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**FOSSIL FUEL
PROCESS PLANTS**

**ALLOY EVALUATION FOR FOSSIL FUEL
PROCESS PLANTS (LIQUEFACTION)**

Annual Report

1 October, 1978 through 30 September, 1979

C.M. Woods and T.E. Scott

**Work Performed Under Contract
No. W-7405-eng-82**

**Ames Laboratory
Iowa State University
Ames, Iowa 50011**

Date Transmitted: November 1979

U. S. DEPARTMENT OF ENERGY



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October 15, 1979

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FOREWORD

This report covers work performed during the period 1 October, 1978 through 30 September, 1979. The work was administered by the Division of Systems Engineering -- Energy Technology with Mr. Wate Bakker, of the Materials Section, as project manager. The report was prepared by Charles M. Woods and T. E. Scott of the Mechanical Properties Section in the Metallurgy and Ceramics Division at the DOE-Ames Laboratory, Ames, Iowa.

The work was performed under the direction of Dr. Scott as principal investigator assisted by: C. M. Woods, S. Shei, C. V. Owen and L. K. Reed.

ABSTRACT

Room temperature mechanical properties were determined for A387-74A-Gr.22-C1.2 steel after various environmental exposures. Exposure conditions include 168 hours at 500 or 800°F in a coal slurry with 4000 psig hydrogen pressure, 1000 hours at 800°F in 4000 psig argon, or 856 hours at 800°F in a coal slurry with 4000 psig hydrogen pressure. Comparison of ambient condition tensile tests of the exposed samples with base property data revealed that no degradation of ambient temperature mechanical properties occurred.

Microstructural examination of samples from all exposure conditions run to date showed no dramatic changes with the exception of the formation of a multilayer scale on the surface of samples exposed to the coal slurry environment. Point source x-ray fluorescence and electron microprobe analyses were carried out on these layers along with the parent metal. The

relative Fe, Cr, Mo, Mn, S, and Si concentrations were determined and a profile plotted.

Scanning electron micrographs were made of the fracture surfaces of unexposed smooth-bar and notched-bar tensile samples and compared to micrographs of like samples exposed for 856 hours in a coal slurry at 800°F in 4000 psig H₂ gas. Fracture surfaces of samples exposed to the slurry exhibited considerably larger voids than those of unexposed samples. The micrographs showed a characteristic ductile-dimple type failure. Spherical particles lying at the base of many of the large voids visible in the fracture surfaces of all the samples examined were analyzed using point source x-ray fluorescence and found to consist of mainly Mn, S, Si and Fe.

Examination of an Inconel rupture disc that failed during the attempted 1000 hour slurry-H₂ exposure revealed considerable pitting and corrosive attack by the environment above the slurry. Further investigation revealed failure occurred by intergranular cracking.

Cross sectional area measurements of specimens exposed to a coal slurry environment for 856 hours showed inward growth of scale with subsequent loss of sample diameter.

OBJECTIVE AND SCOPE

The objective of this program is to evaluate the mechanical properties of liquefaction process plant "dissolver" vessel materials in a "dissolver" vessel environment including coal slurry and pressurized hydrogen gas at temperatures up to 800°F. Originally, the intent was to test at 900°F but we soon learned that above 850°F (455°C) gasification is ignited giving coke and methane. Consequently, all runs originally indicated as 900°F will be run at 800°F to assure there are no excursions above the critical gasification ignition temperature.

Specifically, the degradation of notched-bar and smooth-bar tensile samples of 2 1/4 Cr - 1 Mo will be monitored as a function of exposure time and stress in the "dissolver" vessel environment. Compact tension specimens will be used to monitor the degradation of fracture properties in the "dissolver" vessel environment.

Progress Summary

I. Mechanical Properties Tests

A. Procedure:

Smooth-bar (Fig. 1) and notched-bar (Fig. 2) tensile samples were prepared from longitudinal (parallel to the rolling direction) sections of the plate. Testing was done on a TT-C Instron tensile machine at a constant crosshead rate of 0.05 in/min. Mechanical property data were calculated from the load-elongation curves. Cross-sectional areas and reduction in area at the neck were calculated by measuring across three diameters and averaging. All diameter measurements were made on a Gaertner model 2001 Toolmaker's microscope.

One group each of smooth-bar and notched-bar tensile samples was placed in a pressure vessel and subjected to one of the following exposures: 168 hours with stress (~ 25 ksi) in coal slurry and hydrogen gas at 4000 psig pressure at 500°F or 800°F (tests 51-53, Table 1), 1000 hours with and without stress in 4000 psig argon gas at 800°F (tests 63-66, Table 2), 856 hours with and without stress in coal slurry and hydrogen gas at 4000 psig pressure at 800°F (tests 67-70, Table 2). The coal slurry was a blend of 35 volume percent of -100 mesh Kentucky bituminous (Proximate Analysis, wt.pct.: Moisture, 6.1; Ash, 15.5; Volatile Matter, 36.3; Fixed Carbon, 42.1) and 65 volume percent solvent. The solvent was centrifuged Synthoil product (Ash free wt.pct.: Organic benzene insols, 3.3; asphaltenes, 32.3; Oils, 64.4) from PERC Run FB-61 made from the coal described above. The coal and solvent were graciously supplied by Paul M. Yavorsky of PERC. The slurry was prepared by mixing the fine coal in the solvent which, because of its high viscosity (161 SSF at 180°F) at room temperature, was preheated to 110°F.

Tensile samples were emersed in the coal slurry which was contained inside a 304 stainless steel can; the can was then placed inside one of the specially designed H₂ pressure vessels for subsequent pressure and temperature equilibration. The samples were exposed to conditions given above during which time the H₂ pressure was controlled within ± 100 Psig of the required pressure. The temperature fluctuations were $\pm 10^\circ\text{F}$ at 800°F and $+25^\circ\text{F}$ to -10°F for the 500°F tests. The 500°F runs seemed to exhibit an exothermic reaction between the coal slurry and H₂ gas resulting in the 25°F overshoot. This observation was extremely prevalent and reproducible and was independent of the test cell used for the exposure.

At the end of the exposures, the samples were removed from the pressure vessel and the remaining slurry dissolved away from the samples with a 50:50 mixture of acetone and toluene. Subsequently the threaded grip ends were cleaned with a wire wheel and the samples tensile tested under ambient conditions. The scale layer that formed on the surface of the samples during the exposure was very brittle and fell off upon handling of the samples. Diameter measurements were made to determine whether or not the scaling was an inward growth or an addition to the surface. It was determined to be an inward growth resulting in loss of sample cross sectional area. The new areas were calculated and tensile data obtained were based on these new measurements.

B. Results:

Mechanical properties investigated include 0.2% yield stress, ultimate tensile strength, uniform elongation, total elongation, engineering fracture stress, true fracture stress, reduction in area, and notch tensile strength. Results of the various exposure tests are given in Tables #3-#13. Average values are summarized and compared with the previously determined room temperature tensile properties (Report #IS-4587) in Tables #14 and #15 and shown graphically in Figures #14-#19. These values are statistical averages based on data certified by the 'Q' test at the 90% confidence level. It can be seen from Tables #14 and #15, and particularly from Figures #14-#19 that the values fall within the range of statistical variance of a test of this nature and so it can be concluded that from the tests run to date, the room temperature mechanical properties of A387-74A-Gr.22-C1.2 steel were not greatly affected by any of the exposures to the coal slurry environment.

II. Metallographic Analyses

A. Procedure:

Samples from all exposure conditions run to date (Tests 1-22, Table #16, Tests 39-53, Table #1, and Tests 63-70, Table #2) were prepared by mechanically polishing to Linde B (Al_2O_3 , 0.05 μ particle size) and etching for 60 seconds in 2% Nital (HNO_3 in methanol). Microstructures were examined microscopically and photographed.

Scanning electron micrographs were made of fracture surfaces from smooth-bar and notched-bar tensile samples in both the exposed and unexposed conditions. Tensile samples, exposed for 856 hours in a coal slurry at 800°F in 4000 psig hydrogen, were tensile tested at room temperature and the fracture surfaces were examined with a scanning electron microscope. These fracture surfaces were compared to those of samples in the unexposed condition. Particles lying at the base of many of the large microvoids present in the fracture surfaces were analyzed for elemental content using point source x-ray fluorescence techniques.

Stressed and unstressed tensile samples, exposed for 856 hours in a coal slurry at 800°F in 4000 psig hydrogen, were carefully cleaned with a 50:50 solution of acetone:toluene, sectioned and mounted in copper conducting bakelite. Care was taken not to disturb the surface scale that formed during the slurry exposure. The bakelite-sample composite was then mechanically ground to expose a longitudinal cross section including the surface scale (see Figures #20, #22 and #24). It was then polished to Linde A (Al_2O_3 , 0.3 μ particle size) and analyzed using electron microprobe techniques. The samples were scanned starting in the base metal approximately 50 μ in from the base metal-surface scale interface. Readings were taken

at 2 μ intervals until the end of the scale was reached. The electron beam size (diameter) was $\sim 1 \mu$. Quantitative analyses were obtained for Fe, Cr, Mo, Mn, S and Si. Scanning electron micrographs were taken of the regions scanned (Figs. #20, #22 and #24).

Point source x-ray analyses were also performed individually on each of the different regions of the base metal and scale using an EDAX unit on a scanning electron microscope. Qualitative and semi-quantitative data were obtained this way for Fe, Cr and S.

B. Results:

Examination of microstructures revealed no significant changes relative to the unexposed material for any of the exposure conditions run to date. A typical example of the microstructure is presented in Fig. #3. The presence of spherical and irregular shaped features near the surface was noted for many of the samples exposed to the coal slurry (Figs. #4, #5 and #6).

Scanning electron micrographs of fracture surfaces of unexposed and exposed (856 hrs., 800°F, 4000 Psig H₂, coal slurry) samples revealed a characteristic ductile dimple type fracture with the formation of large microvoids about spherical particles (Fig. #7), determined to be composed of Mn, S, Si and Fe. Both types of samples showed void coalescence with the exposed samples exhibiting slightly larger voids (Figs. #8-#13).

Electron microprobe analyses revealed a multilayer surface scale of varying composition (Figs. #20-#25). For the stress-exposed samples (Figs. #20 and #21), the outermost layer (#4) consists primarily of Fe, S and Mo with the Fe:S ratio at 1:1. The innermost scale layer (#2) is separated from the base metal (#1) and outermost layer (#4) by two thin bands (#3)

that consist primarily of Al, Si and O. In region #4 there appears to be a second phase, which is also rich in Al, Si and O. The innermost layer (#2) varies in composition from inside to outside with the Fe concentration decreasing from ~ 82 at% to ~ 50 at% and the S concentration increasing from ~ 9 at% to 31.5 at%. There is Cr present in the innermost layer (6.5 at%) but none present in the outermost layer, which is confirmed by the disappearance of the Cr peak in the EDAX analyses (Fig. #26).

The unstressed, exposed samples exhibited the same 1:1, Fe:S ratio in the outermost layer (#4, Fig. #24) as the stressed samples did, with the only other species present being Mo. Again the second phase in this layer consists primarily of Al, Si and O. The innermost layer (#2) varies in composition from inside to outside with Fe decreasing from ~ 54 at% to ~ 44.5 at% and S increasing from ~ 25 at% to ~ 35 at%. Again, Cr is present in the inner layer but not in the outer one. In both the stressed and unstressed samples the base metal does not vary significantly in composition of any of the elements analyzed (Figs. #21, #23 and #25).

III. Corrosion

A. Procedure:

An Inconel rupture disc that failed and caused termination of the 1000 hour coal slurry exposure tests at 856 hours was examined microscopically. The disc was examined first in the condition it was removed from the rupture disc assembly. It was then examined after being washed in 50:50 toluene-acetone and rinsed with pure acetone. Finally it was examined after being washed with water and rinsed with acetone.

Tensile samples (tests 67-70, Table #2) were measured for gage diameter

before and after the 856 hour coal slurry exposure. Surface scale was flaked off and saved for analyses yet to be run.

B. Results:

The Inconel rupture disc showed severe pitting that was more evident after the water wash. This indicates that the crystalline substance observed in the pits was water soluble. It has been previously identified as containing mostly nickel and sulfur with traces of Cu, Mg, Fe and Si (Report IS-4587). Further observations showed failure to be intergranular in nature.

The scaling of the 2 1/4 Cr - 1 Mo tensile samples resulted in a smaller base metal cross sectional area. It was previously reported that the scaling was more severe on stressed samples than unstressed samples. This was found to be in error. The scaling does occur by an inward growth mechanism.

WORK FORECAST

The completion of our new high pressure dynamic test facility has been recently accomplished. In situ coal slurry smooth-bar and notched-bar tensile testing will be carried out as well as in situ J_{IC} testing. Bolt loaded compact tension specimens will be exposed to argon or the coal slurry at 800°F, 4000 psig. Cyclic fatigue testing will be done on compact tension specimens in the coal slurry environment at 800°F and 4000 psig H_2 .

