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SWELLING
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MECHANICAL PROPERTY CHANGES
IN
NEUTRON-IRRADIATED COLD-ROLLED
TYPE 316 STAINLESS STEEL

ERDA Research and Development Report

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LMFBR FUELS AND MATERIALS
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IN
NEUTRON-IRRADIATED COLD-ROLLED
TYPE 316 STAINLESS STEEL

By

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ABSTRACT

Samples of cold-rolled Type 316 stainless steel were irradiated in EBR-II to a fluence of about 8 x 10^{26} n/m^2 (E >0.1 MeV) at 500 and 600°C. Three sample configurations were used - small sheet tensile samples, small right-circular cylinders for immersion density, and thin foils for transmission-electron microscopy (TEM). TEM revealed voids in the foils irradiated at both temperatures. Immersion density results, however, indicated swelling only in the sample irradiated at 600°C. Considerable recovery and precipitation were observed in foils irradiated at both temperatures. Results of tensile tests on irradiated samples showed a decrease in yield strength and an increase in ductility compared to unirradiated controls.

I. INTRODUCTION

High fluence swelling and mechanical properties data for cold-rolled Type 316 stainless steel is needed to provide data for design equations so that extrapolations to LMFBR goal fluence levels can be made with more confidence. The work reported here is a continuation of the EBR-II X100 irradiation experiment and extends these observations to a fluence of about 8×10^{26} n/m^2 (E >0.1 MeV).

TABLE 1

COMPOSITION OF TYPE 316 STAINLESS STEEL SAMPLES

Sample Configuration	Pre-Irradiation Treatment	Composition (wt %)										
		C	Mn	P	S	Si	Ni	Cr	Cu	Mo	Co	Fe
Tensile	1120°C/30 min + 25% CR*	0.06	1.68	0.022	0.017	0.51	13.63	17.33	0.075	2.32	0.01	Balance
Cylinder	1120°C/30 min + 25% CR	0.037	1.51	0.021	0.015	0.42	11.56	16.65	0.13	2.39	0.13	Balance
Foil	980°C/1 hr + 20% CR	0.050	1.72	0.018	0.023	0.51	13.70	17.86	--	2.32	--	Balance

*CR = Cold rolled

TABLE 2

IRRADIATION INITIAL AND FINAL TEMPERATURE

Pin	Initial Temperature (°C)	Final Temperature (°C)
A	500	530
B	600	620
C	500	500
D	600	535
E	500	545
F	600	600

II. EXPERIMENTAL

Small sheet tensile samples (0.23 mm by 1.02 mm, with a gage length of 12.7 mm), small right-circular cylinders (3.8 mm diameter by 2.8 mm), and foils (3.7 mm square by 0.013 mm) of Type 316 stainless steel were irradiated in EBR-II Subassemblies X100 and X100A. The composition and pre-irradiation treatment of each type of sample are given in Table 1. These are the same alloy heats and pre-irradiation treatments as those reported earlier for Pins A and B of Subassembly X100.(1)

Data reported here are for samples irradiated in Pins C, D, E, or F to a fluence of about 8.8×10^{26} n/m^2 total with about 8×10^{26} n/m^2 (E >0.1 MeV). These fluences are tentative pending calculations by the Fast Reactor Materials Dosimetry Center (FRMDC) at HEDL. The initial irradiation temperatures and the final temperatures(2) for each pin are given in Table 2. Also included in Table 2 are the temperatures for Pins A and B.

Percent swelling was determined from immersion density measurements* on the right-circular cylinders, and calculated from void size and concentration data for foil samples. Immersion densities for the unirradiated cylindrical samples were obtained from archive samples. Void size and concentrations in the foil samples were obtained from electron photomicrographs using a Zeiss Particle-Size Analyzer.

Tensile testing was done in a vacuum ($\sim 7 \times 10^{-3}$ Pa) at a strain rate of 3.3×10^{-4} sec^{-1}. Samples were tested at either 525 or 575°C, depending on their irradiation temperature. There was a 15-min hold prior to testing to allow for temperature stabilization.

