VON KARMAN CENTER

STRUCTURAL MATERIALS DIVISION

STRESS-CORROSION CRACKING OF HIGH-STRENGTH ALLOYS

A Report To

FRANKFORD ARSENAL

Contract DA-04-495-ORD-3069

Report No. 0414-02-2 (Quarterly) / January 1964 / Copy No. 4

AEROJET
GENERAL TIRE
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AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

This is the eleventh in a series of quarterly progress reports submitted in partial fulfillment of contract DA-04-495-ORD-3069. It constitutes the second quarterly progress report for the second 1-year continuation of the original 2-year program.

This report covers the period 1 October through 31 December 1963. It was written by R. B. Setterlund, who was supervised by A. Rubin.

AEROJET-GENERAL CORPORATION

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CONTENTS

I. OBJECTIVES

The objectives of the program are outlined below.

A. To study the stress-corrosion characteristics of 18%-nickel maraging steel with respect to compositional variation.

B. To study the effect of environmental temperature on the rate of stress-corrosion cracking in three alloys: 18%-nickel maraging steel, a low-alloy martensitic steel, and a hot-worked die steel.

C. To study the electropotential changes occurring in 18%-nickel maraging steel during stress-corrosion exposure and the effect of applied potential.

D. To evaluate the effectiveness and applicability of surface protection on 18%-nickel maraging steel in preventing stress-corrosion cracking.

II. WORK PROGRESS

A. BACKGROUND

The present program is the second 1-year extension of the original 2-year program on stress-corrosion cracking of high-strength alloys. During the first 2-year study, six alloys were evaluated: Ladish D6AC steel; Type 300 M steel; Vascojet 1000 steel; AM 355 cold-worked PH steel; precipitation-hardening 15-7Mo stainless steel; and B120VCA titanium. Significant failures were found to occur with the D6AC, 300 M, and Vascojet 1000 steels in tap, distilled, and salt water, as well as in high-humidity environments; the time to failure for each of the three steels was found to decrease with increasing strength.

During the first 1-year continuation program, attention was focused on three new high-strength steels plus one high-strength titanium alloy. These alloys are (1) 20%-nickel maraging steel, (2) 18%-nickel maraging steel, (3) 9Ni-4Co vacuum melted steel, and (4) 6Al-4V titanium. The titanium alloy showed complete immunity to stress-corrosion failure under all test conditions. Limited susceptibility was noted for the 9Ni-4Co alloy. High susceptibility was noted with both the 20%- and 18%-nickel maraging steels. Since the original study of maraging steels was started, the 18%-nickel grade has received increased attention in the aerospace industry, and is now of primary interest. The present program is therefore directed to the study of the stress-corrosion behavior of this one alloy, with emphasis on compositional variation, effect of environmental temperature, and study of electropotential changes. It is intended to: first, determine the extent of the stress-corrosion problem in 18%-nickel maraging steel by testing four additional heats; second, compare the susceptibility of maraging steel with conventional ultra-high-strength steels; and, third, to investigate further, by electropotential methods, the cause of failure of 18%-nickel maraging steel.

B. TEST PROCEDURES

1. Bent Beam Test

The bent-beam test is the primary test method used in the program. Figure 1 shows an insulated bent-beam fixture with test samples mounted. Polycarbonate blocks 7.000 ± 0.001 in. apart, attached to a stainless-steel holder, support the test specimen and insulate it from the holder. Specimens are cut to exact length to give a maximum outer-fiber stress of 75% of the 0.2% offset yield strength. The length-stress relationship is shown in Figure 2.

2. U-Bend Test

In addition to the bent beam testing, U-bend samples are used to show the effect of elastic stresses combined with plastic deformation. Samples are bent in a special 1 in. radius fixture after heat treatment and cleaning. Samples which were known to have a low ductility were warmed to 100 to 175°F prior to bending.

3. Center Notch Test

Figure 3 shows the test specimen configuration used in the accelerated center-notch test. This consists of a 1-3/4- by 8-in. tensile specimen containing a central notch. The notch is produced by a two-step process. First, a 0.06- by 0.57-in. slot is Elox machined and extended at each end by very narrow

Elox-machined notches of 0.001-in. root radii. Second, an extension of these notches is produced by tension-fatigue cycling to obtain fatigue cracks of controlled dimensions.

These center-notched specimens are tested in Baldwin creep-test machines. The desired loads are obtained by dead weight loading applied to a 20 to 1 lever arm. The test environment is applied by cementing a polyethylene cup to the specimen in the area of the notch. These specimens are well adapted to stress-corrosion studies in that crack growth rate, corrosion potential, or corrosion current can be conveniently measured.

