OCTOBER 1966
REPORT SM-49105-01

STRESS CORROSION CRACKING OF TITANIUM ALLOYS AT AMBIENT TEMPERATURE IN AQUEOUS SOLUTIONS

PROGRESS REPORT FOR PERIOD
JUNE, JULY, AND AUGUST 1966
UNDER CONTRACT NAS 7-488

ASTROPOWER LABORATORY
2121 CAMPUS DRIVE • NEWPORT BEACH, CALIFORNIA

MISSILE & SPACE SYSTEMS DIVISION
DOUGLAS AIRCRAFT COMPANY, INC.
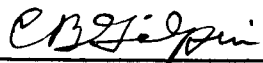### SANTA MONICA/CALIFORNIA

Report SM-49105-Q1

STRESS CORROSION CRACKING OF TITANIUM ALLOYS AT AMBIENT TEMPERATURE IN AQUEOUS SOLUTIONS

Quarterly Progress Report
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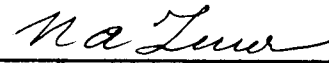
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FOREWORD

This report was prepared by the Astropower Laboratory, Douglas Aircraft Company, Inc., under NASA contract NAS 7-488. The work is administered by Chief, Research SRT NASA Headquarters, Code RRM, with Mr. W. Raring as Project Scientist. This report was prepared by Dr. C. B. Gilpin under the direction of Dr. N. A. Tiner. Dr. T. L. Mackay, Mr. S. M. Toy, and Mr. F. D. Kleist have greatly contributed to the performance of this work.

ABSTRACT

This report covers the work done during the period of July, August, and September 1966 on the study of stress corrosion cracking of titanium alloys at ambient temperature in aqueous solution.

During this period a literature survey was made on subjects related to this problem area. The approach to experimental studies has been discussed.

Preliminary electrochemical evaluations were conducted. Potentiostat curves for Ti-5Al-2.5Sn and Ti-6Al-4V in 3% NaCl, pH 6.5, were determined. Oxygenation was observed to shift the cathodic polarization curves in an electropositive direction, but did not greatly affect the anodic curves. Stress of 80% of the yield strength did not greatly affect the cathodic polarization but did reduce the anodic passivation for both alloys.

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1.0 LITERATURE SURVEY

The stress corrosion cracking of titanium and its alloys at intermediate temperatures of approximately 500 to 800°F has been well documented and has received much attention in experimental studies.[1 - 3] However, until late 1964 it was believed that titanium alloys were completely resistant to stress corrosion cracking at room temperature. At that time Brown was developing a new test for studying stress corrosion cracking of high strength steels.[4,5] That test employed a specimen with a notch that had been sharpened by fatigue. When the same test was applied to titanium alloys, it was discovered that the fracture strength was reduced when the specimen was placed in a 3.5% NaCl environment, in tap water, or in distilled water.[6,7] These results immediately were of grave concern to both the Navy Hull Program and to NASA in connection with the SST. As a result, a tremendous quantity of information has been generated in the last year-and-a-half to develop new alloys and heat treating methods to prevent this susceptibility to stress corrosion.

In addition to the reduction of fracture toughness observed with notched specimens, the effect of an aqueous environment has shown up in two other cases. Judy et al.[8] have shown that the low cycle fatigue crack propagation of Ti-7Al-2Cb-1Ta was greater in aqueous environments at all strain ranges than in air. At a stress of about 60% of the yield strength the growth rate became erratic and much accelerated. The reaction to 3.5% NaCl was more severe than to distilled water. Ti-6Al-4V did not show this effect. Studies of saline waters in titanium loops (alloy not specified) indicated that a tight crevice was necessary for catastrophic corrosion.[9] The attack was favored by low pH, although it has been observed over the pH range of 5.5 to 10.0 at 150°C. The frequency of attack increased from 100°C to 200°C and occurred in either aerated or deaerated solutions.

1.1 Effect of Composition

There have been extensive screening tests conducted to evaluate the effect of composition on susceptibility to sea water stress corrosion. The initial work of Brown et al.[7] indicated that the fracture strengths of Ti-7Al-2Cb-1Ta, Ti-8Al-1Mo-1V, Ti-5Al-2.5Sn, Ti-6Al-4V-1Sn, Ti-6Al-6V-2.5Sn, and Ti-7Al-3Mo were apparently reduced whereas Ti-6Al-4V, Ti-6Al-2Mo,

and unalloyed titanium were less affected. Note that all the alpha alloys appeared to be highly susceptible, whereas some alpha-beta alloys were highly susceptible and some were slightly susceptible.

Lane et al.[10] then evaluated a great number of alloys under consideration in the Navy Titanium Hull Program and broke them into the following two groups:

1. Alloys insensitive to sea water embrittlement were Ti-7Al-2.5Mo, Ti-6Al-2Mo, Ti-6Al-6Sn-1Mo-1V, Ti-6.5Al-5Zr-1V, Ti-6Al-4V, Ti-6Al-2Sn-1Mo-3V, Ti-5Al-2Sn-2Mo-2V.

2. Alloys susceptible to sea water embrittlement were unalloyed titanium (KS-70), Ti-7Al-2Cb-1Ta, Ti-7Al-3Cb, Ti-6Al-2.5Sn, Ti-5Al-2.5Sn, Ti-6Al-3Cb-2Sn, Ti-7Al-3Cb-2Sn, and Ti-8Al-3Cb-2Sn.

