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TESTING FOR THE RELATIVE SUSCEPTIBILITY OF
MATERIALS TO INTERNAL HYDROGEN EMBRITTLE-
MENT BY ELECTROCHEMICAL TECHNIQUES

Dennis L. Dull, et al
Aerospace Corporation

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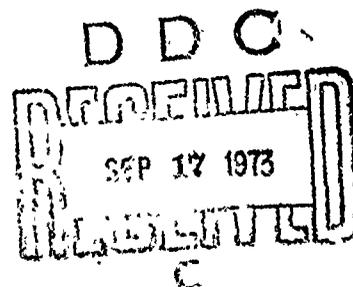
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Testing for the Relative Susceptibility of Materials to
Internal Hydrogen Embrittlement by
Electrochemical Techniques

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Prepared by D. L. DULL and L. RAYMOND
Materials Sciences Laboratory



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Laboratory Operations
THE AEROSPACE CORPORATION

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AIR FORCE SYSTEMS COMMAND
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FOREWORD

This report is published by The Aerospace Corporation, El Segundo, California, under Air Force Contract F04701-73-C-0074.

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Approved



W. C. Riley, Director
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Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



for Ernest L. Lockwood, 1st Lt, USAF
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Galvanic Coupling
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Abstract (Continued)

ABSTRACT

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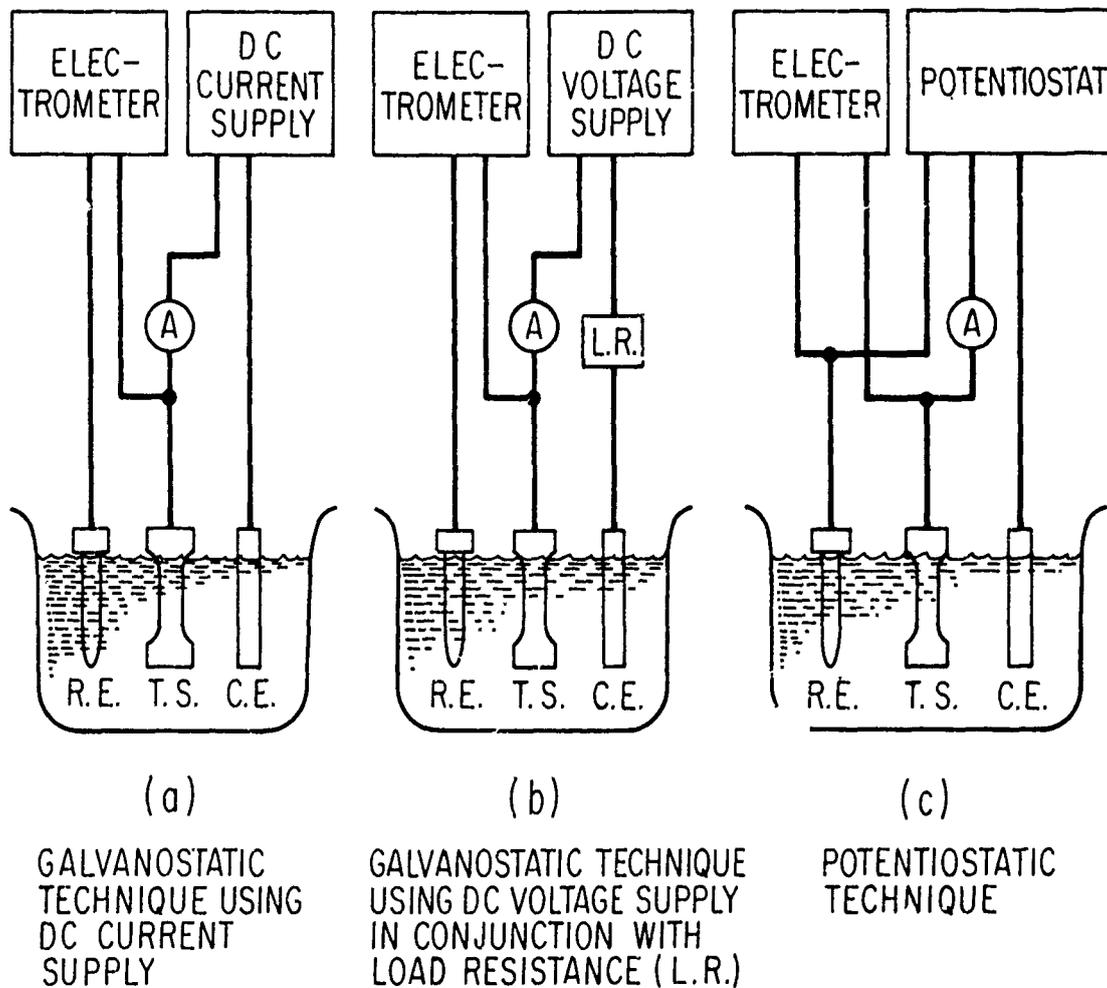
I. INTRODUCTION

Two electrochemical techniques have been used for the introduction of hydrogen into a tensile specimen in a study of material susceptibility to internal hydrogen embrittlement (IHE). The more common technique is the galvanostatic technique in which a constant current is maintained in the circuit containing the tensile test specimen and the counter electrode (Fig. 1). The test cell can also contain a reference electrode in the environment. An electrometer is then used to measure the electrode potential of the test specimen against a standard reference electrode. Either a dc current supply (Fig. 1a) or a dc voltage supply in conjunction with a load resistance (Fig. 1b) may be used as a constant current source. It should be realized that it is the electrode potential and not the constant dc current source that governs the electrochemical reactions occurring at the surface. In the galvanostatic technique, the constant current produces an electrode potential that changes with time as the concentration of the environment changes as the result of precipitation of reaction products and gas evolution.

By comparison, the potentiostatic technique (Fig. 1c) maintains the electrode potential of the tensile test specimen constant as referenced against a standard reference electrode during the test period. Again, the current path is maintained in the circuit containing the tensile test specimen and the counter electrode, but it is not kept constant. Instead, the current is allowed to float and become the dependent variable. With the potentiostatic technique, it is imperative that a reference electrode be used in the electronic circuitry for continuous monitoring of the electrode potential of the test specimen.

The potentiostatic technique offers a method to control the electrode potential of the test specimen within a range that simulates the potentials produced by galvanic coupling of two dissimilar metals under actual service conditions. In contrast, the current densities produced by the galvanostatic

technique can lead to electrode potentials that metals never see in service. This is an important consideration when determining the relative susceptibility to the IHE under actual service conditions. For a more detailed review of the various electrochemical techniques, reference is made to the handbook edited by Ailor [Ref. 1].



