

RECEIVED BY TIC

FEB 27 1979

MASTER

## FRACTURE OF Fe-Cr-Mn AUSTENITIC STEEL

by

G. R. Caskey, Jr.

Savannah River Laboratory  
E. I. du Pont de Nemours and Company  
Aiken, South Carolina 29801

## NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

This paper is to be presented at the  
108th Annual AIME Meeting,  
New Orleans, Louisiana, February 18-22, 1979

This paper was prepared in connection with work under Contract No. AT(07-2)-1 with the U. S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

7b

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

## FRACTURE OF Fe-Cr-Mn AUSTENITIC STEEL\*

by

G. R. Caskey, Jr.

Savannah River Laboratory  
E. I. du Pont de Nemours & Co.  
Aiken, SC 29801

### ABSTRACT

Tensile tests of Tenelon® (U.S. Steel), a nitrogen-strengthened iron-base alloy containing 18% chromium and 15% manganese, demonstrated that cleavage fracture can occur in some austenitic steels and is promoted by the presence of hydrogen. Tensile failure of Tenelon® at 78 K occurred with no detectable necking at low strain levels. The fracture surface contained cleavage facets that lay along coherent twin boundaries oriented transversely to the tensile axis. Charging gaseous hydrogen at 69 MPa pressure and 650 K had no significant effect on the mechanical behavior or fracture mode at 78 K, but raised the ductile-to-brittle transition temperature from less than 200 K to about 250 K.

---

\* The information contained in this article was developed during the course of work under Contract No. AT(07-2)-1 with the U. S. Department of Energy.

## INTRODUCTION

Tenelon® is one of the iron-chromium-manganese-nitrogen alloys developed to fill the need for a nickel-free austenitic stainless steel.<sup>1-7</sup> These steels have a transition from ductile behavior at room temperature to brittle behavior at 78 K. The transition may be gradual or rapid depending upon the steel composition, stress state, and strain rate.<sup>3</sup> The transition is particularly sensitive to the total interstitial content of carbon and nitrogen. Fracture at low temperature is characterized by planar facets on the fracture face that lie along {111} planes.<sup>6</sup> Deformation stacking faults are common, but deformation twins and  $\alpha'$ -martensite are not observed.<sup>6</sup> Low-temperature brittleness has been attributed to deformation faulting,<sup>6</sup> although early studies ascribed the ductility loss to formation of presumably brittle martensite.<sup>3</sup>

Investigation of hydrogen-assisted fracture of austenitic steels at Savannah River Laboratory has shown that Tenelon® is susceptible to both internal and external hydrogen damage at room temperature.<sup>8</sup> Surface cracks developed on surfaces of machined specimens, but were absent if the surface was electropolished.

## RECENT TESTS AT SAVANNAH RIVER LABORATORY

Tensile tests with both smooth-bar and single-edge-notched specimens of Tenelon® have been extended to a wider temperature range (78 to 350 K) and include fractographic as well as x-ray diffraction and metallographic examinations.

## Tensile Tests

Tensile tests of both smooth-bar and single-edge-notched specimens were made at a constant cross-head displacement rate of 0.05 cm/min at temperatures between 77 and 350 K. Specimens were machined from a Tenelon® plate whose chemical composition (Table 1) had been determined by scanning electron microprobe analyses. The axes of the round tensile (smooth-bar) specimens were aligned parallel to the direction in which the plate was rolled at the mill. The long dimensions of single-edge-notched specimens were aligned parallel to the rolling direction, with the notch transverse to the rolling direction.

The specimens had a uniform grain structure in the as-received condition, with an average grain size of 20 to 40  $\mu\text{m}$  (Figure 1). Annealing twins were a common feature in this alloy. Pronounced inclusion stringers were also present. These stringers consisted of fairly massive particles, predominantly manganese and sulfur, and smaller globular particles of undetermined composition. The larger inclusions formed characteristic depressions or mounds on the fracture surface (Figure 2).

Tensile tests demonstrated that Tenelon® is brittle at 78 K whether or not hydrogen is present. However, prior saturation with hydrogen reduced tensile ductility at all test temperatures; the reduction in ductility as measured by area was less than the reduction of ductility as measured by elongation. But the presence of deuterium had little consistent effect on either yield or

ultimate strength (Table 2). Annealing and electropolishing tended to improve the ductility of Tenelon® at 78 K and to increase the ultimate strength. On the other hand, fracture toughness was reduced by annealing (Table 3).

