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EFFECT OF HELIUM PREINJECTION AND PRIOR THERMOMECHANICAL TREATMENT ON THE MICROSTRUCTURE OF TYPE 316 SS*

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EFFECT OF HELIUM PREINJECTION AND PRIOR THERMOMECHANICAL TREATMENT ON THE MICROSTRUCTURE OF TYPE 316 SS*

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SUMMARY

Samples of 316 SS were preinjected with 15 appm helium either hot (650 C) or cold (room temperature) and irradiated with 3 MeV Ni^+ ions to a dose level of 25 dpa at 625 C in order to test the validity of helium preinjection as a means of simulation of transmutant helium production. Results for preinjected and single-ion irradiated samples were compared to samples irradiated with 3 MeV Ni^+ and simultaneously injected with helium at a rate of 15 appm He/dpa (dual-ion irradiated samples). Preinjected samples exhibited bimodal cavity size distributions. Preinjected samples of solution annealed or solution annealed and aged material showed lower swelling than dual-ion irradiated samples. However, He preinjection in 20% cold worked samples showed greater swelling than dual-ion irradiated samples.

INTRODUCTION

The helium produced in materials during irradiation in reactors has an important influence on void nucleation and irradiation damage structure evolution. Helium preinjection (usually 10 to 30 appm) is often used to compensate for the lack of helium in the early nucleation phase of microstructure development in mixed-spectrum reactor irradiations and in ion and electron

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irradiations as well. Unfortunately, we do not yet have a clear picture of the differences between microstructures produced by simultaneous displacement damage and helium production, and those produced by helium preinjection followed by displacement damage.

The objective of this investigation is to study these differences, and thus test the adequacy of helium preinjection as a simulation of transmutant helium production.

MATERIALS AND PROCEDURE

The material for this study was 316 SS from the MFE heat (15893). Irradiations were performed on samples with three prior thermomechanical treatments: solution annealed (0.5 h at 1050 C), solution annealed and aged (0.5 h at 1050 C, 10 h at 800 C), and 20% cold worked. The irradiations were performed at 625 C, which is near the peak swelling temperature for the 3×10^{-3} dpa/sec damage rate [1] that was employed in this study. Nickel-ion irradiation (3.0 MeV Ni^+) was used for displacement damage production and helium preinjection was performed with degraded 0.83 MeV He^+ ions. A detailed description of the facility and the procedure for determining the concentration and distribution of the preinjected He^+ is given in Refs. (2) and (3). Preinjection with 15 appm helium was performed either hot (650 C) or cold (room temperature); the samples were subsequently irradiated with Ni^+ ions to a dose level of 25 dpa.

TEM observations of irradiated samples were performed using a JEM 100-CX electron microscope. The carbon spot method [4] was used to measure the foil thickness of the observed microstructures, and the cavity size distributions were obtained from analysis of photo micrographs with a Zeiss particle size analyzer.

In all cases the micrographs for microstructure analyses were recorded in a (200) two beam diffraction condition for dislocation contrast, or in under-focused absorption contrast to image the cavities. Irradiation conditions are summarized in Table 1.

EXPERIMENTAL RESULTS

Typical microstructures for 25 dpa cold- and hot-preinjected, single ion irradiated samples of solution annealed (SA), solution annealed and aged (SAA) and 20% cold worked (CW) material are presented in Figs. 1, 2, and 3, respectively. For easy comparison with dual-ion microstructures we have included 25 dpa, 15 appm He/dpa dual-ion micrographs in each figure.

The 25 dpa cold-preinjected, hot-preinjected and dual-ion samples are hereafter denoted by 25CP, 25HP and 25D, respectively. Beam histories 25CP, 25HP and 25D produced significantly different microstructures in samples with each of the prior thermomechanical treatments; microstructures in SA and SAA material closely paralleled one another but the effect of preinjection on CW material was entirely different.

In SA and SAA samples, Figs. 1 and 2, the dislocation densities in both 25CP and 25HP samples were significantly lower than for 25D irradiation and the fractional contribution of loops was much higher; i.e., the development of dislocation microstructure appeared to be significantly retarded in preinjected samples. There were many very small cavities that were close to the visibility limit in 25CP and 25HP samples of SA and SAA material, and the large-cavity number densities were a great deal lower than those produced by dual-ion irradiation. Cavities in 25CP samples were significantly smaller than the large cavities in dual-ion samples, while cavities in 25HP samples were significantly larger.

In cold worked samples, Fig. 3, both cold- and hot-preinjection produced large-cavities that were not observed for the dual-ion irradiation. The presence of the large cavities caused an increase in swelling that reverses the trend seen in SA and SAA samples.

The size distribution of cavities in SA, SAA, and CW specimens that contained hot- or cold-preinjected helium and were single-ion irradiated to 25 dpa are presented in Figs. 4, 5 and 6, respectively.

