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NOTCH AND HYDROGEN EFFECTS ON SENSITIZED 21-6-9 STAINLESS STEEL

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ABSTRACT

Type 21-6-9 stainless steel alloy is slightly notch sensitive in the solution annealed condition, a behavior that is aggravated by sensitization anneal at 920 K. The lower toughness of the sensitized alloy is a measure of microstructural embrittlement associated with carbide precipitation in grain boundaries. The tendency toward grain boundary fracture in the sensitized alloy is accentuated by stress concentration at the notch. Also, there is an increase in notch sensitivity when the alloy is tested in a high pressure (69 MPa) hydrogen environment, due to susceptibility of the grain boundaries to hydrogen embrittlement.

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INTRODUCTION

Intergranular fracture is commonly associated with either a precipitate or segregation of an impurity in the grain boundary (1). The grain boundary region is therefore weakened with respect to the bulk grain, thus providing an easier fracture path. Often fracture initiates by decohesion of the precipitate-alloy interface or by cracking of the precipitate particles. Sensitization of austenitic stainless steels can lead to grain boundary failure through formation of chromium-rich carbides of the general form $M_{23}C_6$ (2). The possible relation of this compositional modification to hydrogen-assisted fracture of austenitic stainless steel is of interest, as the chromium-depleted boundary region might be expected to be more susceptible to hydrogen damage. Hydrogen-assisted cracking of sensitized Type 309S and Type 304 stainless steel alloys has been examined with differing results (3,4). Hydrogen compatibility of Type 309S stainless steel alloy was unchanged by sensitization, whereas sensitized Type 304 stainless steel alloy was more susceptible to hydrogen damage then solution-annealed material.

The current study extends the investigation of sensitization effects on hydrogen-assisted cracking to a stable but more complex austenitic steel alloy, Type 21-6-9 steel or *Nitronic* 40.* Solution-annealed and sensitized specimens from a single heat were tested in high-pressure hydrogen environments or following gas-phase charging at high pressure.

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*Trademark, Armco Steel Company.

MECHANICAL TESTS

Tensile specimens were machined from bar stock of a single heat of 21-6-9 alloy with the analysis shown in Table I. Smooth bar specimens had a nominal diameter of 0.48 cm with a 2.5-cm gauge-length. A stress concentration factor of about 3 was attained in several specimens by machining a V-notch with a 0.013-cm root radius and 30° flank angle to a depth of 0.1 cm. Both solution-annealed and sensitized specimens were tested. The sensitization anneal was at 920 K with either a 2 or 24 hr treatment. Microstructures of the alloy are shown in Figure 1. Grain boundary ditches or grooves are evident in the sensitized samples. Tensile tests were at 200 or 300 K in air or at 300 K in high-pressure (69 MPa) helium or hydrogen.

DISCUSSION

Notch Effect

An external notch reduces the toughness and strength of 21-6-9 alloy; the notch bar tensile strength is slightly less than the smooth bar tensile strength (Table I). The overall fracture is flat rather than cup-cone due to the constraint of the notch. Fracture mode remains dimpled (microvoid coalescence), but dimple sizes appear larger on the average in the notched specimen (Figure 2).

Sensitization Effect

The sensitization treatment reduces both the strength and ductility of 21-6-9 alloy tested in air (Table II) and introduces grain boundary fracture. Smooth and notched specimens differ in appearance (Figure 3). The fracture path in a smooth specimen is dimpled and is possibly transgranular in the transverse plane and along grain boundaries in a direction parallel to the tensile axis. In contrast, the fracture path in a notched specimen is nearly wholly along grain boundaries. This difference is evident at 300 K as well as at 200 K (Figures 2 and 3).

