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ANODIC POLARIZATION OF TITANIUM AND ITS ALLOYS IN HYDROCHLORIC ACID

Technical Report by

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ANODIC POLARIZATION OF TITANIUM AND ITS ALLOYS IN HYDROCHLORIC ACID

ABSTRACT

Commercially pure titanium and the alloys studied exhibited active to passive transitions in HCl. Increasing the acid concentration increased the critical current for passivity and shifted the critical potential for passivity in the noble direction. Increasing the temperature served only to increase the critical current for passivity. Activation energies for the anodic polarization process were the right order of magnitude for a reaction controlled by reactivity at the metal surface. For the alpha-beta alloy 6Al-6V-2Sn the critical current for passivity increased as the ratio of amounts of beta-to-alpha phase decreased with increasing strength level. The dissolution is accelerated, probably by the galvanic effects and the unfavorable area ratio. Ti-75A, a commercially pure metal, had a smaller critical current for passivity value than did the alloys, which was expected. Secondary current density increases were observed for all the alloys at potentials above +1.0 v. For the 13V-11Cr-3Al alloy such behavior was attributed to transpassivity. For the other alloys the phenomena was attributed to pitting or localized corrosion. The commercially pure metal remained passive in this potential range. The addition of Fe^{3+} and Cu^{2+} (0.03 *M*) to the HCl facilitated passivation of the metal and its alloys. The maximum dissolution current densities for the metal and alloys in HCl were double those in H_2SO_4. Pitting of several of the alloys occurred in HCl but not in H_2SO_4.

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INTRODUCTION

In previous work[1] the anodic behavior of alpha-beta titanium alloy 6Al-6V-2Sn was studied in sulfuric acid as a function of temperature, acid concentration, selected ion additions to the electrolyte, yield strength level, and microstructure of the alloy. For comparison, commercially pure alpha titanium and a beta alloy containing 13V-11Cr-3Al were also studied. The present investigation extends this study to hydrochloric acid media, additional commercially available titanium alloys, and considers the directionality of the metal specimen.

EXPERIMENTAL PROCEDURE

The metal and alloys studied were commercially pure alpha-Ti-75A in the annealed condition, alpha-beta Ti-6Al-6V-2Sn heat treated to strength levels of 140, 160, and 180 ksi, beta Ti-13V-11Cr-3Al in the as-received mill-annealed condition, alpha-beta Ti-8Al-1Mo-1V in both the single and duplex annealed condition, and annealed alpha-beta Ti-6Al-4V alloy.

The potential sweep method of potentiostatic polarization (sometimes called potentiodynamic polarization) was used. The electrode potential was continuously changed at a constant rate of 5,000 mv/hr (83.3 mv/min) and current was simultaneously recorded. A Wenking Potentiostat in conjunction with a motor potentiometer for automatic programming of the operating potential and an x-y recorder were employed to automatically record current versus voltage. The automatic potential sweep technique (as opposed to potential step) yields the most reproducible results because of the high resolution offered by continuous recording. The polarization cell and general procedure has been described previously.[1]

Polarization measurements were made in 5, 12, and 20 percent hydrogen-saturated hydrochloric acid electrolytes at temperatures of 35, 50, and 65 C ± 1 degree.

For the directionality studies cylindrical specimens of 1 sq cm surface area were machined both parallel (longitudinal) and perpendicular (transverse) to the working direction. Because of the many similarities in behavior, a number of the polarization curves obtained are not presented.

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[1] LEVY, M. *Anodic Behavior of Titanium and Commercial Alloys in H_2SO_4.* Corrosion, v. 23, no. 8, 1967, p. 236-244; also Army Materials and Mechanics Research Center, AMRA TR 67-16, May 1967.

RESULTS AND DISCUSSION

Polarization of Ti-6Al-6V-2Sn

Effect of Concentration of Electrolyte

Figure 1 shows the anodic behavior of Ti-6Al-6V-2Sn (180 ksi strength level) at 65 C in 5, 12, and 20 percent HCl. Increasing the acid concentration increases the critical current for passivity or maximum dissolution current and the critical potential for passivity is shifted in the more noble direction. Similar behavior was observed at other strength levels and temperatures except at the lowest temperature (35 C) where the curves were characterized by the extremely small currents required to passivate the alloy and the negligible extent of their active regions.

