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EFFECTS OF PULSED DUAL-ION IRRADIATION ON MICROSTRUCTURAL DEVELOPMENT*

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The effect of pulsed irradiation on the development of microstructure during Ni ion bombardment has been investigated in a simple austenitic alloy similar to type 316 stainless steel. Bombardment conditions were 10 dpa, 940 K, pulsing with equal on/off times of either 0.5 or 60 s, and the addition of 20 appm He/dpa to some specimens either by room temperature preimplantation or by dual-beam coimplantation. Particular care was taken to minimize thermal pulses from beam heating (to < 2°C). The results show that pulsing has a subtle influence, and the effects on specific cavity parameters are complex. Pulsing produced a small increase in swelling in the helium-free case, but a slight decrease for helium-implanted specimens, and it seems to have counteracted the usual stimulative effects of helium on cavity nucleation. An assessment is made of the often-conflicting observations in the literature; there is a need for further, well-controlled experiments.

INTRODUCTION

Many concepts of magnetic confinement fusion reactors (MFR) envision their operation as a regular sequence of on ("burn") and off periods. The implications for materials behavior of this unique "pulsing" have been identified as a major unknown area in fusion technology.† In particular, the influence of periodic interruptions of displacement damage, relative to that of continuous exposure, upon the radiation effects engendered in the reactor first wall has been only lightly explored, with generally ambiguous results [1–5]. The present experiment seeks to explore such effects, building upon a base of previous work [6–8] (in part reported at the previous conference in this series) which dealt with the influence of fusion-level helium upon damage in the same material.

Experimental Conditions

The material studied is an austenitic alloy, designated P7, whose major constituents (Fe–17 wt % Cr, 16.7 Ni, 2.5 Mo) are similar to type 316 stainless steel, but with low (0.005 wt %) carbon and low residual elements (less than 0.1% each). In the prior steady-irradiation experiments the material swelled readily and remained single-phase even to high dose. Pulsing was made possible by the addition of solenoid-actuated pivoting beam stops to both the Ni and the He beam lines. The beam stops can be actuated simultaneously and provide any combination of on/off times greater than about 0.5 s. Details of the dual accelerator facility and our bombardment procedure have been given elsewhere [9,10].

Rapid simulation of neutron irradiation by charged particle bombardment almost inevitably entails some amount of additional heating when the beam is on. Until the specimen temperature controller can respond by adjusting the provided heating, this beam heating can lead to initial temperature excursions of 20 K or more. In the case of pulsed irradiation, such concurrent temperature fluctuations were considered particularly detrimental, insofar as they would complicate the interpretation of results. Therefore, for this experiment, an instantaneous damage rate in the peak damage region of ~2 × 10^{-3} dpa s^{-1}, about a factor of 3 lower than in the previous work, was deliberately employed so that the temperature excursions caused by beam heating were held to only 2–3 K, as observed with an infrared pyrometer which could monitor the bombarded surface of each specimen. All bombardments were carried out at one temperature to a common dose, nominally 940 K and 10 dpa. Helium was introduced either by dual beam coimplantation at a rate of 20 appm/dpa (appropriate to fusion) or by room temperature preimplantation of 200 appm; nonimplanted specimens were also studied.

The pulsing options were: fast pulse (0.5 s on/off), slow pulse (60 s on/off), and continuous bombardment as a control. The shorter pulse period is somewhat longer than the vacancy lifetime at the chosen bombardment temperature (which is approximately the peak swelling temperature, adjusted downward ~15 K to compensate for the lower dose rate). Pre- and postbombardment specimen preparation, and transmission electron microscope (TEM) examination, was accomplished in the same manner as described previously [6]. The depth of "microexcavation"

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†There is an important qualitative difference between pulsing taken to mean sequential interruptions of "normal" intensity irradiation (as in the MFR environment) and pulsing referring to the compaction of irradiation into brief periodic events of very high intensity, typical of Inertial Confinement Fusion Reactor (ICFR) designs. The current experiment relates only to the former concept of pulsing.

previously [6]. The depth of "microexcavation" of the bombarded surface was adjusted to 0.5–0.55 μm, in accord with a recent depth-dependence investigation in Ni [11] which also found cavitation not to vary strongly with depth between 0.45–0.9 μm. Swelling values were computed directly from the observed cavity sizes and concentrations per ASTM recommendations [12]. At least two specimens were normally examined for each bombardment condition. Specimen-to-specimen variations in cavity parameters were less than ±15% for the helium-free and coimplanted conditions, although preimplanted specimens exhibited greater scatter. Still, considering the sundry sources of error possible in any ion bombardment experiment, full trust ought not be attached to variations in cavity concentration or swelling less than a factor of ~2.