4. Test Environments

The test environments in this program include those that the results of the previous year's program indicated were the cause of the most rapid failure of maraging steel; these are: continuous immersion in aerated distilled water; continuous immersion in aerated distilled water containing 3% by weight chemically pure sodium chloride; water-saturated air at 140°F; and natural seacoast atmospheric exposure. In addition, two new environments are being employed: distilled water at a thermostatically regulated temperature of 120 ± 0.1°F; and distilled water thermostatically regulated to 160 ± 0.1°F. All baths are changed every 10 to 14 days.

C. PROGRAM STATUS

1. Compositional Variation

In order to determine the effect of compositional variation on stress corrosion of 18%-nickel maraging steel, Aerojet is testing four commercial heats of the material in environments that were found, in the previous year's work, to produce the most rapid failures. The compositional variation of these four heats are shown in Table 1, Group b. The only element showing wide variation is titanium, which varies from 0.23 to 0.55%. Mechanical properties of the four heats are shown in Table 2, Group b.

Triplicate bent-beam and U-bend specimens of each of these four heats of material have been exposed to aerated distilled water (Table 3), aerated 3%-sodium chloride solution (Table 4), and 140°F water-saturated air (Table 5).

The most severe test condition has been found to be water-saturated air (high humidity) at 140°F. All specimens from each of the four heats failed; the individual lifetimes ranged from 3 to 27 days for U-bend specimens and 7 to 65 days for bent beam specimens. No well-defined relationship between failure time and compositional variation has been obtained, although the 0.23%-titanium alloy is generally the least susceptible. Failures have been found to be intergranular. Three of the four heats of material have shown failures with both U-bend and bent beam specimens in the aerated distilled water and aerated salt water environments. The fourth heat has had a relatively short exposure time to date.

Specimens of D6AC steel are being exposed to these same environments. The data so far indicates that, in each of the three environments, the D6AC steel is more susceptible than 18%-nickel maraging steel at the 237-ksi yield-strength level (600°F) but is far less susceptible at the 203-ksi yield-strength level (1100°F).

2. Environmental Temperature

The second objective is to study the effect of environmental temperature on the rate of stress-corrosion cracking in three alloys: 18%-nickel maraging steel, a low-alloy martensitic steel (D6AC), and a hot-worked die steel (Vascojet 1000). To accomplish this, we are repeating the ambient tests in distilled water at temperatures of 120°F and 160°F. To date, only the tests at 120°F have been started. These results, Table 6, show slightly more rapid failures at 120°F than at ambient temperature for both the 18%-nickel maraging steel and the low-alloy martensitic steel.

3. Electrochemical Measurements

The third objective of the program is to measure the electrochemical changes occurring in 18%-nickel maraging steel during stress-corrosion exposure and to determine the effect of applied potential on failure time. A preliminary test has been conducted on a center-notched specimen of heat 3960502. The test setup is shown in Figure 4. Using this test procedure the stress-potential relationship shown in Figure 5 was obtained. As the stresses at the crack tip, K, increase by dead weight loading of the specimen, the metal is found to become more chemically active. This study is continuing.

4. Surface Preparation

The fourth objective of the present program is to evaluate the effectiveness and applicability of surface protection in preventing stress-corrosion cracking on 18%-nickel maraging steel. Three coating systems, which have shown some degree of effectiveness in preventing stress-corrosion cracking of H-11 steel that has been heat treated to a susceptible level, are being evaluated on a single heat of 18%-nickel maraging steel. Each of the three coatings offers a different means of protection. The polyurethane coating forms a dense barrier between the environment and the metal. The inorganic zinc coating serves to provide cathodic protection to the metal, while the inhibited-epoxy coating protects the metal both by forming a barrier and by chromate compounds within the coating. As shown in Table 7, maraging steel coated with the zinc-bearing coating fails more rapidly than uncoated material, while the polyurethane coating offers some protection. The chromate-inhibited epoxy coating is the most effective of the three in preventing stress-corrosion cracking on either H-11 or 18%-nickel maraging steel.

D. FUTURE WORK

Work during the remainder of the contract period will involve: (1) evaluation and environmental testing of the Vascojet 1000 material for comparison with materials in testing; (2) testing of all materials in 160°F distilled water and in seacoast environments, (3) electrochemical experiments to determine the effect of applied potential on stress corrosion and (4) continued evaluation of results to establish correlations.

