

Alloy Evaluation for Fossil Fuel Process Plants (Liquefaction)

Quarterly Report for

Period

1 April, 1978 through 30 June, 1978

C. M. Woods and T. E. Scott

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FOREWORD

This report covers work performed during the period 1 April, 1978 through 30 June, 1978. The work was administered by the Division of Materials and Exploratory Research with Mr. Wate Bakker 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 have been determined for A387-74A-Gr. 22-C1. 2 steel after exposure to a coal slurry environment at various temperatures and H_2 pressures. Comparison with base property data revealed that no degradation of ambient temperature mechanical properties occurred.

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.

Progress Summary

I. 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. All data were calculated from the stress-strain 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.

Smooth-bar and notched-bar tensile tests were carried out under ambient test conditions on samples which had been subjected to 168 hour emersions in

a vessel containing coal slurry and hydrogen gas. Test conditions (39-46, Table 10) include exposures to 2000 and 4000 Psig H_2 gas at temperatures of 500, 800 and 900°F. 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 insol, 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 H_2 pressure vessels for subsequent pressure and temperature equilibration. The samples were exposed for 168 hours during which time the H_2 pressure was controlled within \pm 100 Psig of the required pressure. The temperature fluctuations were \pm 10°F at 800°F and + 25°F to - 10°F for the 500°F tests. The 500°F runs seemed to exhibit an exothermic reaction between the coal slurry and H_2 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. The threaded portions of the samples were subsequently cleaned with a wire wheel and subsequently tensile tested with the surface film intact.

II. 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 1-8. Average values are summarized and compared with the previously determined room temperature tensile properties (Report #FE-7318-4) in Table 9. These values are statistical averages based on data certified by the 'Q' test at the 90% confidence level. It can be seen from Table 9, that the room temperature mechanical properties of A387-74A-Gr. 22-C1. 2 steel were not appreciably affected by prolonged (168 hour) exposures to the coal slurry environment under any of the test conditions run to date.

The only noted trend in the slurry exposure data is a slight increase in the uniform elongation over that of the other test conditions. This trend, however, still falls within the realm of the statistical variance of these tests.

Work Forecast

Stressed sample exposure runs (Tests 47-54, Table 10) and micro-structural investigation of samples from the various slurry exposures are presently underway. Electron microprobe and Auger analyses of the surface oxide layers will be performed. These data, along with the composition of a crystalline precipitate that forms on the pressure vessel rupture disc during the course of a run will be reported in the next report.

TABLE #1
 Test 39: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min.}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°F , 2000 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)
TS111	.0352	81.0	96.9	23.3	13.2	56.1	73.4	215.6
TS112	.0353	80.7	96.3	23.9	14.0	55.2	74.5	216.9
TS113	.0353	80.7	96.3	24.7	14.5	54.5	75.0	218.1
TS114	.0353	80.0	95.8	24.8	14.6	53.8	74.5	211.3
TS115	.0353	80.0	95.8	25.1	14.9	53.8	75.5	219.4
TS116	.0353	79.3	94.9	24.5	14.4	53.1	75.5	216.5
TS117	.0353	79.0	94.9	24.3	14.0	53.1	75.5	216.5
TS118	.0350	78.6	94.3	21.9	12.9	52.9	74.8	209.6
TS119	.0353	80.0	94.9	24.2	14.6	54.5	75.0	218.1
TS120	.0353	78.8	94.9	26.6	14.5	52.4	75.0	209.6

TABLE #2
 Test 40: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\varepsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°F , 2000 psig H_2
 AND COAL SLURRY ENVIRONMENT
 NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS111	.0362	.001	63.5	143.6
NS112	.0364	.001	64	142.9
NS113	.0361	.001	64	144.0
NS114	.0361	.001	65	144.0
NS115	.0361	.001	65	143.5
NS116	.0360	.001	64	144.7
NS117	.0360	.001	64	144.2
NS118	.0361	.001	64	144.0
NS119	.0356	.001	65	146.6
NS120	.0359	.001	63	144.8

TABLE #3
 Test 41: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 900°F , 2000 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)
TS121	.0353	78.6	95.5	24.5	14.5	55.2	74.5	216.9
TS122	.0350	78.9	95.7	23.8	14.2	54.3	74.3	211.3
TS123	.0350	78.6	95.4	22.3	13.0	55.0	74.3	214.1
TS124	.0351	78.3	96.0	24.0	14.4	55.6	74.9	221.0
TS125	.0352	78.1	95.2	22.2	12.9	54.0	74.5	211.3
TS126	.0350	79.3	96.3	23.7	13.7	55.0	73.8	210.1
TS127	.0352	78.8	95.7	24.1	13.9	54.0	74.5	211.3
TS128	.0350	77.1	92.9	20.3	12.6	55.7	74.8	221.0
TS129	.0346	78.8	95.4	22.2	13.2	55.6	75.0	222.3
TS130	.0350	78.6	95.0	22.1	13.2	54.9	74.3	213.5