Helium analysis was determined by mass spectrometry(3) at Atomics International (AI) on pieces cut from the grip ends of tensile samples. The analysis showed 8.6 appm for samples irradiated to 3.8×10^{26} n/m^2 and 17.8 appm for samples irradiated to about 8×10^{26} n/m^2. The fluence of 3.8×10^{26} n/m^2 was calculated at the FRMDC.(4) Fluences quoted in this report are for E >0.1 MeV, except where noted.

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*Density measurements were made at ANL's Idaho Facility, Analytical Laboratory.

TABLE 3

IMMERSION DENSITY AND TEM SWELLING RESULTS

Pin	Sample Type	T_i* (°C)	T_f† (°C)	Fluence (n/m^2, E >0.1 MeV)	$(\rho_o - \rho_i)/\rho_o$ (%)	$\Delta V/(V-\Delta V)$ (%)	ρ_V (V/m^3)	$\bar{d}$ (nm)
A	Cylinder	500	530	3.8×10^{26}	-0.1 ± 0.2			
B	Cylinder	600	620	3.8×10^{26}	+0.1 ± 0.2			
C	Cylinder	500	500	$\sim 8 \times 10^{26}$	+0.1 ± 0.2			
D	Cylinder	600	535	$\sim 8 \times 10^{26}$	-1.0 ± 0.2			
E	Cylinder	500	545	$\sim 8 \times 10^{26}$	+0.0 ± 0.2			
F	Cylinder	600	600	$\sim 8 \times 10^{26}$	-3.3 ± 0.2			
A	Foil	500	520	3.8×10^{26}		-0.01 ± 0.01	4×10^{19}	15.4
B	Foil	600	630	3.8×10^{26}		-0.1 ± 0.03	2×10^{19}	31.7
C	Foil	500	500	$\sim 8 \times 10^{25}$		-2.0 ± 0.7	3.8×10^{20}	39.5
F	Foil	600	600	$\sim 8 \times 10^{25}$		-7 ± 2	6.4×10^{20}	53.0

*T_i = Initial temperature
†T_f = Final temperature

III. RESULTS AND DISCUSSION

A. SWELLING

Swelling results from immersion density and TEM measurements are summarized in Table 3. The errors associated with the immersion-density results are one standard deviation, while those for the TEM results are ±30%, due to foil thickness uncertainties.

Garner et al.,(5) folded the immersion density data for the high fluence samples irradiated at 500 and 600°C, Pins C and F, into their revised design correlation equation. They found that the 600°C data is best represented by taking a shorter incubation period for the onset of swelling compared to the other data; the 500°C data fit the revised equation without modification.

Swelling data for samples cold rolled 20 to 27% and irradiated to a fluence greater than 7.0 x 10^{26} n/m^2 have been plotted in Figure 1. Data from Subassemblies X098, X100, and X157 above 600°C appear to be in good agreement and sufficient to define the swelling curve in this region. Below 600°C there are