TABLE 1

MILL-CERTIFIED CHEMICAL ANALYSIS OF PROGRAM MATERIALS

Trade Name	Supplier	Heat No.	Composition, % C	Mn	P	S	Si	Ni	Co	Mo	Al	Cr	Zr	Ti	Ca	B	V
*(a) Maraging Steel from Previous Program																	
RSM 250	Republic Steel	3960502	0.02	0.08	0.007	0.006	0.15	18.48	7.00	4.84	0.21	0.10	0.035	0.50	-	0.0036	-
--**	Allegheny-Ludlum	448	0.029	0.002	0.004	0.008	0.009	18.51	8.48	4.92	0.089	-	-	0.52	-	-	-
Almar 18	Allegheny-Ludlum	W-24178	0.012	0.01	0.003	0.005	0.01	18.69	8.90	4.92	0.029	-	0.003	0.62	0.006	0.002	-
--**	Allegheny-Ludlum	476	0.02	0.08	0.006	0.005	0.014	18.60	9.05	4.90	0.078	-	-	1.00	-	-	-
Almar 20	Allegheny-Ludlum	W-24254	0.009	0.09	0.002	0.005	0.06	20.41	-	-	0.29	-	0.002	1.40	0.004	0.003	-
(b) Maraging Steel for Present Program																	
RSM 200	Republic Steel	3960523	0.029	0.06	0.005	0.006	0.05	17.79	8.50	3.48	0.13	-	-	0.23	-	-	-
Vascomax 250	Vanadium Alloys	07868	0.02	0.09	0.004	0.005	0.10	17.75	7.60	4.60	0.08	-	0.017	0.52	0.05	0.004	-
Marvac 18	Latrobe Steel	C56858	0.03	0.03	0.004	0.008	0.05	18.34	8.00	4.75	0.11	-	0.03	0.49	-	0.004	-
Vascomax 300	Vanadium Alloys	07268	0.03	0.05	0.004	0.006	0.04	18.54	9.06	4.88	0.09	-	0.088	0.55	0.02	0.003	-
(c) Conventional High-Strength Steels																	
Vascojet 1000	Vanadium Alloys	07914	0.38	0.21	0.010	0.008	0.92	-	-	1.33	-	4.75	-	-	-	-	0.51
Ladish D6AC	Allegheny-Ludlum	W-23217	0.495	0.62	0.009	0.003	0.20	0.57	-	0.94	-	1.00	-	-	-	-	0.05

*Some material from previous program will be used to obtain supplementary data.
**Experimental 400-lb heats.

TABLE 2

MECHANICAL PROPERTIES OF PROGRAM MATERIALS (AEROJET DATA)

Trade Name	Supplier	Heat No.	Heat Treatment Temp, °F	Heat Treatment Time, hours	0.2% Offset Yield Strength ksi	Ultimate Tensile Strength ksi	% Elongation	% Reduction in Area	Rc Hardness	Crack Growth Energy (Gc) in.-lb/sq in.
(a) Maraging Steel from Previous Program										
RSM 250	Republic Steel	3960502	900	3	249.9	254.7	4.0	37.0	50.5	670.0
**	Allegheny-Ludlum	448	900	3	255.4	265.9	5.0	9.0	52.0	634.0
Almar 18	Allegheny-Ludlum	W-24178	900	3	283.0	294.0	8.0	38.0	53.5	552.0
**	Allegheny-Ludlum	476	900	3	323.3	330.0	2.5	27.0	56.0	402.0
Almar 20	Allegheny-Ludlum	W-24254	850	4	291.3	302.2	3.0	17.0	54.0	58.3
(b) Maraging Steel for Present Program										
RSM 200	Republic Steel	3960523	900	3	181.5	190.7	5.0	43.0	42.0	638.0
Vascomax 250	Vanadium Alloys	07868	900	3	248.2	248.2	4.0	-	49.0	692.0
Marvac 18	Latrobe Steel	C56858	900	3	269.7	275.7	5.0	34.0	51.5	640.0
Vascomax 300	Vanadium Alloys	07268	900	3	279.1	288.1	4.0	18.0	52.0	560.0
(c) Conventional High-Strength Alloys										
Vascojet 1000	Vanadium Alloys	07914	(To be evaluated)							
Ladish D6AC	Allegheny-Ludlum	W-23217	600	temper****	237.4	281.3	6.0	25.0	51.5	182.0
			800	temper	214.5	241.2	7.0	38.0	45.5	323.0
			900	temper	204.6	226.4	7.5	43.5	44.0	385.0
			1100	temper	203.1	218.5	11.0	46.0	44.0	563.0

* Some material from previous program will be used to obtain supplementary data.

