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INFLUENCE OF HELIUM GENERATION RATE
AND TEMPERATURE HISTORY ON MECHANICAL
PROPERTIES OF MODEL Fe-Cr-Ni ALLOYS
IRRADIATED IN FFTF AT RELATIVELY LOW
DISPLACEMENT RATES

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INFLUENCE OF HELIUM GENERATION RATE AND TEMPERATURE HISTORY ON MECHANICAL PROPERTIES OF MODEL Fe-Cr-Ni ALLOYS IRRADIATED IN FFTF AT RELATIVELY LOW DISPLACEMENT RATES - M.L. Hamilton, F.A. Garner and D.J. Edwards (Pacific Northwest Laboratory)^a

OBJECTIVE

The objective of this effort is to determine the influence of high helium generation rates on evolution of mechanical properties of simple model fcc alloys.

SUMMARY

In agreement with earlier studies conducted at higher displacement rates, the evolution of mechanical properties of model Fe-Cr-Ni alloys irradiated at lower displacement rates in the ⁵⁹Ni isotopic doping experiment does not appear to be strongly affected by large differences in helium generation rate. This insensitivity to helium/dpa ratio is exhibited during both isothermal and non-isothermal irradiation. The overall behavior of the model alloys used in this study is dominated by the tendency to converge to a saturation strength level that is independent of thermomechanical starting state and helium/dpa ratio, but which is dependent on irradiation temperature and alloy composition.

PROGRESS AND STATUS

Introduction

Until recently, it has been almost impossible to conduct experiments in which spectrum-related parameters such as helium/dpa ratio were varied without also accepting variations in other important parameters such as displacement rate or temperature history. A technique currently being used, however, allows the study of the influence of helium alone on density change, microstructural evolution and mechanical properties. This technique utilizes isotopic tailoring to vary the helium production rate without introducing changes in specimen composition, neutron spectrum or displacement rate (1,2). This generates substantial variations in He/dpa ratio without varying any other important parameter by using alloys whose only difference is the presence or absence of ⁵⁹Ni, an isotope that does not occur naturally (3), and irradiating doped and undoped specimens side-by-side in the appropriate reactor spectrum (1).

The extensive ⁵⁹Ni isotopic tailoring experiment conducted in the FFTF reactor since 1985 to study the effects of helium generation rate on mechanical properties is now approaching completion. Whereas previous papers (3,4) have concentrated on the irradiation sequences conducted at higher displacement rates, this report focuses on the tests conducted at lower displacement rates, reaching cumulative displacement levels ranging from 0.2 to 13.9 dpa. These two groups explored both LMR - relevant helium/dpa ratios and more fusion-relevant ratios of 4.4 and 20, as well as a higher-than-fusion rate of 62 appm/dpa. The alloys studied were Fe-15Cr-25Ni (both with and without 0.04 P) and Fe-15Cr-45Ni, all in weight percent. Two of the three irradiation series reported here involved

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comparison of isothermal and non-isothermal irradiations, since the details of temperature history are now known to have dominated many earlier experiments directed toward the influence of helium (5).

Experimental Details

The alloys employed in this study were irradiated in both the cold worked and annealed conditions. The acquisition of the ^{59}Ni , and the production of the ^{59}Ni -doped miniature tensile specimens and microscopy disks, as well as their irradiation conditions, are described by Simons and coworkers (1). The miniature tensile specimens measured 5.1, 1.0, and 0.25 mm in gauge length, width and thickness, respectively. They were tested at room temperature at a strain rate of $4.7 \times 10^{-4} \text{ s}^{-1}$ in a horizontal test frame described in ref. 6. Yield strengths were determined at 0.2% offset. The majority of test conditions involved single specimens, but two specimens were tested for most alloy conditions in the 600°C series.

Figure 1 shows the discharges from the final three irradiation sequences of the ^{59}Ni experiment, each defined by its target irradiation temperature and its location in the Materials Open Test Assembly (MOTA) in the FFTF reactor. The experiment was initiated in the MOTA 1D irradiation segment, but one-third of the way through the irradiation segment there was a pronounced temperature excursion of relatively short duration (~1 hr), referred to as an over-temperature event. This event compromised the integrity of many of the experiments in the MOTA and a programmatic decision was therefore made to run the MOTA in the helium-purged mode for the remainder of FFTF cycles 7 and 8 while a series of reactivity feedback tests were conducted. The majority of the MOTA canisters therefore operated at variable but lower than target temperatures until the end of the MOTA 1D. Isothermal irradiation at the original target temperatures then proceeded in MOTA segments 1E, 1F and 1G.

Specimens in the out-of-core irradiation sequence conducted at 465°C were thought to have been affected very little by the temperature history event and their irradiation was continued, with discharges

Fig 1. Schematic representation of the low displacement rate irradiations sequences for the ^{59}Ni isotopic tailoring experiment. "600°C(R)" and "490°C(R)" refer to the replacement sequences substituted for the original compromised sequences at 600 and 490°C. Damage levels shown represent cumulative totals.

occurring at 0.14, 0.26 and 0.40 dpa. The specimens in the original in-core sequence at 490°C were not returned to the reactor after reaching 2.0 dpa, but were replaced with a repeat sequence in MOTA 1E through 1G with discharges at 2.1, 4.2 and 6.7 dpa. The specimens in the in-core sequence at 600°C were very strongly impacted by the temperature history, both in the over-temperature and under-temperature portions, and irradiation was discontinued after 8.7 dpa. A decision was made later, however, to repeat this sequence for two irradiation segments in MOTA 2B and 2C. Unfortunately, both groups of specimens at 600°C had to be retrieved when FFTF was shut down before MOTA 2C was initiated. Only 13.9 dpa was attained in this repeat sequence.