Tests in High-Pressure Hydrogen Environments

The tensile properties of Type 21-6-9 stainless steel alloy, when tested in either high pressure helium or hydrogen environments, were not changed significantly by the sensitization treatment (Table III). However, the specimens exposed to hydrogen exhibited modified fractures and displayed some grain boundary cracking (Figure 4). The fracture surface branches along grain boundaries and shows a rough irregular appearance, in contrast to the relatively smooth cup-cone failure seen in solution annealed specimens (Figure 4). The transition to grain boundary failure is nearly complete if an external notch is present on a sensitized specimen tested in a hydrogen environment (Figure 5).

Hydrogen Saturated Specimens

The sensitization heat treatment in combination with super-saturation of the alloy with hydrogen degraded the mechanical properties of smooth bar tensile specimens of Type 21-6-9 stainless steel more than either factor alone (Table IV). Of itself, hydrogen introduced by exposure of the test specimens for several weeks at 620 K to 69 MPa hydrogen gas increased the tensile strength of Type 21-6-9 alloy and decreased its ductility. In contrast, a loss of strength and a further reduction in ductility occurred when the alloy was sensitized prior to hydrogen exposure.

A distinct grain-boundary fracture path with patches of dimpled failure can be seen in the sensitized specimen shown on Figure 6. Grain boundary cracks are visible on longitudinal sections in areas away from the fracture face. The proportion of the fracture following grain boundaries is obviously greater in the hydrogen-charged specimen than in the specimen tested without exposure to hydrogen. Figure 6 also shows that fracture of the sensitized 21-6-9 alloys is not the same as for the solution-annealed 21-6-9 alloy test specimen. In the latter case, fracture is mixed with isolated facets in a matrix of dimpled fracture. Markings on the facets appear to arise from slip traces intersecting the face. Grain faces of the sensitized specimens are different; the patterns suggest microvoid coalescence on a fine scale or flute-shaped voids and ridges.

Grain faces in sensitized Type 21-6-9 steel alloy appear to be enriched in chromium and manganese relative to dimpled regions in the solution annealed alloy. In addition, there is evidence of increased aluminum and silicon concentration at grain faces. The relative chemical compositions of grain faces and dimpled areas were estimated from the intensities of characteristic x-rays measured with an energy-dispersive X-ray spectrometer on the scanning electron microscope.

SUMMARY

Sensitization of Type 21-6-9 stainless steel alloy at 920 K increased the susceptibility to hydrogen damage relative to the solution-annealed alloy. In this regard, Type 21-6-9 stainless steel is similar to Type 304 stainless steel, but differs from Type 309S stainless steel alloy which is unaffected.

Type 21-6-9 stainless steel test specimens were slightly notch sensitive in the solution-annealed condition, a behavior that was aggravated by sensitization.

Sensitization increased the extent of hydrogen damage resulting from gas-phase charging at 69 MPa hydrogen pressure. The cause for increased susceptibility must be associated with microstructural and compositional alterations at the grain boundaries, but the mechanism is undefined as yet.

Sensitization induced grain boundary failure which was further enhanced by exposure to a hydrogen environment.

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TABLE I

Analyses of Type 21-6-9 Stainless Steel Alloy

Element	*wt %*	*Element*	*wt %*
Cr	20.20	C	0.034
Ni	7.15	N	0.30
Mn	9.00	P	0.024
Mo	0.20	Si	0.74
Cu	0.31	S	0.005

TABLE II

Mechanical Properties of Type 21-6-9 Alloy: Heat Treatment and Notch Effects

Test Temp., K[a]	*Heat Treatment*	*Specimen*	*Strength, MPa*		*Elongation, %*		*Plastic Strain*
			Yield	*Ultimate*	*Uniform*	*Total*	
300	Solution Anneal	Smooth bar	700	1170	41	51	1.59
		Notch bar	800	1160	24	27	0.74
	Sensitize	Notch bar	750	1070	18	18	0.53
200	Solution Anneal	Smooth bar	880	1550	48	58	1.57
		Notch bar	1130	1500	19	19	0.72
	Sensitize	Smooth bar	720	1490	51	60	1.03
		Notch bar	1120	1250	10	10	0.17

a. Air environment.

