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CLADDING AND OTHER STRUCTURAL MATERIALS

5. MECHANICAL PROPERTIES OF ALLOYS IN REACTOR ENVIRONMENTS AND DEVELOPMENT OF LMFBR CLADDING AND STRUCTURAL MATERIALS

J. R. Weir, Jr. H. E. McCoy, Jr.

This program is concerned with the effects of irradiation on the mechanical properties of various metals of potential use in nuclear reactors. The materials of primary concern are types 304 and 316 stainless steel, both materials of interest for liquid-metal fast breeder reactors (LMFBR). These alloys are being exposed to high neutron fluences, and the resulting changes in density, microstructure, and mechanical properties are being measured. Other materials under study include Incoloy 800 and V. Some attention is also being given to the use of particle accelerators to simulate neutron damage.

Austenitic Stainless Steels

Irradiation Damage to Type 304 Stainless Steel at 370 to 800°C
(E. E. Bloom, J. O. Stiegler)

The effect of irradiation temperature from 370 to 800°C on the immersion density and microstructural changes in type 304 stainless steel at a fluence of about 4×10^{22} neutrons/cm² (> 0.1 Mev) was determined. Materials irradiated at 370 to 380°C and 430 to 440°C were obtained by examining portions of the safety-rod thimble (flat C of the 3-D-1 thimble) from the Experimental Breeder Reactor-II (EBR-II). Irradiation temperatures along the length of the thimble were calculated at Argonne National Laboratory.¹ Materials irradiated at 570 to 630°C and at 770 to 840°C were obtained by examining specimens of types 304 and 304L stainless steel irradiated in EBR-II subassembly XO-34. In all cases, the irradiation temperatures were calculated with the lower and upper temperatures in each case based on gamma heating rates of 3 and 4 w/g at the reactor midplane of row 2 at a reactor power of 50 Mw. The calculated heating rate for this position is about 3.6 w/g. The

ranges on the irradiation temperature are believed to be the largest possible.

Changes in immersion density for these specimens are listed in Table 5.1. These results show that the density decrease (at this fluence) for type 304 stainless steel reaches a maximum at 450 to 500°C and then decreases rapidly with increasing irradiation temperature.

Table 5.1. Density Decrease of Types 304 and 304L Stainless Steel Irradiated at 370 to 800°C

Type Stainless Steel	Unirradiated Density (g/cm ³)	Irradiation Temperature (°C)	Fluence [neutrons/cm ² (> 0.1 Mev)]	Change in Density (%)
			$\times 10^{22}$	
304 ^a	7.8875	370-380	4.3	-0.60
	7.8875	430-440	4.2	-1.33
304 ^b	7.8724	570-630	4.0	-0.19
	7.8724	770-840	4.0	+0.01
	7.9724	770-840	4.0	-0.01
304L ^a	7.8911	570-630	4.0	-0.06
	7.8911	570-630	4.0	-0.07

^aSafety rod thimble, EBR-II.

^bEBR-II subassembly XO-34.

Transmission electron microscopy of these specimens showed that irradiation temperature markedly influenced both dislocation and void structures. Below 470°C, faulted dislocation loops like those shown in Fig. 5.1(a) formed during irradiation. During irradiation at 570 to 630°C, a structure consisting of a dislocation network and a few unfaulted dislocation loops formed [Fig. 5.1(b)] in types 304 and 304L stainless steel. The transition in dislocation structure appears to be governed primarily by irradiation temperature rather than composition or other variables. At irradiation temperatures of about 400°C, these same heats of stainless steel contained faulted dislocation loops. At 770 to 840°C no dislocation structure was produced by irradiation.

Fig. 5.1. Effect of Irradiation Temperature on the Dislocation Structure in Type 304 Stainless Steel. (a) Faulted dislocation loops formed during irradiation below 470°C; (b) unfaulted dislocation loops and dislocation network formed during irradiation at 570 to 630°C.