The successful conduct and interpretation of these experiments is contingent on measurement of the helium levels attained. The details of the helium measurements are presented in refs. 7-9. The displacement levels were calculated from dosimetry measurements using the standard NRT model for dpa.

Results and Discussion

The yield strength and total elongation data for the full series of irradiations at 490, 465°C and 600°C are shown in Figures 2-4. At 490°C the yield strengths of the cold worked and annealed specimens appear to be converging in each of the three alloys studied. The elongation data exhibit a similar convergence. This behavior is in agreement with a similar tendency toward convergence observed in many other irradiation experiments (10). While the yield strengths of both the annealed and cold worked alloys exhibit essentially no dependence on helium/dpa level, there is possibly a small effect of helium on the total elongation, but only for the annealed Fe-15Cr-25Ni-0.04P alloy. However, the maximum difference between doped and undoped specimens is comparable to the range of variation observed in the unirradiated specimens. The cold worked alloys clearly do not exhibit an influence of helium on elongation. The impact of the non-isothermal temperature history appears to be significant only for the Fe-15Cr-25 Ni alloy in both starting conditions.

Convergence in tensile properties also appears to be occurring in the high-helium, low-dose sequence at 465°C, with very little or no consistent influence of helium on the yield strength. Approximately 25 appm have accumulated in the doped specimens at an exposure of 0.4 dpa in this low dose rate experiment. There may be some small and possibly increasing influence of helium, however, on the elongation of the cold worked Fe-15Cr-25Ni-0.04P alloy with increasing dpa level.

At 600°C convergence in the tensile properties also occurs in the isothermal sequence, but very little change is occurring in the annealed condition. The impact of the over-temperature/under-temperature history is very pronounced, but with one possible exception (total elongation, annealed 45% Ni alloy), there appears to be no significant effect of the differences in helium generation rate. Convergence in the non-isothermal sequence is approached by both the cold worked and annealed specimens, reflecting the lower average temperature over the last two-thirds of the sequence. Once again, however, the helium generation rate does not appear to be a significant factor.

The microstructural origins of the non-isothermal response at 600°C and 8.7 dpa have been presented previously (11), but microscopy has not yet been performed for the isothermal repeat sequence. Assuming that the response differs only in the details of the microstructure, it is expected that changes in tensile properties can be successfully related to the densities and sizes of cavities, dislocation loops, and dislocation segments, as well as phosphide precipitates in the case of the Fe-15Cr-25Ni-0.04P alloy. Mitchell has shown, however, that the superposition rules employed to estimate the combined influence of various microstructural components must be modified when needle-like phosphides exhibit large aspect ratios, and also when the phosphides are directly associated with cavities (12).

Fig. 2. Influence of thermomechanical starting state, isotopic doping and temperature history on yield strength and elongation following irradiation just above the top of the FFTF core at 490°C. The dotted lines correspond to the isothermal repeat sequence. The boxes on the y-axis show the range of values obtained for the unirradiated specimens.

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Fig. 3. Influence of thermomechanical starting state and isotopic doping on yield strength and elongation following irradiation high above the FFTF core at 465°C.

Whereas success has been attained in describing the microstructural origins of strength changes at high displacement rates in terms of relatively large microstructural components (11-14), the annealed Fe-15Cr-25Ni alloy in the low exposure sequence at 465°C was found not to develop cavities or dislocations, but to be dominated by the formation of many small point defect clusters (15). In this case, high levels of helium were observed to decrease the density of such clusters, with a concurrent

Fig. 4. Influence of themomechanical starting state, isotopic doping and temperature history on yield strength and total elongation following irradiation at the top of the core at 600°C. The dotted lines correspond to the isothermal repeat sequences.

increase in their mean size. This helium-induced difference led, in turn, to a small but measurable change in alloy hardness (15) that may account for the difference observed in the total elongation of the annealed Fe-15Cr-25Ni alloy.

CONCLUSIONS

In agreement with other studies (3,4,10) these results demonstrate the strong tendency of austenitic alloys to converge during irradiation to a yield strength that is independent of the thermomechanical starting state.

When the results of this and the previous study at higher displacement rates (4) are compared, it appears that the influence of helium/dpa ratio on the evolution of mechanical properties in three model Fe-Cr-Ni alloys is small or insignificant when compared to the scatter exhibited by the unirradiated material, or when compared to the action of compositional variations, temperature or cold work level. The impact of temperature history on tensile properties is often quite strong, but is somewhat dependent on alloy composition. The role of helium/dpa ratio in these alloys is small even during non-isothermal irradiation, however.

FUTURE WORK

This work will continue, using the TEM specimens irradiated at 600°C to conduct shear punch tests for small specimens development.

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