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DMIC Report S-31

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EFFECTS OF HYDROGEN GAS ON METALS AT AMBIENT TEMPERATURE

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DMIC Report S-31
April, 1970

EFFECTS OF HYDROGEN GAS ON METALS AT AMBIENT TEMPERATURE

by

J. E. Campbell

to

OFFICE OF THE DIRECTOR OF DEFENSE RESEARCH AND ENGINEERING

DEFENSE METALS INFORMATION CENTER
Battelle Memorial Institute
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EFFECTS OF HYDROGEN GAS ON METALS AT AMBIENT TEMPERATURE

J. E. Campbell*

SUMMARY

Recent information on the properties of ferrous and nonferrous alloys in hydrogen-gas environments at ambient temperature indicates that many engineering alloys become embrittled when subjected to tensile loads in high-purity hydrogen gas. This information will be important for those who are concerned with selecting or specifying materials for systems in which hydrogen gas is used as a fuel, propellant, or coolant.

On the basis of the information available, steels (ferritic, martensitic, and bainitic), nickel-base alloys, and titanium alloys become embrittled in pure-hydrogen-gas environments at ambient temperature. The embrittling effect is detected by making tension tests on sharp-notched specimens in an environment of high-purity hydrogen gas and, for comparison, tests on similar specimens in an inert gas at the same temperature and pressure. If the material is embrittled by hydrogen, its notch tensile strength will be reduced. The effect is more pronounced as the hydrogen-gas pressure is increased, but in some cases the embrittling effect has been observed at 1 atmosphere of pressure. The effect is more pronounced for the high-strength steels and high-strength nickel and titanium alloys than for the low-strength alloys. In unnotched specimens exposed to a pure-hydrogen environment, hydrogen embrittlement manifests itself as a decrease in ductility.

The data on sharp-notched or precracked specimens must be considered in connection with the performance of actual components in hydrogen-gas environments, because most components contain areas where there are stress concentrations or small cracks or flaws. If the alloy is subject to hydrogen-environment embrittlement, the effects will be noted first in the areas where there are high strain gradients.

Results of tests on stable austenitic stainless steels such as Types 310 and 316, or certain aluminum alloys such as 6061-T6, 2219-T6, and 7075-T73, and beryllium copper indicate that there is no significant evidence of embrittlement of these alloys in hydrogen gas at pressures up to 10,000 psi.

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INTRODUCTION

Problems associated with embrittlement of steels caused by hydrogen induced into the steel during manufacture, processing, pickling, plating, and exposure to hydrogen gas at elevated temperatures have been discussed in the literature.(1-4) Embrittlement of steels and nonferrous alloys resulting from exposure to hydrogen gas at ambient temperature has been studied only quite recently. Consequently many of the people who are concerned with selecting or specifying materials for advanced systems involving the use of hydrogen-gas storage and associated tubing, fittings, and controls may not be familiar with the limitations of materials for such applications.

Dissemination of this information is particularly important at the present time, because there has been a growing interest in the design of propulsion or power systems (including fuel cells) for space and undersea vehicles which require hydrogen gas at high pressures. Other space applications such as equipment for hydrogen-gas cooling by the Joule-Thompson effect of sensors in the Mariner 69 space vehicle also require hydrogen storage at high pressures.

Since there have been no problems with the portable steel storage tanks for laboratory and industrial supplies of hydrogen gas, one might assume that the selection of alloys for hydrogen environments is not critical. However, the use of certain engineering alloys in hydrogen-gas environments can be very hazardous, particularly at high pressures.(5) The purpose of this report is to point out, on the basis of available information, those alloys that become embrittled in hydrogen gas at ambient temperature and those that do not. The available information is somewhat limited, but is sufficient to permit selection of materials that can be used to avoid embrittlement problems at ambient temperature.

A tentative list of papers to be presented at a symposium in May, 1969, on "Effects of Gaseous Hydrogen on Metals" is presented in the Appendix.

The effects of hydrogen gas on metals and alloys are discussed by metal or alloy group as follows:

- Pure iron, carbon steels, and alloy steels
- Stainless steels
- Aluminum and aluminum alloys
- Copper and beryllium copper
- Nickel and nickel-base alloys
- Titanium and titanium alloys

PURE IRON, CARBON STEELS, AND ALLOY STEELS

Results of tension tests, burst tests, and fatigue tests on specimens of high-purity iron and carbon and alloy steels are discussed in the following sections. Each series of tests indicates that hydrogen gas at high pressure can cause a reduction in notch strength and in ductility of ferrous materials as compared with similar tests in air or in helium.

Effects on Tensile Properties

At Rocketdyne, the tensile properties of Armco iron and several carbon and alloy steels have been evaluated in hydrogen gas at 10,000 psi pressure.(6,7) The alloys and their chemical compositions are shown in Table 1. In this program, the tensile properties were determined in air at 1 atmosphere pressure, in helium gas at 10,000 psi pressure, and in hydrogen gas at 10,000 psi pressure. The hydrogen gas contained less than 5 ppm impurities. Unnotched specimens were 0.250 inch in diameter and the notched specimens had a stress-concentration factor (K_t) of 8.4. A special pressure cell was positioned around each specimen

TABLE 1. CHEMICAL COMPOSITIONS OF ARMCO IRON, CARBON STEELS, AND ALLOY STEELS IN THE ROCKETDYNE PROGRAM(6)

Material	Composition, percent by weight								
	C	Mn	P	S	Si	Ni	Cr	Mo	Other
Armco Iron (3/8-inch rod)	0.033	0.036	0.007	0.015	0.001				
AISI 1020 (3/8-inch rod)	0.17	0.47	0.011	0.037					
AISI 1042 (3/8-inch rod)	0.44	0.76	0.008	0.020	0.20				
AISI 4140 (3/8-inch rod)	0.405	0.83	0.009	0.014	0.31		0.93	0.20	
HY-80 (1/2-inch plate)	0.13	0.30	0.016	0.021	0.22	2.49	1.46	0.43	0.001Ti, 0.002V
HY-100 (1/2-inch plate)	0.16	0.32	0.010	0.019	0.22	2.57	1.67	0.42	0.05Cu, 0.001Ti, 0.002V
ASTM A-302, Grade B with Ni (5 1/2-inch plate)	0.25	1.31	0.016	0.021	0.27	0.63			0.003B
ASTM A-515 Gr.70 (3/8-inch plate)	0.27	0.71	0.011	0.018	0.19				
ASTM A-517, Grade F (3/8-inch plate)	0.16	0.80	0.010	0.016	0.21	0.79	0.54	0.43	0.25Cu, 0.04V, 0.002B
ASTM A-372, Class IV (0.817-inch-thick pipe)	0.46	1.60	0.010	0.022	0.21			0.22	
9Ni-4Co-0.20C (7/16-inch rod)	0.17	0.27	0.005	0.005	0.02	9.10	0.78	1.01	4.45Co, 0.78V
18Ni(250) Maraging Steel (3-inch-square block)	0.006	0.02	0.003	0.007	0.02	18.70		4.80	0.15Al, 0.40Ti, 7.58Co, 0.012Zr, 0.002B, 0.05Ca

TABLE 2. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF ARMCO IRON, CARBON STEEL, AND ALLOY STEELS IN AIR, IN HELIUM AT 10,000 PSI PRESSURE, AND IN HYDROGEN AT 10,000 PSI PRESSURE(6)

			Tensile Properties in Air				Tensile Strength, ksi			Elongation, percent		Reduction in Area, percent		
Material	Heat Treatment	Specimen Type(a)	Yield Strength, ksi	Tensile Strength, ksi	Elongation, percent	Reduction in Area, percent	In 10,000 Psi He	In 10,000 Psi H_2	Reduction in strength, percent(b)	In 10,000 Psi He	In 10,000 Psi H_2	In 10,000 Psi He	In 10,000 Psi H_2	Vacuum Purged(c)
Armco Iron	Annealed	UN	30	66	19	79	56	57	–	18	15	83	50	Yes
		N		133		6.0	121	105	14			6.4	1.7	No
AISI 1042	Normalized 1650 F	UN	66	97	30	58	90	89	–	29	22	59	27	No
		N		161		8.3	153	115	25			8.5	2.8	No
AISI 1042	Quenched and tempered(d)	UN	–	–	–	–	–	187	–	–	0	–	0	Yes
		N		236		2.3	236	53	78			0.6	0.6	Yes
ASTM A-302 Grade B+Ni	Quenched and tempered(e)	UN	–	–	–	–	119	116	–	19	17	66	33	No
		N					227	176	22			15	2.8	No
AISI 4140	Quenched and tempered(f)	UN	93	108	29	68								
		N		195		10.5	182	156	15			14	7.1	Yes
AISI 4140	Quenched and tempered(g)	UN	186	197	14	56	186	178	4	14	2.6	48	8.8	Yes
		N		318		3.9	313	125	60			2.8	0.9	Yes
HY-100	Heat treated by supplier	UN	106	122	25	65	113	115	–	20	18	76	63	Yes
		N		234		7.3	224	164	27			7.3	3.8	Yes
ASTM A-372 Class IV	Tempered at 875 F	UN	94	125	18	47	118	116	1.7	20	9.6	53	18	Yes
		N		206		2.9	200	148	26			2.0	2.8	Yes
9Ni-4Co-0.20C	Quenched and tempered(h)	UN	202	208	16	66	199	175	12	15	0.5	67	15	Yes
		N		373		6.8	367	89	76			6.3	0.2	Yes
18Ni(250) Maraging Steel	Aged 900 F for 6 hours	UN	253	259	10.5	–	250	171	32	8.2	0.2	55	2.5	Yes
		N		436		3.1	423	50	88			2.5	0.0	Yes

(a) UN-unnotched specimen, 0.250 inch in diameter in test section, 1-inch gage length.
N-notched specimen with 60-degree V-notch, root radius 0.00095 inch ± 0.0001, and diameter at notch root 0.150 inch (K_t = 8.4).
(b) For specimens tested in hydrogen compared with those tested in helium.
(c) If vacuum purging of test cell was not done, purge consisted of three pressurization-depressurization cycles with hydrogen (or helium) to 10,000 psi and to 1 atmosphere. For vacuum purging, test cell was evacuated to 20 microns, backfilled with hydrogen three times, and then given three pressurization cycles to 10,000 psi depressurized to 1 atmosphere before final pressurization to 10,000 psi.

(d) Austenitized 1575 F for 1 hour, quenched in water, tempered at 400 F for 2 hours.
(e) Austenitized 1650 F for 1 hour, quenched in water, tempered at 1270 F for 1 hour.
(f) Austenitized 1550 F for 1 hour, quenched in oil, tempered at 1300 F for 2 hours.
(g) Austenitized 1550 F for 1 hour, quenched in water, tempered at 900 F for 2 hours.
(h) Austenitized 1550 F for 1 hour, quenched in oil, tempered twice at 1000 F for 2 hours.

subjected to the high-pressure gas environments. Information regarding the purging technique is presented in Table 2 along with the tensile property data. Vacuum purging was found to be preferable and was used exclusively in later phases of the program. Tensile-property data in Table 2 were obtained by starting the tensile test immediately after the final pressurization cycle. Strain rates for the unnotched specimens were 0.002 minute^{-1} to the yield load and 0.04 minute^{-1} from the yield load to the fracture load. All tensile tests were made at ambient temperature.

The tensile data in Table 2 indicate that, even for the short duration of the tensile tests, each of the steel was embrittled to some extent by the high-pressure hydrogen gas. This embrittlement is most obvious for the notched-specimen data, but the reduction in ductility of the unnotched specimens tested in the hydrogen environment is also an indication of the embrittling effect of the hydrogen. The configuration of the notched specimens is such that when there is no embrittlement, the notch strength is higher than the unnotch strength. Notch tensile properties are [illegible] more sensitive toward embrittling conditions than are unnotch tensile properties. In general, the reduction in notch strength under these conditions of high-pressure hydrogen gas is more pronounced for the high-strength steels than for the lower-strength steels.

Since the series of steels in Table 2 includes a carbon steel with two different heat treatments, several low-alloy steels, and high-alloy steels of two types [9Ni-4Co-0.20 C and 18 Ni (250) maraging steel], the data indicate that steels, other than austenitic types, are in general embrittled in hydrogen gas at high pressures.