The temperature dependence of reduction-in-area, Figure 3, is comparable to that reported by Schaller and Zackay for an alloy of 16.4% Cr, 14.5% Mn, 0.025% C, and 0.49% N.<sup>3</sup> Steels of slightly differing composition retain relatively high ductility except at the lowest test temperature,<sup>3,6</sup> indicating that the ductile-to-brittle transition is extremely sensitive to composition.

Tensile tests in hydrogen at high pressure (69 MPa) showed reduced ductility and strength (Table 4). The ductility loss, about 10%, was more than the 3% ductility loss determined previously by reduction-in-area of electropolished specimens of Tenelon® under comparable test conditions.<sup>8</sup> In contrast, the ductility loss for a machined surface can be as much as 30%.<sup>8</sup>

Response of a Magna-Gage® (American Instrument Company) indicated that, except for deformation at 198 K, Tenelon® was stable during deformation with respect to transformation to ferromagnetic martensite. At 198 K, Magna-Gage® readings of 30 to 130 units above background were obtained from the hydrogen-free specimen; the hydrogen-charged specimen did not respond to the Magna-Gage® in spite of a plastic strain of about 20%. These results indicate that hydrogen tends to suppress the strain-induced

transformation of austenite to  $\alpha'$ -martensite, an effect also observed in Type 304L stainless steel.<sup>9</sup>

### Fractography

Three fracture modes were observed, depending upon specimen treatment, hydrogen content, and test temperature: dimpled rupture, intergranular failure, and transgranular cleavage. Dimpled rupture was the only fracture mode observed in hydrogen-free specimens tested at 200 to 350 K (Figure 4). Intergranular failure was observed in hydrogen-free tensile specimens annealed at 1170 K and tested at 78 K (Figure 5). However, a mixed intergranular-transgranular cleavage fracture mode was observed in single-edge-notched specimens annealed at 1170 K and tested at 78 and 198 K (Figure 6). The stress state apparently had an influence on the fracture path. Tensile and single-edge-notched specimens of as-received Tenelon® tested at 78 K failed by mixed transgranular cleavage and dimpled rupture, regardless of their hydrogen content, as did hydrogen-charged specimens tested at 200 to 273 K.

Transgranular cleavage at 78 K was not significantly affected by hydrogen. The size, shape, and surface markings on the facets were identical whether or not the specimens were saturated with hydrogen before testing (Figure 7). Facets on Type 304L stainless steel have a similar appearance, although precise details differ slightly, which suggests a common mechanism of formation independent of chemical composition.<sup>10</sup>

Microcracks commonly formed along coherent twin boundaries and occasionally along grain boundaries (Figure 8). The faces of these microcracks were probably the facets observed on the fracture surface. Deformation bands intersecting the microcracks caused the markings on the facet. Both facets and microcracks extended across one grain only and never across grain boundaries. Consequently, the fracture surfaces had facets surrounded by areas of microvoid coalescence.

The incidence of transgranular cleavage was directly related to both test temperature and hydrogen content of the specimens. Microcracks occurred throughout the reduced-gauge portion of hydrogen-charged tensile specimens more frequently at 273 and 198 K than at 78 K (Table 5). Similar hydrogen-free specimens tested at 273 and 198 K were free of microcracks. Hydrogen-free specimens tested at 78 K contained very few microcracks and these were close to the fracture surface, although ductility was very low and the fracture itself was predominantly transgranular cleavage. At 78 K, the initial formation of microcracks apparently triggered an avalanche of cracks and early failure, so that crack formation was never widespread.

There was no direct evidence for a strain-induced martensite phase in Tenelon® deformed at 78 K. Wide-angle x-ray scattering patterns from the fracture faces of specimens tested at 78 K revealed two phases: (1) an austenitic phase with an FCC lattice having a lattice parameter of  $3.62 \times 10^{-8}$  cm, and (2) a second

phase probably with a hexagonal lattice. This latter phase could arise from deformation faulting of the austenite. The absence of a martensitic phase is confirmed by the x-ray and electron diffraction results reported by Defilippi for similar steel.<sup>6</sup>

## DISCUSSION

Two hypotheses have been proposed to explain the brittle fracture of Cr-Mn-N stainless steels at low temperature:

