EFFECTS OF RAPIDLY PULSED ION BOMBARDMENT ON MICROSTRUCTURE
AND PHASE STABILITY IN A Ti-MODIFIED STAINLESS STEEL*

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Abstract

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A low-swelling Ti-modified austenitic stainless steel has been bombarded with pulsed 4 MeV Ni^{++} and in some cases simultaneously with 0.2-0.4 MeV He^+ ion beams at 948 K over a dose range from 1 to 70 dpa. The interruption periods studied were 60, 1, 10^{-3} , or 10^{-5} s on time with equal off time. Continuous irradiation was also employed as a comparison. At 1 dpa, pulsed irradiation caused interstitial dislocation loops (and subsequent MC precipitates) to be more refined with decreasing pulse periods down to the realm of the vacancy lifetime (10^{-1} to 10^{-3} s); still faster pulsing at 10^{-5} s yielded little difference from steady irradiation. At higher doses, 40-70 dpa, pulsing reduced the amount of radiation-induced G phase. Long pulse periods promoted the thermally-stable MC phase, while shorter periods like 10^{-3} s aided M_6C and M_{23}C_6 development. In helium-implanted material bubbles were present for all pulsing conditions but bias-driven voids only developed in continuously-irradiated specimens. The results are interpreted with regard to the inherent fluctuations in point defect concentrations that result from cascade formation. The sensitivity of damage microstructure to pulsed bombardment is seen as a possible new tool for the control of microstructure in ion implantation experiments.

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1. Introduction

The influence of beam pulsing is a topic of interest in both radiation effects and ion beam modification research (in particular, the use of rastered beams for ion implantation). A heavy ion bombardment facility with beam chopping capability is a powerful tool to study the kinetics of the processes induced by atomic vacancies and interstitials produced by irradiation. In our previous studies, the effect of pulsed irradiation was studied using a solenoid-actuated beam chopper which could interrupt the beam for intervals as short as 0.5 s [1]. In the experiments to be described in the present paper, a recently developed electrical chopper was used to produce beam interruptions as short as 10 microseconds. Such times approach the interstitial lifetime and are significantly shorter than the vacancy lifetime for these irradiation conditions.

The focus of this paper is to investigate the effects of pulsed irradiation on microstructural evolution in a Ti-modified austenitic stainless steel. Significant differences in microstructure and phase stability were found previously for slow pulsed irradiation (1 s on/1 s off, 60 s on/60 s off) compared to continuous irradiation [1]. The results were interpreted in terms of theoretical concepts in radiation effects [2]. Additional information now has been obtained using 10^{-3} s on/ 10^{-3} s off and 10^{-5} s on/ 10^{-5} s off pulse intervals. The greatest changes were manifest in loop growth and precipitate formation.

2. Experimental

Solution annealed (1373 K) Ti-modified austenitic stainless steel (Fe-15Cr-15Ni-2Mo-2Mn-1Si-0.2Ti-0.05C in wt %) was studied with pulsed-beam irradiation using the ORNL dual ion Van de Graaff accelerator facility. The bombardments employed either 4 MeV Ni-ions alone or simultaneously with 0.2-0.4 MeV helium ions. Beam pulsing was carried out in two ways. For the 60 s and 1 s pulsing, both the Ni^{++} and the He^+ beams were simultaneously interrupted using synchronized mechanical choppers (one for each beam line). For the fast 10^{-3} and 10^{-5} s pulsing, the Ni-ion beam was deflected at the ion source prior to acceleration using a square wave ac field generated by a push-pull circuit. Details of the latter pulsing technique have been described [3]. In all cases pulsing was carried out with equal beam on/beam off intervals. Helium was introduced into the slow-pulsed specimens at a rate of 20 appm He/dpa with the chopped He^+ beam. The fast-pulsed specimens received a continuous injection of helium at an average rate of 10 appm He/dpa, whether the Ni-beam was on or off, to yield the same total helium concentration as in the slow-pulsed specimens. Specimen disks 3 mm-diameter by 0.35 mm-thick were irradiated to doses of 1, 40, and 70 dpa at 948 K (near the peak swelling temperature). The irradiated specimens were then thinned for electron transparency in the region 0.6 to 0.8 μm below the surface, near the peak damage depth. Specimens were examined using a JEM 120CX analytical electron microscope equipped with an energy dispersive x-ray spectrometer. A general procedure for microstructural examination and precipitate particle analysis has been described [1].