RESULTS

For this material and these bombardment conditions, the specific influence of pulsed irradiation seems to be complex and subtle, rather than clear-cut. As is evident in Fig. 1 and Table 1, the relative effects of fast and slow pulsing as well as continuous irradiation upon the damage microstructure vary with the presence or absence of helium. Irradiation without He produced the greatest contrast between fast and slow pulsing: fast pulsing gave rise to significantly larger and fewer cavities, while those resulting from slow pulsing were more numerous and slightly smaller than the no-pulse controls. Both fast and slow pulsing did increase the swelling relative to that of continuous irradiation. With helium added, the results were reversed: fast pulsing gave smaller cavities than did slow, and both types of pulsing produced considerably fewer cavities and less swelling than did continuous bombardment. Compared with each other, although the cavity sizes and concentrations shifted around, our slow pulse condition consistently generated more swelling and a higher dislocation content than did the fast pulsing. The dislocations were always in coarse network form, including segments of large loops.

There was an additional population of small matrix cavities (very likely gas-stabilized bubbles) engendered by the addition of helium. These cavities were sparse in coimplanted specimens but profuse for preimplantation; their characteristics are shown in Table 2 and their contribution to the swelling is included in Table 1, although they do not contribute to that table's statistics for $\bar{d}$ and N. Their sizes and numbers were not greatly affected by pulsed bombardment in general, or by fast vs slow pulsing in particular.

Besides the small matrix cavities, small grain boundary cavities were also observed in all helium implanted material. Based on a rough, qualitative analysis the grain boundary cavities in pulsed (fast or slow) dual beam specimens seemed to be about one fourth as numerous and ~50% larger than their continuously-irradiated counterparts. For preimplanted material, pulsing yielded somewhat smaller boundary cavities of approximately equal concentration compared with continuous bombardment.

DISCUSSION

What *are* the effects of pulsed irradiation on cavity formation? The earlier investigations exposed a considerable number of influential variables, such as pulse period, duty factor, temperature, dose, material, etc. But even controlling for these, the literature presents a surprising range of conflicting observations. Scanning these briefly, the early work of Sprague and Smidt [1,2] found that fast (2.5 ms on/off) raster-pulsing of pure Ni to 3 dpa yielded swelling (S) generally comparable to continuous irradiation, but with a markedly-reduced cavity mean diameter $\bar{d}$: down by a factor of 5 at 873 K. On the other hand, Taylor et al. [3] who also studied pure Ni at 873 K with fast (13 ms on/off) pulsed vs continuous irradiation to 7 dpa found that pulsing increased $\bar{d}$ by nearly double, although with an offsetting ten-fold decrease in the cavity concentration N which resulted in (again) little net difference in S. Taylor's work also investigated longer pulse times (2–6 s on-time) and varied duty factors at 773 K, reporti[illegible] somewhat enhanced S and $\bar{d}$ for equal 6.75 [illegible]ff pulsing. They also found a reduced S an[illegible] lower duty factor. Overall, though, they co[illegible]ded that such pulsing in their system did not produce large effects. That was not the conclusion of Powell and Odette [4], however, who conducted pulsed (2, 20, or 60 s on/off) HVEM irradiations of an Fe–15Cr–25Ni ternary alloy to 4 dpa at 873 K. Powell saw a twenty-fold reduction in swelling rate for 60 s on/off pulsing (less reduc[illegible]r the shorter pulse periods), stemming fro[illegible]gnificantly reduced $\bar{d}$ and somewhat increased N. The remaining study, by Kaletta [5], is not comparable to the others because it employed 2 MeV He^{+} ions alone to create low -- only 0.28 dpa — damage but very high helium concentrations in a V target. The resulting damage structure at 848 K consisted of profuse He bubbles, and the effect of 300 s on/off pulsing was to greatly increase S and $\bar{d}$ while reducing N.