The effect of holding time for periods up to 100 days in 10,000 psi hydrogen gas on the tensile properties of unnotched and notched specimens of several steels also was determined on the Rocketdyne program.(6) These specimens were subjected to sustained loading during the exposure time. The specimens then were tested in tension in the same environment at the end of the holding time. The specimens were AISI 1020 steel (prestrained 1 percent), ASTM A-515 Grade 70, HY-80, and AISI H-11 tool steel.

Elongation of unnotched specimens of AISI 1020 steel was slightly lower for tests in 10,000 psi hydrogen than for tests in 10,000 psi helium, but was not significantly affected by holding time or holding stress. Reduction in area was considerably lower for specimens tested in the hydrogen-gas environment than for those tested in the helium environment.

Elongation of unnotched specimens of ASTM A-515 Grade 70 steel was significantly lower in 10,000 psi hydrogen gas than in 10,000 psi helium, but it was not affected by holding time or holding stress. Reduction in area was also lower for these specimens when tested in hydrogen then when tested in helium.

Elongation of unnotched specimens of HY-80 steel was practically the same for all test conditions, but the reduction in area was slightly less for the specimens tested in hydrogen in comparison with those tested in helium. Unnotched specimens of H-11 tool steel (heat treated to Rockwell C 55) had no measurable elongation or reduction in area under any conditions in the 10,000 psi hydrogen environment.

Results of tension tests on notched specimens of the same four steels in 10,000 psi hydrogen after holding for periods up to 100 days at selected holding stresses are shown in Table 3. When tested in the gas atmosphere without any prior exposure period; the notched specimens of AISI 1020 steel failed at 20 percent lower stress in hydrogen than in helium. However, a reduction in strength was not observed for the notched specimens of AISI 1020 steel that were held in the hydrogen environment for 1 or 100 days at 76,000 psi stress.

For the other three steels in Table 3, there was a marked reduction in notch strength in the hydrogen environment, and this reduction was greatest for the specimens that had not been exposed to the hydrogen prior to testing. Apparently, extended exposure time under load does not increase the extent of the hydrogen embrittlement and, in fact, appears to diminish the effect slightly.

Additional tests were made on two steels using specimens of three notch configurations to obtain stress-concentration factors (K_t) of about 4, 5.9, and 8.3. Any of these specimens will fracture at notch strengths greater than the tensile strength for unnotched specimens when used to test ductile steels in air. Results of tests in air, in helium, and in hydrogen are shown in Table 4. The strength of these notched specimens when tested in 10,000 psi hydrogen was 15 to 25 percent lower as compared with strengths obtained in similar tests in 10,000 psi helium. The notch acuities of these specimens represent a range of stress concentrations but do not represent the degree of stress concentration that might be expected at the apex of a natural flaw.

An additional series of tests was conducted on the Rocketdyne program to determine the effect of hydrogen pressure at 100, 1,000, and 10,000 psi on the tensile properties of unnotched and notched specimens of the ASTM A-302-B steel. For the unnotched specimens, the major effect was on the reduction in area, which decreased progressively from 68 to 33 percent as the hydrogen pressure was increased from 0 to 10,000 psi. For the notched

TABLE 3. AVERAGE TENSILE PROPERTIES OF NOTCHED SPECIMENS OF SEVERAL STEELS AFTER EXPOSURE TO HELIUM OR HYDROGEN WHILE UNDER LOAD(6)

	In 10,000 Psi Helium				In 10,000 Psi Hydrogen				
Material	Holding Time, days	Holding Stress, ksi	Tensile Strength,(a) ksi	Reduction in Area, percent	Holding Time, days	Holding Stress, ksi	Tensile Strength,(a) ksi	Reduction in Area, percent	Reduction in Strength,(b) percent
AISI 1020	0	0	105	14	0	0	84	8.2	20
(hot rolled)	1	76	101	19	1	76	101	8.7	–
	10	76	103	14	100	76	104	8.4	–
ASTM A-515	0	0	106	8.1	0	0	74	3.4	32
Grade 70					1	67	83	3.4	22
(hot rolled)					100	67	83	3.7	22
HY-80	0	0	190	8.6	0	0	151	3.9	20
(mill processed)					1	136	161	4.2	15
					100	136	155	4.3	18
H-11	0	0	252	0.0	0	0	57	0.0	77
(R_c 55)					1	54	78	0.0	69
					100	54	112	0.0	56

(a) Notched-specimen design same as in Table 2 (K_t = 8.4).
(b) For specimens tested in hydrogen compared with those tested in helium.

TABLE 4. AVERAGE TENSILE PROPERTIES OF NOTCHED SPECIMENS OF THREE STEELS IN AIR, HELIUM, AND HYDROGEN SHOWING EFFECT OF NOTCH ACUITY[6]

			In Air		In 10,000 Psi He		In 10,000 Psi H_2		
Material	Heat Treatment	Notch Acuity, K_t	Tensile Strength, ksi	Reduction in Area, percent	Tensile Strength, ksi	Reduction in Area, percent	Tensile Strength, ksi	Reduction in Area, percent	Reduction in Strength,[a] percent
ASTM A-302	1650 F 1 hr, WQ,	1.0			120		116	67	3.3
Grade B + Ni	1270 F 1 hr, AC	4.0	221	19	207	19	177	4.8	15
		5.9	221	15	212	16	168	4.9	21
		7.5-8.3	220	15	227	11	176	2.7	23
ASTM A-517	1625 F 1/2 hr,	1.0	128	65			121	63	6
Grade F	WQ, 1225 F	3.7	243	13	227	12	181	2.8	20
(T-1 steel)	1 hr	5.8	243	11	230	12	172	2.0	25
		8.3-8.7	236	7.4	222	5.7	173	2.1	23

(a) Percentage reduction in strength for specimens tested in hydrogen environment as compared with strength of those tested in helium environment.

specimens, the notch strength decreased progressively from 235 to 176 ksi as the hydrogen pressure was increased over the same range.

For room-temperature tensile tests on steel specimens, the effects of pure-hydrogen-gas environments are dependent, therefore, on the hydrogen gas pressure, stress concentration, alloy content, and microstructure of the steel. Because of the potential degradation of the properties of engineering steels in hydrogen environments, especially at high pressures, special precautions should be taken when using steels in such environments or consideration whould be given to using materials that are not degraded by hydrogen.

Effects on Notch Tensile Properties of AISI C 1025 and 4140

Notched specimens of AISI C1025 and AISI 4140 steels were tested in high-pressure nitrogen and hydrogen environments at the Rensselaer Polytechnic Institute.[8, 9] Specimens with two notch configurations were employed. Specimens with a "sharp notch" configuration had major diameters of 0.306 inch, notch diameters of 0.150 inch in 60-degree V-notches, and root radii of 0.0046 inch, resulting in a stress-concentration factor of 4.2. Specimens with a "blunt notch" configuration were the same except that the root radii were 0.0313 inch. Specimens with these notch configurations normally have notch strengths higher than the corresponding tensile strengths for these steels. The specimens were cleaned with carbon tetrachloride and inserted in the test cells, which were similar to those used in the Rocketdyne program discussed previously. Smooth-bar tensile properties of the steels are given in Tables 5 and 6 along with the notch-strength data. For the data in Table 5, the specimens were exposed to the pressurized gas environment for 24 hours before loading was started and for the duration of the test.

The data in Table 6 provide further evidence of the embrittlement resulting from high-pressure hydrogen environments. Several specimens were subjected to delayed-failure tests in 10,000 psi hydrogen environments. However, such tests were discontinued when it was found that the stress range within which delayed failure occurred was very narrow for reasonable failure times. It was concluded that the results of the notch tensile tests were sufficient evidence of the degree of embrittlement caused by the hydrogen environment.

These tests demonstrated that the effect of the hydrogen environment is more severe for the steel heat treated to a high strength level than for the same steel at a lower strength level.

TABLE 5. EFFECT OF HIGH-PRESSURE HYDROGEN ON THE NOTCH STRENGTH OF AISI C 1025 AND 4140 STEELS AT ROOM TEMPERATURE[8]

Steel Type	Unnotch Tensile Strength, ksi	Environment for Notch Tensile Tests[a]	Notch Strength, ksi
AISI C 1025	65.0	10,000 psi N_2	106
		10,000 psi H_2	80
AISI 4140	135	10,000 psi N_2	241
		6,000 psi H_2	207
		10,000 psi H_2	204
AISI 4140	228	10,000 psi N_2	362
		2,000 psi H_2	135
		6,000 psi H_2	121
		10,000 psi H_2	89

(a) Specimens exposed to environment 24 hours before loading and for duration of test. Notched specimens had a stress-concentration factor of 4.2.

Furthermore, the effect of hydrogen pressure is more pronounced for steel specimens in the high-strength condition, than for specimens of the same steel at a lower strength level.

Effects on Burst Disk Specimens

The effects of hydrogen on sheet steels and the protection offered by various surface finishes and coatings have been studied by the French Atomic Energy Commission using disk-shaped specimens that were subjected to burst tests.[10] The environmental gas was used as the pressurizing medium. The specimens were rated for hydrogen-gas embrittlement by the ratio of the pressure of helium gas to cause fracture to the pressure of hydrogen gas to cause fracture, i.e, P_{He}/P_{H_2}. Tensile stresses were induced on the high-pressure side of the disk as a result of bending in the disks near the seating area of the fixture. Fracture occurred in a circular pattern. Tests have been conducted using this equipment to show the extent of environmental-hydrogen embrittlement for some of the high-strength martensitic steels meeting certain French specifications, including maraging steel. Results of these disk tests showed the same trends as the previously discussed tension tests in hydrogen conducted at the Rensselaer Polytechnic Institute.[8, 9]

TABLE 6. EFFECT OF HIGH-PRESSURE HYDROGEN ON THE NOTCH STRENGTH OF AISI 4140 STEEL AT ROOM TEMPERATURE AND AT -320 F[9]

Unnotch Tensile Strength, ksi	Notch Configuration	Testing Conditions for Notched Specimens	Notch Strength, ksi	Change in Strength, percent
154	Sharp[a]	In air, room temp	261	0
		In 10,000 psi H_2 18 to 22 hr	215	18 loss
		In 10,000 psi H_2 18 to 22 hr; H_2 removed prior to testing	304	16 gain
		In 10,000 psi H_2 18 to 22 hr; H_2 removed, tested within 3 hr	256	2 loss
		In N_2 at -320 F	361	38 gain
		In H_2 2 hr at room temp tested at -320 F	377	4 gain[c]
154	Blunt[b]	In air, room temp	287	0
		In 10,000 psi H_2 18 to 22 hr	254	11 loss
		In 10,000 psi H_2, tested immediately	252	12 loss
		In 10,000 psi H_2 18 to 22 hr; H_2 removed prior to testing	278	3 loss
		Stressed to 180 ksi, added H_2, and tested	264	8 loss
230	Sharp[a]	In air, room temp	375	0
		In 10,000 psi H_2 18 to 22 hr	[illegible]	[illegible] loss
		In 10,000 psi H_2 18 to 22 hr; H_2 removed prior to testing	[illegible]	[illegible]
		Stressed to 175 ksi, tested after adding 4000 psi H_2	[illegible]	50 loss
		Stressed to 125 ksi; tested after adding 4800 psi H_2	140	63 loss
		Stressed to 75 ksi, tested after adding 10,000 psi H_2	106	72 loss
		In N_2 at -320 F	265	29 loss
		In 10,000 psi H_2 18 to 22 hr at room temp, tested at -320 F	238	10 loss[c]
		[illegible] 10,000 psi H_2 [illegible] to 22 hr, tested at -320 F	247	7 loss[c]
		Cooled to -320 F; stressed to 19? ksi, added 10,200 psi H_2; tested at -320 F.	229	14 loss[c]
230	Blunt[b]	In air, room temp	377	0
		In 10,000 psi H_2 5 min	240	36 loss
		In 10,000 psi H_2 18 to 22 hr	177	52 loss
		In 10,000 psi H_2 18 to 22 hr; H_2 removed prior to testing	310	17 loss

(a) Sharp-notch configuration, 0.306-inch major diameter, 60-degree V-notch with 0.150 inch root diameter and 0.0046 inch root radius.
(b) Blunt-notch configuration, same as above but with 0.0313 inch root radius.
(c) Relative to specimen tested in N_2 at -320 F.