Hatch et al.[11] observed that for 0.025 inch thick material Ti-65A and Ti-6Al-4V were not sensitive, whereas Ti-8Al-1Mo-1V, Ti-13V-11Cr-3Al, Ti-5Al-2.5Sn, and Ti-8Mn were susceptible in that order. Thicker sections of Ti-8Al-1Mo-1V and Ti-6Al-4V were both susceptible.

Reactive Metals, Inc.,[12] found Ti-8Al-1Mo-1V, Ti-7Al-3Cb-1Ta, Ti-5Al-2.5Sn, Ti-6Al-4V, and Ti-8Mn to be susceptible, whereas Ti-6Al-2Cb-1Ta-0.8Mo and pure titanium were not susceptible. Many additional studies of composition have been made, but the above results illustrate the effect of composition.

These results indicated that the stress corrosion cracking may be related to aluminum content or to combinations of aluminum and tin. Of course aluminum is not necessary since the Ti-8Mn alloy is very susceptible, as is unalloyed titanium (with high oxygen content). It also appeared that the presence of beta stabilizers such as Mo, V, and Cb decreased sensitivity — for example, Ti-7Al-3Cb was sensitive, Ti-7Al-3Cb-0.2Mo less sensitive, and Ti-7Al-3Cb-0.8Mo insensitive for certain heat treatments.[10] Reactive Metals[12] has shown for the Ti-xAl-2Cb-1Ta and Ti-xAl-3Cb alloys that increasing aluminum from 6 to 7% increases susceptibility, whereas adding molybdenum to both Ti-7Al-2Cb-1Ta and Ti-7Al-3Cb alloys decreases susceptibility. From these results Reactive Metals has had quite good success in relating the beta stabilizer composition and an ordering parameter

to susceptibility. Diagrams of this type show regions in which all heat treatments are susceptible, regions in which some heat treatments are susceptible and some are not, and regions in which all heat treatments are not susceptible.

Lane et al.[10] suggested that the stress corrosion of titanium is related to the ordering observed in various titanium alloys[13, 14] that causes a reduction in toughness in the 6 to 10 weight per cent aluminum range when heat treated from $900^{\circ}F$ to $1300^{\circ}F$. Aging at $1100^{\circ}F$ increases the sensitivity in several alloys. In addition, the faster the cooling from the beta field the less sensitive the sea water stress corrosion. Reactive Metals[12] has observed the same effect on Ti-7Al-2Cb-1Ta. The susceptibility decreases as the cooling rate goes from furnace cooled to air cooled to water quenched from either $1950^{\circ}F$ or $1650^{\circ}F$. The sensitivity of Ti-6Al-4V apparently is greatly affected by the annealing temperature. Annealing around $1450^{\circ}F$ resulted in susceptibility, whereas annealing at $1650^{\circ}F$ resulted in nonsusceptibility.[10]

Due to these great effects of heat treatment and quenching rate on susceptibility, attempts have been made to produce a time, temperature, susceptibility diagram similar to time, temperature, transformation diagrams. Reactive Metals[12] has produced such a diagram for Ti-8Al-1Mo-1V alloy.

1.2   Effect of Microstructure

Several attempts have been made to relate microstructure to the degree of susceptibility to sea water stress corrosion. Hatch et al.[11] has shown that the typical Ti-8Al-1Mo-1V microstructure that exhibits a continuous alpha matrix is very susceptible. However, by heat treating the same alloy to produce discontinuous alpha in a beta matrix there is no reduction in toughness in sea water. Lane et al.[10] observed an apparent correlation between susceptibility and microstructure for a great number of alloys. Microstructures with coarse and long matrix platelets were susceptible, whereas materials with fine platelets and alpha island dispersions were not susceptible. Quench structures that showed needle-like alpha were not susceptible, but aging made the material susceptible without any apparent change in microstructure.

All of the above observations were made with a light microscope. Fine details such as aging must be examined by electron microscopy, but work

in this area has not yet been very complete. Sanderson and Scully[15] have shown by transmission electron microscopy of Ti-5Al-2.5Sn that hydrides were formed on the surface when a metal was plastically deformed while immersed in 3% NaCl. This occurred only in susceptible alloys and suggested hydride formation as an essential feature of cracking.

1.3 Fracturing Behavior

Hatch et al.[11] have studied the mechanical conditions necessary for failure and found that susceptibility is related to the thickness of the test section. For Ti-8Al-1Mo-1V alloy, a 0.045 inch sheet is highly susceptible, whereas a 0.025 inch sheet is not. For Ti-6Al-4V in the mill annealed condition the thickness must be greater than 0.045 inch for susceptibility. Wald[16] has shown similar results for both Ti-8Al-1Mo-1V and Ti-6Al-4V. He also showed that this was due to thickness alone and not to processing variables by starting with a sheet 0.063 inch thick and grinding to various thicknesses. Feige and Murphy[17] have reported minimum thicknesses of 0.025 inch for Ti-8Al-1Mo-1V and 0.045 inch for Ti-6Al-4V. Hatch et al.[11] further showed that a fatigue crack from which a plane strain failure initially propagates is an essential condition. Cracks put in by shear failure did not propagate. These results suggest that the effect of thickness is related to approaching a plane strain condition.

Lane et al.[10] found that fractures did not follow any definite metallographic path such as prior beta grain boundaries or between plates of a Widmanstatten structure. Fracture was transgranular through beta grains and alpha platelets. Furthermore, cracks changed direction when going from one group of platelets of similar orientation to another group of different orientation. Hatch et al.[11] found that the fracture is a low energy failure and is accompanied by very little deformation. There was very little slip or twinning adjacent to the crack. These authors did conclude that fracture was primarily along grain boundaries.