TABLE III

Mechanical Properties of Sensitized Type 21-6-9 Stainless Steel Alloy Tested in a High-Pressure Hydrogen Environment at Room Temperature

Specimen Condition	*Test Atmosphere*	*Strength, MPa*		*Elongation, %*		*Plastic Strain to Failure*
		Yield	*Ultimate*	*Uniform*	*Total*	
Solution Annealed	69 MPa He	650	1050	42	52	1.11
	69 MPa H_2	670	1060	41	50	1.22
920K-2 hr	69 MPa He	640	1100	43	50	1.51
	69 MPa H_2	640	1080	42	49	1.50
920K-24 hr	69 MPa He	625	1110	46	53	1.38
	69 MPa H_2	620	1100	46	52	1.10
920K-24 hr	69 MPa He[a]	760	1060	16	16	0.32
	60 MPa H_2[a]	700	760	9	9	0.06

a. Notch bar specimen.

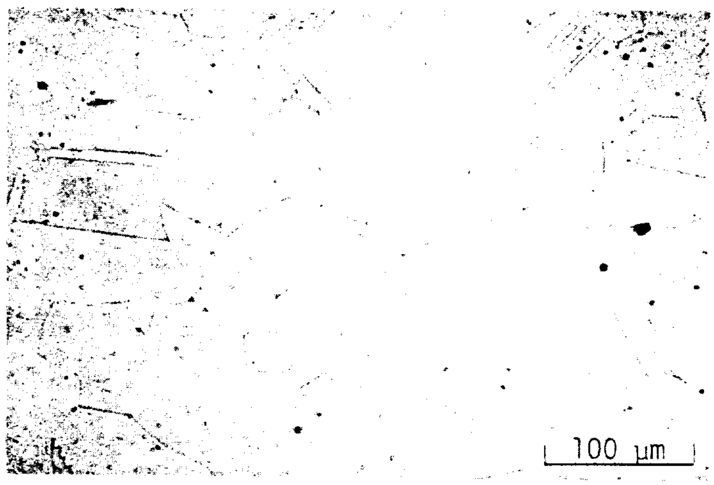
TABLE IV

Mechanical Properties of Sensitized Type 21-6-9 Stainless Steel Alloy Saturated with Hydrogen

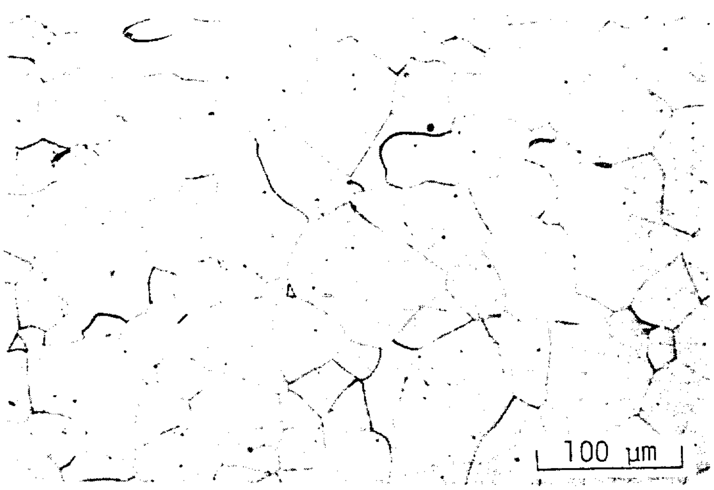
Temp., K	*Treatment*[a]	*Exposure*	*Strength, MPa*		*Elongation, %*		*Plastic Strain to Failure*
			Yield	*Ultimate*	*Uniform*	*Total*	
200	Solution Anneal	None	970	1550	48	58	1.57
	Solution Anneal	69 MPa H_2	1060	1650	44	48	0.66
	920K-24 hr	None	790	1490	51	60	1.03
	920K-24 hr	60 MPa H_2	920	1470	37	37	0.33
	920K-24 hr	69 MPa H_2[b]	900	1350	35	40	0.45

a. Smooth bar tensile specimens.

b. Crosshead speed, 0.5 cm/sec; all others, 0.05 cm/sec.