For tests to evaluate the effect of surface treatments and protective coatings, the treated or coated sides of the disks were subjected to hydrogen pressure during testing in the burst-test fixture. Specimens of the French steel 35 NiCrMo 16 were finished on one surface by milling, turning, shot peening with glass beads, and electrolytic polishing. Information on composition, heat treatment, and bursting-pressure ratios is given in Table 7. The peening treatment and electropolishing produced surfaces that appeared to be more resistant to hydrogen-gas embrittlement than were the machined surfaces.

Electrolytic deposits of zinc, cadmium, copper, and nickel plus gold (brush plates), gold and aluminum deposits produced by vacuum metallizing, and nickel carbonyl and diffused chromium deposits produced by vapor plating also were evaluated using disk specimens of 35 NiCrMo 16 steel. Zinc, copper, nickel, and chromium coatings did not give favorable results. Cadmium under certain conditions and gold and aluminum coatings produced by vacuum metallizing were beneficial. A tin coating brush plated on a nickel flash plating also provided some resistance to hydrogen environment embrittlement.

Effects on Fatigue Properties of ASTM A-302 and A517

Specimens of ASTM A-302 Grade B steel modified with nickel and ASTM A-517 Grade F steel were subjected to low-cycle fatigue loading in tension in 10,000 psi helium and in 10,000 psi hydrogen on the Rocketdyne program.[6] The most significant data were obtained on notched specimens that had been fatigue cycled in air to develop shallow cracks in the notches prior to low-cycle fatigue loading in the high-pressure gas environments. The same loading equipment and gas-pressure cells were

TABLE 7. INFLUENCE OF SURFACE CONDITION ON THE RELATIVE RUPTURE PRESSURE OF DISK SPECIMENS OF 35 NiCrMo 16 STEEL[10]

Surface Condition	P_{He}/P_{H_2} for Rupture
Turned	5.45
Milled	4.50
Shot peened	4.02
Electrolytically polished	3.00

Notes:
Composition of 35 NiCrMo 16 steel in percent by weight: 0.38C, 4.3Ni, 1.9Cr, 0.50Mo, 0.37Mn, 0.30Si, 0.010P, 0.005S.
Heat treatment: austenitized at 1600 F for 1 hour in helium, cooled in helium, cooled to - 110 F for 2 hours, tempered twice at 450 F for 1 hour each time.
Tensile properties: yield strength 226 ksi, tensile strength 275 ksi, elongation 7.3 percent.

used for fatigue cycling as for the tensile tests discussed previously. Load cycling was between zero load and a maximum applied load [illegible] internal pressure [illegible] friction. Results of these tests are shown in Figures 1 and 2. Hydrogen at 10,000 psi obviously has a marked effect in reducing the fatigue strength of the two steels under these conditions as compared with that of steels tested in 10,000 psi helium or air at 1 atmosphere pressure.

Effects on Fatigue Properties of 18 Ni (250) Maraging Steel

Center-notch plate specimens of 18Ni (250) maraging steel were subjected to fatigue loading at the United States Steel Corporation in environments of argon, humid argon, hydrogen, and humid hydrogen to determine the effect of these environments on the crack growth rate.[11] The specimens were 0.250 inch thick, 2.75 inches wide, and 12 inches long, with center cracks initially about 0.140 inch long. Fatigue cycling was conducted at room temperature at 150 cycles per second for zero-to-tension loading. During testing, fatigue cycling was interrupted every 10,000 cycles to make crack-length measurements by means of an electric-potential method while the specimen was under static mean load. An environmental chamber was clamped in place on each specimen prior to fatigue cycling. The environments were maintained at 1 atmosphere pressure and the hydrogen gas was of 99.999 percent purity. The specimens had been annealed at 1700 F and aged at 900 F for 3 hours. The yield and tensile strengths of the 18 Ni maraging steel were 246,000 psi and 257,000 psi respectively.

Crack-growth-rate data were practically the same for the argon, humid argon, and humid hydrogen environments. The data are plotted in Figure 3 for the humid and dry hydrogen. Crack growth rates under conditions of cyclic loading were about three times greater for specimens tested in the dry hydrogen than for those tested in humid hydrogen. Apparently the water vapor present in the hydrogen gas counteracts the effect of the hydrogen itself.

Effects on Subcritical Crack Growth in Alloy Steels

Crack growth in center-cracked sheet specimens of H-11 steel in hydrogen gas and other environments has been studied in a continuing program at Cornell University.[12, 13] The specimens were 3 inches wide, 12 inches long, and 0.065 inch thick, with 1-inch-long transverse center slots. The ends of the slots were sharpened to about 0.001-inch radius by Elox machining. These H-11 steel specimens had been heat treated to a yield strength of 230,000 psi. During loading in the testing

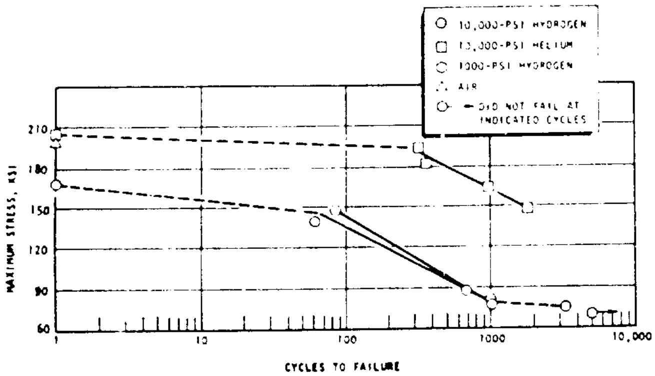

FIGURE 1. LOW-CYCLE FATIGUE CURVES FOR PRE-CRACKED SPECIMENS OF ASTM A-302 STEEL(6)

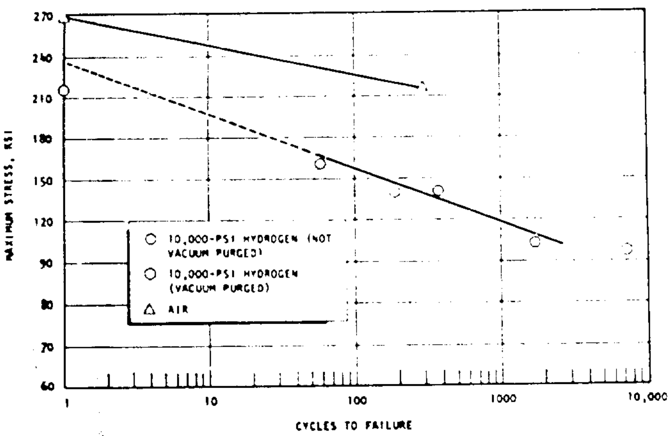

FIGURE 2. LOW-CYCLE FATIGUE CURVES OF PRE-CRACKED SPECIMENS OF ASTM A-517 STEEL(6)

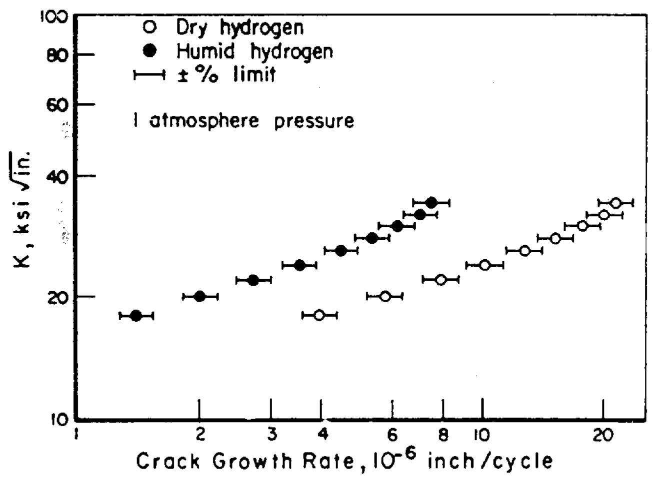

FIGURE 3. RATE OF FATIGUE-CRACK PROPAGATION FOR SPECIMENS OF 18Ni (250) MARAGING STEEL TESTED IN HYDROGEN ENVIRONMENTS(11)

ΔK is range of stress-intensity factor on fatigue cycling.

environment, crack propagation was measured by an electric-potential method. In a purified hydrogen environment at 1 atmosphere pressure, crack growth occurred at a stress-intensity factor of 11 ksi $\sqrt{\text{in.}}$, but in a dry-argon atmosphere, crack growth did not occur until the stress intensity factor reached 40 ksi $\sqrt{\text{in.}}$

Since a continuous record of crack growth could be obtained by means of the electric-potential instrumentation, the relative rates of crack growth could be determined with different environments for the same specimen. The crack growth at a stress-intensity factor of about 18.5 ksi $\sqrt{\text{in.}}$ in pure hydrogen at 1 atmosphere pressure is compared with that at a stress intensity factor of about 22.5 ksi $\sqrt{\text{in.}}$ in humidified argon in Figure 4. The crack growth rate is seen to be significantly greater in the hydrogen environment. However, as shown in Figure 5, the introduction of only 0.6 percent oxygen into the hydrogen is sufficient to stop crack growth under the conditions for which the data in Figure 5 were obtained.

Crack growth rates in precracked specimens of hardened AISI 4130 steel were investigated in hydrogen-gas environments under constant stress intensity in a program at Ames Research Center.(14) The specimens used were tapered double-cantilever-beam type, such as have been used in some laboratories for obtaining plane-strain fracture toughness (K_{Ic}) data. Because of the tapered design, the stress-intensity factor does not change as the crack length is increased if the same load is applied. Crack growth was measured by a compliance technique. After a fatigue

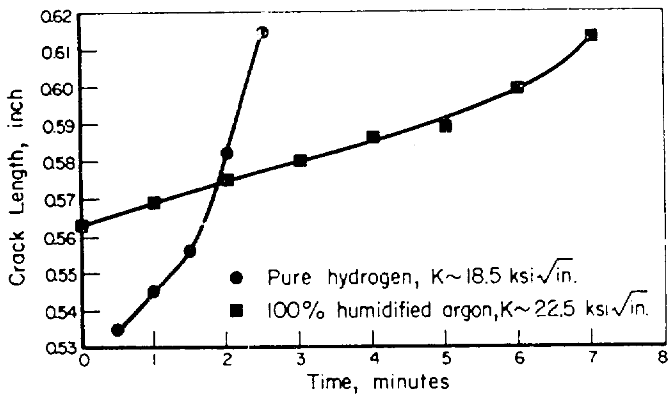

FIGURE 4. SUBCRITICAL CRACK GROWTH IN H-11 STEEL SPECIMENS IN ENVIRONMENTS OF PURE HYDROGEN AND HUMIDIFIED ARGON AT ONE ATMOSPHERE PRESSURE(12)

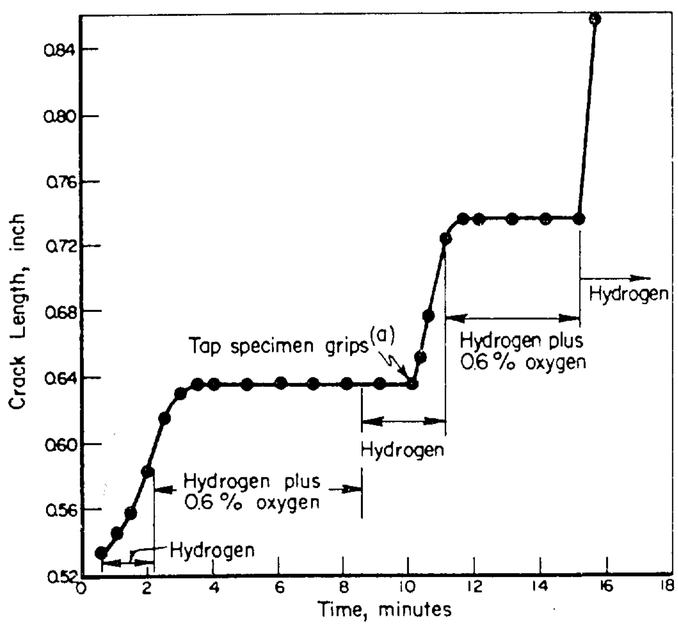

FIGURE 5. SUBCRITICAL CRACK GROWTH IN H-11 STEEL SPECIMENS IN ENVIRONMENTS OF PURE HYDROGEN AND A HYDROGEN-OXYGEN MIXTURE(12)

(a) At this point, the specimen grips were tapped to initiate the reaction.

crack was developed in the specimen in the evacuated chamber, hydrogen was introduced and the maximum static load on the specimen was adjusted to obtain a stress-intensity factor of 45 ksi $\sqrt{in.}$ After slow crack growth had started, the load was reduced to produce a stress-intensity factor of 36 ksi $\sqrt{in.}$ Results of tests conducted in the temperature range from -8 to 48 C at a hydrogen-gas pressure of 8.5 psi absolute indicate that the crack growth rate was relatively constant over this temperature range. Results of tests at room temperature with hydrogen-gas pressures over the range from 1.26 to 14.5 psi absolute are shown in Figure 6. On this log-log plot, the crack growth rate versus hydrogen pressure can be represented by a straight line. For a constant stress-intensity factor, the crack growth rate increases as the hydrogen pressure increases. These data were used in developing a proposed mechanism for hydrogen-gas embrittlement which apparently is not the same as the mechanism for hydrogen embrittlement from water or saturated vapor environments. A discussion of these mechanisms is outside the scope of this report.