Through precise observations of the small cavities, the size distributions were revealed to be bimodal in He preinjected samples of SA, SAA and CW material; this has not been previously reported except in specimens with high concentrations of preinjected helium [5]. The average diameters of large-cavities in the HP, CP and D specimens were highest in 25HP samples and lowest in 25CP samples for SA and SAA material as shown in Fig. 7. Dual-ion irradiation produced no large cavities in CW specimens, but both 25CP and 25HP produced large cavities and bimodal cavity size distributions.

Regarding the average size of small cavities, the helium preinjection produced average cavity sizes that were very close to the visibility limit. Figure 8 shows the dependence of the cavity number density on irradiation dose from ref. 1, together with the values from this study. For both large and small cavities, helium preinjection followed by single-ion irradiation produced cavity number densities that were significantly lower than in dual-ion irradiated specimens. In 25HP samples, the cavity number density was higher than in 25CP for both large cavities and small cavities in materials with all three prior thermomechanical treatments.

The dependence of the average cavity volume fraction on irradiation for hot- and cold-preinjection followed by single ion irradiation and for dual-ion irradiation is shown in Fig. 9. In cold worked samples both hot- and cold-preinjection enhanced swelling relative to dual-ion irradiation instead of retarding it as in SA and SAA material. It can also be seen that swelling in preinjected cold worked samples appears to be as high or possibly higher than the swelling in preinjected SA and SAA samples. This unusual feature will require further examination.

DISCUSSION

In SA and SAA materials, the development of the dislocation microstructure appeared to be significantly retarded in helium preinjected samples. Even at the 25 dpa level, Frank loops were dominant. This retarding effect of dislocation development is difficult to understand if we assume that helium atoms exist in the same state in the preinjected samples and in the dual-ion irradiated samples. Bauer and Wilson [6] showed that helium-interstitial activation energies for motion were less than 1 eV and detrapping from substitutional sites results in activation energies larger than 2 eV, in general. The results of helium release as a function of annealing temperature showed that in helium preinjected samples, a major portion of the injected helium was at substitutional sites; this does not mean that the helium is not clustered. In dual-ion irradiated samples, helium atoms may be in substitutional sites more easily than in samples that are preinjected, because of the high vacancy concentrations produced by the heavy-ion irradiation. Here we will assume that isolated helium atoms in helium preinjected samples are interstitials and those in dual-ion irradiated samples are in substitutional sites. This would be likely to produce the lower number densities of larger bubbles (at similar He levels) in preinjected samples relative to dual-ion irradiated samples. The results obtained in this study suggest that there are significant differences in the diffusivity and distribution of helium atoms in materials preinjected with helium and in materials simultaneously helium injected.

The most significant feature of microstructure development in helium preinjected samples is that the cavity size distribution is bimodal in nature for all conditions investigated. This can be understood if the helium preinjection to 15 appm produces bubbles that are as large as or very close to the critical cavity size. As to the bubble formation by helium preinjection, hot-preinjection is likely to generate lower number densities of larger bubbles than cold-preinjection. Nevertheless, hot-preinjection could produce higher number densities of bubbles that are larger than the critical size for transition from pressure-driven to bias-driven growth than for cold-preinjection. This explanation agrees with the results obtained here for all three prior thermomechanical treatments; i.e., the average size and number density of large cavities are larger for hot-preinjected samples than for cold-preinjected samples. The lower swelling values in preinjected SA and SAA materials than those in dual-ion samples may be due to the low amount of injected helium (15 appm) compared with the 25D samples (375 appm). This is also supported by the fact that both the small-cavity and large-cavity number densities are lower in helium preinjected samples than in dual-ion irradiated samples.

Conversely, swelling was enhanced by helium preinjection rather than dual-ion irradiation in CW materials. The dual-ion results showed that almost all cavities were bubbles up to 25 dpa, therefore the swelling rate and swelling values were very low. On the other hand, in helium preinjected samples, bubble nucleation and growth apparently took place at the initial stage of single-ion irradiation to 25 dpa and a low density of cavities larger than the critical size for bias-driven growth grew rapidly, resulting in higher swelling values than that in dual-ion irradiated samples.

This result indicates that helium preinjection experiments are sensitive to the amount of helium injection, the injection rate, and the injection temperature as well as the microstructure of the irradiated materials. Therefore, one must be careful in using helium preinjection as a simulation of transmutant helium production.

CONCLUSIONS

1. The dislocation densities in helium preinjected samples were significantly lower than in dual-ion irradiated samples and the fractional contribution of Frank loops was much higher.
2. The cavity size distributions were bimodal in helium preinjected samples.
3. Helium preinjection retarded swelling relative to dual-ion irradiation in SA and SAA materials.
4. Helium preinjection enhanced swelling relative to dual-ion irradiation in CW material.
5. Swelling in preinjected CW samples was higher than in preinjected SA and SAA samples; hot-preinjection produced higher swelling than cold-preinjection.
6. Helium preinjection as a simulation of transmutant helium production should be used with caution.

