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THE EFFECT OF HYDROGEN ISOTOPES AND HELIUM ON THE
TENSILE PROPERTIES OF 21-6-9 STAINLESS STEEL (U)

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by

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THE EFFECT OF HYDROGEN ISOTOPES AND HELIUM ON THE TENSILE PROPERTIES OF 21-6-9 STAINLESS STEEL (U)

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High-energy-rate-forged (HERF) stainless steels are used as the materials of construction for pressure vessels designed for the containment of hydrogen and its isotopes. Hydrogen and helium, the decay product of tritium, are known to embrittle these materials (1-4). HERF stainless steels have a relatively good resistance to hydrogen-and-helium-induced embrittlement when compared to annealed stainless steels due to their high number density of dislocations, which act as traps for hydrogen and helium. However, the degree of embrittlement in these materials can vary considerably because of microstructure and yield strength variations introduced during the forging process.

In this study the effect of hydrogen and tritium on the tensile properties of 21-6-9 stainless steel was measured as a function of HERF yield strength in the range of 660 to 930 MPa. The effect of microstructure was studied also by conducting tensile tests with HERF and annealed samples.

Tensile tests were conducted using samples machined from 21-6-9 stainless steel forgings that were supplied (from Oxnard, CA) in the form of forward extruded cylinders. The forgings were approximately 10 cm long and 3.8 cm in diameter. The forgings had been HERF'ed to produce nominal yield strengths of 660, 760, 870, and 930 MPa. A few samples from the 930 MPa forging were subsequently annealed at 1144 K for 5 minutes to produce a recrystallized microstructure having a yield strength of 517 MPa. The tensile samples had a 19.1 mm gage length and a 4.8 mm diameter.

One set of samples was exposed to hydrogen at 623 K and 69 MPa for 6 weeks. This treatment saturated the samples with approximately 9500 atomic parts per million (appm) hydrogen based on available diffusivity and solubility data (4). Another set of samples was exposed to tritium gas at 423 K and 31 MPa for 9 months, and aged for 12 more months at 298 K for helium build-in from tritium decay. The average tritium and helium concentrations were calculated to be 2470 appm and 390 appm respectively. The samples were pulled at room temperature in air using a screw-driven testing machine and a crosshead speed of 0.0085 mm/s.

The mechanical properties of the unexposed, hydrogen-exposed, and tritium-exposed samples are summarized in Table I. Yield strengths ranged from 500 MPa for annealed samples to 918 MPa for the highest strength HERF samples. In general, hydrogen-charged and tritium-charged-and-aged samples had higher strengths and lower ductilities than uncharged samples. The yield strength increase caused by hydrogen was dependent on the initial strength and ranged from about 11% for the 500 MPa samples to 5% for the 918 MPa samples. The yield strength increase over uncharged samples that was observed in the tritium-charged-and-aged

samples ranged from 25% for the 500 MPa samples to 10% for the 918 MPa samples. The results indicate that helium is more effective than hydrogen isotopes on a "per-atom" basis in affecting strength.

The effects of hydrogen and tritium on ductility as a function of annealed and HERF yield strengths are plotted in Figure 1. The ductility of unexposed HERF samples was reduced by about 11% as the HERF yield strength was increased from 712 to 918 MPa. Over this same range of yield strengths, the samples containing 9500 appm hydrogen showed on average 40% lower ductilities than the unexposed samples. The HERF tritium-exposed-and-aged samples showed 33% lower ductilities than the unexposed samples over the same range of strengths. Tritium-charged-and-aged samples that had been annealed prior to charging had much lower ductilities than similarly charged HERF samples (Figure 1). Annealed samples failed prior to the onset of necking with ductilities less than 15%. HERF samples failed after the onset of necking with ductilities greater than 30 %. Fracture surface examination revealed that unexposed samples and all hydrogen-and-tritium-charged HERF samples failed by a microvoid nucleation and growth process. In contrast, annealed samples that were tritium-charged-and-aged fractured along grain and twin boundaries in a brittle manner.

In summary, hydrogen-charged and tritium-charged-and-aged samples had higher yield strengths and lower ductilities than uncharged samples. The ductility was not strongly affected by yield strength. HERF microstructures were less susceptible to helium embrittlement effects than annealed microstructures.

Acknowledgement

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2. S. L. Robinson, "Tritium and Helium Effects on Plastic Deformation in AISI 316 Stainless Steel", *Materials Science and Engineering*, 96 (1987), p. 7-16.
3. A. J. West, Jr. and McIntyre R. Louthan, Jr., "Hydrogen Effects on the Tensile Properties of 21-6-9 Stainless Steel", *Metallurgical Transactions A*, 13A, (1982) p. 2049-2058.
4. G. R. Caskey, Jr., "Hydrogen Effects in Stainless Steels," *Hydrogen Degradation of Ferrous Alloys*, eds. J.P. Hirth, R. W. Oriani, and M. Smialowski, (Park Ridge, NJ: Noyes Publication, 1985), p. 882-862.