3. Results

The greatest effect of pulsing was manifest at the loop growth stage (~ 1 dpa). At higher doses pulsing effects were subtle and mainly appeared in precipitate development.

3.1 Microstructure at 1 dpa

The microstructures at 1 dpa were dominated by faulted interstitial loops. Figures 1 and 2 show the loop microstructures that developed during various pulsed irradiations, without and with helium injection, respectively. The most striking features are that pulsing restricts loop growth, particularly for longer beam-on times (>1 s), and adding helium further refines loop sizes and generally increases loop concentration. Table 1 summarizes the measured average loop diameters and their concentrations for various irradiation conditions. Values for loop concentration were difficult to obtain; the estimated uncertainty is about a factor of two, due primarily to place-to-place variations. Figure 3 depicts the variation of average loop diameters as a function of pulse duration. As the pulsing frequency increased, from right to left in the figure, the average loop diameter initially decreased slightly showing a minimum at 1 s on/1 s off pulses but increased again for further reduced periods. For a half-pulse interval of 10^{-5} s, the average loop diameters approached those of continuous irradiation. An unusual presence of line dislocations was, however, observed in the specimen irradiated with a 10^{-3} s half-pulse duration without helium injection (Fig. 1c). Since such an early development of line dislocations was not otherwise observed in this series, this

singular behavior may have originated from prebombardment handling rather than being of intrinsic nature.

The loops were often distorted and revealed black-white contrast when they were very small; on further growth, they exhibited irregular shapes with numerous lobes. Similar loops that were termed "crenelated" or "rosette-like" were observed previously in type 316 steel irradiated with 1 MeV electrons [4] and in 304 stainless steel irradiated with neutrons [5]. Our high magnification micrographs now reveal that the shape is caused by impeded growth, which is due in turn to pinning by tiny MC particles and/or small helium bubbles nucleated at the loops. Figure 4 shows examples of loops pinned by MC and helium bubbles. There was no consistent correlation of damage conditions with the degree of crenelation of the loops, but it seemed that fast pulsing together with helium increased the effect. MC particles were nucleated on a much finer scale when helium was injected in large amounts. This primarily was due to the coprecipitation of MC and helium bubbles [6,7]. In measuring the loop diameters, the longest diameter across the loop was used. Since most loop morphologies were ill-defined because of pinning, there were some uncertainties in actual loop sizes, but loop growth showed definite effects of pulsing and helium. A few G- and eta-phase particles were observed at 1 dpa, but the presence of these particles became prominent only at higher doses.

3.2 High Dose Microstructure, Without Helium

The effect of pulsing on precipitation was not obvious until high doses. Although a cursory check of TEM micrographs suggested similar

precipitate microstructures irrespective of pulsing rates, careful analysis indicated that there were subtle differences in precipitate formation with pulsing conditions. At 40 dpa, the microstructure was essentially established and further irradiation only caused dislocation motion and precipitate growth. Figure 5 shows TEM micrographs after 40 dpa at 943 K without helium injection for various pulse intervals. The damage microstructure comprised dislocation network and various precipitate phases. This behavior continued to 70 dpa.