TABLE #4

Test 42: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$ HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 900°F , 2000 psig H_2

AND COAL SLURRY ENVIRONMENT

NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS121	.0360	.00075	65	142.5
NS122	.0362	.001	65	143.4
NS123	.0359	.001	65	142.9
NS124	.0356	.001	65	143.8
NS125	.0362	.001	65	144.5
NS126	.0361	.001	65	143.5
NS127	.0359	.001	65	144.6
NS128	.0356	.001	64	144.7
NS129	.0359	.00075	64	144.3
NS130	.0358	.001	65	143.0

TABLE #5
 Test 43: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°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)
TS131	.0353	79.9	95.8	25.2	15.6	55.2	74.5	216.9
TS132	.0351	80.3	96.2	23.8	14.0	54.8	74.4	214.1
TS133	.0351	79.5	95.7	23.8	14.1	55.6	73.9	212.9
TS134	.0350	79.4	95.4	23.8	14.0	54.3	74.8	215.3
TS135	.0350	79.1	95.0	24.0	14.4	53.6	74.8	212.5
TS136	.0353	79.0	94.9	24.3	14.3	53.1	74.5	208.5
TS137	.0352	79.0	94.9	26.6	15.8	53.3	75.9	220.7
TS138	.0350	79.3	95.0	24.1	14.3	53.6	75.3	216.5
TS139	.0351	79.5	95.2	26.4	15.6	53.4	74.9	212.5
TS140	.0348	78.7	94.8	24.2	14.3	53.9	75.1	216.5

TABLE #6
 Test 44: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 500°F , 4000 psig H_2
 AND COAL SLURRY ENVIRONMENT
 NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS131	.0346	.00075	65	148.6
NS132	.0357	.001	65	147.1
NS133	.0360	.001	65	147.2
NS134	.0359	.001	65	147.6
NS135	.0358	.001	65	148.0
NS136	.0355	.001	65	148.2
NS137	.0356	.001	65	147.8
NS138	.0364	.001	65	145.9
NS139	.0362	.001	65	147.5
NS140	.0362	.001	65	146.7

TABLE #7
 Test 45: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 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)
TS141	.0353	77.9	94.6	24.9	15.7	54.5	73.6	206.3
TS142	.0353	77.2	94.3	24.9	14.9	53.8	74.5	211.3
TS143	.0351	76.9	94.0	23.5	14.0	54.1	74.9	215.3
TS144	.0352	77.0	94.0	23.5	14.4	54.7	74.0	210.1
TS145	.0353	77.2	94.2	26.1	15.9	52.4	75.0	209.6
TS146	.0351	76.2	93.4	25.7	15.8	52.7	75.3	213.7
TS147	.0353	77.3	94.3	25.2	15.4	53.8	74.5	211.3
TS148	.0353	77.3	94.3	24.2	15.0	55.2	74.0	212.9
TS149	.0349	77.4	94.6	22.5	13.3	53.9	74.7	213.0
TS150	.0353	77.2	94.2	24.2	14.8	54.1	75.0	216.4

TABLE #8
 Test 46: $T=72^{\circ}\text{F}$ $P=1\text{ATM AIR}$ $\dot{\epsilon}=.05 \text{ min}^{-1}$
 HISTORY: MATERIAL EXPOSED FOR 168 HOURS AT 800°F , 4000 psig H_2
 AND COAL SLURRY ENVIRONMENT
 NOTCHED BAR TENSILE SAMPLES

Sample #	Notch Area (in ²)	Notch Radius (in)	Notch Angle (°)	Notch Tensile Stress (ksi)
NS141	.0353	.001	65	148.0
NS142	.0355	.00125	65	147.6
NS143	.0357	.00125	65	146.5
NS144	.0354	.00125	65	147.2
NS145	.0356	.00125	65	145.8
NS146	.0353	.001	65	148.2
NS147	.0353	.00125	65	147.3
NS148	.0355	.001	65	147.3
NS149	.0356	.001	65	146.3
NS150	.036	.001	65	146.7

TABLE #9

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)	79.2 (4)	94.5 (4)	22.5 (4)	8.8 (4)	61.0 (4)	138.8 (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)