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Table 1. Irradiation Conditions

	SOLUTION ANNEALED		SOLUTION ANNEALED AND AGED		20% COLD WORKED	
	Temp. (C)	dpa (dpa/sec)	Temp. (C)	dpa dpa/sec)	Temp.(C)	dpa (dpa/sec)
15 appm He cold-preinjected and 25 dpa Ni-ion	648.6	24.1 (3.1×10^{-3})	651.4	25.3 (3.2×10^{-3})	647.1	24.1 (3.1×10^{-3})
15 appm He hot-(650 C) preinjected and 25 dpa Ni-ion	627.8	23.6 (3.1×10^{-3})	642.9	24.9 (3.2×10^{-3})	640.7	23.3 (3.0×10^{-3})
25 dpa dual-ion with 15 appm He/dpa	617.5	23.5 (3.0×10^{-3})	621.6	24.7 (3.2×10^{-3})	623.1	23.7 (3.1×10^{-3})

FIGURE CAPTIONS

Fig. 1. Dislocation and cavity microstructure in 25 dpa samples of solution annealed material that were cold-preinjected, hot preinjected, or dual-ion irradiated

Fig. 2. Dislocation and cavity microstructure in 25 dpa samples of solution annealed and aged material that were cold-preinjected, hot preinjected, or dual-ion irradiated

Fig. 3. Dislocation and cavity microstructure in 25 dpa samples of 20% cold-worked material that were cold-preinjected, hot preinjected, or dual-ion irradiated

Fig. 4. Size Distribution of Cavities in Solution Annealed Type 316 SS (average cavity diameter: small cavities, open symbol; large cavities, filled symbol).

Fig. 5. Size Distribution of Cavities in Solution Annealed and Aged Type 316 SS (average cavity diameter: small cavities, open symbol; large cavities, filled symbol).

Fig. 6. Size Distribution of Cavities in 20% Cold-Worked Type 316 SS (average cavity diameter: small cavities, open symbol; large cavities, filled symbol).

Fig. 7. The Dependence of the Average Cavity Diameter on Irradiation Dose in Cold (room temperature: open symbols) and Hot (650 C: filled symbols) Helium Preinjected and Ion-Irradiated Type 316 SS.

Fig. 8. Dependence of Cavity Number Density on Irradiation Dose for Cold (blank mark) and Hot (filled mark) Helium Preinjected Type 316 SS.

Fig. 9. Dependence of Average Cavity Volume Fraction on Irradiation Dose for Hot (filled symbols)- or Cold (half-filled symbols)- Helium Preinjection Followed by Ion Irradiation and for Dual-Ion Irradiation (open symbols) of Type 316 SS.

