

DEPARTMENT OF ENERGY, MINES AND RESOURCES MINES BRANCH OTTAWA

STRESS-CORROSION CRACKING TESTS ON SOME HIGH-STRENGTH STEELS, USING THE USNRL CANTILEVER METHOD

G. J. BIEFER AND J. G. GARRISON

PHYSICAL METALLURGY DIVISION

MAY 1969

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The Queen's Printer Ottawa, Canada 1969 Mines Branch Technical Bulletin TB 114 STRESS-CORROSION CRACKING TESTS ON SOME HIGH-STRENGTH STEELS, USING THE USNRL CANTILEVER METHOD

by

G.J. Biefer* and J.G. Garrison**

ABSTRACT

A stress-corrosion cracking test developed at the U.S. Naval Research Laboratory, Washington, D.C., has been used at the Physical Metallurgy Division to test the cracking susceptibility of a number of high-strength steels.

Results are reported which were obtained in stresscorrosion tests on an 18% Ni maraging steel (both parent and weld metal), an HP-9-4-25 steel, a copper-nickel low-alloy steel developed at the Physical Metallurgy Division, and a 17/4 PH stainless steel in each of the H900 and H1000 conditions. For each of these steels, the tendency to fracture under dry conditions was compared with that resulting from immersion in 3.5% NaCl solution in the unpolarized state. The effects of cathodic protection by 5083 aluminum alloy and zinc were also investigated.

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Direction des mines

Bulletin technique TB 114

ESSAIS DE FISSURATION PAR CORROSION SOUS TENSION PRATIQUÉS SUR CERTAINS ACIERS À HAUTE RÉSISTANCE, PAR LA MÉTHODE CANTILEVER DE L'USNRL

par

G.J. Biefer* et J.G. Garrison**

RÉSUMÉ

Un essai de fissuration par corrosion sous tension mis au point par le U.S. Naval Research Laboratory, à Washington, D.C., a été employé par la Division de la métallurgie physique pour déterminer la susceptibilité à la fissuration d'un certain nombre d'aciers à haute résistance.

Le présent bulletin décrit les résultats d'essais de fissuration par corrosion sous tension exécutés sur un acier "maraging" à 18% de Ni (métal de base et métal de fusion), sur un acier HP-9-4-25, sur un acier faiblement allié au cuivre et au nickel mis au point par la Division de la métallurgie physique, et sur un acier inoxydable 17/4 PH dans les conditions H900 et H1000. Dans chacun de ces aciers, la tendance à la fissuration à sec a été comparée avec celle qui résulte de l'immersion du métal dans une solution de NaCl à 3.5 p. 100 à l'état non polarisé. Les auteurs ont également étudié les effets de la protection cathodique au moyen de zinc et d'un alliage d'aluminium 5083.

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INTRODUCTION

Stress-corrosion cracking (SCC)* measurements were carried out previously in the Physical Metallurgy Division, using an apparatus in which a small, thin strip of highstrength steel was held in a bent configuration under spring loading. Differences in cracking susceptibility were demonstrated for different steel types, and effects of cathodic protection potential were observed, particularly in the presence of pre-existing cracks⁽¹⁾. However, the results obtained using these methods were of only qualitative significance, because stresses were not known quantitatively. In addition, the chemically produced cracks were not uniform (in number or depth) from one specimen to the next.

Furthermore, B.F. Brown of the U.S. Naval Research Laboratory, Washington, D.C., has pointed out that SCC may be very difficult to propagate in a thin specimen as compared with a thick specimen, for reasons which are purely geometrical $^{(2)}$. Because of this, considerable caution must be exercised in attempting to relate laboratory results, obtained with thin specimens, to service applications in which relatively thick plate is used. In addition, Brown is critical of "time-tofailure" criteria of resistance to SCC. He has stated that, in many cases, what is really being measured is the rate of pitting corrosion, because SCC failures occur with extreme rapidity once a pit has become large enough to initiate a crack. To eliminate what are sometimes lengthy induction periods in SCC tests, during which pits are growing, Brown has developed a simple test in which a notched bar, pre-cracked at the notch root by fatiguing, is loaded as a cantilever beam. The pre-cracked area of the bar is surrounded by a plastic vessel containing the test solution usually 3.5% sodium chloride.

*In this report, SCC is used as a general term and includes hydrogen embrittlement cracking (HEC).