The void structures produced by irradiation are shown in Fig. 5.2. Several qualitative observations can be made. With increasing irradiation temperature to 570 to 630°C, the concentration of voids decreases, the size increases, and the shape becomes more polyhedral. In agreement with previous observations² made on specimens irradiated to relatively low fluences at 370 to 470°C (which, thus, contained low concentrations of voids), a large fraction of the voids in the specimen irradiated at 570 to 630°C were associated with dislocation lines and precipitate particles. The specimen irradiated at 770 to 840°C contained cavities both within the matrix and at the grain boundaries. The cavities within the matrix had maximum diameters of about 100 Å and those at grain boundaries had maximum diameters of about 275 Å. The lack of dislocation structure, the presence of cavities at grain boundaries, and the fact that the cavities are smaller than the voids in the corresponding specimen irradiated at 570 to 630°C suggest that the cavities present after irradiation at high temperatures are bubbles of He. Experiments are in progress to determine the behavior of these cavities during annealing after irradiation.

Fig. 5.2. Effect of Irradiation Temperature on the Concentration and Size of Voids in Type 304 Stainless Steel Irradiated to 3.7×10^{22} neutrons/cm² (> 0.1 Mev). The irradiation temperatures were (a) 370 to 380, (b) 430 to 440, (c) 570 to 630, and (d) 770 to 840°C.

The concentration of voids is shown as a function of irradiation temperature in Fig. 5.3. At each fluence level, the concentration of voids decreases with increasing irradiation temperature. The curves also reflect the previous observation that the concentration of voids increases at a faster rate at 460 to 470°C than at 370 to 380°C.

Fig. 5.3. Void Concentration as a Function of Irradiation Temperature for Type 304 Stainless Steel.

Previous results showed that the maximum diameter of the voids is essentially independent of the fast-neutron fluence for fluences greater than about $1 \times 10^{22} \text{ neutrons/cm}^2$ ($> 0.1 \text{ MeV}$). Figure 5.4 shows that the maximum diameter of the voids is a sensitive function of the irradiation temperature: it increases as the temperature increases.

The Development of Concentration Gradients Around Voids in Irradiated Stainless Steel (J. O. Stiegler, E. E. Bloom, B.T.M. Loh)

Although macroscopic swelling due to the formation of microscopic voids is a common feature of metals and alloys irradiated at elevated temperatures, the nature of the void population depends greatly on the material under consideration. For example, the fluence at which voids

Fig. 5.4. Maximum Diameter of Voids as Function of Irradiation Temperature for Specimens Irradiated to Fluences Above 1×10^{22} neutrons/cm². The numbers in parentheses indicate the number of observations.

are first observed in Al of commercial purity is three orders of magnitude higher than that at which they are first seen in high-purity (99.9999%) Al (ref. 3). A similar difference exists between Ni and type 304 stainless steel.^{4,5} There are significant differences in the concentration and size of voids even among stainless steels of similar composition irradiated under identical conditions.

A description of these differences is of both fundamental and applied interest. Such observations may guide our development of theoretical models that describe the process, and, of course, existing models must explain the differences to remain under consideration. Practically, an understanding of the factors that influence the concentration and size of voids will aid in the development of materials that are more resistant to swelling.

The striking contrast between the dependence of void concentrations and sizes on fluence in stainless steel and in Ni is illustrated in Figs. 5.5 and 5.6. The transmission electron photomicrographs of

Fig. 5.5. Void Structure in Type 304 Stainless Steel Irradiated in the Experimental Breeder Reactor-II at About 470°C to a Fluence of (a) 3×10^{21} and (b) 2×10^{22} neutrons/cm².