NOTE: R.E. IS REFERENCE ELECTRODE; T.S. IS TEST SPECIMEN; C.E. IS COUNTER ELECTRODE; AND A IS AMMETER

Fig. 1. Schematic of electrochemical techniques

II. BACKGROUND

In 1958, H.H. Johnson, et al. [Ref. 2] demonstrated the effect of hydrogen introduced into steel by galvanostatically charging unstressed SAE 4340 notched round bars in sulfuric acid solutions. The authors showed that the time to failure (TTF) and the threshold stress increased as the hydrogen concentration in the steel decreased. It was shown also that the TTF increased as the stress decreased. Later, B.F. Brown [Ref. 3] galvanostatically polarized smooth bar tensile specimens while under stress and demonstrated that the TTF is a function of environment while under cathodic or anodic polarization. Bhatt and Phelps [Ref. 4] summarized the effects of electrochemical polarization on high strength steels and identified the failures under cathodic polarization as those due to IHE.

In 1967, Smialowski and Rychcik [Ref. 5] demonstrated the use of potentiostatic techniques to provide extremely reproducible TTF data. Every experiment was repeated at least six times with very little scatter in data. Uhlig and Cook [Ref. 6] further demonstrated with the potentiostatic technique that the TTF is a function of environment and electrode potential. Leckie [Ref. 7] in 1969 showed that the TTF and the threshold stress are functions of the impressed potential. The results were very sensitive also to a variation in heat treatment for 12Ni Maraging precracked cantilever bend specimens.

In summary, a variety of specimen configurations have been used to demonstrate that steels are susceptible to IHE. The experimental variables are composition, state of stress, environment, electrode potential, and, most important, the heat treatment. Specimens have been cathodically charged without stress in an environment and then had the load applied, while others have been cathodically charged while under stress. The former technique has primarily been applied to evaluation of the severity of hydrogen pick-up during plating of those steels susceptible to IHE; the latter technique has been applied to evaluation of the relative susceptibility

of steels to IHE. In the former, a material recognized to be susceptible, e. g. , SAE 4340, is used, and the emphasis is to avoid deleterious conditions during manufacturing and processing that can lead to IHE. Performance under service conditions is totally ignored, often leading to in-service failures.

The purpose of this study is to demonstrate the usefulness of the potentiostatic technique in generating a standard to evaluate the relative susceptibility of materials to IHE, which is typically related to cathodic polarization conditions. It has generally been accepted that crack growth resulting from anodic polarization conditions is to be termed stress corrosion cracking (SCC). This has implied that two different crack growth mechanisms are prevalent. It is our feeling that substantial evidence has been shown to clearly not warrant such a distinction between these mechanisms in steels.

For instance, anodic polarization does not totally eliminate cathodic reactions from occurring, it only suppresses them with increasing anodic polarization. Furthermore, for materials that can be readily passivated, anodic polarization will enhance pitting corrosion where these pits can serve as nucleation sites for cracks. More important, the localized environment within a pit cannot be typified as the same as the bulk environment. It has been demonstrated by Brown, et al. [Ref. 8] and Smith, et al. [Ref. 9] that the pH within a crack is approximately 3.7 in steel in a 3.5 percent NaCl solution. Smith, et al., further show that the pH within the crack, is essentially independent of the bulk pH of the environment. Thus, SCC is defined as any cracking process in an environment that requires the simultaneous action of the environment and stress. In this study, IHE is a particular SCC mechanism that is characteristic to steels and independent of the polarization direction, i. e. , cracking resulting from anodic polarization is attributed to IHE.

For the determination of the relative susceptibility, it is imperative that the metal-environment system be included. The implication is that

the relative susceptibility depends on a specified critical hydrogen concentration for failure; but, more important, the relative susceptibility is application sensitive. Thus, a given material can have a different rating depending on the specific environment; this environment includes not only the surrounding aqueous solution but also the metal to which it is coupled. A material might have a good rating with regard to performance in an aluminum structure but very poor in a titanium structure. For economical testing, short TTF may be obtained by use of notched round bars at a high stress level. The TTF results are monitored as a function of electrode potential of the tensile test specimen in the environment.

III. EXPERIMENTAL PROCEDURE

A. MATERIALS AND MECHANICAL PROPERTIES

The materials and their heat treatments used in this study are listed in Table 1. They have been categorically separated into three strength classes: Class I, below 220 ksi (1520 MN/m^2) UTS; Class II, 220-250 ksi ($1520\text{-}1720 \text{ MN/m}^2$) UTS; and Class III, above 250 ksi (1720 MN/m^2) UTS.

The mechanical properties for each material are summarized in Table 2. A 20,000-lb (90 kN) Instron testing machine and a 60,000-lb (270 kN) Tinius Olsen testing machine were used for determination of the mechanical properties. The tensile bar specimen and the notch bar specimen are shown in Fig. 2. The stress concentration factor for the notched bar specimen was 5.2.

B. TEST EQUIPMENT AND ENVIRONMENT

A schematic of the test setup is presented in Fig. 3. The potentiostat used was an Anotrol Model 4700 M. The impressed potential and corrosion potential were measured with a Keithley Model 610C electrometer against a saturated calomel electrode (SCE). A Luggin probe was used to reduce the IR drop of the solution during measurement of the potential. A platinum wire served as a counterelectrode during testing.

The environments used were a 3.5-percent NaCl solution and a 5.8-percent (1 M) NaCl solution acidified with concentrated sulfuric acid to a pH 1. All the chemicals were reagent grade; the water was deionized to $>1 \text{ M}\Omega\text{-cm}$ before distillation with a Corning AG-1b distilling apparatus. The 3.5-percent NaCl solution was initially used for testing with all materials. If this environment did not cause failure under an impressed cathodic polarization condition, the more aggressive environment was used. The use of this environment was justified on the basis that, within a pit, the pH can be as low as 2 to 4, the Cl^- ion tends to break down the passive film, and the Cl^- ion concentration is higher [Refs. 8, 10].

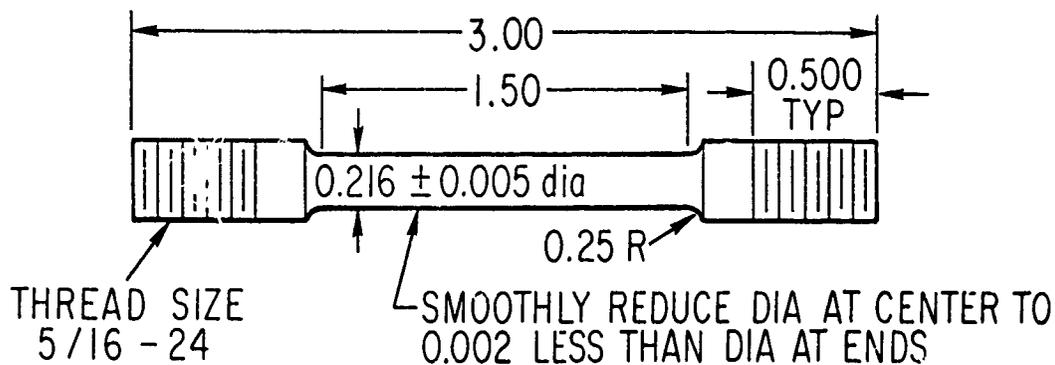
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Table 1. Heat Treatment of Materials Used in Study