Effect of Temperature

The critical current for passivity for the alloy (140, 160, 180 ksi strength levels) in 5, 12, and 20 percent HCl increases considerably as temperature is increased from 35 C to 65 C. The critical potential for passivity is unaffected. Figure 2 shows log critical current for passivity plotted against the reciprocal of absolute temperature. The result is a linear dependency which can be expressed by the Arrhenius equation and an activation energy of 21 kcal/mol calculated. This activation energy is the right order of magnitude for a reaction controlled by chemical reactivity at the surface rather than a diffusion-controlled reaction.

Effect of Strength Level

Anodic polarization curves for Ti-6Al-6V-2Sn at strength levels of 140, 160, and 180 ksi in 20 percent HCl at 65 C are shown in Figure 3. The critical current for passivity increases with increasing strength level. The critical passive potential is unchanged. X-ray diffraction analysis showed that the beta-phase content decreases from 36 to 23 to 17 percent with a corresponding increase in alpha phase as the strength level increases from 140 to 160 to 180 ksi. It is probable that the alloying additions produce a significant potential difference between the alpha and beta phases which causes the relatively high dissolution currents. The dissolution current increases as the ratio of amounts of beta to alpha phases decreases with increasing strength level. Dissolution may be accelerated both by the galvanic effects and the unfavorable area ratio.

Effect of Iron and Copper Additions

Fe^{3+} and Cu^{2+} were added to the HCl electrolyte in the form of $FeCl_3$ and $CuSO_4$. Figure 4 shows the effect of the Fe addition on the polarization behavior of Ti-6Al-6V-2Sn (180 ksi) in 20 percent HCl at 65 C. The critical current for passivity decreases with increasing Fe concentrations. At all concentrations of Fe there is some inhibition of the anodic reaction. At the maximum Fe concentration (0.03 *M*) the polarization current becomes negative

