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STRESS CORROSION CRACKING SUSCEPTIBILITY OF 18 Ni MARAGING STEEL

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16. ABSTRACT

The stress corrosion cracking (SCC) resistance of 18Ni maraging steel (grades 200, 250, 300, and 350) has been determined in 3.5 percent salt (NaCl) solution, synthetic sea water, high humidity, and outside MSFC atmosphere. All grades of the maraging steel were found to be susceptible to SCC in varying degrees according to their strengths, with the lowest strength steel (grade 200) being the least susceptible and the highest strength steel (grade 350), the most susceptible to SCC.

The SCC resistance of 250 grade maraging steel was also evaluated in salt and salt-chromate solutions using fracture mechanics techniques. The threshold value, K_{ISCC}, was found to be approximately 44 $MN/m^2\sqrt{m}$ (40 ksi$\sqrt{in.}$) or 40 percent of the K_Q value.

17. KEY WORDS:
Stress Corrosion Cracking (SCC)
18Ni Maraging Steel (Grades 200, 250, 300, and 350)
Alternate Immersion
High Humidity
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INTRODUCTION

The International Nickel Company in 1959 announced the development of a series of age hardenable high nickel martensitic steels, called maraging steels, which were of great interest to users of high strength alloys. Yield strengths ranging from 1379 MN/m^2 (200 ksi) to 2413 MN/m^2 (350 ksi) can be obtained by a simple aging process (3 hours at 750°K or 900°F) and the steel is classified according to yield strength (grade 200 to 350). Based on a combination of strength, ductility, fabricability, and fracture toughness characteristics, the 18 percent nickel maraging steels have been utilized in a variety of design applications which included large rocket casings, aerospace hardware, landing gear and structural components for aircrafts, and pressure vessels. The material is also found in the rail and oil industries, and it is being evaluated for gun tubes, machine gun barrels, and for use in submarines.

Although maraging steels are more resistant to rusting than most of the non-corrosion resistant steels, some type of protective coating is normally used for prolonged service life. These steels also are known to be susceptible to SCC, and the objective of this investigation was to determine the degree of susceptibility among these maraging steels.

EXPERIMENTAL PROCEDURE

Four types of specimens were used to test the stress corrosion cracking (SCC) resistance of the 18Ni maraging steels in at least two directions of grain orientation. Flat tensile specimens (Figure 1) were used for testing sheet material and were beam loaded by constant deflection. Round tensile specimens (Figure 2), stressed in uniaxial tension, were used for testing the longitudinal and long transverse grain directions of plate and the longitudinal grain direction of bar stock. The transverse grain direction of bar was evaluated using C-rings (Figure 2) loaded by constant deflection. Compact tension specimens (CPT) dead weight loaded in the transverse grain direction, were used in the fracture mechanics approach to SCC (Figure 3).

All heat treated round and flat tensile specimens were wet-grit blasted with fast-cut (quartz) #325 abrasive to remove surface oxides. These specimens were washed in cold running water and then given an alcohol rinse to facilitate drying. C-ring specimens were fabricated from aged bar and required no oxide removal.

Specimens were deflected or strained to the desired stress levels (40 to 90 percent of the directional yield strength) which were calculated from measured mechanical properties. The specimens were wiped with alcohol prior to exposure in the alternate immersion (AI) tester, high humidity, and outside MSFC atmosphere. Unstressed tensile specimens were exposed under identical conditions to determine the corrosiveness of the test media. The alternate immersion tests were conducted at room temperature in ferris wheel type testers (Figure 4) containing either a 3.5 percent solution (deionized water) of salt (NaCl) with an adjusted pH of 6.8 to 7.2 or synthetic sea water having a pH of 8.2. The exposure cycle was ten minutes in solution followed by fifty minutes of drying above the solution. The humidity cabinet was maintained at 98 percent relative humidity and a temperature of 310°K (95°F). The test period ranged from failure in one day to 180 days for the AI and humidity tests, and the atmospheric test is continuing after one and two years of exposure. The formulas for calculating deflection and strain and the methods of loading and testing are given in Reference 1.

Compact tension specimens were fatigue cracked and the K_Q value (conditional K_{IC} - see appendix) was established using the tentative method of test for Plane Strain Fracture Toughness of Metallic Materials (ASTM Designation: E-399-70-T). These SCC specimens were dead weight loaded using Model C creep testers (Figure 5), manufactured by Satec System Inc., Grove City, Pennsylvania. The specimens were loaded to stress intensities of 40 to 80 percent of the K_Q value and were tested completely immersed in the following solutions prepared with deionized water.

1. 20% NaCl having an initial pH of 6.4 to 6.8 (Reference 2).
2. 1% NaCl, 2% $K_2Cr_2O_7$ with pH adjusted to 4.0 (Reference 3).

3. 3.5% NaCl, 0.60% $Na_2Cr_2O_7 \cdot 2H_2O$, 0.95% $NaC_2H_3O_2 \cdot 3H_2O$, plus CH_3COOH to adjust pH to 4.0 (Reference 4).

4. 2% NaCl, 0.5% Na_2CrO_4 with an initial pH of 6.6 to 7.0.
At the end of 1000 hours, all specimens that had not failed were fractured mechanically and inspected for crack growth.

High humidity and alternate immersion in both 3.5 percent salt water and synthetic sea water were used as SCC test media for smooth specimens because there exists no universally accepted accelerated test medium for steels, and it was deemed advisable to compare the SCC performance in the most frequently used test media. Outside atmosphere was employed as a normal long term exposure. The four salt solutions were used as SCC test media for CPT specimens because no universally accepted test medium exists for precracked steel specimens, and the media used for smooth specimens were not suitable for the CPT specimens without changing the methods of loading. Where no specified test medium exists, comparison of SCC data from several exposure media is needed to assure confidence in the results.