(1) martensite formation,<sup>3</sup> and (2) high localized strain arising from deformation faulting.<sup>6</sup> The first explanation is questionable because several steels in this class of Cr-Mn-N stainless steel deform and fracture in a brittle manner at 78 K with no detectable martensite formation.<sup>6</sup> Furthermore, the transition temperature does not correlate with the volume fraction of strain-induced martensite.<sup>6</sup>

Deformation faulting is common in austenitic stainless steels and can be a precursor of deformation twinning or strain-induced transformation to the hexagonal close-packed configuration.<sup>11</sup> Low values of the stacking fault energy (a measure of the ease with which faults are formed) lead to widespread faulting. In Tenelon®, the stacking fault energy is low, about 20 mJ/m<sup>2</sup>, and faulting is extensive, developing readily observable bands in the microstructure (Figure 8). As the deformation temperature is lowered, the stacking fault energy decreases and faulting becomes

easier in many austenitic steels.<sup>12</sup> Defilippi suggested that the high local strain associated with the faults and intersections of faults could give rise to local stresses sufficient to nucleate microcracks,<sup>13</sup> but a detailed mechanism was not proposed. The present investigation supports this latter explanation.

## CONCLUSIONS

There is a ductile-to-brittle transition in Tenelon.<sup>®</sup> The transition temperature is raised by the presence of hydrogen introduced by gas-phase charging.

Brittle fracture at 78 K as well as hydrogen-assisted fracture at 200 to 280 K proceeds by microcracking across individual grains followed by ductile rupture of the intervening material. The ductility loss depends on the relative amount of brittle fracture.

Microcrack formation and cleavage facets on the fracture face of Tenelon<sup>®</sup> are similar in appearance to those observed in hydrogen-assisted fracture of Type 304L stainless steel between 200 and 280 K.

The mechanism of formation is believed to be the same for both steels and is related to deformation faulting and  $\epsilon$ -phase formation.

Neither  $\alpha'$ -martensite nor hydrogen is necessary for this fracture mode, but hydrogen does promote cleavage fracture.

## REFERENCES

1. Chi-Mei Hsiao and E. J. Dulis. "Precipitation Reactions in Austenitic Cr-Mn-C-N Stainless steels." *Trans. ASM* 49, 655 (1957).
2. Chi-Mei Hsiao and E. J. Dulis. "Phase Relationship in Austenitic Cr-Mn-C-N Stainless Steels." *Trans. ASM* 50, 773 (1958).
3. F. W. Schaller and V. F. Zackay. "Low Temperature Embrittlement of Austenitic Cr-Mn-N-Fe Alloys." *Trans. ASM* 51, 609 (1959).
4. Chi-Mei Hsiao and E. J. Dulis. "Effect of Interstitial Carbon Plus Nitrogen and Precipitation Reactions on the Properties of Austenitic Cr-Mn-C-N Steels." *Trans. ASM* 52, 855 (1960).
5. J. C. Shyne, F. W. Schaller, and V. F. Zackay. "The Tensile and Yield Strength of Cr-Mn-N Steels at Low Temperatures." *Trans. ASM* 52, 848 (1960).
6. J. D. Defilippi, K. G. Brickmer, and E. M. Gilbert. "Ductile to Brittle Transition in Austenitic Chromium-Manganese-Nitrogen Stainless Steel." *Trans. Met. Soc. AIME* 245, 2141 (1969).
7. G. T. Haddick, L. D. Thompson, E. R. Parker, and V. F. Zackay. "New Nickel-Free Austenitic Stainless Steel for Ambient and Cryogenic Service." *Metal Progress* 114 (6), 37 (1978).
8. M. R. Louthan, Jr. "Effects of Hydrogen on the Mechanical Properties of Low Carbon and Austenitic Steels." page 53, *Hydrogen in Metals*. Ed. by I. M. Bernstein and A. W. Thompson, ASM (1974).
9. G. R. Caskey, Jr. "The Role of Twinning and Transformation in Hydrogen Embrittlement of Austenitic Stainless Steel." page 437, *Environmental Degradation of Engineering Materials*. Ed. by M. R. Louthan and R. P. McNitt, Virginia Polytechnic Institute (1977).
10. G. R. Caskey, Jr. "Fractography of Hydrogen Embrittled Type 304L Stainless Steel." *Scripta Met.* 11, 1077 (1977).
11. F. Lecroisey and A. Pineau. "Martensitic Transformation Induced by Plastic Deformation in the Fe-Ni-Cr-C System." *Met. Trans.* 3, 387 (1972).