Fig. 5.6. Void Structure in Nickel 270 Irradiated in the Experimental Breeder Reactor-II at About 470°C to a Fluence of (a) 1.4×10^{20} and (b) 1.5×10^{22} neutrons/cm².

stainless steel irradiated at 470°C to fluences of 3×10^{21} and 2×10^{22} neutrons/cm² (> 0.1 Mev) in Fig. 5.5 show that in this material the concentration of voids increases with increasing fluence but that the maximum void size, reached at 3×10^{21} neutrons/cm², increases little, if any, as the fluence is increased by nearly an order of magnitude. Measurements of the concentration and maximum size of voids as functions of fluence for irradiation temperatures of 370 and 470°C , shown in Figs. 5.7 and 5.8, confirm this observation. Identical behavior was noted for material irradiated at intermediate temperatures to fluences as high as 7×10^{22} neutrons/cm². Over the temperature range in which voids have been observed (350 to 650°C), the concentration of voids increases with increasing fluence, and the maximum size

Fig. 5.7. Concentration of Voids as a Function of Fluence for Type 304 Stainless Steel Irradiated in the Experimental Breeder Reactor-II at 370 to 470°C .

Fig. 5.8. Maximum Size of Voids as a Function of Fluence for Type 304 Stainless Steel Irradiated in the Experimental Breeder Reactor-II at 370 to 470°C.

of the voids varies little with fluence but increases with increasing irradiation temperature.

Photomicrographs of Nickel 270, shown in Fig. 5.6, illustrate essentially the opposite behavior; that is, the concentration of voids seems to become saturated at a relatively low fluence, but the size of the voids appears to increase without limit. In fact, in Fig. 5.6(b) a large fraction of the voids have diameters larger than the foil thickness and result in the circular, indistinct images. An increase in fluence from 1.4×10^{20} to 1×10^{22} neutrons/cm² at 470°C results in no appreciable change in void concentration (about 10^{14} /cm³) but does cause an increase from 225 to about 1500 Å in the maximum size of the voids.

Lauritzen *et al.*⁶ recently reported significant differences in swelling and in the concentrations and sizes of voids in types 304 and 347 stainless steel irradiated in the EBR-II to a fluence of 3.4×10^{22} neutrons/cm² (total) at about 660°C. The voids in the type 347 stainless steel were significantly smaller, although slightly more numerous, than those in type 304 stainless steel. In addition, the type 347 stainless steel swelled less. Bloom and Steigler⁷ compared swelling and microstructures in a standard type 304 stainless steel and a similar alloy modified by the addition of 0.18 wt % Ti.

They found for irradiation to a fluence of about 1.6×10^{22} neutrons/cm² (> 0.1 Mev) at $450 \pm 50^\circ\text{C}$ that the Ti-modified alloy swelled less due to a decreased concentration of voids; the distributions of void sizes were equivalent in the two alloys.

These observations show that, even within the austenitic stainless steels, minor variations in composition can significantly change void concentrations and maximum and average sizes. A model describing void formation must be sensitive to these changes in composition and explain the apparent occurrence of a limiting void size in stainless steel and the lack of a limiting void size in Ni. It must also predict the saturation of void concentration at low fluences in Ni but not in stainless steel. We shall attempt to rationalize these effects by examining some of the factors that influence the growth of voids.

Growth of voids has been considered to be limited by diffusion of vacancies to the void or by adsorption of vacancies onto void surfaces or other locations.

Bullough et al.⁸ derived an expression for the growth rate of voids when that growth is limited by absorption of vacancies at steps on the surface of the void. Their equation for growth rate is independent of the void radius. That is, if growth is limited by this process, a small void will experience the same change in radius as a large void during a given radiation increment. The curve for the distribution of the void sizes, then, reflects the nucleation rate. The shape of that end of the curve that represents the large void sizes should not change as the irradiation proceeds but rather should be translated as a unit to larger void sizes. The newly generated voids make up the portion of the curve that represents small void-size range.

Such a translation of the curve for distribution of void sizes is not observed experimentally as the irradiation progresses. In stainless steel, the maximum void size does not increase (for fluences greater than 3×10^{21} neutrons/cm²), but the concentration of voids in the largest size interval continually increases. In high-purity Al, the maximum void size increases during the irradiation, but the curve changes shape.⁹ We conclude that in these materials surface absorption does not

limit void growth. Studies of the annealing of dislocation loops¹⁰ and voids^{11,12} in thin foils have shown that dissolution is governed by diffusion of vacancies and not surface emission. Accordingly, we shall treat void growth as a process limited by diffusion of vacancies to the void and examine the factors that might account for the limiting void size.