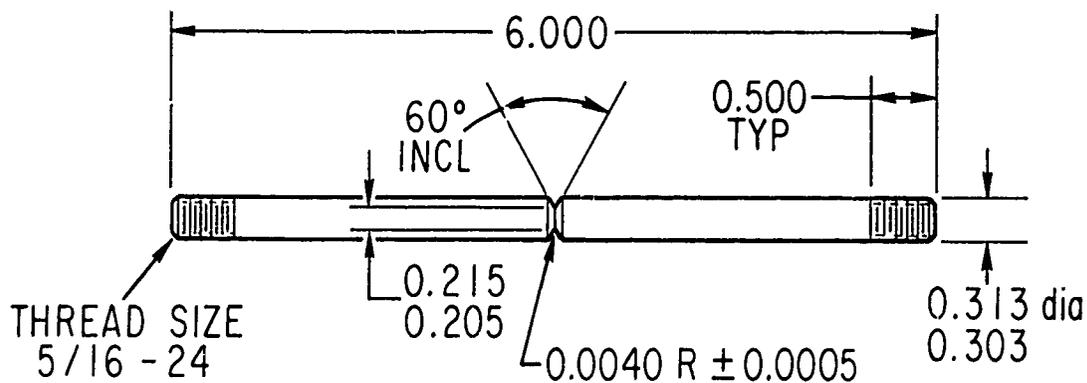
Material	Heat Treatment
<u>Class I (below 220-ksi UTS)</u>	
a. 17-4 PH (H900-1h)	Solution treat 1038 C (1900 F), 1 h; oil quench + age 482 C (900 F), 1 h; and air cool
b. 17-4 PH (H950-4h)	Solution treat 1038 C (1900 F), 1 h; oil quench + age 510 C (950 F), 4 h; and air cool
c. A-286	Solution treat 954 C (1750 F), 1 h; oil quench + cold work 50% + age 677 C (1250 F), 8 h; furnace cool to 621 C (1150 F), 8 h; and air cool
<u>Class II (220-250-ksi UTS)</u>	
a. H-11	Austenitize 996 C (1825 F), 1 h; air cool + triple temper (2 h + 2 h + 2 h) 579 C (1075 F)
b. Inconel 718	Solution treat 954 C (1750 F), 1 h; oil quench + cold work 19% + age 718 C (1325 F), 8 h; furnace cool to 635 C (1175 F), 8 h; and air cool
c. PH 13-8 Mo	Solution treat 954 C (1750 F), 1 h; oil quench + cold treat to -73 C (-100 F) 2 h; air warm + age 510 C (950 F), 4 h; and air cool
<u>Class III (above 250-ksi UTS)</u>	
a. H-11	Austenitize 996 C (1825 F), 1 h; air cool + triple temper (2 h + 2 h + 2 h) 566 C (1050 F)
b. Maraging 300	Solution treat 816 C (1500 F), 1 h; oil quench + age 482 C (900 F), 3 h; and air cool
c. MP35N	Cold work 48% + age 593 C (1100 F), 4 h; air cool

Table 2. Mechanical Properties of Materials Used in Study

Material	Yield Strength (0.2% Offset) ksi (MN/m ²)	Ultimate Tensile Strength (UTS), ksi (MN/m ²)	Notched Tensile Strength (NTS), ksi (MN/m ²)	NTS/UTS
<u>Class I (below 200-ksi UTS)</u>				
a. 17-4 PH (H900-1 h)	190 (1310)	205 (1410)	340 (2440)	1.66
b. 17-4 PH (H950-4 h)	177 (1220)	185 (1280)	310 (2140)	1.68
c. A-286	204 (1410)	219 (1510)	320 (2210)	1.46
<u>Class II (220-250-ksi UTS)</u>				
a. H-11	206 (1420)	248 (1710)	350 (2410)	1.41
b. Inconel 718	231 (1590)	243 (1680)	350 (2410)	1.44
c. PH 13-8 Mo	211 (1460)	227 (1570)	325 (2240)	1.43
<u>Class III (above 250-ksi UTS)</u>				
a. H-11	235 (1620)	280 (1930)	370 (2550)	1.32
b. Maraging 300	267 (1840)	273 (1880)	425 (2930)	1.56
c. MP35N	270 (1860)	275 (1900)	340 (2440)	1.24



