ANL/ED/CP-94626
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in Iron-Chromium-Nickel Alloys***

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Conference Paper to be submitted for presentation at the
Symposium on Effects of Radiation on Materials
Seattle, WA
June 16-18, 1998

*Work supported by the U.S. Department of Energy, Materials/Chemistry, Materials Characterization, under Contract W-31-109-ENG-38.

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The Correlation Between Swelling and Radiation-Induced Segregation in Iron-Chromium-Nickel Alloys

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Abstract

The magnitudes of both void swelling and radiation-induced segregation (RIS) in iron-chromium-nickel alloys are dependent on bulk alloy composition. Because the diffusivity of nickel via the vacancy flux is slow relative to chromium, nickel enriches and chromium depletes at void surfaces during irradiation. This local composition change reduces the subsequent vacancy flux to the void, thereby reducing void swelling. In this work, the resistance to swelling from major element segregation is estimated using diffusivities derived from grain boundary segregation measurements in irradiated iron-chromium-nickel alloys. The resistance to void swelling in iron- and nickel-base alloys correlates with the segregation and both are functions of bulk alloy composition. Alloys that display the greatest amount of nickel enrichment and chromium depletion are found to be most resistant to void swelling, as predicted. Additionally, swelling is shown to be greater in alloys in which the RIS profiles are slow to develop.

Introduction

Void swelling in austenitic alloys can cause unacceptable dimensional changes in reactor structural materials. After void swelling was discovered in irradiated austenitic stainless steels [1], many studies were undertaken to determine not only the magnitude of the swelling problem but also the variables that influenced swelling (A comprehensive review of void swelling was recently written by Garner [2]). One of the major factors that has been shown to affect void swelling is the bulk composition of the structural component. A relationship between swelling and bulk composition of iron-chromium-nickel alloys was initially discovered by Johnston et al. using heavy ion irradiations [3]. At 675°C and 140 dpa, for an alloy series with constant chromium and varying nickel concentration, swelling decreased strongly with increasing bulk nickel concentration. For alloys with 15 wt% chromium, the swelling decreased from 120% for an alloy with 15 wt% nickel to around 1 % for an alloy with 45 wt% nickel. The swelling reached a minimum at intermediate nickel concentration (between 45 and 60 wt%). At higher nickel concentrations, the swelling increased slowly with increasing nickel concentration. For alloy series with constant nickel and varying chromium concentration, swelling increased with increasing bulk chromium concentration. Studies of iron-chromium-nickel alloys irradiated with neutrons to low fluence showed similar trends in void swelling as a function of bulk alloy composition [4-6]. Garner showed that the dependence of swelling on bulk composition occurs in the transient regime that precedes steady-state swelling [7]. Following the transient period, the rate of swelling in austenitic alloys reaches a constant rate of about 1%/dpa.

Radiation-induced segregation (RIS) is the non-equilibrium segregation that occurs at grain boundaries and other defect sinks such as dislocation loops and voids during irradiation of an alloy at high temperature (30 to 50 percent of the melting temperature) [8]. In irradiated iron-chromium-nickel alloys, nickel enriches and chromium depletes at defect sinks. Iron either enriches or depletes depending on the bulk alloy composition. Marwick noted that RIS of the slow diffusing nickel to void surfaces in iron-chromium-nickel alloys could

reduce the rate of void growth by reducing the arrival rate of vacancies to the void surface [9]. The nickel atoms surrounding the void surface can inhibit both void nucleation and void growth. Marwick postulated that differences in diffusion rates between different iron-chromium-nickel alloys might explain the differences in swelling behavior.

This work explores the relationship between RIS, swelling behavior, and bulk composition described by Marwick. A recent analysis of RIS in seven different iron-chromium-nickel alloys indicated that grain boundary segregation is driven primarily by differing rates of interaction between atoms and the vacancy flux [10,11]. Additionally, the rate at which chromium, nickel, and iron diffuse via vacancies changes with bulk composition. Therefore, changes in bulk composition alter the amount of segregation at any defect sink, including a void. In this work, resistance to void swelling as a function of bulk alloy composition is shown to correlate with major element segregation.