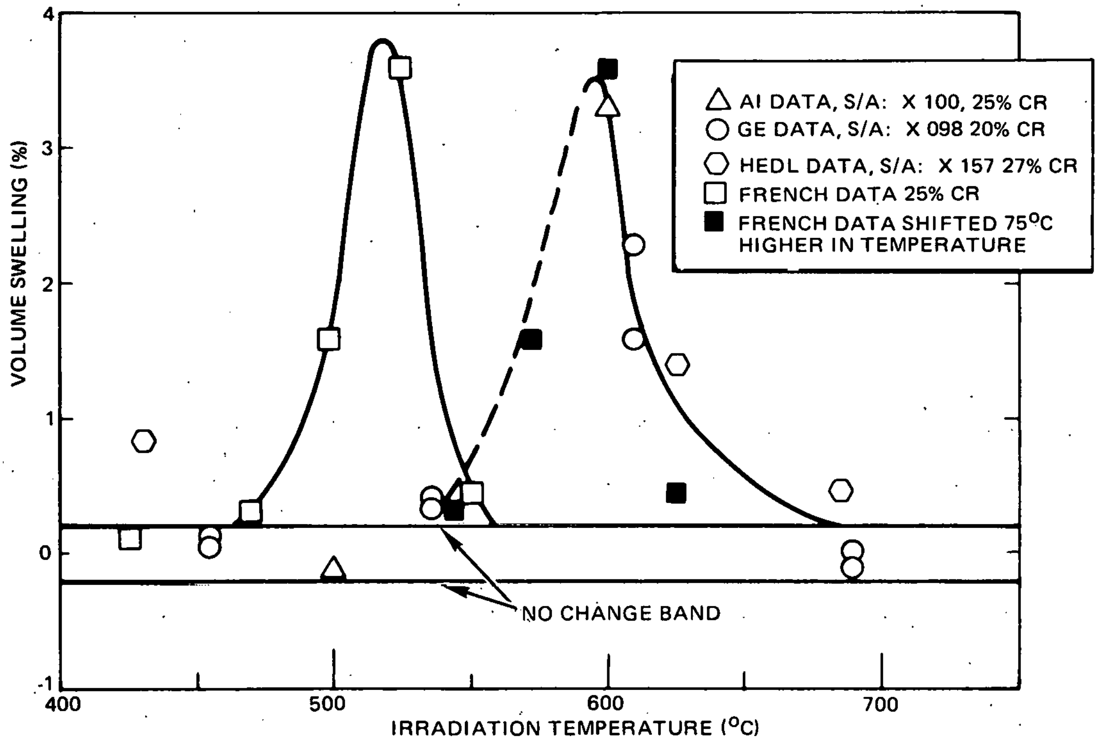

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Figure 1. Swelling vs Irradiation Temperature for Type 316 Stainless Steel, 20 to 27% Cold-Rolled and Irradiated to a Fluence >7 x 10^{26} n/m^2 (E >0.1 MeV)

insufficient data to interpret the shape of the curve and it is, therefore, shown as dashed in that region. The French data, obtained from Dupouy,(6) are peaked about 75°C lower in temperature than the U.S. data. This discrepancy is probably due to differences in the models and gamma heating rates used to calculate the temperature profile in a subassembly. Although it cannot be stated which data are correct, it was decided to arbitrarily shift the French data 75°C higher in temperature (shown as solid symbols) for comparison with the U.S. data. The correlation is quite good. From the available U.S. data, the peak swelling would be placed between 590 and 600°C.

The data from samples irradiated in Pins D and E are not included here because of the large temperature shift during irradiation. However, it should be noted that the sample in Pin D, which had a large temperature decrease, had a swelling of 1.0%, whereas the sample in Pin E, which had a temperature increase, did not show any change in density. Assuming that (1) the samples were always between their initial temperatures and their final temperatures, and (2) the change in temperature was a function of the swelling of the capsule and/or surrounding subassembly components, then the results can be explained with reference to Figure 1. The Pin D sample was always in a temperature region conducive to swelling, 600 to 535°C. The sample in Pin E, however, probably remained in the region indicating no change for most of its radiation history, and what small increment of swelling due to voids that may have taken place was only sufficient to offset densification caused by precipitation.

B. METALLOGRAPHY

Metallographic observations for samples irradiated to 3.8×10^{26} n/m^2 were reported earlier.(1) Irradiation to the higher fluence of about 8×10^{26} n/m^2 resulted in considerably more recovery and precipitation. The precipitates that were examined by selected area diffraction were identified as $M_{23}C_6$ type. Although considerable recovery occurred in both samples irradiated to the higher fluence, no obvious cell structure was evident after irradiation at 500°C. After irradiation at 600°C, however, polygonization and cell walls were evident. Some grain boundary migration was evident in all the irradiated samples. Although rare, small helium bubbles were observed in the sample irradiated at 600 to 620°C to 3.8×10^{26} n/m^2;(1) no helium bubbles were observed in the sample irradiated to about 8 x 10^{26} n/m^2 at 600°C. These observations of helium bubbles are in good agreement with the work of Brager and Straalsund.(7)