STAINLESS STEELS

Available information on the effect of high-pressure hydrogen on stainless steels is limited. However, results of the program discussed in the following paragraphs indicate that the stable austenitic stainless steels are not embrittled while the martensitic, ferritic, and semiaustenitic PH stainless steels are subject to embrittlement in hydrogen-gas environments at ambient temperatures.

Effects on Tensile Properties

Several stainless steels were included in the Rocketdyne program discussed previously.[6] The steel types and compositions are shown in Table 8. Unnotched tensile specimens were 0.250 inch in diameter in the test sections (except for Type 304L and Type 305 which were 0.150 inch in diameter). Notched tensile specimens had a stress-concentration factor (K_t) of 8.4.

From a comparison of the tensile data in Tables 9 and 10 for corresponding specimens tested in the helium and hydrogen environments, it is evident that the most stable austenitic stainless steels of the series (Types 310 and 316) and the precipitation-hardening austenitic stainless steel (Type A-286) were not affected by the high-pressure hydrogen. The other austenitic stainless steels (Types 304L and 305) were only slightly affected. The martensitic and ferritic stainless steels and the precipitation-hardening semi-austenitic 17-7 PH stainless steel were substantially embrittled by the high-pressure hydrogen. Failure of Bourdon tubes of the 400 series stainless steels subjected to gaseous hydrogen is discussed in Reference (5). The stable austenitic stainless steels represent one of the few classes of alloys that are resistant to embrittlement by a hydrogen-gas environment.

Effects on Low-Cycle Fatigue Properties of Type 310

Specimens of Type 310 stainless steel in the Rocketdyne program were subjected to low cycle fatigue tensile loading in 10,000 psi helium and in 10,000 psi hydrogen.[6] These were round-notched specimens of the design used in other phases of this program, but each specimen had been fatigue loaded in air to develop a small crack around the specimen at the root of the notch. They were tested in the same equipment used for the tensile tests and the same environmental chambers were used around the specimens as in the other tests.

Results of the fatigue tests are shown in Figure 7. These limited test results indicate that the low-cycle fatigue properties

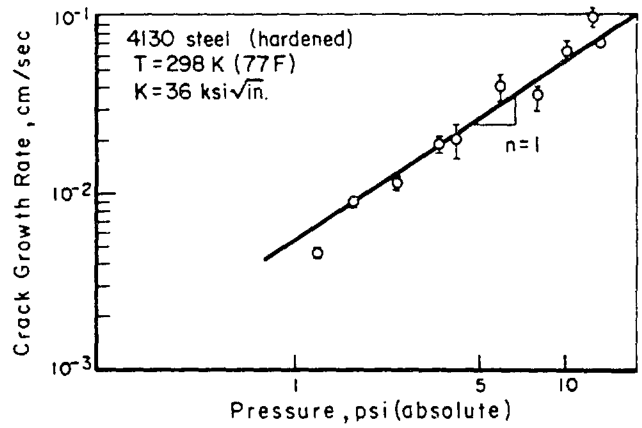

FIGURE 6. EFFECT OF HYDROGEN-GAS PRESSURE ON CRACK GROWTH RATE OF AISI 4130 STEEL[14]

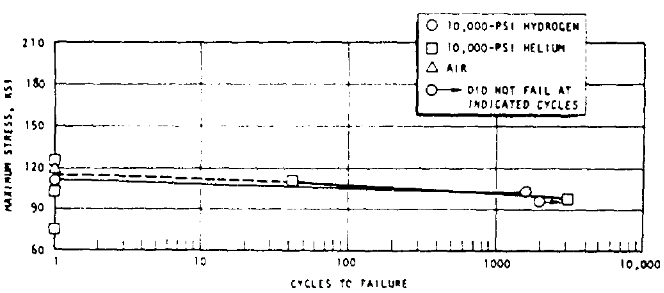

FIGURE 7. LOW-CYCLE FATIGUE CURVES FOR PRE-CRACKED SPECIMENS OF AISI TYPE 310 STAINLESS STEEL TESTED IN HELIUM OR HYDROGEN[6]

TABLE 8. CHEMICAL COMPOSITIONS OF STAINLESS STEELS IN THE ROCKETDYNE PROGRAM[6]

	Composition, percent by weight								
Material(a)	C	Mn	P	S	Si	Ni	Cr	Mo	Other
Type 304 L	0.020	1.78	0.014	0.011	0.49	9.78	18.50	0.09	0.10Cu
Type 305	0.07	0.86	0.025	0.025	0.68	11.99	18.19	--	
Type 310	0.05	1.34	0.013	0.014	0.64	20.4	24.8	0.14	0.11Cu
Type 316	0.05	1.73	0.024	0.022	0.56	12.45	17.52	2.67	0.22Cu
Type 410	0.145	0.71	0.016	0.007	0.60	0.40	12.26	0.08	0.11Cu, 0.02Al
Type 430 F	0.096	1.07	0.015	0.293	0.63	0.24	16.33	0.40	0.07Cu
Type 440 C	0.96	0.48	0.013	0.024	0.33	0.36	17.33	0.48	
Type A-286	0.052	1.47	0.019	0.010	0.61	25.58	15.07	1.35	0.13Al, 1.93Ti, 0.30V, 0.005B
17-7-PH	0.072	0.68	0.028	0.005	0.64	7.10	17.29		0.90Al

(a) Specimens were machined from 3/8-inch rod except for Type A-286.

TABLE 9. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF STAINLESS STEELS IN AIR, IN HELIUM AT 10,000 PSI, AND IN HYDROGEN AT 10,000 PSI(6)

			Tensile Properties in Air				Tensile Strength, ksi			Elongation, percent		Reduction in Area, percent		
Material	Heat Treatment	Specimen Type(a)	Yield Strength, ksi	Tensile Strength, ksi	Elongation, percent	Reduction in Area, percent	In 10,000 Psi He	In 10,000 Psi H_2	Reduction in Strength, percent(b)	In 10,000 Psi He	In 10,000 Psi H_2	In 10,000 Psi He	In 10,000 Psi H_2	Vacuum Purged
Type 304 L (austenitic)	1950 F 1 hr, air cooled	UN	24.0	80.0	79	81	77	76	–	86	79	78	71	No
		N		110		15	102	89	13			21	11	No
Type 305 (austenitic)	Annealed, cold drawn	UN	–	90.1	–	–	90	87	3	63	65	78	75	Yes
		N					165	147	11			19	17	Yes
Type 316 (austentic)	Annealed, cold drawn	UN	78	96	48	72	94	99	–	59	56	72	75	No
		N		177		17	161	161	–			18	19	No
Type 410 (martensitic)	1850 F 1 hr, OQ, 1000 F 1 hr	UN	194	221	15	60	211	166	21	7.2	1.3	13	12	Yes
		N		396		2.2	386	82	78			–	0.6	Yes
Type 430 F (ferritic)	Annealed	UN	84.0	89.0	20	60	80	78	2.5	22	14	64	37	No
		N		166		2.8	152	104	32			1.9	0.6	No
Type 440 C (martensitic)	1900 F 1 hr, OQ, 400 F 2 hr	UN	249	301	3.4	3.1	293	119	60	3.2	0	2.6	0	Yes
		N		–			149	74	50			0.6	0	No
Type A-286 (precipitation hardening)	1650 F 2 hr, WQ, aged 1325 F 16 hr	UN	118.9	161.2	24.1	42.2	158	162	–	26	29	44	43	No
		N		250		4.2	233	227	3			5.6	6.2	No
17-7PH (precipitation hardening)	1950 F 1 hr, AC, 1400 F 1.5 hr, AC, aged 1050 F 1.5 hr	UN	161.7	176.0	15	46.2	164	151	8	–	1.7	–	2.5	Yes
		N		312		0.6	302	70	77			–	0.4	Yes

(a) UN, unnotched specimens, 0.250 inch in diameter in test section, 1-inch gage length.
N, notched specimen with 60-degree V-notch, root radius 0.00095 inch ± 0.0001, and diameter at notch root 0.150 inch (K_t = 8.4).
(b) For specimens tested in hydrogen compared with those tested in helium;

TABLE 10. TENSILE PROPERTIES OF TYPE 310 STAINLESS STEEL IN HELIUM OR HYDROGEN AT VARIOUS PRESSURES(6)

		Tensile Properties in Helium				Tensile Properties in Hydrogen		
Specimen Type(a)	Helium Pressure, psi	Tensile Strength, ksi	Elongation, percent	Reduction in Area, percent	Hydrogen Pressure, psi	Tensile Strength, ksi	Elongation, percent	Reduction in Area, percent
UN					100	87	52	66
	1000	90	54	67	1000	89	55	67
	10,000	77	56	64	10,000	78	56	63
N					100	123	–	18
					1000	123	–	16
	10,000	116	–	20	10,000	108	–	18

(a) UN, unnotched; N, notched; see Table 2 for dimensions.
Note: Tensile properties in air: Unnotched, tensile strength 87 ksi, elongation 54 percent, reduction in area 66 percent; Notched, tensile strength 124 ksi, reduction in area 19 percent.

of precracked specimens of Type 310 stainless steel are not affected by a 10,000 psi hydrogen environment up to about 2,000 cycles. This behavior substantiates the tensile test data in the same environments.

ALUMINUM AND ALUMINUM ALLOYS

Although only limited information is available, aluminum and aluminum alloys represent one of the few classes of metals that do not appear to be embrittled to any appreciable extent by high-purity hydrogen gas at ambient temperature. Evidence for this view is presented below.

Effects on Tensile Properties

Notched and unnotched tensile specimens of 1100-O aluminum and 6061-T6 and 7075-T73 aluminum alloys were tested in air, in helium at 5,000 to 10,000 psi, and in hydrogen at 5,000 or 10,000 psi at Rocketdyne to determine the effect of the high-pressure hydrogen environment.(6) The unnotched specimens were 0.250 inch in diameter in the test section and the notched specimens contained a 60-degree annular V-notch designed for a stress-concentration factor (K_t) of 8.4. For specimens tested in helium or hydrogen, a small pressure vessel was slipped over the specimen with the threaded ends of the specimen protruding from the ends of the pressure vessel. Pressure seals in the ends of the pressure vessel were seated on shoulders adjacent to the threads of the tensile specimen. The hydrogen after pressurization was determined to contain <0.5 ppm oxygen, 2.6 ppm nitrogen, <0.5 ppm total hydrocarbons, < 0.5 ppm CO and CO_2, and < 0.5 ppm water. For most of the tests in 10,000 psi helium and 10,000 psi hydrogen, the system was vacuum purged as noted in Table 11. The gas pressure was then raised to 10,000 psi and the test was started immediately. The unnotched specimens were loaded at a strain rate of 0.002 minute^{-1} to the yield load and then at 0.04 minute^{-1} to fracture. The notched specimens were loaded at 0.0007 to 0.005 inch ipm to fracture. The loads were corrected for effect of pressure on the shoulder fillets and notches. The tests on specimens of 1100-O aluminum were made with 5000 psi gas pressure so the correction would be less than the yield load.