** Experimental 400-lb heats.

*** Received 50% cold reduced, lab-annealed for 1 hour at 1500°F.

**** Normalized, 1675°F, for 1 hour, A.C.; Austenitized, 1650°F for 15 min, O.Q.

TABLE 3

STRESS-CORROSION TEST RESULTS
AERATED DISTILLED WATER

Material	Heat No.	Yield Strength ksi	Failure* Ratio	Failure Time, hours** Mean	Failure Time, hours** Range
		Bent Beam Tests***			
20%-Ni Maraging Steel	W-24254	291.3	3/3	11	10.2-18
18%-Ni Maraging Steel	3960523	181.5	0/3	-	NF 1830
	07868	248.2	2/3	1030	980-NF 1080
	3960502	249.9	3/3	68	50-85
	C56858	267.7	0/3	-	NF 430
	07268	279.1	3/3	500	483-512
18%-Ni Maraging Steel	W-24178	283.0	3/3	34.5	20.5-27.5
D6AC Steel	W-23217	203.1	0/3	-	NF 750
		204.6	0/3	-	NF 750
		214.5	0/3	-	NF 750
D6AC Steel	W-23217	237.4	2/3	725	700-NF 750
		U-Bend Tests****			
20%-Nickel Maraging Steel	W-24254	291.3	2/2	3.5	1.4-5.5
18%-Ni Maraging Steel	3960523	181.5	1/3	1980	1900-NF 2070
	07868	248.2	2/3	880	480-NF 1540
	3960502	249.9	1/2	625	600-NF 650
	C56858	267.7	0/3	-	NF 24
18%-Ni Maraging Steel	07268	279.1	2/3	1530	407-NF 2350
D6AC Steel	W-23217	203.1	0/2	-	NF 940
		204.6	0/2	-	NF 940
		214.5	1/2	690	432-NF 940
D6AC Steel	W-23217	237.4	2/2	4.0	1.2-6.8

*Ratio of samples failed to samples tested.
**"NF" indicates no failure at the time given.
***Samples stressed to 75% of yield point.
****Samples stressed beyond yield point.

TABLE 4

STRESS-CORROSION TEST RESULTS
AERATED SALT WATER

Material	Heat No.	Yield Strength ksi	Failure* Ratio	Failure Time, hours** Mean	Failure Time, hours** Range
		Bent Beam Tests***			
20%-Ni Maraging Steel	W-24254	291.3	3/3	7.3	6-8.5
18%-Ni Maraging Steel	3960523	181.5	0/3	-	NF 1830
↓	07868	248.2	0/3	-	NF 1080
↓	3960502	249.9	3/3	430	140-700
↓	C56858	267.7	0.3	-	NF 430
↓	07268	279.1	3/3	1070	119-1970
18%-Ni Maraging Steel	W-24178	283.0	3/3	52	19-100
D6AC Steel	W-23217	203.1	0/2	-	NF 750
↓	↓	204.6	0/3	-	NF 750
↓	↓	214.5	0/3	-	NF 750
D6AC Steel	W-23217	237.4	1/3	580	240-NF 750
		U-Bend Tests****			
20%-Ni Maraging Steel	W-24254	291.3	2/2	2.4	1.9-2.9
18%-Ni Maraging Steel	3960523	181.5	0/3	-	NF 2070
↓	07868	248.2	1/3	1130	312-NF 1540
↓	3960502	249.9	0/0	-	-
↓	C56858	267.7	0/3	-	NF 24
18%-Ni Maraging Steel	07268	279.1	0/3	-	NF 2350
D6AC Steel	W-23217	203.1	0/2	-	NF 940
↓	↓	204.6	0/2	-	NF 940
↓	↓	214.5	0/2	-	NF 940
D6AC Steel	W-23217	237.4	2/2	1.0	0.8-1.2

*Ratio of samples failed to samples exposed.
**"NF" indicates no failure at the time given.
***Samples stressed to 75% of yield strength.
****Samples stressed to beyond yield strength.