TABLE II
Summary of Tensile Results
HERF 21-6-9 Stainless Steel

Condition	Nominal Strength (MPa)	Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)	Ductility (%)
<i>Uncharged</i>					
Annealed	517	500	811	68.6	74.8
HERF	662	712	932	34.3	71.1
HERF	758	819	969	22.5	56.1
HERF	869	825	1029	28.4	64.3
HERF	931	918	1032	39.5	63.3
<i>Hydrogen Charged</i>					
Annealed	517	555	839	71.1	59.7
HERF	662	776	974	29.1	42.8
HERF	758	1005	1093	23.5	33.0
HERF	869	836	948	----	----
HERF	931	965	1073	38.9	39.2
<i>Tritium Charged and Aged</i>					
Annealed	517	627	782	16.5	13.7
HERF	662	829	996	34.0	46.1
HERF	758	863	1019	21.2	47.7
HERF	869	882	1025	23.8	35.1
HERF	931	1013	1094	37.2	42.8

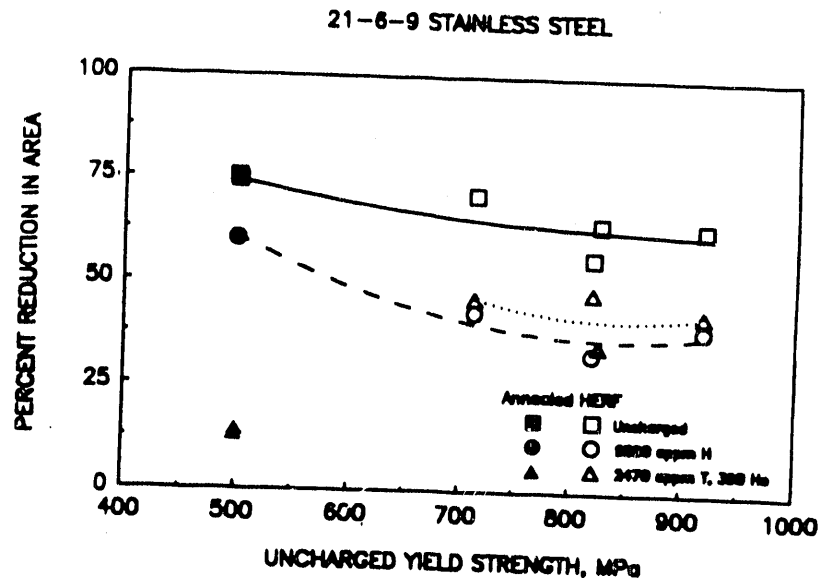


Figure 1. Ductility of Unexposed, Hydrogen-Exposed, and Tritium-Exposed-and-Aged Samples as a Function of the Yield Strength of Unexposed Samples.

**The Effects of Hydrogen Isotopes and Helium
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Lawrence Livermore National Laboratory, April 24-26, 1990**

OUTLINE

Summary

Background

Experimental Procedure

Experimental Results

Conclusions and Future Objectives

SUMMARY

PURPOSE

To support tritium reservoir reliability programs by measuring the effects of hydrogen isotopes and helium on the mechanical properties of tritium reservoir materials.

RESULTS

Yield Strength: Hydrogen-charged and Tritium-charged-and-aged increased the yield strength of HERF 21-6-9 stainless steel.

Ductility: Hydrogen-charged and Tritium-charged-and-aged decreased the ductility of HERF 21-6-9 stainless steel. Ductility was not strongly affected by HERF yield strength.

Microstructural Effect: A recrystallization annealing heat treatment increased the ductility of uncharged and hydrogen charged samples, but reduced the ductility of tritium charged samples.

BACKGROUND

High-Energy-Rate-Forged (HERF) 21-6-9 Stainless Steel is a commonly used tritium reservoir material.

Reservoirs are reclaimed and reused.