Electron fractographic studies of the fracture have added a great deal to our knowledge about the mode of failure. Hatch et al.[11] found that the fracture propagates by a combination of quasi-cleavage and ductile rupture. Judy et al.[8] have conducted the most complete fractographic studies on low

cycle fatigue in different environments. For the Ti-7Al-2Cb-1Ta alloy tested in air the fracture showed typical fatigue striations. For specimens tested in distilled water there was a mixture of quasi-cleavage and fatigue striations. For specimens tested in 3.5% NaCl the fracture surface was a mixture of fatigue striations and quasi-cleavage facets — the major portion being quasi-cleavage. Thus the amount of quasi-cleavage was directly related to the rate of crack growth. The fracture surface of the Ti-6Al-4V alloy was not affected by environment. These fracture surface studies generally agree with those made by Brown et al.[6] In notched stress corrosion tests above the threshold stress, failure always occurred in a few minutes by quasi-cleavage, whereas below the threshold, failure was of the ductile dimpled rupture type.

Meyn[18] has attempted to study the crystallographic orientation of the cleavage facets observed on Ti-7Al-2Cb-1Ta. The specimens were chill cast to produce large grains. After fracture the orientation of cleavage facets was determined by X-ray Laue back reflection. The angle between the micro cleavage planes and the $(0001)_\alpha$ was $16^\circ \pm 2^\circ$ and was insensitive to its relation to the principle stress axis. The poles of the cleavage planes lay in symmetrically equivalent zones displaced 10° from the $[01\bar{1}0]$ zones and 16° from the $[0001]$ pole.

1.4 Electrochemical Aspects

Detailed mechanism studies of sea water stress corrosion are underway at several laboratories. Feige and Murphy[17, 19] have shown that titanium alloys that can be pitted with anodic potentials of less than 20 volts are susceptible to stress corrosion failure. Specimens cannot be protected by anodic potential but can be protected by about 1000 mv vs. SCE of cathodic potential. They interpret these results and others in terms of film rupture models of stress corrosion cracking. The following steps are proposed:

1. Film rupture at the surface due to applied load.
2. Cl^-, Br^-, and I^- will not passivate this area, whereas OH^- or $O^=$ will.
3. H^+ will be discharged at the cathode which is the surface protective film.
4. Dissolution of titanium takes place.
5. The rate of reaction is dependent upon the rate of slip and the rate of passivation.

Murphy and Feige[20] have observed that cathodic polarization causes the crack to form inside of the specimen. This may be similar to the internal crack initiation in delayed failure of steel observed by Johnson et al.[21] However, Murphy and Feige tend to rule out hydrogen embrittlement as a cause since cathodic protection increases the strength level both above and below the hydrogen evolution potentials. However, at potentials this high, the formation of any type of hydride film at the crack is probably being prevented.

Deaeration of sea water causes a decrease in stress necessary to cause fracture. Deaeration can either increase anodic dissolution by retarding the cathodic reaction or can increase hydrogen embrittlement by eliminating oxygen reduction at the cathode.

Brown[22] has reported that for the Ti-8Al-1Mo-1V alloy, magnesium coupled with titanium will stop crack propagation, zinc will not stop crack propagation but if it is stopped will prevent it from continuing, and cadmium will not stop the crack.

2.0 THEORETICAL CONSIDERATIONS

From these many observations there obviously still are many areas of research necessary to elucidate the mechanism of sea water stress corrosion of titanium alloys. We will direct this investigation towards one of these problem areas, the role of hydrogen. Electrochemical studies that presently exist are inconclusive. They can be interpreted either to favor the importance of hydrogen or to rule out hydrogen completely. During the past several years Astropower Laboratory has been studying the role of hydrogen in stress corrosion cracking of steel[23, 24, 25, 26] and has noted many similarities between that problem and the present one of titanium.

The stress corrosion cracking of smooth 4340 steel specimens in aqueous solutions can be separated into three periods: (1) an incubation period that is very long and is associated with intergranular attack; (2) a stable crack growth period that takes a very short period of time and is associated with both intergranular and transgranular failure; and (3) final rupture resulting from mechanical overload. The stable crack growth period shows many characteristics which can be related to stress corrosion cracking of titanium. If a sharp notch is introduced into a specimen, the incubation period is eliminated, but the stable crack growth period is still present. It is characterized on the fracture surface by transgranular regions exhibiting quasi-cleavage mixed with dimpled rupture and intergranular region. The faster the crack growth (i.e., towards the end of the period) the more dimpled ductile the rupture. These are similar to the observations reported for titanium by Judy et al.[8]

A detailed study of stable crack growth in 4340 steel was carried out by Tiner and Gilpin[23, 24, 25, 26] by combining radioactive tracers of hydrogen (i.e., tritium) and electron microautoradiography. It was shown that hydrogen was introduced into the lattice during stress corrosion and was found only in the cleavage areas in the stable crack growth region. Furthermore, this hydrogen was retained in the microstructural features (in this case epsilon carbides). A similar attempt will be made in the present program on titanium to determine the role of hydrogen, if any.

2.1 Hydrogen Distribution in Titanium

There have been few attempts to determine the distribution of hydrogen in titanium alloys. Huber et al.[27] introduced tritium into a Ti-3Mn-1Fe-1Cr-1Mo-1V alloy and observed the tritium distribution after heat treatment using autoradiography. They found that hydrogen concentrates at alpha-beta boundaries in the form of hydrides after duplex heat treatments involving aging in the 800 to 1100°F range. There also was some evidence that hydrogen partitioned to the beta phase in preference to the alpha phase. There is still a great deal that can be learned about hydrogen and hydride distribution in titanium alloys.