Quantitative x-ray EDS analysis indicated that the relative distribution of precipitate phases varied with pulsing rate. The major precipitate phases found in this experiment were G, MC, eta (M_6C), and $M_{23}C_6$. Among them MC, eta, and $M_{23}C_6$ also form during thermal aging but were enhanced or retarded depending upon irradiation conditions. Under ion irradiation, diffusional migration of Si and Ni are particularly important. Segregation of these elements to loops or dislocations leads to the formation of G-phase [8]. The G-phase does not occur during thermal aging and it dissolves during post-irradiation annealing: it is a true radiation-induced phase. A detailed description of phases that are found during irradiation or aging has been described previously [8]. A brief summary of the structure and typical compositions of the four major phases that occurred during irradiation is presented in Table 2. Since the morphologies of G and eta phases are very similar, the distinction between them was mostly based on EDS x-ray analysis of extracted particles. The analyses presented here are qualitative but the trends are unmistakably

clear. The major phases in continuously irradiated samples were G and MC, while few eta phase particles were present.

This situation changed when the beam was pulsed. Pulsing stimulated MC nucleation and also promoted the eta and $M_{23}C_6$ phases. As the pulsing rate increased, the MC particle concentrations increased and their average sizes decreased, with the greatest refinement at 10^{-3} s pulse duration. Although the particle concentration could not be measured accurately because of coarsening and coalescence along dislocations, it was estimated to be in the range of $3 \times 10^{21}/m^3$. That value was about two to three times higher than in continuously-irradiated specimens. A striking fact was that large $M_{23}C_6$ particles were also nucleated at grain boundaries in 10^{-3} s and 10^{-5} s pulsed specimens. This $M_{23}C_6$ phase was not observed in slow pulsed or continuously irradiated specimens. Although pulsing (especially at high frequencies) caused the enhancement of eta phase, G-phase was still the most numerous of the large-particle-size precipitate in all specimens. This trend changed, however, when helium was injected.

3.3 Helium Effects

Bubbles and cavities were not observed in samples without helium injection. As described already, when helium was injected loop size was restricted and MC nucleation was stimulated at 1 dpa. At higher doses helium caused bubble formation, aided cavity growth, and affected precipitate development. Figures 6 and 7 show TEM micrographs after irradiation to 40 and 70 dpa, respectively. Small bubbles were visible in all samples but large cavities were observed only in continuously irradiated samples,

Figs. 6a and 7a. Helium promoted MC, ϵ , and $M_{23}C_6$ phases and this effect was further enhanced under pulsed irradiation. Pulsing alone refined MC particles, and helium further promoted MC nucleation. Thus, the greatest number of MC particles and helium bubbles appeared when both pulsing and helium were applied. Among the large precipitates, the ϵ phase now became the major phase, surpassing G-phase in volume fraction. The $M_{23}C_6$ phase was also formed at grain boundaries in specimens irradiated with pulse half-periods of 10^{-3} s and 10^{-5} s. There was a direct correlation between MC particles and helium bubbles. Since MC phase has a positive misfit with the matrix and nucleates more readily at a helium bubble, the MC particle concentration increased as the bubble density increased. The concentrations of MC particles were, in fact, comparable to helium bubble concentrations and were estimated to be about 1 to $3 \times 10^{22}/m^3$, taking into account the coalescence effects. Bubbles were often very small (<1 nm) and difficult to image. However, high magnification TEM micrographs yielded estimated observable bubble concentrations in the range of 1 to $3 \times 10^{22}/m^3$ with average diameters of 3 to 4 nm. As can be seen in Figs. 6c and 7c, the finest MC particles were observed in samples with 10^{-3} s pulse duration and there must have been many bubbles there too small to be seen; a bubble concentration much higher than $3 \times 10^{22}/m^3$ is estimated for the 10^{-3} s pulsed condition. In pulsed irradiations, bubbles ranging up to 10 nm diameter did not display bimodal size distributions (indicative of bias-driven cavity growth). On the other hand, in the continuous irradiation mode some cavities, particularly bubbles at G-phase precipitates, exhibited bias-driven growth at 40 dpa. In these cases a

bimodal cavity size distribution was well developed by 70 dpa. The distribution consisted of small bubbles, at a concentration of about $3 \times 10^{22}/\text{m}^3$, with an average diameter of 3.2 nm and maximum cut-off diameter about 10 nm, plus large cavities numbering about $3.6 \times 10^{20}/\text{m}^3$ with an average diameter of 41 nm and producing about 2% swelling. For continuous irradiation G-phase was the dominant phase and there was considerable dissolution of MC in the irradiation period between 40 and 70 dpa, while G-phase particles were growing. Dislocation densities approached $2 \times 10^{14}/\text{m}^2$ irrespective of irradiation conditions at 70 dpa.

