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SUMMARY

In 316 stainless steel and other alloys, nickel and silicon appear to be major determinants of the radiation-induced swelling behavior. In this study it has been shown that the fast-diffusing species concept proposed by Venker and Ehrlich appears to be a viable mechanism for suppression of void nucleation by silicon atoms. While the role of silicon can be ascribed at least partially to the fast-diffusion effect, the role of nickel cannot, and its influence lies in the operation of other physical mechanisms.

The enhanced diffusivity of the alloy which results from the addition of silicon leads to a substantial increase in the free energy barrier to void nucleation, particularly at higher temperatures. The fast-diffusion mechanism operates in addition to the interstitial-solute binding effect, and the combined fast-diffusion/interstitial binding model dispenses with the requirement of unrealistically large interstitial-solute binding energies needed for the solute-binding concept. Solute binding may account for most of the observed silicon segregation behavior while fast-diffusion accounts for the void suppression.

The addition of slow-diffusing elements has an opposite but less pronounced effect on void nucleation.

INTRODUCTION

There now exists ample evidence that certain minor alloying elements have a pronounced effect of delaying neutron-induced void formation in many metals and alloys. This suppression of nucleation is effective only as long as the active elements remain in solution. It appears that during irradiation these elements are inevitably removed from the alloy matrix, however, which can lead to substantial alteration of the matrix composition due to coprecipitation of major alloy components into various second phases. $^{(1-3)}$ Venker, Ehrlich and Giesecke $^{(4,5)}$ have recently conjectured that there exists a correlation between the suppression of void formation and the presence of fast-diffusing solute or solvent elements. They did not demonstrate, however, that such a correlation arises from a specific physical mechanism.

The conjecture of Venker et al appears to be an alternative to the concept that defect trapping at solute elements reduces void formation. This possibility

has been proposed and analyzed by several authors. $^{(6)}$ The trapping of interstitials and/or vacancies at solute atoms or impurities enhances the probability of recombination, and thereby lowers the supersaturation of vacancies. However, for parameters typically assumed for nickel the enhanced recombination by trapping is only significant for vacancy-solute binding energies of 0.2 - 0.3 eV or greater $^{(6)}$ or for interstitial-solute binding energies of the order of or greater than the vacancy migration energy. Binding energies of this magnitude have not been confirmed by experiment for interstitial-solute complexes in fcc metals, and measured vacancy-solute binding energies are generally 0.2 eV or less for substitutional solute elements. $^{(7)}$ The action of trapping and detrapping may be expressed in terms of reducing the effective diffusion coefficient for the point defect, as has been shown by Mansur. $^{(6)}$ Enhanced recombination by trapping implies slower diffusion rather than faster diffusion.

To determine the relative contributions of the trapping mechanism and the conjecture of Venker et al, it is important to realize that the former process requires immobile or slowly diffusing solute atoms, whereas the latter assumes highly mobile solute atoms. It will be shown in this report that when fast-diffusing solute atoms enhance the vacancy mobility, the vacancy supersaturation is reduced, and the void nucleation rate reduced. Conversely, slow-diffusing solute elements reduce the vacancy mobility, and thereby enhance void nucleation. This appears to contradict the prediction of Mansur based on the action of immobile traps. (6)

However, as shown in the following section the difference between the two results is due to different assumptions concerning the role of trapped vacancies as void nucleation sites. Whereas Mansur considers the traps to be saturable and therefore assumes that bound vacancies cannot be sites for void nucleation, the treatment presented in this paper allows both free and bound vacancies to function not only as nucleation sites but also as defects capable of incorporation into other vacancy clusters.

In the following section the concept of effective vacancy diffusion is examined in a metal with substitutional impurities. The effective vacancy diffusion coefficient is then used in the void nucleation theory to demonstrate that fast-diffusing solutes can significantly reduce void nucleation. The results and their correlation with experimental evidence are examined in the last section.