TABLE 5

STRESS-CORROSION TEST RESULTS
140°F WATER-SATURATED AIR

Material	Heat No.	Yield Strength ksi	Failure* Ratio	Failure Time, hours** Mean	Failure Time, hours** Range
		Bent Beam Tests***			
20%-Ni Maraging Steel	W-24254	291.3	3/3	100	22-174
18%-Ni Maraging Steel	3960523	181.5	3/3	1500	1318-1560
↓	07868	248.2	3/3	600	480-722
↓	3960502	249.9	3/3	370	170-475
↓	056858	267.7	3/3	190	167-236
↓	07268	279.1	3/3	965	320-2040
18%-Ni Maraging Steel	W-24198	283.0	3/3	21	20.5-21.5
D6AC Steel	W-23217	203.1	0/2	-	NF 750
↓	↓	204.6	1/3	676	528-NF 750
↓	↓	214.5	3/3	333	309-381
D6AC Steel	W-23217	237.4	3/3	142	72-213
		U-Bend Tests****			
18%-Ni Maraging Steel	3960523	181.5	4/4	252	120-407
↓	07868	248.2	3/3	378	282-426
↓	3960502	249.9	2/2	167	167-167
↓	056858	267.7	3/3	70	70-70
18%-Ni Maraging Steel	07268	279.1	3/3	527	470-640
D6AC Steel	W-23217	203.1	0/2	-	NF 940
↓	↓	204.6	2/2	372	244-500
↓	↓	214.5	2/2	217	191-244
D6AC Steel	W-23217	237.4	2/2	1	1

*Ratio of samples failed to samples tested.
**"NF" indicates no failure at time given
***Samples stressed to 75% of yield strength.
****Samples stressed beyond yield strength.

TABLE 6

STRESS-CORROSION TEST RESULTS
120°F DISTILLED WATER

Material	Heat No.	Yield Strength ksi	Failure* Ratio	Failure Time, hours** Mean	Range
		Bent Beam Tests***			
18%-Ni Maraging Steel	396052	181.5	0/3	-	NF 770
↓	67868	248.2	3/3	532	500-596
↓	3960502	249.9	2/2	165	164-166
↓	C56858	267.7	3/3	348	324-396
18%-Ni Maraging Steel	07268	279.1	3/3	447	336-668
D6AC Steel	W-23217	203.1	0/3	-	NF 750
↓	↓	204.6	0/3	-	NF 750
↓	↓	214.5	3/3	695	644-740
D6AC Steel	W-23217	237.4	3/3	245	213-310
		U-Bend Tests****			
20%-Ni Maraging Steel	W-24254	291.3	3/3	347	180-668
18%-Ni Maraging Steel	396052	181.5	3/3	359	245-500
↓	07868	248.2	3/3	196	180-218
↓	3960502	249.9	2/2	133	121-144
↓	C56858	267.7	0/3	-	NF 24
18%-Ni Maraging Steel	07268	279.1	3/3	414	331-500
D6AC Steel	W-23217	203.1	0/2	-	NF 940
↓	↓	204.6	1/2	915	890-NF 940
↓	↓	214.5	2/2	537	335-740
D6AC Steel	W-23217	237.4	2/2	2.3	1.6-3.0

* Ratio of samples failed to samples exposed
** "NF" indicates no failures at time given.
*** Samples stressed to 75% of yield strength.
**** Samples stressed beyond yield strength.