Theory

Compositional gradients in the vicinity of the defect sinks affect the rate at which defects reach the sinks. In iron-chromium-nickel alloys, chromium diffuses more rapidly via a vacancy mechanism than does iron or nickel, and therefore depletes at grain boundaries. Nickel diffuses more slowly via a vacancy mechanism than does iron or chromium and therefore enriches at grain boundaries. The enrichment of the slow diffuser nickel and depletion of the fast diffuser chromium at a defect sink reduces the continued flux of vacancies to the sink. Therefore, the reduced vacancy flux caused by RIS should reduce the rate of void swelling in iron-chromium-nickel alloys.

The flux of the vacancy concentration to a void is given by:

$$J_V = N \left[-D_V \nabla C_V + C_V \alpha (d_{CrV} \nabla C_{Cr} + d_{FeV} \nabla C_{Fe} + d_{NiV} \nabla C_{Ni}) \right] \quad (1)$$

where D_V is the diffusion coefficient of vacancies, C_V is the vacancy concentration, α is the thermodynamic factor, C_j is the concentration of the j th ($j=Fe, Cr, Ni$) atom, and d_{jv} is the diffusivity of the j th atom. The term $-D_V \nabla C_V$ is the flux of vacancies from the bulk to the defect sink caused by the gradient in the vacancy concentration. The term $C_V \alpha (d_{AV} \nabla C_A + d_{BV} \nabla C_B + d_{CV} \nabla C_C)$ is the flux of vacancies away from the defect sink due to gradients in the atom concentrations (vacancy back diffusion). In a ternary iron-chromium-nickel alloy:

$$C_{Cr} + C_{Fe} + C_{Ni} = 1, \quad (2a)$$

or

$$\nabla C_{Fe} = -\nabla C_{Cr} - \nabla C_{Ni}. \quad (2b)$$

Substituting (2b) into (1) and simplifying gives

$$J_V = N C_V \nabla C_V \left[\frac{-D_V}{C_V} + \alpha \left[\frac{(d_{CrV} - d_{FeV}) \nabla C_{Cr}}{\nabla C_V} + \frac{(d_{NiV} - d_{FeV}) \nabla C_{Ni}}{\nabla C_V} \right] \right] \quad (3)$$

Void growth depends on the flux of vacancies to the void (the magnitude of which is determined by $\frac{-D_V}{C_V}$), and the back diffusion of vacancies from the void (the magnitude of

which is determined by $\alpha \left[\frac{(d_{CrV} - d_{FeV}) \nabla C_{Cr}}{\nabla C_V} + \frac{(d_{NiV} - d_{FeV}) \nabla C_{Ni}}{\nabla C_V} \right]$). Those alloys

with a smaller flux of vacancies to the void and a greater back diffusion away from the void are less likely to swell.

The two flux terms can be analyzed further. The bulk vacancy concentration is related to the vacancy diffusion coefficient by [12]:

$$C_v \propto \left(\frac{1}{D_v} \right)^{1/2}. \quad (4)$$

The flux of vacancies to the boundary therefore depends on $D_v^{3/2}$, with the diffusion coefficient for vacancies given by:

$$D_v = d_{CrV}C_{Cr} + d_{FeV}C_{Cr} + d_{NiV}C_{Cr}. \quad (5)$$

The diffusivity of the j th atom via vacancies is given by:

$$d_{jv} = d_0^j \exp\left(\frac{-E_{vm}^j}{kT}\right), \quad (6)$$

where d_0 is the pre-exponential factor and E_{vm}^j is the migration energy via vacancies.

The back diffusion of vacancies away from the void depends on the magnitude of the difference in the diffusivities of chromium and iron ($d_{CrV} - d_{FeV}$) and on the magnitude of the difference in the diffusivities of nickel and iron ($d_{NiV} - d_{FeV}$). It also depends on

the ratio of the chromium gradient to the vacancy gradient $\frac{\nabla C_{Cr}}{\nabla C_V}$ and the ratio of the nickel gradient to the vacancy gradient $\frac{\nabla C_{Ni}}{\nabla C_V}$.