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Brown states that, for a particular alloy in a selected heat-treatment condition, there will be a characteristic stressintensity value $K_{\rm ISCC}$ (kpsi/in.) above which it can be stated with confidence that cracking will occur. He has used some of the equations of linear elastic fracture mechanics to calculate values of $K_{\rm ISCC}$ for specimens exposed to a 3.5% salt solution at room temperature (3, 4). Brown states that it is not known for certain that stress-corrosion cracking will never occur at stress intensities below $K_{\rm ISCC}$.

It was decided to attempt to use Brown's SCC test at the Physical Metallurgy Division, in work aimed at assessing various high-strength steels with respect to their suitability for use in hydrofoil seacraft such as the Canadian FHE 400. The present report describes the methods used and the results obtained in the initial measurements.

EXPERIMENTAL

Materials

The steels used in this work were obtained in the form of plate, 0.5 in. to 1 in. thick. Some information concerning the steels appears in Tables 1 and 2. Notched specimens were cut according to the drawing shown in Figure 1, the length of the bar lying in the rolling direction of the plate. It was specified that the faces shown to be 6.375 in. x 0.375 in. in Figure 1 were to be parallel to the original plate surfaces, i.e., the dimension shown to be 0.750 in. corresponded to the original plate thickness. In one case (17/4 PH stainless steel), the specimens were cut sideways so that the 6.375 in. x 0.750 in. faces lay parallel to the original plate surfaces. Heat treatments were carried out on the as-machined specimens. Table 1 lists the various heat treatments used.

TABLE 1

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General Description of the Steels

Steel	Source and As-Received Condition	Additional Heat Treatments Given	Nominal Yield Strength After Heat Treatment, kpsi
18% N ickel Maraging	Vanadium-Alloys Steel Co. Vascomax 250 Consumable-arc vacuum-melted 3/4-in. plate, in the solution- annealed condition.	3 hr at 482°C (900°F).	250
18% Nickel Maraging	As above, but 1/2-in. plate from Heat 09081.	After MIG welding, 3 hr at 482°C (900°F)	250
HP 9-4-25	Vacuum-melt from Republic Steel Co., obtained as 3/4-in. plate, quenched and tempered at 538°C (1000°F).	None.	190
P.M.D. Cu-Ni Alloy	Commercially produced 10-ton heat, forged to 1-in. plate, water quenched, then tempered at 205°C (400°F). See reference 7.	None.	160
Armco 17/4 PH Stainless	Heat 64190-2C, G. O. Carlson, Inc., obtained as 1/2-in. plate in the solution- treated "A" condition.	l hr at 482°C (900°F) to produce the "H900" condition.	180
	· .	4 hr at 538°C (1000°F) to produce the "H1000" condition.	150

τ 1

Steel	с	Mn	Si	S .	P	Cu	Co	Cr	Мо	Ni	Other Elements
Maraging Steel (a) (3 4-in. plate)	0.03 max	0.10 max	0.10 max	0.010 max	0.010 max	-	7.50	-	4.80	18.50	Al 0.10; Ti 0.40; B 0.003; Zr 0.02 added; Ca 0.05 added.
Maraging Steel (b) $(\frac{1}{2} - in. plate)$	0.02	0.06	0.04	0,009	0.003	-	7.63	-	4.78	18.48	Al 0.05; Ti 0.36; B 0.004; Zr 0.012; Ca 0.05.
HP-9-4-25 (c)	0.27	0.21	0.02	0.014	0.005	-	3,86	0.39	0.51	9,48	V 0.08.
PMD Cu-Ni Alloy (d)	0.16	0.78	0.28	0.007	0.004	1.11	-	-	-	3.78	
17/4 PH Stainless (e)	0.039	0.20	0.58	0,008	0.017	3.49	-	15.91	-	4,28	СЪ 0.20; Та 0.02.

TABLE 2

Chemical Analyses of the Steels

(a) Nominal Analysis, Vanadium-Alloys Steel Co.

(b) Heat Analysis supplied by Vanadium-Alloys Steel Co.

(c) Analysis at the Department of Energy, Mines and Resources.
(d) Supplied by R. K. Buhr⁽⁷⁾
(e) Supplied by G. O. Carlson, Inc.