One generally neglected parameter that may underlie these discrepancies is the dose rate. If the dose rate is high, concomitant *temperature* pulsing due to beam heating may be an unavoidable complication. The Sprague beam-rastering experiment utilized a very high $8 \times 10^{-[illegible]}$ dpa s^{-1} dose rate, while that of Taylor was 6×10^{-3} (time averaged). Even the lower of these two rates suffices, in our own facility, to gene[illegible] 15–20 K initial beam heating for bombardments conducted at ~873 K. The Powell and Odette experiment featured a lower dose rate (1.3×10^{-3} dpa s^{-1}), but since this was confined

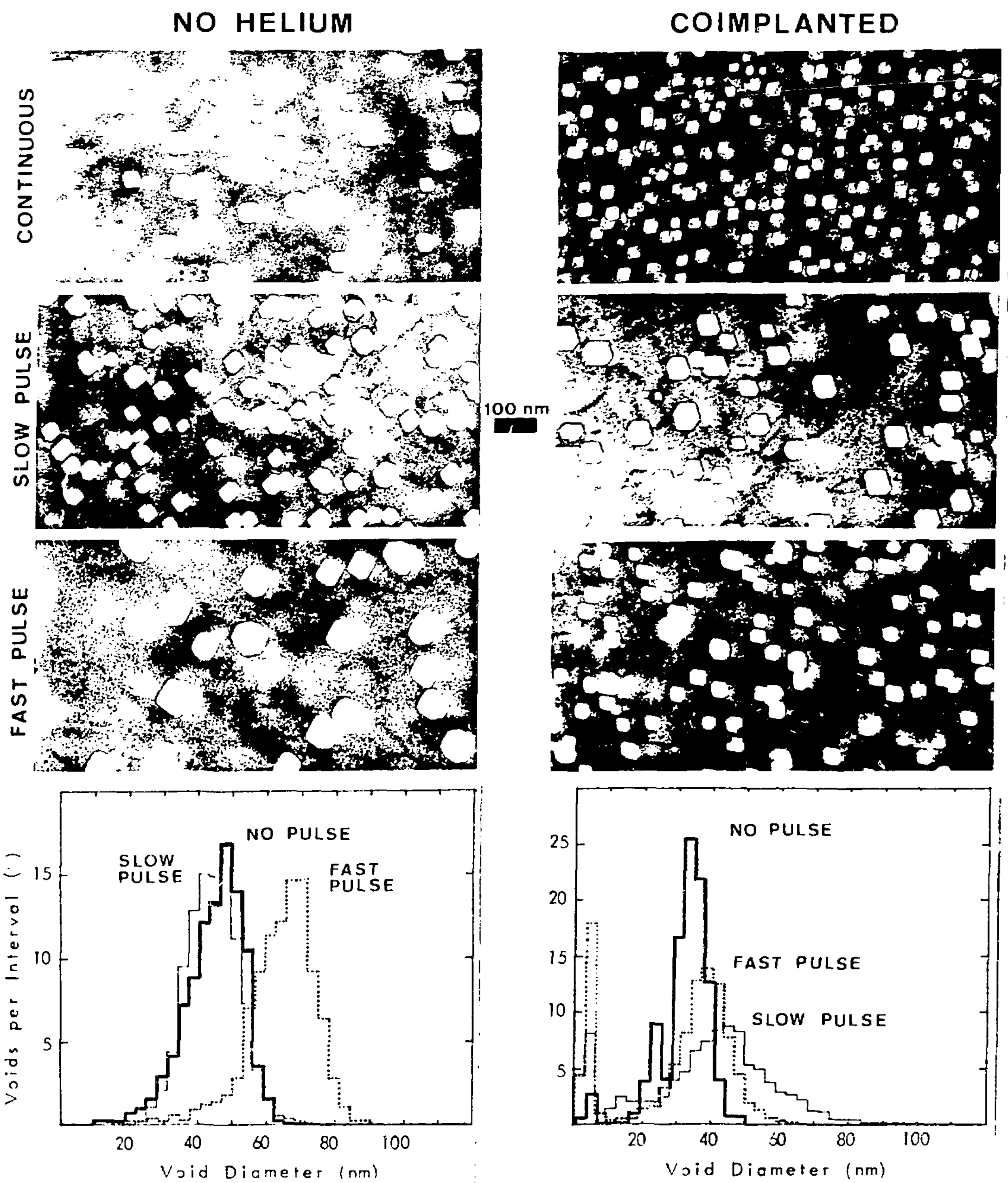

Fig. 1: Swelling resulting from pulsed ion bombardment, either without helium (left) or with dual-beam coimplantation of 20 appm He/dpa (right). Pulsing conditions are: continuous bombardment (top), slow pulse (middle), fast pulse (bottom).