12. L. Remy and A. Pineau. "Twinning and Strain-Induced FCC  $\rightarrow$  HCP Transformation in the Fe-Mn-Cr-C System." *Mat. Sci. and Eng.* 28, 99 (1977).
13. K. H. G. Ashbee. "Intersecting Stacking Faults in Face-Centered Cubic Lattices." *Acta Met.* 15, 1129 (1967).

TABLE 1  
Chemical Composition of Tenelon® Plate

Element	Weight %
Chromium	17.44 ±0.06
Manganese	15.31 ±0.20
Nitrogen	0.40 to 0.60
Nickel	0.22 ±0.02
Silicon	0.53 ±0.01
Phosphorus	0.02
Sulfur	0.02
Iron	66.47 ±0.24
Carbon	0.1 maximum

TABLE 2  
Temperature Dependence of Mechanical Properties of Tenelon®

Test Temp., K	Hydrogen Exposure	Specimen Condition	Strength, MPa		Elongation, %		Area Reduction, %
			Yield	Ultimate	Uniform	Total	
350	None	As-machined	675	1265	48	59	76
	69 MPa D <sub>2</sub>	As-machined	700	1300	53	60	61
273	None	As-machined	830	1480	50	58	68
	69 MPa D <sub>2</sub>	As-machined	920	1540	48	50	40
198	None	As-machined	1050	1960	59	66	50
		As-machined	1020	1625	40	40	30
78	None	As-machined	1735	1780	19	19	8
	None	Electropolished	1730	2040	22	22	7
	None	1170 K - 24 hr	1670	2120	25	25	12
	None	1270 K - 24 hr	1450	1730	21	21	13
	69 MPa D <sub>2</sub>	As-machined	1720	1775	20	20	6

TABLE 3  
Fracture Toughness of Tenelon®

<i>Test Temp, K</i>	<i>Specimen Condition</i>	<i>Fracture Toughness, MPa<math>\sqrt{m}</math></i>
78	As-received	68.6
	Anneal 1170 K	36.5
	Anneal 1270 K	71.1
200	As-received	127.8
	Anneal 1170 K	99.6
	Anneal 1270 K	120.5

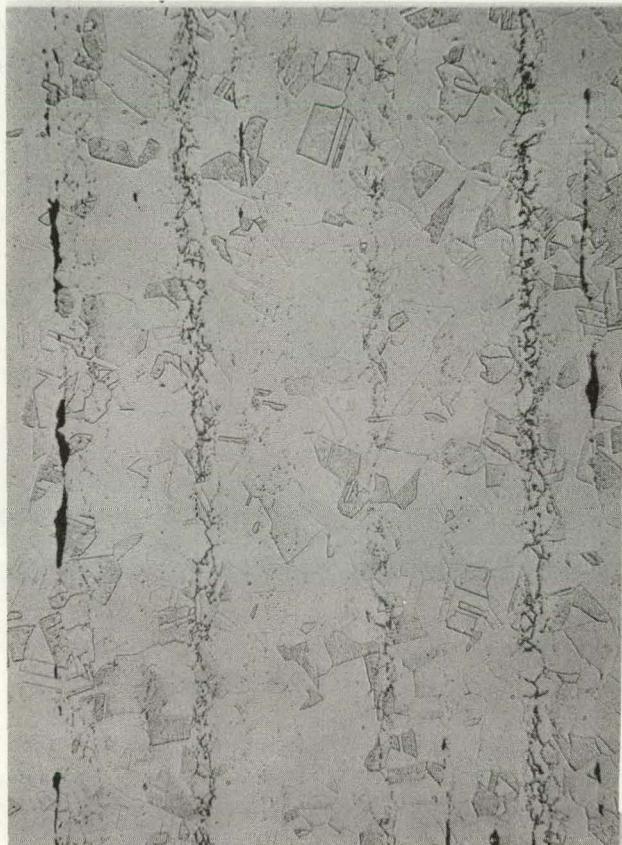
TABLE 4  
Effect of High-Pressure Hydrogen on Tensile Properties of Tenelon®

<i>Test Atmosphere</i>	<i>Strength, MPa</i>		<i>Elongation, %</i>		<i>Area Reduction, %</i>
	<i>Yield</i>	<i>Ultimate</i>	<i>Uniform</i>	<i>Total</i>	
60 MPa He	700	1410	55	62	73
69 MPa H <sub>2</sub>	685	1330	51	58	66