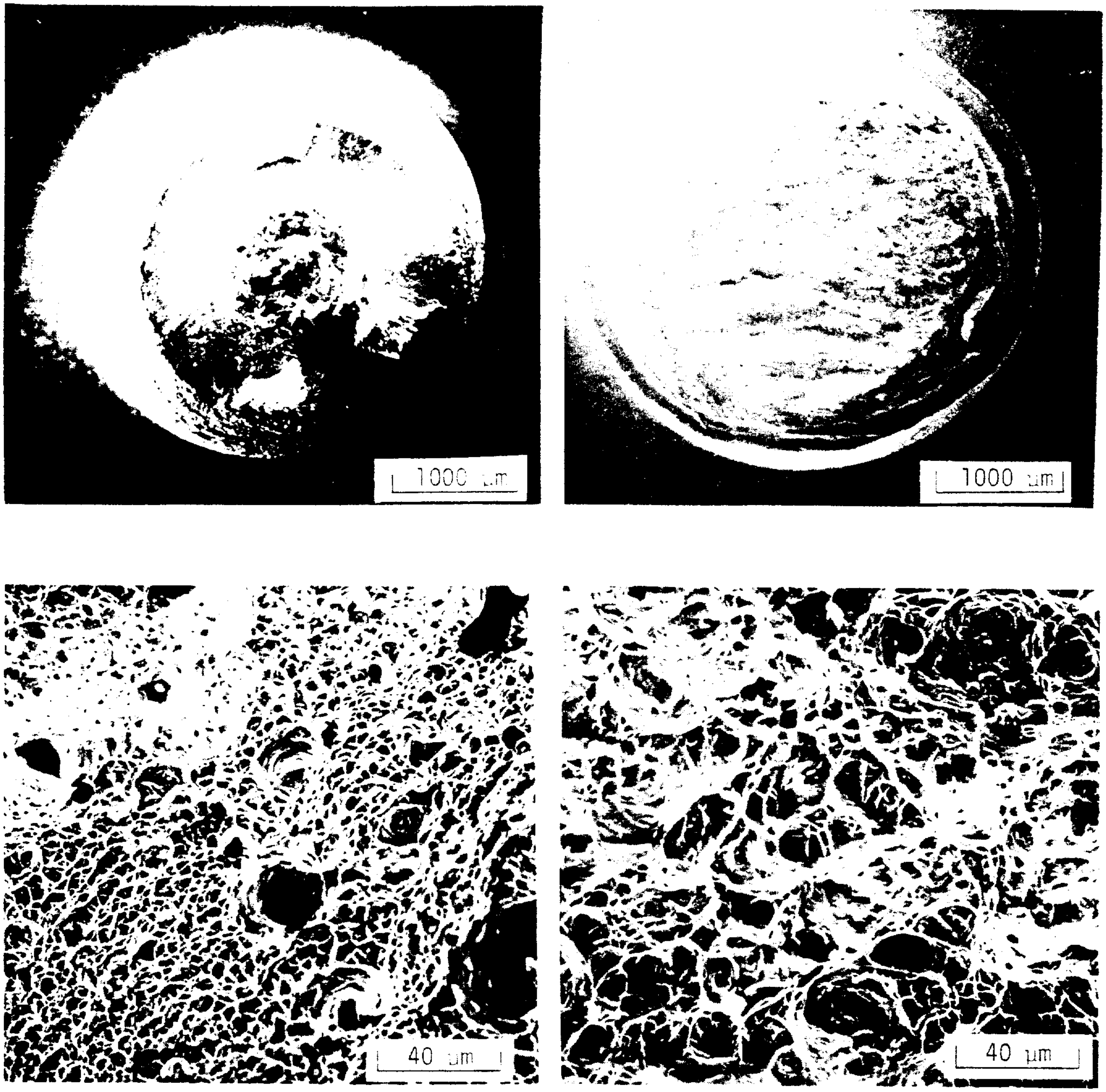


Solution Annealed



Sensitized: 24 hours at 920 K

FIGURE 1. Effect of Heat Treatment on the Microstructure of Type 21-6-9 Stainless Steel Alloy