The Effective Vacancy Migration Coefficient

The normal diffusion of substitutional impurity atoms occurs by vacancy migration. For dilute concentrations of impurities, diffusion is commonly analyzed in

terms of the five-frequency model, $^{(8)}$ from which Howard and Lidiard $^{(9)}$ have derived an effective diffusion coefficient for vacancy migration. In the present work, we employ the somewhat simpler expression by Flynn, $^{(10)}$

$$D_{V}^{eff} = (D_{V} + K C_{S} D_{S})/(1 + K C_{S}),$$
 (1)

where $D_{\rm V}$ is the diffusion coefficient for migration of a free vacancy, and $D_{\rm S}$ is the diffusion coefficient for a solute-vacancy pair. $C_{\rm S}$ is the solute concentration, and

$$K = 12 \exp \left(E_{h}/kT \right) \tag{2}$$

is the rate constant for the solute-vacancy dissociation in an fcc lattice, and \mathbf{E}_{b} is the solute-vacancy binding energy.

As shown by Howard and Lidiard, equation (1) is valid if the concentration of bound vacancies is small compared to the solute concentration C_s . Considering the fact that the steady-state vacancy concentration in metals irradiated in a fast neutron flux is of the order of 10^{-6} per atom or less, this condition is met. A further restriction on the validity of equation (1) is that the jump frequency of a free vacancy into a bound position must not differ greatly from the jump frequency of an isolated vacancy. Once formed, however, the vacancy-solute pair may possess a jump frequency that differs substantially from that of the free vacancy.

It should be noted that equation (1) is equally applicable when the flow of solute is in the same or opposite direction to the flow of vacancies. Therefore, both solute drag and inverse Kirkendall effects are compatible with this treatment.

Equation (1) was derived assuming a quenched-in vacancy supersaturation in the absence of self-interstitials. Therefore, its application to metals subject to irradiation is suspect. Mansur (11) has derived an expression for D_V^{eff} which is more general than equation (1), and which incorporates the effect of trapping and the recombination of interstitials and bound vacancies, as well as the transport of bound vacancies to sinks. He has shown, however, that trapping and bound recombination reactions can be neglected whenever the binding energy $E_b \stackrel{\sim}{<} 5$ kT. Transport of bound vacancies were shown to exert a lesser influence than free vacancies on the effective diffusion coefficient in equation (1) when

$$E_{b} \gtrsim [10 + \ln (D_{v}/D_{s})] kT.$$
 (3)

As long as $E_b \stackrel{\sim}{<} 5$ kT, condition (3) is also satisfied when D_s is no larger than about 100 D_v .

All these conditions are met for vacancy-solute binding energies in the range of measured values. Therefore, equation (1) may be used as the effective vacancy diffusion coefficient in the conventional rate theory in which the interstitial mobility is much greater than the vacancy mobility.

The vacancy diffusion coefficient for nickel is given by

$$D_{v} = 0.0153 \exp(-E_{v}^{m}/kT)$$
, (4)

with E_{V}^{m} = 1.4 eV. For an imaginary solute atom in nickel we assume a diffusion coefficient of

$$D_{s} = 0.0153 \exp(-E_{s}^{m}/kT)$$
, (5)

where E_S^m differs from E_V^m at the most by ± 0.2 eV.

Figure 1 shows the effective vacancy diffusion coefficient D_{V}^{eff} for the case that E_{S}^{m} = 1.2 eV and E_{b} = 0.05 eV, and for solute concentrations up to 5 at %. (Above 1% the dilute solute assumption may not be valid, according to reference 6.) It is seen that small additions of a fast diffusing solute element greatly enhance the vacancy mobility. Note that relatively minor solute additions are quite effective and that additional solute has a proportionally smaller effect. With increasing solute content, D_{V}^{eff} approaches D_{S} at a rate governed by the sign and magnitude of the binding energy E_{b} .