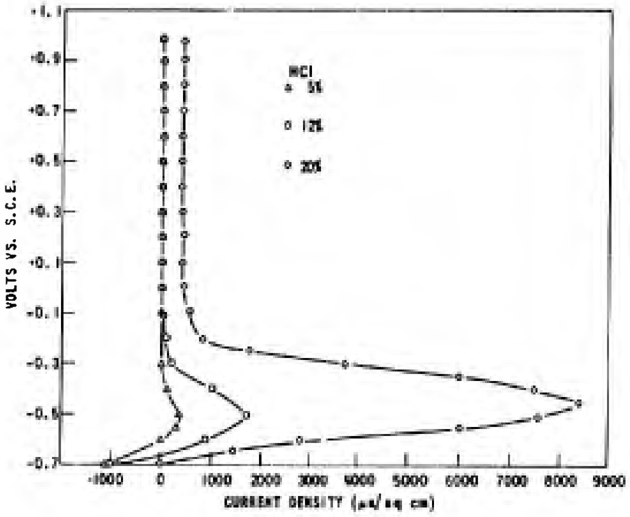

Figure 1. EFFECT OF CONCENTRATION OF HCl ON POLARIZATION OF Ti-6Al-6V-2Sn (180 KSI) AT 65 C

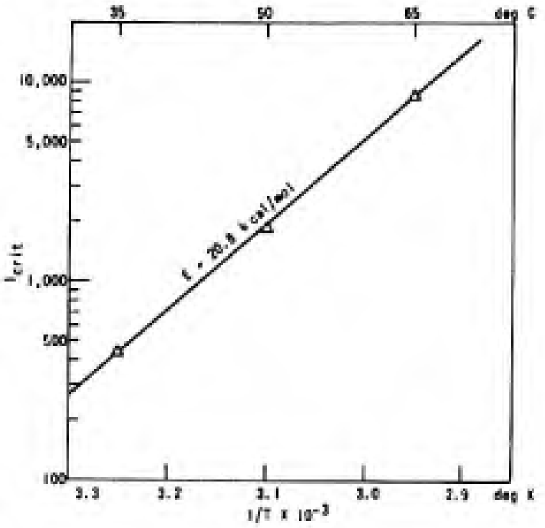

Figure 2. ARRHENIUS PLOT FOR Ti-6Al-6V-2Sn (180 KSI) IN 20% HCl

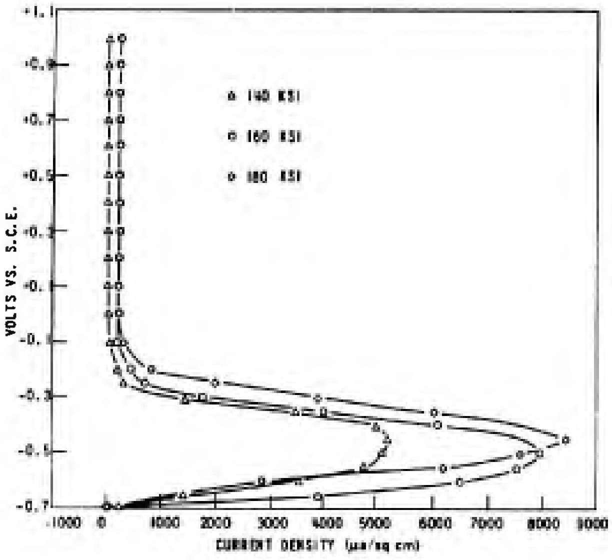

Figure 3. EFFECT OF STRENGTH LEVEL ON POLARIZATION OF Ti-6Al-6V-2Sn IN 20% HCl AT 65 C

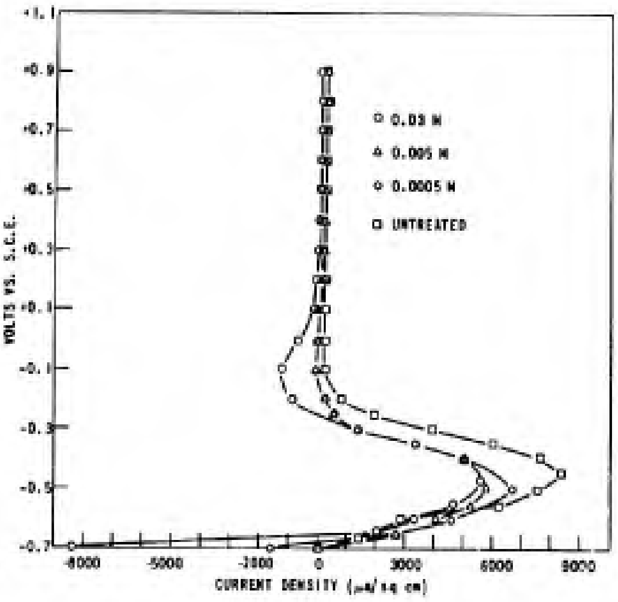

Figure 4. EFFECT OF $FeCl_3$ CONCENTRATION ON POLARIZATION OF Ti-6Al-6V-2Sn (180 KSI) IN 20% HCl AT 65 C

and recrosses the zero current to a positive value at some point in the passive range. This behavior indicates that passivity is stable and a stable passive potential exists. The electrochemical process associated with the cathodic currents is probably hydrogen ion reduction, particularly because the cathodic current occurred at a potential more active than the hydrogen electrode value of the electrolyte. The effect of cupric ions on the polarization behavior of the alloy in 20 percent HCl at 65 C is shown in Figure 5. The Cu^{2+} ion reduces the critical current for passivity. The reduction increases with increasing Cu^{2+} concentration. At 0.03 *M* concentration the maximum dissolution current is approximately one third that of the untreated solution. The Cu^{2+} ion achieves greater inhibition than the Fe^{3+} ion.