Hydrogen Isotopes and Helium increase the yield strength and decrease the ductility of the stainless steels

EXPERIMENTAL PROCEDURE

Material: HERF 21-6-9 Stainless Steel

Nominal Yield Strengths: 75 ksi (135 ksi annealed for 5 minutes at 8570C)
96 ksi
110 ksi
126 ksi
135 ksi

Exposure Conditions: As-received (Control)

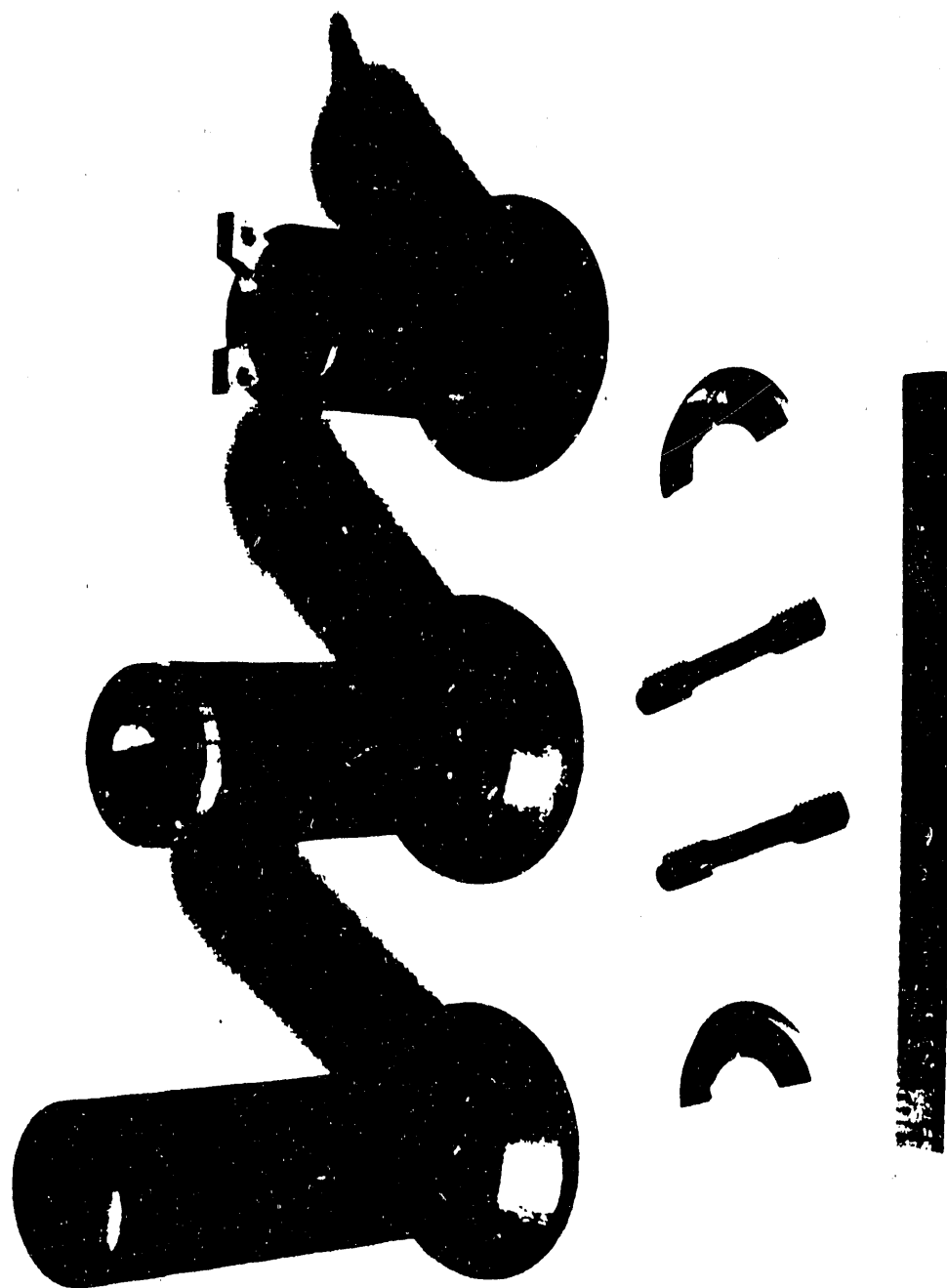
Hydrogen-charged (10k psi, 3500C, 6 weeks)
9500 appm H