TABLE #1

FY 1978 TEST MATRIX

Test	Type	Exposure Conditions				Time (HRS)	Exposure ^A Stress (ksi)	Test Conditions ^{A,B}			
		Temp (°F)	Pressure (Psig)	Environment	Temp. (°F)			Pressure (Psig)	Environment	Number of samples	
21	Smooth	500	2000	Slurry-H ₂	1	-	500	2000	Slurry-H ₂	10	
24	Notched	500	2000	Slurry-H ₂	1	-	500	2000	Slurry-H ₂	10	
25	Smooth	800	2000	Slurry-H ₂	1	-	800	2000	Slurry-H ₂	10	
26	Notched	800	2000	Slurry-H ₂	1	-	800	2000	Slurry-H ₂	10	
27	Smooth	500	2000	Argon	1	-	500	2000	Argon	10	
28	Notched	500	2000	Argon	1	-	500	2000	Argon	10	
29	Smooth	800	2000	Argon	1	-	800	2000	Argon	10	
30	Notched	800	2000	Argon	1	-	800	2000	Argon	10	
31	Smooth	500	4000	Argon	1	-	500	4000	Argon	10	
32	Notched	500	4000	Argon	1	-	500	4000	Argon	10	
33	Smooth	800	4000	Argon	1	-	800	4000	Argon	10	
34	Notched	800	4000	Argon	1	-	800	4000	Argon	10	
35	Smooth	500	4000	Slurry-H ₂	1	-	500	4000	Slurry-H ₂	10	
36	Notched	500	4000	Slurry-H ₂	1	-	500	4000	Slurry-H ₂	10	
37	Smooth	800	4000	Slurry-H ₂	1	-	800	4000	Slurry-H ₂	10	
38	Notched	800	4000	Slurry-H ₂	1	-	800	4000	Slurry-H ₂	10	
39	Smooth	500	2000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
40	Notched	500	2000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
41	Smooth	800	2000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
42	Notched	800	2000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
43	Smooth	500	4000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
44	Notched	500	4000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
45	Smooth	800	4000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
46	Notched	800	4000	Slurry-H ₂	16B	0	Ambient	Ambient	Air	10	
47	Smooth	500	2000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
48	Notched	500	2000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
49	Smooth	800	2000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
50	Notched	800	2000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
51	Smooth	500	4000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
52	Notched	500	4000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
53	Smooth	800	4000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
54	Notched	800	4000	Slurry-H ₂	16B	25 ^C	Ambient	Ambient	Air	10	
55	Smooth	500	4000	Argon	Failure ^D	Various	-	-	-	12 ^F	
56	Notched	500	4000	Argon	Failure ^D	Various	-	-	-	12 ^F	
57	Smooth	800	4000	Argon	Failure ^D	Various	-	-	-	12 ^F	
58	Notched	800	4000	Argon	Failure ^D	Various	-	-	-	12 ^F	
59	Smooth	500	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^F	
60	Notched	500	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^F	
61	Smooth	800	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^F	
62	Notched	800	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^F	

A All loading is in tension
 B Constant cross-head rate of 0.05 inch/min.
 C Loading via precompressed rings
 D Stress-rupture tests: Until failure or 1000 Hours whichever comes first
 F Two samples at each of 6 different stress levels

TABLE #2
1000 HR. EXPOSURE TEST MATRIX

Test	Type	Exposure Conditions				Exposure ^A Stress (ksi)	Test Conditions ^{A,B}			
		Temp. (°F)	Pressure (Psig)	Environ- ment	Time (HRS.)		Temp. (°F)	Pressure (Psig)	Environ- ment	No. of Samples
63	Smooth	800	4000	Argon	1000	25 ^C	Ambient	Ambient	Air	5
64	Notched	800	4000	Argon	1000	25 ^C	Ambient	Ambient	Air	5
65	Smooth	800	4000	Argon	1000	0	Ambient	Ambient	Air	5
66	Notched	800	4000	Argon	1000	0	Ambient	Ambient	Air	5
67	Smooth	800	4000	Slurry-H ₂	1000	25 ^C	Ambient	Ambient	Air	5
68	Notched	800	4000	Slurry-H ₂	1000	25 ^C	Ambient	Ambient	Air	5
69	Smooth	800	4000	Slurry-H ₂	1000	0	Ambient	Ambient	Air	5
70	Notched	800	4000	Slurry-H ₂	1000	0	Ambient	Ambient	Air	5

A - All loading is in tension.

B - Constant cross-head rate of 0.5 in/min.

C - Loading via precompressed rings.

TABLE #3

Test 51: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°F, 4000 psig H₂

AND COAL SLURRY ENVIRONMENT

AND STRESS = 27.2 ± .5 ksi

SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in ²)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS171	.0350	80.3	96.0	23.5	13.0	53.6	75.3	216.5
TS172	.0352	80.6	96.5	24.1	13.6	53.9	75.4	219.4
TS173	.0350	79.9	95.6	23.2	13.0	53.5	75.7	220.7
TS174	.0351	79.6	95.5	23.4	13.3	53.5	75.8	220.7
TS175	.0352	80.1	95.5	26.0	14.5	52.8	75.9	219.0
TS176	.0352	79.8	95.4	24.8	14.2	52.5	75.9	217.8
TS177	.0350	79.1	94.8	23.3	13.1	53.5	74.8	212.5
TS178	.0350	79.4	95.0	24.0	14.1	54.9	74.8	217.6
TS179	.0353	79.4	94.7	25.6	14.8	52.5	75.9	217.8
TS180	.0352	78.1	93.7	24.6	13.8	52.2	75.9	216.6

TABLE #4

Test 52: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°F, 4000 psig H₂
 AND COAL SLURRY ENVIRONMENT
 AND STRESS = 24.0 ± 1.0 ksi
 NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS171	.0353	.001	65	147.5
NS172	.0365	.001	65	146.5
NS173	.0354	.001	65	147.0
NS174	.0354	.001	65	149.6
NS175	-	.001	65	-
NS176	.0354	.001	65	147.0
NS177	.0358	.001	65	146.5
NS178	.0352	.00125	65	147.7
NS179	.0359	.001	65	147.5
NS180	-	.001	65	-

TABLE #5

Test 53: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 800°F, 4000 psig H₂

AND COAL SLURRY ENVIRONMENT

AND STRESS = $22.7 \pm 1.0 \text{ ksi}$

SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in ²)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS181	.0343	78.2	93.7	22.6	13.5	53.7	75.7	220.8
TS182	.0351	77.7	93.2	25.5	15.5	52.7	76.3	222.0
TS183	.0351	76.9	93.4	24.8	14.5	52.4	76.7	225.2
TS184	.0345	79.1	95.7	21.7	12.6	55.8	74.4	218.1
TS185	.0352	78.1	94.3	24.2	14.3	53.3	75.9	220.7
TS186	.0350	76.8	92.8	23.3	14.1	52.8	75.7	217.8
TS187	.0350	76.3	92.3	22.7	13.3	52.1	76.7	223.3
TS188	.0350	77.2	94.1	24.0	14.1	52.9	75.7	217.8
TS189	.0346	78.1	94.3	21.2	12.4	54.2	75.0	216.5
TS190	.0350	78.2	94.2	23.7	14.4	53.9	75.7	222.5

TABLE #6

Test 63: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 1000 HOURS AT 800°F, 4000 psig ARGON

AND STRESS = $24.3 \pm 2.0 \text{ ksi}$

SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in ²)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS1001	.0353	79.3	97.2	25.8	14.5	53.8	75.0	215.3
TS1002	.0352	80.2	97.8	25.0	14.0	54.0	74.9	215.3
TS1003	.0354	79.7	97.3	24.4	13.7	55.1	74.1	212.9
TS1004	.0349	79.5	97.1	23.6	13.4	54.4	74.7	215.3
TS1005	.0352	79.0	96.6	24.7	14.1	53.3	74.9	212.5

TABLE #7

Test 64: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 1000 HOURS AT 800°F, 4000 psig ARGON

AND STRESS = $23.5 \pm 2.8 \text{ ksi}$

NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS1001	.0355	.001	65	147.9
NS1002	.0358	.001	65	147.3
NS1003	.0359	.001	65	147.1
NS1004	.0354	.001	65	151.1
NS1005	.0353	.0015	65	152.0

TABLE #8

Test 65: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 1000 HOURS AT 800°F, 4000 psig ARGON

SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in ²)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS1006	.0352	78.9	97.3	25.9	14.6	54.0	75.4	219.4
TS1007	.0353	78.5	96.3	25.3	15.4	54.5	74.5	214.1
TS1008	.0349	79.4	97.9	22.5	12.8	55.1	74.2	214.1
TS1009	.0348	78.7	96.3	24.6	14.0	53.9	75.1	216.5
TS1010	.0350	79.8	97.2	23.2	13.2	55.8	74.3	216.9

TABLE #9

Test 66: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 1000 HOURS AT 800°F, 4000 psig ARGON

NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS1006	.0355	.001	65	149.6
NS1007	.0358	.001	65	150.7
NS1008	.0354	.001	65	150.7
NS1009	.0362	.001	65	148.2
NS1010	.0361	.001	65	149.7

TABLE #10

Test 67: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 856 HOURS AT 800°F, 4000 psig H₂

AND COAL SLURRY ENVIRONMENT

AND STRESS = $24.7 \pm 1.6 \text{ ksi}$

SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in ²)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS1011	.0334	79.6	96.4	22.5	13.6	57.8	73.6	218.7
TS1012	.0336	78.6	95.5	23.6	13.7	54.2	76.2	227.2
TS1013	.0345	78.3	94.5	24.1	14.3	53.3	76.3	225.2
TS1014	.0344	76.5	91.9	25.0	14.9	51.6	76.7	221.5

TABLE #11

Test 68: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 856 HOURS AT 800°F, 4000 psig H₂

AND COAL SLURRY ENVIRONMENT

AND STRESS = 24.8 ± 1.5 ksi

NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS1011	.0352	.001	65	141.5
NS1012	.0357	.00075	65	143.1
NS1014	.0355	.00075	65	145.1
NS1015	.0355	.001	65	146.1

TABLE #12

Test 69: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 856 HOURS AT 800°F , 4000 psig H_2
 AND COAL SLURRY ENVIRONMENT
 SMOOTH BAR TENSILE SAMPLES

Sample #	Gage Area (in^2)	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elong. (%)	Engr. Fracture Stress (ksi)	Reduction Area (%)	True Fracture Stress (ksi)
TS1016	.0349	79.1	96.6	24.7	14.5	54.4	75.2	219.4
TS1017	.0344	78.5	95.9	23.9	13.7	53.8	75.3	217.8
TS1018	.0347	79.3	97.1	24.1	14.4	56.2	74.6	221.0
TS1020	.0348	77.6	94.8	24.0	14.7	53.9	75.6	220.7

TABLE #13

Test 70: T=72°F P=1ATM AIR $\dot{\epsilon}=.05 \text{ min.}^{-1}$

HISTORY: MATERIAL EXPOSED FOR 856 HOURS AT 800°F, 4000 psig H₂
 AND COAL SLURRY ENVIRONMENT
 NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS1016	.0360	.001	65	147.5
NS1017	.0356	.001	65	147.5
NS1018	.0356	.0125	65	145.8
NS1019	.0358	.001	65	145.3