When hydrogen is present in titanium alloys it is known to cause a low strain rate embrittlement or delayed failure. If hydrides are present the material is embrittled even at impact speeds. Quigg and Troiano[28] have pointed out that the amount of hydrogen necessary to embrittle titanium is an order of magnitude greater than in steel. They proposed that hydrogen migrates to regions of inhomogenous plastic strain (as to the root of a notch or along slip planes). This occurs in alpha, alpha-beta, and beta alloys. Beta alloys required 420 ppm and failed very rapidly because of the high diffusivity of hydrogen in beta, whereas alpha alloys were sensitive only over a narrow range of applied stress because of the low diffusivity of hydrogen in alpha.

In the notch stress corrosion test, the requirement of a region of inhomogenous strain is met at the root of the fatigue crack. In this respect Gilpin et al.[29] have shown by electron microautoradiography that hydrogen does segregate to the root of a notch during delayed failure tests in high strength martensitic steels. For alpha titanium alloys relatively long times (one hour or more) are required for delayed failure because of the slow diffusivity of hydrogen.[28] Alpha-beta alloys fail more rapidly, and in beta alloys failure can occur during a notched tensile test (i.e., very rapidly).

The above description is dependent on hydrogen in the titanium metal before diffusing to the root of the notch. In the case of stress corrosion cracking, hydrogen could be introduced into the lattice by chemical attack. This hydrogen would then diffuse to the tip of the crack which is the same place corrosion is taking place. If this is the case, one would expect the

alpha alloys to be more sensitive to stress corrosion failures than the alpha-beta alloys. Since the diffusivity of hydrogen in alpha is low, hydrogen cannot diffuse away and will concentrate at the root of the notch. However, in alpha-beta alloys, hydrogen can diffuse rapidly in the beta phase and will be carried away before it can concentrate sufficiently for cracking to take place. Whether such a mechanism indeed exists will be tested in the present work.

Such considerations may very well explain the observations made by Feige and Murphy[17] for Ti-7Al-2Cb-1Ti, that increasing notch sharpness decreases the load necessary to cause failure (see Table I). Johnson et al.[21] have shown that delayed failures in steel (due to hydrogen) nucleate at the region of maximum triaxial stress which is displaced into the specimen from the actual root of the notch. The sharper the notch, the closer to the root tip this region of triaxial stress is. Gilpin et al.[29] have verified by electron microautoradiography that hydrogen is most concentrated at approximately 0.020 inch from the tip of a 0.010 radius notch. If the sea water stress corrosion is associated with hydrogen diffusion, the sharper the notch the less distance the hydrogen would need to diffuse to a region of stress triaxiality after being introduced into the specimen from the corrosion reaction.

At the present time it is not clear that hydrogen can be introduced into the titanium lattice from a corrosion reaction. Tomashov and Modestova[30] have shown that during corrosion in acid media, alpha titanium alloys under stress do absorb hydrogen. Furthermore, this hydrogen was observed to form hydrides along slip planes (i.e., the region of inhomogenous strain mentioned previously). Sanderson and Scully[15] observed that chemically polished Ti-5Al-2.5Sn immersed in 3% NaCl, pH 0.75, and bent while immersed developed hydrides and cracks within six weeks. Specimens not chemically polished did not develop cracks. Thus it appears that hydrogen can be introduced into titanium, but the conditions for this to occur are not clear.

TABLE I

EFFECT OF NOTCH RADIUS ON FAILURE LOAD[17]

Radius	Air	Load, pounds Sea Water	1 part HCl 1-1/2 parts Sea Water
Unnotched	11,600	11,600	11,600
0.010"	10,560	9,320	7,440
0.005"	10,040	9,630	7,320
0.001"	9,680	8,250	6,280
0.010" fatigue crack	8,280	6,000	—

## 3.0   EXPERIMENTAL EVALUATIONS

During the first quarter electrochemical studies were conducted in order to evaluate the possible electrochemical reactions that should be considered during the stress corrosion of Ti alloys. The studies were limited to mill annealed Ti-6Al-4V (6.0 Al, 4.0 V, 0.13 Fe, 0.12 Ni, 0.025 C, 0.008 H) and Ti-5Al-2.5Sn (5.2 Al, 2.4 Sn, 0.34 Fe, 0.26 C, 0.15 H, 0.028 N). The effect of oxygenation and stress were evaluated.

Sheet tensile specimens, 0.025 inch thick, were abraded with 600 grit silicon carbide and then coated with a lacquer impervious to the solution. A part of this lacquer was then stripped off leaving an area of 0.5 cm^2 of bare metal exposed to the environment. A potentiostat curve for the no stress condition was obtained. The open area was remasked and another open area of the same size made prior to running the potentiostat curve for the stressed condition.

Potentiostat curves were obtained with a Wenking Potentiostat Model 61-TR. The experimental arrangement is shown schematically in Figure 1. All potentials reported are with respect to a saturated calomel electrode. Oxygenation was accomplished by bubbling oxygen through the solution during test and deoxygenation by bubbling nitrogen. The stirrer was located 0.2 cm from the surface being measured. The solution was 3% NaCl, pH 6.5.

The measurements were made by applying a given potential (usually starting at -1400 mv but on occasion up to -2000 mv) and measuring the current after stabilization (usually about one minute). Successive values of potential were measured from negative toward positive to prevent formation of an anodic film. The results are presented in Figures 2, 3, 4, and 5.