4. Discussion

The nature of the "pulsed" irradiation employed in this and essentially all prior ion bombardment experiments is a sequence of irradiations (ON times) interspersed with anneals (OFF times). The net effect corresponds to the "burn" and "off" times of a pulsed Tokamak fusion reactor, although the ion bombardment pulse periods are generally much shorter than the Tokamak--corresponding to the major speed-up in damage generation itself. (By contrast, the highly concentrated nanosecond pulses of an Inertial Confinement Fusion Reactor have not been simulated by this or any other experiment to date.)

It is important from a fundamental perspective to see if basic changes or special effects result from the use of pulse periods that are in the realms of the vacancy or interstitial lifetimes. From cascade diffusion theory one can recognize that the primary damage events--cascade formation

and diffusion of the point defects--give rise to inherently "pulsed" defect fluxes [9,10]. A given location in the material will experience, during "steady" ion irradiation, a rapidly varying vacancy concentration as well as spikes of interstitials on the order of 10^{-6} s apart. Given these discontinuities, it follows that it should not be possible to discern effects of irradiation that is interrupted for times shorter than the lifetime of the controlling defect (vacancy or interstitial), since a higher frequency pulsing cannot be distinguished from the intrinsic focal fluctuations that take place during steady irradiations. For the conditions of this experiment--stainless steel vacancy migration energy $E_v^m \sim 1.3$ eV, irradiation temperature 950 K, and apparent sink densities (at 1 dpa, $S_v \sim 2 \times 10^{13} \text{ m}^{-2}$ was observed in small loops calculated vacancy lifetime for loss to sinks, τ_v , is about 0.2 to 0.5 τ^* while the interstitial lifetime is on the order of 10^{-6} s.

The effects of slowly pulsed (60 s and 1 s on/off) irradiation on the low dose microstructure were largely attributable to the expected influences of periodic anneals and irradiation "restarts" [1]. Pulsing and

$$^*\tau_v = (L_v S_v)^{-1}; \text{ with } S_v = S_v(\text{cavities}) + S_v(\text{loops}) = 4\pi r_c N_c Z_v^c + 2\pi r_l N_l Z_v^d,$$

$$\text{and } D_v = D_v^0 \exp(-E_v^m/kT)$$

where $r_{c,l}$ = radii of cavities or loops

$N_{c,l}$ = concentrations of cavities or loops

$Z_v^{c,d}$ = bias factors of cavities or dislocations for vacancies, and

E_v^m = activation energy for long-range migration of vacancies

helium, separately or together, refined the interstitial loop populations at 1 dpa. It is now clear that this behavior changes for shorter pulse periods (Fig. 3). Very fast pulsing (10^{-5} s) does not alter the loop microstructure; such brief cycles evidently are not distinguished from the $\sim 10^{-6}$ s inherent interstitial "pulses" that are always experienced.

At an in-between pulse period of 10^{-3} sec, the results are complex: apparently no effect (i.e., like steady irradiation) for helium-free specimens but a substantial loop refinement for the helium-injected material. The loop statistics for the He-free specimens are comparatively uncertain, but the loops that were observed were comparable to those of continuous irradiation. Based on the previous discussion, if the loop development is vacancy controlled, a null effect might be expected at this pulsing frequency since 10^{-3} s on/off times are much less than the predicted vacancy lifetime, which was calculated above from irradiation temperature and observed sink strength to be $\sim 10^{-1}$ s. So the lack of a pulsing effect for helium-free irradiation makes sense. Nevertheless, a pronounced loop refinement was experienced by helium-implanted material. We suggest that this latter effect arises because the vacancy lifetime is in fact reduced to $\sim 10^{-3}$ s in this case through a hundred-fold increase in the sink strength. Such a raised sink strength might well result from the introduction of the 10 at. ppm helium atoms which could provide a cavity sink strength of up to $S_v = 10^{15} \text{ m}^{-2}$ (several-fold less if in small clusters). The effectiveness of helium in trapping vacancies has been widely demonstrated. The upshot is that pulsing (viewed as a sequence of anneals and restarts) does affect loop evolution, but as the pulse frequency