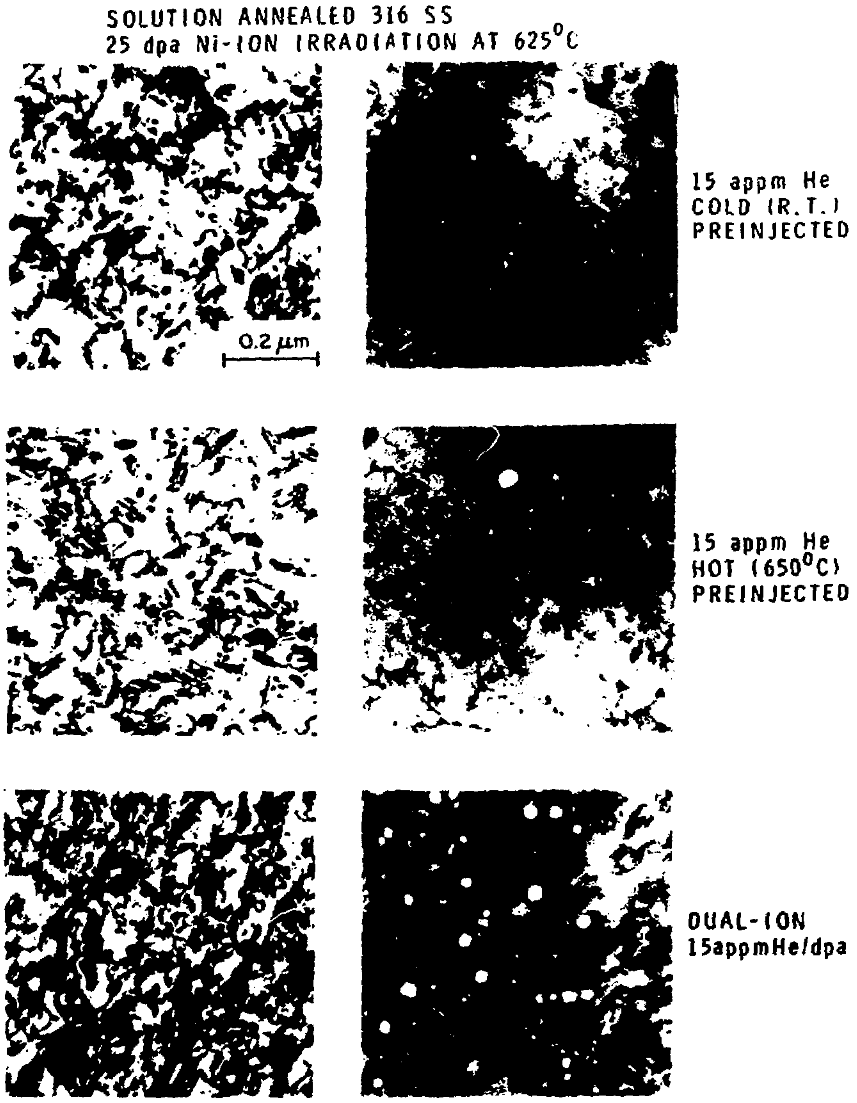


Fig. 1. Dislocation and cavity microstructure in 25 dpa samples of solution annealed material that were cold-preinjected, hot preinjected, or dual-ion irradiated

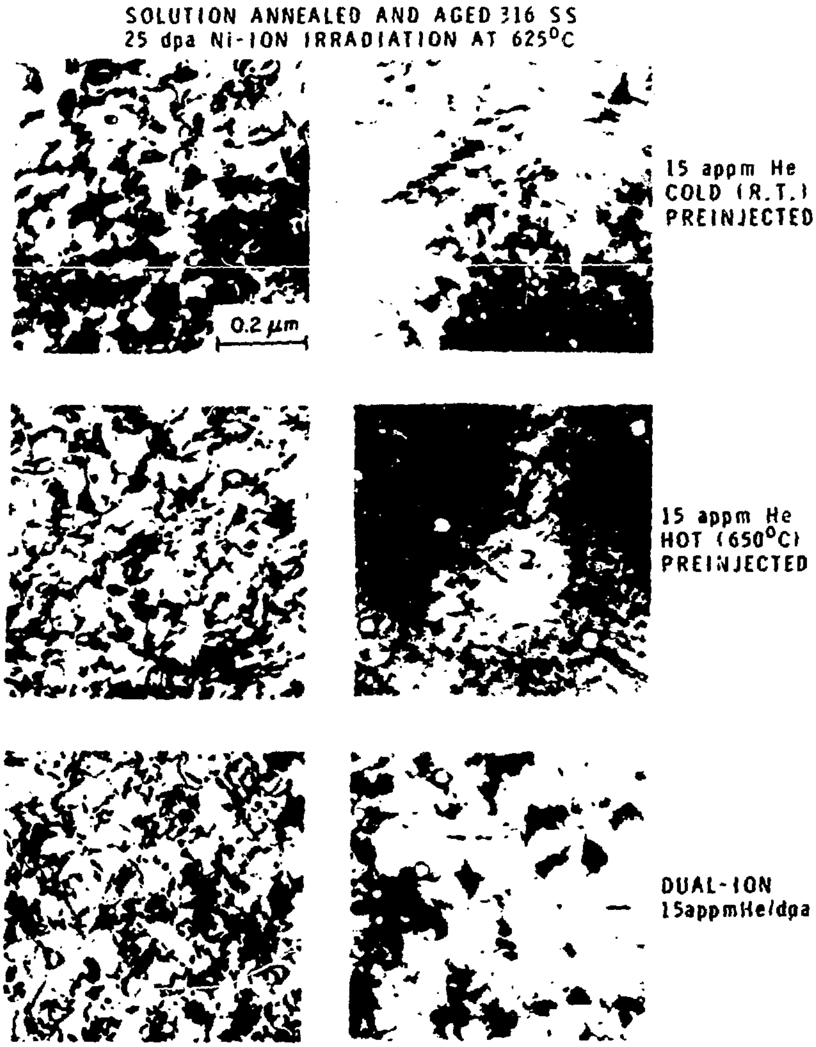


Fig. 2. Dislocation and cavity microstructure in 25 dpa samples of solution annealed and aged material that were cold-preinjected, hot preinjected, or dual-ion irradiated