Results of these tests are summarized in Table 11. Since the tests in 10,000 psi helium involved the same pressure vessel setup as the tests in 10,000 psi hydrogen, the property values obtained in those provide a logical basis for comparison. Comparison of the data in Table 11 indicates that no substantial reduction in strength or ductility results from exposure of these alloys to the 10,000 psi hydrogen environment.

Effects on Properties of Precracked Specimens of 2219 Alloy

Aluminum Alloy 2219 was included in a program for evaluating effects of high-pressure high-purity hydrogen gas on tankage materials at Boeing when it was discovered that Alloy 718 (nickel base) was severely affected by the hydrogen environment.(15, 16)

Specimens of 2219 aluminum alloy were obtained from one 0.75-inch-thick plate and one 1.0-inch-thick plate. Welded specimens were prepared by two different weld-joint configurations and weld settings. Design of the part-through-cracked specimens is shown in Figure 8. Heat treatment of the parent metal and welded specimens consisted of solution treating at 995 F for 4 hours, water quenching, straightening, aging at room temperature for several hours, aging at 350 F for 12 hours, and cooling in air. This treatment is designated by Boeing as T6E46. Tensile properties and fracture-toughness data obtained in air at room temperature are included in Table 12.

Precracked specimens (which had been precracked by fatigue loading) were subjected to a high-pressure hydrogen-gas environment by clamping a hydrogen pressure cup to the specimen so that the cracked face of the specimen was exposed to the high-pressure hydrogen at 5200 psi. At regular intervals, the hydrogen was

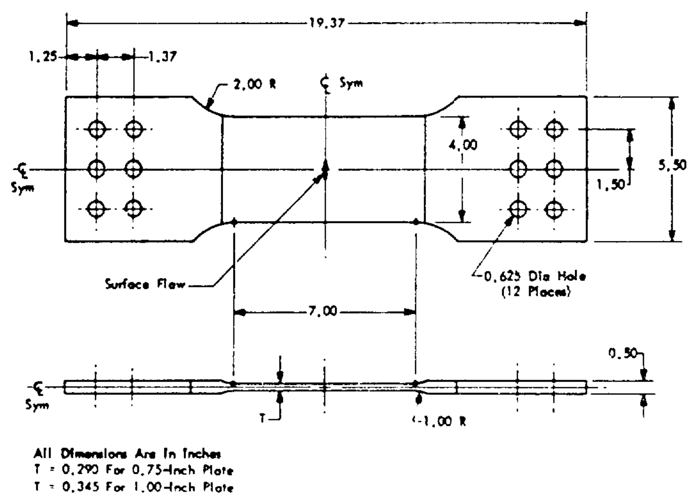

FIGURE 8. DESIGN OF PART-THROUGH-CRACK SPECIMEN OF 2219-T6E46 ALUMINUM ALLOY(15)

For welded specimens, the welds extended across the specimen at the center line and the surface flaw was in the weld metal.

TABLE 11. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF ALUMINUM ALLOYS IN AIR, IN HELIUM AT 10,000 PSI PRESSURE AND IN HYDROGEN AT 10,000 PSI PRESSURE(6)

		Tensile Properties in Air				Tensile Strength, Ksi		Elongation, percent		Reduction in Area, percent		
Material	Specimen Type(a)	Yield Strength, ksi	Tensile Strength, ksi	Elongation, percent	Reduction in Area, percent	In 10,000 Psi He	In 10,000 Psi H_2	In 10,000 Psi He	In 10,000 Psi H_2	In 10,000 Psi He	In 10,000 Psi H_2	Vacuum Purged(b)
1100-O	UN	7	14	36	90	16(b)	16(b)	42	39	93	93	Yes
	N		21		20	18(b)	25(b)			20	21	Yes
6061-T6	UN	40	46	17	56	39	40	19	19	61	66	No
	N		90		6	72	78			9.5	11	Yes
7075-T73	UN	66	74	12	32	66	65	15	12	37	35	No
	N		118		2.8	116	114			3.8	2.3	No

(a) UN, unnotched specimens, 0.250 inch in diameter in test section; N, notched specimens, 60-degree V-notch, root radius 0.00095 $\pm$ 0.0001 inch, diameter at notch root 0.150 inch ($K_t = 8.4$).

(b) Vacuum purging consisted of evacuation and backfilling with the test gas three times plus three pressurization-depressurization cycles; when not vacuum purged, three pressurization-depressurization cycles were used to establish the test environment.

(c) 5000 psi helium and hydrogen.

TABLE 12. FRACTURE TOUGHNESS IN AIR OF 2219-T6E46 ALUMINUM-ALLOY PLATE AT ROOM TEMPERATURE USING PART-THROUGH-CRACK SPECIMENS(15)

	Specimen	Thickness inch	Flaw Depth, inch	Flaw Length, inch	Fracture Stress, ksi	K_{Ic},(a) ksi $\sqrt{in.}$	Yield Strength, ksi	Tensile Strength, ksi
Parent Metal(b)	6B-1	0.292	0.170	0.722	41.9	36.9	39.4	58.0
(1-inch plate)	6B-2	0.342	0.155	0.714	43.7	34.6		
Welded Specimens	6W-1	0.341	0.164	0.722	41.2	33.4	42.1	57.8
	6W-2	0.403	0.158	0.713	40.3	30.6		

(a) Based on Kobayashi's equation: $K_I = 1.1 M_k \frac{\sigma\sqrt{\pi a}}{\sqrt{Q}}$, where M_k and Q values are dependent on flaw size and fracture stress and may be obtained from curves, σ is gross stress at failure, and a is crack depth.
(b) Transverse specimen orientation.

vented off and fresh hydrogen was added to the pressure cup to maintain the pressure. The hydrogen gas was 99.999 percent pure. In the sustained-load tests, the precracked specimens were loaded to a specified level of the stress-intensity factor and held at this load for specific lengths of time. At the end of the time period, the specimens were again fatigue loaded without the hydrogen pressure cup to develop additional fatigue cracking to mark the extent of sustained-load cracking in the fracture. The specimens were then loaded in tension to fracture. The fracture surfaces were examined to determine whether sustained-load crack growth had occurred. Results of these tests on 2219-T6E46 aluminum-alloy parent-metal specimens are shown in Figure 9. These results indicate that the threshold stress-intensity factor (K_{TH}) for this alloy exposed to hydrogen gas at 5200 psi was about 28.0 ksi $\sqrt{in.}$ for specimens from the 0.75- and 1.00-inch plates. Corresponding data (Figure 10) for weld metal in welded specimens indicated that the threshold stress-intensity factor was about 26.0 ksi $\sqrt{in.}$ for the hydrogen environment. The numbers next to the points in Figures 9 and 10 indicate the extent of crack growth in thousandths of an inch.

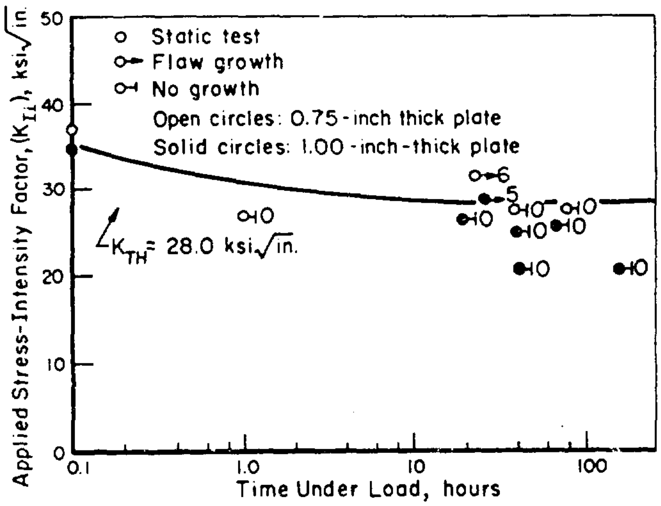

FIGURE 9. SUSTAINED-LOAD FLAW GROWTH FOR PRE-CRACKED SPECIMENS OF 2219-T6E46 ALUMINUM-ALLOY PLATE IN HYDROGEN GAS AT 5200 PSI(15)

The K_{Ic} data and the threshold stress-intensity-factor data (K_{TH}) for the parent metal and weld metal were used in plotting the curves in Figures 11 and 12. These curves relate applied stress to critical flaw depths, assuming a flaw depth-to-length ratio of 0.25. Assumed operating- and proof-stress levels are indicated in the figures. From Figure 11, a flaw greater than 0.187 inch deep in a pressure vessel wall 0.345 inch thick theoretically will cause failure at a proof pressure that develops a wall stress of 38.6 ksi in a circumferential direction (assuming a cylindrical tank with the flaw in a plane passing through the longitudinal axis of the cylinder). Therefore, proof testing with an inert fluid at this pressure will indicate, if no failure occurs, that there are no flaws larger than 0.187 inch deep in the tank walls. When the tank is pressurized with hydrogen gas to develop the operating stress level of 24.1 ksi in the tank wall, the intersection of the 24.1-ksi line with the K_{TH} curve indicates that no flaw less than 0.255 inch deep would grow under these conditions. If the tank were not proof tested and a flaw were present approximately 0.255 inch or more in depth from the inside surface, the flaw could grow in a pressurized hydrogen environment. In time, the flaw could grow to 0.315 inch depth, which is less than the wall thickness. At this point, catastrophic failure would occur. The same kind of exercise can be performed using the data plotted in Figure 12 for flaws in welds, assuming a proof stress of 29.9 ksi and an operating stress of 18.7 ksi. If the operating stress were 16.0 ksi, a large flaw would grow through the wall thickness before reaching the critical level of stress-intensity factor and leakage would occur before catastrophic failure when the tank was pressurized with hydrogen.

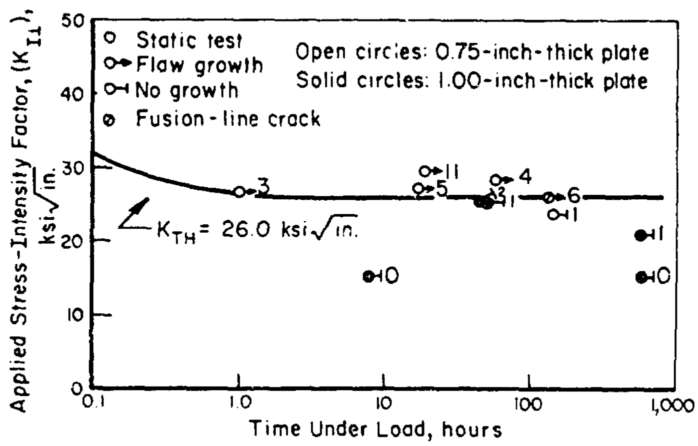

FIGURE 10. SUSTAINED-LOAD FLAW GROWTH IN WELD METAL OF PRECRACKED SPECIMENS OF 2219-T6E46 ALUMINUM-ALLOY PLATE IN HYDROGEN GAS AT 5200 PSI(15)

Boeing's study has indicated that the 2219-T6E46 aluminum alloy is superior for high-pressure hydrogen-gas tankage to such alloys as Alloy 718 and Ti-6Al-4V, which are discussed later.