The effect of oxygen was to shift the cathodic polarization curves in an electropositive direction. The Tafel slope, b_c, for Ti-5Al-2.5Sn was 0.07 V which agrees with Stern and Wissenberg's[31] value for the hydrogen reaction of titanium in hydrogen saturated 20% H_2SO_4. Assuming the transfer coefficient $\alpha = 1/2$, and since $b = 0.059/\alpha n$ then $n = 1.64 \cong 2$. This would indicate that one of the cathodic reactions in 3% NaCl is $2e + 2H^+ \rightarrow 2H$.
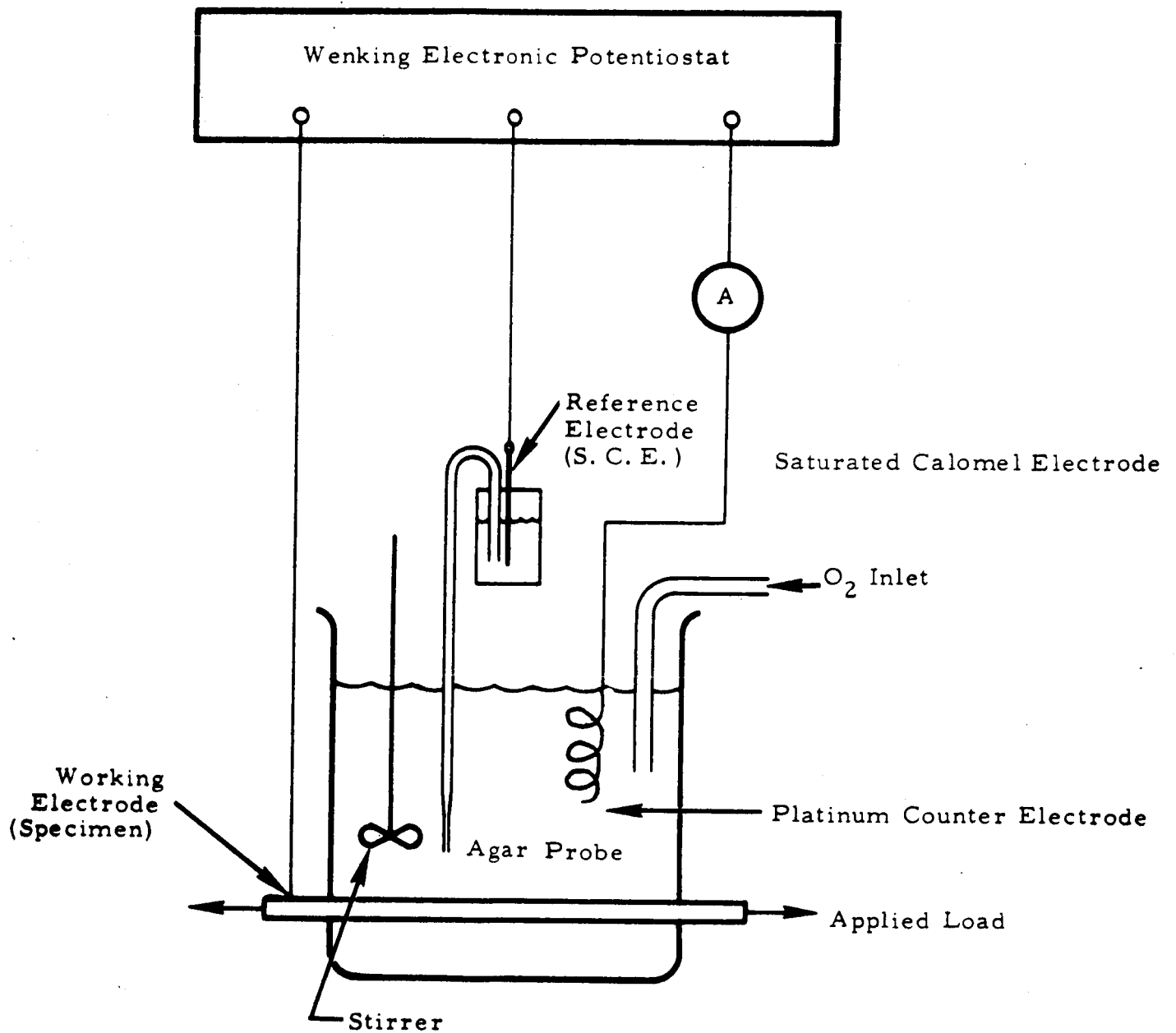
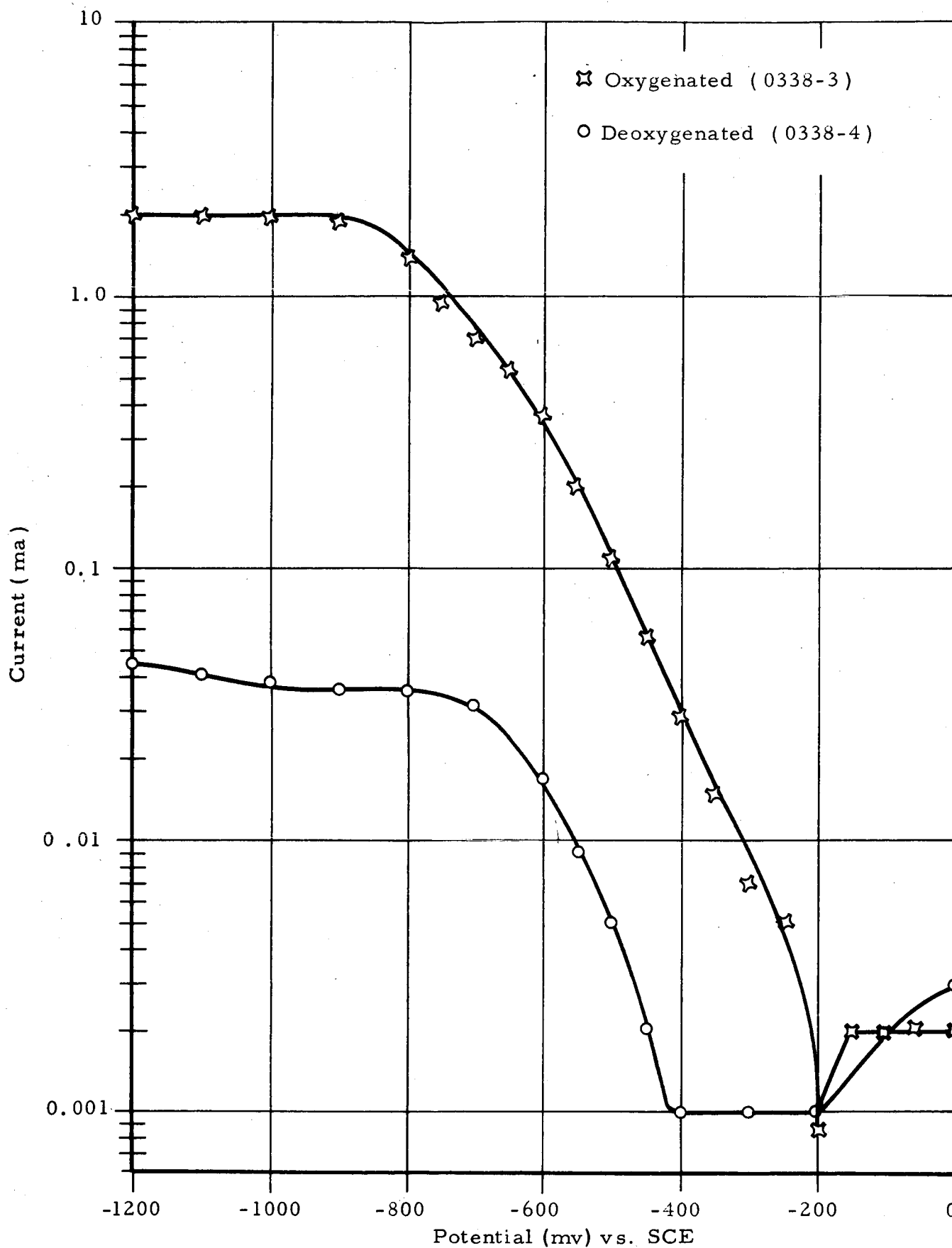Figure 1. Schematic of Potentiostatic Experimental Arrangement