The magnitude of the term

$$\frac{(d_{CrV} - d_{FeV})\nabla C_{Cr}}{\nabla C_V} + \frac{(d_{NiV} - d_{FeV})\nabla C_{Ni}}{\nabla C_V}, \quad (7)$$

determines the resistance to void swelling due to back diffusion. The gradient ratios ($\frac{\nabla C_{Cr}}{\nabla C_V}$ and $\frac{\nabla C_{Ni}}{\nabla C_V}$) can be estimated from the steady-state solution to the equations describing RIS. The equations describing RIS have been solved by Wiedersich et al. [13] to obtain the relationship between an atom gradient and the vacancy gradient at *steady-state* for a binary alloy. The ratio of these two gradients at steady-state has been termed the determinant (M) by Watanabe et al. [14]. For a binary alloy, the determinant for atom A is:

$$M_A = \frac{\nabla C_A}{\nabla C_V} = \frac{C_A C_B d_{Ai} d_{Bi}}{\alpha (d_{Bi} C_B D_A + d_{Ai} C_A D_B)} \left(\frac{d_{Av}}{d_{Ai}} - \frac{d_{Bv}}{d_{Bi}} \right). \quad (8)$$

If the term $\left(\frac{d_{Av}}{d_{Ai}} - \frac{d_{Bv}}{d_{Bi}} \right)$ is positive, the gradient of atom A is in the same direction as the vacancy gradient and atom A would deplete. Watanabe et al. [14] derived an expression for the determinant in a ternary alloy. The determinant for atom j (where j is Fe, Cr, or Ni in a ternary alloy) is:

$$M_j = \frac{\nabla C_j}{\nabla C_v} = \frac{\frac{d_{jv}C_j}{D_j} \sum_{k \neq j} \frac{d_{ki}C_k}{D_k} - \frac{d_{ji}C_j}{D_j} \sum_{k \neq j} \frac{d_{kv}C_k}{D_k}}{\alpha \sum_k \frac{d_{ki}C_k}{D_k}}. \quad (9)$$

If the determinant for the j th atom is positive, then the j th atom depletes during irradiation.

Although the determinants ($\frac{\nabla C_{Cr}}{\nabla C_v}$ and $\frac{\nabla C_{Ni}}{\nabla C_v}$) are a steady-state solution, the resistance to swelling from back diffusion throughout the irradiation can be estimated using the determinants.

Since atom diffusivities and vacancy diffusion coefficients vary significantly from iron-base to nickel-base alloys, the flux terms are more easily compared by normalizing them to the nickel diffusivity. Carrying out this normalization, equation 3 becomes:

$$J_v = N d_{NiV} C_v \nabla C_v \left[\frac{-\left(D_v/d_{NiV}\right)}{C_v} + \alpha \left[\frac{(d_{CrV} - d_{FeV})}{d_{NiV}} \frac{\nabla C_{Cr}}{\nabla C_v} + \frac{(d_{NiV} - d_{FeV})}{d_{NiV}} \frac{\nabla C_{Ni}}{\nabla C_v} \right] \right]. \quad (10)$$

Since the vacancy concentration is proportional to $D_v^{3/2}$, the magnitude of the forward diffusion term can be defined as:

$$\left(D_v/d_{NiV}\right)^{3/2} \equiv \text{FDM}. \quad (11)$$

The magnitude of the back diffusion term can be defined as:

$$\propto \left[\frac{(d_{CrV} - d_{FeV})}{d_{NiV}} \frac{\nabla C_{Cr}}{\nabla C_V} + \frac{(d_{NiV} - d_{FeV})}{d_{NiV}} \frac{\nabla C_{Ni}}{\nabla C_V} \right] \equiv \text{BDM}. \quad (12)$$

The ratio $\frac{\text{FDM}}{\text{BDM}}$ is an indication of the propensity to swell. The larger $\frac{\text{FDM}}{\text{BDM}}$, the more likely an alloy of a given composition will swell. The propensity to swell could also be defined as the difference FDM-BDM. Either formulation is sufficient. In this work, the ratio $\frac{\text{FDM}}{\text{BDM}}$ will be used to define the propensity to swell.

To calculate the FDM and BDM for a given alloy, the diffusivities must be known *at the temperatures at which swelling and RIS occur*. Using the diffusivities and steady-state determinants obtained from RIS measurements, the FDM and BDM for different iron-chromium-nickel alloys can be calculated. The trends in the ratio $\frac{\text{FDM}}{\text{BDM}}$ as a function of alloy composition can be compared to the trends in void swelling as a function of composition to show that the reduction in swelling corresponds directly to the magnitude of the vacancy flux in these alloys.