Void Nucleation Rates

To study the impact of fast-diffusing solute elements on void formation, the nucleation theory of Katz, Wiedersich and Russell (12) (KWR) is utilized in conjunction with the standard rate theory. In the latter, however, the effective vacancy diffusion coefficient D_{v}^{eff} is substituted for D_{v} . The nucleation barrier energy $\Delta G(x)$ has been given previously, (13,14) and can be written in the form

$$\Delta G(x)/kT = -\sum_{n=2}^{x-1} \ln \left\{ \left[\lambda(n) \Delta C_{v} + C_{v}^{\circ}(n) \right] / \left[\Delta C_{v} + C_{v}^{eq} \right] \right\}, \qquad (6)$$

where

$$\lambda(n) = (1 + \frac{1}{n})^{1/3} \frac{Z_{1}^{\circ}(n+1)}{Z_{v}^{\circ}(n)} \frac{\overline{Z}_{v}}{\overline{Z}_{i}}, \qquad (7)$$

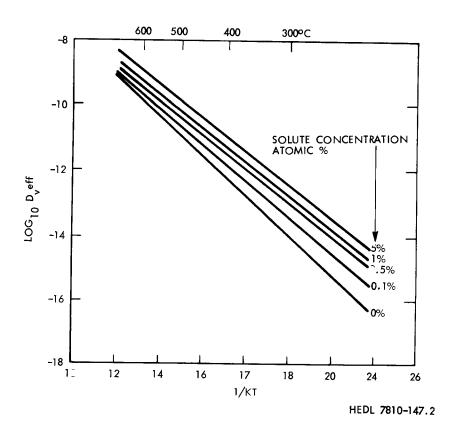


FIGURE 1. Calculated Effective Diffusion Coefficient for Vacancies in Nickel with Various Concentrations of Fast-Diffusing Solute.

 ΔC_V is the radiation-produced vacancy concentration over and above the average thermal vacancy concentration \bar{C}_V^{eq} in equilibrium with the sinks, and $C_V^{\circ}(n)$ is the vacancy concentration in equilibrium with a void containing n vacancies. Z_1° and Z_V° are the interstitial and vacancy capture efficiencies of voids, and \bar{Z}_1 and \bar{Z}_V are average capture efficiencies of all sinks. All these quantities have been given and discussed extensively elsewhere. (13,14)

The excess vacancy concentration $\Delta C_{_{\mbox{$V$}}}$ can easily be obtained from the rate equations. Using the materials parameters for nickel as given in Reference 14 and a displacement rate of 10^{-6} dpa/sec, the nucleation barrier $\Delta G(x)/kT$ for "pure nickel" is obtained from equation (6) as a function of the number of vacancies, x, contained in the void, and the results are shown in Figure 2 for different temperatures. The numbers in brackets are the steady-state void nucleation rates given by

$$I = 2 (6\pi^{2}\Omega)^{1/3} D_{v}^{eff} (\Delta C_{v} + \overline{C}_{v}^{eq})^{2} \left\{ \sum_{x=2}^{\infty} \frac{\exp \left[\Delta G(x)/kT\right]}{x^{1/3} Z_{v}^{o}(x)} \right\}^{-1}.$$
 (8)

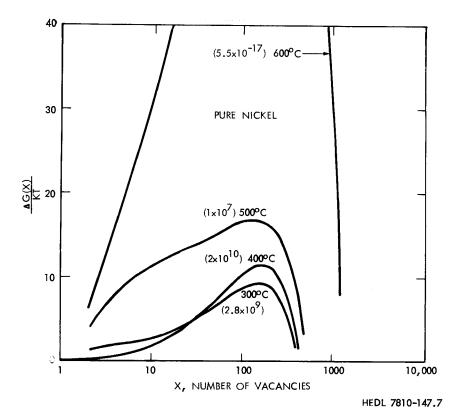


FIGURE 2. Void Nucleation Barrier Calculated for "Pure Nickel." Numbers in brackets are the steady-state nucleation rates per cm³ and sec.

In the following, the void nucleation rate for the pure material, i.e., when $D_V^{eff} = D_V$, will be designated by I_O . Note that the nucleation rate for 600°C irradiation is very low because the influence of helium atoms has not been included. It has been shown earlier, however, that the ratios I/I_O are relatively insensitive to the presence of gas when studying the parametric influence of variables such as stress. (14)

The effect of 1 at. % of solute atoms whose activation energy for migration is $E_s^m = 1.2$ eV and whose binding energy with vacancies is $E_b = 0.05$ eV is shown in Figure 3.