TENSILE SPECIMEN



NOTCHED ROUND BAR SPECIMEN

NOTE: DIMENSIONS ARE IN INCHES

Fig. 2. Specimen configuration used in this study

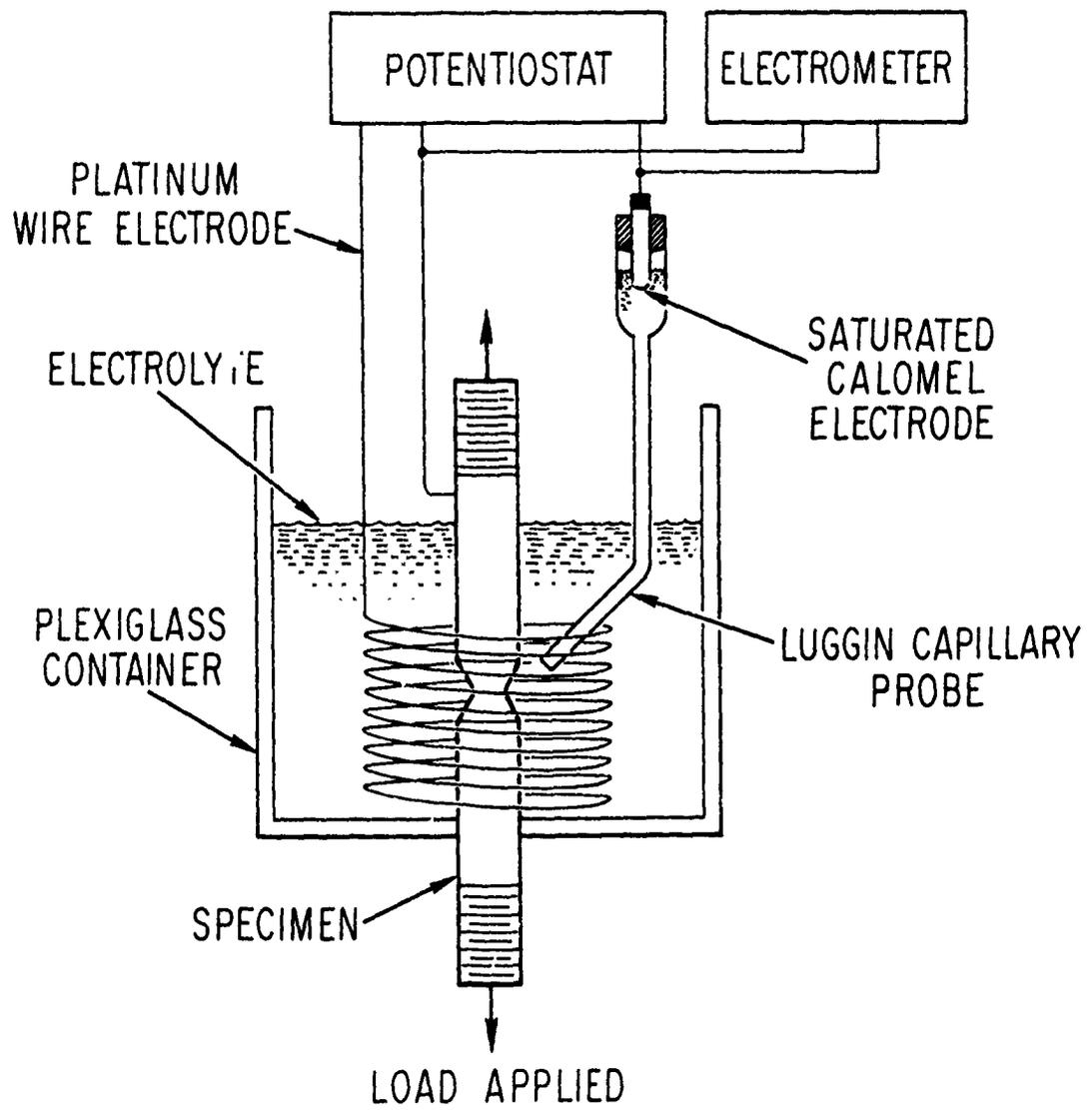


Fig. 3. Potentiostatic stress-corrosion test setup

C. TEST PROCEDURE

Notched round bars were painted with Mil-P-23377 epoxy-polyamide paint to within 1/4 in. of each side of the notch. This was done to reduce the total current flow during polarization. Each specimen was cleaned with isopropyl alcohol and rinsed with distilled water. It was then mounted in a plexiglass container and placed in a 12,000-lb creep rupture frame, at which time the environment was added and the corrosion potential ϕ_{corr} was measured. The specimen was loaded to 0.9 NTS and was potentiostatically polarized to a preselected impressed potential versus the SCE. These potentials were determined from two criteria, i. e., (1) the measured corrosion potential ϕ_{corr} of the material in the environment, and (2) the total potential range, which was from +0.200 to -1.250 V versus SCE. This potential range was felt to best represent the actual service potentials that a typical material would be subjected to if galvanically coupled in a 3.5-percent NaCl solution. Passivated titanium or stainless steel would represent the +0.200 V versus SCE for the more noble potentials, whereas the aluminum or magnesium would represent the -1.250 V versus SCE for the more active potentials.

It is important to realize that the corrosion potentials of most materials in 3.5-percent NaCl will fall within this range. Subjecting a material to potentials beyond this range is unrealistic in normal service conditions; thus, the TTF data have only academic significance.

IV. RESULTS

Results for each material are shown in Figs. 4 - 8. The abscissa is the TTF, and the ordinate is the potential. The plotted curve is being referred to as a potentiostatic stress corrosion life curve (PSCLC). It should also be noted that a specimen without an impressed potential was loaded to 0.9 NTS. It is specifically indicated on the PSCLC with the corrosion potential symbol ϕ_{corr} .

In Fig. 4, the PSCLC for 17-4 PH materials in a 3.5-percent NaCl solution is presented. The effect of the overaging treatment (H950-4 h) of the 17-4 PH material shifts the PSCLC to the right approximately one order of magnitude. This supports previous work [Ref. 11] that shows that an overaging heat treatment reduces the susceptibility of 17-4 PH material to IHE. These curves emphasize the dependence of the time to failure on the impressed potential. This issue will be treated in more detail in the discussion.

In Fig. 5, the PSCLC for H-11 and PH 13-8 Mo materials in 3.5-percent NaCl solution is presented. The shape of the PH 13-8 Mo material PSCLC is similar to the 17-4 PH (Class I) material. The shape of the H-11 material for the potential range more noble than -0.800 V differs from the PH 13-8 Mo material. This behavior is attributed to the different types of corrosion behavior observed in these materials. The PH 13-8 Mo material tends to form pits, whereas, the H-11 material uniformly corrodes with only slight pitting in the early stages of corrosion. In this region, the TTF is considered independent of the impressed potential. This issue will be treated in more detail in Sec. V.

In Fig. 5, the PSCLC for H-11 and PH 13-8 Mo materials in 3.5-percent NaCl solution is presented. The shape of the PH 13-8 Mo material PSCLC is similar to the 17-4 PH (Class I) material. The shape of the H-11 material for the potential range more noble than -0.800 V differs from the PH 13-8 Mo material. This behavior is attributed to the different types of corrosion behavior observed in these materials. The PH 13-8 Mo material