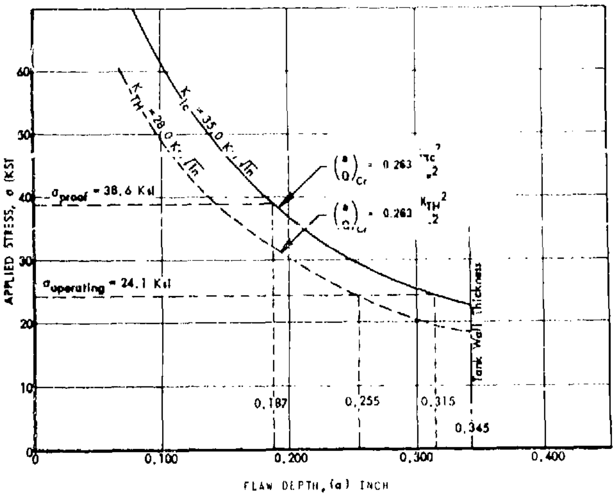

FIGURE 11. THRESHOLD FLAW DEPTH FOR 2219-T6E46 ALUMINUM ALLOY IN HYDROGEN GAS AT 5200 PSI(15)

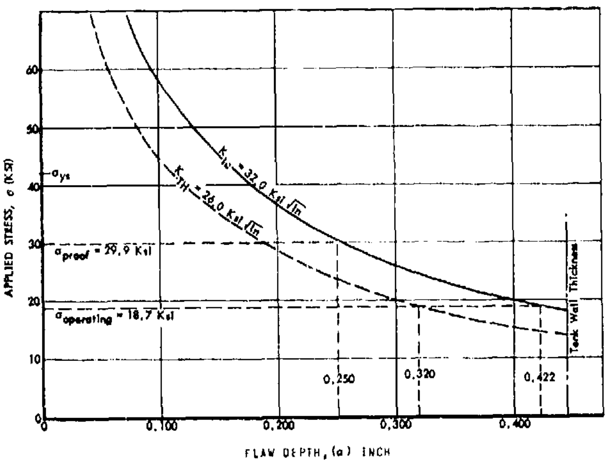

FIGURE 12. THRESHOLD FLAW DEPTH FOR WELDS IN 0.445 INCH THICK PLATE OF 2219-T6E46 ALUMINUM ALLOY IN HYDROGEN GAS AT 5200 PSI(15)

COPPER AND BERYLLIUM COPPER

Tensile specimens of OFHC copper and beryllium copper were evaluated in 10,000 psi hydrogen gas along with specimens of other alloys in the Rocketdyne program.(6) The tensile specimen design was the same as that used for other specimens in the program. Environmental chambers were positioned around the specimens to provide environments of helium at 10,000 psi pressure or hydrogen at 10,000 psi pressure. Results of the tests, shown in Table 13, indicate that copper and the beryllium-copper alloy apparently are not embrittled by environmental hydrogen at high pressures.

NICKEL AND NICKEL-BASE ALLOYS

Several nickel-base alloys, particularly Alloy 718, have been used for certain aerospace applications that required relatively high strength at high temperatures or relatively high strength, good toughness, and compatibility with liquid oxygen and other cryogenic fluids at low temperatures. However, no tests had been made on notched specimens of nickel or nickel-base alloys in hydrogen-gas environments at ambient temperature until the past few years, when the data discussed in this section were obtained. These data indicate that nickel, Alloy 718, and René 41 are embrittled in high-pressure hydrogen at ambient temperature.

Effects on Tensile Properties of Nickel, Alloy 718, and René 41

Tensile specimens of nickel, Alloy 718, and René 41 were evaluated in hydrogen at high pressure on the Rocketdyne program.(6) Specimen designs and equipment were the same as discussed in previous sections. Heat treatments and tensile properties are presented in Table 14. The strength of the notched specimens tested in 10,000 psi hydrogen was from 30 to 73 percent lower than that of specimens tested in 10,000 psi helium. In 1,000 psi helium and 1,000 psi hydrogen, the unnotch tensile properties of Alloy 718 were nearly the same as those in air. However, the notch strengths of the Alloy 718 specimens were 282 ksi in 1,000 psi helium and 169 ksi in 1,000 psi hydrogen. This represents a 42 percent reduction in notch strength at the lower hydrogen pressure.

The extent of embrittlement is somewhat dependent on strength level, alloy, and hydrogen pressure, but these data indicate that a hydrogen environment can cause embrittlement in nickel and nickel-base alloys.

Effects on Center-Notch Specimens of Alloy 718

Alloy 718 has been used extensively in aerospace tankage for liquid oxygen. This alloy is readily fabricated and has relatively high strength and toughness in the solution-treated-and aged condition at room temperature and at cryogenic temperatures. Because of these factors, Alloy 718 was used in constructing tanks for hydrogen gas for the Mariner 69 space vehicle. When data from the Rocketdyne program indicated that Alloy 718 could be embrittled by hydrogen gas, a program was initiated at Boeing to provide further information on embrittlement of this alloy in hydrogen.(15, 16)

In Boeing's program, flat plate specimens of Alloy 718 were machined in the longitudinal direction from forged bars about 4.5 inches in diameter. To conserve test material and reduce the amount of sawing, each test plate was 4.1 inches long, and one piece of 3/8-inch plate of Alloy 718 was welded to each end of each of the test plates to produce parent-metal specimens 12 inches long. Weld-metal specimens were prepared by welding two 0.312 x 3.5 x 2.8-inch pieces together and then welding one piece of 3/8-inch plate on each end.

The parent metal specimens were solution annealed at 1750 F for 1 hour, air cooled to room temperature, duplex aged at 1325 F for 8 hours, furnace cooled to 1150 F, then held at 1150 F for a combined total aging time of 18 hours. The weld-metal-specimen blanks were solution annealed at 1750 F for 1 hour and then air cooled to room temperature before welding. The gas-tungsten-arc process was used in welding the weld-metal test plates together. One welding pass was made on each side. These welded specimens were then subjected to the same duplex aging treatment as that for the parent-metal specimens. Configurations of the finish-machined specimens are shown in Figures 13 and 14. A part-through crack was produced at the midsection of each of these specimens by electrical discharge machining small notches in the surface and subjecting each specimen to fatigue loading to develop the crack. The crack sizes are given in the tables along with the other data.

Preliminary tests on several of these precracked specimens in air at room temperature indicated that both the parent-metal and weld-metal specimens were too thin to be used for the generation of valid K_{Ic} (plane-strain fracture toughness) values. Several

TABLE 13. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF COPPER AND BERYLLIUM COPPER IN AIR, IN HELIUM AT 10,000 PSI, AND IN HYDROGEN AT 10,000 PSI(6)

Material	Condition	Specimen Type(a)	Tensile Properties in Air: Yield Strength, ksi	Tensile Properties in Air: Tensile Strength, ksi	Tensile Properties in Air: Elongation, percent	Tensile Properties in Air: Reduction in Area, percent	Tensile Strength, ksi: In 10,000 Psi He	Tensile Strength, ksi: In 10,000 Psi H_2	Reduction in Strength, percent	Elongation, percent: In 10,000 Psi He	Elongation, percent: In 10,000 Psi H_2	Reduction in Area, percent: In 10,000 Psi He	Reduction in Area, percent: In 10,000 Psi H_2	Vacuum Purged
OFHC Copper (99.99 Cu)	Cold drawn	UN	–	52	14	89	42	41	–	20	20	94	94	Yes
		N		98		22	87	86	–			20	24	Yes
Be-Cu Alloy 25, (1.86 Be, 0.22Co)	Condition H(b)	UN	92	104	18	67	94	93	–	22	22	72	71	Yes
		N		209		11	195	181	7.0			12	13	Yes

(a) UN-unnotched specimen, 0.250 inch in diameter in test section, l-inch gage length. N-notched specimen with 60-degree V-notch, root radius 0.00095 inch ± 0.0001 inch, and diameter at notch root 0.150 inch (K_t = 8.4).
(b) Heat treated by supplier.

TABLE 14. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF NICKEL AND NICKEL-BASE ALLOYS IN AIR, IN HELIUM AT 10,000 PSI, AND IN HYDROGEN AT 10,000 PSI(6)

Material	Heat Treatment	Specimen Type(a)	Tensile Properties in Air: Yield Strength, ksi	Tensile Properties in Air: Tensile Strength, ksi	Tensile Properties in Air: Elongation, percent	Tensile Properties in Air: Reduction in Area, percent	Tensile Strength, ksi: In 10,000 Psi He	Tensile Strength, ksi: In 10,000 Psi H_2	Reduction in Strength, percent	Elongation, percent: In 10,000 Psi He	Elongation, percent: In 10,000 Psi H_2	Reduction in Area, percent: In 10,000 Psi He	Reduction in Area, percent: In 10,000 Psi H_2	Vacuum Purged
Nickel 270 (99.9Ni)	Hot rolled	UN	22	58.5	56	90	48	50	–	–	52	–	66	Yes
		N		85		24	78	54	30			23	6.9	Yes
Alloy 718	1750 F 1 hr, AC, 1325 F 8 hr, FC to 1150 F, held 10 hr, AC	UN	193	213	16	27	208	193	7	17	1.5	26	0.8	Yes
		N		282		1.7	274	126	54			1.7	0.2	Yes
René 41	1950 F 1 hr, WQ, 1400 F 16 hr, AC	UN	180	206	21	30	196(b)	165	16	11	4.3	14	11	Yes
		N		290			280(b)	77	73			1.9	0.2	Yes

(a) UN-unnotched specimen, 0.250 inch in diameter in test section, l-inch gage length. N-notched specimen with 60-degree V-notch, root radius 0.00095 inch ± 0.0001 inch and diameter at notch root 0.150 inch (K_t = 8.4).
(b) Strength in air corrected for effect of pressure on environmental cell.
Note: Chemical analyses, percent by weight: Alloy 718, 0.04C, 0.16Mn, 0.33Si, 18.37Cr, 3.13Mo, 19.8Fe, 0.52Al, 0.92Ti, 0.12Cu, 5.39Cb+Ta; René 41, 0.082C, 18.88Cr, 9.79Mo, 10.9Co, 0.64Fe, 1.51Al, 3.09Ti, 0.006B.

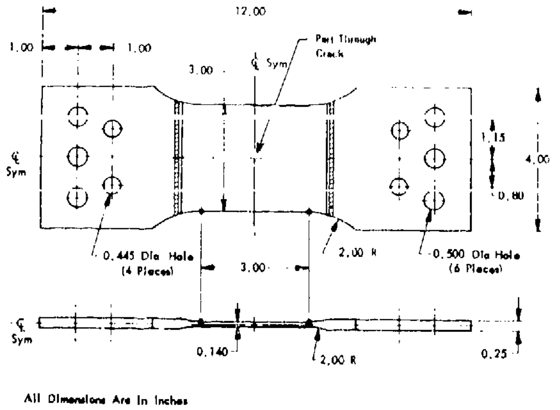

FIGURE 13. SPECIMEN CONFIGURATION FOR PART-THROUGH-CRACKED PARENT-METAL SPECIMENS OF ALLOY 718(15)

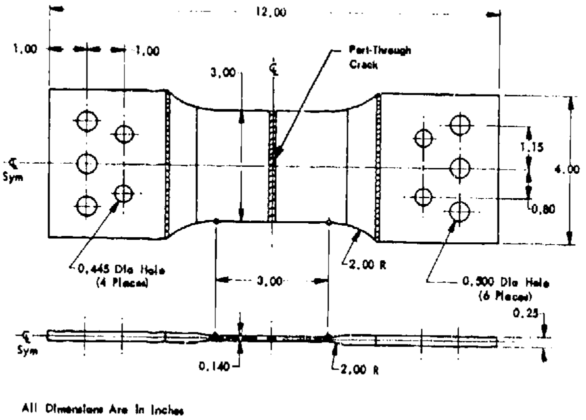

FIGURE 14. SPECIMEN DESIGN FOR PART-THROUGH-CRACKED WELDS IN SPECIMENS OF ALLOY 718(15)

additional specimens were prepared nearly twice as thick in order to obtain a better approximation of the fracture toughness of the parent metal and weld metal. The data obtained from testing these specimens in air at room temperature are shown in Table 15. The information was needed to provide base values so that the extent of damage from the hydrogen environment could be determined. Only one of the precracked parent-metal specimens failed at stresses less than the yield strength, and the calculated K_{Ic} value for this specimen was 150 ksi $\sqrt{\text{in.}}$ by Kobayashi's method (See Table 15). The average K_{Ic} value for two weld-metal specimens was 95.0 ksi $\sqrt{\text{in.}}$

Results of the sustained-load flaw-growth tests for Alloy 718 parent metal when exposed to hydrogen gas at 5200 psi are shown in Figure 15. The curve represents the lower bound line for detectable flaw growth. On the basis of these data, the threshold stress-intensity factor under these conditions was located at 22.0 ksi $\sqrt{\text{in.}}$ Corresponding data for preflawed 6-inch-diameter tanks of Alloy 718 also subjected to hydrogen-gas pressure are shown in Figure 15. Results of sustained-load flaw-growth data in 718 weld metal in high-pressure hydrogen are shown in Figure 16.