RESULTS AND DISCUSSION

The chemical compositions of the four maraging steels are given in Table I and the mechanical properties and heat treatments are shown in Table II. It may be noted that the compositions of the four steels are similar with the greatest difference being the cobalt and titanium content of grade 350. All steels (grade 200-350) reached the desired mechanical properties as indicated by the yield strengths of Table II.

All the maraging steels were found to be susceptible to SCC to some degree with the susceptibility varying according to strength. The lowest strength steel (grade 200) was the least susceptible to SCC, and the highest strength (grade 350) was the most susceptible. A decrease in the SCC resistance with increase in strength is normal for most alloy systems.

As indicated in Table III, grades 200 and 250 did not fail in salt water at loads up to 90 percent of their respective yield strengths. Although both of these materials failed in high humidity and MSFC atmosphere, the 250 grade was more susceptible because its failure times were shorter than those of the 200 grade. Both the 300 and 350 grades of steel failed in all exposures including salt water. Grade 350 failed rapidly (<2 days) in salt water when stressed to 55 percent of yield, whereas the 300 grade did not fail after six months in salt water when stressed to 50 percent of yield but failed at 75 percent. As may be seen in Figures 6 and 7, all failures appeared to have resulted from SCC in that the attack was primarily intergranular with significant secondary branching. Figure 6 shows the intergranular and branching attack of a longitudinal and transverse specimen of 200 grade steel after exposure to high humidity and Figure 7 shows similar attack of a 350 grade specimen after exposure in 3.5 percent salt. Examination of a 250 grade failed surface using a Scanning Electron Microscope showed a mud crack pattern (Figure 8) which is indicative of SCC.

High humidity caused more failures than alternate immersion in either salt or sea water and therefore is considered a better accelerated medium for SCC testing 18 Ni maraging steels. This is not too surprising because other investigators have found that aerated distilled water and water saturated air causes SCC of maraging steels in relatively short periods (References 5 and 6). Failure of maraging and other martensitic steels in aqueous solutions reportedly does not depend on specific ions such as chloride (Reference 7). Benjamin and Steigerwald stated that the presence of chlorides (3N NaCl) in distilled water had little or no effect on incubation times for slow crack growth or failure times for D6AC, H11, and 18 Ni maraging steels (Reference 8).

There was no particular difference in the resistance of the maraging steels to general surface corrosion or rusting. None of the test environments caused any significant corrosion of the test specimens although alternate immersion in salt or sea water was somewhat more aggressive than the other two environments as indicated by visual examination and percent loss in tensile strength (Table III).

The SCC resistance of 250 maraging steel was evaluated using fracture mechanics techniques in addition to the smooth specimen method. The K_Q value in air of this steel was found to be 109 $MN/m^2\sqrt{m}$ (100 ksi$\sqrt{in.}$). Fatigue precracked compact tension specimens dead-weight loaded in the transverse grain direction yielded roughly similar failure rates and SCC threshold in all four salt and salt-chromate solutions (Table IV). Relatively short times to failure were encountered in all solutions at high stress intensities (80 percent of the K_Q value) and the failure time increased as the stress intensity was decreased until at 40 percent of K_Q no failure occurred in 1000 hours. This point at which failure does not occur is designated the threshold or K_{ISCC} which describes the stress intensity below which subcritical cracks do not extend to a critical size in a given environment.

CONCLUSIONS

The results of this investigation of the SCC resistance of 18Ni maraging steel (grades 200, 250, 300, and 350) revealed that:

1. All four grades of maraging steel were susceptible to SCC with the susceptibility varying according to strength. The lowest strength steel (grade 200) was the least susceptible to SCC and the highest strength (grade 350), the most susceptible.

2. High humidity (98 percent relative and 310°K, 95°F) is superior to alternate immersion in either 3.5 percent salt water or synthetic sea water as an accelerated medium for the SCC evaluation of maraging steel.

3. The threshold for SCC (K_{ISCC}) of grade 250 plate is approximately 44 $MN/m^2 \sqrt{m}$ (40 ksi$\sqrt{in}$) or 40 percent of the K_Q value, based on the results of dead weight loaded, fatigue cracked, compact tension specimens totally immersed for 1000 hours in four salt solutions.

Additional tests are planned to investigate the effects of temperature and the presence or absence of sodium chloride in the corrosive media on the SCC of maraging steel.

APPENDIX

1. Nomenclature - Fracture Mechanics

   K_{IC} - Plane strain fracture toughness as defined in ASTM E-399-70-T, 4.2.

   K_Q - Conditional value of K_{IC} as defined in ASTM E-399-70-T, 8.1.

   K_{Io} - Original stress intensity.

   K_{ISCC} - Stress intensity below which subcritical cracks do not extend to a critical size in a given environment.

2. Chemical Formulas

   NaCl - Sodium chloride

   Na_2CrO_4 - Sodium chromate

   $Na_2Cr_2O_7$ - Sodium dichromate

   $NaC_2H_3O_2$ - Sodium acetate

   $K_2Cr_2O_7$ - Potassium dichromate

   CH_3COOH - Acetic Acid

3. Conversion of customary U. S. Units to SI Units

Physical Quantity	Customary U.S. Units	Conversion Factors	SI Units
Length	in.	2.54	cm
Load	lb.	4.45	N
Stress	ksi	6.9	N/m^2
Temperature	F	5/9 (F+460)	K
Stress Intensity	ksi $\sqrt{in.}$	1.09	$MN/m^2 \sqrt{m}$