TABLE #14

Comparison of Room Temperature Tensile Properties of A387-74A-Gr. 22-C1. 2 **

	0.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elongation (%)	RA (%)	Notch Tensile (ksi) Strength
ASTM Code Specifications	45.0 min.	75.0 to 100.0	22% min.	-	45 min.	-
ASTM Specification Verification Tests (Longitudinal)	78.3 (5)*	94.2 (5)	22.5 (5)	9.3 (5)	72.5 (5)	146.0 (5)
ASTM Specification Verification Tests (Transverse)	74.2 (4)	94.5 (4)	22.5 (4)	B.H (4)	61.0 (4)	174.8 (4)
Base Data Tests	78.7 (10)	95.6 (10)	24.7 (10)	14.2 (10)	75.5 (10)	148.2 (10)
Exposed Sample Tests (500°F, 2000 Psig Argon)	79.6 (10)	96.4 (9)	24.5 (10)	14.3 (10)	75.8 (10)	148.6 (10)
Exposed Sample Tests (900°F, 2000 Psig Argon)	78.2 (10)	95.7 (10)	24.3 (10)	14.0 (10)	75.0 (10)	147.9 (10)
Exposed Sample Tests (500°F, 4000 Psig Argon)	80.0 (10)	96.8 (10)	23.7 (10)	13.6 (10)	75.0 (10)	146.4 (10)
Exposed Sample Tests (900°F, 4000 Psig Argon)	79.2 (10)	97.2 (10)	23.5 (10)	12.9 (10)	74.7 (10)	146.6 (10)
Stress Exposed Sample Tests (500°F, 2000 Psig Argon, 46.1 ± 6.0 ksi)	79.2 (9)	95.8 (10)	22.9 (10)	13.1 (10)	74.7 (10)	147.0 (10)
Stress Exposed Sample Tests (900°F, 2000 Psig Argon, 19.1 ± 1.3 ksi)	78.2 (10)	94.6 (10)	22.8 (10)	13.5 (10)	74.4 (9)	147.8 (10)
Stress Exposed Sample Tests (900°F, 2000 Psig Argon, 41.4 ± 7.2 ksi)	81.3 (10)	95.2 (9)	22.4 (9)	12.0 (10)	73.9 (10)	149.2 (9)
Stress Exposed Sample Tests (500°F, 4000 Psig Argon, 48.6 ksi)	80.7 (10)	94.7 (10)	22.1 (10)	11.9 (10)	75.1 (9)	148.0 (10)
Stress Exposed Sample Tests (900°F, 4000 Psig Argon, 19.1 ± 1.3 ksi)	77.1 (10)	94.3 (10)	23.6 (10)	13.9 (10)	74.6 (10)	145.0 (10)
Exposed Sample Tests (500°F, 2000 Psig H ₂ Coal Slurry Environment)	79.8 (10)	95.5 (10)	24.3 (10)	14.2 (10)	75.0 (9)	144.0 (9)
Exposed Sample Tests (900°F, 2000 Psig H ₂ Coal Slurry Environment)	78.7 (9)	95.6 (9)	23.2 (9)	13.6 (10)	74.6 (9)	143.7 (10)
Exposed Sample Tests (500°F, 4000 Psig H ₂ Coal Slurry Environment)	79.4 (10)	95.3 (10)	24.6 (10)	14.6 (10)	74.8 (10)	147.5 (10)
Exposed Sample Tests (800°F, 4000 Psig H ₂ Coal Slurry Environment)	77.2 (8)	94.3 (10)	24.5 (10)	14.9 (10)	74.6 (10)	147.1 (10)
Stress Exposed Sample Tests (500°F, 2000 Psig H ₂ Coal Slurry Environment, 24.0 ± 1 ksi)	77.9 (9)	94.1 (10)	22.6 (9)	13.5 (9)	74.9 (10)	146.8 (5)
Stress Exposed Sample Tests (800°F, 2000 Psig H ₂ Coal Slurry Environment, 22.5 ± 1.7 ksi)	76.1 (8)	91.5 (10)	22.9 (10)	14.0 (10)	75.7 (10)	143.4 (10)
Stress Exposed Sample Tests (500°F, 4000 Psig H ₂ Coal Slurry Environment, 27.2 ± .5 ksi)	79.6 (10)	95.3 (10)	24.3 (10)	13.8 (10)	75.5 (10)	147.1 (7)
Stress Exposed Sample Tests (800°F, 4000 Psig H ₂ Coal Slurry Environment, 22.7 ± 1 ksi)	77.7 (10)	93.8 (10)	23.4 (10)	13.9 (10)	75.8 (10)	-

* The number in () following each value indicates the number of samples averaged.

** All exposures were for 168 hours.

TABLE #15

Comparison of Room Temperature Tensile Properties of A387-74A-Gr.22-C1.2

	.2% YS (ksi)	UTS (ksi)	Total Elongation (%)	Uniform Elongation (%)	RA (%)	Notch Tensile Strength (ksi)
Base Data Tests	78.7 (10)***	95.6 (10)	24.7 (10)	14.2 (10)	75.5 (10)	148.2 (10)
Stress Exposed [*] Sample Tests (800°F, 4000 psig Argon, 24.3 ± 2.0 ksi)	79.5 (5)	97.2 (5)	24.7 (5)	13.9 (5)	74.9 (4)	149.1 (5)
Exposed [*] Sample Tests (800°F, 4000 psig Argon)	79.1 (5)	97.0 (5)	24.3 (5)	14.0 (5)	74.7 (5)	149.8 (5)
Stress Exposed ^{**} Sample Tests (800°F, 4000 psig H ₂ , Coal Slurry Environ- ment, 24.7 ± 1.6 ksi)	78.3 (4)	94.6 (4)	23.8 (4)	14.1 (4)	76.4 (3)	144.0 (4)
Exposed ^{**} Sample Tests (800°F, 4000 psig H ₂ , coal slurry environment)	78.6 (4)	96.1 (4)	24.2 (4)	14.3 (4)	75.2 (4)	146.5 (4)

* Exposed for 1000 hours.

** Exposed for 856 hours.

*** Number in () stands for number of samples averaged.

TABLE #16

Test	Type	Exposure Conditions			Time (HRS.)	Exposure Stress (KSI) ^A	Test Conditions ^{A,B}			Number of samples
		Temp. (F°)	Pressure (Psig)	Environment			Temp. (F°)	Pressure (Psig)	Environment	
1	Smooth	-	-	-	-	-	Ambient	Ambient	Air	10
2	Notched	-	-	-	-	-	Ambient	Ambient	Air	10
3	Smooth	-	-	-	-	-	500	Ambient	Air	10
4	Notched	-	-	-	-	-	500	Ambient	Air	10
5	Smooth	-	-	-	-	-	900	Ambient	Argon	10
6	Notched	-	-	-	-	-	900	Ambient	Argon	10
7	Smooth	500	2000	Argon	168	0	Ambient	Ambient	Air	10
8	Notched	500	2000	Argon	168	0	Ambient	Ambient	Air	10
9	Smooth	900	2000	Argon	168	0	Ambient	Ambient	Air	10
10	Notched	900	2000	Argon	168	0	Ambient	Ambient	Air	10
11	Smooth	500	4000	Argon	168	0	Ambient	Ambient	Air	10
12	Notched	500	4000	Argon	168	0	Ambient	Ambient	Air	10
13	Smooth	900	4000	Argon	168	0	Ambient	Ambient	Air	10
14	Notched	900	4000	Argon	168	0	Ambient	Ambient	Air	10
15	Smooth	500	2000	Argon	168	48 ^C	Ambient	Ambient	Air	10
16	Notched	500	2000	Argon	168	48 ^f	Ambient	Ambient	Air	10
17	Smooth	900	2000	Argon	168	25 ^C	Ambient	Ambient	Air	10
18	Notched	900	2000	Argon	168	25 ^C	Ambient	Ambient	Air	10
19	Smooth	500	4000	Argon	168	48 ^f	Ambient	Ambient	Air	10
20	Notched	500	4000	Argon	168	48 ^C	Ambient	Ambient	Air	10
21	Smooth	900	4000	Argon	168	25 ^C	Ambient	Ambient	Air	10
22	Notched	900	4000	Argon	168	25 ^C	Ambient	Ambient	Air	10

A All loading is in tension

B Constant cross-head rate of 0.05 inch/min.

C Loading via precompressed rings

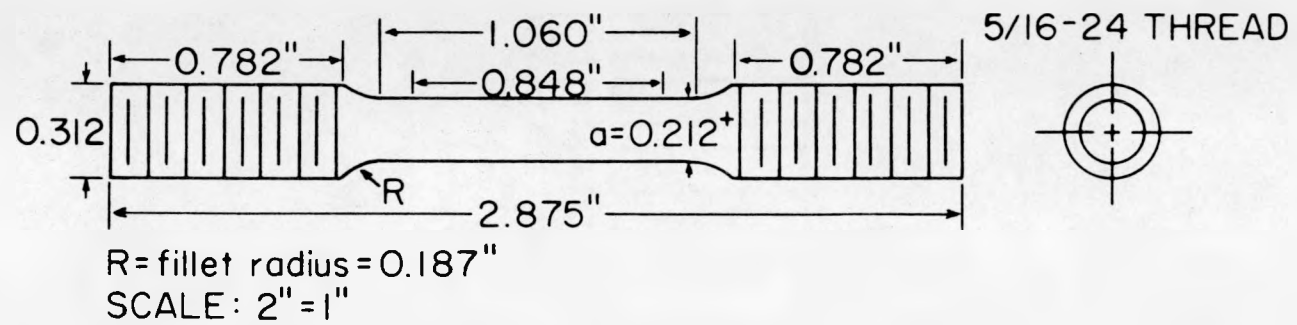
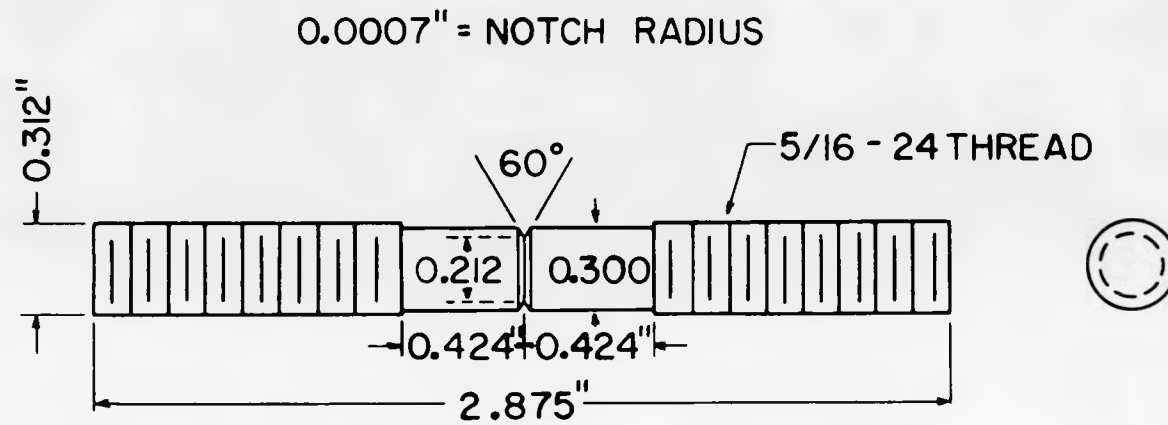


Fig. 1: Base Property Determination Smooth Tensile Sample.



SCALE: 2"=1"

Fig. 2: Base Property Determination Notched Tensile Sample.

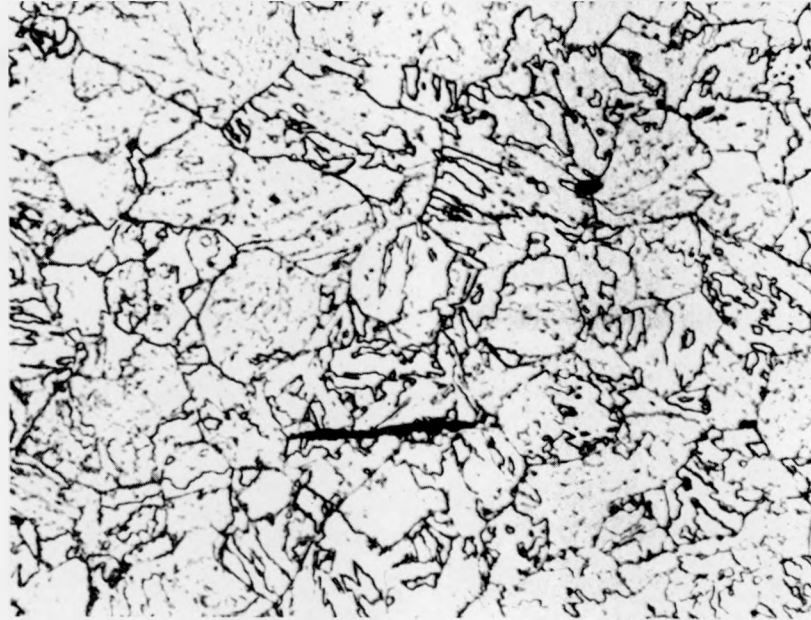


Fig. 3: Microstructure of 2 1/4 Cr-1 Mo steel. Specimen was exposed for 168 hours at 800°F in a coal slurry in 4000 Psig hydrogen gas. (400X) = Magnification.

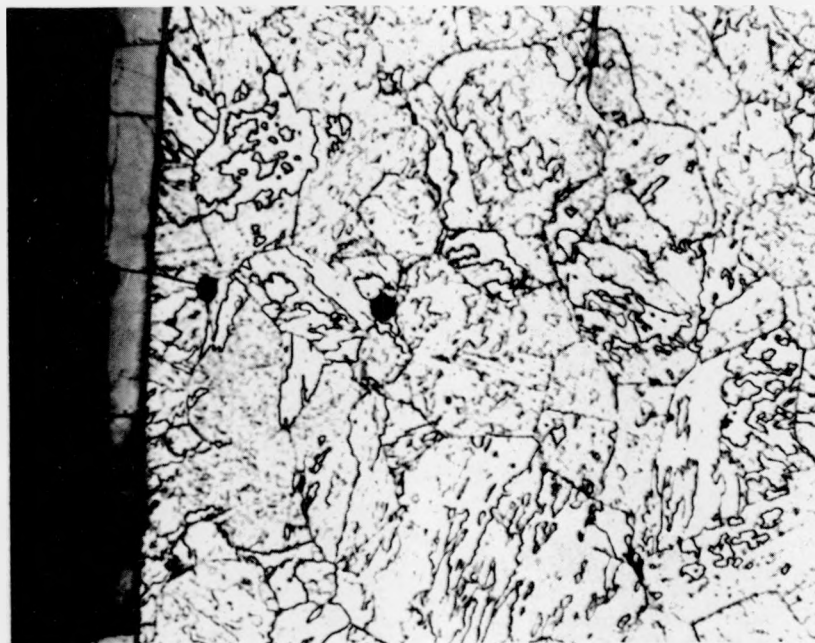


Fig. 4: Microstructure of 2 1/4 Cr-1 Mo steel showing spherical features near surface of sample. Specimen was exposed for 168 hours at 500°F in a coal slurry in 4000 Psig hydrogen and stress of $27.2 \pm .5$ ksi. (400X) = Magnification.