Due to the enhanced mobility of vacancies, the nucleation barrier is raised for temperatures where recombination does not dominate the point defect concentrations and the steady state nucleation rate, I, is decreased. This is more clearly illustrated in Figure 4 which gives the relative nucleation rates I/I_0 as a function of the solute concentration C_S .

These results not only confirm the conjecture of Venker et al, but at the same time demonstrate that fast-diffusing solute additions reduce the void nucleation rate and thereby increase the incubation dose for void swelling. The effect can be interpreted in a simple manner. The enhancement of vacancy mobility by addition of fast-diffusing solutes is analogous to an increase in temperature. In both cases the vacancy supersaturation is reduced, and the void nucleation rate depressed.

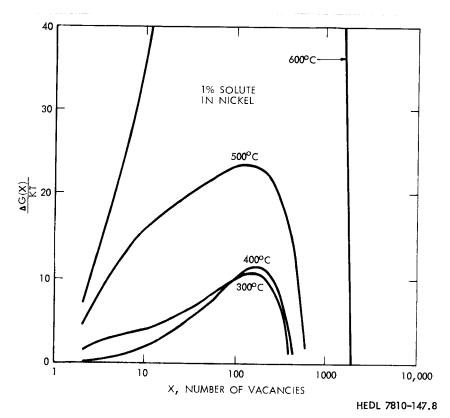


FIGURE 3. Free-Energy Barriers to Void Nucleation Calculated for Nickel-1% Solute Alloy.

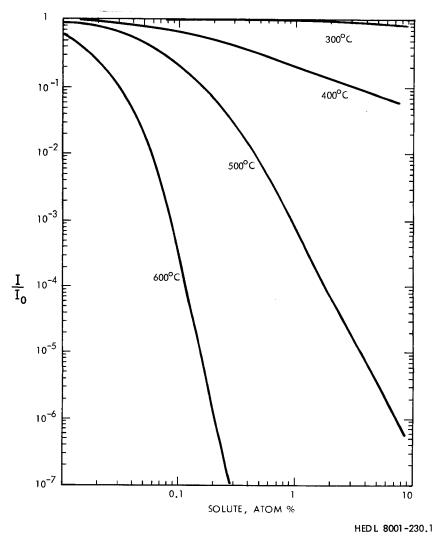


FIGURE 4. Relative Void Nucleation Rates for Solute Additions with $E_{\rm S}^{\rm m}$ = 1.2 eV and $E_{\rm b}$ = 0.05 eV.

This argument can be carried further by considering the influence of slow-diffusing solute additions. Accordingly, several cases have been investigated wherein the solute possesses a migration energy of $E_S^m=1.6~{\rm eV},$ i.e., 0.2 eV higher than E_V^m . Figure 5 gives the results for 0.1 at. % solute additions. The lower solid curve shows again the fast-diffusing case for solutes with $E_S^m=1.2~{\rm eV}$ and $E_D^m=0.05~{\rm eV}$, whereas the upper solid curve is for a slow-diffusing solute with $E_S^m=1.6~{\rm eV}$ and $E_D^m=0.05~{\rm eV}$. Although the nucleation rate is increased with slow-diffusing solutes, the effect is less pronounced than the corresponding suppression of nucleation by fast-diffusing solutes, provided the binding energy is small.

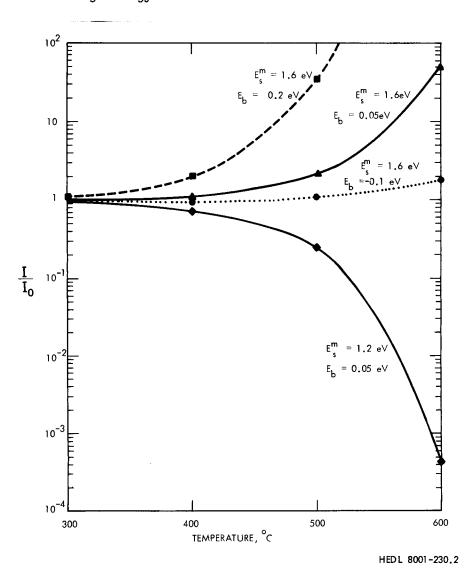


FIGURE 5. Effect on the Relative Void Nucleation Rate of Slow and Fast-Diffusing Solute with 0.1 at. % Concentration.