Void formation after irradiation at 500°C to a fluence of about 8 x 10^{26} n/m^2 occurs primarily between the remains of original deformation bands, and is still somewhat inhomogeneous. After irradiation at 600°C to the same fluence, the voids approach a homogeneous distribution. This is probably due to the greater degree of recovery that has taken place in the sample. Figure 2 shows the pre-irradiation microstructure of a foil sample while Figures 3 to 6 show the microstructure following irradiation at 500 or 600°C to a fluence of 3.8 x 10^{26} n/m^2 and about 8 x 10^{26} n/m^2.

C. TENSILE TESTS

Results of the tensile tests are shown in Table 4. Also included are results of samples irradiated to a fluence of 3.8 x 10^{26} n/m^2.(1) These results indicate that irradiation in the temperature region of 500 to 600°C leads to a reduction in both the yield and ultimate strengths and an increase in both the uniform and total elongations.

Fahr et al.,(8) and Fish et al.,(9) have reported similar observations on irradiated 20% cold-rolled Type 316 stainless steel tested in the same temperature region. Both also reported that the yield strength of samples that were cold rolled and aged, but not irradiated, was essentially that of the cold-rolled and irradiated samples.

Metallographic examination of 20% cold-rolled foils, irradiated in the same capsules as the tensile samples, revealed an obvious loss of cold-rolled structure in all cases except those irradiated at 500°C to a fluence of 3.8 x 10^{26} n/m^2. Thus, the reduction in strength appears to be due to thermal processes, independent of irradiation, in the temperature range of 500 to 600°C.