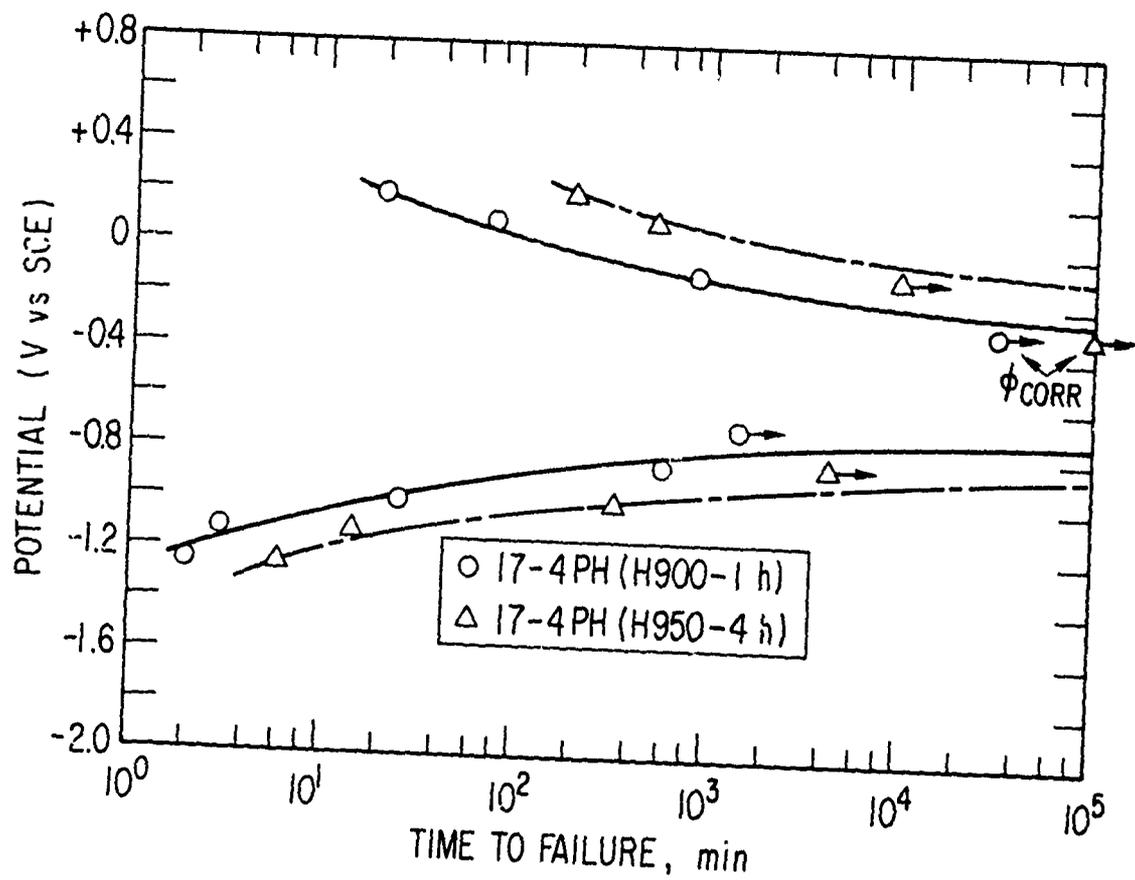


Fig. 4. PSCLC for Class I materials below 220 ksi (1520 MN/m²) UTS in 3.5-percent NaCl solution

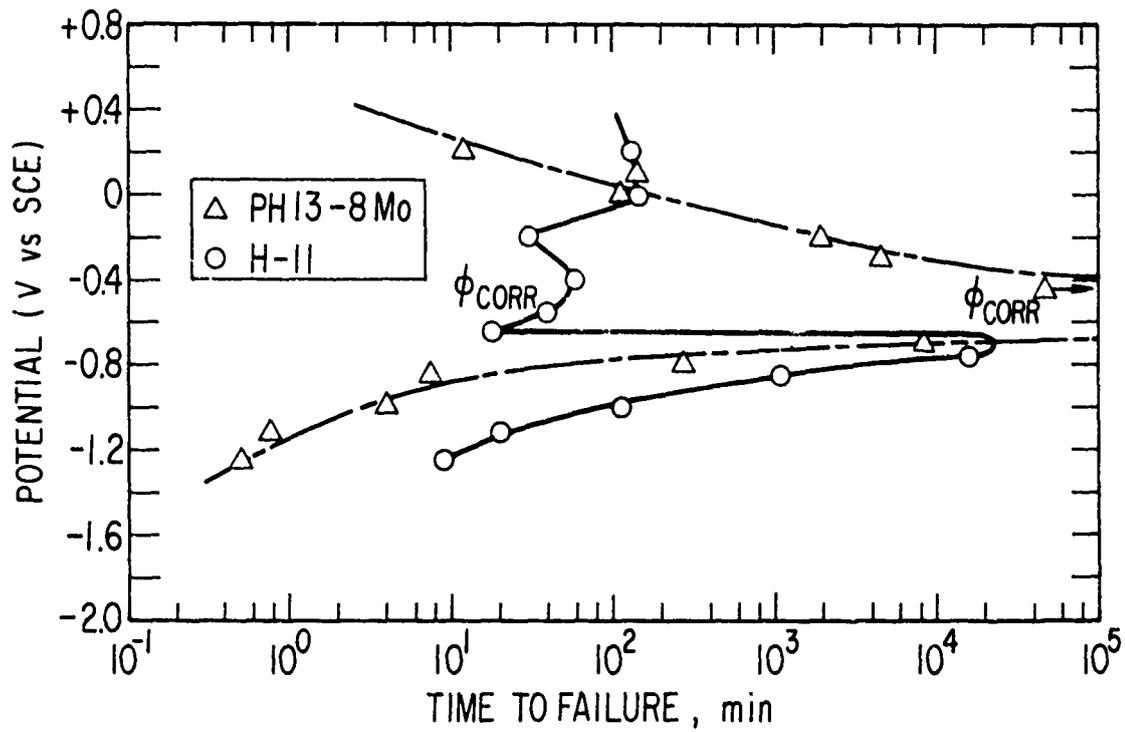


Fig. 5. PSCLC for Class II materials, 220-250 ksi (1520-1720 NM/m²) UTS, in 3.5-percent NaCl solution

tends to form pits, whereas, the H-11 material uniformly corrodes with only slight pitting in the early stages of corrosion. In this region, the TTF is considered independent of the impressed potential.

In Fig. 6, the PSCLC for Maraging 300 and H-11 materials in a 3.5-percent NaCl solution is presented. Under these stress conditions, it is not dependent on the impressed potential. The shape of the PSCLC for Maraging 300 material shows dependency on the TTF for potentials more active than -0.700 V. For potentials more noble than -0.700 V, the TTF is not dependent on the impressed potential, which is similar to the H-11 material already presented.

In Fig. 7, the PSCLC for H-11 material for Class II and Class III are plotted together. Although the shape of the curves remains essentially the same, the shift of the PSCLC for H-11 Class II appears to be, to the right, at least an order of magnitude and upwards ~ 0.2 V. This shift is significant because it shows that tempering H-11 material to a lower strength level will improve its resistance to IHE at all selected potentials.

The PSCLC's for A-286 (Class I), Inconel 718 (Class II), and MP35N (Class III) in a 5.8-percent NaCl solution acidified to a pH 1 are presented in Fig. 8. The A-286 material is shown to fail only in a noble region. Failure is attributed to metal dissolution in the form of pitting with eventual tensile overload. Inconel 718 material is shown to be susceptible in both the noble and active regions. The MP35N material is shown to be susceptible only in the active region. Failures do not occur in the noble region because of its high pitting resistance.