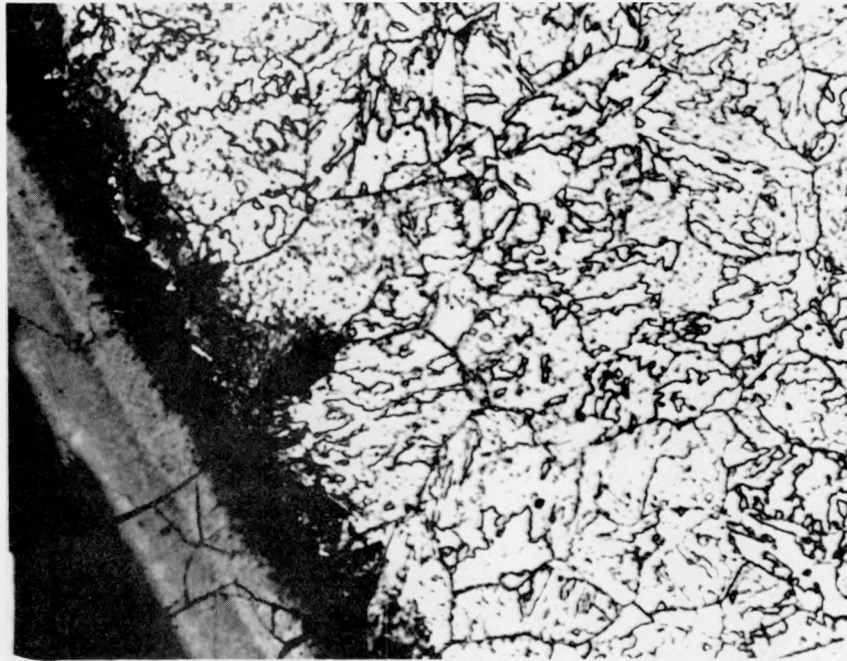


Fig. 5: Microstructure of 2 1/4 Cr-1 Mo steel showing irregular shaped features near surface of sample. Specimen was exposed for 168 hours at 800°F in a coal slurry in 2000 Psig hydrogen and stress of 22.5 ± 1.7 ksi. (400X) = Magnification.

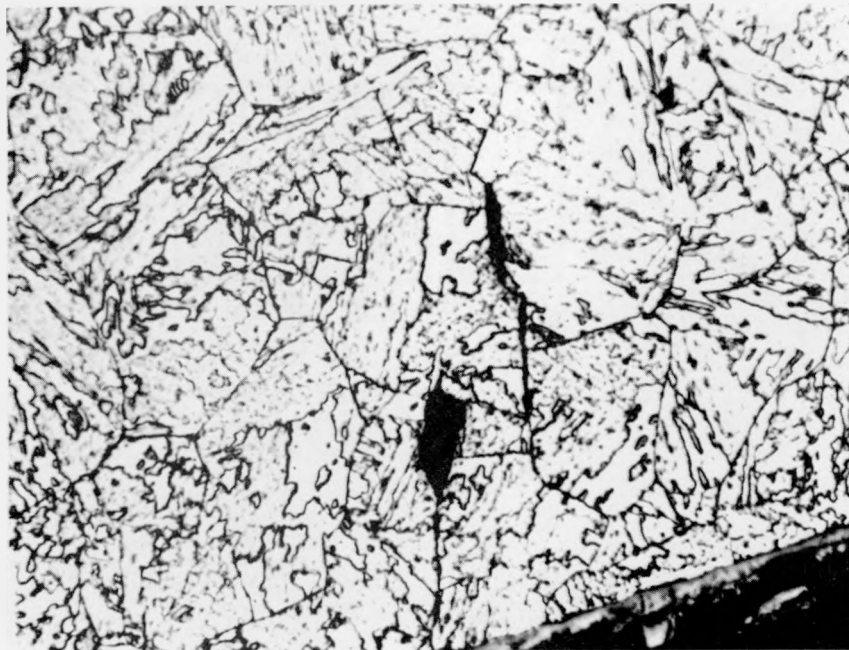


Fig. 6: Microstructure of 2 1/4 Cr-1 Mo steel showing irregular shaped features near surface of sample. Specimen was exposed for 168 hours at 500°F in a coal slurry in 2000 Psig hydrogen and stress of 24.0 ± 1.0 . (400X) = Magnification.

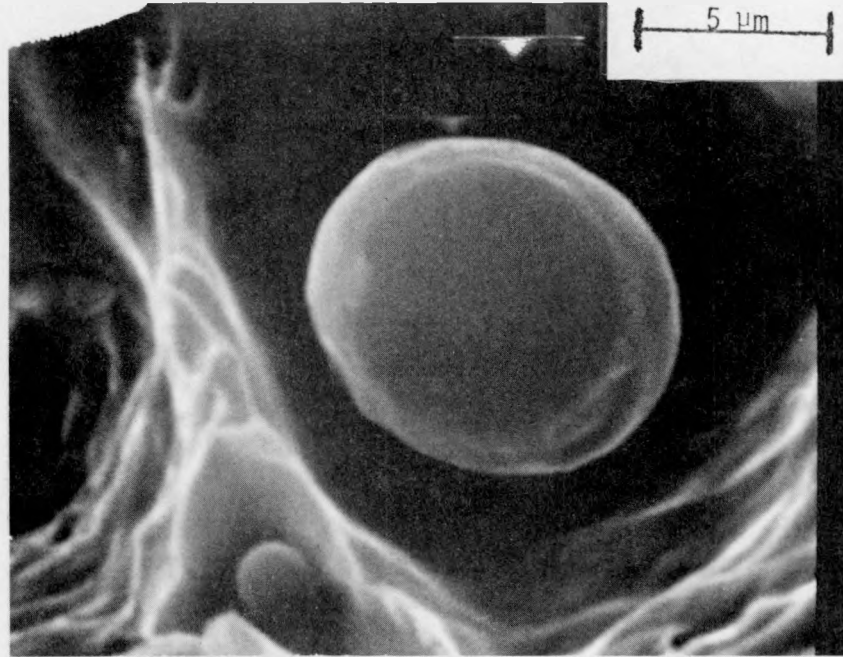


Fig. 7: Scanning electron micrograph of a MnS particle at the base of a large microvoid in the fracture surface of an unexposed 2 1/4 Cr-1 Mo notch tensile sample.

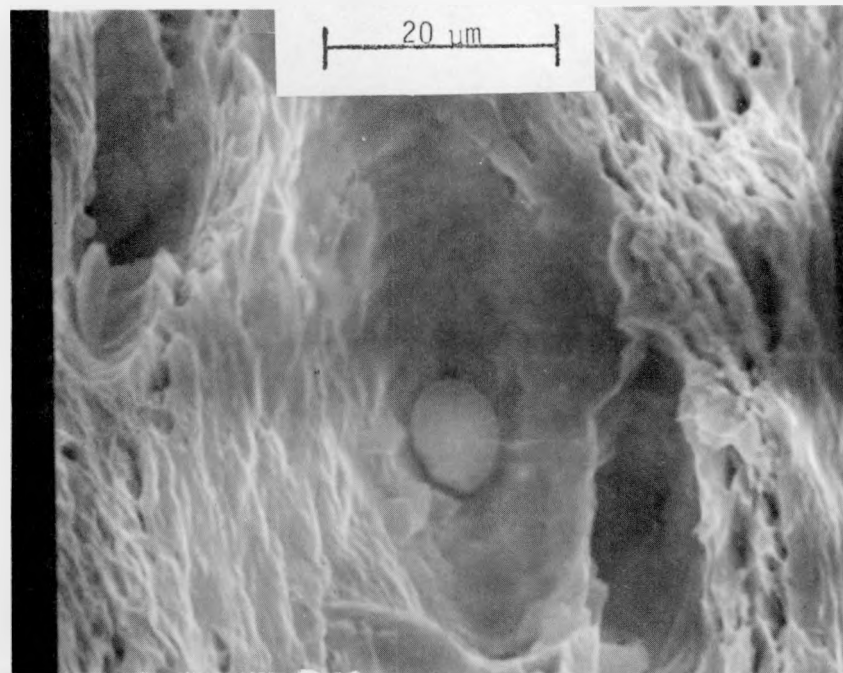


Fig. 8: Scanning electron micrograph of a MnS particle lying in a large microvoid in the fracture surface of an unexposed 2 1/4 Cr-1 Mo notch tensile sample surrounded by characteristic ductile dimple type fracture.

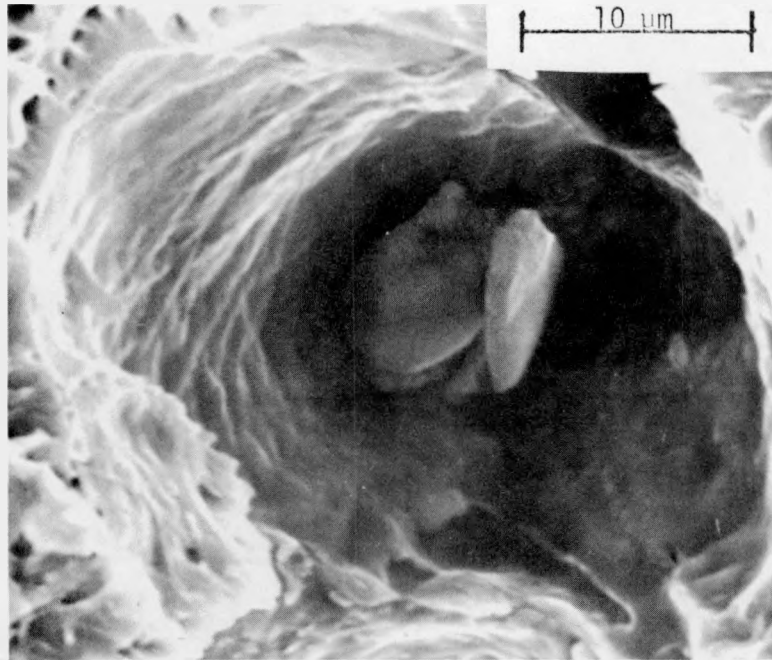


Fig. 9: Scanning electron micrograph of a MnS particle in the fracture surface of a 2 1/4 Cr-1 Mo notched-bar tensile sample exposed for 856 hours to an 800°F coal slurry in 4000 Psig H₂.

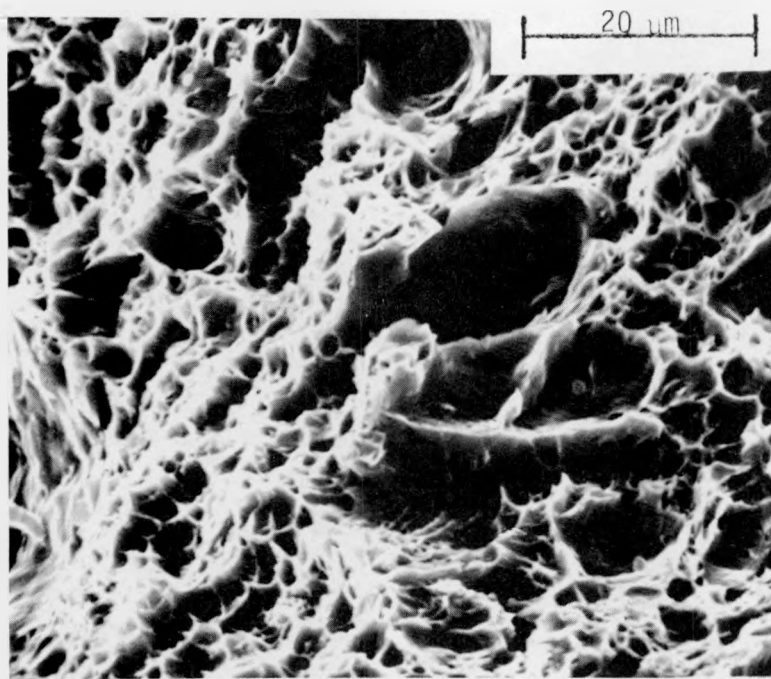


Fig. 10: Characteristic ductile dimple type fracture exhibited by an unexposed smooth-bar tensile sample of 2 1/4 Cr-1 Mo.

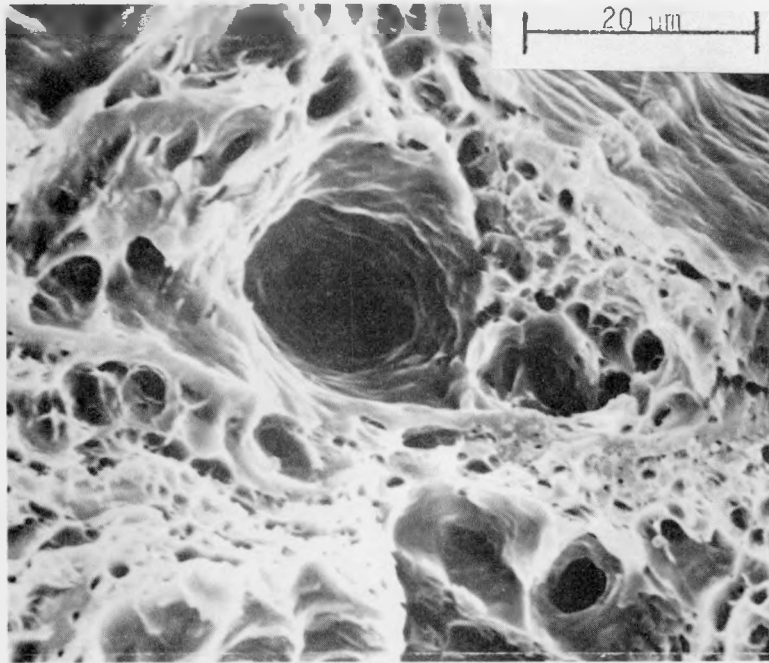


Fig. 11: Large Microvoid observed in the fracture surface of a 2 1/4 Cr-1 Mo unexposed smooth-bar tensile sample.