For strong binding with E_b = 0.2 eV, the slow-diffusing solute (E_s^m = 1.6 eV) produces a more significant increase in the void nucleation rate, as shown by the upper dashed curve. This latter case is considered a very important demonstration that solute trapping of vacancies does not necessarily lead to a reduction in void swelling. In fact, for reasonable binding energies and for a slower but finite mobility of the solute the present analysis leads to the opposite conclusion. This in no way contradicts earlier work on the effectiveness of immobile traps on reducing swelling (15) and irradiation creep. (16) Rather, it emphasizes the fact that effective trapping requires probably small precipitates or clusters of impurities. Conversely, solutes with low mobility are enhancing void nucleation, and a higher void number density may be obtained.

As mentioned previously Mansur⁽⁶⁾ predicted that immobile traps would decrease void nucleation, whereas in this paper slowly-diffusing elements are predicted to enhance nucleation. The apparent contradiction arises not from the treatment of migration or binding energies but in the role of bound vacancies as nucleation sites. The consequences of the different assumptions can be easily demonstrated. Note that in equation (8) that the nucleation rate depends on the second power of the vacancy concentration $C_V = \Delta C_V + \overline{C}_V^{eq}$. The second power expresses the requirement that the nucleation rate is proportional to the number of vacancies C_V and the number of impinging vacancies D_V^{eff} C_V . In this paper it is assumed that C_V is comprised of the sum of free vacancies C_V^f and bound vacancies C_V^b . In contrast the bound traps of Mansur are considered to be saturable and therefore not available for void nucleation. Therefore in comparable notation Mansur's nucleation rate I' is given by

$$I' = 2 (6\pi^{2}\Omega)^{1/3} D_{v}^{eff} (C_{v} C_{v}^{f}) \left\{ \sum_{x=2}^{\infty} \frac{\exp \left[\Delta G(x) / kT \right]}{x^{1/3} Z_{v}^{o}(x)} \right\}^{-1}.$$
 (9)

The ratio of the two predicted nucleation rates is

$$\frac{I}{I^{T}} = \frac{C_{V}}{C_{V}^{f}} = 1 + \frac{C_{V}^{b}}{C_{V}^{f}} , \qquad (10)$$

and according to the definition of the effective vacancy diffusion coefficient $^{(6)}$

$$D_{\mathbf{v}}^{\mathsf{eff}} C_{\mathbf{v}} = D_{\mathbf{v}} C_{\mathbf{v}}^{\mathsf{f}} + D_{\mathsf{s}} C_{\mathbf{v}}^{\mathsf{b}}, \qquad (11)$$

where $\mathbf{D}_{\mathbf{S}}$ was earlier defined as the solute diffusion coefficient. Thus,

$$\frac{C_{\mathbf{v}}^{\mathbf{b}}}{C_{\mathbf{v}}^{\mathbf{f}}} = \frac{D_{\mathbf{v}} - D_{\mathbf{v}}^{\mathbf{eff}}}{D_{\mathbf{v}}^{\mathbf{eff}} - D_{\mathbf{s}}} . \tag{12}$$

The effective diffusion coefficient is also defined in equation (1), leading to the conclusion that for a given solute concentration C_S

$$\frac{I}{I'} = 1 + K C_s . \tag{13}$$

For strong binding conditions typical of that considered by Mansur the nucleation rate ratio

$$\frac{I}{I}$$
 % K C_s , (14)

and is always larger than unity. Thus, the nucleation rates defined in this study are always larger than those of Mansur for conditions of strong binding. The assumption that both bound and free vacancies can function as nucleation sites is considered to be the more realistic assumption.