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TABLE I

CHEMICAL ANALYSIS OF THE MARAGING STEELS

	200	250	300	350
	Composition, Wt %			
Nickel	18.49	18.42	18.57	18.31
Cobalt	7.87	7.34	8.67	10.79
Molybdenum	4.19	4.57	4.75	4.50
Titanium	0.20	0.40	0.67	1.25
Carbon	0.013	0.020	0.017	0.022
Aluminum	0.07	0.08	0.08	0.07
Manganese	0.042	0.041	0.033	0.031
Silicon	<0.1	<0.1	<0.1	<0.1
Phosphorus	0.005	0.003	0.004	0.005
Sulfur	0.010	0.005	0.009	0.005
Calcium	0.008	0.010	0.009	0.008
Zirconium	<0.02	<0.02	<0.02	<0.02
Iron	Remainder	Remainder	Remainder	Remainder

TABLE II

HEAT TREATMENT AND MECHANICAL PROPERTIES OF THE MARAGING STEELS

Heat Treatment	Grain Direction	Mechanical Properties TS MN/m^2	(ksi)	YS MN/m^2	(ksi)	El %
200 Grade 1.59 cm (0.625 in.) Plate						
Received solution treated,	Longitudinal	1538	(223)	1489	(216)	11.0
Aged 3 hrs at 760°K (900°F)	Transverse	1579	(229)	1551	(225)	10.3
250 Grade 0.635 cm (0.250 in.) Plate						
Received solution treated,	Longitudinal	1737	(252)	1717	(249)	12.2
Aged 3 hrs at 760°K (900°F)	Transverse	1779	(258)	1765	(256)	10.0
250 Grade 1.59 cm (0.625 in.) Bar						
Heat treated by vendor	Longitudinal	1848	(268)	1806	(262)	10.0
300 Grade 0.25 cm (0.10 in.) Sheet						
Received solution treated,	Longitudinal	2062	(299)	2027	(294)	3.50
Aged 3 hrs at 760°K (900°F)	Transverse	2034	(295)	2013	(292)	3.75
350 Grade 1.59 cm (0.625 in.) Bar						
Heat treated by vendor	Longitudinal	2432	(353)	2365	(343)	4.0

TABLE III
STRESS CORROSION CRACKING RESULTS OF 18Ni MARAGING STEEL

Environment	Type Spec.	Stress Direction	Applied Stress MN/m² (ksi)	%YS	Failure Ratio	Days to Failure	% Loss TS(1)
			200 Grade 1.59 cm (0.625 in.) Plate				
3.5% NaCl	RT	Trans.	- -	0	0/3	-	N-10
			1165 (169)	75	0/3	-	18-21
			1400 (203)	90	0/3	-	N-24
		Long.	- -	0	0/3	-	N
			1117 (162)	75	0/3	-	N-17
			1338 (194)	90	0/3	-	N-13
High Humidity	RT	Trans.	696 (101)	45	1/3	149	N
			931 (135)	60	1/3	137	10-11
			1165 (169)	75	3/3	87, 98, 156	-
			1400 (203)	90	3/3	77, 87, 98	-
		Long.	1117 (162)	75	0/3	-	N
			1338 (194)	90	2/3	155, 164	N
MSFC Atmos.	RT	Trans.	1165 (169)	75	3/3	430, 440, 440	-
			1400 (203)	90	3/3	425, 425, 430	-
		Long.	1117 (162)	75	0/3(2)	-	-
			1338 (194)	90	2/3(2)	500, 530	-
			250 Grade 0.635 cm (0.250 in.) Plate				
3.5% NaCl	RT	Trans.	- -	0	0/3	-	N
			1324 (192)	75	0/3	-	7-8
			1593 (231)	90	0/2	-	N-10
		Long.	- -	0	0/3	-	N
			1289 (187)	75	0/3	-	N-23
			1544 (224)	90	0/3	-	N-9
High Humidity	RT	Trans.	883 (128)	50	2/2	67, 67	-
			1324 (192)	75	3/3	29, 29, 32	-
			1593 (231)	90	3/3	9, 16, 21	-
		Long.	1289 (187)	75	3/3	60, 90, 105	-
			1544 (224)	90	3/3	36, 38, 60	-
MSFC Atmos.	RT	Trans.	1324 (192)	75	2/2	270, 300	-
			1593 (231)	90	2/2	200, 250	-
		Long.	1289 (187)	75	0/2 (2)		-
			1544 (224)	90	0/2 (2)		-

TABLE III
STRESS CORROSION CRACKING RESULTS OF 18Ni MARAGING STEEL
(CONTINUED)

Environment	Type Spec.	Stress Direction	Applied Stress MN/m^2 (ksi)	%YS	Failure Ratio	Days to Failure	% Loss TS(1)
			250 Grade 1.59 cm (0.625 in.) Bar				
3.5% NaCl	CR	Trans.	758 (110)	40	0/3	-	-
			1240 (180)	70	0/3	-	-
High Humidity	CR	Trans.	758 (110)	40	3/3	64,105,105	-
			1240 (180)	70	3/3	46,47,47	-
MSFC Atmos.	CR	Trans.	758 (110)	40	3/3	140, 850, 850	-
			1240 (180)	70	3/3	260, 365, 480	-
			300 Grade 0.25 cm (0.10 in.) Sheet				
3.5% NaCl	FT	Trans.		0	0/6	-	N
			1007 (146)	50	0/6	-	N
			1510 (219)	75	3/6	102-135	N
		Long.	- -	0	0/6	-	N
			1014 (147)	50	0/5	-	N
			1524 (221)	75	5/5	64-151	-
Synthetic Sea Water	FT	Trans.	- -	0	0/3	-	N
			1007 (146)	50	0/3	-	N
			1510 (219)	75	1/3	11	N
		Long.	- -	0	0/3	-	N
			1014 (147)	50	0/3	-	N
			1524 (221)	75	2/3	166, 172	N
High Humidity	FT	Trans.	1510 (219)	75	2/2	18,20	-
		Long.	1524 (221)	75	2/2	7,11	-
			350 Grade 1.59 cm (0.625 in.) Bar				
3.5% NaCl	RT	Long.	1282 (186)	55	3/3	1	-
MSFC Atmos.	RT	Long.	1282 (186)	55	3/3	27-31	-
High Humidity	RT	Long.	1282 (186)	55	3/3	1-2	-

RT-Round tensile, CR-C ring, FT-flat tensile, N-negligible (<5)

Notes: (1) All specimens from which % loss in TS was calculated were exposed for 180 days. Loss in TS is not available for specimens exposed in atmosphere because these test are continuing.