TABLE 5  
Microcrack Occurrence in Tenelon® Tensile Specimens

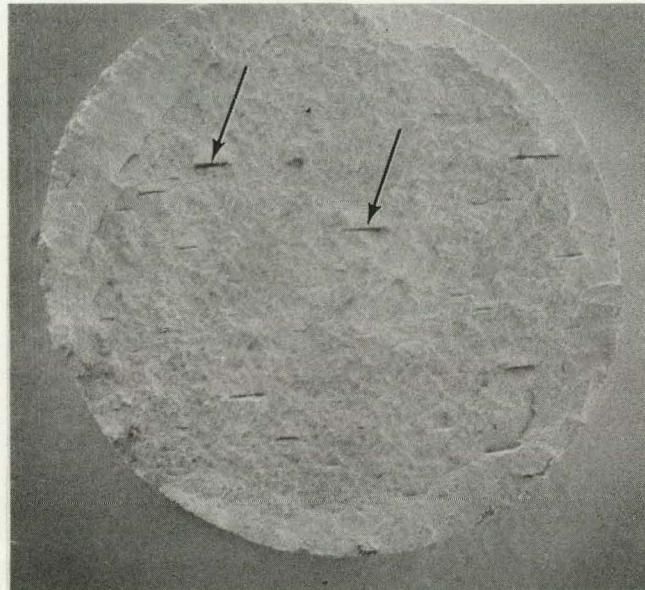
<i>Test Temp, K</i>	<i>Microcrack Frequency, number/cm<sup>2</sup></i>	
	<i>Hydrogen-Charged</i>	<i>Not Hydrogen-Charged</i>
273	900	0
198	1100	0
78	(0) <sup>a</sup>	(0) <sup>a</sup>

*a.* Several microcracks close to fracture surface.