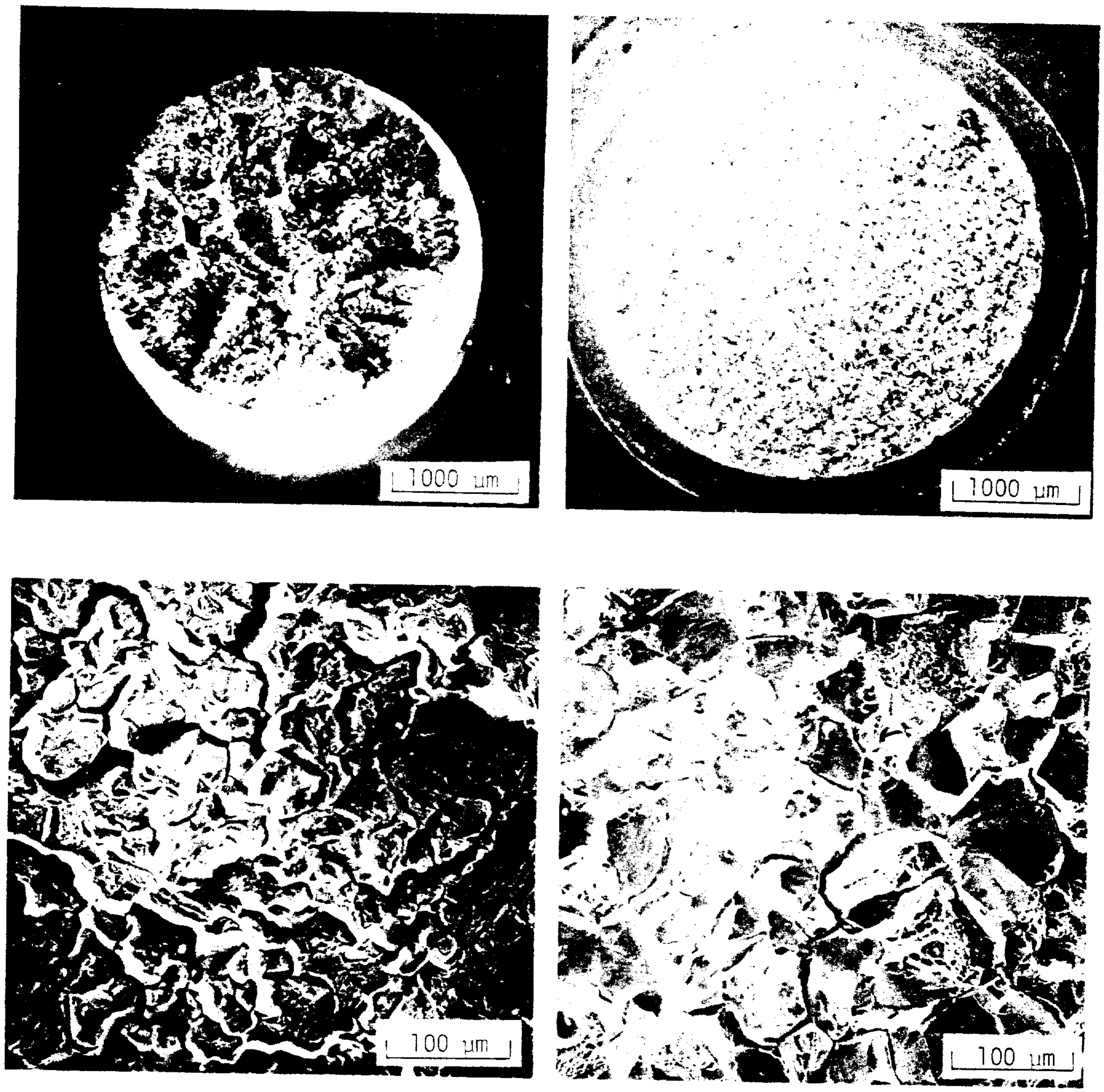


Smooth Bar

Notch Bar

Test Temperature: 300 K
Test Environment: Air

FIGURE 2. Fracture of Solution-Annealed Type 21-6-9 Stainless Steel Alloy

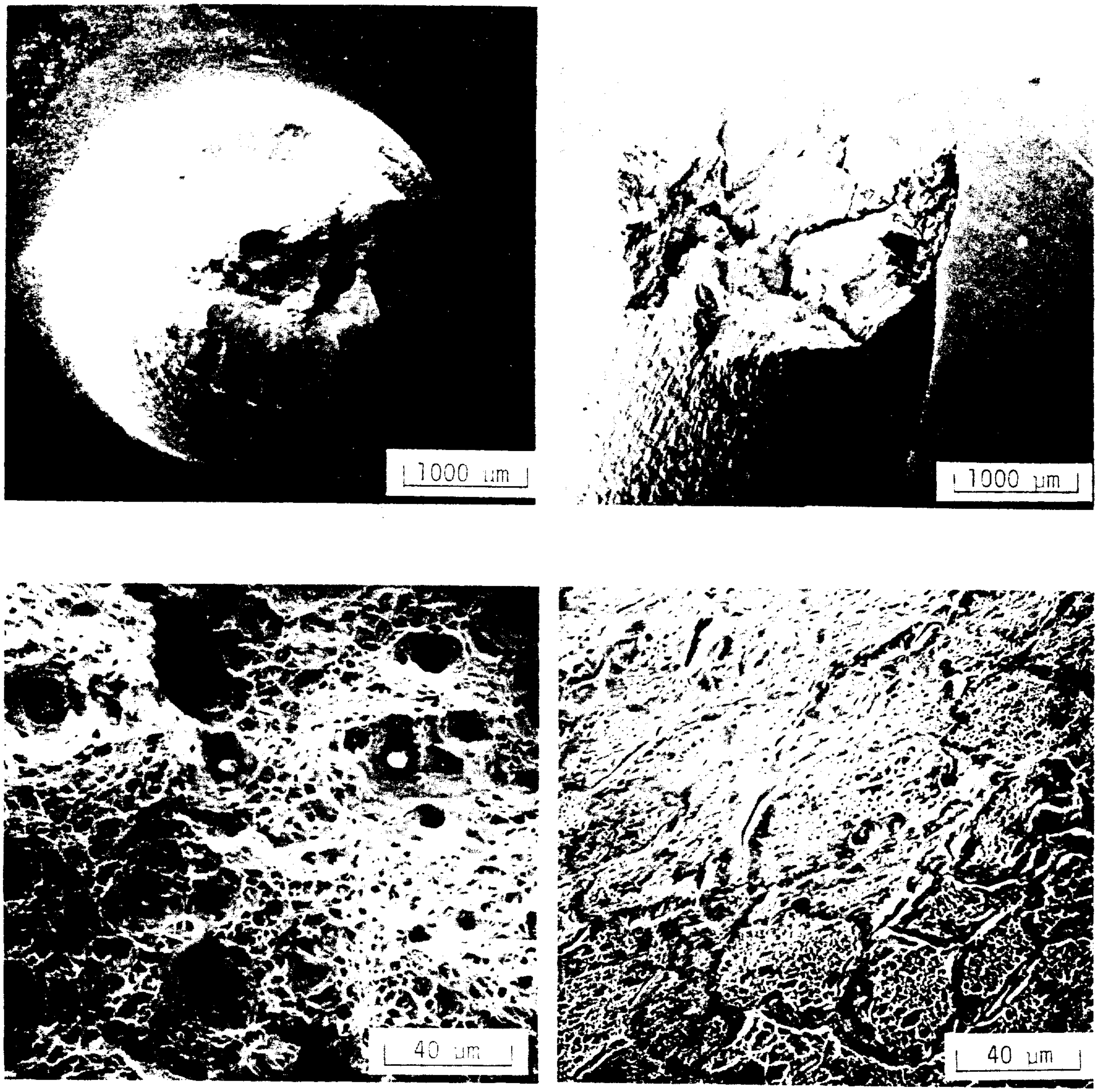