For the case in which void growth is controlled by diffusion and the vacancy concentration, N_v , is much greater than that in equilibrium with a cavity of radius, r , we may write $dr/dt = (N_v D_v)/r$, where D_v is the coefficient for diffusion of vacancies and t is the time. We expect that N_v will vary with time since concentrations of voids and loops (i.e., sink concentrations) change with time:

$$rdr = D_v N_v dt \quad (5.1)$$

$$\frac{r_2^2}{2} - \frac{r_1^2}{2} = D_v \int_{t_1}^{t_2} N_v(t) dt .$$

Without knowing the time variation of N_v we cannot integrate the expression on the right. We can, however, evaluate it for a given fluence increment by examining the change in radius of certain voids.

At a fluence of 4.8×10^{21} neutrons/cm² at 370°C, we observed 2×10^{15} voids/cm³ greater than 50 Å in diameter (our limit of resolution) and a maximum void size of 200 Å. At a fluence of 5×10^{22} neutrons/cm², we counted 10×10^{15} voids/cm³ greater than 50 Å in diameter and 2×10^{15} voids/cm³ greater than 150 Å in diameter. The maximum void size was again 200 Å. Therefore, during this fluence increment, an appreciable number of voids were nucleated or grew from an invisible size to a diameter of 150 Å. If we evaluate the integral in Eq. (5.1) for the most conservative case in which a void grows from just below the limit of visibility to 150 Å ($r_1 = 25$ Å and $r_2 = 75$ Å), Eq. (5.1) then becomes

$$\begin{aligned} (7.5 \times 10^{-7})^2 - (2.5 \times 10^{-7})^2 &= 2 D_v \int_{t_1}^{t_2} N_v(t) dt \quad (5.2) \\ &= 50 \times 10^{-14} \text{ cm}^2 . \end{aligned}$$

A void that was 100 Å in radius at the beginning of this increment should grow to a final size given by

$$\begin{aligned} r_2^2 - (10 \times 10^{-7})^2 &= 50 \times 10^{-14} \\ r_2^2 &= 150 \times 10^{-14} \\ r_2 &= 122 \text{ Å} . \end{aligned} \tag{5.3}$$

That is, a void that was 200 Å in diameter at 4.8×10^{21} neutrons/cm² should grow to a diameter of 244 Å at 5×10^{22} neutrons/cm². We are capable of clearly detecting this amount of growth. Since we do not observe it, we conclude that the apparent limiting void size observed in stainless steel is not due solely to a depletion of the supersaturation of vacancies as a result of an increase in sink concentration. The only other term in Eq. (5.1) that can vary is the coefficient for diffusion of vacancies.

Variation of the coefficient for diffusion of vacancies is not inconceivable in an alloy in which solute segregation may be induced by a vacancy flow. Segregation will change local solute concentrations and, consequently, coefficients for diffusion of vacancies. This situation is the inverse of the well-known Kirkendall effect, which occurs when two dissimilar metals or alloys are joined together in a temperature range at which diffusion can occur. The different atomic species generally diffuse at different rates, and, since diffusion occurs by a vacancy process, a vacancy flow also occurs to counter the flow of the faster moving component. That is, a concentration gradient induces a vacancy flow. In a homogeneous alloy, the inverse process occurs during neutron irradiation at temperatures at which vacancies are mobile. Early in the irradiation, the lattice becomes supersaturated with vacancies, the concentration depending on the flux, temperature, and mechanisms of their annihilation. The situation changes, however, when a void is nucleated; the void acts as a vacancy sink and results in a vacancy concentration gradient around it. This sets up a net flow of vacancies into the void, which in turn induces a flow of atoms away from the void. If the different atoms in the alloy diffuse at different rates, as is generally the case, a shell of material with lower

diffusivity will gradually build up around the void. This is, in a sense, an inverse Kirkendall effect.