Comparison of swelling data to swelling resistance from segregation

The largest set of RIS data for iron-chromium-nickel alloys has been generated using proton irradiations [10,11,15-22]. Seven different iron-base alloys (Fe-16Cr-24Ni, Fe-20Cr-24Ni, Fe-24Cr-24Ni, Fe-24Cr-19Ni, Fe-20Cr-9Ni, Ni-18Cr-9Fe, and Ni-18Cr) were irradiated at five different temperatures (200°C-600°C at 100°C intervals), to five

different doses (0.1, 0.3, 0.5, 1.0, and 3.0 dpa). Grain boundary compositions were measured using both Auger electron spectroscopy (AES) and scanning transmission electron microscopy with energy dispersive x-ray spectroscopy (STEM/EDS), resulting in over 1100 grain boundary composition measurements.

From the grain boundary composition measurements, the diffusivities of iron, chromium, and nickel were determined for each alloy. The diffusivities, which are input to RIS model calculations, were determined by changing the diffusivities (model inputs) until the model best fit the measured data [16,17]. These "best-fit" diffusivities followed the trends of self-diffusion coefficients for all of the alloys involved indicating that preferential coupling with the vacancy flux was the primary mechanism of RIS in these alloys. The pre-exponential factors and vacancy migration energies that best predicted the measured segregation are listed in Table 1. The fit of the data was better for the iron-base alloys and therefore, the uncertainty in the diffusivities of the nickel-base alloys is greater. The ratio $\frac{FDM}{BDM}$ was calculated using equations 11-12 at two temperatures (510°C and 675°C) where swelling measurements as a function of composition exist. The calculations assumed that the interstitial diffusivities were equal for chromium, iron, and nickel [20].

As described, void swelling should be inhibited in those alloys where the ratio $\frac{FDM}{BDM}$ is a maximum. Figure 1 plots the measured void swelling and $\frac{FDM}{BDM}$ at 675°C for Fe-xCr-(19-29)Ni alloys where the chromium concentration x varies from 8 to 31 at%. Figure 2 plots the measured void swelling and $\frac{FDM}{BDM}$ at 510°C for Fe-xCr-(19-24)Ni alloys where the chromium concentration x varies from 8 to 31 at%. For both irradiation temperatures, the

mean swelling and the $\frac{\text{FDM}}{\text{BDM}}$ (propensity to swell) increase as bulk chromium concentration increases.

The relationship between swelling and back diffusion can also be examined in constant chromium alloys. Figure 3 plots the measured void swelling and calculated BDM at 675°C for Fe-(16-24)Cr-xNi alloys where the nickel concentration x varies from 14 to 33 at%. Figure 4 plots the measured void swelling and calculated BDM at 510°C for Fe-(16-24)Cr-xNi alloys where the nickel concentration x varies from 14 to 34 at%. The swelling in iron-base alloys decreases as bulk nickel concentration increases. At both temperatures, the ratio $\frac{\text{FDM}}{\text{BDM}}$ follows the trends of the swelling as bulk nickel concentration changes. The only place in which the ratio $\frac{\text{FDM}}{\text{BDM}}$ does not follow the trends in swelling is for the Ni-18Cr alloy irradiated at 510°C. In all other cases, reduction in swelling correlates with swelling resistance from vacancy diffusion.

Alloys that take the longest to reach steady-state segregation should also swell the most. Garner has shown that the composition dependence of swelling occurs in the transient and not the steady-state swelling regime [23]. The end of the transient swelling regime should correlate roughly with the onset of steady-state grain boundary segregation. Measurements of RIS have shown that the time required for the grain boundary composition to reach steady-state differs with bulk nickel concentration [24]. Figure 5 shows the grain boundary chromium and nickel concentrations for Fe-20Cr-9Ni, Fe-20Cr-24Ni, and Ni-18Cr-9Fe irradiated with protons at 400°C to various doses. While Fe-20Cr-24Ni and Ni-18Cr-9Fe reach a relatively stable concentration by around 1 dpa, the grain boundary composition in Fe-20Cr-9Ni is still increasing between 1 and 3 dpa. The time to steady-

state segregation in the Fe-20Cr-9Ni alloy is longer than in Fe-20Cr-24Ni and Ni-18Cr-9Fe.

