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MICROSTRUCTURE OF 9 Cr-1 MoVNB STEEL IRRADIATED TO 40 dpa AT ELEVATED TEMPERATURES IN HFIR*

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As part of an effort by the Office of Fusion Energy to evaluate the irradiation behavior of ferritic steels, a 9 Cr-1 MoVNB alloy was irradiated in HFIR to a dose of ~36 dpa at temperatures of 300, 400, 500, and 600°C. In addition to the displacement damage produced during irradiation, a transmutation reaction of nickel during HFIR irradiation resulted in the simultaneous production of ~30 at. ppm He. Electron microscopy disks in the normalized and tempered condition were irradiated, and the microstructures were evaluated as a function of irradiation temperature. A few small cavities were observed after irradiation at 300, 500, and 600°C. However, a pronounced cavity microstructure was found after irradiation at 400°C. At this temperature, the cavities had a volume-averaged diameter of 15 nm and a concentration of $1.1 \times 10^{21} \text{ m}^{-3}$, resulting in a void-swelling contribution of 0.19%. The cavities at 400°C were homogeneously distributed throughout the tempered martensite matrix, and showed no preference for lath boundaries or precipitate interfaces. The results are compared to those recently reported on a similarly irradiated 12 Cr-1 MoVW ferritic steel.

1. INTRODUCTION

Over the last three years, a surge of interest in ferritic steels has been generated within the Alloy Development for Irradiation Performance (ADIP) program of the United States fusion energy research effort. This class of steels offers several potential advantages as a fusion first-wall structural material. Among the favorable attributes, the good thermal properties and resistance to swelling during irradiation are foremost. Early investigations have shown that swelling in these materials is extremely low.¹⁻⁶

When considering applications in fusion energy devices, one must consider the effect of helium generation during irradiation. Under fusion irradiation conditions, a helium generation rate of ~10 at. ppm/dpa is expected for ferritics. Therefore, this additional factor has been included in the effort to evaluate the swelling response of ferritic steels.

Since fusion reactor conditions for material testing are not presently available, simulation is necessary. One technique for simulating

helium production during irradiation is to irradiate nickel-bearing alloys in the High Flux Isotope Reactor (HFIR) facility at Oak Ridge. The thermal portion of the mixed-neutron spectrum in HFIR results in helium production means of a two-step transmutation reaction of nickel.⁷ Thus, simultaneous helium production and displacement damage are possible in nickel-bearing alloys.

The 9 Cr-1 MoVNB is one of the primary ferritic alloys being evaluated in the ADIP program. This steel contains 0.11% Ni and is expected to produce approximately 1 at. ppm He/dpa in HFIR. The objective of this paper to evaluate the microstructure of a 9 Cr-1 Mo alloy after irradiation in HFIR. The postirradiation cavity microstructure will be compared with a similar normalized and tempered ferritic steel, 12 Cr-1 MoVW, which was irradiated under the same conditions and was reported on elsewhere.⁸

2. EXPERIMENTAL PROCEDURE

The steel used in this investigation was experimental heat of 9 Cr-1 MoVNB (heat XA359

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A chemical analysis of the major constituents is given in Table 1. The heat was hot rolled to 3 mm, and then softened by heating to 900°C, furnace cooling to 700°C, annealing at 700°C for 16 h, and air cooling. After pickling, the plate was cold rolled to 0.25 mm. Disks of 3 mm diameter were punched from the final sheet and given a normalizing-and-tempering treatment. Normalizing consisted of austenitizing at 1040°C for 0.5 h followed by cooling in flowing helium gas; tempering was at 760°C for 1 h followed by cooling in helium gas.

Table 1. Composition of 9 Cr-1 MoVNb (Heat XA3590) in Weight Percent

Cr	8.62	V	0.21	Si	0.08
Mo	0.98	Ni	0.11	Nb	0.06
Mn	0.36	C	0.09	N	0.05
				Fe	Bal

The ferritic steel disks were irradiated as part of the HFIR-CTR-30 experiment.⁹ Irradiation temperatures of 300, 400, 500, and 600°C were achieved with gamma heating by varying the size of the gas gap around the sample holders. Evaluation of temperature and flux monitors is currently under way to confirm the experimental conditions. Based on the evaluation of flux monitors from similar experiments irradiated to lower fluence,^{10,11} the fluence achieved was 4.9×10^{26} neutrons/m² ($E > 0.1$ MeV), corresponding to a damage level of 36 dpa (a nominal 40 dpa) and a helium concentration of 30 at. ppm.