Smooth Bar

Notch Bar

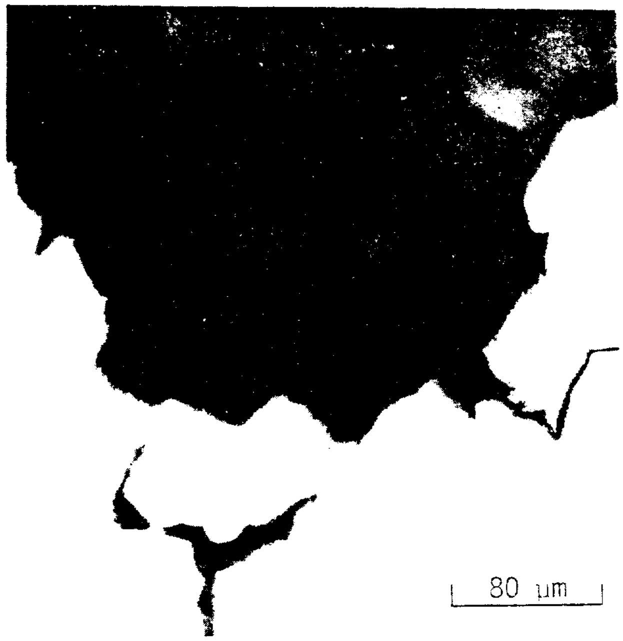
Test Temperature: 200 K
Test Environment: Air

FIGURE 3. Fracture of Sensitized Type 21-6-9 Stainless Steel Alloy

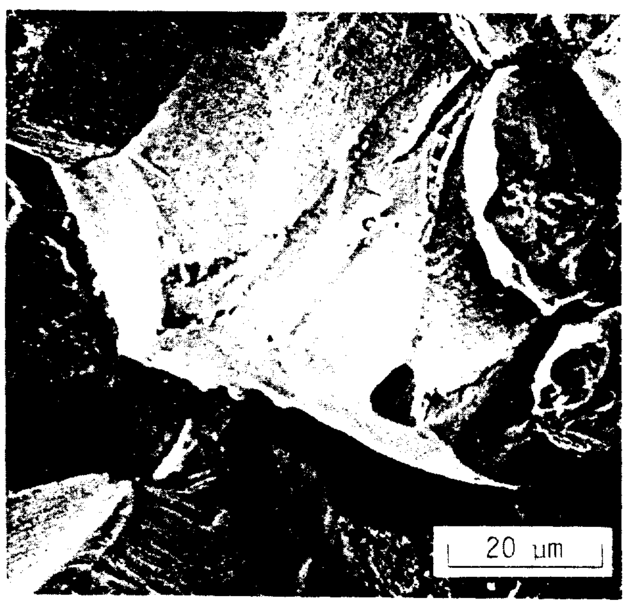


Solution Annealed   Sensitized

FIGURE 4. Fracture of Type 21-6-9 Stainless Steel Alloy in High-Pressure Hydrogen



Longitudinal Section



Fracture Face

FIGURE 5. Sensitized and Notched Type 21-6-9 Stainless Steel Alloy Tested in High-Pressure Hydrogen