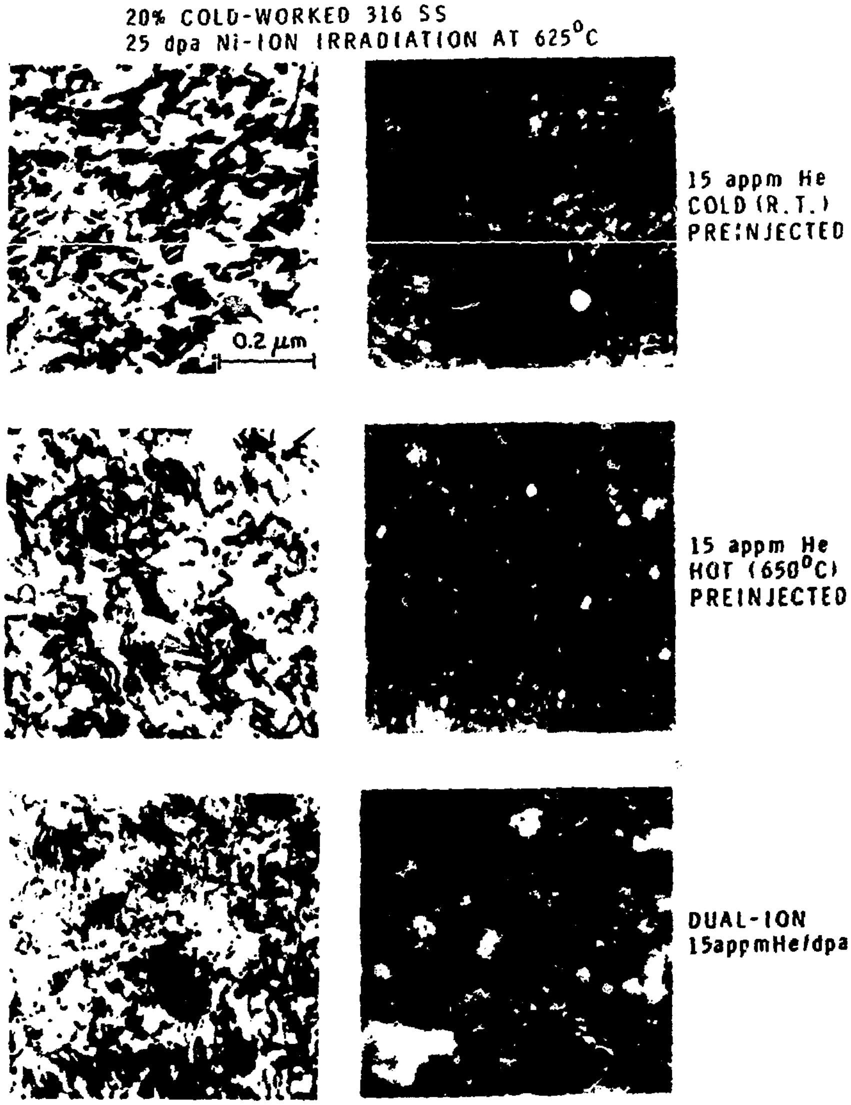
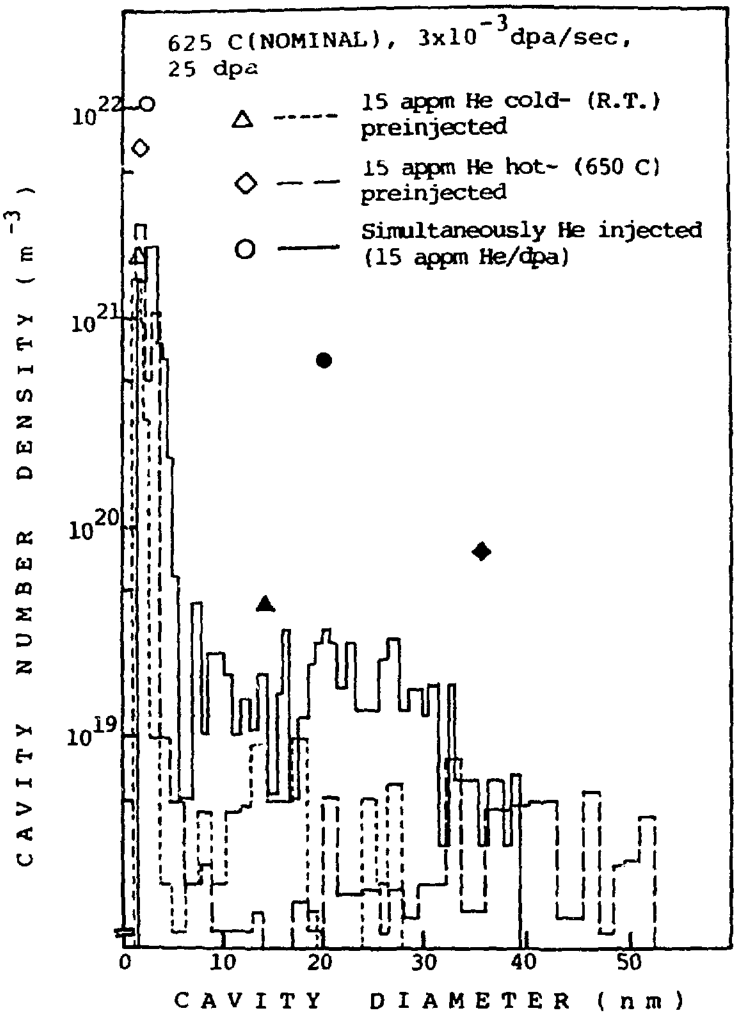
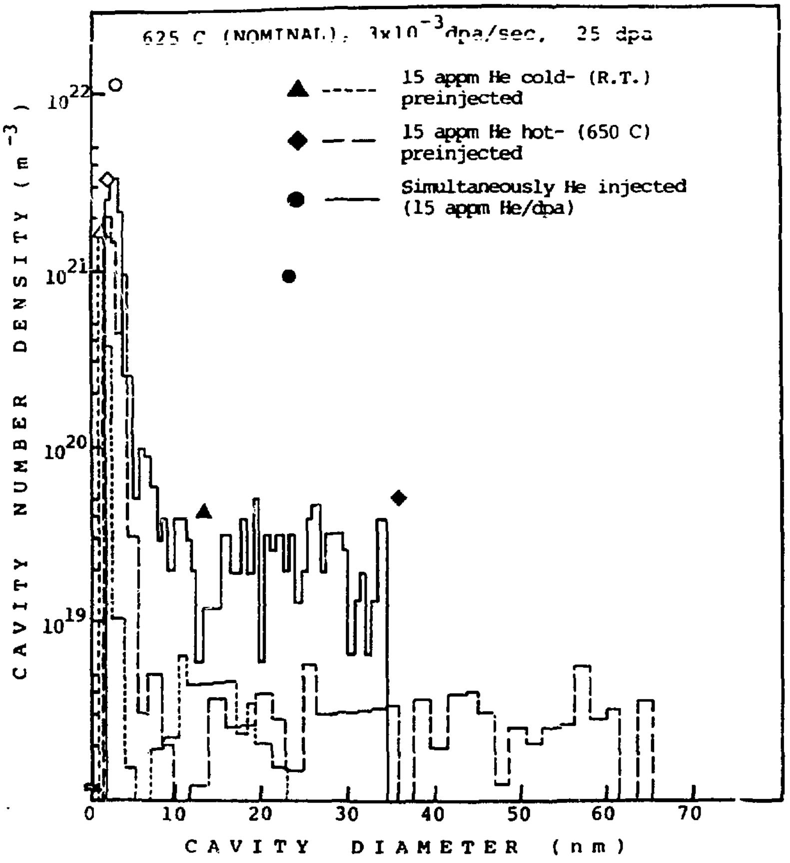