The disks were electropolished remotely in a hot cell with a twin-jet polisher. A 7:1 methanol:sulfuric acid electrolyte was used at -12°C with a current of 140 mA and a voltage of approximately 20 V. The specimens were examined within 24 h of electropolishing with a JEM 100C transmission electron microscope equipped with a low magnetic field objective lens polepiece.

3. RESULTS

3.1 Unirradiated Microstructure

The austenitizing treatment results in a fully austenitic structure, and due to the high hardenability of the alloy, gas cooling produced a martensitic lath microstructure, without any significant amount of ferrite [Fig. 1(a)]. Following tempering, the structure consisted of a tempered lath martensite network with carbide precipitates along prior-austenite grain boundaries and to a lesser degree along lath boundaries [Fig. 1(b)]. The tempered martensite structure contained 1.4 wt % precipitate consisting of mostly chromium-rich $M_{23}C_6$ but with smaller amounts of vanadium and/or niobium-rich carbides or nitrides.¹²

FIGURE 1
Microstructure of unirradiated 9 Cr-1 MoVNb in the (a) normalized and (b) normalized and tempered conditions.

3.2 Irradiated microstructure

Foils from each of the four irradiation temperatures were examined. In specimens irradiated at 300°C, a few small cavities were observed, homogeneously distributed throughout the matrix. These were approximately 4 to 6 nm in diameter. Although the precipitates were not analyzed in detail, their morphology and distribution were unchanged by the irradiation, implying no new precipitation occurred.

The microstructure after irradiation at 400°C was remarkably different. A well-developed cavity microstructure was observed, as shown in Fig. 2. Cavities ranged in size from 4 nm to over 30 nm in diameter. A composite size distribution from several micrographs is presented in Fig. 3. Since the cavity microstructure was more developed after irradiation at 400°C, it was analyzed in detail. The volume-averaged cavity diameter was 15 nm and the cavity concentration was calculated as $1.1 \times 10^{21} \text{ m}^{-3}$. The cavitation swelling contribution was 0.19%. The cavities were homogeneously distributed with no apparent preferential formation at lath boundaries, precipitate interfaces, or prior-austenite grain boundaries. As in the

FIGURE 3
Cavity size distribution in 9 Cr-1 MoVNB aft
irradiation in HFIR at 400°C to 36 dpa.

case for the 300°C irradiation, no apparent change in the nature of the precipitation was noticed.

A different cavity microstructure was observed after irradiation at 500 and 600°C. both of these temperatures, very few cavities on the order of 4 to 6 nm in diameter, were found. They were preferentially located at microstructural defects such as dislocations and lath boundaries. The foil irradiated at 500

FIGURE 2
Microstructure of 9 Cr-1 MoVNB following irradiation in HFIR at 400°C to 36 dpa.

showed occasional areas with large elongated precipitates, shown in Fig. 4. Analysis of these precipitates is in progress. However, the precipitate microstructure was unchanged after irradiation at 600°C.

FIGURE 4
Elongated precipitates found in 9 Cr-1 MoVNB after irradiation in HFIR at 500°C to 36 dpa.

4. DISCUSSION

The observation of cavities in irradiated 9 Cr-1 MoVNB is not new. Gelles and Thomas found a small number of voids, up to 20 nm in diameter, in 9 Cr-1 MoVNB after irradiation at 400°C in EBR-II to a dose of ~11 dpa (ref. 13). No voids were observed by Gelles and Thomas after irradiation at 500 or 550°C. The fact that considerably more swelling was observed in the current study after irradiation in HFIR may be attributed to two factors. The HFIR samples were irradiated to a higher dose than the EBR-II specimens of Gelles and Thomas (36 vs 11 dpa) and thus cavity development is expected to be greater in the former material. In addition, it was recently observed that the increased helium present after HFIR irradiation of 12 Cr-1 MoVW increased the cavity formation rate.⁸ Since the preirradiation microstructures of 9 Cr-1 MoVNB

and 12 Cr-1 MoVW are similar tempered lath martensite structures, it is expected that the enhanced cavity development due to helium generation in HFIR applies to 9 Cr-1 MoVNB. However, the relative degree to which these two factors affect the microstructure is impossible to assess at present.