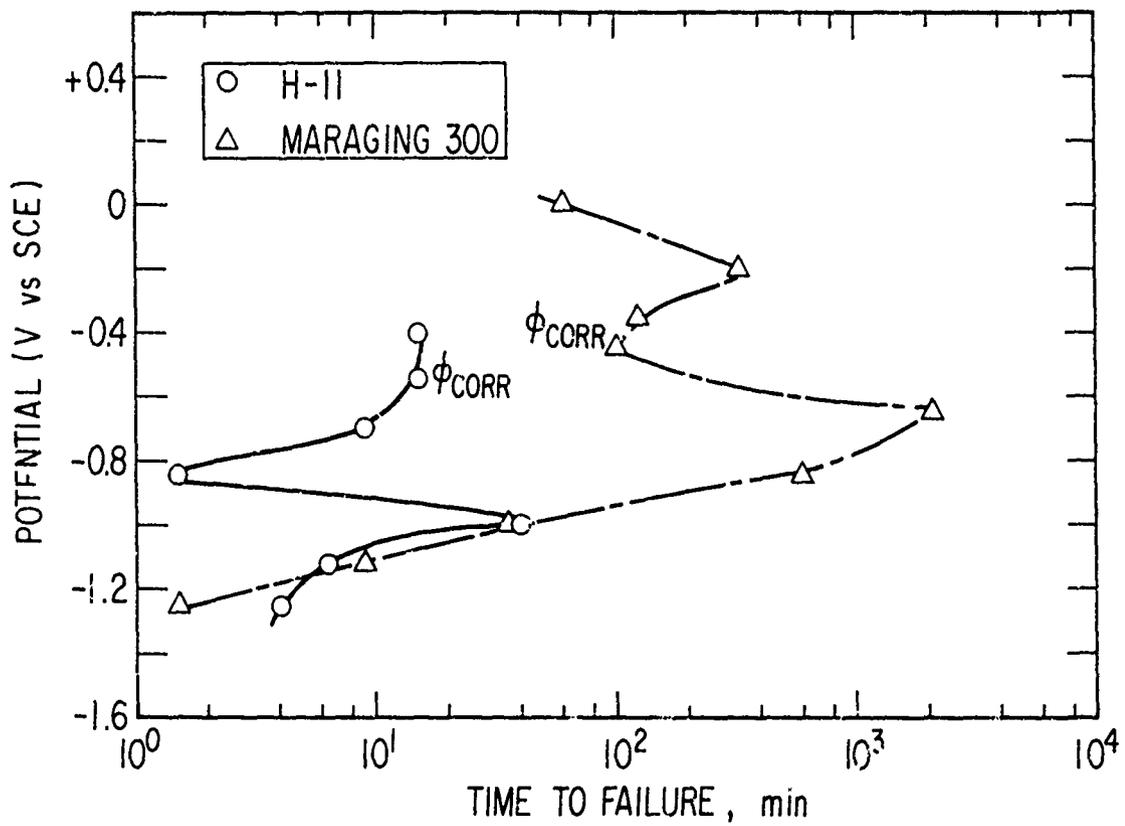


Fig. 6. PSCLC for Class III materials above 250 ksi (1720 MN/m²) UTS in 3.5-percent NaCl solution

