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Influence of cold work to increase swelling of pure iron irradiated in the BR-10 reactor to ~6 and ~25 dpa at ~400°C

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Abstract

Irradiation of pure iron in several starting conditions at 400°C has been conducted in the BR-10 fast reactor. Contrary to expectations, cold working appears to significantly accelerate the onset of void swelling. When compared to a similar experiment conducted in this reactor at the same time, it appears that iron experiences a rather long transient duration before the onset of steady-state swelling. The transient appears to be shortened by both cold-working and lower atomic displacement rates. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

A recent reanalysis of previously published swelling data from the EBR-II and FFTF fast reactors by Garner et al. [1] showed that the steady-state swelling rate of Fe–Cr binary ferritic alloys is much larger than previously anticipated, being at least 0.2%/dpa and perhaps as large as 0.5%/dpa. The swelling transient regime of these Fe–Cr alloys was also shown to be much more sensitive to details of temperature, atomic displacement rate and chromium content than are Fe–Cr–Ni ternary austenitic alloys irradiated in the same experiments. Sencer and Garner [2] recently confirmed these conclusions in a separate study on Fe–Cr binary alloys. In general, it was concluded that ferritic alloys experience more difficulty in nucleating voids than austenitic alloys.

It was further demonstrated that some commercial ferritic and ferritic/martensitic alloys also appear to be developing larger than expected swelling rates after very long transient periods [1]. Such observations call into question the general perception that pure iron and iron-base ferritic alloys will always swell at very low rates under all irradiation conditions.

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One previous study by the Russian authors of this paper provides some support for this perception of ferritic swelling behavior [3]. The results of an experiment

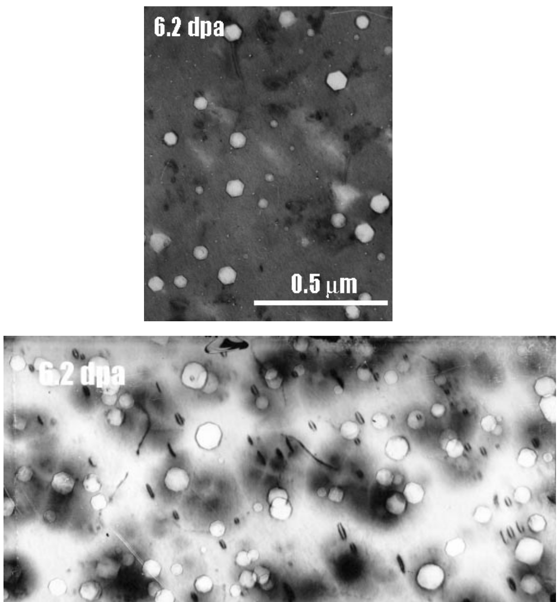

Fig. 1. Voids observed in 10% cold-worked pure iron irradiated in BR-10, reported in a previous study [3].

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was reported involving pure iron and Fe–XCr alloys ($X = 2$, 6, 12, and 18 at.%) irradiated at ~400°C in the BR-10 fast reactor in Obninsk, Russia. These metals were in the ~10% cold-worked condition and reached doses of 5.5–7.1 dpa at $\sim 1 \times 10^{-7}$ dpa/s. The Fe–Cr alloys exhibited void, dislocation and precipitate behaviors consistent with that observed in other studies involving alloys in the annealed condition. Surprisingly, however, the cold-worked pure iron exhibited the largest swelling, 3.0% at only 6.2 dpa, as shown in Fig. 1.

This result implies not only that cold working accelerates the onset of swelling under these difficult nucleation conditions, but also suggests an average swelling rate of almost 0.5%/dpa, an observation that begs for additional confirmation. Unfortunately, there were no annealed specimens irradiated together with the cold-worked specimens in the same subcapsule.

In the same irradiation vehicle, however, there was another subcapsule that provides an opportunity to test the concept that cold work can accelerate the onset of swelling in pure iron. This paper addresses the effect of the starting metallurgical state on swelling of pure iron.

2. Experimental details

Strips 5 mm in thickness of pure iron used in the earlier study [3] were rolled into foils of 0.12–0.15 mm thick, all deformations accomplished in one pass. The average level of cold work in the resulting foils was therefore ~250%. From some cold-worked foils strips 6×15 mm^2 were prepared. Other foils were annealed at 1100°C for 5 min and then quenched in water. Afterwards, 6×15 mm^2 strips were prepared. The composition of the starting iron was <0.015 C, <0.005 P, <0.05 S, 0.012 Si, 0.001 Al, 0.0015 Cr, 0.0015 Cu, 0.02 Ni, <0.0015 Mg, 0.001 Mo, 0.18 O, 0.003 Ni, wt%.