Figure 1. Shop drawing of cantilever stress-corrosion test bar. Note that the height (0.750 in. in the drawing) was varied in the range about 0.5 to 1.0 in.

Methods

After the specimens had been heat-treated, they were pre-cracked in the notch by fatiguing, using a Krouse Reverse-Bend Plate Fatigue Testing Machine operating at a rate of 1725 cycles/min. A special extension arm was used so that nominal stresses at the notch, "s", could be obtained from the equation:

In this equation, W is the load at one end of the specimen, A is the distance from the notch to the loaded end, and B_{N} and D_{N} are, respectively, the width and the height of the test bar at the notch.

Cracks were generally produced in approximately 30 sec (~900 cycles) of fatiguing at about half the yield strength. (During the fatiguing the apparatus was stopped every 5 sec (~150 cycles) at the point of maximum tensile stress in the notch, and the specimen was examined in the notch for signs of crack formation with an "Otoscope" diagnostic light equipped with a low-power magnifying glass.)

The cantilever SCC apparatus is shown in Figure 2. It was constructed according to shop drawings supplied by B.F. Brown of the U.S. Naval Research Laboratory, Washington, D.C. A few minor modifications were made subsequently.

The metal specimen is mounted between the two sets of grips shown at the left centre of Figure 2. It may be enclosed by a polyethylene bottle with its top cut off and holes of appropriate size cut through its sides, as shown in Figure 2. When this was done, the joints were sealed externally with G.E. Silicone Construction Sealant. An enlarged view of this region (Figure 3) shows the notched specimen immersed in the usual test solution of 3.5% NaCl. A probe is shown which, leading through a salt bridge to a saturated calomel electrode, permitted measurements of specimen potential during the tests, by means of a vacuum tube voltmeter. Figure 3 also shows sacrificial anodes, either of 99.999% Zn or of 5083 aluminum alloy, which were used to provide cathodic protection (c.p.) during some of the tests. As can be seen from Figure 3, electrical connections to the test bar were external. In a single test in which a specimen of steel was polarized cathodically at a d-c current density of -1 mA/cm^2 , a platinum anode was used.

In tests on unpolarized specimens, the 3.5% NaCl solution was not replenished. However, in the tests in which cathodic protection was used, the solution was replenished at the rate of 4 litres/day.

As is shown in Figure 2, weights can be positioned on a holder hanging from the right-hand end of the cantilever arm. An auto jack, fitted with a special platform, was used to remove the tension from the test bars when weights were being added, and also served as a landing pad for the weights when specimens fractured. The latter action deactivated a timer which was operated through a microswitch recessed in the landing pad.

A height gauge, shown at the right above the cantilever arm, was also used, in some cases, to follow the movement of the arm subsequent to the addition of weights. Because the gauge itself increases the tension on the notch, it was found necessary to either refrain from using it, or make a correction, for specimens breaking at relatively low loads.

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Figure 2. View of the cantilever stress-corrosion cracking test apparatus, showing auxiliary equipment.



Figure 3. The notched specimen, immersed in salt solution, is shown under cathodic protection by a sacrificial anode. The glass probe used in measuring the specimen potential can also be seen. To perform a test on a dry specimen, an initial weight of from 10 to 20 lb was usually added. The total weight was then increased, usually at the rate of 1 lb/min, until fracture occurred. For specimens of relatively low height (height = 0.750 in. in Figure 1) or specimens known to be highly susceptible to cracking, fractures were known to occur at lower loads and the additions were made in $\frac{1}{4}$ -lb or $\frac{1}{2}$ -lb increments. Such measurements yielded what B.F. Brown terms KI_i values, in the units kpsi $\sqrt{in.}$, from the equation

 K_{I_1} is the nominal stress intensity parameter, based on the initial crack geometry, and β is a function of a/D (where a is the pre-crack depth, including the notch, and D is the specimen height). M is the bending moment on the notch, i.e., the arm length x load at the end of the arm. B_N is the specimen width at the notch, B the width at other than the notch, and $\left(\frac{B}{B_N}\right)^{\frac{1}{2}}$ the Freed-Krafft correction for the side-notching⁽⁸⁾.

Calculation of the β values was made by means of Brown's⁽³⁾ "graphical shortcut method" (see Figure A-1, Appendix, page 31), the total pre-crack depth being measured (after the fracture) under a low-power stereomicroscope with a calibrated scale. Since most of the cracks had an inverted thumbnail profile, the crack depth showed variation across the specimen. For simplicity, the crack depth at the centre of the specimen was used in the calculation. It represented a minimum depth in most cases.