The interaction of a vacancy with a substitutional solute atom may be repulsive rather than attractive, in which case E_b is negative. This possibility may particularly exist with slow-diffusing solute additions. Therefore, such a case was considered wherein $E_s^{m} = 1.6$ eV and $E_b = -0.1$ eV. The repulsive interaction of a vacancy and a solute offsets the contribution of the solutes to the effective vacancy mobility, and void nucleation is not affected much (as also shown in Figure 5).

DISCUSSION

Silicon as a Fast-Diffusing Species in AISI 316: An Assessment

The above calculations demonstrate that fast-diffusing substitutional elements suppress void nucleation even at very low solute concentrations. This phenomenon is independent of any solute-defect binding processes and exhibits a saturation behavior with increasing solute. The calculations also show that the degree of suppression varies with temperature. The important question to answer is whether this proposed mechanism is one of the dominant mechanisms by which solutes such as silicon influence void growth in AISI 316. Recently acquired data (3) on the effect of silicon content on the swelling of this alloy indicate that silicon

indeed exerts a strong temperature-dependent influence on void nucleation while in solution but is inevitably removed into various second phases, (1) whereupon void nucleation commences.

In thermal environments, silicon is thought to migrate in nickel by vacancy exchange mechanisms, as evidenced by its activation energy for diffusion (61.7 kcal/gram atom). (17) It has also been shown that the addition of silicon leads to a reduction in void number density in 316 stainless steel at low displacement levels in both neutron and electron irradiations. Other alloys also show a suppression of void nucleation with silicon. Since its diffusivity is from two to three orders of magnitude greater than that of nickel, silicon appears to be an excellent example of the fast-diffusing solute. (17)

There is, however, some tendency in the radiation-effects community to view the fast-diffusion model as being in opposition to another postulated mechanism, that of solute drag by formation of bound interstitials. This latter mechanism involves the formation of di-interstitial complexes with the smaller atoms of the lattice, lowering the effective interstitial diffusion coefficient. (18) Silicon is indeed one of the smaller atoms in austenitic alloys (29) and there is substantial evidence that solute drag effects occur. The segregation of silicon to various sinks is often cited as evidence of solute-drag effects since silicon could migrate with interstitials, while it would migrate away from the net vacancy flow. Segregation of silicon at sinks cannot be considered prima facie evidence for solute drag effects as the only operative mechanism, however. Wolfer $^{(19)}$ notes that all sinks have preferences for interstitials over vacancies and the growth of voids results only from dislocations having higher preferences for interstitials than do voids. It should be noted, however, that the net flow of interstitials over vacancies is very small for all sinks, and the enhanced diffusivity of unbound silicon would eventually result in its sampling all lattice sites. The segregation of silicon might therefore result from random migration to sinks at which the free energy of silicon is lowered. Segregation would also result if there were vacancy-solute binding. (20)

There is one type of evidence which argues very convincingly that solute drag effects operate on the silicon atoms. (21) Silicon is known to migrate to specimen surfaces during irradiation and form silicon-rich phases. If the irradiation ceases while the temperature is maintained the silicon-rich phases almost immediately dissolve. This means that the free energy state is not lower at this type of sink and the silicon must have been carried or dragged up an otherwise insurmountable energy barrier in order to form such phases.

There is therefore no doubt that solute drag effects exist, but the fastdiffusing and solute drag concepts are not necessarily mutually exclusive models. Both theories require that the bound defect concentration is relatively small compared to the total solute level, so direct competition of the two mechanisms would not be a major factor. It is quite likely that a combination of these two mechanisms is operating, and the combination would alleviate one of the major problems with the solute-binding model. While solute-binding effects can account for segregation at surfaces, exceptionally high binding energies are required to affect void nucleation. In effect, the interstitial would have to become the slowest-moving defect, which requires binding energies ≥ 0.7 eV. Some parameter studies by Okamoto et al (22) have indicated that at low damage rates $(10^{-6} \text{ dpa/sec})$ even a binding energy of interstitials to 0.1% of silicon atoms of 1.5 eV has a negligible effect in increasing vacancy-interstitial recombination. Marwick (23) has shown that even 1% of interstitial traps with binding energy of 1 eV would have a negligible effect on radiation-enhanced diffusion at a damage rate of 10^{-5} dpa/sec. Recent measurements of loop formation during electron irradiation suggest that the silicon-interstitial binding energy is only on the order of 0.26 eV, (24) although the experiment was conducted in a manner which did not take into account possible silicon segregation to foil surfaces.