Strips of the same dimensions were also cut from iron foils of another melt with slightly different chemical compositions, 0.07 C, 0.005 P, 0.004 S, 0.02 Si, 0.02 Al, 0.02 Cr, 0.01 Cu, 0.03 N. These foils were annealed at 750°C for 1 h and cooled in air.

The three groups of foils were lightly tied together with a wire and then placed in the same irradiation capsule used in the experiment reported earlier. The specimens were in contact with flowing sodium during irradiation. No attack of the sodium was observed on the specimen surfaces after irradiation.

Whereas the 6.2 dpa experiment was irradiated at a level of 280–340 mm above the core midplane, the current experiment was conducted at 140–155 mm above

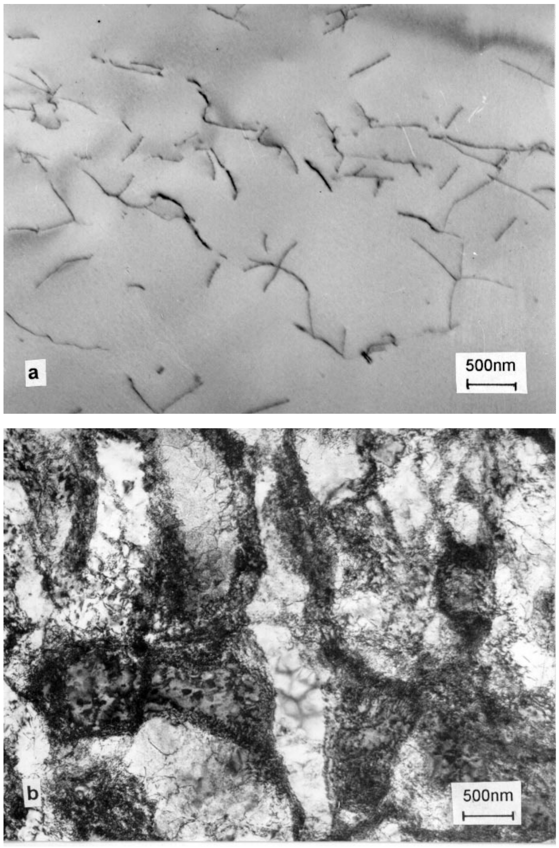

Fig. 2. The dislocation structure in unirradiated pure Fe at two different pre-treatment conditions: (a) 750°C/1 h, cooled in air; (b) 250% cold worked.

Table 1
Microstructural data[a]

Treatment	750°C/1 h AC	1100°C/5 min WQ	250% CW
$\langle d_v \rangle$, nm	45	55	120
N_v, 10^{14} cm^{-3}	2	1.2	0.32
$\Delta V/V$, %	1.2	1.7	4.5
$\langle d_L \rangle$, nm	68	117	85
N_L, 10^{14}, cm^{-3}	1.7	0.75	0.9
ρ_d, cm^{-2} (unirrad.)	$<10^9$	$<10^9$	$1\text{–}2 \times 10^{11}$
ρ_d, cm^{-2} (irrad.)	4×10^9	3×10^9	4×10^9

[a] $\langle d \rangle$, N_v, $\langle d_L \rangle$, N_L denote the mean diameter and number density of voids and loops, respectively. $\Delta V/V$ and ρ_d are the swelling and dislocation density.

the core midplane. A dose of 25.8 dpa was attained at a dose rate of 4×10^{-7} dpa/s, approximately four times the rate experienced by the experiment reported earlier. The irradiation capsule was designed such that the temperature at all levels of interest was 400°C, with an uncertainty of ±15°C.

After removal from the irradiation capsule 5 disks of 3 mm in diameter were punched from each plate. Microscopy specimens were prepared from these disks, using polishing condition reported earlier [3]. Similar specimens were prepared from non-irradiated archive foils. Examination was conducted in a JEM-100CX electron microscope. The thinned foils were examined in the regions ranging from 100 to 150 nm thick as determined by measurements of the width of grain or subgrain boundary projections. The accuracy of the thickness determinations is thought to be ±20%.

3. Results

The pre-irradiation dislocation microstructures are shown in Fig. 2. Note that while the annealed specimens have relatively low densities of dislocations, probably introduced mostly during the preparation of the thin foils, the ~250% cold-worked specimen has a very dense cell structure.

The irradiation to 25.8 dpa at 400°C resulted in the formation of voids, dislocation loops and dislocation segments in all the three types of specimens. The microstructural details are tabulated in Table 1 and shown in Fig. 3.

Dislocation loops observed in pure iron at this dpa level have the same Burgers vector and habit plane as the loops at 6.2 dpa, namely, $b = a\langle 1\,0\,0\rangle$ and $\{1\,0\,0\}$ planes, respectively. All three starting conditions have reached the same network dislocation density at $3–4 \times 10^9$ cm^{-2}.