100  $\mu\text{m}$

FIGURE 1. Grain Structure and Inclusion Stringers  
in Tenelon® Plate



Arrows indicate  
large inclusion  
stringers

1000  $\mu\text{m}$

FIGURE 2. Fracture of Tenelon® Tensile Specimen at 273 K

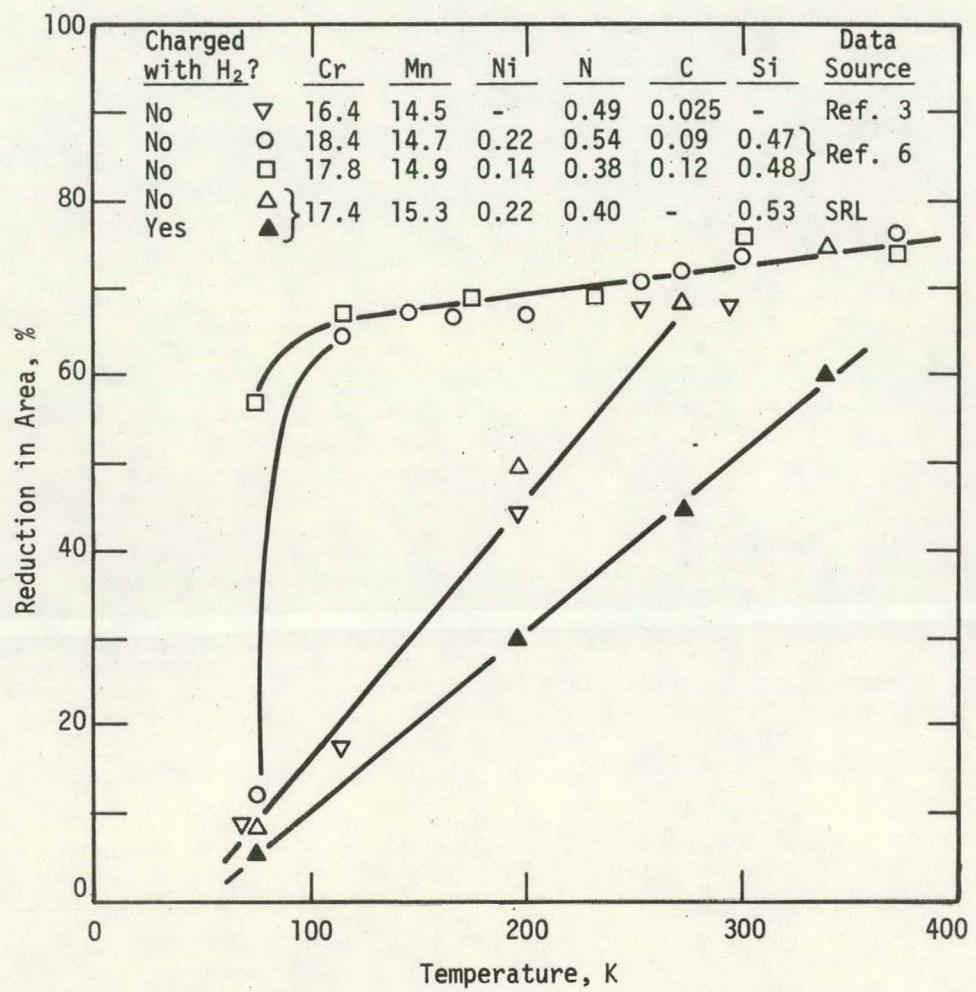
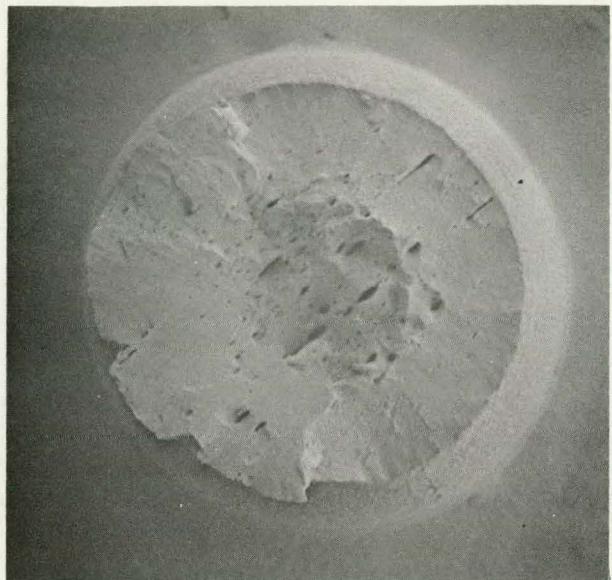
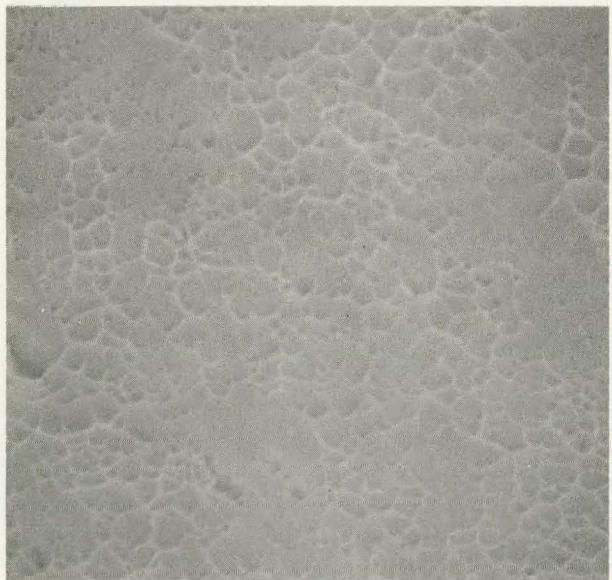