Table 1. Cavity Characteristics in P7 Austenitic Alloy Bombarded to 10 dpa at ~940 K

	Total Swelling S (%)	Cavity Diam $\bar{d}$ (nm)	Cavity Conc. N ($10^{20} m^{-3}$)	Dislocation Density L ($10^{14} m^{-2}$)
No Helium				
No Pulse	1.8	55	2.5	1.2
Slow Pulse	2.8	50	4	1.8
Fast Pulse	2.4	65	1.4	1.5
Coimplanted He				
No Pulse	3.2	35	15	1.9
Slow Pulse	3.3	55	4	1.7
Fast Pulse	2.6	44	6	1.5
Preimplanted He				
No Pulse	0.30–1.0	19	8–19	0.4–1.5
Slow Pulse	0.14	21	2	0.2
Fast Pulse	0.06–0.09	13–16	0.7–2	0.07–0.2

Table 2. Characteristics of the Additional Small Matrix Cavities in He-Implanted Specimens

	Cavity Diam $\bar{d}$ (nm)	*Cavity* Conc. N ($10^{22} m^{-3}$)
Coimplanted He		
No Pulse	4.2	2
Slow Pulse	5.2	1
Fast Pulse	5.2	4
Preimplanted He		
No Pulse	4.4	100
Slow Pulse	4.9	80
Fast Pulse	4.9	80

within an HVEM foil, they had to quote a value of beam heating "less than 10°C," which may still be non-negligible. The present experiment has sought to carefully investigate the influence of pulsing in 0.5-mm-thick (i.e., not thin foil) specimens with beam heating effects minimized. The incorporation of added helium breaks new ground, also.

With the microstructural sink strength data of Table 1, it is possible to calculate, after the fact, the approximate vacancy lifetime, τ_v, in effect at an interruption of bombardment. The relation

$$\tau_v \sim (D_v S_v)^{-1} \qquad (1)$$

is used with the customary expressions

$$D_v = D_v^0 \exp(-E_v^m/kT)$$

and

$$S_v = Z_v^c \, 2\pi \bar{d}_c N_c + Z_v^d L \quad .$$

Values from Mansur [13], thought to be reasonable for stainless steel, of

$D_v^0 = 0.014$ cm^2 s^{-1}, $E_v^m = 1.38$ eV, and

$Z_v^c \approx Z_v^d \approx 1$, together with the Table 1 data combine to yield an estimated value of $\tau \approx 0.1$ s for all of the no-helium cases. This is shorter, but still comparable, to our fast pulse on/off times.

The general finding of this experiment is that pulsing, under our conditions, produced relatively small effects (if any). For the bombardments without He, fast (0.5 s) pulsing gave increased $\bar{d}$ and S. An increased size is understandable in terms of current cavity growth theory, *because during the frequent "off"* periods, interstitial point defect concentrations plummet immediately, while the vacancy supersaturation persists awhile (as calculated above) and may be augmented by emission from submicroscopic vacancy clusters [14]. Thus the fraction of defects recombining may be reduced. Enhanced cavity growth should be the result.

Adding helium either prior or simultaneously had the expected [6] strong stimulative effect on the cavity concentration N for *continuous* bombardment, but — for some reason — much less change in N for pulsed specimens. Pulsing appears to have caused a significant change in the helium-vacancy transport mechanism. There does not seem to be a satisfactory void nucleation model that will help explain these results. One observation that *does* seem explicable, however, is the relative insensitivity of the secondary small matrix cavity sizes and numbers to the presence and type of pulsing. Such probably pressure-stabilized sinks ought to be relatively oblivious to the perturbed point defect fluxes existing during the off periods.

CONCLUSIONS

Under a limited range of conditions for a high swelling, phase stable alloy, this experiment has addressed the critical *question of the effects of* pulsing on cavity swelling. The observations indicate that pulsed, compared with continuous, heavy-ion bombardment produced relatively small-scale but intriguing changes in the damage microstructure:

- For helium-free irradiations, swelling was somewhat higher for pulsed compared with continuous bombardment.

- For specimens with helium added, either by simultaneous or preimplantation, pulsing slightly reduced the swelling.

- Addition of helium in pulsed irradiation did not cause the marked increase in cavity concentration that it did in continuous irradiation.

- Slow (60 s on/off) pulsing gave slightly greater swelling than did fast (0.5 s) pulsing.

- A secondary matrix population of small cavi-ties (probable helium bubbles) in the He-implanted specimens was relatively insensitive to pulsing condition.

These apparently-simple trends must be regarded with caution, because the microstructural parameters that comprise "swelling" — cavity size and concentration —varied in an extremely complex fashion. Work in progress is exploring the effect of pulsing at other bombardment temperatures in this material [15].

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