For the specimens fractured in 3.5% NaCl, a different loading schedule was adopted. Each loading level was maintained for a period of at least one hour (unless a fracture occurred) before going on to the next higher load level. If a specimen had not fractured at the end of a working day, the load was left on overnight. If a break had not occurred, loading was continued in the same way the next morning. For fractured specimens, K_{I_i} values were calculated using the methods employed with the dry specimens.

RESULTS

The results obtained in the measurements, in terms of the K_{Ii} values at fracture, appear in Table 3. Additional information regarding the measurements is given in Figures 4-9, which indicate the number of loading levels and total duration of each of the tests. The figures also show, for each specimen, the K_{Ii} values of the lowest loads, the K_{Ii} values for loads sustained for two hours or more, the K_{Ii} values of the loads just below the level causing fracture, and the K_{Ii} values at which fracture occurred.

Figures 4-9 also show horizontal straight lines representing the best estimate of K_{ISCC} , the threshold stress intensity above which cracking will definitely occur for an unprotected specimen in 3.5% NaCl solution(2,3,4). In these figures, special attention is drawn to the K_{Ii} values at fracture for the specimens tested under cathodic protection, by indicating the metal used for cathodic protection for these points only.

The appearance of the fracture faces of the specimens was of interest, and is shown in Figures 10-15. In these figures, identification of the individual specimens is facilitated by the fact that they are in the same order, left to right, as listed in Table 3. Furthermore, under each specimen is the K_{Ii} value at which fracture occurred. It should be noted that the specimens were not de-rusted, and thus there are some trivial markings. Comments on each steel type follow.

K _I ,	Values	Obtained	Using	USNRL	Stress-Corrosion	Cracking	Test

TABLE 3

		Nominal	K_ (kpsi√in.)	K _{li} (kpsi√in. at Potentia	.) for Specimens in 3. Is with respect to $S_{\bullet}C$	5% NaCl Solution
Steel	Description	Yield Strength (kpsi)	I for Dry Specimens	-430 to -610 mV (Unpolarized)	-750 to -840 mV (5083 Al)	-1020 to -1090 mV (Zinc)
18% Ni Maraging	3/4-in, plate, parent metal	250	89, 85	69, 68.5	-	37.5
18% Ni Maraging	1/2-in, plate, weld metal	250	51.5	44	44	34.5
HP 9-4-25	3/4-in. plate, parent metal	. 190	115	100, 102, 100	114	48
P.M.D. Cu-Ni Alloy	l-in, plate, parent metal	160	63.5,62	49.5,51	49.5	35.0 (32.5*)
17/4 PH Stainless	3/4-inthick specimens from 1/2-in. plate, H900 condition	180	46.5	46,50	50	32.5
17/4 PH Stainless	3/4-inthick specimens from 1/2-in. plate, H1000 condition	150	93.5 (264**)	91.5 (258**)	71 (210**)	47 (132**)

* Under a cathodic impressed current of -1 mA/cm² (-1270 to -1340 mV, with respect to S.C.E.).

** σ values in kpsi⁽⁶⁾, calculated using Equation 1.



Figure 4. Results obtained on 18% nickel maraging steel (parent metal).



Figure 5. Results obtained on 18% nickel maraging steel (weld metal).

Γ.



Figure 6. Results obtained on HP-9-4-25 steel.



Figure 7. Results obtained on Cu-Ni alloy developed at the Physical Metallurgy Division(7).



Figure 8. Results obtained on 17/4 PH stainless steel, H900 condition.



Figure 9. Results obtained on 17/4 PH stainless steel, H1000 condition.



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Figure 10. Fracture faces of 18% Ni maraging steel (parent metal) specimens, showing K values (kpsivin.).



~X2

Figure 11. Fracture faces of 18% Ni maraging steel (welded) specimens, showing K_I values (kpsi√in.).





Figure 13. Fracture faces of Cu-Ni (PMD) steel specimens, showing K values (kpsi√in.).



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Figure 14. Fracture faces of 17/4 PH (H900) stainless steel specimens, showing K_I values (kpsi/in.). i



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Figure 15. Fracture faces of 17/4 PH (H1000) stainless steel specimens, showing K_I values (kpsi√in.).