FIGURE 3. Temperature Dependence of Tensile Ductility of Fe-Cr-Mn Alloys

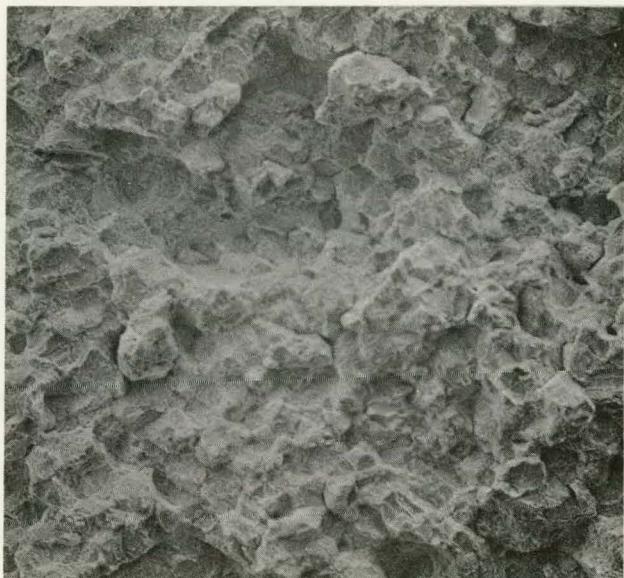


1000  $\mu\text{m}$

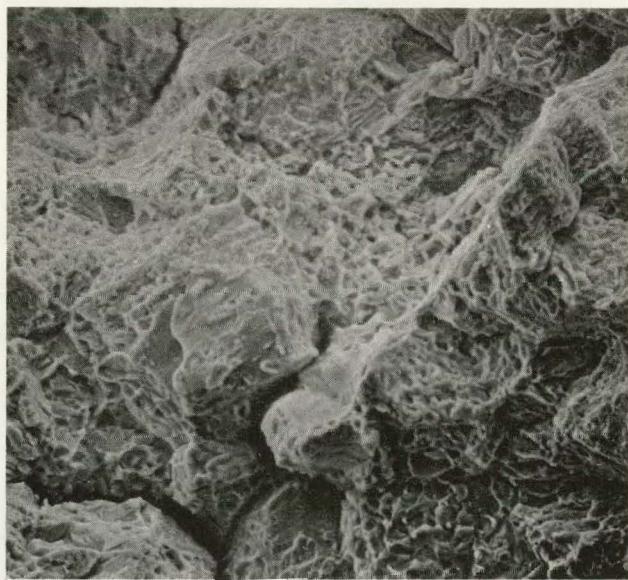


20  $\mu\text{m}$

FIGURE 4. Ductile Rupture of Tenelon® at 350 K



100  $\mu\text{m}$



20  $\mu\text{m}$

FIGURE 5. Intergranular Failure of Tenelon<sup>®</sup>  
at 78 K Following 1170 K Anneal

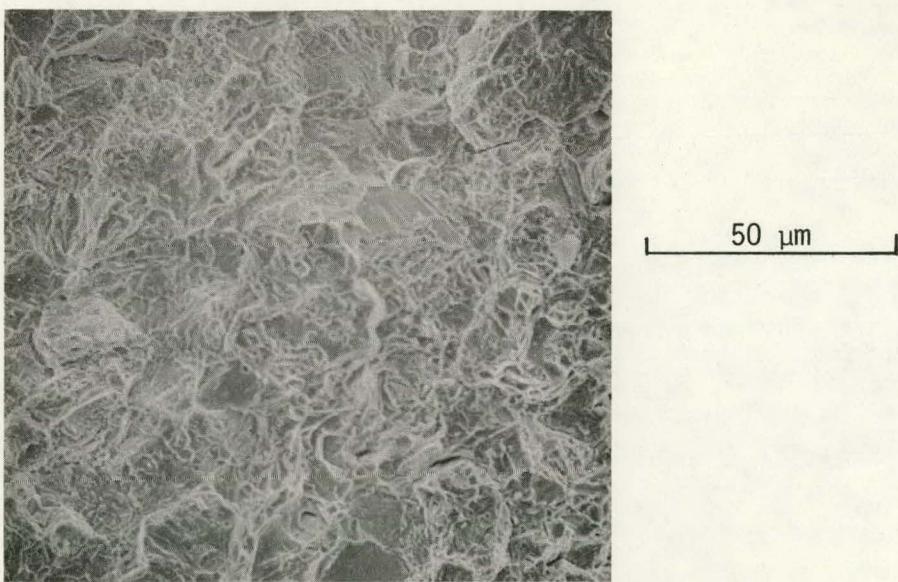
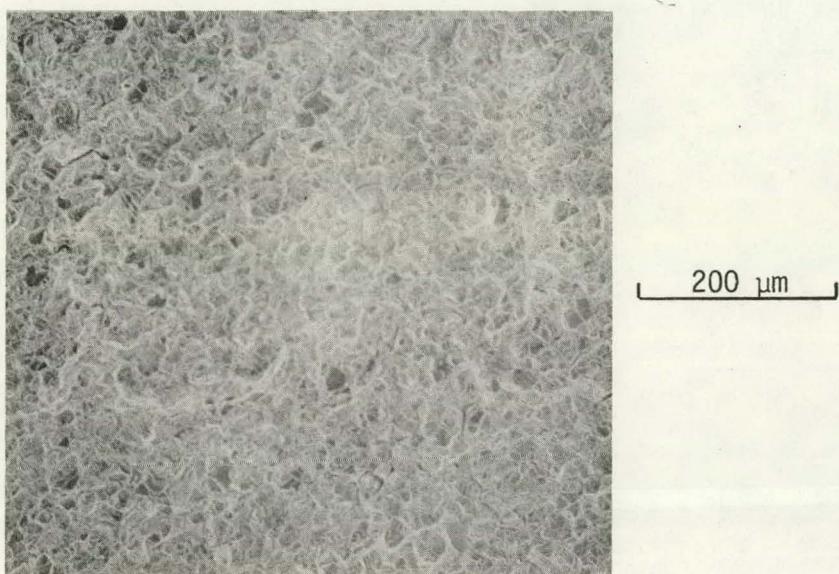
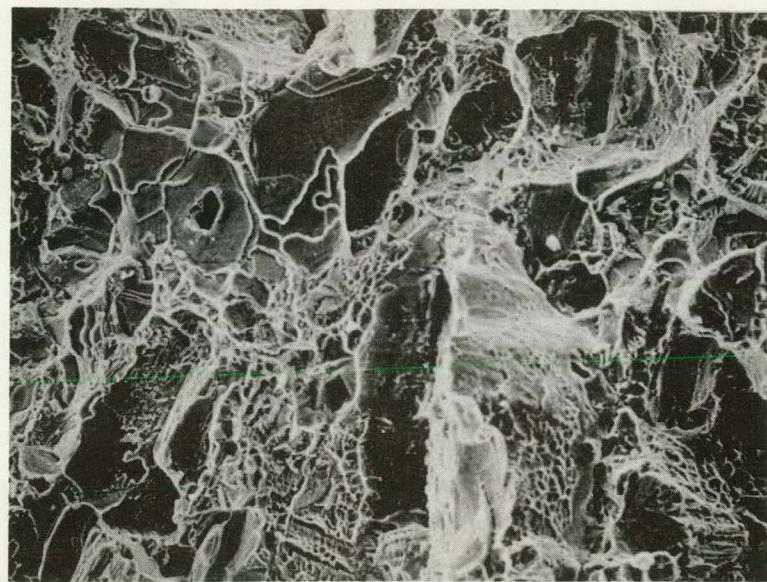