There are several sets of data which support the combined fast diffusion/ interstitial binding model. Several researchers have observed silicon depletion in the irradiated zone of ion-bombarded specimens, a process which results in silicon enrichment both at the surface and beyond the ion range. (25,26) This would be the inevitable result of solute-interstitial binding. Solute-vacancy binding would not yield this result. (25) Marwick and Pillar have shown, however, that addition of silicon to nickel reduces the mobility of implanted tracer chromium atoms during ion bombardment. (23) This demonstrates a strong effect of silicon on the vacancy mobility and the subsequent diffusion of other substitutional components. Assassa and Guiraldenq (27) have recently shown that silicon additions to a Fe-16Cr-14Ni alloy increase the frequency factors for diffusion of all three solvent atoms, which also signals silicon's influence on vacancy diffusion. A similar effect was also recently found in Fe-15Cr-20Ni by Rothman and coworkers. (28)

Fast Diffusion by Solvent Atoms: An Assessment

Nickel is the slowest diffusing component of Fe-Ni-Cr ternary alloys. It also has the smallest partial molar volume of Fe-Ni-Cr-Si $^{(29)}$ alloys so that one would expect nickel to concentrate at sinks both by nickel-interstitial binding effects and by preferential out-migration of faster-diffusing Fe and Cr atoms.

Such behavior would be consistent with the coprecipitation of nickel and silicon observed in bulk material (1-3,30,31) if the coprecipitation were occurring on microstructural sinks. For example, void surfaces in AISI 316 have been observed to become enriched in nickel during irradiation, and correspondingly depleted in other solvent atoms, particularly chromium. (32) This segregation has been interpreted as evidence of the inverse-Kirkendall effect. At external surfaces, however, the situation is not quite so clear. Nickel has been observed to be depleted at surfaces in 71Fe-15Cr-14Ni $^{(23)}$ during ion bombardment, but accumulated at surfaces in 73Fe-18Cr-8Ni-1Si. $^{(18)}$ The differences in nickel behavior may represent slower segregation phenomena involving ordered phases such as Ni₃Fe. In any event the fast-diffusion effect involving solvent atoms would be expected to be slower than that involving silicon. This may explain why an irradiation-induced phase such as γ' in 316 stainless steel requires thousands of hours to form. $^{(1)}$

There does appear to be some relationship between solute and solvent migration, however. Not only do silicon and nickel coprecipitate under irradiation but the addition of silicon changes the ratios of diffusivities of the various solvent atoms in thermal diffusion studies at $1334^{\circ}\text{C.}^{(33)}$ However, these authors also showed that the fast-diffusion effect is not a viable mechanism for solvent atoms in Fe-Ni-Cr alloys.

CONCLUSION

The proposition advanced by Venker and Ehrlich that the swelling phenomenon is sensitive to the different diffusivities of its components appears to be applicable to the void nucleation process, but is only important for large differences in diffusivity. In Fe-Ni-Cr alloys the differences are large enough to produce substantial segregation at sinks of the slower-diffusing elements in response to the inverse-Kirkendall effect, but these differences are not thought to be sufficient to affect void nucleation. The element silicon not only has a large diffusivity but also has been shown to increase the diffusivity of the solvent atoms as well. Silicon in solution thus leads to a substantial change in the void nucleation barrier and results in a suppression of void nucleation. The evidence also suggests that silicon plays several other roles as well. It is involved in some type of binding interaction with point defects and strongly influences the development of second phases. The latter role is important in that it controls the matrix content of nickel, a slow-diffusing element in Fe-Ni-Cr alloys. While slow-diffusing elements such as nickel have a proportionately smaller influence on void nucleation than fast-diffusing elements, other evidence suggests that nickel influences the swelling phenomenon strongly by other mechanisms. (1,3,34)

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