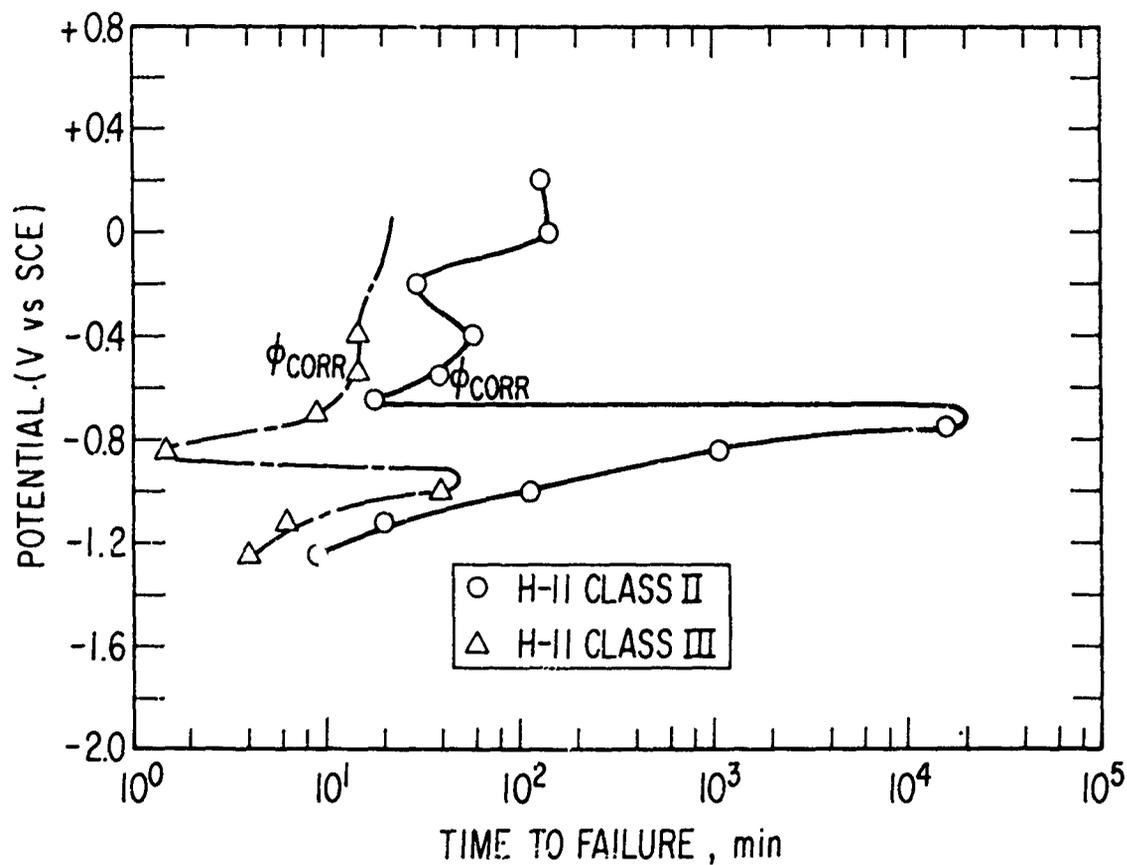


Fig. 7. Effect of heat treatment on H-11 material

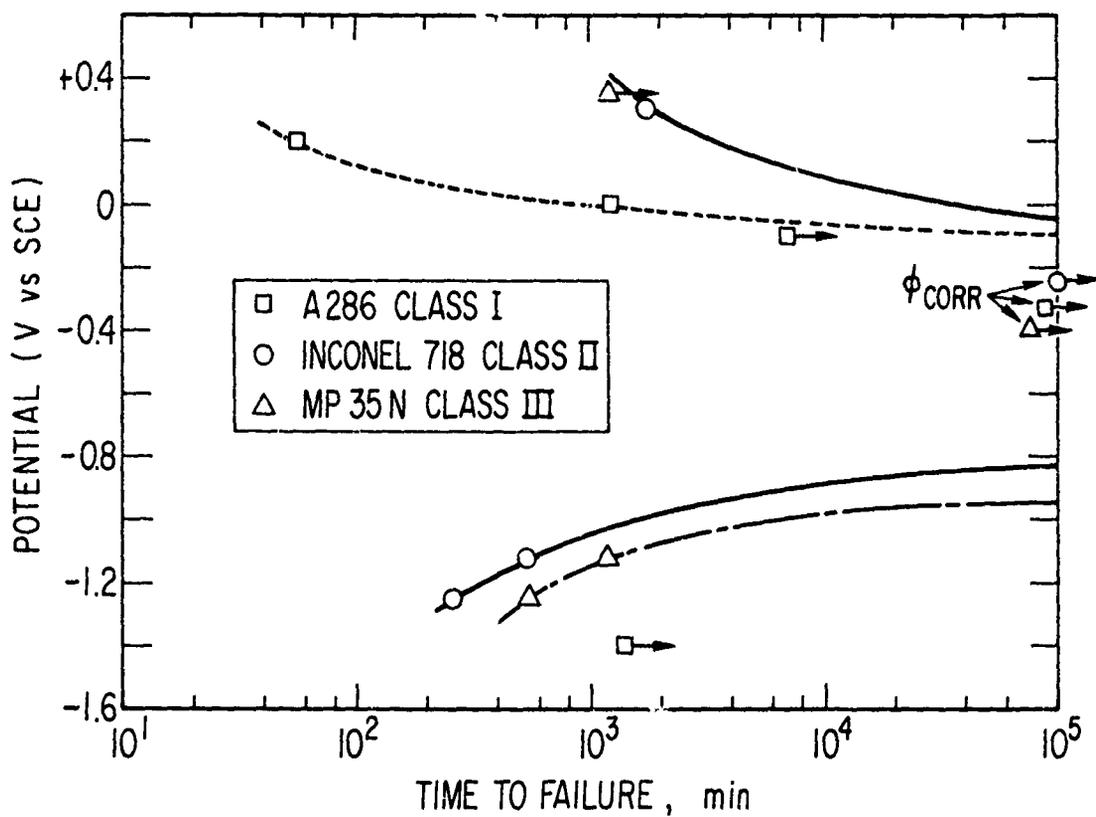


Fig. 8. PSCLC of A286, Inconel 718, and MP35N in 1-M NaCl solution (pH = 1)

V. DISCUSSION

In a selection involving two or more materials for a particular application, difficulty arises because the TTF scatter bands from testing generally overlap. Laboratory results generally do not agree with actual service TTF. This makes it difficult to determine which material is really best. The problem is that TTF is dependent on the electrode potential of the test specimen. The galvanic couple potential ϕ_{gc} , which results when materials form a galvanic couple and which can occur in structures, platings, and other such places, determines the TTF. This potential lies between the ϕ_{corr} of the materials to be coupled and is not easily predicted. The galvanic couple potential ϕ_{gc} depends on the coupling materials, the ratio and condition of the exposed areas, the kinetics of the reactants, and the temperature.

The test method presented in this study can be used to select the best IHE resistant material for a particular application since all foreseeable service potentials can be simulated. This is done in two steps. First, the PSCLC of specific materials in the selected environments are superimposed. Secondly, the anticipated potential range of the galvanic couple is partitioned. The material requiring the longest TTF is then selected. This is illustrated in Fig. 9 for two materials, A and B. With the PSCLC for each material, B is the obvious choice. The test results of this approach are summarized in Table 3. Although the results are not presented for A-286, Inconel 718, and MP35N in a 3.5-percent NaCl solution, it is apparent that these materials, in the more aggressive environments, are still the best choice in each of the stress classes. When coupled to either an aluminum or a titanium structure, Class I and Class III show no change in the order of selection of the most resistant material; however, Class II shows a change in the second and third choices. This change in position is a result of the corrosion resistance of these materials. In the noble region, PH 13-8 Mo material is more

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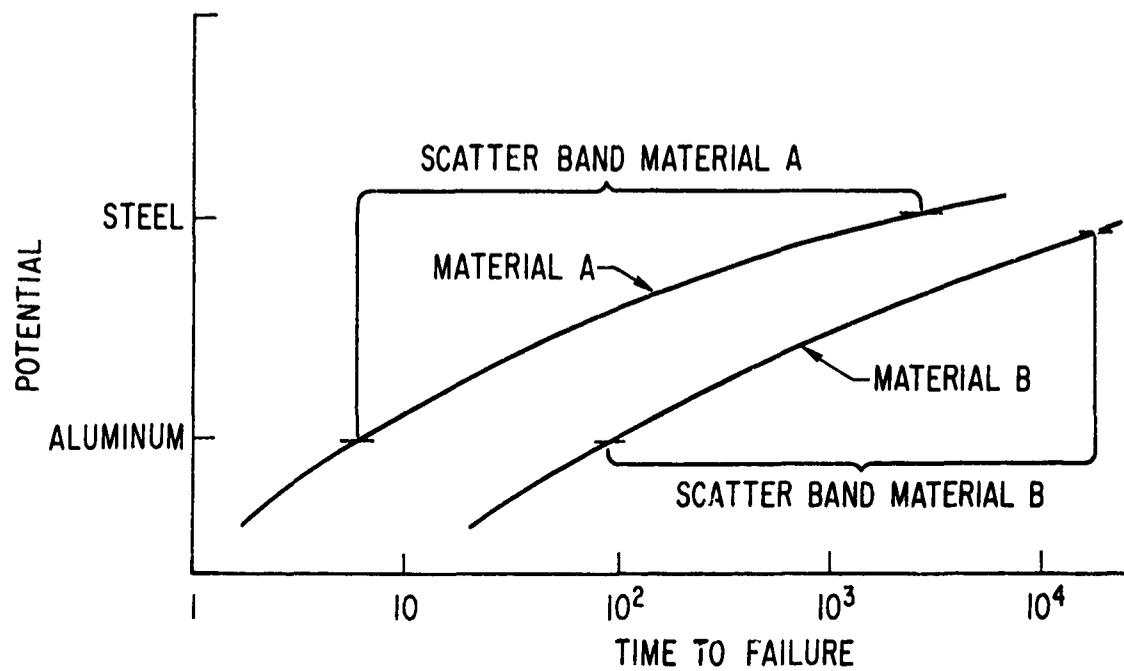


Fig. 9. Selection of steel material to be used in an aluminum structure; material B is the obvious choice

Table 3. Summary of Results of Potentiostatic Techniques on
in Situ Notched Round Bar Specimens

Potentiostatically Simulated Couple to Aluminum ^a	Potentiostatically Simulated Couple to Titanium ^a
<u>Class I</u>	
1. A-286	1. A-286
2. 17-4 PH (H950-4 h)	2. 17-4 PH (H950-4 h)
3. 17-4 PH (H900-1 h)	3. 17-4 PH (H900-1 h)
<u>Class II</u>	
1. Inconel 718	1. Inconel 718
2. H-11	2. PH 13-8 Mo
3. PH 13-8 Mo	3. H-11
<u>Class III</u>	
1. MP35N	1. MP35N
2. Maraging 300	2. Maraging 300
3. H-11	3. H-11

^aMaterials are rated 1, 2, and 3 in order of resistance: most resistant, next-most resistant, and least resistant, respectively.

corrosion resistant than H-11. This gives it more resistance to nucleation and thus more resistance to IHE. In the active region, however, PH 13-8 Mo is quite susceptible to IHE, because the material surface now acts as an absorber of hydrogen without the need for an exterior nucleation site for the occurrence of the necessary reactions when notched bar specimens are used.

Another significant use for the PSCLC is that the effect of galvanic coupling on IHE can be determined; 17-4 PH (H900-1 h) Class I material and H-11 Class III material are two extreme cases. The failure of 17-4 PH material is shown to be dependent on the potential in both the noble and active regions. It should be noted that in a standard static test this material would be resistant to IHE in a 3.5-percent NaCl solution. However, a PSCLC would indicate failure if the 17-4 PH material were galvanically coupled to a different material. The PSCLC for this material in this potential region indicates that the 17-4 PH material is quite susceptible to IHE. The effect of physically coupling 17-4 PH material to aluminum by placing an aluminum block next to a 17-4 PH notched round bar in a salt water environment has been demonstrated [Ref. 11]. Failure by IHE occurred only when the block was attached.