**Tritium-charged-and-aged (10k, 1500C, 9 months, aged
for 12 months at 250C) 2470 appm H, 390 appm He**

Mechanical Testing: air tested, room temperature, 0.02"/minute

Fractography

Metallography



**TRANSVERSE SECTION MICROSTRUCTURES
FROM FORWARD EXTRUDED HERF' ING**



**UNANNEALED
135 KSI**



**ANNEALED
75 KSI**

**LONGITUDINAL SECTION MICROSTRUCTURES
FROM FORWARD EXTRUDED HERF'ING**

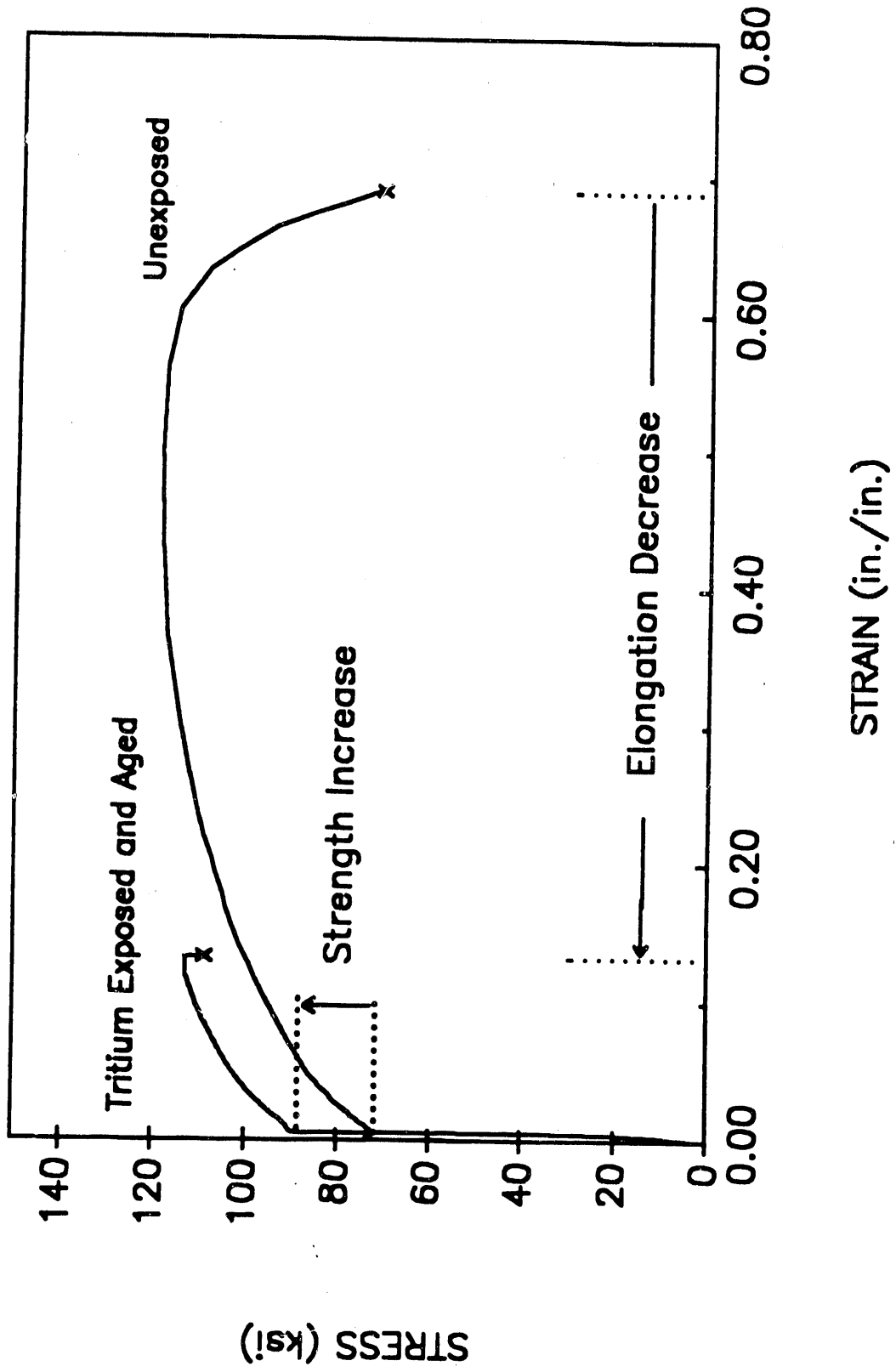


**UNANNEALED
135 KSI**

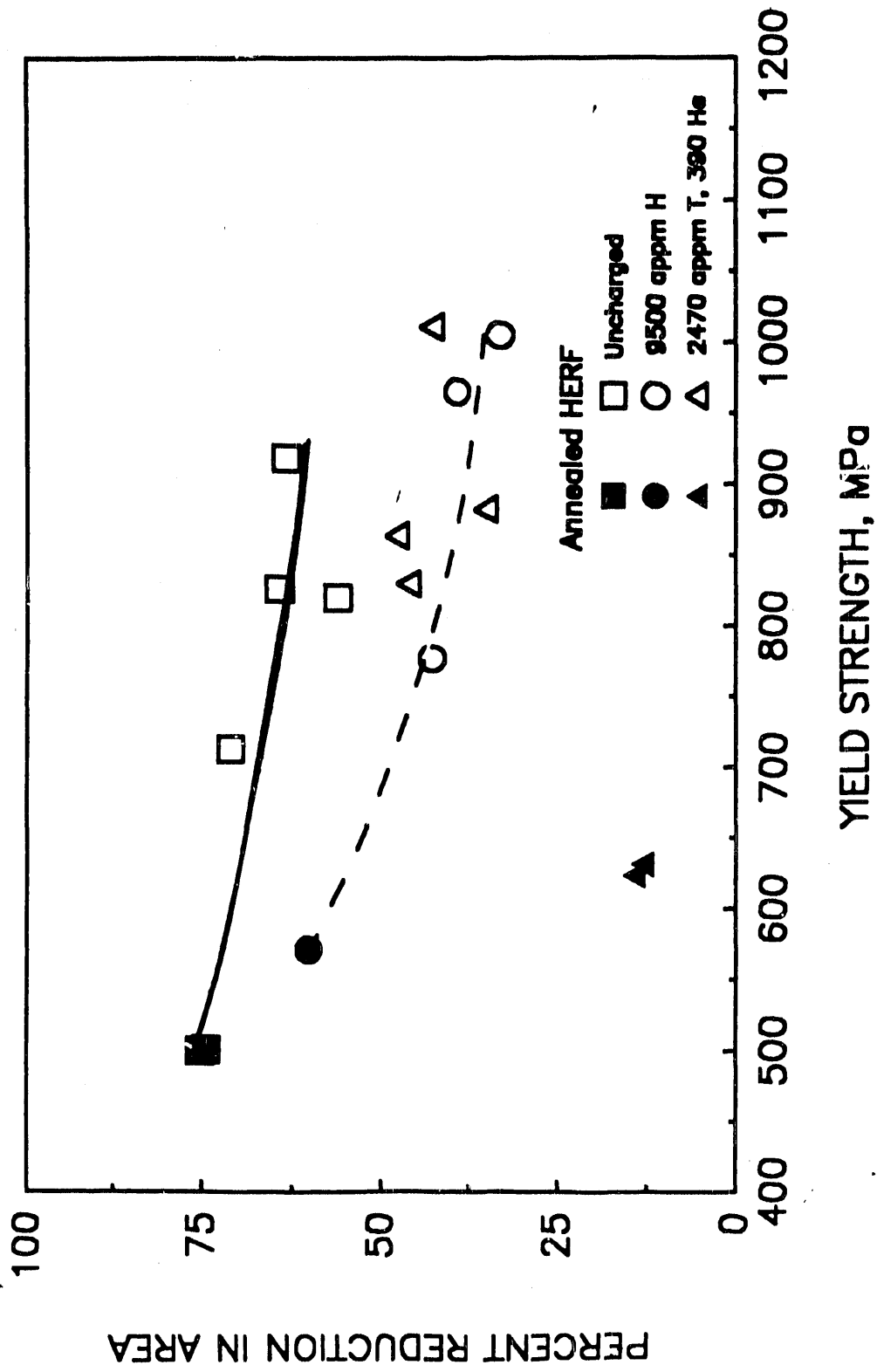


**ANNEALED
75 KSI**

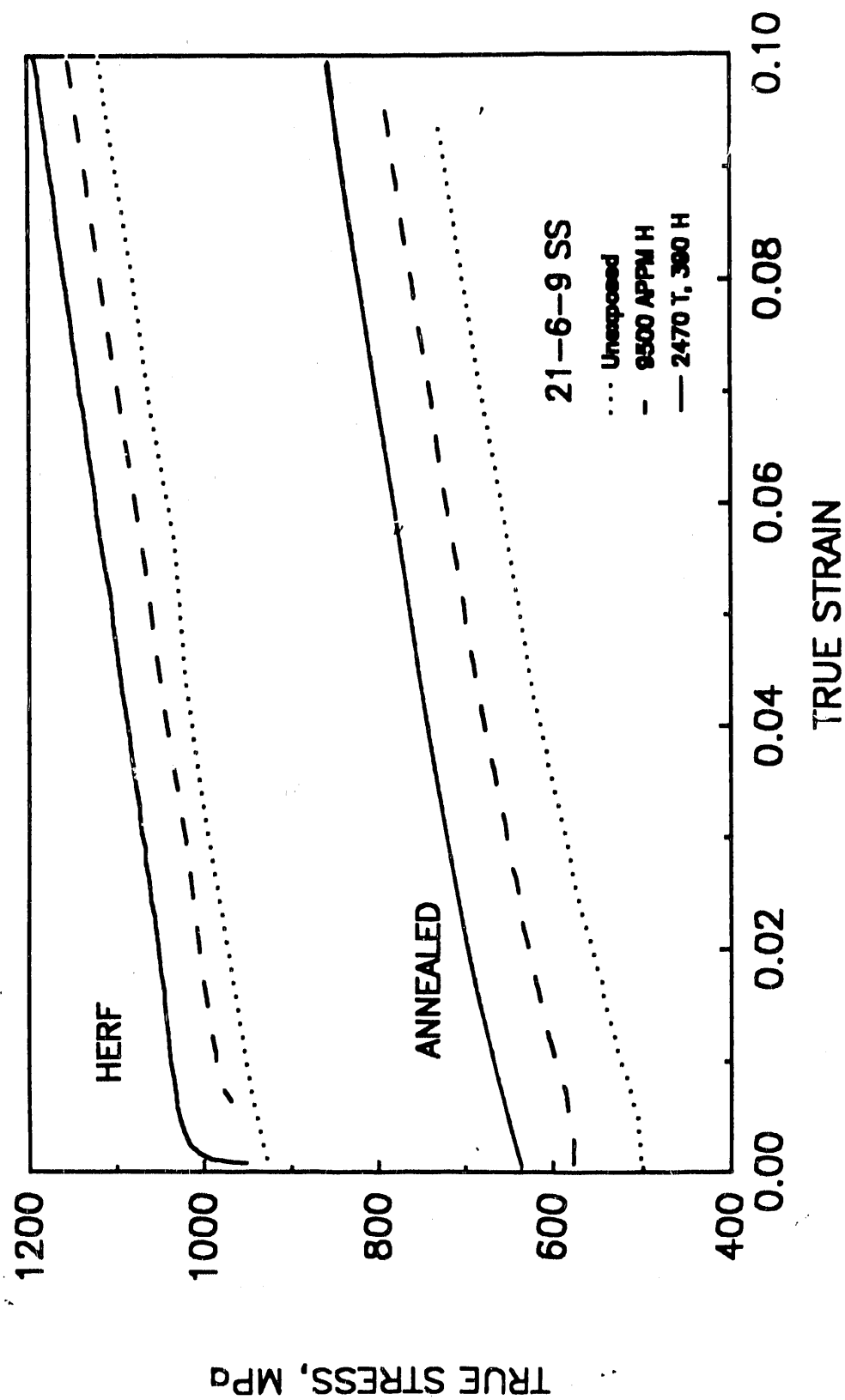
EFFECT OF TRITIUM AND HELIUM ON TENSILE PROPERTIES 21-6-9 Stainless Steel



Ductility Decreased by Hydrogen and Helium



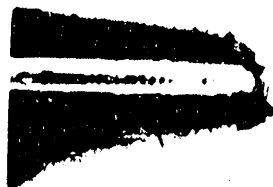
Flow Stress Increased by Hydrogen and Helium



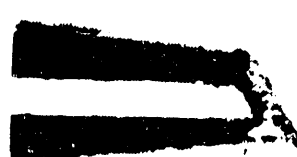
Fracture Modes in Tritium-Exposed-and-Aged Tensile Samples (U)