For these proton irradiated alloys, bulk swelling was not measured, but the swelling can be calculated from the measured void size and density [24]. Figure 6 plots the calculated swelling as a function of dose. The Fe-20Cr-9Ni alloy, which is the slowest to reach steady-state (figure 5), has the largest swelling. The resistance to swelling created by the development of a segregation profile takes longer to develop in the Fe-20Cr-9Ni alloy.

Figure 6 also lists the propensity to swell ($\frac{\text{FDM}}{\text{BDM}}$) calculated at 400°C. The propensity to swell is largest in the Fe-20Cr-9Ni alloy which has the greatest swelling. The swelling in Fe-20Cr-24Ni is larger than the swelling in Ni-18Cr-9Fe, even though the propensity to swell is smaller. This discrepancy is most likely due to greater uncertainty in the diffusivity of Ni-18Cr-9Fe which affects the FDM/BDM calculation.

Conclusion

The degree of both swelling and RIS in austenitic iron-chromium-nickel alloys vary with bulk alloy composition. Resistance to void swelling in iron- and nickel-base alloys has been shown to correlate with major element segregation. The resistance to void swelling can be estimated by calculating the magnitude of the vacancy diffusion to the void using the diffusivities determined from RIS measurements. Enrichment of nickel and depletion of chromium reduces the vacancy flux to voids and thus limits the rate of void growth. Additionally, the swelling is greater in alloys in which the RIS profiles are slow to develop. These alloys take a longer time to reach the steady-state swelling regime, thereby reducing the amount of void swelling. These conclusions hold true for both constant chromium,

varying nickel iron-base alloys and constant nickel, varying chromium iron-base alloys. Alloys with greater nickel enrichment and chromium depletion are less prone to swelling.

Acknowledgments

The authors gratefully acknowledge R. D. Carter, J. M. Cookson, D. Damcott , and J. Gan for their insight and assistance. Thanks go out to S. M. Bruemmer at Pacific Northwest National Laboratory for his support. This research was supported by the U.S. Department of Energy under grant DE-FG02-93ER-12310 and by the Associated Western Universities-Northwest under U.S. Department of Energy grant DE-FG02-89ER-7552. Support was also provided by the Materials Sciences Branch, Office of Basic Energy Sciences, under contract DE-AC06-76LO1830, through Pacific Northwest National Laboratory. ANL support ????

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Table 1. Diffusivities determined from segregation measurements

Alloy	$\frac{d_{vm}^{Cr}}{d_{vm}^{Ni}}$	$\frac{d_{vm}^{Fe}}{d_{vm}^{Ni}}$	E_{vm}^{Cr}	E_{vm}^{Fe}	E_{vm}^{Ni}
Fe-16Cr-24Ni	2.54	1.68	1.32	1.305	1.30
Fe-20Cr-24Ni	2.48	1.60	1.32	1.305	1.30
Fe-24Cr-24Ni	2.39	1.47	1.32	1.305	1.30
Fe-24Cr-19Cr	2.32	1.34	1.32	1.305	1.30
Fe-20Cr-9Ni	2.31	1.28	1.32	1.305	1.30
Ni-18Cr-9Fe	2.54	1.67	0.83	0.815	0.80
Ni-18Cr	2.54	-----	1.07	-----	1.04

Bulk Cr Concentration (at%)

Figure 1. Comparison of swelling with forward and back diffusion at 675°C. Swelling from Ni-ion irradiations. FDM/BDM calculated using diffusivities from segregation measurements from proton irradiations.

Figure 2. Comparison of swelling with forward and back diffusion at 510°C. Swelling from neutron irradiations. FDM/BDM calculated using diffusivities from segregation measurements from proton irradiations.

Figure 3. Comparison of swelling with forward and back diffusion at 675°C. Swelling from Ni-ion irradiations. FDM/BDM calculated using diffusivities from segregation measurements from proton irradiations.

Figure 4. Comparison of swelling with forward and back diffusion at 510°C. Swelling from neutron irradiations. FDM/BDM calculated using diffusivities from segregation measurements from proton irradiations.

Figure 5. Dose dependence of segregation as a function of alloy composition for Fe-(18-20)Cr-xNi alloys (x=9, 24, 72)

Figure 6. Dose dependence of calculated swelling as a function of alloy composition for Fe-(18-20)Cr-xNi alloys (x=9, 24, 72)