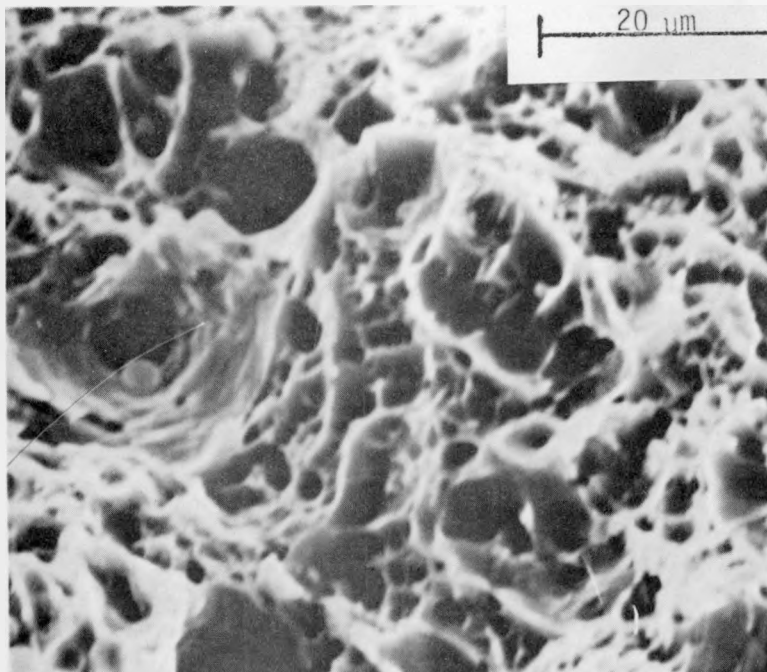


Fig. 12: Fracture surface of a 2 1/4 Cr-1 Mo smooth-bar tensile sample exposed to a coal slurry for 856 hours at 800°F in 4000 Psig H₂. Displays characteristic ductile dimple type fracture.

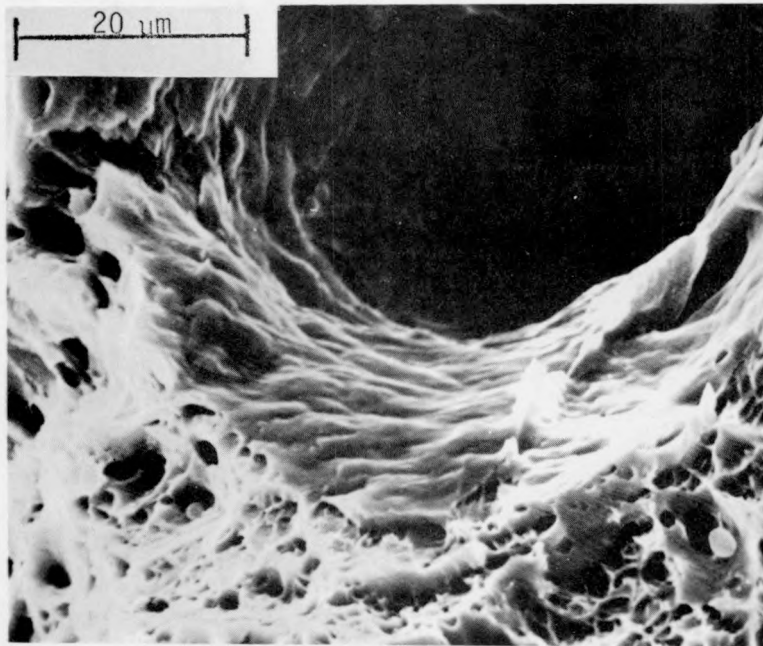
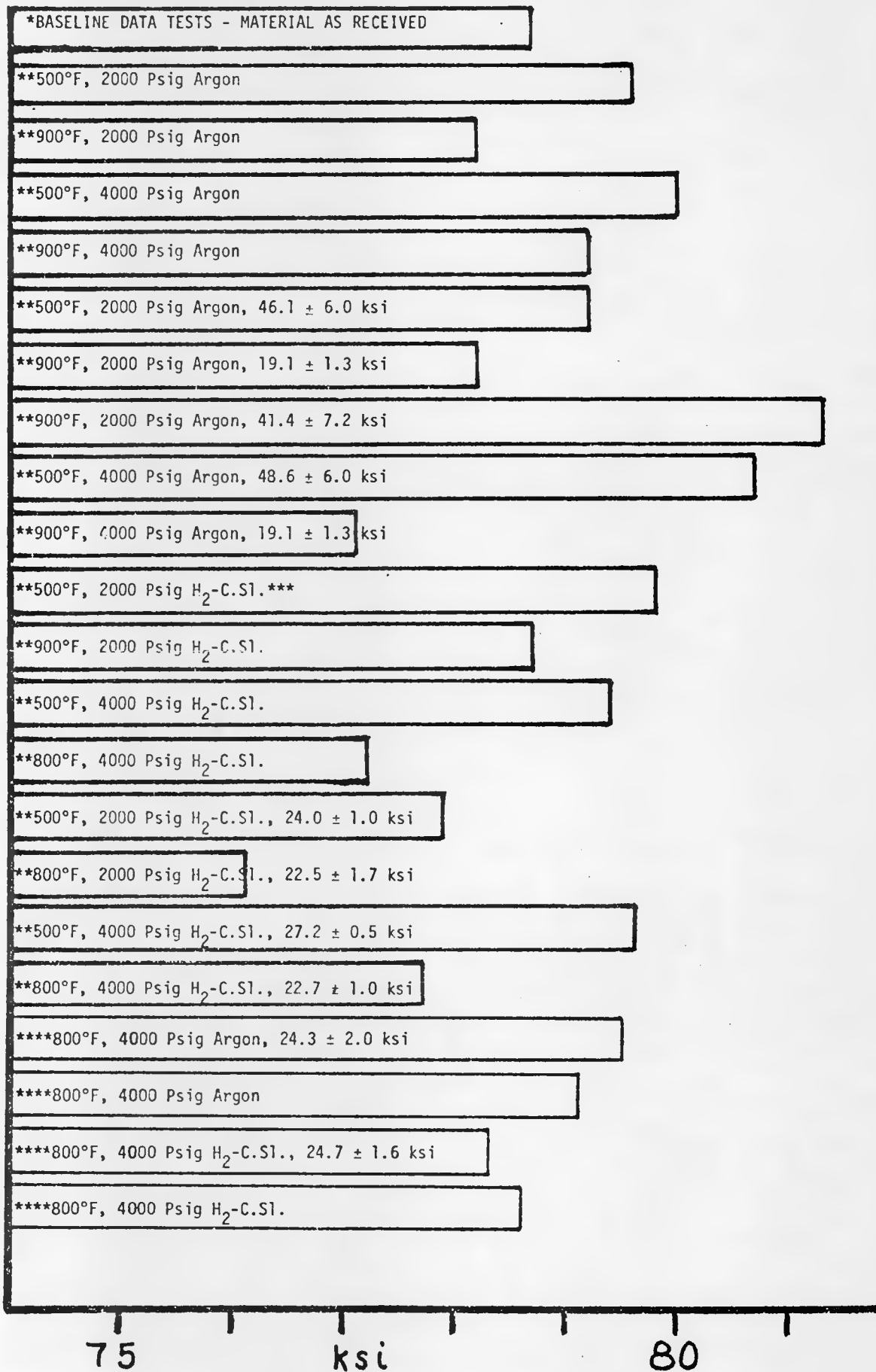


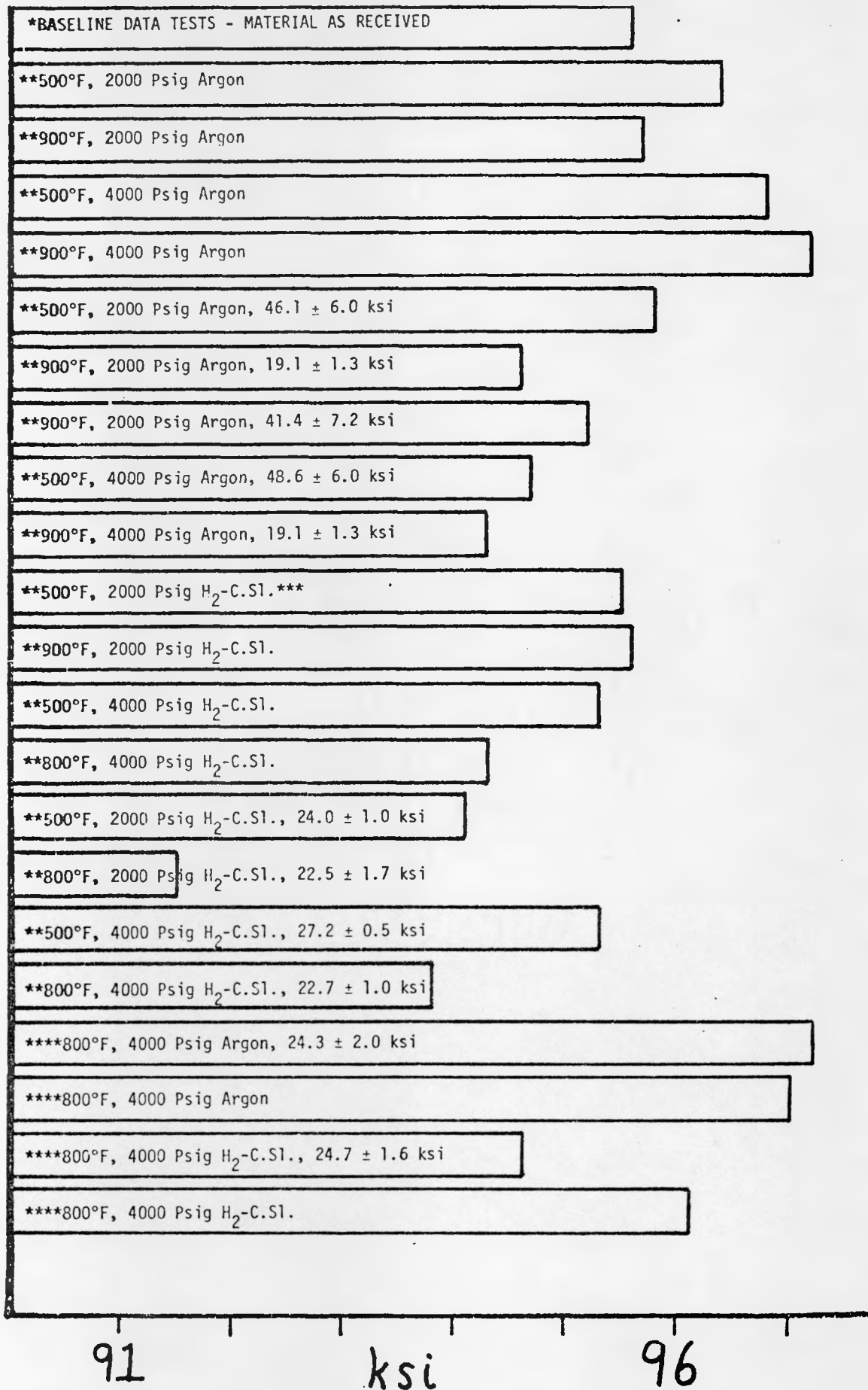
Fig. 13: Very large microvoid observed in the fracture surface of a 2 1/4 Cr-1 Mo smooth-bar tensile sample exposed to a coal slurry for 856 hours at 800°F in 4000 Psig H₂.

Fig. 14: Comparison of Room Temperature 0.2% Yield Strengths of A387-74A-Gr.22-C1.-2.



*Material as received from vendor (no exposure).
 **168 hour exposures at conditions given.
 ***Hydrogen gas plus coal-slurry environment.
 ****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

Fig. 15: Comparison of Room Temperature UTS of A387-74A-Gr.22-C1.2.