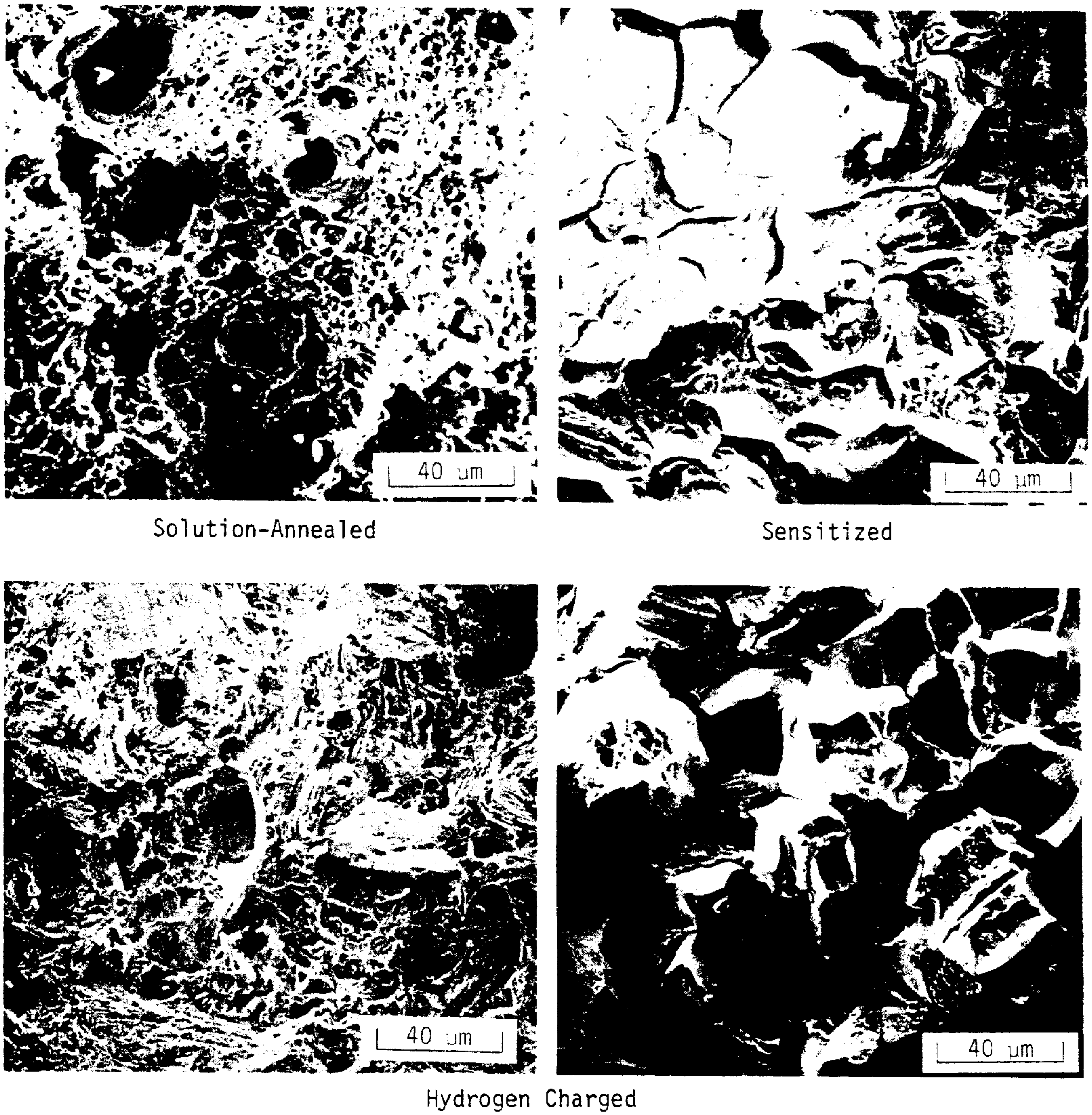


FIGURE 6. Alteration of Fracture Mode with Sensitization and Hydrogen Charging