Figure 2.   Effect of Oxygenation on the Polarization Curves
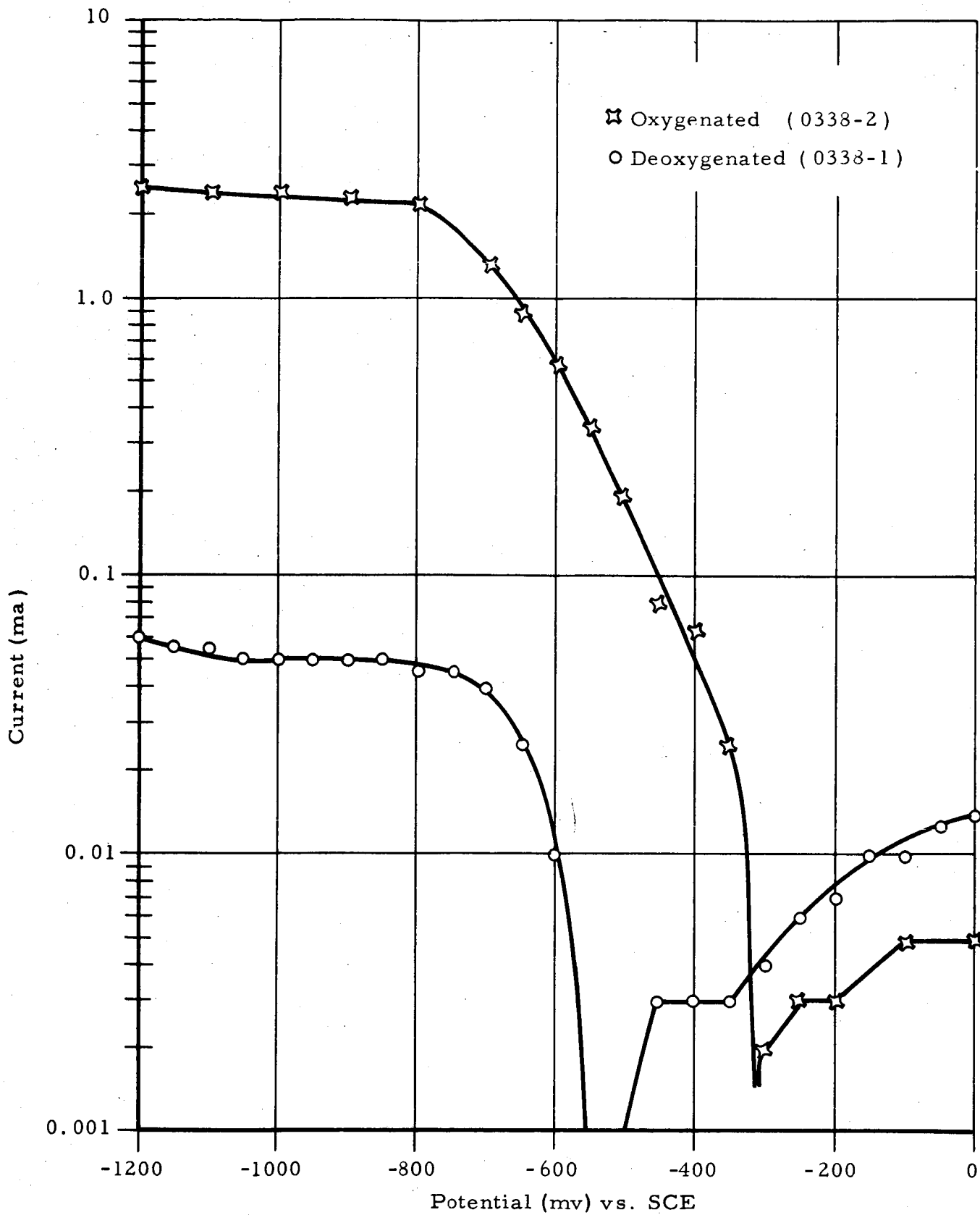of Ti-5Al-2.5Sn in 3% NaCl