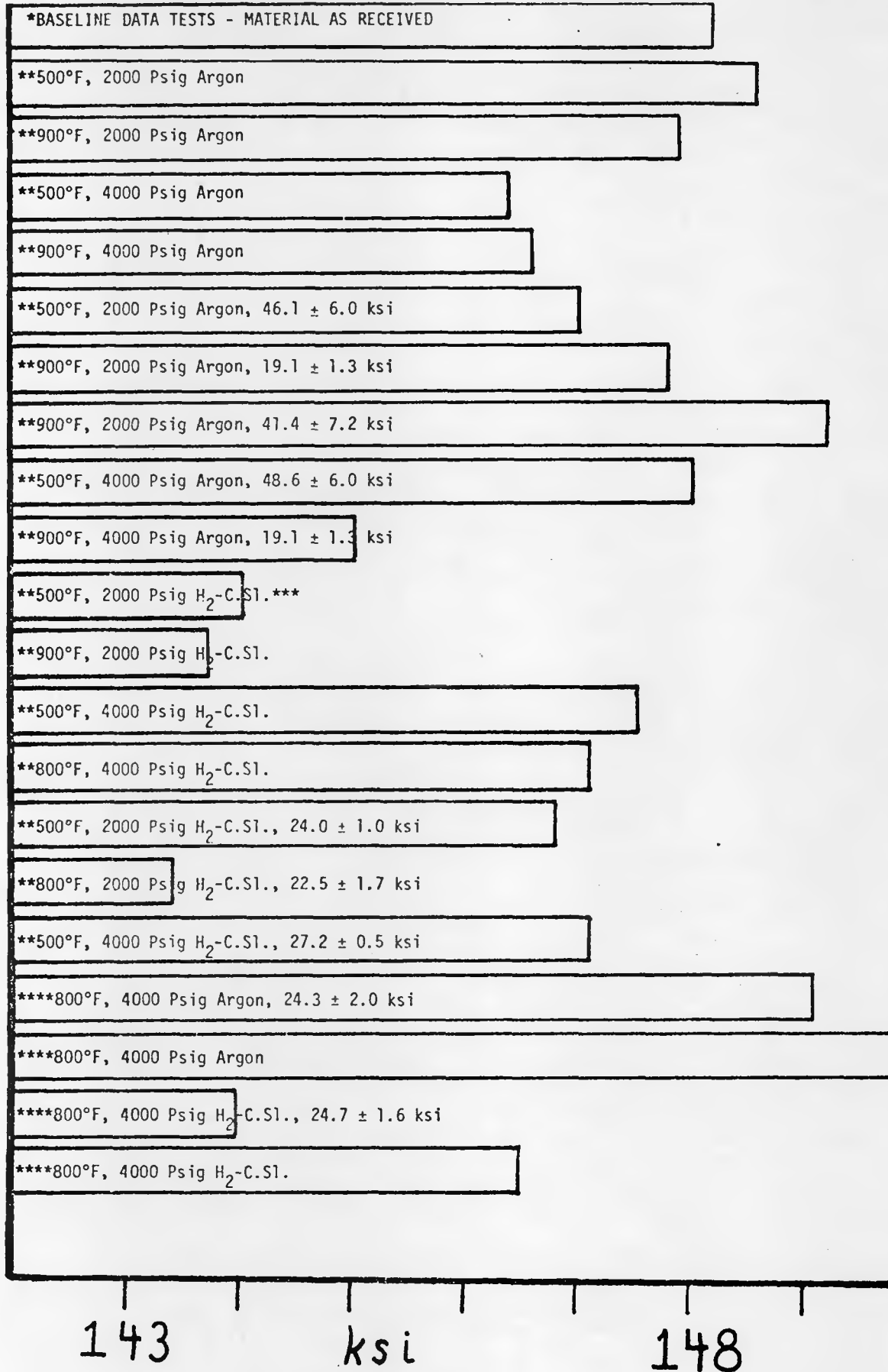
*Material as received from vendor (no exposure).

**168 hour exposures at conditions given.

***Hydrogen gas plus coal-slurry environment.

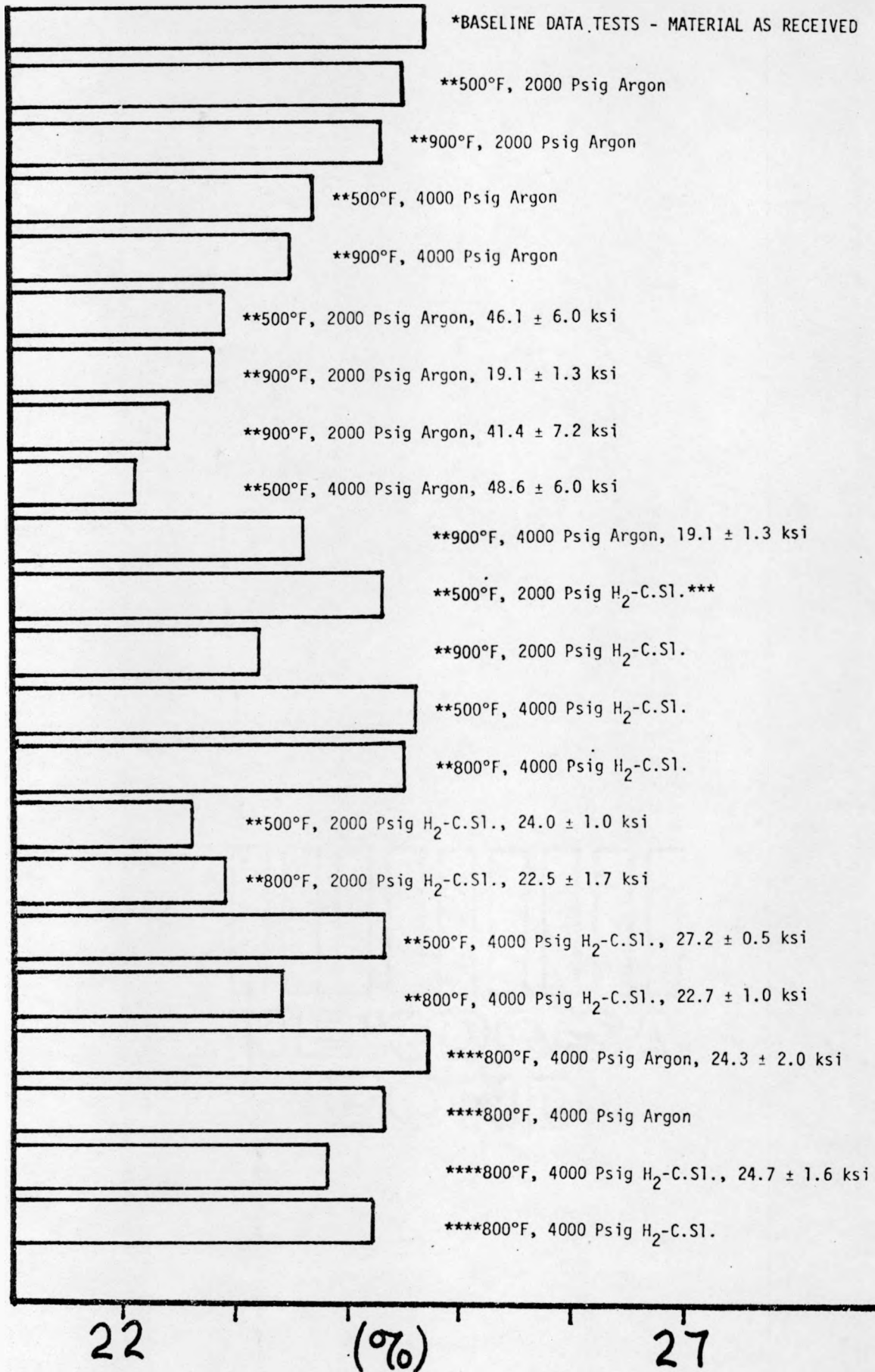
****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

Fig. 16: Comparison of Room Temperature Notch Tensile Strengths of A387-74A-Gr.22-C1.2.



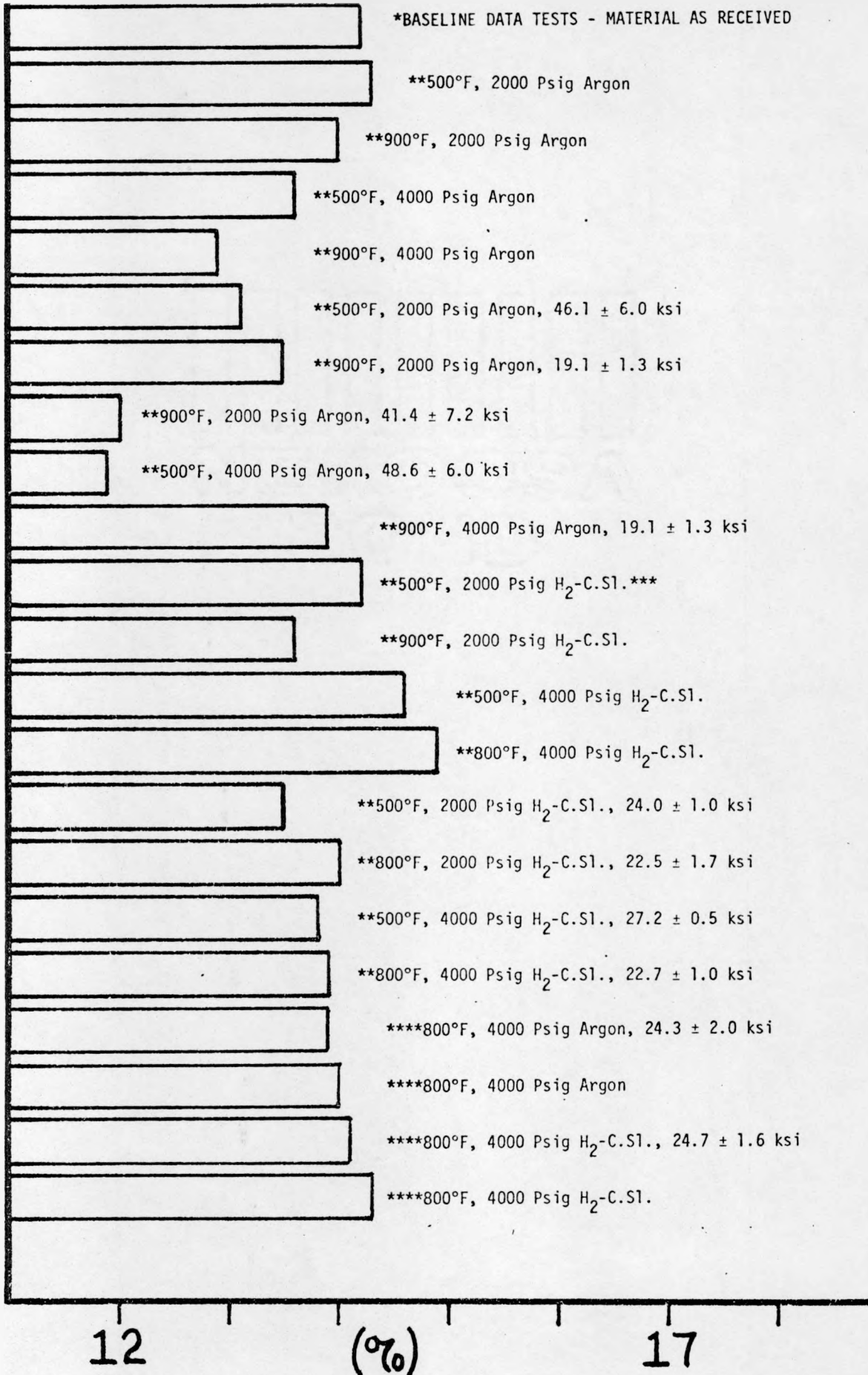
*Material as received from vendor (no exposure).
 **168 hour exposures at conditions given.
 ***Hydrogen gas plus coal-slurry environment.
 ****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

Fig. 17: Comparison of Room Temperature Total Elongation of A387-74A-Gr.22-C1.2.