Such segregation as a result of a vacancy flow may also occur during heating, cooling, or sintering. The general aspects of the problem were treated theoretically by Anthony,¹³ and experimental evidence for segregation during sintering was obtained by Kuczyuski *et al.*¹⁴ By metallographic examination of the necked regions of sintered wires of a Cu-8% In alloy in which vacancy currents were induced by differences in radius of curvature, Kuczyuski *et al.* observed precipitation of an In-rich phase that could have occurred only by movement of the faster moving In atoms into the necked region.

Anthony pointed out that segregation may be induced by vacancy flow by two processes: (1) the reverse flow of atoms, mentioned above, which is a consequence of vacancy flow, and (2) the dragging of solute atoms by vacancies. The latter occurs when the energy of attraction between a vacancy and solute atom is much greater than the thermal energy. Following Anthony, we shall consider the distribution of solute around a void for the former (weak binding) case.

Consider a thin shell of material surrounding a void in a binary alloy composed of A and B atoms. The composition in the shell will change until the fluxes of solute and solvent atoms (J_A and J_B) entering and leaving the shell are proportional to their atom fractions in the shell (C_A and C_B); that is, until $J_A/J_B = C_A/C_B$. Anthony showed that, for a dilute solution of B in A and a weak interaction between solute atoms and vacancies, the solute concentration for a one-dimensional gradient (direction X) can be described by

$$\frac{\partial \ln C_B}{\partial X} = \frac{\partial \ln C_V}{\partial X} \frac{(D_B - D_A)}{D_B} \quad (5.4)$$

where C_V is the concentration of vacancies and D_B and D_A the diffusion coefficients for B and A atoms, respectively. If the diffusivity of the solute is less than that of the solvent ($D_B < D_A$), the region in the vicinity of the void will be enriched in solute, the degree of enrichment depending on the vacancy concentration gradient and the

difference in diffusivities of the elements. If $D_B > D_A$, the region in the vicinity of the void will be enriched in solute, the degree of enrichment depending on the vacancy concentration gradient and the differences in diffusivities of the elements. If $D_B < D_A$, the region adjacent to the void will be depleted of solute. In either case the diffusion coefficient near the surface of the void will be lower than that in the original alloy.

The difference between D_B and D_A must necessarily decrease with increasing temperature. This would require a larger void before sufficient segregation could occur to reduce the growth rate to a negligible level. The increase in the apparent limiting void size with increasing temperature is consistent with this interpretation.

Such a process could, of course, occur only in an alloy, not in a pure metal. It should significantly slow the void growth rate but not totally stop it. It is possible that the greatly reduced rate of void growth observed in stainless steel results from a combination of the vacancy depletion effect, discussed earlier, and solute segregation.

It is interesting to consider the observations of Lauritzen et al.⁶ in this light. They found that voids formed in type 347 stainless steel were considerably smaller than those formed in type 304 stainless steel. It is difficult to make any meaningful calculations of the nature and extent of segregation in an alloy as complex as stainless steel, but we can speculate that differences in composition lead to buildup of a layer of sufficiently low diffusivity at a smaller void size in the type 347 stainless steel.

It is also possible that this process may explain the saturation of void concentration observed in Ni and the apparent lack of saturation observed in stainless steel. If He atoms generated by (n, α) reactions are involved in the nucleation of voids, the rate of nucleation will be related to the concentration of gas dissolved in the lattice. If the gas is mobile during irradiation, the void population will serve as effective sinks, at least in the case of Ni, where there is no barrier to diffusion of gas atoms into the voids. In the case of stainless steel, however, the development of a layer of low diffusivity around

voids could reduce their effectiveness as sinks and thereby increase the rate of void nucleation because of the enforced solution of the gas.