In the latter case, the H-11 material is shown to be independent of the impressed potential in either the noble or the active region. This behavior is attributed to its poor corrosion resistance in the NaCl solution and its extreme susceptibility to IHE in the NaCl solution. The combination of these effects has made the impressed potential only a secondary effect. The impressed potential plays an important part when the material is tempered to a lower strength level, as evidenced by H-11 Class II material (Fig. 7). In the active region, the TTF is dependent on the impressed potential.

For further emphasis of the usefulness of the potentiostatic technique, polarization curves were determined for PH 13-8 Mo, Inconel 718, and H-11 materials in an aerated 3.5-percent NaCl solution by use of standard electrochemical techniques. These results are shown in Fig. 10. If one was to select an anodic current density of 10^{-4} A/cm², the measured

potential for the three materials would read 0.00 V for PH 13-8 Mo, -0.58 V for H-11, and +0.28 V for Inconel 718. The significance of these potentials is that Inconel 718 is being subjected to an impressed potential that would not occur in actual service conditions. Secondly, by comparing these potentials for the H-11 and PH 13-8 Mo materials to the PSCLC for these materials (Fig. 5), it can be shown that the TTF are approximately equal (100 min). If they were compared at the same potential of -0.58 V, the PH 13-8 Mo material would be the better material. It is even conceivable that by application of the appropriate anodic current density, the H-11 material would appear to be the better selection (in this case, the electrode potential would be more noble than 0.0 V). In the case of applying cathodic currents for these materials, little difference is noted in the polarization curves in the 3.5-percent NaCl solution. However, in other solutions such as acids, bases, and organic solvents, the materials would be expected to exhibit definite characteristic differences. Such cases, as previously discussed for the noble potential region, would also be expected to occur.

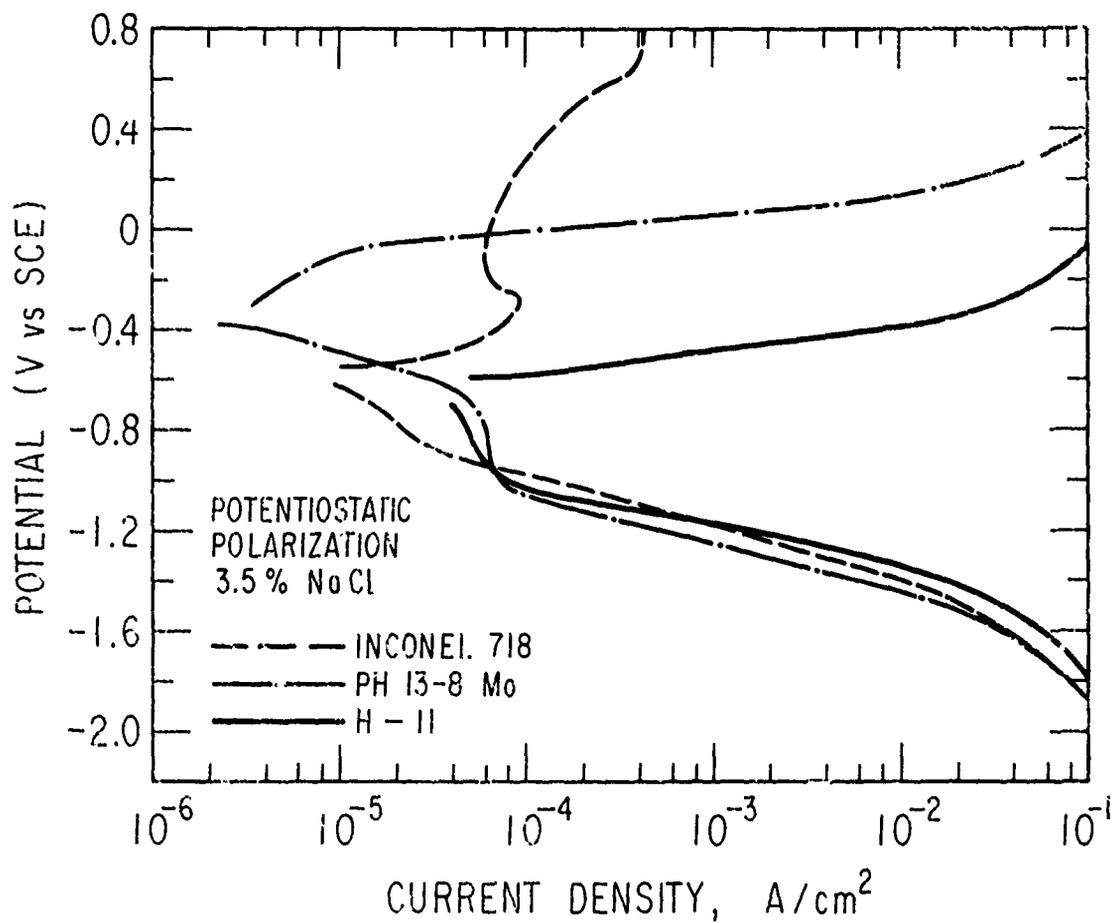


Fig. 10. Polarization curves of Inconel 718, PH 13-8 Mo, and H-11 materials in aerated 3.5-percent NaCl solution

VI. SUMMARY

It is well documented that TTF data of material susceptible to IHE can be influenced by impressing currents or potentials. The current electrochemical techniques employed are the galvanostatic and potentiostatic techniques. In this study, the use of the galvanostatic technique for determination of the relative susceptibility of materials to IHE is criticized because it offers no way of controlling the electrochemical reactions on the material surface with easy reproducibility; electrochemical reactions are potential-dependent. It further allows materials to easily obtain potentials that are not readily found in actual service conditions. This leads to allowing reactions to occur that could result in erroneous conclusions when the susceptibility of two or more materials to IHE in a specific environment is rated.

In this study, stressed notched round bars were potentiostatically polarized and failed by an IHE mechanism. This led to the development of PSCLC for various materials. The significances of these curves are as follows: (1) Experimental scatter obtained from either the standard specification methods or galvanostatic techniques can be explained; (2) the curves allow superpositioning of PSCLC from various materials, so that they may be rated for resistance to IHE; and (3) the curves show the effects of galvanic coupling. The advantages of using the potentiostatic technique on in-situ specimens are as follows: (1) It allows potentials of actual service conditions to be easily simulated; (2) it overrides the problem of the same current density not corresponding to the same potential; and (3) understanding the specific IHE mechanism to rate the various materials is not necessary. Though it was not performed in this study, it is not unreasonable to propose that a test data catalog could be generated for each material with the effects of different environments, temperature, stress, surface coating, and manufacture processing included. Of the electrochemical methods available, the potentiostatic technique is the most suited method for determination of the relative susceptibility of materials to IHE.

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