75 KSI
(Annealed)

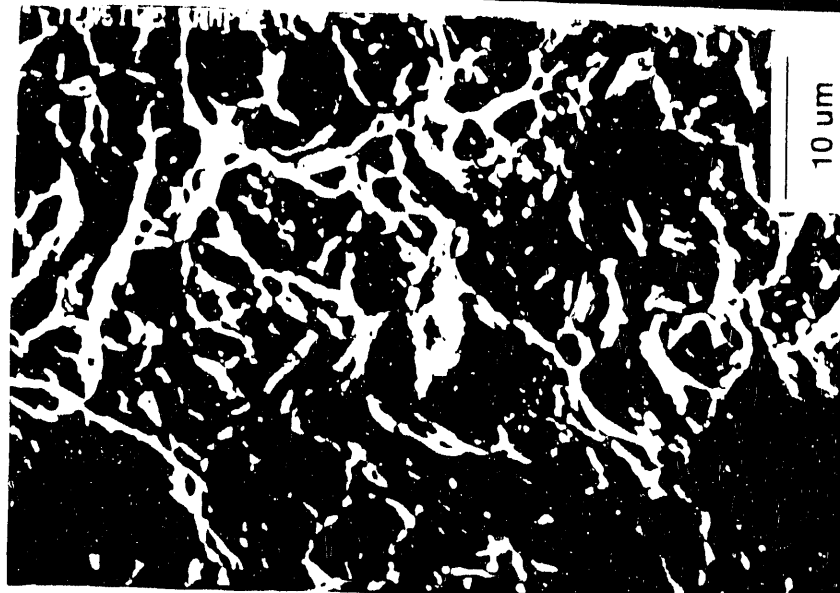


96 KSI
(HERF)

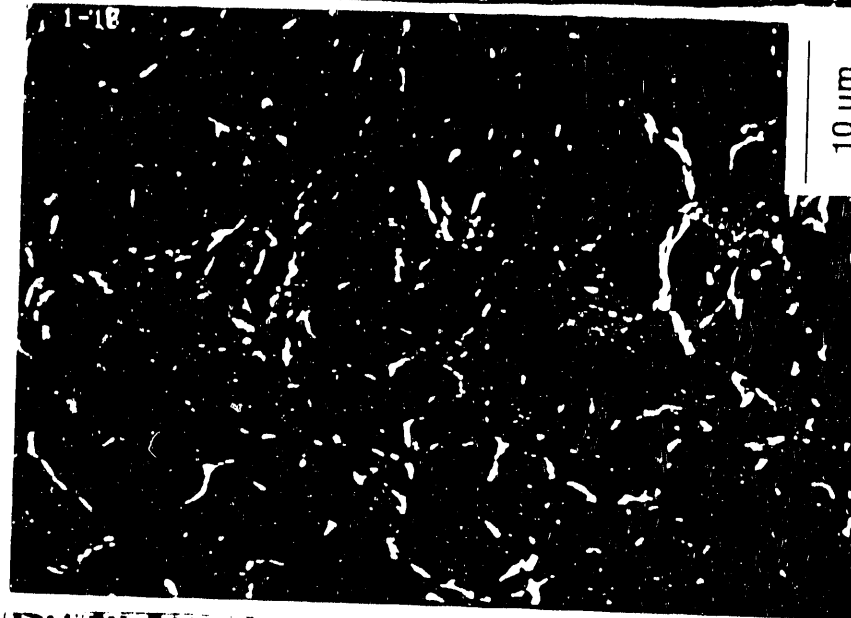


135 KSI
(HERF)

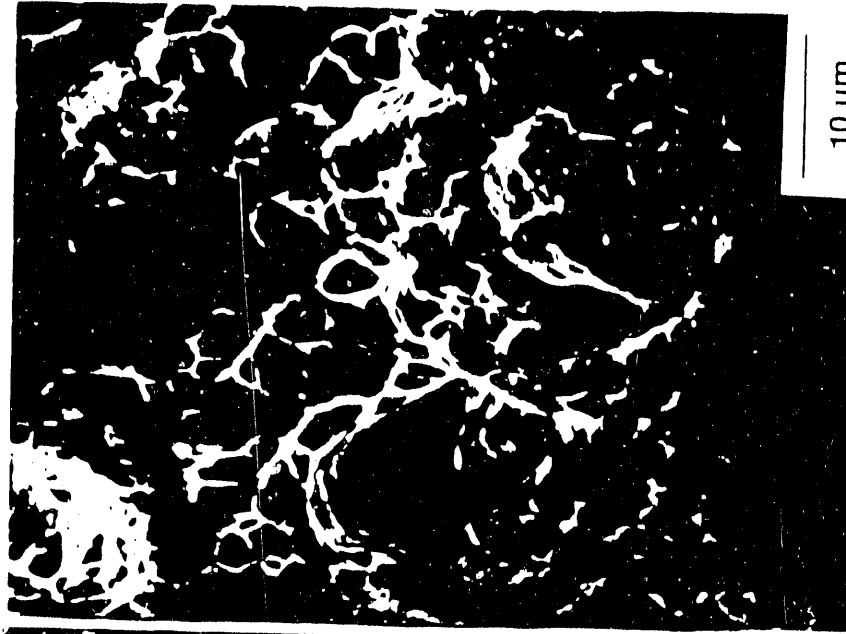
**FRACTURE APPEARANCE
135 KSI (HERF) TENSILE SAMPLES**



**TRITIUM-EXPOSED
-AND-AGED**

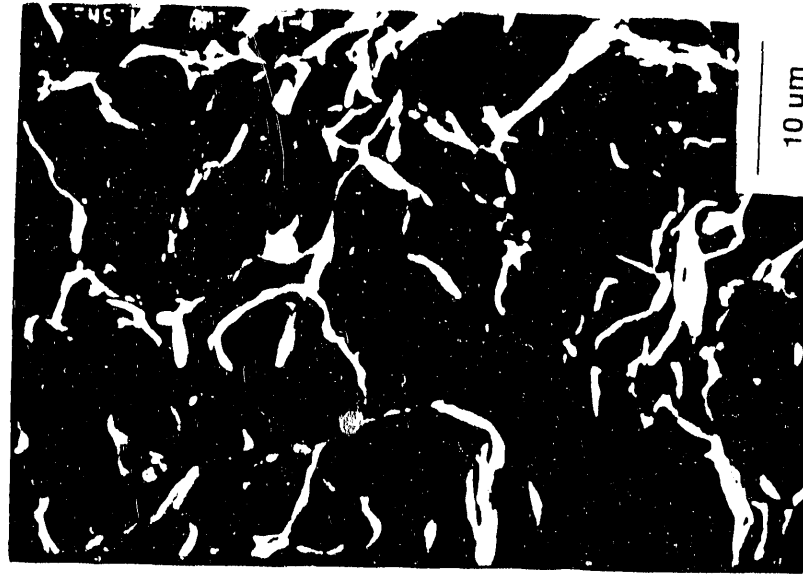


HYDROGEN-EXPOSED

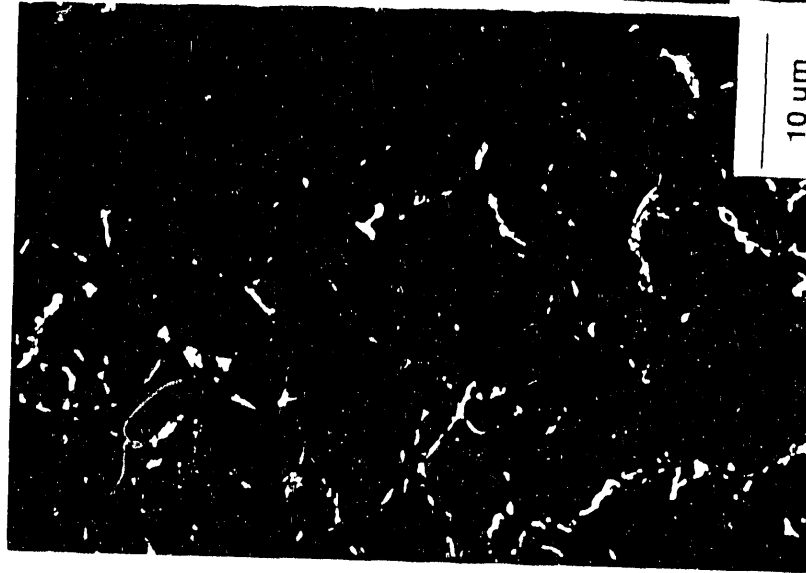


CONTROL

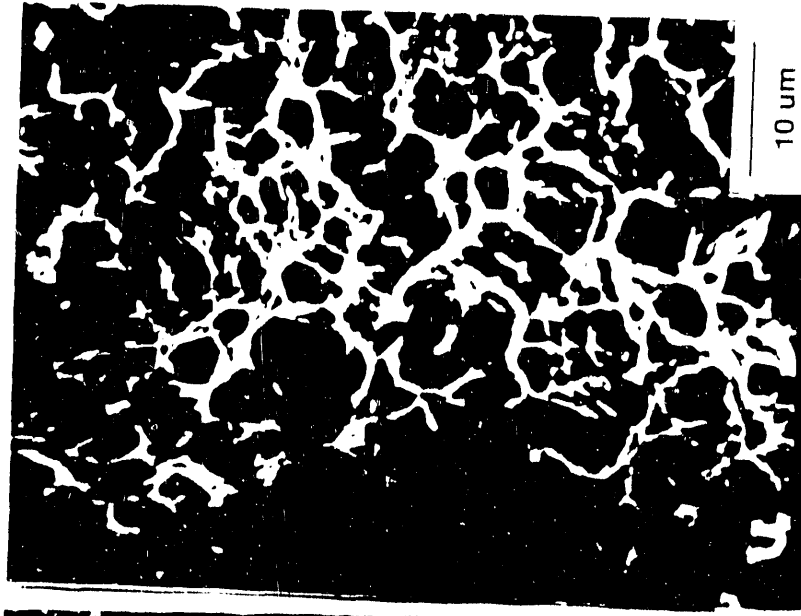
**FRACTURE APPEARANCE
75 KSI (ANNEALED) TENSILE SAMPLES**



**TRITIUM-EXPOSED
-AND-AGED**



HYDROGEN-EXPOSED



CONTROL

CONCLUSIONS

21-6-9 HERF microstructures were less susceptible to helium embrittlement than the annealed microstructures.

Yield Strength is increased and ductility decreased in hydrogen-charged and tritium-charged-and-aged samples.

Ductility was not strongly influenced by yield strength.

FUTURE OBJECTIVES

Measure the effect of helium content on the tensile properties.

Correlate tensile results to the fracture toughness results of similarly conditioned material.

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