TABLE 7

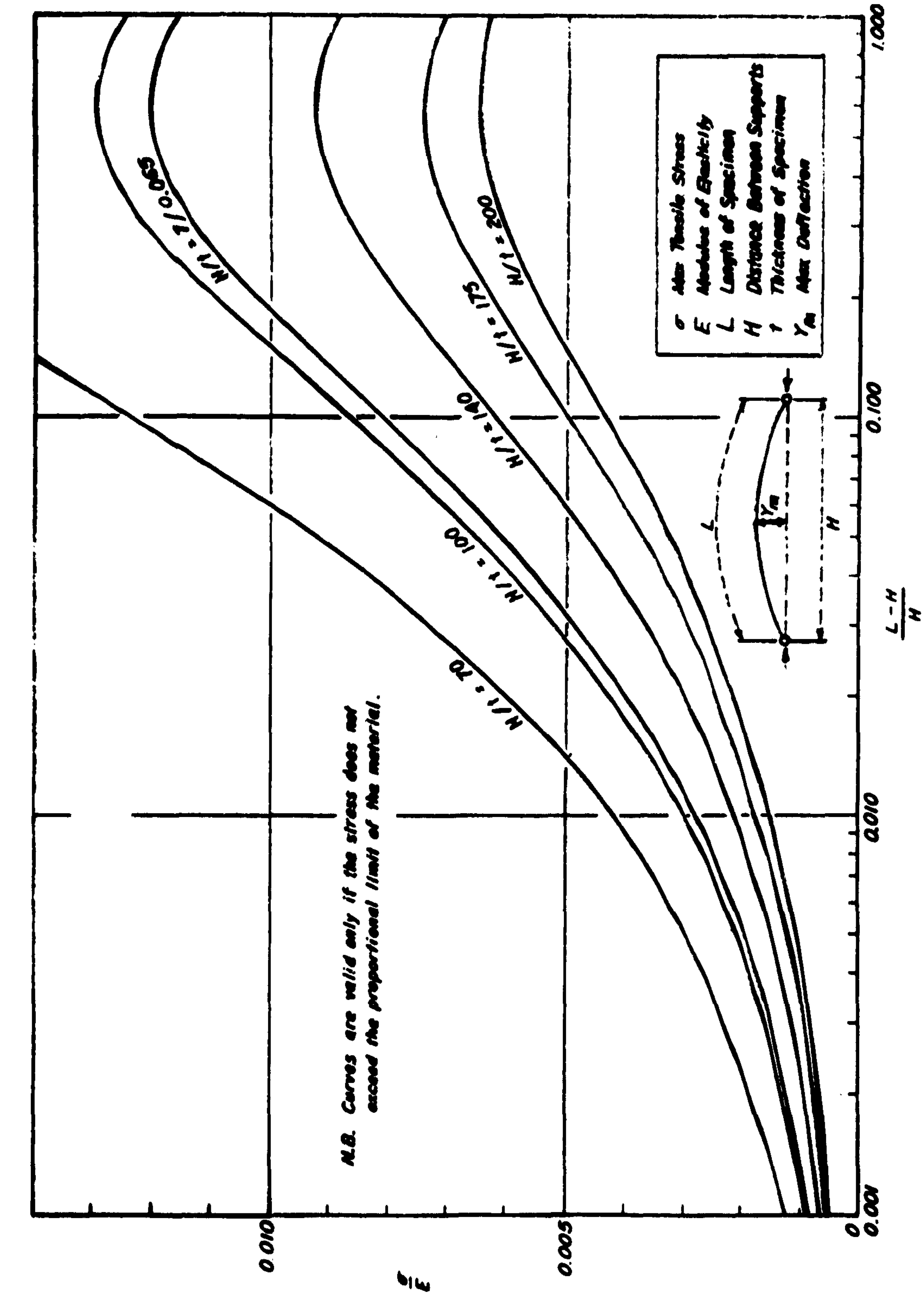
BENT-BEAM STRESS CORROSION TESTS
FOR COATING EVALUATION

Base Material	Coating	Aerated 3%-NaCl Solution: Failure Ratio*	Aerated 3%-NaCl Solution: Failure Time, hours, Mean	Aerated 3%-NaCl Solution: Failure Time, hours, Range	140°F Water-Saturated Air: Failure Ratio*	140°F Water-Saturated Air: Failure Time, hours, Mean	140°F Water-Saturated Air: Failure Time, hours, Range
H-11 Steel	None	4/4	1.6	0.8-2.5	2/2	64	48-70
	Polyurethane X-500	3/3	149	144-168	6/6	3500	2830-5500
	Inorganic Zinc 11	2/2	687	674-702	2/2	821	728-819
H-11 Steel	Inhibited Epoxy 454-1-1	0/2	-	NF 3100	3/3	2720	2590-2850
18%-Ni Maraging Steel	None	3/3	1068	119-1970	3/3	965	320-2040
	Polyurethane X-500	0/3	-	NF 1750	3/3	1513	1250-1728
	Inorganic Zinc 11	3/3	339	72-648	3/3	150	140-170
18%-Ni Maraging Steel	Inhibited Epoxy 454-1-1	0/3	-	NF 1750	1/3	1550+	1150-NF 1750

*Ratio of samples failed to samples exposed.