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

TABLE #10

FY 1978 TEST MATRIX

Test	Type	Exposure Conditions					Exposure Stress ^A (KSI)	Test Conditions ^{A,B}			
		Temp. (°F)	Pressure (Psig)	Envir- onment	Time (HRS)	Temp. (HRS)		Temp. (°F)	Pressure (Psig)	Envir- onment	Number of samples
23	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	168	0	Ambient	Ambient	Air	10	
40	Notched	500	2000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
41	Smooth	900	2000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
42	Notched	900	2000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
43	Smooth	500	4000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
44	Notched	500	4000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
45	Smooth	800	4000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
46	Notched	800	4000	Slurry-H ₂	168	0	Ambient	Ambient	Air	10	
47	Smooth	500	2000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
48	Notched	500	2000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
49	Smooth	800	2000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
50	Notched	800	2000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
51	Smooth	500	4000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
52	Notched	500	4000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
53	Smooth	800	4000	Slurry-H ₂	168	25 ^C	Ambient	Ambient	Air	10	
54	Notched	800	4000	Slurry-H ₂	168	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 ^E	
61	Smooth	800	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^E	
62	Notched	800	4000	Slurry-H ₂	Failure ^D	Various	-	-	-	12 ^E	

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

E Two samples at each of 6 different stress levels

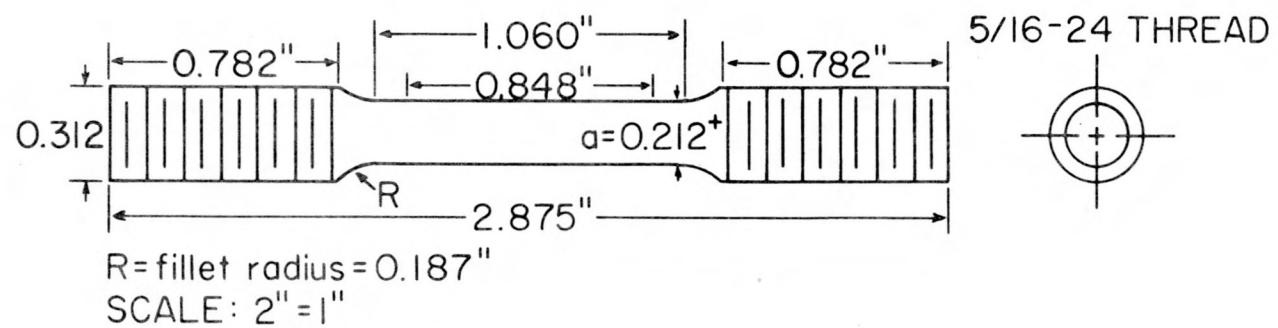


Fig. 1: Base Property Determination Smooth Tensile Sample.

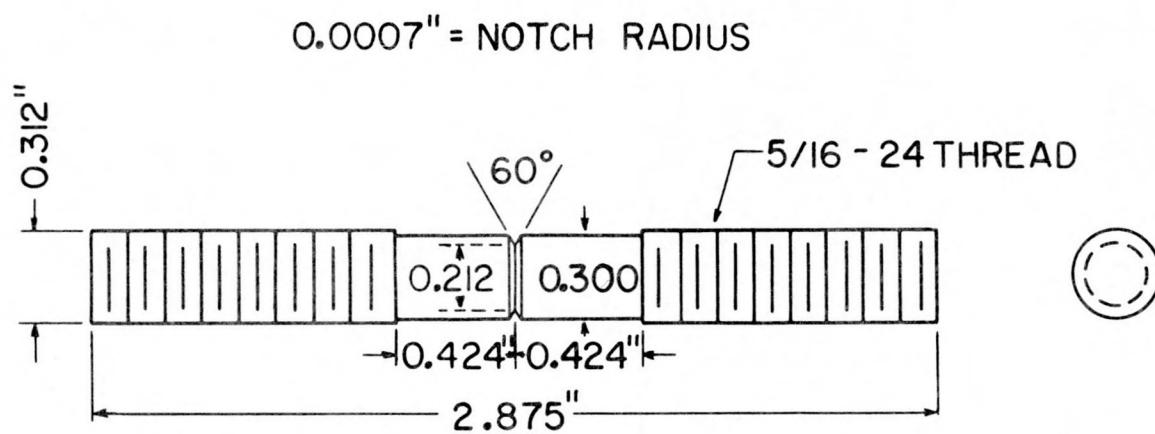


Fig. 2: Base Property Determination Notched Tensile Sample.

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