Fig. 3. Dislocation and cavity microstructure in 25 dpa samples of 20% cold-worked material that were cold-preinjected, hot preinjected, or dual-ion irradiated



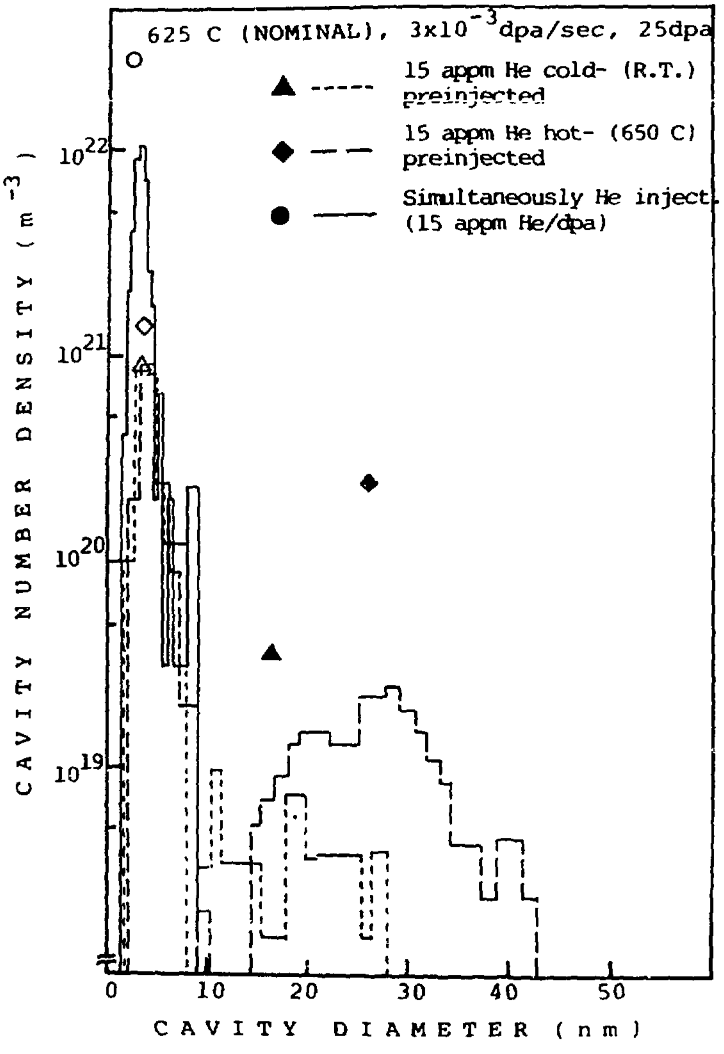
SIZE DISTRIBUTION OF CAVITIES IN SOLUTION ANNEALED Type 316 SS
(average cavity diameter: small cavities, open symbol; large cavities, filled symbol)