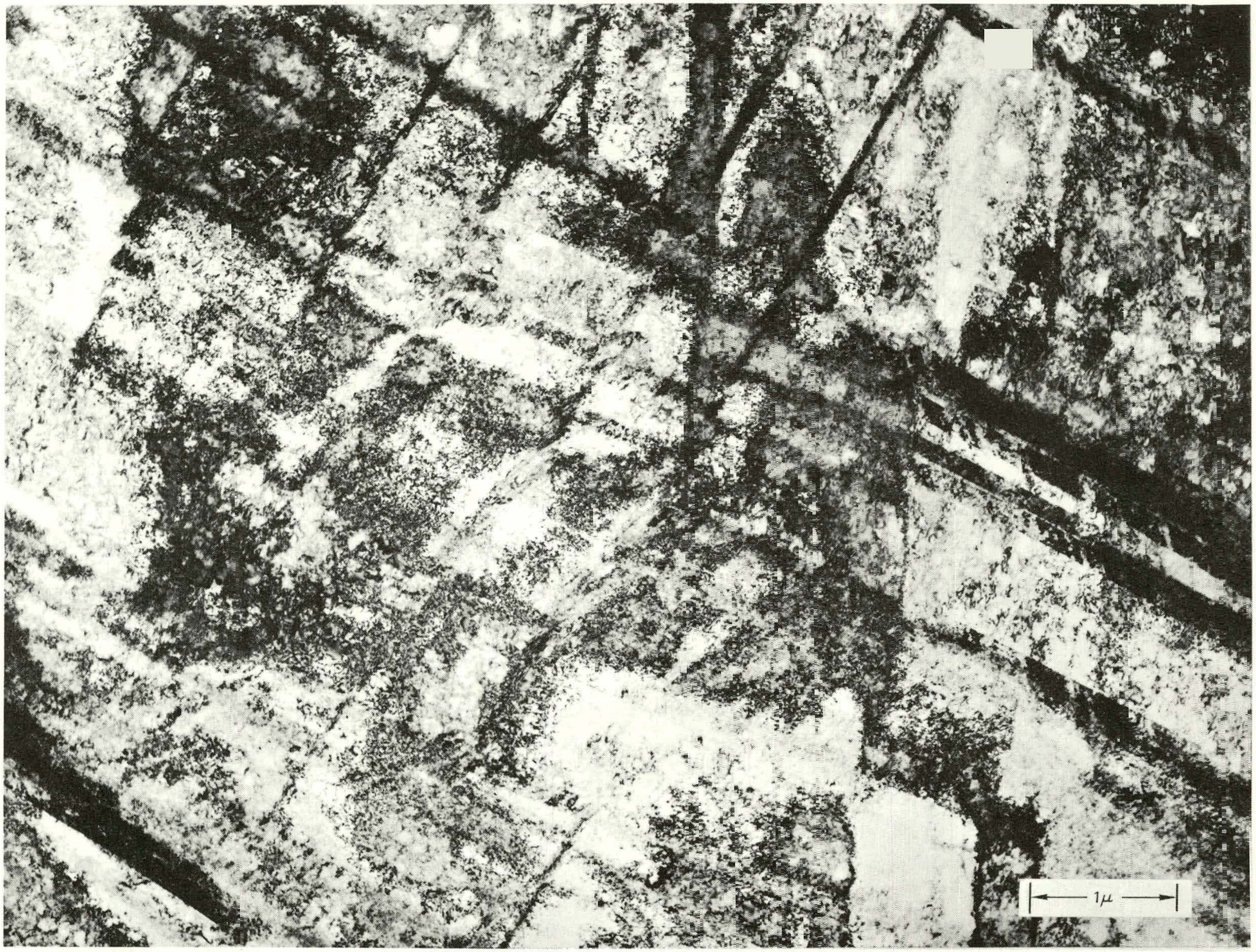

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Figure 2. Pre-irradiation Microstructure of 20% Cold-Rolled Type 316 Stainless Steel

18-279

Figure 3. Microstructure of 20% Cold-Rolled Type 316 Stainless Steel after Irradiation at 500 to 530°C to a Fluence of 3.8×10^{26} n/m^2 (E >0.1 MeV)