(2) Test continuing after one year of exposure for grade 250 specimens and two years for grade 200.

TABLE IV
STRESS CORROSION CRACKING RESULTS OF 250 GRADE MARAGING STEEL USING FRACTURE MECHANICS(1)

Load N (lb)	K_{Io}/K_Q	Crack Length Initial cm (in.)	Crack Length Final cm (in.)	Time To Failure (hr)
		20% NaCl Solution		
7503 (1686)	0.80	1.42 (0.560)	1.57 (0.620)	7
5945 (1336)	0.70	1.49 (0.585)	1.80 (0.710)	22
5531 (1243)	0.60	1.44 (0.565)	1.84 (0.725)	92
4495 (1010)	0.50	1.45 (0.570)	1.87 (0.735)	709
3689 (829)	0.40	1.44 (0.565)	1.44 (0.565)	1000(2)
		1% NaCl, 2% $K_2Cr_2O_7$ Solution		
7076 (1590)	0.80	1.46 (0.575)	1.64 (0.645)	12
6453 (1450)	0.70	1.44 (0.565)	1.84 (0.725)	24
5830 (1310)	0.60	1.40 (0.550)	1.83 (0.720)	48
4730 (1063)	0.50	1.41 (0.555)	1.97 (0.775)	54
4690 (1054)	0.45	1.28 (0.505)	1.38 (0.545)	1000(3)
3596 (808)	0.40	1.45 (0.570)	1.56 (0.610)	1000(3)
		3.5% NaCl, 0.524% $Na_2Cr_2O_7$, 0.574% $NaC_2H_3O_2$, CH_3 COOH Solution		
7378 (1658)	0.80	1.44 (0.565)	Not Measured	7
6190 (1391)	0.70	1.46 (0.575)	1.70 (0.670)	22
5830 (1310)	0.60	1.40 (0.550)	1.82 (0.715)	32
4423 (994)	0.50	1.46 (0.575)	2.00 (0.790)	65
4259 (957)	0.45	1.41 (0.555)	1.96 (0.770)	96
3596 (808)	0.40	1.45 (0.570)	1.86 (0.735)	1000(3)
		2% NaCl, 0.5% Na_2CrO_4 Solution		
4762 (1070)	0.60	1.52 (0.600)	1.94 (0.765)	25
4610 (1036)	0.50	1.44 (0.565)	1.91 (0.750)	34
3751 (843)	0.40	1.42 (0.560)	1.45 (0.570)	1000(3)

K_{Io} - original crack intensity, K_Q - conditional K_{IC}

Notes: 1. Test Method

a. Procedure: Compact tension specimens dead weight loaded were completely immersed in the test solutions until failure or 1000 hours.

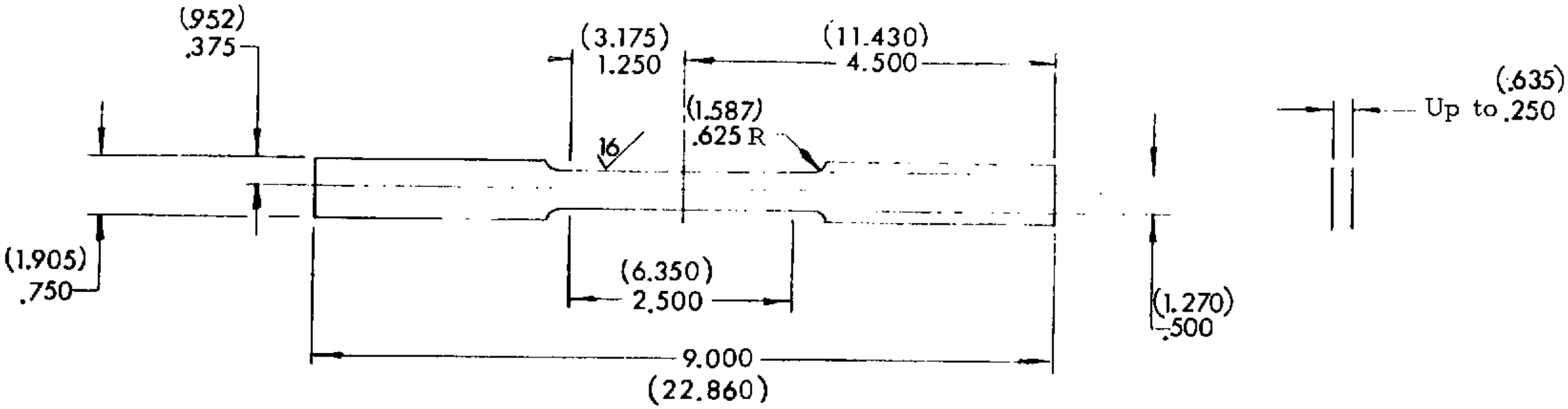
TABLE IV
STRESS CORROSION CRACKING RESULTS OF 250 GRADE MARAGING STEEL USING FRACTURE MECHANICS[1]
(CONTINUED)

b. Fatigue precrack conditions: Stress intensity amplitude ratio, K min/K max. = 0.1. Maximum stress intensity applied during precracking ranged from 25.6 to 33.0 $MN/m^2\sqrt{m}$ (23.5 to 30.3 ksi$\sqrt{in.}$

c. The measured K_Q value was 109 $MN/m^2\sqrt{m}$ (100 ksi $\sqrt{in.}$)

2. Specimen did not fail and suffered no crack extension.

3. Specimen did not fail but suffered some crack extension.



Dimensions In Inches
Dimensions in Centimeters ()