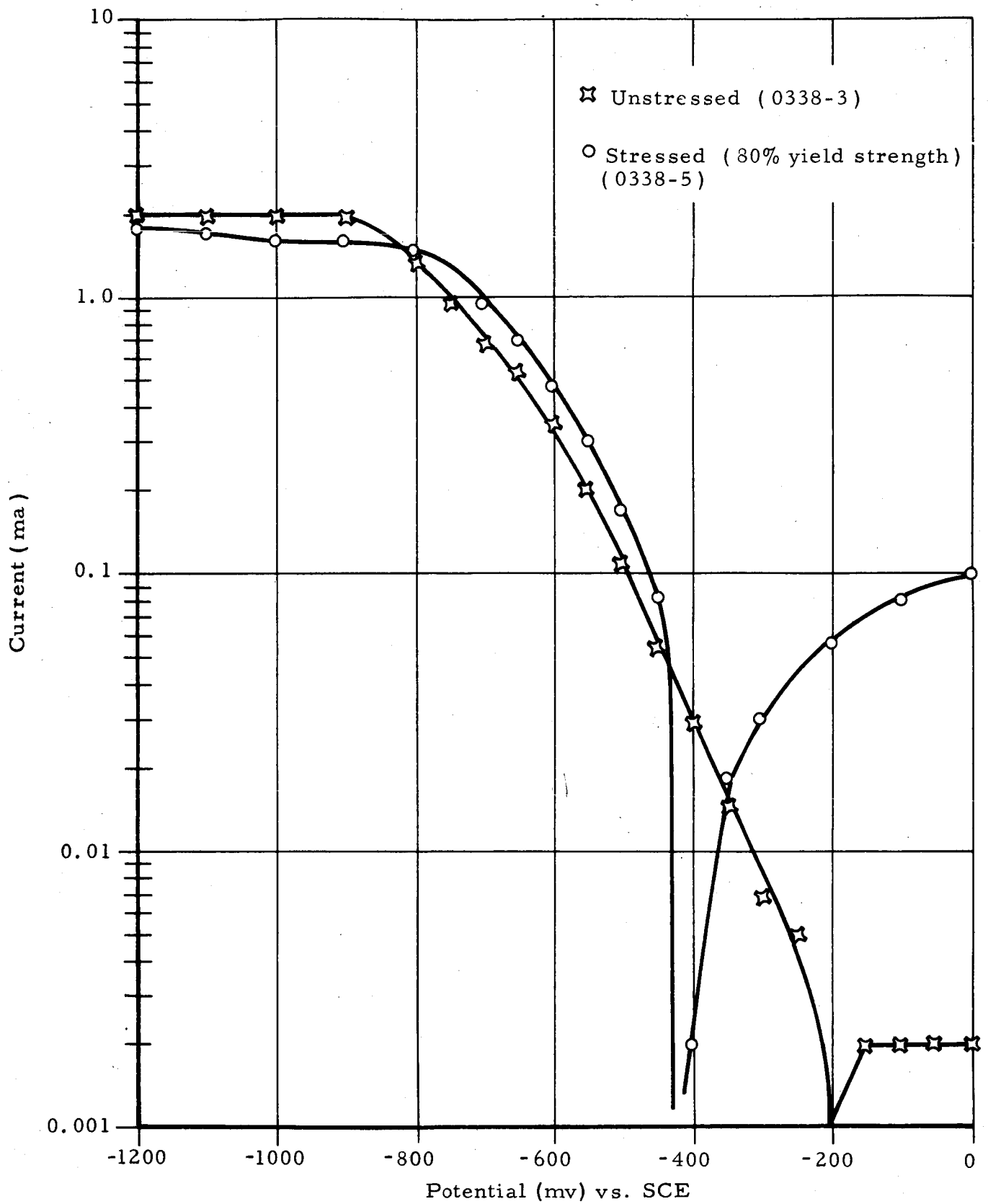Figure 3.   Effect of Oxygenation on the Polarization Curves
of   Ti-6Al-4V in 3% NaCl