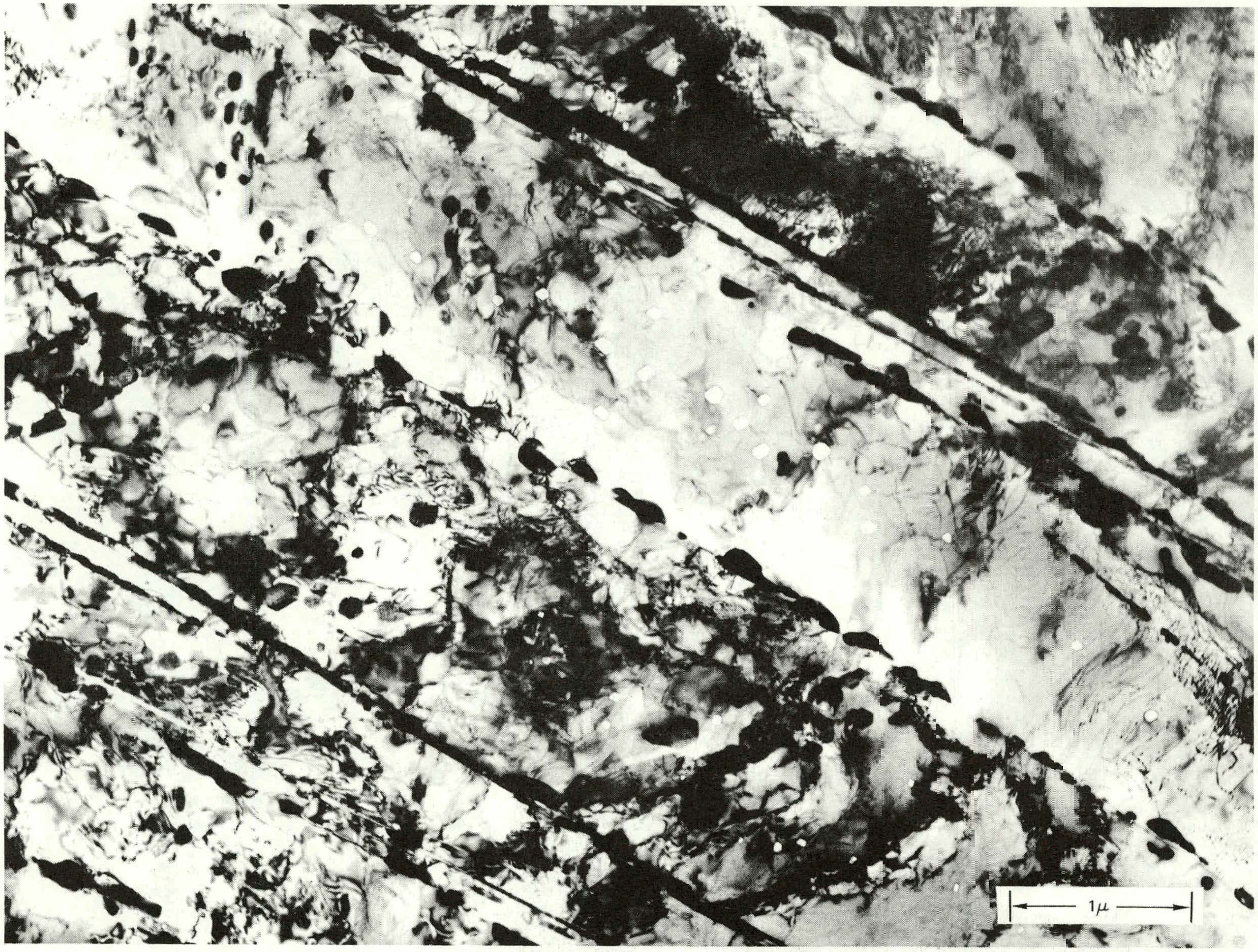

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Figure 4. Microstructure of 20% Cold-Rolled Type 316 Stainless Steel after Irradiation at 600 to 620°C to a Fluence of 3.8×10^{26} n/m^2 (E >0.1 MeV)