The curves in Figure 17 show the relationship between flaw depth (a) and applied stress (σ) in an air environment for K_{Ic}

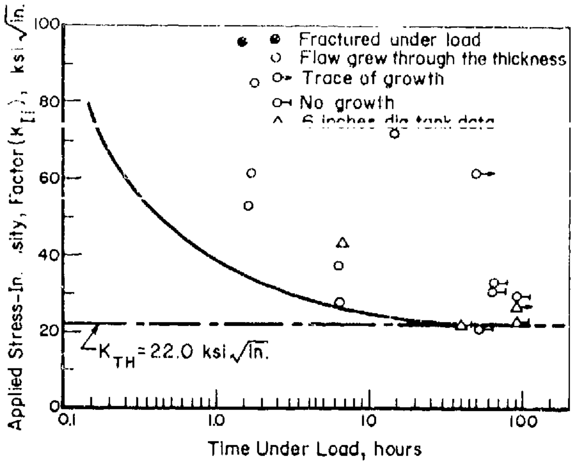

FIGURE 15. SUSTAINED-LOAD FLAW GROWTH FOR PRECRACKED SPECIMENS OF NICKEL-BASE ALLOY 718 IN HYDROGEN GAS AT 5200 PSI(15)

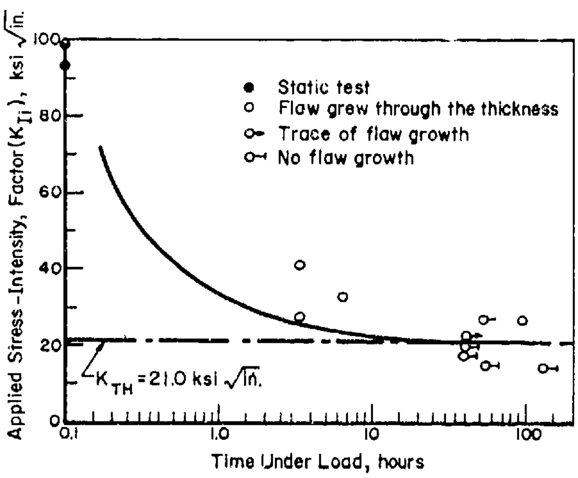

FIGURE 16. SUSTAINED-LOAD FLAW GROWTH IN WELD METAL OF PRECRACKED SPECIMENS OF NICKEL-BASE ALLOY 718 IN HYDROGEN GAS AT 5200 PSI(15)

of 150 ksi $\sqrt{\text{in.}}$ and in high-pressure hydrogen for K_{TH} of 20 ksi $\sqrt{\text{in.}}$ For these curves, the flaw depth-to-length ratio is 0.25 and the assumed wall thickness is 0.120 inch. Values for yield strength (σ_{ys}) and assumed operating stress (σ_{op} = 66.0 ksi) are indicated on the stress scale. For the high-pressure hydrogen environment, the initial flaw depth should not exceed 0.032 inch, corresponding to a crack length of 0.128 inch. Any internal flaw larger than this would grow if the tank were pressurized with hydrogen at the operating-stress level. Under these conditions, the crack would eventually grow through the wall (to a depth of 0.120 inch). Since the operating stress is considerably lower than the K_{Ic} curve at 0.120-inch, it is expected that a leak-before-fracture condition would occur (rather than catastrophic failure). Because of the high toughness of Alloy 718 in air and the relatively low K_{TH} value in high-pressure hydrogen, a proof test with an inert pressurizing fluid could not be used to eliminate pressure vessels containing flaws 0.032 inch deep. Such flaws must be located by nondestructive tests.

The curves in Figure 18 apply to the K_{Ic} value of 95.0 ksi $\sqrt{\text{in.}}$ for Alloy 718 weld metal and the threshold K_{TH} value

TABLE 15. FRACTURE TOUGHNESS OF ALLOY 718 IN AIR AT ROOM TEMPERATURE USING PART-THROUGH-CRACK SPECIMENS(15)

	Specimen	Thickness, inch	Width, inches	Flaw Depth, inch	Flaw Length, inch	Fracture Stress, ksi	K_{Ic},(a) ksi $\sqrt{in.}$	Yield Strength, ksi	Tensile Strength, ksi
Parent Metal	1B-3	0.256	2.51	0.171	0.812	153.8	150	174	195
Weld metal	1W-1	0.153	2.93	0.094	0.387	141.0	92.8		
	1W-2	0.246	2.46	0.140	0.570	128.9	98.7		

(a) Based on Kobayashi's equation: $K_I = 1.1 M_k \frac{\sigma\sqrt{\pi a}}{\sqrt{Q}}$ where M_k and Q values are obtained from curves, σ is gross stress, and a is crack depth.

Note: Composition, percent by weight: 0.02C, 0.11Mn, 0.010S, 0.12Si, 18.1Cr, 52.8Ni, 5.16Cb+Ta, 1.05Ti, 0.55Al, 3.11Mo, 0.03Cu, 0.05Co, 0.004B, balance Fe.

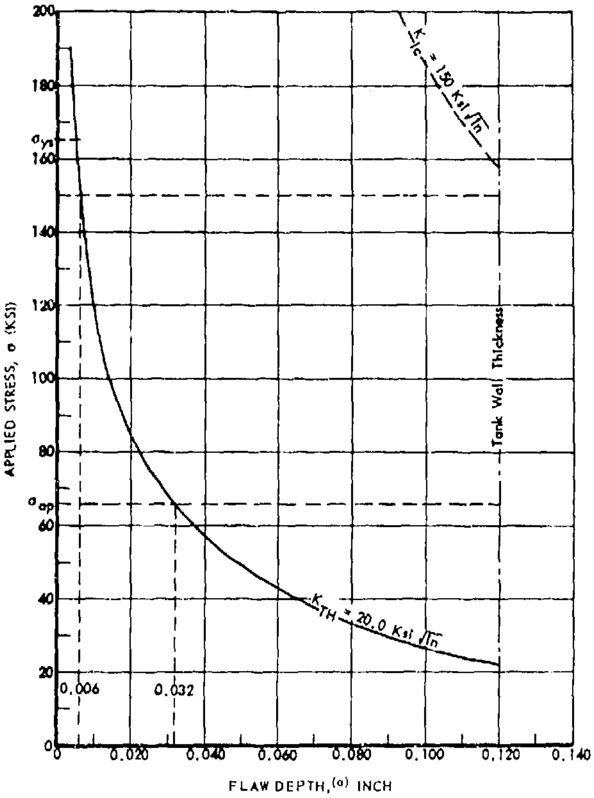

FIGURE 17. THRESHOLD FLAW DEPTH FOR NICKEL-BASE ALLOY 718 IN HYDROGEN GAS AT 5200 PSI(15)

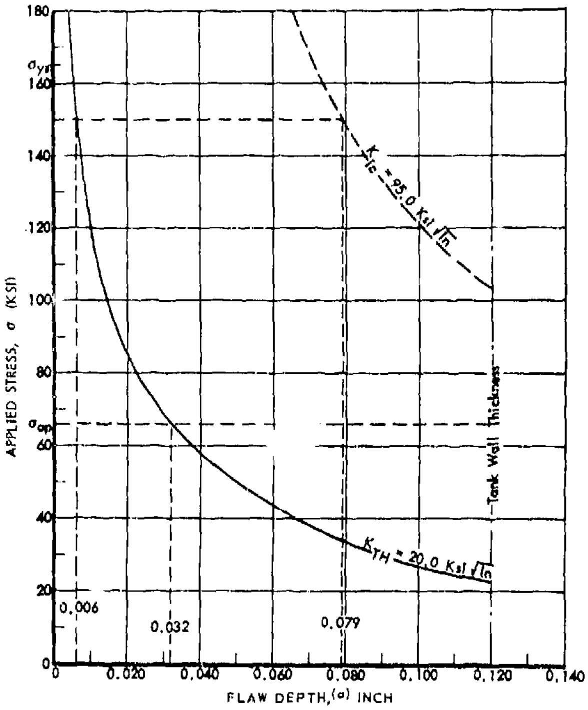

FIGURE 18. THRESHOLD FLAW DEPTH FOR WELDMENTS IN NICKEL-BASE ALLOY 718 IN HYDROGEN GAS AT 5200 PSI(15)

of 20 ksi $\sqrt{in.}$ for Alloy 718 in high-pressure hydrogen. As in the parent metal, flaws deeper than 0.032 inch in the weld metal would eventually grow through the wall at a stress of 66.0 ksi in the high-pressure hydrogen environment. At a 150-ksi stress level for the hydrogen environment, flaw growth would occur for flaws 0.006 inch deep or greater. As soon as the flaw depth in the weld metal reached 0.079 inch, catastrophic fracture would occur, since the K_{Ic} curve passes through the 150-ksi stress level at 0.079-inch flaw depth. However, proof testing with an inert fluid could not be used to locate flaws 0.032 inch deep in the weld metal. This condition again would place full responsibility for flaw detection on the nondestructive inspection facilities.

Because of the extensive reduction in fracture toughness resulting from a high-pressure high-purity hydrogen environment, Alloy 718 is not recommended for gaseous hydrogen tankage.

Effects on Notch Strength of K Monel

K Monel was one of the alloys evaluated in high-pressure nitrogen and hydrogen environments at Rensselaer Polytechnic Institute.(8) The major diameter of the specimens was 0.306 inch and the stress-concentration factor (K_t) of the notched specimens was 4.2. Unnotch tensile properties of the annealed alloy in air were 47-ksi yield strength, 100-ksi tensile strength, and 40.1 percent elongation in 1-1/4 inches. Notch strength in 10,000 psi nitrogen was 144 ksi and that in 10,000 psi hydrogen was 105 ksi. There is a 27 percent reduction in notch strength in the high-pressure hydrogen.

Unnotch tensile properties of the precipitation-hardened K Monel in air were 139-ksi tensile strength and 8.7 percent elongation in 1-1/4 inches. Notch strength in 10,000 psi nitrogen was 251 ksi and that in 10,000 psi hydrogen was 113 ksi for the precipitation-hardened alloy, representing a 55 percent reduction in notch strength. As for other alloys, the effect of the hydrogen environment is greater at the higher strength level.

TITANIUM AND TITANIUM ALLOYS

Because of its favorable strength-weight ratio and its good toughness at -423 F, the Ti-5Al-2.5Sn (ELI) alloy is often selected for liquid-hydrogen tankage for spacecraft applications. Fittings and tubing for these applications often are either titanium or one of the titanium alloys. These titanium and titanium-alloy components, therefore, are exposed to hydrogen gas at temperatures from -423 F to above ambient temperature at variable pressures. A failure in one of these systems was cause for an extensive study of the effects of hydrogen environments on titanium and titanium alloys.(17, 18, 19) Leakage had occurred at a weld between titanium tubing and a fitting as a result of the formation of titanium hydride and the spalling away of the hydride in the area of the weld. However, the hydride formation was not limited to the weld.

A research program was initiated at Battelle to determine what conditions promote and inhibit the hydride reaction between hydrogen gas and titanium.(18, 20) Experiments conducted in this program demonstrated that titanium hydrides could be formed at 1-atmosphere pressure of high-purity hydrogen gas at ambient temperature. However, there are a number of variables that produce rather subtle effects when one tries to obtain consistent results. Factors which enhance the reaction are increased hydrogen pressure, increased reaction time, abrasion of the titanium surface immediately before exposure to hydrogen, galling of the surface with other metals (especially in the presence of oil), and vacuum annealing to dissolve surface oxides before hydrogen exposure. The amount of hydriding for a given set of conditions was greater for acicular structures than for equiaxed structures. Unalloyed titanium and Ti-5Al-2.5Sn alloy appeared to be more reactive with hydrogen gas than did Ti-6Al-4V alloy when surface oxidation was a factor. However, when oxidation of the surface was prevented, the Ti-6Al-4V alloy was more reactive than titanium and Ti-5Al-2.5Sn alloy. The beta-containing alloys appear to be more susceptible than the alpha alloys to severe damage from the reaction to hydrogen gas, because of the higher solubility of hydrogen in the beta phase. The rate of the hydride reaction was not consistently affected by stress on the specimen.