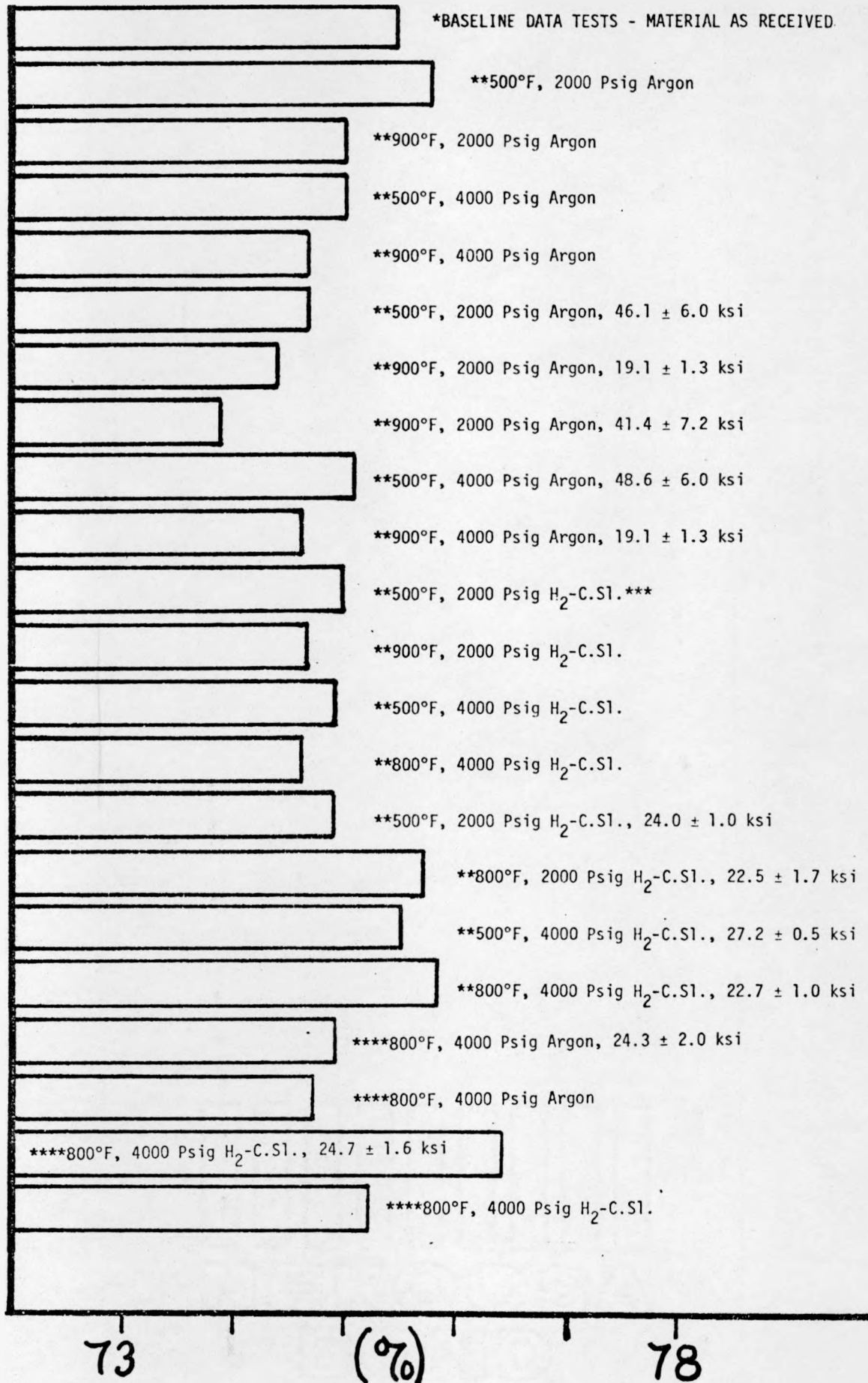
*Material as received from vendor (no exposure).
 **168 hour exposures at conditions given.
 ***Hydrogen gas plus coal-slurry environment.
 ****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

Fig. 18: Comparison of Room Temperature Uniform Elongation of A387-74A-Gr.22-C1.2.



*Material as received from vendor (no exposure).
 **168 hour exposures at conditions given.
 ***Hydrogen gas plus coal-slurry environment.
 ****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

Fig. 19: Comparison of Room Temperature Reduction in Area of A387-74A-Gr.22-C1.2.



*Material as received from vendor (no exposure).
 **168 hour exposures at conditions given.
 ***Hydrogen gas plus coal-slurry environment.
 ****1000 hours (Argon) or 856 hours (coal-slurry) exposures at conditions given.

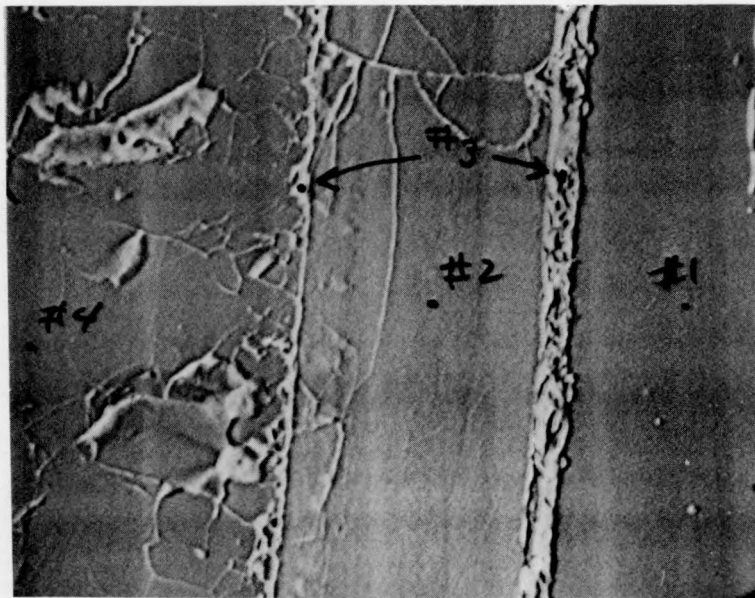


Fig. 20: Scanning electron micrograph of surface region of A387 after an 856 hour stressed (24.7 ± 1.6 ksi) exposure to an 800°F , 4000 psig H_2 -coal-slurry environment. #1 is base metal, the others are surface scale layers. Magnification=580X.

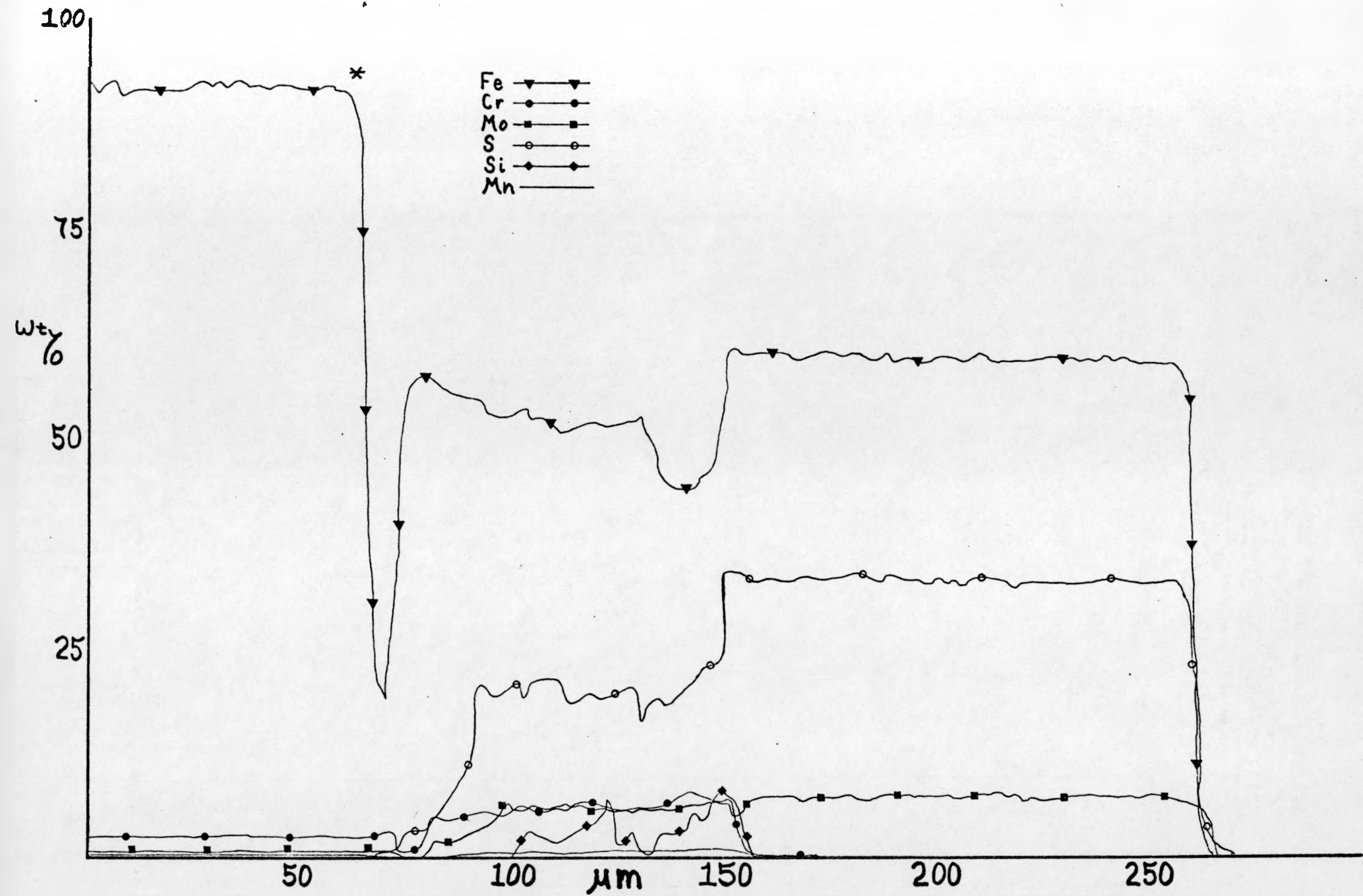


Fig. 21: Weight % concentration profile of Fe, Cr, Mo, S, Si and Mn as determined by electron microprobe analyses when scanning from the base metal to the outside of the surface scale. * indicates base metal-innermost scale layer interface. Corresponds to micrograph of Fig. 20.

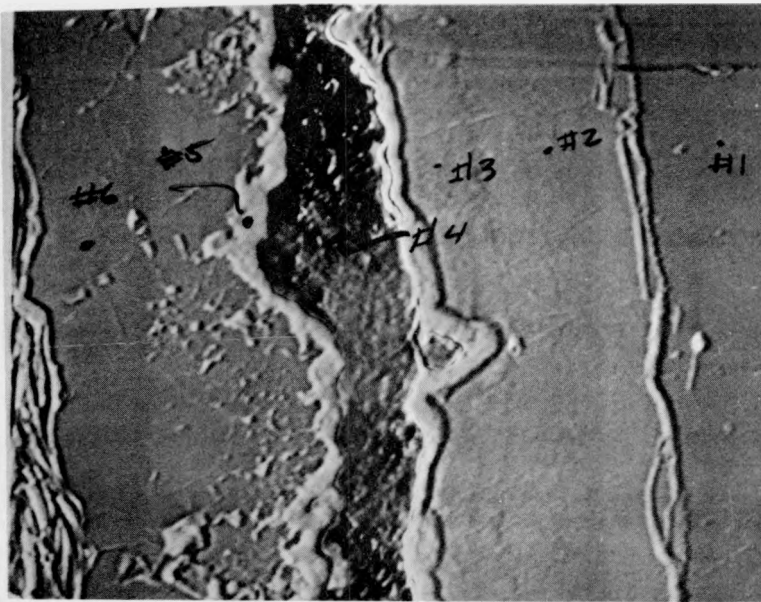


Fig. 22: Scanning electron micrograph of surface region of A387 after an 856 hour exposure to an 800°F, 4000 psig H₂-coal-slurry environment. #1 is base metal, #4 is a crack, the others are surface scale layers. Magnification=400X.

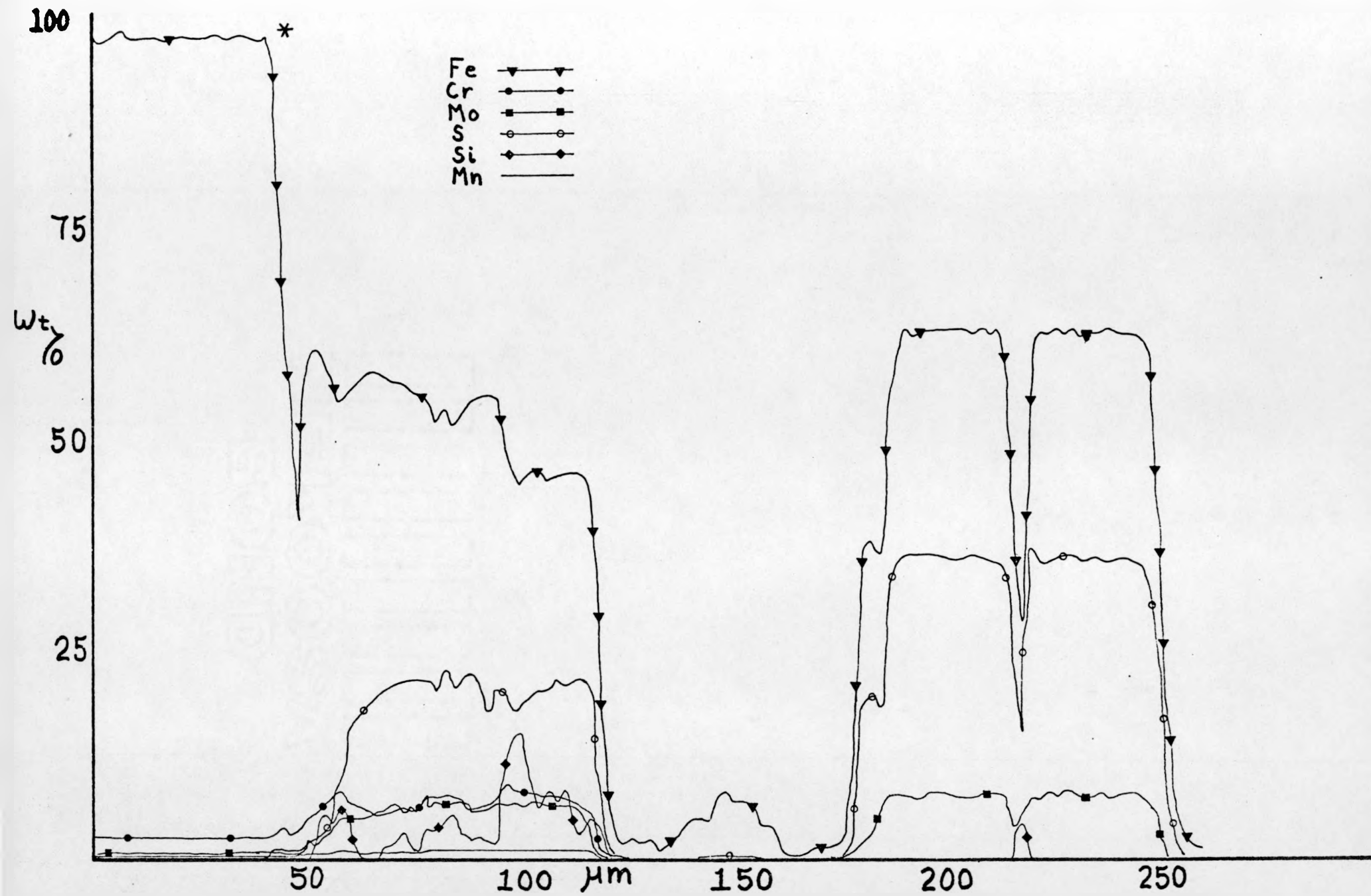


Fig. 23: Weight % concentration profile of Fe, Cr, Mo, S, Si and Mn as determined by electron microprobe analyses when scanning from the base metal to the outside of the surface scale. * indicates base metal-innermost scale layer interface. Corresponds to micrograph of Fig. 22.