Figure 4. Effect of Stress on the Polarization Curve of
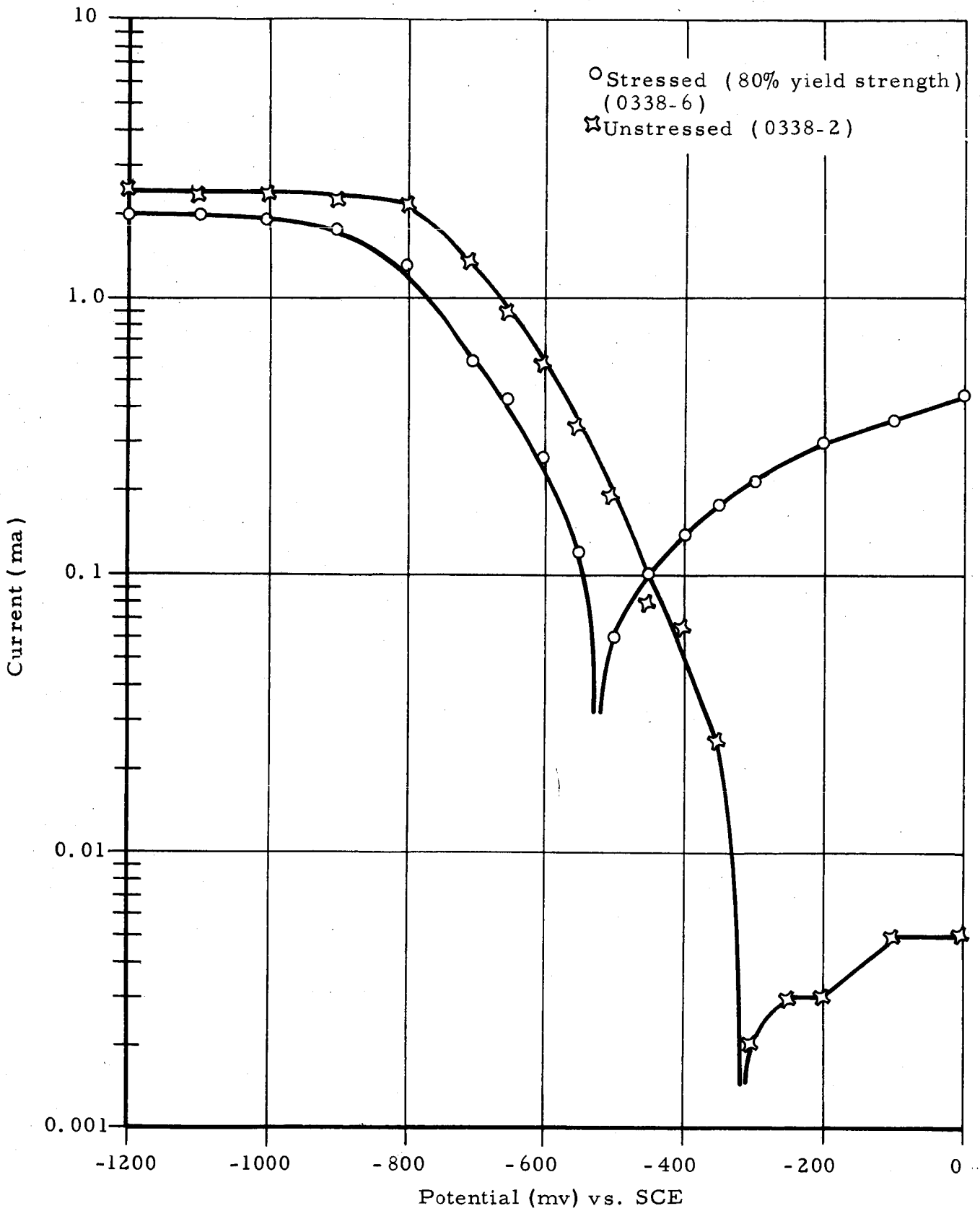Ti-5Al-2.5 Sn in 3% NaCl

Figure 5. Effect of Stress on the Polarization Curve of
Ti-6Al-4V in 3% NaCl

The large increase in current density with oxygenation indicates the strong depolarization effect due to oxygen. Thus it is apparent that cathodic reduction of oxygen $O_2 + 4H^+ + 4e \rightarrow 2H_2O$ also contributes to the cathodic polarization curve. Bubbling air through the solution produced the same results as did oxygen. Oxygenation did not greatly affect the anodic potential.

Stressing the specimens up to 80% of the yield strength did not greatly affect the cathodic polarization. The anodic branch, however, was greatly affected. Stress apparently reduced anodic passivation for both alloys. Thus the current at a given anodic potential was increased when stress was present.

The results of the electrochemical measurements must be considered preliminary until further experimentation assists in elucidating the electrochemical reactions taking place.

4.0   FUTURE WORK

Based on the literature survey, the following titanium alloys have been selected for detailed investigation of the mechanism of stress corrosion cracking:

1.   Ti-5Al-2.5Sn (an alpha alloy)
2.   Ti-6Al-4V (an alpha + beta alloy)
3.   Ti-8Al-1Mo-1V (a super alpha alloy)
4.   Ti-13V-11Cr-3Al (a beta alloy)

Single-edge notched specimens will be fabricated from sheet material (approximately .075" thick).   Stress corrosion tests will be conducted in distilled water and 3% NaCl.   Fracture time will be determined for each of the alloys as a function of load.   The mode of crack propagation during stress corrosion will be evaluated by electron fractography.

In addition to determining the fracturing modes in which hydrogen may play an important part, the distribution of hydrogen in various phases in titanium alloys will be studied.   Specimens 1/2 x 1 inch will be metallographically polished and etched on one surface, cathodically charged with tritium, H^3, and examined by electron microautoradiography.

The sites of local dissolution of titanium alloy matrix will be evaluated by transmission electron microscopy.   Thin foils of titanium alloys suitable for transmission of electron beam will be prepared.   The foils will be examined and then dipped in distilled water or 3% NaCl and reexamined in the electron microscope.   Preferential attack can easily be detected by increased transmission of the electron beam.

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