Impurities in the hydrogen gas and an oxide layer on the titanium (resulting from exposure to air) inhibited the reaction with hydrogen.

Effects on Tensile Properties

Effect of 10,000 psi hydrogen on the tensile properties of titanium, Ti-5Al-2.5 Sn alloy, and Ti-6Al-4V alloy for unnotched and notched specimens evaluated in the Rocketdyne program are shown in Table 16.(6) Reduction in strength resulting from exposure to the high-pressure hydrogen environment was observed for the notched specimens only. These tests represent relatively short times for loading to fracture. Since hydride formation is time dependent, the short-time tensile tests do not indicate the effect of an extended-time exposure.

Effects on Fatigue Properties of Ti-6Al-4V

Annealed Ti-6Al-4V specimens from 0.080-inch sheet were used in a study at McDonnell Douglas to show the effects of a hydrogen-gas environment and other environments on the fatigue properties of precracked specimens.(21) Yield strength of the alloy was 139 ksi, tensile strength 144 ksi, and elongation 14 percent. The alloy contained 0.23 percent carbon, 0.08 percent iron, 6.0 percent aluminum, 4.1 percent vanadium, 0.010 percent nitrogen, 0.12 percent oxygen, and 0.004 percent hydrogen.

The fatigue specimens were 1 inch wide in the test sections. Each specimen had a 0.100-inch-long, part-through fatigue crack in one surface at the center of the test section. Each fatigue crack was produced by making an Eloxed starter notch at the center of the test section and fatigue cycling the specimen in flexure until a 0.100-inch-long fatigue crack had been developed at the notch. The purpose of the preliminary cracks, which were produced in an air atmosphere, was to provide uniform starting conditions for the crack-propagation tests.

The precracked specimens were cycled under axial loading conditions in a controlled atmosphere which was maintained in an insulated sealed retort around the specimen. Before starting each test, the test chamber was vacuum purged and backfilled with nitrogen, helium, or hydrogen gas. Oxygen content of the hydrogen gas was not more than 1 ppm. A gas pressure of 2 psi above atmospheric pressure was maintained in the test chamber throughout the test. The lower stress during tension-tension cycling was 12.5 ksi and the upper stress was 62.5 ksi on the uncracked area of the test section. Cyclic rate was 80 cycles per minute. Low temperatures were obtained by either spraying the specimens with cold gas from a liquefied-gas storage dewar or by submersing the specimens in liquid nitrogen or liquid hydrogen in a cryostat.

Results of the fatigue tests are summarized in Figure 19. The number of cycles to failure is the number of cycles, starting with the precracked specimen, to cause failure at the maximum cyclic gross stress of 62.5 ksi. As crack growth occurred during cycling, a clean fracture surface was continually produced for exposure to the environment. The number of stress cycles to fracture for specimens exposed to different environments depended on the environment and the test temperature. The data in Figure 19 indicate that the hydrogen-gas environment had a marked effect

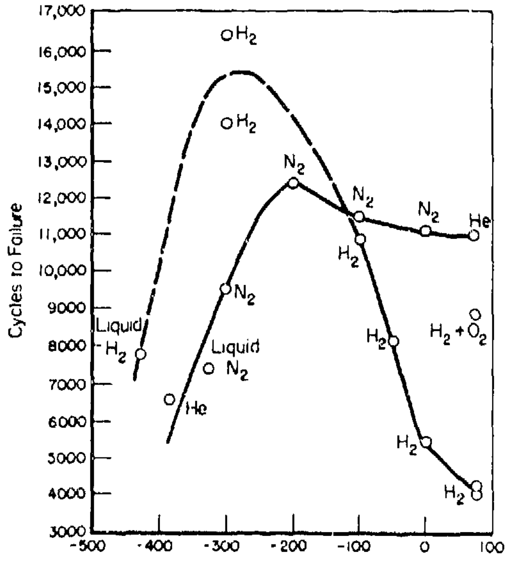

FIGURE 19. FATIGUE LIVES OF PRECRACKED SPECIMENS OF Ti-6Al-4V ALLOY TESTED IN HYDROGEN GAS AND OTHER ENVIRONMENTS AT A PRESSURE OF 2 PSI ABOVE ATMOSPHERIC PRESSURE UNDER TENSION-TENSION LOADING AT A LOWER STRESS LEVEL OF 12.5 KSI AND UPPER STRESS LEVEL OF 62.5 KSI(21)

TABLE 16. AVERAGE TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED SPECIMENS OF TITANIUM AND TITANIUM-ALLOY SPECIMENS IN AIR, IN 10,000 PSI HELIUM, AND IN 10,000 PSI HYDROGEN(6)

			Tensile Properties in Air				Tensile Strength, ksi			Elongation, percent		Reduction in Area, percent		
Material	Condition	Specimen Type(a)	Yield Strength, ksi	Tensile Strength, ksi	Elongation percent	Reduction in Area, percent	In 10,000 Psi He	In 10,000 Psi H_2	Reduction in Strength, percent	In 10,000 Psi He	In 10,000 Psi H_2	In 10,000 Psi He	In 10,000 Psi H_2	Vacuum Purged
Titanium (Commercially pure)	Hot rolled	UN	56	70	25	52	63	61	–	32	31	61	61	Yes
		N		141		11	126	120	5			10	7.3	Yes
Ti-5Al-2.5Sn(b)	Annealed 1400 F	UN	116	121	18	46	113	114	–	20	18	45	39	Yes
		N		205		3.7	201	162	19			3.1	1.8	Yes
Ti-6Al-4V(c)	Annealed	N					243	156	36			2.2	1.9	Yes
Ti-6Al-4V(d)	Annealed	N					227	166	27			2.2	1.7	Yes

(a) UN-unnotched; N-notched ($K_t = 8.4$).
(b) Composition, percent by weight: 5.47Al, 2.70Sn, 0.09Fe, 0.011C, 0.0122 H_2, 0.09 O_2.
(c) Composition, percent by weight: 6.0Al, 4.1V, 0.15Fe, 0.24C, 0.024N_2, 0.008 H_2, 0.019 O_2.
(d) Composition, percent by weight: 6.4Al, 4.2V, 0.15Fe, 0.023C, 0.014N_2, 0.008 H_2, 0.14 O_2.

in increasing the crack growth rate at all testing temperatures above -100 F as compared with crack growth rates for precracked specimens tested in nitrogen or helium gas. When the hydrogen gas was contaminated with oxygen, the crack growth rate was retarded, as shown in Figure 19 for the point marked $H_2 + O_2$. When the hydrogen gas was contaminated with oxygen, a protective oxide probably formed on the new fracture surfaces and inhibited the hydrogen-titanium reaction. This behavior is consistent with that indicated by information from other sources.

At temperatures below -100 F, specimens tested in the hydrogen-gas environment had fatigue lives longer than those of corresponding specimens tested in nitrogen or helium gas. This indicates that the cold hydrogen gas in a liquid-hydrogen storage tank would not have an adverse effect on the fatigue life of the tank if it were annealed Ti-6Al-4V alloy. The downward trend of the fatigue curves in Figure 19 for tests made at temperatures from -200 F to -423 F is probably the result of increasing embrittlement (increasing notch sensitivity) as the testing temperatures were decreased.

Hydrogen analysis of filings from the fracture surface of the specimen tested at 0 F in hydrogen indicated a hydrogen content 16 times greater than that for the unexposed alloy. Examination of the fractures by electron-microscope techniques indicated distinct differences between the fracture surfaces produced in helium or nitrogen environments and those produced in the hydrogen environment at ambient temperature, 0 F, and -50 F. Fracture surfaces of specimens tested in hydrogen gas at temperatures above -100 F contained discontinuous cracks at the fatigue striations. Fracture surfaces of specimens tested in helium or nitrogen at the same temperatures contained relatively flat fields of continuous fatigue striations.

Since this program has shown that exposure to pure hydrogen gas at a pressure as low as 2 psi above atmospheric pressure can cause marked reductions in fatigue lives for precracked specimens of Ti-6Al-4V alloy at temperatures above -100 F as compared with specimens tested in nitrogen and helium gases, the use of this alloy is not recommended for service in hydrogen environments above -100 F. Hydrogen-gas environments at higher pressures are likely to cause even greater reductions in fatigue lives. It must be assumed that other titanium alloys will behave similarly under these conditions until additional tests have been conducted to provide additional information for them.

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APPENDIX

PROGRAM OF TMS-AIME SYMPOSIUM

"EFFECTS OF GASEOUS HYDROGEN ON METALS"

A one-day workshop session cosponsored by the Non-Ferrous Committee of TMS-AIME and the Aerospace Panel of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals will be held at the spring meeting of the Non-Ferrous Metallurgy Committee, Institute of Metals Division, Metallurgy Society of AIME, in Las Vegas, May 11-14, 1970. The tentative program is as follows:

Chairman: D. C. Goldberg
Co-Chairman: R. Raring

Session 1. Definition of the Problem, Test Techniques Employed, Data Accumulated Pertaining to the Problem

(a) Film "Hydrogen Challenge" J. P. Fidelle, Commissariat a L'Energy Atomique (16 mm - 18 minutes)

(b) Discussion of Several Aspects of GH_2 Embrittlement of Metals, D. P. Williams, H. G. Nelson of NASA-AMES

*(c) Fracture Mechanics Examination of H_2-Metal Reactions, R. G. Forman, NASA-MSC

*(d) Slow Crack Growth in Ti Alloys Exposed to Low Pressures of H_2 Gas, H. L. Marcus, B. S. Hickman, J. C. Williams, G. Garmong, and P. Stocker, Science Center, North American Rockwell

*(e) Effects of Temperature and Pressure on the Hydriding of Ti-5Al-2.5Sn (ELI), R. L. Kesterson, Westinghouse Astronuclear Laboratory

(f) Effect of Pure Gaseous H_2 on Tensile Properties of Ni-Base Alloys, I. M. Rehn, P. P. Dessau, C. E. Dixon and C. W. Funk, Aerojet-General Corporation, Sacramento

Session 2. Development of Rationale for the Damage Mechanism in Various Materials

*(a) Environmental H_2 Embrittlement of a Ferrous Alloy: Embrittlement in Dissociated Hydrogen, Howard G. Nelson, Dell P. Williams, Alan S. Tetelman, of University of California, Los Angeles and NASA-AMES.

(b) Rocket Engine Materials in High Pressure Hydrogen, G. R. Janser, Aeroject-General Corporation, Sacramento

(c) Discussion

*Papers available for publication.

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13. ABSTRACT

On the basis of the information available, steels (ferritic, martensitic, and bainitic), nickel-base alloys, and titanium alloys become embrittled in pure-hydrogen-gas environments at ambient temperature. The embrittling effect is detected by making tension tests on sharp-notched specimens in an environment of high-purity hydrogen gas and, for comparison, tests on similar specimens in an inert gas at the same temperature and pressure. If the material is embrittled by hydrogen, its notch tensile strength will be reduced. The effect is more pronounced as the hydrogen-gas pressure is increased, but in some cases the embrittling effect has been observed at 1 atmosphere of pressure. The effect is more pronounced for the high-strength steels and high-strength nickel and titanium alloys than for the low-strength alloys. In unnotched specimens exposed to a pure-hydrogen environment, hydrogen embrittlement manifiests itself as a decrease in ductility.

Results of tests on stable austenitic stainless steels such as Types 310 and 316, or certain aluminum alloys such as 6061-T6, 2219-T6, and 7075-T73, and beryllium copper indicate that there is no significant evidence of embrittlement of these alloys in hydrogen gas at pressures up to 10,000 psi.

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
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High pressure						
Hydrogen						
Mechanical properties						
Nickel-base alloys						
Pressure vessels						
Steels						
Tensile strength						
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