The temperature dependence of the cavity microstructures is similar for both 9 Cr-1 MoV and 12 Cr-1 MoVW. A maximum in swelling was observed for both alloys after irradiation at 400°C. Furthermore, after irradiation at 300 and 400°C, the cavities were homogeneously distributed throughout the matrix, without any preferential formation at lath boundaries or precipitate interfaces. After irradiation at 500 and 600°C, the cavities in 12 Cr-1 MoVW were small and were found primarily at microstructural defects such as dislocations and lath boundaries.⁸ Although far fewer cavities were observed in 9 Cr-1 MoVNB irradiated at 500 and 600°C they were distributed in the same manner as in 12 Cr-1 MoVW. It was concluded that in the case of 12 Cr-1 MoVW, helium production during irradiation assisted in the development of voids at 400°C, and that the cavities found at 500 and 600°C were primarily helium bubbles. The same can be inferred of the nature of the cavities in 9 Cr-1 MoVNB. The fact that far fewer bubbles are found after the 500 and 600°C irradiations agrees with the lesser amount of helium produced in 9 Cr-1 MoVNB during irradiation. In support of this conclusion, following irradiation in EBR-II, where very little helium was produced, Gelles and Thomas found no cavities in 9 Cr-1 MoVNB irradiated at temperatures above 450°C.

In comparison to 12 Cr-1 MoVW, the amount of swelling at 400°C is noticeably greater in 9 Cr-1 MoVNB (0.19% vs 0.07%). This occurs in spite of the fact that the helium concentration in the HFIR-irradiated 12 Cr-1 MoVW is over three times

greater than in the 9 Cr-1 MoVNB alloy. The fact that 9 Cr-1 MoVNB swells more readily than 12 Cr-1 MoVW agrees with earlier observations by Gelles and Thomas.¹³ It was suggested by Gelles and Thomas that G phase formation in 12 Cr-1 MoVW inhibits void swelling. However, G phase was not found in the HFIR-irradiated 12 Cr-1 MoVW even though it also showed less swelling than 9 Cr-1 MoVNB. It seems another explanation is needed. Although the preirradiated microstructures are quite similar, differences do exist. More than twice as much carbide precipitates in 12 Cr-1 MoVW during tempering and the distribution of $M_{23}C_6$ carbide is more uniform through the matrix. In 9 Cr-1 MoVNB, the large $M_{23}C_6$ carbides are more concentrated on prior-austenite grain boundaries. In addition, the tempering treatments prior to irradiation differed, with an over-tempering treatment being used for 12 Cr-1 MoVW. Finally, minor compositional differences may play a role, such as the higher nickel level or lower niobium concentration in 12 Cr-1 MoVW. The question of which of these factors is important needs to be addressed in future work.

After irradiation at 500°C, some evidence of phase instability was found. Although the elongated precipitates in Fig. 4 have not been identified, their presence indicates additional precipitation occurred during irradiation. Gelles and Thomas¹³ found similar rod-shaped precipitates after irradiation of 9 Cr-1 MoVNB in EBR-II at 400°C and identified them as Cr_2C . Evidence for precipitation during irradiation was not found in HFIR-irradiated 12 Cr-1 MoVW.⁸ Thus, still another dissimilarity between these two ferritic steels exists.

The present results indicate that it is likely that helium plays a role in cavity development during irradiation of 9 Cr-1 MoVNB. However, it is impossible to assess how much of the swelling found at 400°C is due to helium

assisted void growth. In comparison to an optimized austenitic stainless steel (PCA) irradiated in HFIR to the same dose but containing significantly greater amounts of helium, the temperatures of maximum swelling differ.¹⁴ Austenitics show a maximum at 600°C while the ferritics exhibit a maximum at 400°C. The degrees of maximum swelling are comparable—to 0.5% in the PCA austenitic stainless steel and 0.19% in the 9 Cr-1 MoVNB. Finally, with the observed preferential distribution of bubbles at dislocations and boundaries after high temperature irradiations, one needs to be aware of the possibility of embrittlement in 9 Cr-1 MoVNB at higher helium concentration levels.

5. CONCLUSIONS

The cavity microstructures of 9 Cr-1 MoVNB were examined following irradiation in HFIR to a dose of approximately 36 dpa. The following conclusions may be drawn from this study:

1. A maximum in swelling of 0.19% is found after irradiation at 400°C. Although some cavities were observed after irradiation at 300, 500, and 600°C, the degree of swelling was negligible.

2. The cavities found after irradiation at 300 and 400°C are uniformly distributed throughout the matrix, and are assumed to be of void character. However, the cavities present after irradiation at 500 and 600°C are distributed preferentially at dislocations and lath boundaries, and are concluded to be helium bubbles.

3. At the maximum swelling temperature of 400°C, the degree of swelling in 9 Cr-1 MoVNB is greater than comparably irradiated 12 Cr-1 MoVW.

4. Signs of precipitation during irradiation at 500°C were found, but the precipitates were not identified.

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