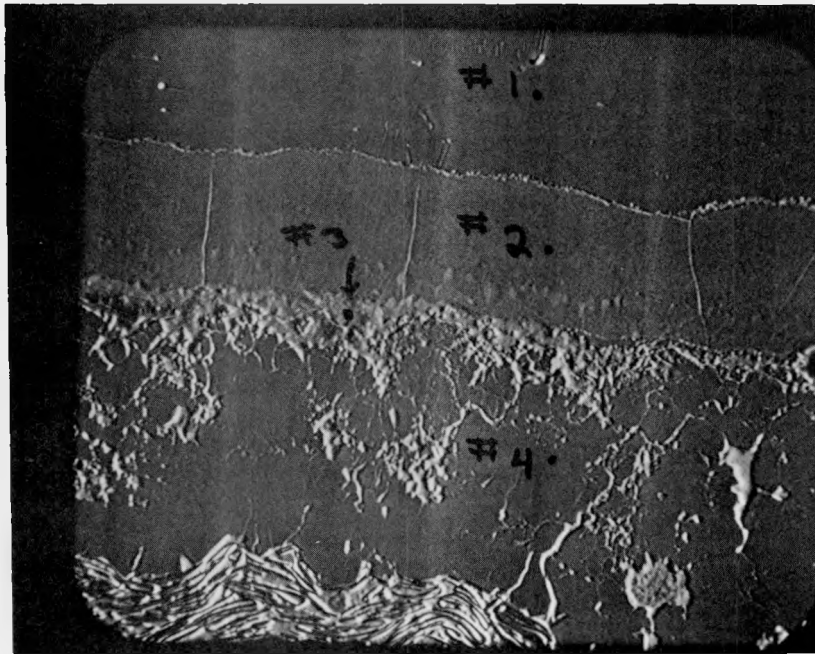


Fig. 24: Scanning electron micrograph of surface region of A387 after an 856 hour exposure to an 800°F, 4000 psig H₂-coal-slurry environment. #1 is base metal, the others are surface scale layers. Magnification=349X.

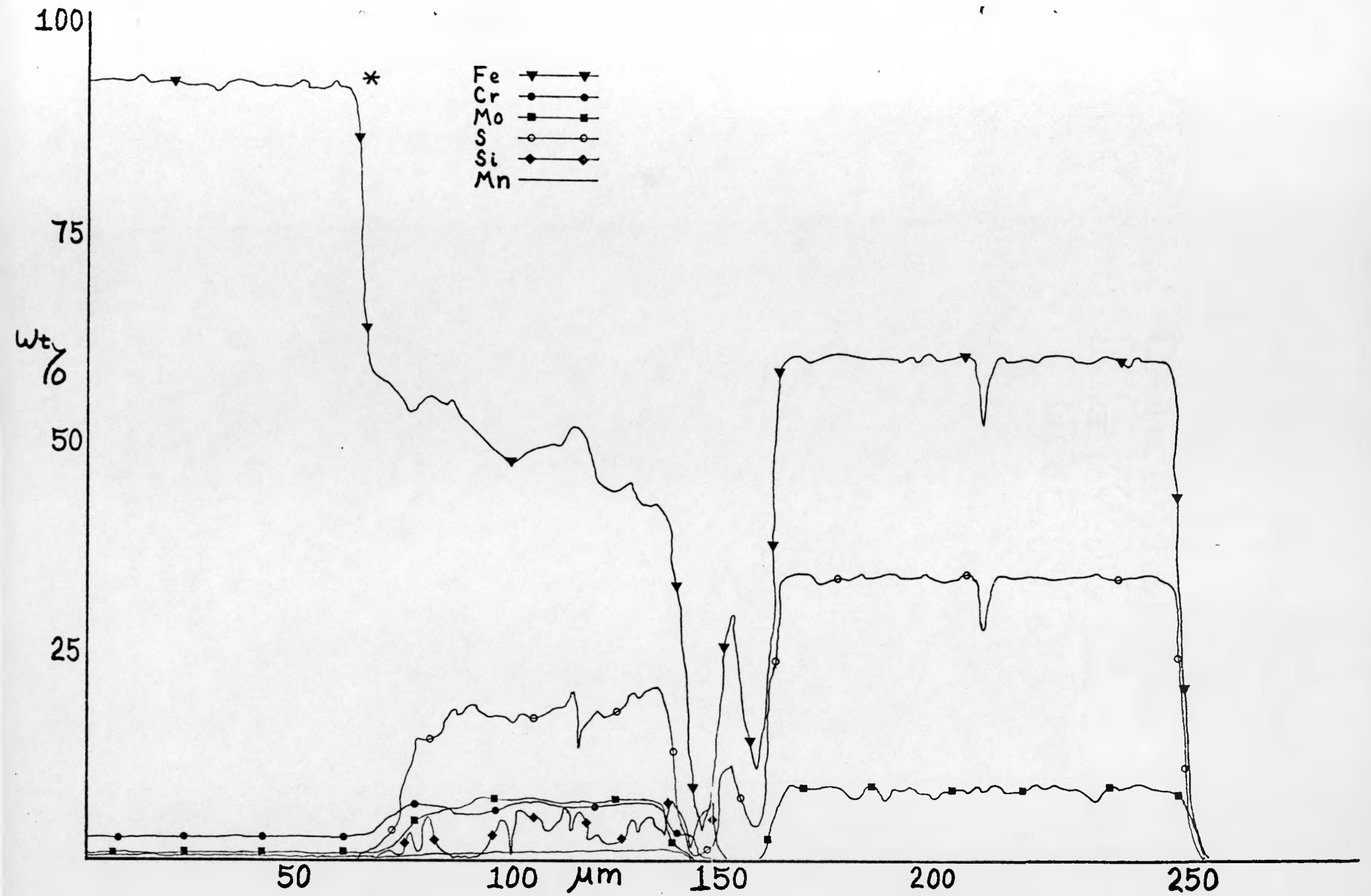
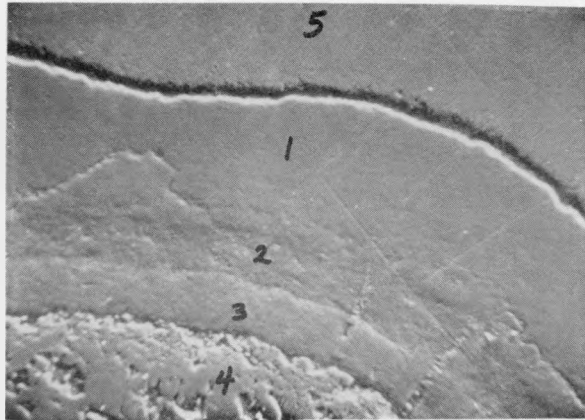


Fig. 25: Weight % concentration profile of Fe, Cr, Mo, S, Si and Mn as determined by electron microprobe analyses when scanning from the base metal to the outside of the surface scale. * indicates basic metal-innermost scale layer interface. Corresponds to micrograph of Fig. 24.

Fig. 26: EDAX point source x-ray fluorescence analyses of A387 exposed for 856 hours to an 800°F, 4000 psig H₂-coal-slurry environment.

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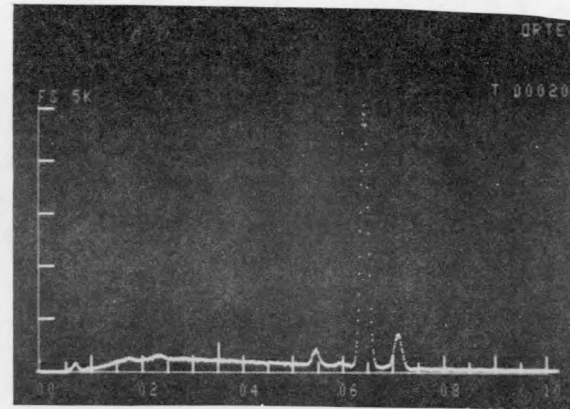


880X

TS 1019 A

Fig. 26, continued on next page

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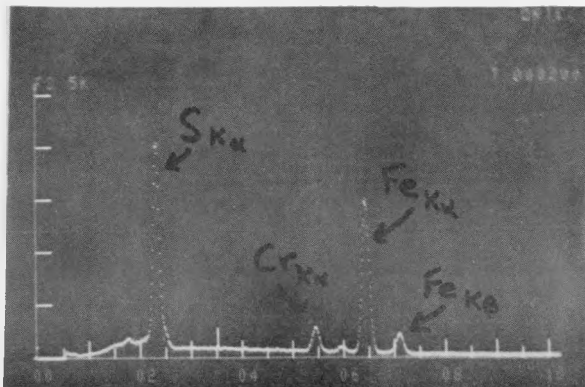


TS 1019 A

Phase 5

Region 5: Base metal

7-17-79

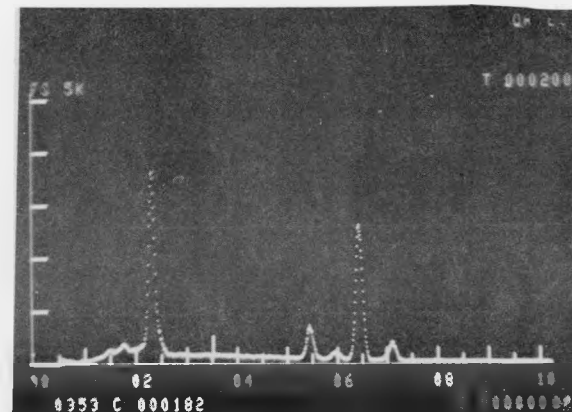


TS 1019 A

Phase 1

Region 1: 1st scale layer

7-17-79

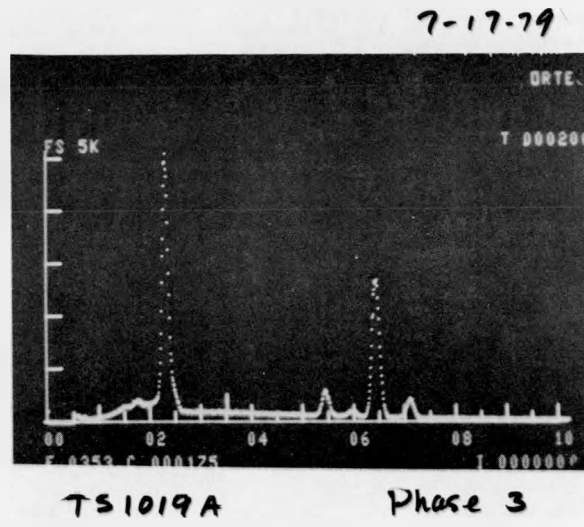


TS 1019 A

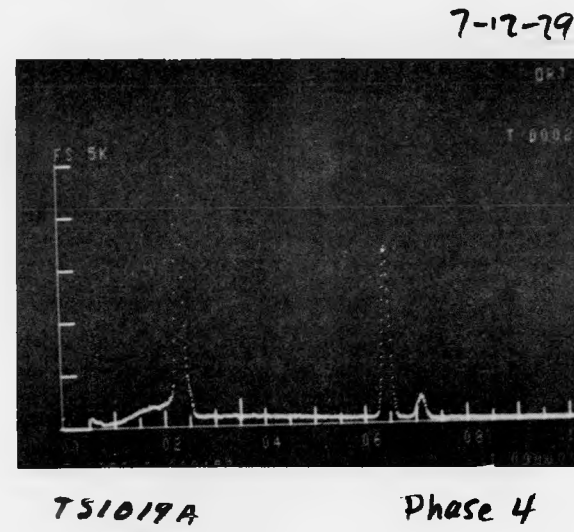
Phase 2

Region 2: Different relief zone of 1st scale layer

Fig. 26: Continued



Region 3: High sulfur region of 1st scale layer



Region 4: Outer scale layer

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