NOTE: Overall dimension will vary with the strength and the desired stress level

FIGURE 1 - FLAT TENSILE SPECIMEN CONFIGURATION

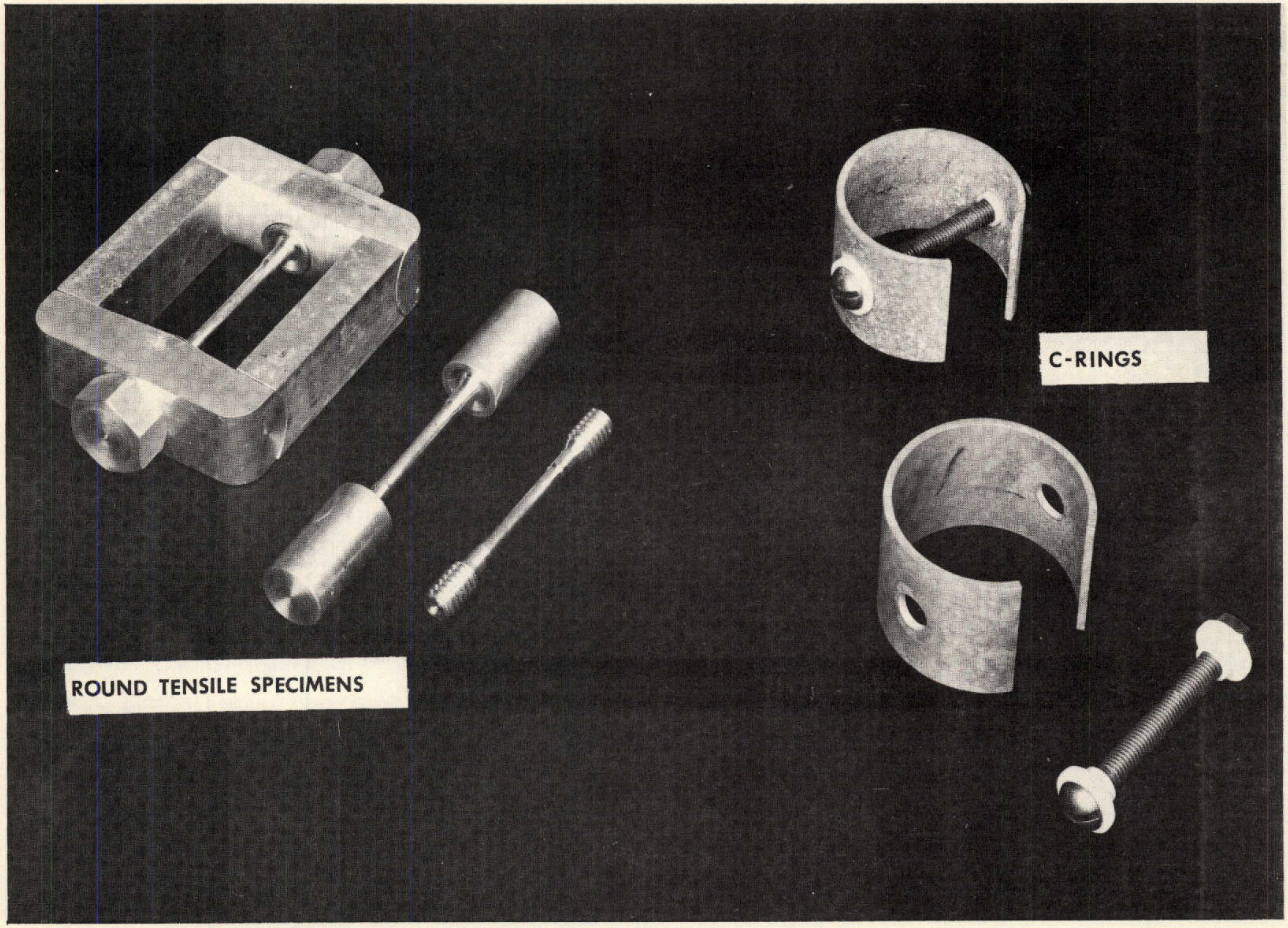
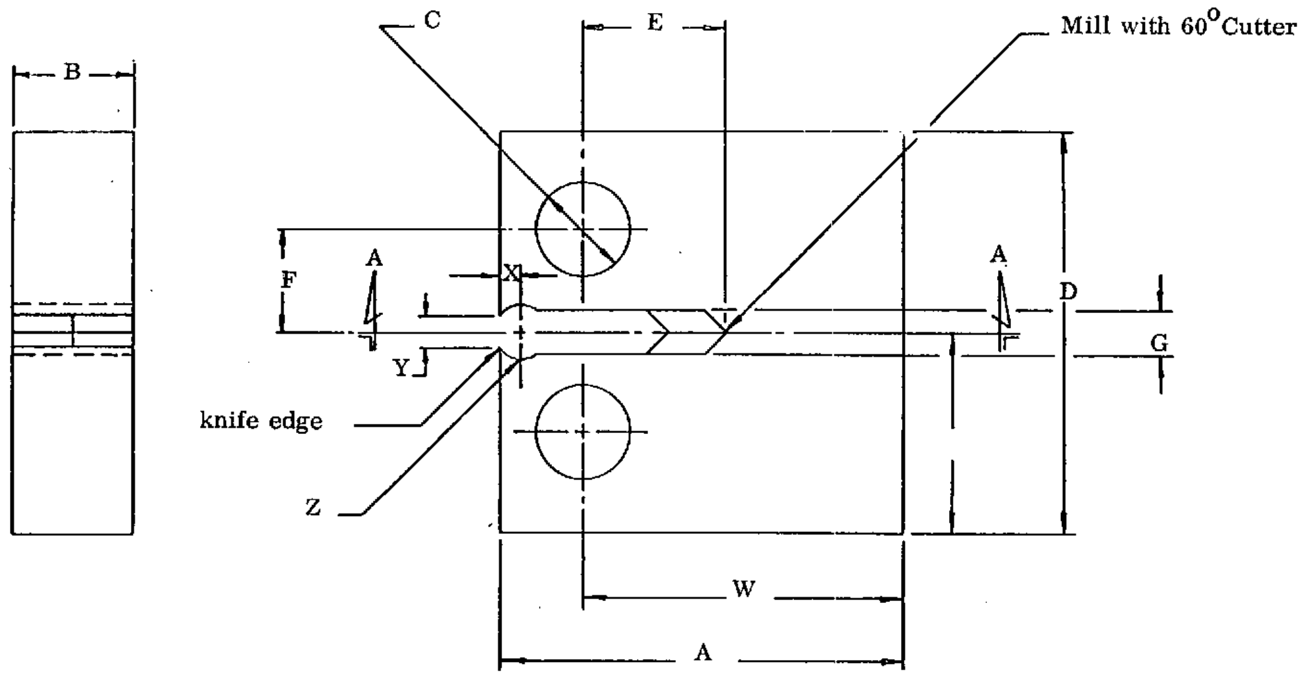
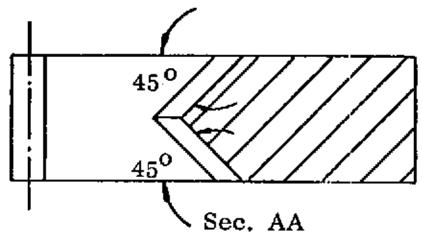