Polarization of Commercially Pure Ti-75A

Effect of Concentration of HCl

Figure 6 shows anodic polarization curves for Ti-75A in 5, 12, and 20 percent HCl at 65 C. The maximum dissolution current increases with increasing HCl concentration. The critical potential for passivity is essentially the same in both 12 and 20 percent solutions. At the lowest HCl concentration, the curve is characterized by the negligible extent of the active region and a very small current is required to passivate the metal. At a potential of +1.5 v different behavior is observed in 5 percent HCl solution. A secondary active-passive transition is observed. Although Levy[1] reported transpassive behavior for Ti-13V-11Cr-3Al in H_2SO_4, transpassivity has not been reported for commercially pure titanium. Why it should occur only in 5 percent HCl is not understood. Perhaps it does not represent true transpassive behavior but rather is an artifact.

Effect of Temperature

Figure 7 shows the effect of temperature on the polarization of the metal in 20 percent HCl. Paralleling the effect of concentration, the critical current for passivity increases with increasing temperature. The rate of increase corresponds to an activation energy of about 20 kcal/mol. At 35 C, even at the 20 percent acid concentration, a relatively small current is required to passivate the metal. The dissolution current in the passive range is of very low order of magnitude at all temperatures. Of course, the lower the value of the corrosion current in the passive state, the more stable the passive state.

Effect of Fe^{3+} Additions

Figure 8 shows the effect of $FeCl_3$ additions on the polarization behavior of Ti-75A in 20 percent HCl at 65 C. $FeCl_3$ in concentrations of 0.005 *M* and 0.0005 *M* accelerates anodic dissolution. A concentration of 0.03 *M* $FeCl_3$ reduces the maximum dissolution current slightly. At this concentration cathodic currents are observed in the passive range which indicates a more stable passive potential exists.