(a) 18% Nickel Maraging Steel Parent Metal

The two breaks made under dry conditions show a finegrained surface, relatively flat and smooth at low magnification. The fatigue pre-cracks are clearly visible in Figure 10. Note that the specimen with a KI_i value of 85 acquired a light coating of rust after testing.

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The two specimens with K_{Ii} values of 69 and 68.5 (which had been broken in 3.5% solution in the unpolarized condition) both showed fringes of SCC extending from the base of the fatigue crack, at an angle to the plane of the fatigue crack. Subsequent to the development of these stress-corrosion cracks (which are difficult to see in Figure 10), a rapid fracture occurred, similar to that in the two specimens broken dry.

The specimen which was tested while under c.p. by zinc $(K_{I_1} = 37.5)$ had also shown initial crack propagation from the base of the fatigue crack. However, there was a considerably larger roughened area - approximately 50% of the fracture - before the relatively smooth, flat final fracture mode (resembling the dry fractures) occurred.

(b) 18% Nickel Maraging Steel Weld Metal

Examination of these specimens indicated that the fatigue pre-cracks did not always lie in a plane transverse to the specimen axis. In this respect, this group of steels differed from all others.

It is immediately obvious (see Figure 11) that two of the specimens -- the one broken in unpolarized condition, and the one broken while under c.p. by zinc $(K_{I_1} = 34.5)$ -- showed a pronounced fringe of SCC adjacent to the fatigue crack. In both cases the region of SCC exhibits a columnar appearance and occupies about $\frac{1}{4}$ of the area of the fracture face. Over the remainder of the face, rapid fracture (as with the specimen broken dry) had then occurred. The specimen broken dry ($K_{I_i} = 51.5$) showed the usual relatively smooth and planar face, whereas the specimen broken while under c.p. by 5083 aluminum (second from right, Figure 11) showed what appeared to be a narrow fringe of SCC adjacent to the fatigue crack. This cracking was very nearly in the plane of the fatigue crack, which was transverse to the specimen axis in this case.

(c) HP-9-4-25 Steel

Aside from one exceptional specimen, fracture surfaces appeared to be of the relatively smooth and flat type, similar to that seen in a specimen broken under dry conditions. Perhaps because of the presence of rust, in general, SCC could not be seen adjacent to the fatigue crack.

The exceptional case was the specimen broken while under c.p. by zinc ($K_{I} = 48$). This specimen showed SCC proceeding from the base of the fatigue crack at an angle with respect to the plane of the fatigue crack, and a roughened zone extended over about 80% of the fracture surface.

(d) P.M.D. Copper-Nickel Alloy

In all cases, fractures were of the relatively smooth and flat variety. Perhaps due to the presence of rust, zones of SCC could not be detected.

(e) 17/4 PH Stainless Steel, H900

With the exception of the specimen under c.p. with zinc $(K_{I} = 32.5)$, the fractures were similar to each other, and of theⁱrelatively smooth and flat variety generally shown by specimens fractured in air.

The specimen which had had c.p. by zinc differed from the others in that it showed a region in which the crack had propagated at an angle relative to the plane of the fatigue precrack. This region was not adjacent to the fatigue crack; apart from it there was no severe surface roughening.

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(f) 17/4 PH Stainless Steel, H1000

The specimen cracked under dry conditions ($K_{I_1} = 93.5$) had a much rougher surface than the other steels showed after cracking in dry conditions. Comparison with some photographs of fracture surfaces supplied by S.T. Rolfe⁽⁵⁾ (and bearing in mind the relatively low yield strength of the subject steel) suggested that this specimen may have cracked in a more ductile manner than it would have under plane-strain conditions. In such cases Rolfe avoids using "K" values and, instead, uses the nominal fracture stress σ_{NF} as an index of resistance to SCC⁽⁶⁾. Values of σ_{NF} for this steel (using Equation 1) were, therefore, included in Table 3.

The specimen which had been tested unpolarized in 3.5% NaCl was similar in appearance to the specimen which had been broken dry. However, the two specimens which had received c.p. showed a considerable surface roughening adjacent to the fatigue pre-crack, particularly the one which had received c.p. by zinc $(K_{Ii} = 47)$.