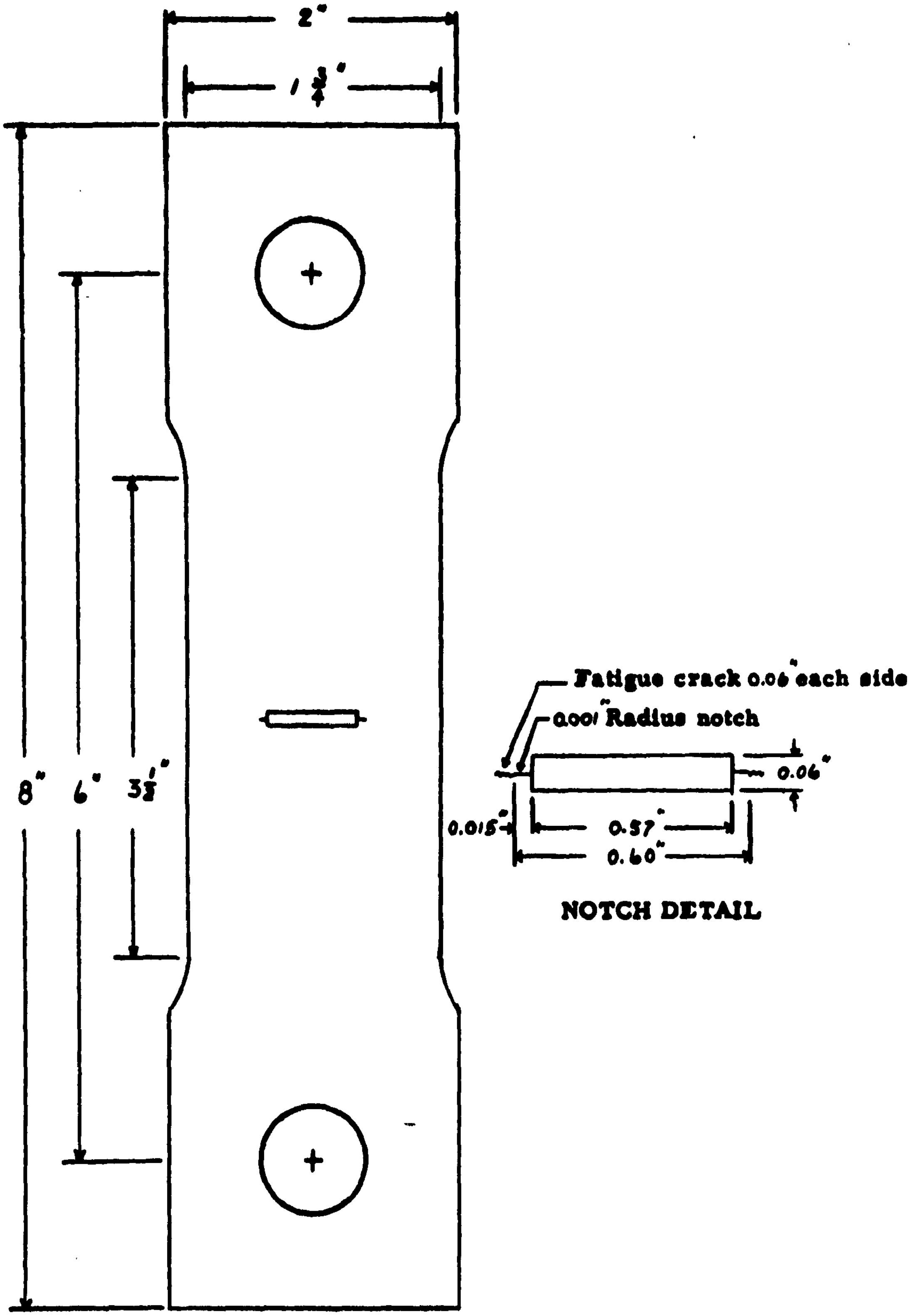
Bent-Beam Test Fixture and Specimens

Figure 1



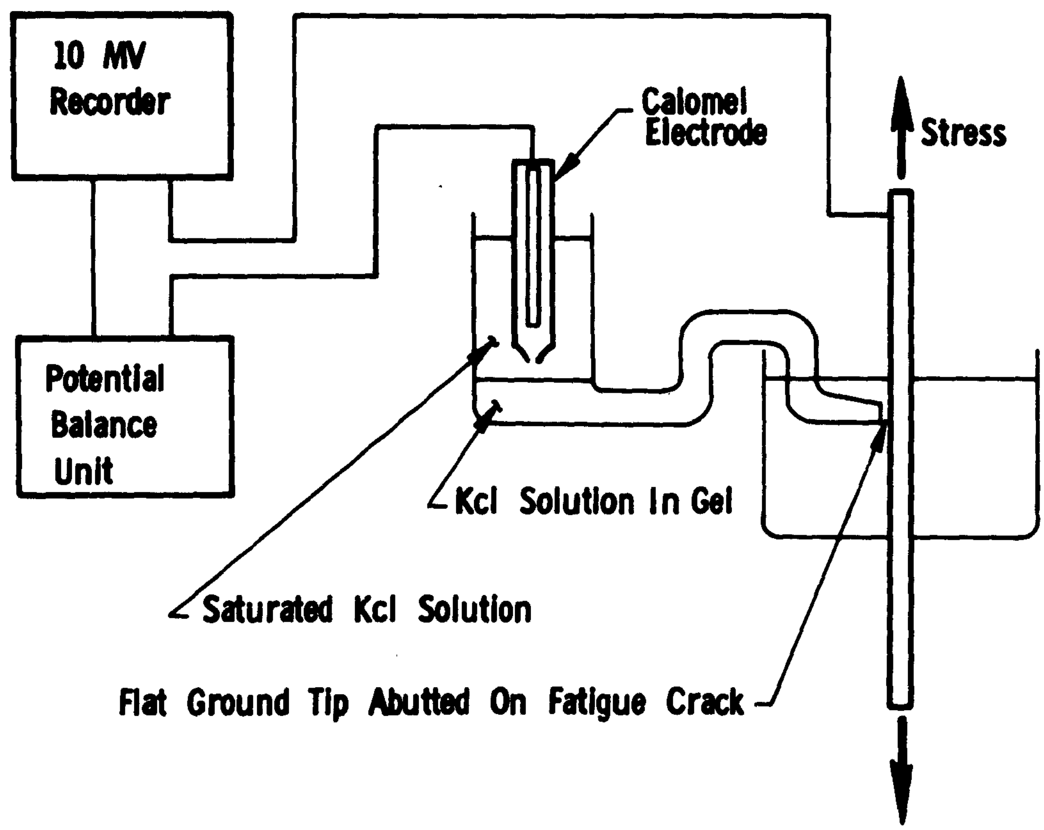
Beam Length-Stress Relationship

Figure 2



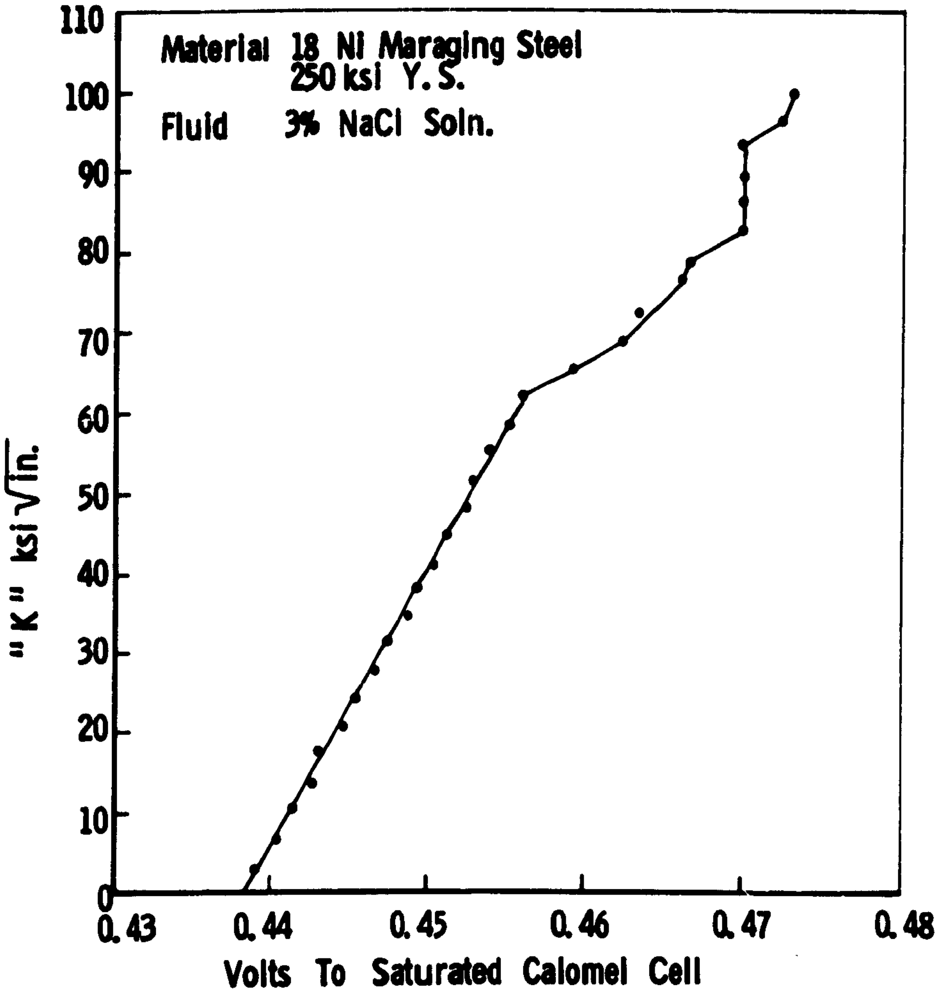
Center-Notch Specimen Configuration

Figure 3



STRESS-CORROSION POTENTIAL MEASURING APPARATUS

Figure 4



EFFECT OF STRESS FIELD PARAMETER, K, ON CRACK-TIP CORROSION POTENTIAL

Figure 5