will be generated in a fusion environment are expected to lead to significant degradation of mechanical properties relative to that developed in fast reactors at comparable displacement doses (47). It is also expected that radiation-induced dimensional changes will be sensitive to the helium/displacement ratio. In an effort to study this possibility specimens are being irradiated with dual ion beams (48,49) and neutrons in mixed-spectrum reactors such as HFIR (50). It has been shown that the larger levels of helium in these environments lead to alterations in the temperature regime of swelling (51), as well as alterations in the Frank loop, void and precipitation response (51-54). It also appears that under some conditions the correlated development of voids and precipitates can lead to cavity/precipitate interactions that greatly accelerate swelling (49).

The modeling of these effects requires not only a description of the gas contribution to cavity growth (55-57) but also measurements of the initial distribution and subsequent mechanisms and rates of redistribution of helium and hydrogen. At the moment this area comprises the major uncertainty in modeling of the effect of transmutation-produced gases.

Another area requiring experimental input is the time-dependent distribution of helium generation. The use of mixed-spectrum irradiations and the nickel two-step transmutation reaction (50) will lead to non-uniform helium generation in and around those areas where nickel segregates. Thus helium deposition will be greatest at the surface of large precipitates (58) and grain boundaries. Since voids and cavities tend to nucleate preferentially on these microstructural components, the mixed-spectrum simulation experiments will develop a distribution of voids and cavities that are possibly quite atypical of that obtained in both fast reactor and fusion device spectra.

Correlated Microstructural Development

Many examples have been published in this and other reports of the correlated development of microstructural components. The successful modeling of these processes requires that detailed data be collected on the nature of the various physical associations, the sequence of nucleation of correlated pairs (the old chicken or egg question) and the direction and sequence of elemental flows to and across boundaries.

When collecting such data it is important to provide sufficient evidence to allow the modeler to judge whether the association is a direct consequence of the interfaces involved, or whether the two components nucleated separately and then grew in such a way as to join.

Needless to say it is clearly insufficient to collect only void or dislocation data. Given the heterogeneity of the evolution in some alloys, all such data should be derived from one region of the specimen if possible.

Austenite \rightarrow Ferrite Transformation

The question arises whether the $\gamma \rightarrow \alpha$ transformation observed in several studies occurs by a nucleation and growth process during irradiation or by a martensitic inversion upon cooling. Porter (59) has deduced that the transformation is a continuous process involving nucleation and growth on stacking faults, citing as evidence the nature of the $\gamma \rightarrow \alpha$ boundaries and the absence of twinning in the α -phase. Mazey and co-workers (60) arrive at the opposite conclusion from an ion irradiation study of a series of 12Cr-15Ni-Fe alloys. Their conclusion was based on the observation that the nickel content was reduced by precipitation of Ni_3Si and other phases, which should lead to an increase in the martensitic transformation temperature. They also note that x-ray analysis shows the remaining austenite and the ferrite have essentially identical compositions, whereas they would expect substantially different nickel or nickel-equivalent compositions for a diffusion and growth process. Mazey also notes that voids were found in the ferrite regions which are known to resist swelling. Brager and Garner disagree with Mazey's conclusions while citing essentially the same type of evidence derived from neutron irradiations of silicon-modified AISI 316 alloys (31).

The issue here centers on whether the $\sim 2\%$ volume change that accompanies the transformation occurs during irradiation or only on cool-down. The latter possibility allows the loss of this volume change upon reheating.

Effect of Stress on Microstructural Development

The various roles of stress on radiation-induced microstructural development have been clearly demonstrated in a number of studies (11,12,16-18,61). It has been shown, however, that the operating stresses are not always determinate, particularly when the material experiences large (and possibly anisotropic) strains due to precipitate-related volume changes (17,18). In mater-

ials possessing low intrinsic swelling and creep rates this can lead to the influence of internally-generated stresses overwhelming the microstructural record of the externally-applied stresses (18). The acquisition of component and stress orientation data in complex microstructures may require the development of additional data collection and analysis techniques.

The development of preferred orientations of Frank loops during irradiation under stress (12,18,38) leads to the prediction that the resulting network dislocations will also exhibit a corresponding but slightly different anisotropy. This possibility has not yet been experimentally demonstrated and has large consequences in the modeling of irradiation creep, particularly in response to changes in stress magnitude or direction.

It also appears that the microstructural record of various irradiation creep processes must be interpreted very carefully in that the record changes as the number of competing components increases (18). This requires the experimenter to choose carefully the specimens with which the record is sought.