DISCUSSION AND SUMMARY OF RESULTS

In agreement with B.F. Brown's statements, the K_{I_1} values for fracture obtained for unpolarized test bars in contact with salt solution seemed to be independent of the time the bar had been under load. That is, there appeared to be a definite stress-intensity level K_{ISCC} at which stress-corrosion cracks began to propagate rapidly, whilst below this stress intensity there was little or no crack propagation. However, this tentative conclusion only held within the limits of loading times for the few replicate determinations in the present work. It needs to be tested by breaking specimens at slower and/or much faster loading rates (e.g. that used for specimens broken under dry conditions). For the bars which were tested under c.p., no replicate tests were run, so that no information was gained on the effect of total loading time.

Other main points appearing from this work follow:

- (a) For all specimen environments tested, the highest K_I, values were exhibited by HP-9-4-25 steel. (Note that yield strength is an important factor, SCC susceptibility increasing with increased yield strength.)
- (b) In 18% nickel maraging steel, weld metal was found to be distinctly more susceptible to SCC than the parent metal.
- (c) For 17/4 PH stainless steel, the K_{ISCC} values (in 3.5% solution) were equal to the dry K_I values within the experimental error. That is, for the unpolarized specimens, the presence of salt solution did not increase the tendency for cracks to propagate. In this respect, 17/4 PH stainless, in both the H900 and H1000 conditions, differed from the other steels tested.
- (d) In all cases, specimens under c.p. by 5083 aluminum alloy showed higher K_{I_i} values than specimens under c.p. by zinc. With one exception (17/4 PH, H1000 condition), the K_{I} values obtained under c.p. with the aluminum alloyⁱ were equal to or higher than the K_{ISCC} values determined on unpolarized specimens.
- (e) Under c.p. by zinc, all the specimens showed distinctly lower K_I values than were obtained in the other three environments. Under this condition, differences between the alloys were very nearly obliterated, all K_I values falling in the rather narrow range of 32.5-48 kpsi√in. (contrast the behaviour of the dry specimens, which fractured at values of K_I ranging from 46.5 to 115 kpsi /in.).
- (f) The single specimen tested under c.p. by an impressed current of -1 mA/cm^2 (-1720 to -1340 mV) did not yield a K_{I_1} value differing significantly from that obtained under c.p. by zinc.

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- (g) Specimens showing significant fracture-face roughening adjacent to the fatigue pre-crack always showed K_I values significantly lower than those of the same alloy ⁱ broken dry. (The specimens tested under c.p. by zinc showed surface roughening to an unusual degree.)
- (h) For those specimens which had been broken in salt solution, relatively smooth fracture faces tended to be associated with higher K_I values. However, the Physical Metallurgy Division¹Cu-Ni alloy provided results which were exceptional, a specimen with a K_I value of only 35 exhibiting a relatively smooth fracture surface.

CONCLUSIONS AND FUTURE PROGRAM

The most important conclusions from this work are the following:

- (a) The U.S. Naval Research Laboratory SCC test appears capable of yielding reproducible determinations of a stress-intensity factor, K_{ISCC}, which is thought to be related to the tendency of unpolarized metals to fail by SCC.
 - (b) If cathodic protection is used, there appears to be much less danger of rapid crack propagation if potentials are held in the range -750 to -840 mV (5083 aluminum) rather than in the range -1020 to -1090 mV (high-purity zinc). Potentials are relative to the saturated calomel electrode.

The following further work is proposed :

- (a) To obtain more data on the relationship of total time under load to K_{I_i} value;
- (b) determine to what extent c.p. by zinc or high cathodic impressed current embrittles a high-strength steel;

- (c) determine more precisely (using a potentiostat) the effect of c.p. potential on resistance to cracking;
- (d) utilize electron fractography and metallographic methods to obtain more definite information on the cracking mechanisms;
- (e) carry out evaluation tests on other high-strength materials, e.g. HY 140 steel and welded HP-9-4-25 steel.

ACKNOWLEDGEMENTS

The assistance of Dr. B.F. Brown, of the U.S. Naval Research Laboratory, Washington, D.C., in providing information on experimental techniques and apparatus design is gratefully acknowledged.

The stress-corrosion tester was fabricated by the Technical Services Division of the Mines Branch, Department of Energy, Mines and Resources, Ottawa.

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GJB: JGG: (PES)GT



APPENDIX