increases there comes a point at which the pulse period approximates the vacancy lifetime and beyond which the deliberate beam pulses become indistinguishable from inherent fluctuations. That period turns out to be $\sim 10^{-1}$ s for helium-free irradiation and $\sim 10^{-3}$ s for the helium-injected case.

With increased dose the interstitial loops unfaulted and interacted to form a dislocation network which proved to be quite independent of pulsing conditions. Such an insensitivity of the network dislocation density has also been observed in all other pulsing studies as well [11].

At high doses (40–70 dpa), pulsed irradiation influenced the balance between thermally-stable phases like MC and the radiation-induced G phase. In pulsed irradiation the point defect currents responsible for radiation-induced segregation (RIS) tend to decay during the beam-off intervals and solute can flow back into the matrix. For the particular case of silicon, an undersized atom that evidently couples with interstitials for transport, even very brief interruptions like 10^{-5} s might perturb the RIS process that culminates in the Si- and Ni-rich G phase. (The outflow of Si would have to employ the substantial level of radiation-produced vacancies, since interstitials would be almost instantly gone after the start of a beam-off interval.) During the same beam-off times, thermal phases like MC experience a net influx of solute. Since G phase is purely radiation-induced while eta phase is a thermally-stable but radiation-enhanced phase that incorporates less, yet still elevated, levels of Si and Ni, and MC is both thermally-stable and Si-free, the observed trend of $G \rightarrow \eta \rightarrow MC$ for increasing beam-off time is understandable.

These experiments showed MC precipitates, and also helium bubbles, to be most profuse for 10^{-3} s pulsing. There was also a widespread association of MC precipitates with dislocation loops or lines. It is thought the peak refinement of loops at 10^{-3} s pulsing in turn imposed the corresponding refinement of the MC particles and the bubbles. Despite the great profusion of fine MC at 10^{-3} s, however, the total volume fraction of MC phase was apparently greater at longer pulse periods. A smaller total mass of MC at short pulse periods would have left more carbon in the matrix to accommodate eta phase (M_6C) formation, and perhaps also the modest amounts of $M_{23}C_6$ phase observed only for 10^{-3} and 10^{-5} s pulsing.

It is noteworthy that any growth of cavities from the class of small bubbles to large bias-driven voids occurred only for continuous irradiation and not for pulsed irradiation at any frequency. These observations lend support to the hypotheses that void growth is aided by a "collector mechanism" [12] through symbiotic attachment to growing G precipitates, and retarded if helium has to be distributed among a higher number of sinks [13]. Pulsing at both high and low frequencies retarded G-phase and generated more loops, bubbles, and MC precipitates, all of which are effective dispersed traps for helium, as Fig. 4 well illustrates.

Finally, even though the irradiation conditions employed in this experiment were chosen to represent fusion reactor first wall parameters, the findings are quite relevant to the ion implantation field. Pulsed irradiation has been seen to amount to a sequence of irradiation and anneal periods, the details of which can broadly influence the final damage

microstructure. If ion implantation is conducted with a rastered beam, as a means of uniformly covering an extended area, then each location in the material actually receives the implantation in pulses, the nature of which depends on the rastering parameters. Thus, whether the use of pulsed implantation comes about as a side effect (e.g. rastering) or by intentional interruptions (chopping), its application with deliberately chosen pulse periods is an additional tool to control the microstructure (point defect clusters, precipitates, etc.) and thereby the properties of the implanted region.