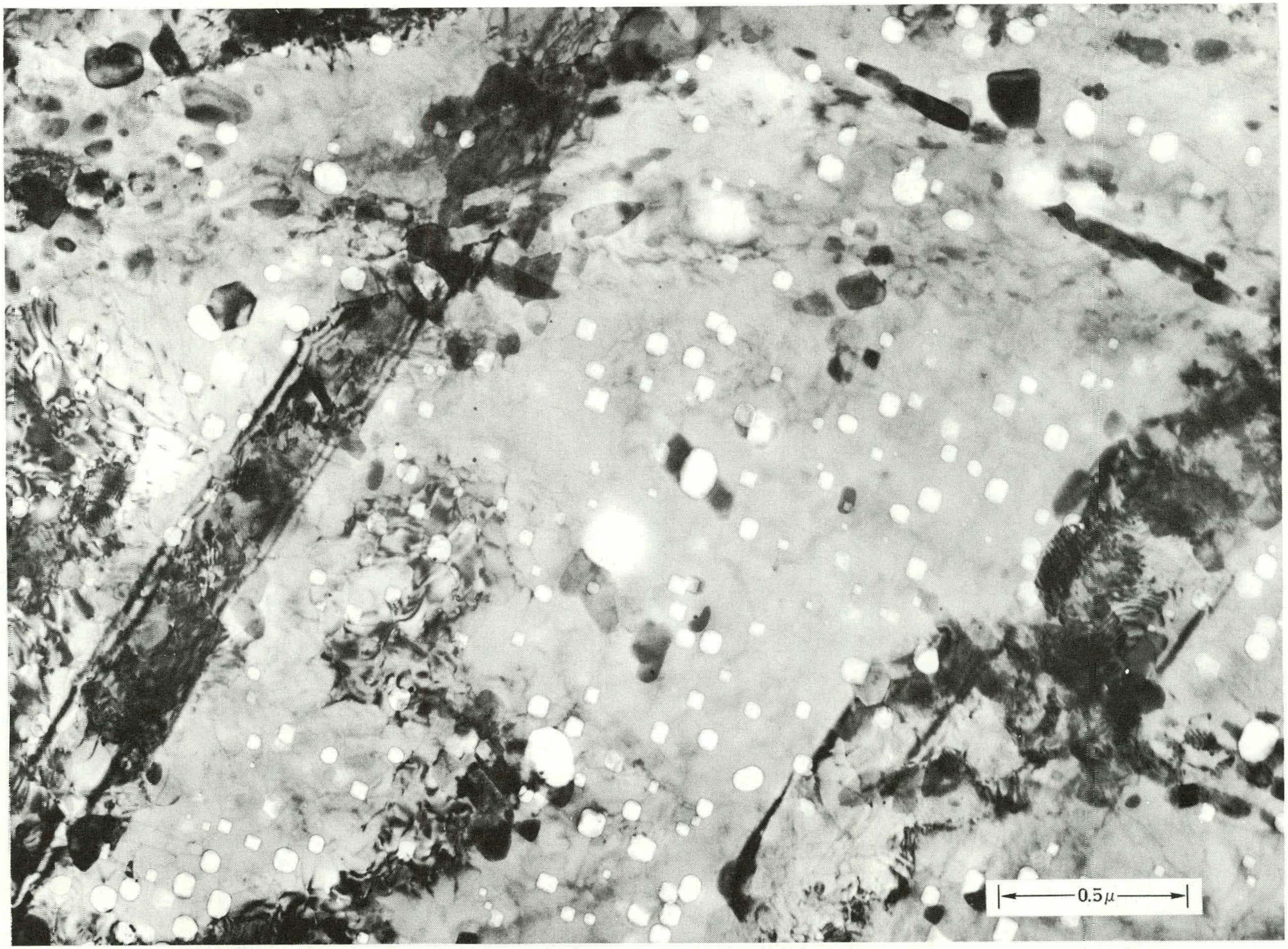

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Figure 5. Microstructure of 20% Cold-Rolled Type 316 Stainless Steel after Irradiation at 500°C to a Fluence of $\sim 8 \times 10^{26} n/m^2$ (E>0.1 MeV)

10-369

Figure 6. Microstructure of 20% Cold-Rolled Type 316 Stainless Steel after Irradiation at 600°C to a Fluence of ~8 x 10^{26} n/m^2 (E>0.1 MeV)

TABLE 4

SUMMARY OF TENSILE DATA FOR 25% COLD-ROLLED TYPE 316 STAINLESS STEEL

Sample	Fluence (E >0.1 MeV) x 10^{26} (n/m^2)	Irradiated Temperature (°C)	Test Temperature (°C)	Strength		Elongation	
				Yield (MPa)	Ultimate (MPa)	Uniform (%)	Total (%)
Control	--	--	500	825	863	2.9	3.1
Irradiated	3.8	500-530	500	595	667	3.2	3.6
Control	--	--	600	698	756	2.6	2.9
Irradiated	3.8	600-620	600	438	519	4.5	5.0
Control	--	--	525	796	831	2.6	2.9
Irradiated	~8	500-545	525	463	547	5.0	5.4
Control	--	--	575	719	786	2.6	2.8
Irradiated	~8	600-535	575	407	478	4.0	4.3

IV. CONCLUSIONS

Significant amounts of recovery occur in 20% cold-rolled Type 316 stainless steel irradiated at 500 or 600°C to a fluence of about 8 x 10^{26} n/m^2 (E >0.1 MeV).

Swelling occurred in the samples irradiated at 600°C while none occurred in the samples irradiated at 500°C, as measured by immersion density.

Voids were observed in samples irradiated at both temperatures.

Irradiation at 500 to 545°C and 600 to 535°C results in a reduction in yield strength and an increase in ductility.

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