Fig. 4



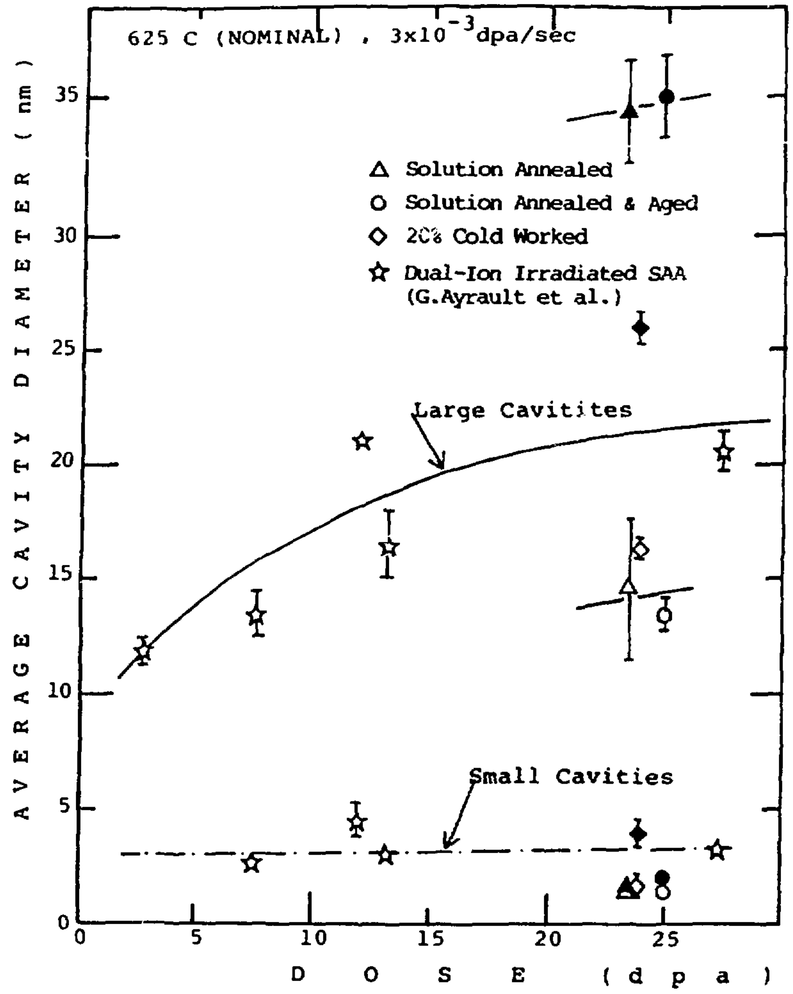
SIZE DISTRIBUTION OF CAVITIES IN SOLUTION ANNEALED AND AGED Type 316 SS
(average cavity diameter: small cavities, open symbol; large cavities, filled symbol)