FIGURE 6. Mixed Fracture of Single-Edge-Notched Specimen Tested at 78 K

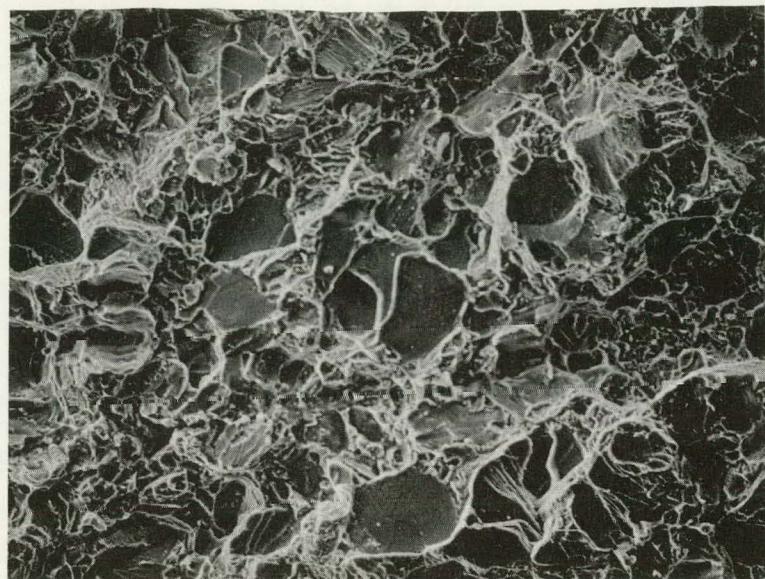


40  $\mu\text{m}$



4  $\mu\text{m}$

FIGURE 7A. Facets on Fracture Surfaces of Hydrogen-Saturated Specimen Tested at 78 K



40  $\mu\text{m}$



4  $\mu\text{m}$

FIGURE 7B. Facets on Fracture Surfaces of Specimen  
Not Charged with Hydrogen Before Testing at 78 K



Twin Boundary



Twin Boundary



Grain Boundary

FIGURE 8. Microcracks Along Coherent Twin Boundaries and Grain Boundaries