FIGURE 2 - STRESS CORROSION SPECIMENS





X - 0.3/cm (.122 in.)
Y - 0.5/cm (.20 in.)
Z - 0.8 cm (.312 in.) dia.

	A		W		H		D		E		F		G		B		C	
	cm	in.	cm	in.	cm	in.	cm	in.	cm	in.	cm	in.	cm	in.	cm	in.	cm	in.
	3.175	1.250	2.540	1.000	1.524	0.600	3.048	1.200	1.27	0.500	0.699	0.275	0.127	0.050	0.635	0.250	0.635	0.250

FIGURE 3 - COMPACT TENSION SPECIMEN CONFIGURATION

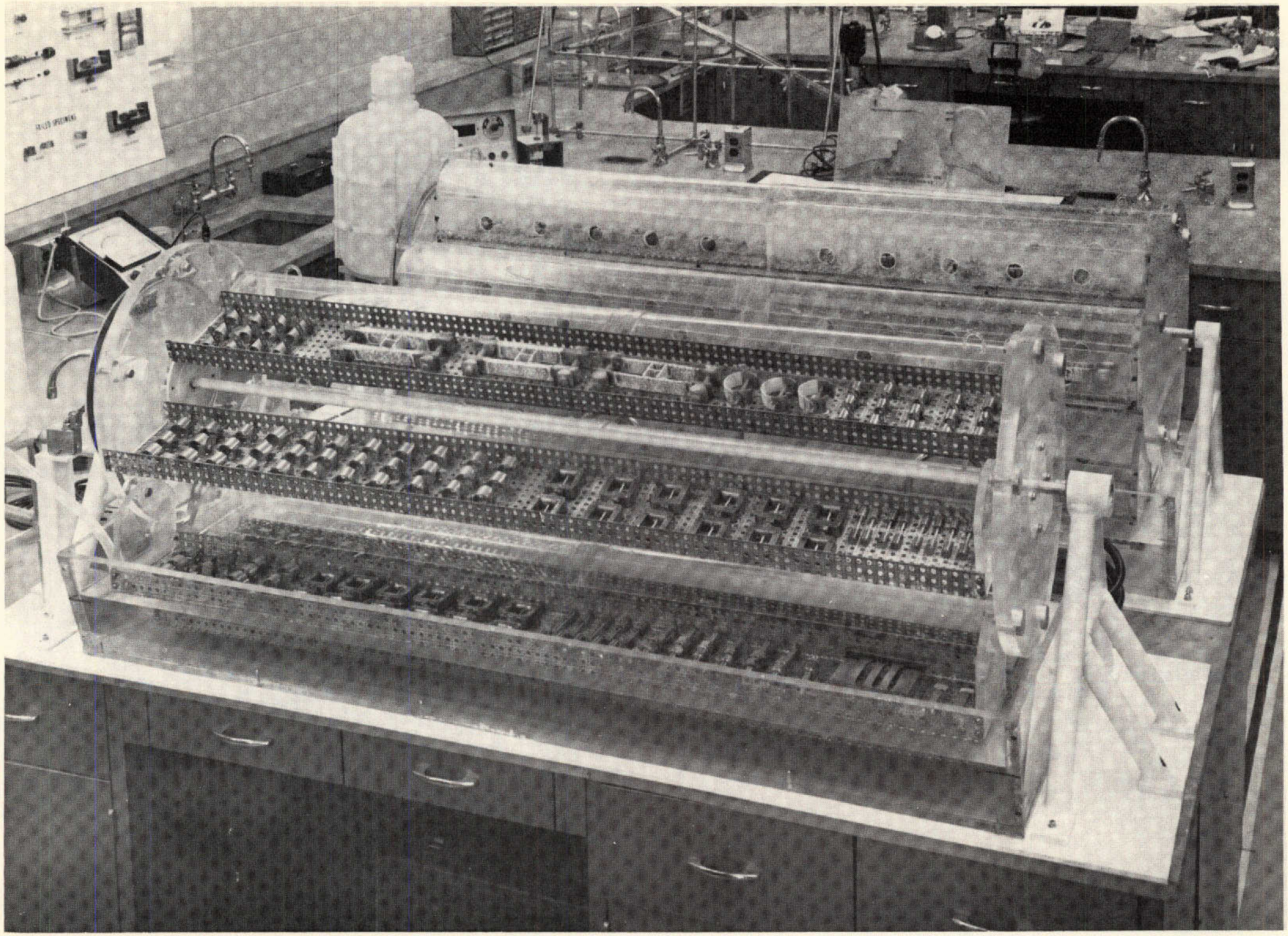


FIGURE 4 - ALTERNATE IMMERSION TESTER

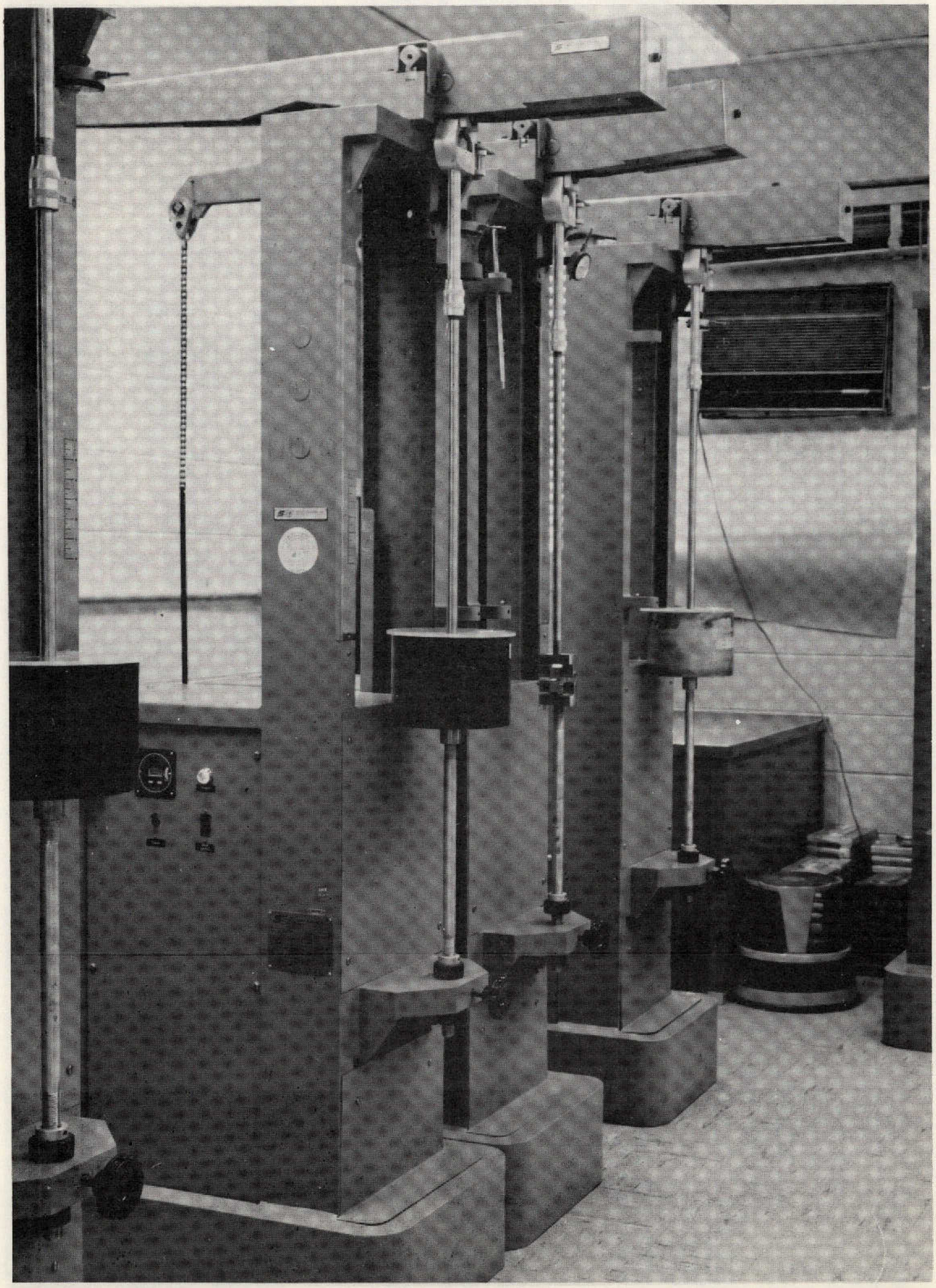
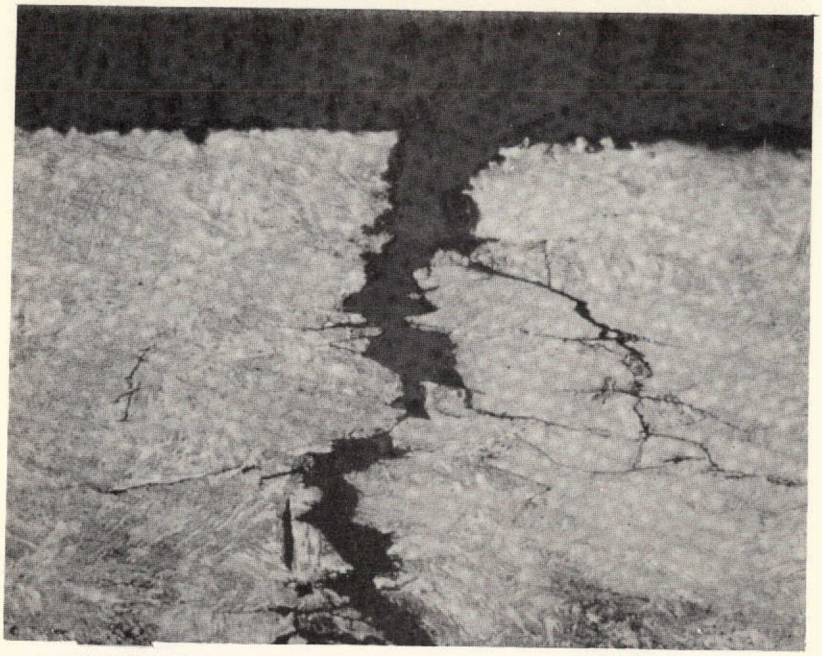
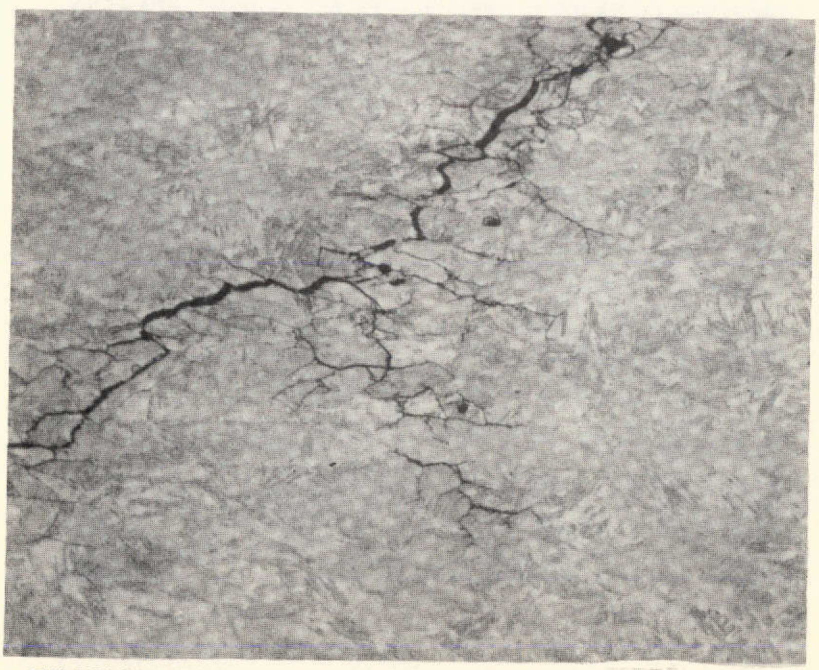