5. Conclusions

Pulsed single (Ni^{++}) or dual (Ni^{++} , He^+) ion beams with beam on/off times ranging from 10^{-5} to 60 s have been used to irradiate a low swelling stainless steel at 948 K over the dose range of 1 to 70 dpa with the following findings:

- The effects of very fast pulsing can be indistinguishable from the inherent fluctuations (e.g. cascades) of damage generation.
- At 1 dpa, the pulsing-induced refinement of interstitial loops is at a maximum for pulse periods that correspond roughly to the calculated vacancy lifetime (10^{-3} s and 10^{-1} s for helium-implanted and helium-free material, respectively).
- The pulsing frequency can affect phase evolution, presumably by altering the balance between radiation-driven and thermal processes.

- Growth of small cavities into voids failed to occur for any pulsing condition, perhaps because of the pulse-induced suppression of β -phase and/or the dilution of available helium among a higher density of sinks.
- Pulsed ion implantation (including beam rastering) at deliberately chosen frequencies can be an additional tool to alter the final implanted microstructure.

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FIGURE CAPTIONS

- Fig. 1. Dislocation loops after bombardment at 948 K to 1 dpa with 4 MeV pulsed Ni^{++} ions (pulse half-periods indicated).
- Fig. 2. Loops after 1 dpa irradiation with concurrent 4 MeV pulsed Ni^{++} ions (pulse half-periods indicated) and 0.2–0.4 MeV He^+ ions (to a final helium level of 20 appm).
- Fig. 3. Dependence of average loop diameter on pulse half-period for both single and dual beam bombardments.
- Fig. 4. High magnification images of loops pinned by small helium bubbles (left) and MC precipitates (right) in continuously-irradiated material.
- Fig. 5. Precipitates resulting from bombardment to 40 dpa with pulsed Ni^{++} ions only (pulse half-periods indicated).
- Fig. 6. Precipitates from 40 dpa bombardment with concurrent pulsed Ni^{++} ions and He^+ ions (to a final level of 800 appm helium).
- Fig. 7. Microstructure after 70 dpa dual beam bombardment revealing very small bubbles for the pulsed irradiations and ~2% void swelling for continuous irradiation.

Table 1. Loop Parameters at 1 dpa

Pulse sec on/off	Helium appm/dpa	Loop	
		Concentration $10^{19}/\text{m}^3$	Diameter nm
Continuous	0	4.5	143
$10^{-5}/10^{-5}$	0	2.3	150
$10^{-3}/10^{-3}$	0	$<1.0^*$	130^*
60/60	0	9.3	68
Continuous	$20/0^\dagger$	7.4	128
$10^{-5}/10^{-5}$	10/10	5.0	130
$10^{-3}/10^{-3}$	10/10	12.0	40
1/1	20/0	7.3	24
60/60	20/0	6.9	45

*Most loops are unfaulted.

† When Ni beam is on/when Ni beam is off.

Table 2. Structures and Typical Compositions of Four Major Phases
Which Occur During Ni-Ion Irradiation

Phase	Effect of Radiation		Structure, Lattice Para.	Typical Composition (wt %)*						
	Continuous	Pulsed		Fe	Cr	Ni	Si	Ti	Mn	Mo
Matrix	--	--	Cubic Fm3m 0.36 nm	64	15	15	1	0.2	2	2
G	Induced	Less	Cubic Fm3m 1.12 nm	7	7	51	21	4	5	5
MC	Enhanced	More	Cubic Fm3m 0.43 nm	1	1	--	--	75	--	23
η	Enhanced	More	Cubic Fd3m 1.08 nm	15	32	30	7	--	--	16
$M_{23}C_6$	Retarded	Enhanced	Cubic Fm3m 1.06 nm	22	60	5	1	--	--	12

*Carbon is not included.

1 dpa (No Helium)

1 dpa (Helium)

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40 dpa (No Helium)

40 dpa (Helium)

70 dpa (Helium)