We suggest that the differences in the irradiation behavior of stainless steel and Ni may be due in part to the development of a layer of low diffusivity around voids in the stainless steel as a result of solute segregation induced by vacancy flow. Such a layer would reduce the rate of growth of existing voids and reduce the effectiveness of the voids as sinks for He atoms. The increased He concentration in the lattice would tend to increase the rate at which voids were nucleated. The increased concentration of voids would, in turn, further deplete the vacancy supersaturation and further decrease the rate of growth.

We believe that solute segregation should occur as a result of vacancy flow. Whether or not it is the cause of the observed behavior is a matter of speculation. More careful experiments on simpler binary alloys may provide the proof. In situ measurements of the annealing kinetics of voids by means of the electron microscope may show anomalous behavior that can be related to the concentration gradient. That is, the coefficient for diffusion of vacancies may appear to increase with decreasing void size. We believe that it is important to understand the effect, for compositional modifications that decrease the limiting void size in alloys may prove useful in controlling the swelling of reactor materials.

Void Distributions as Monitors of Irradiation Temperature and Fluence (J. O. Stiegler, E. E. Bloom)

A preliminary survey of the concentrations and sizes of voids formed in type 304 stainless steel irradiated in EBR-II at 370 to 650°C and to fluences of 2 to 70×10^{21} neutrons/cm² (> 0.1 Mev) indicated that the configuration of voids is a unique function of the irradiation temperature and fluence. Specifically, at constant fluence, the concentration of voids decreases and the maximum size of the voids increases with increasing irradiation temperature. At constant temperature, the concentration of voids increases with increasing fluence, but the maximum size of the voids changes little at fluences above about 1×10^{22} neutrons/cm².

Quantitative plots of void concentrations and maximum void sizes as functions of fluence for irradiation temperatures of 370 and 470°C are shown in Figs. 5.7 and 5.8. A curve depicting the maximum size of voids as a function of irradiation temperature for fluences above 1×10^{22} neutrons/cm² is given in Fig. 5.4. In order to use microstructural characteristics as monitors of temperature and fluence one must first determine the maximum size of the voids as an indication of the irradiation temperature. Once the temperature is known, the concentration of voids can be read directly from the plots developed by Straalsund *et al.*¹⁵ for void concentration, temperature, and fluence, since the concentration of voids is a unique function of fast-neutron fluence. The maximum size of the voids must be used, since the average size of the voids increases with increasing fluence, but the maximum size does not. As an additional temperature reference we observe that interstitials precipitate as faulted dislocation loops at irradiation temperatures below about 550°C and as unfaulted loops at higher temperatures.

Present data are not extensive or accurate enough to permit quantitative determinations of irradiation conditions, but they do allow evaluation of reported conditions and at least a qualitative determination of temperature and fluence for cases where no monitors were present. As additional data become available, the accuracy of estimates based on microstructural characteristics should improve.

We can illustrate the use of this technique by evaluating some observations made by Lauritzen *et al.*⁶ on type 304 stainless steel irradiated in the EBR-II to a total fluence of 3.4×10^{22} neutrons/cm² at 660°C, as determined by fusible-metal thermometry. Unfortunately, the specimens were taken from gage and grip regions of a tensile specimen that had been tested at 704°C; therefore, no evidence was retained of the original dislocation-loop structure. A maximum void diameter of 550 Å was observed in the gage sections and in one specimen taken from a grip region.¹⁶ In a second grip specimen, a similar distribution of void sizes was obtained with the exception that a few voids were as large as 900 Å in diameter.¹⁶ The average concentration of voids was about 1×10^{15} voids/cm³.

On the basis of our observation that the maximum size of voids is a function of irradiation temperature, we suggest that this material was irradiated at 550 to 600°C. The presence of some very large voids in one region of the grip may signify that part of the specimen ran at an appreciably higher temperature, perhaps as high as 660°C, for at least part of the irradiation. We conclude from the microstructural characteristics that the bulk of the specimen was not irradiated at 660°C but rather at a temperature 50 to 100°C lower. A local hot spot or transient heating effect may have been responsible for the higher irradiation temperature established by fusible-metal thermometry.

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