Fig. 5



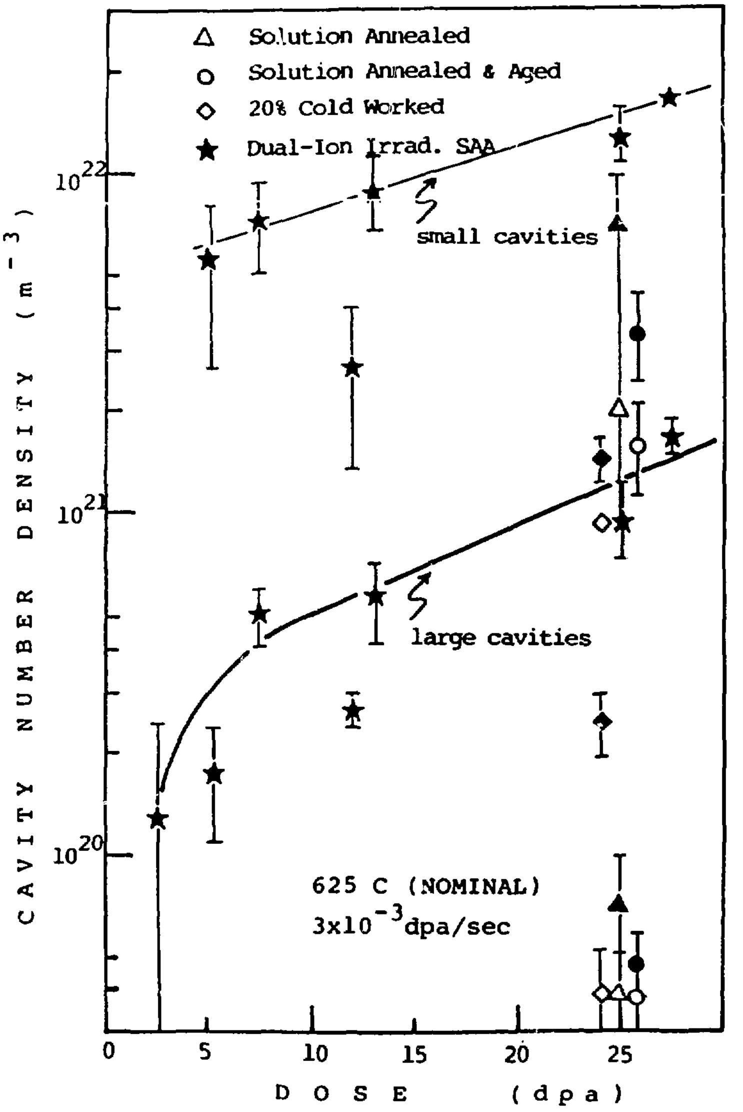
SIZE DISTRIBUTION OF CAVITIES IN 20% COLD WORKED Type 316 SS
(average cavity diameter: small cavities, open symbol; large cavities, filled symbol)

Fig. 6



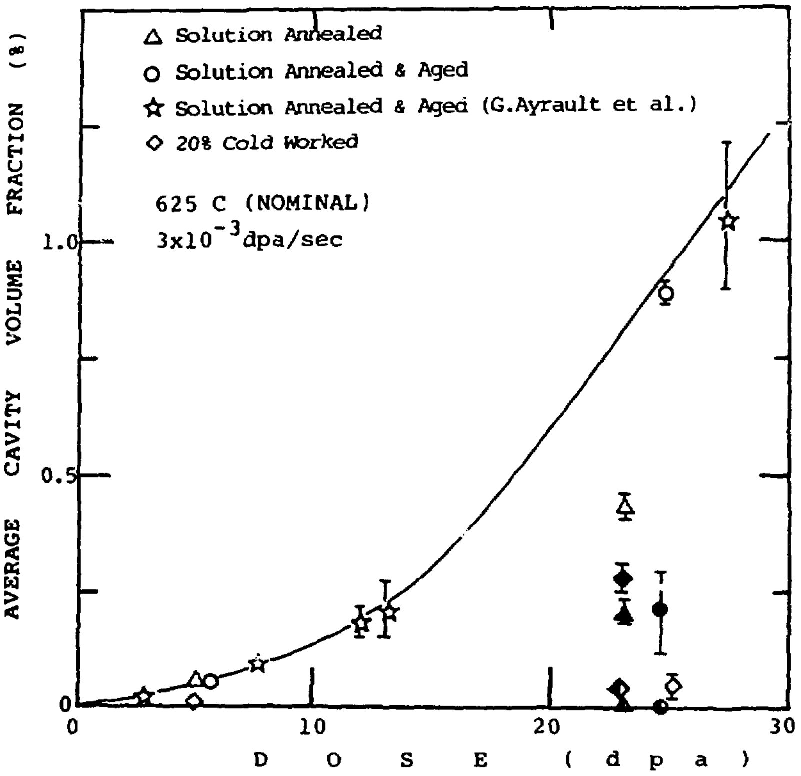
THE DEPENDENCE OF AVERAGE CAVITY DIAMETER ON IRRADIATION DOSE IN COLD(room temperature:blank mark) AND HOT(650 C:filled mark) He PREINJECTED AND ION IRRADIATED Type 316 SS

Fig. 7



DEPENDENCE OF CAVITY NUMBER DENSITY ON IRRADIATION DOSE FOR COLD (blank mark) and HOT (filled mark) He PREINJECTED Type 316 SS

Fig. 8



DEPENDENCE OF AVERAGE CAVITY VOLUME FRACTION ON IRRADIATION DOSE FOR HOT(filled mark)- or COLD (half-filled mark)- HELIUM PREINJECTION FOLLOWED BY ION IRRADIATION AND FOR DUAL-ION IRRADIATION (blank mark) OF Type 316 SS

Fig. 9