The effect of tensile stress on swelling of AISI 316 and other alloys has recently been definitively determined to be related to changes in the incubation behavior (61). The effect of stress is most pronounced at high temperatures and this sensitivity has been shown to be related to the stress-sensitivity of intermetallic phase formation, as shown in Figure 11. This insight requires that the swelling equation developed for non-tensile stress states be quite different than originally envisioned. The low-temperature radiation-stable precipitates do not appear to be stress-sensitive. This has been verified experimentally for the γ' phase in AISI 316 and has been inferred for the G-phase from the relative stress-insensitivity of swelling of annealed AISI 316 at low temperature (62).

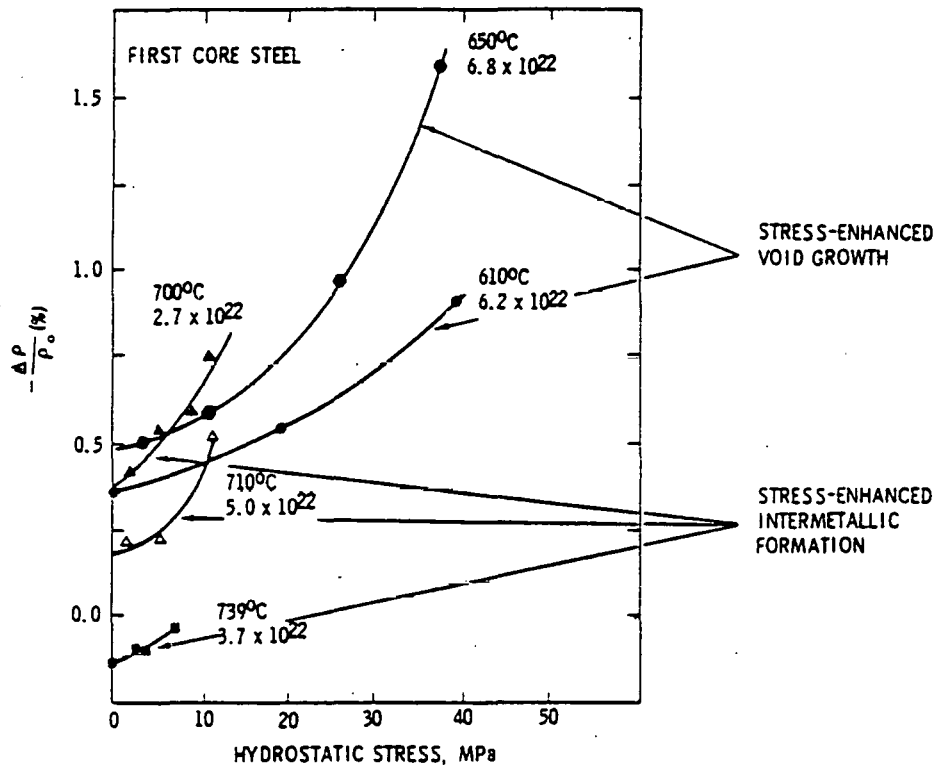


FIGURE 11. Stress-enhanced density changes observed in 20% cold worked AISI 316 (61). Note that dilational strains associated with formation of intermetallic phases can be observed at temperatures and fluences where void swelling has not commenced.

The major problem with all of the above studies is that they were conducted in tensile biaxially-stressed specimens. In order to confirm the results of these studies and confidently extrapolate into torsional or compressive environments, data must be generated in these stress states.

Guidelines for Data Acquisition

The preceding discussion has spotlighted a number of principles which can be condensed as guidelines for experimenters who anticipate the further use of their data in modeling and analysis activities.

- (1) The choice of specimens to examine is often the critical step. Given the tremendous complexity and synergistic nature of the processes involved it is best to choose whenever possible specimens for comparison which clearly differ in only a single variable. Since many of the most important processes are transient by nature, it is best to examine specimens just before, during, and just after the transient. Examinations conducted long past the terminus of the transient will often suffer erasure of the microstructural or microchemical records.
- (2) After having chosen the specimen it is important that the experimenter anticipate the presence of fine structure and pursue it even in the absence of foreknowledge of its existence.
- (3) The microstructure should be characterized in its entirety, including associated microchemical gradients and concentrations. Wherever possible all data should be extracted from a single region of the specimen, recognizing the relatively heterogeneous nature of the microchemical evolution. The authors recognize of course the difficulty of obtaining fine and coarse structure in the same foil thickness.
- (4) The experimenter should also recognize the need for mass balances in modeling efforts.
- (5) In studies involving the effect of stress it is important to retain knowledge of the relative orientation of the microstructural components and the stress state. One should also be alert for signs that signal the large role of internally-generated stresses.

Conclusions

It appears that the need is increasing for the coupled use of advanced microstructural characterization techniques and modeling/analysis activities. Substantial challenges are posed by recent insights on fundamental processes and the needs of materials people in development of descriptions of component response in fission and fusion environments.

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