FIGURE 5 - METHOD OF LOADING AND TESTING CPT SPECIMENS

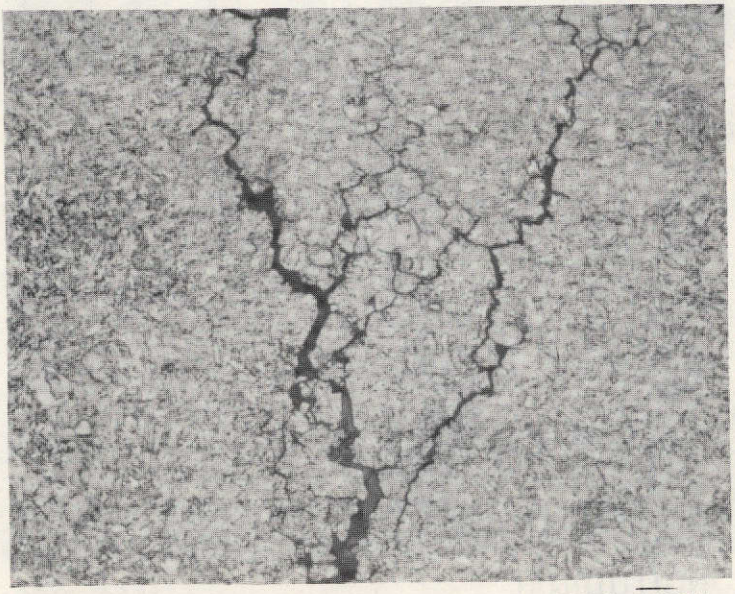


LONGITUDINAL MAG. 200X



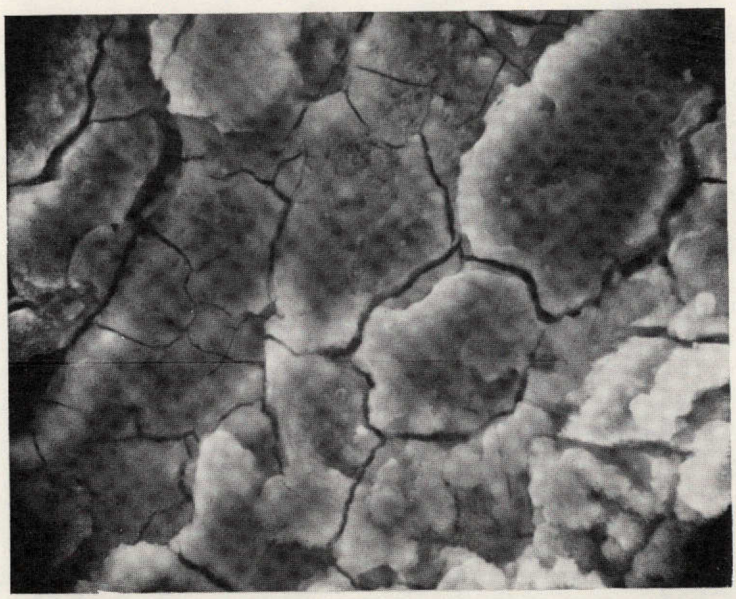
TRANSVERSE MAG. 200X

FIGURE 6 - SCC OF 200 MARAGING STEEL EXPOSED TO HIGH HUMIDITY



LONGITUDINAL MAG. 200X

FIGURE 7 - SCC OF 350 MARAGING STEEL EXPOSED TO SALT WATER



TRANSVERSE SEM 2,000X

FIGURE 8 - SCC OF 250 MARAGING STEEL EXPOSED TO SALT WATER

APPROVAL

STRESS CORROSION CRACKING SUSCEPTIBILITY
OF 18Ni MARAGING STEEL

By

T. S. Humphries
Eli E. Nelson

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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