4. Discussion

The prevailing perception is that cold work almost always results in a suppression of void swelling. Garner et al. [1] have shown, however, that when void nucleation is impeded by difficulty in forming or maintaining a dislocation network, cold working can accelerate swelling. This behavior has been observed in pure nickel, pure molybdenum and Fe–Cr–Ni ternary alloys at high nickel levels and relatively high temperatures [4–6].

The most striking result of this experiment is the significant enhancement of swelling in the heavily cold-worked iron, 4.5% vs. 1.7% in the annealed condition of the same starting composition. The other annealed heat of iron swelled a little less, 1.2% vs. 1.7%, as a consequence of its slightly different composition and annealing condition.

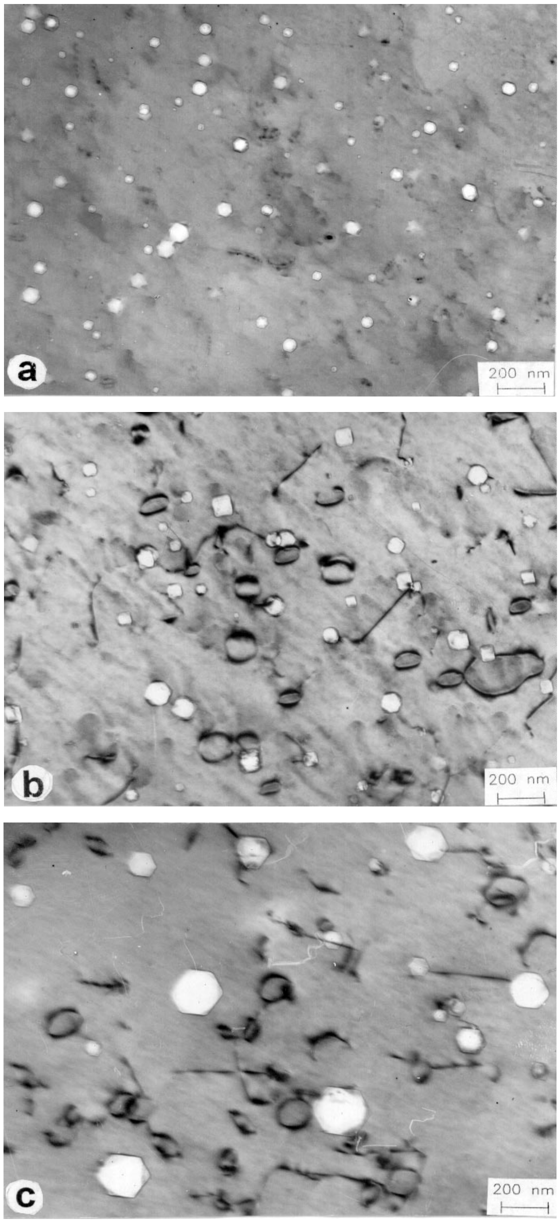

Fig. 3. Void and dislocation microstructures observed in pure iron irradiated at 400°C to 25.8 dpa in different pre-treatment conditions: (a) 750°C/1 h cooled in air; (b) 1100°C/5 min quenched in water; (c) 250% cold worked.

The enhancement of swelling by cold work is expressed primarily in the larger void size. This observation suggests that the voids were nucleated earlier in the cold-worked specimen and have grown for a longer time. The average swelling rate over 25.8 dpa is approximately 0.17%/dpa in the cold-worked iron. If there was any transient lower swelling rate, however, the swelling rate toward the end of the irradiation was obviously larger. Such a conclusion is consistent with that of both the earlier study on pure iron (0.5%/dpa at 6.2 dpa) [3] and that of Garner and co-workers for Fe–Cr binary alloys (0.2%/dpa or greater) [1].

Having reached 3.0% at 6.2 dpa, it appears at first glance that a swelling level significantly larger than 4.5% should have been reached at 25.8 dpa. If there were no differences in displacement rate one might conclude that swelling in iron saturates between 6 and 26 dpa. Garner et al. [1] noted, however, that relatively small increases in displacement rate could significantly extend the duration of the transient regime of swelling in Fe–Cr alloys. Perhaps a similar dependence of transient swelling behavior on displacement rate occurs in pure iron. The factor of four difference in dpa rate in the two experiments conducted simultaneously in BR-10 is comparable to the difference in dpa rate between EBR-II and FFTF, where a very large difference in transient duration was observed for Fe–Cr binary alloys.

5. Conclusions

It appears that cold working of pure iron can significantly increase the swelling of pure iron at 400°C, most likely by shortening the transient regime of void nucleation. The transient duration may also increase with increasing displacement rate, as observed in experiments involving Fe–Cr binaries. The results of this experiment provide some support for the proposal that the intrinsic steady-state swelling rate of pure iron and Fe–Cr ferritic alloys is larger (⩾0.2%/dpa) than previously envisioned.

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