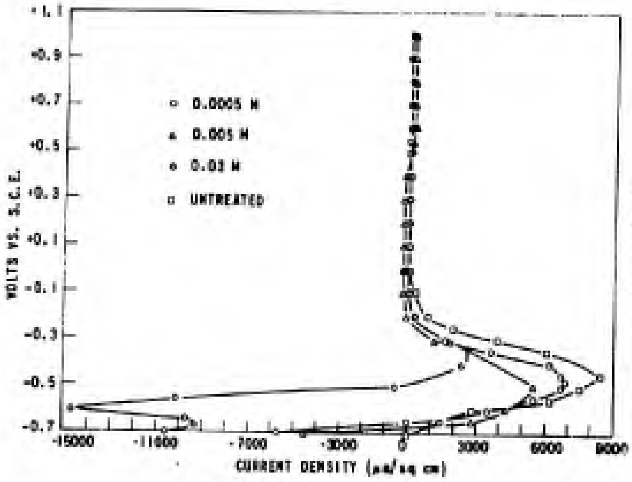

Figure 5. EFFECT OF $CuSO_4$ CONCENTRATION ON POLARIZATION OF Ti-6Al-6V-2Sn (180 KSI) IN 20% HCl AT 65 C

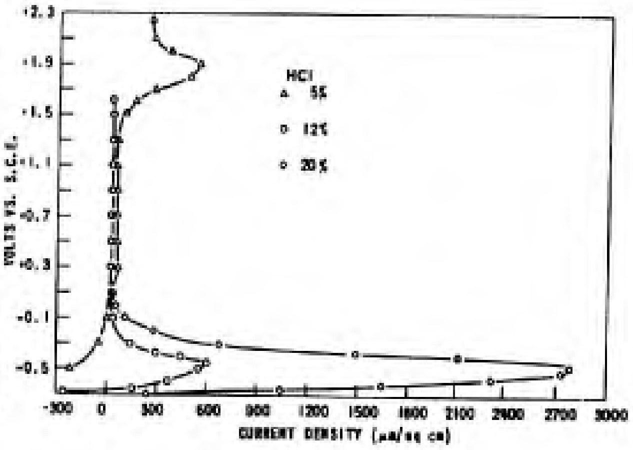

Figure 6. EFFECT OF HCl CONCENTRATION ON POLARIZATION OF COMMERCIALLY PURE Ti-75A AT 65 C

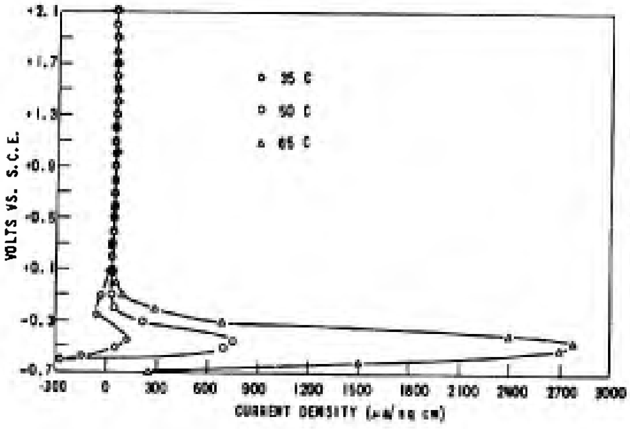

Figure 7. EFFECT OF TEMPERATURE ON POLARIZATION OF COMMERCIALLY PURE Ti-75A IN 20% HCl

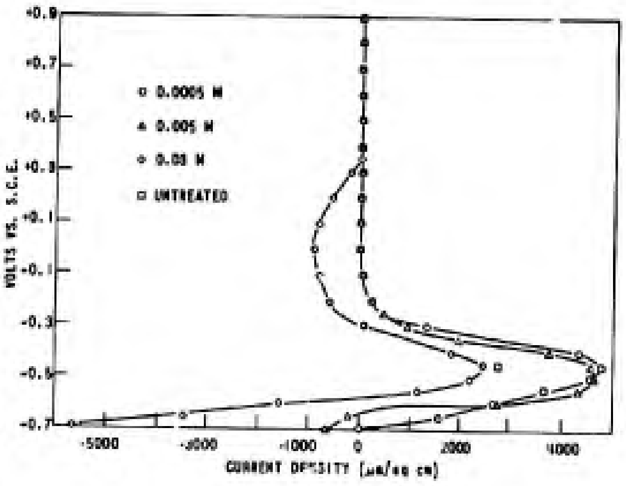

Figure 8. EFFECT OF $FeCl_3$ CONCENTRATION ON POLARIZATION OF COMMERCIALLY PURE Ti-75A IN 20% HCl AT 65 C

Polarization of Ti-8Al-1Mo-1V

Concentration of HCl

Figure 9 shows anodic polarization curves for the alloy as a function of HCl concentration at 65 C. The maximum dissolution current increases with increasing concentration. The critical potential for passivity remains essentially the same. Note the negligible extent of the active region in 5 percent HCl even at 65 C. At potentials between +1.0 v to +1.25 v, depending upon the concentration, a secondary anodic reaction occurred. Such behavior is usually attributed to transpassivity. However, pitting occurred at these potentials indicating that the reaction was not one involving general dissolution or transpassivity.

Effect of Temperature

Figure 10 shows anodic polarization curves for the alloy in 20 percent HCl as a function of temperature. The critical current for passivity increases with increasing temperature at a rate corresponding to an activation energy of about 20 kcal/mol. There is no significant change in the critical potential for passivity. Pitting occurred at all temperatures at potentials above +1.1 v where the current density increased appreciably. The potential at which pitting occurred was temperature dependent.

Effect of Directionality

The anodic behavior of the alloy in both longitudinal and transverse directions in 20 percent HCl and 65 C is shown in Figure 11. The maximum dissolution current density for the specimen fabricated in the transverse direction (machined normal to the working direction) was slightly greater than that fabricated in the longitudinal direction (parallel to the working direction). Pitting occurred in both transverse and longitudinal specimens but at a more noble potential in the longitudinal specimen (+1.3 v versus +0.9 v).

Effect of Heat Treatment

The effect of heat treatment on the polarization of Ti-8Al-1Mo-1V in 20 percent HCl at 65 C is shown in Figure 12. The critical current for passivity and the critical potential for passivity are essentially the same for the alloy in the single- and the duplex-annealed conditions. Localized corrosion (pitting) of the single-annealed alloy occurred at a potential of +1.0 v and of the double-annealed alloy at +1.3 v.

Polarization of Ti-13V-11Cr-3Al

Effect of Concentration of Electrolyte

Figure 13 shows the polarization behavior of the alloy in 5, 12, and 20 percent HCl solutions at 65 C. The critical current for passivity

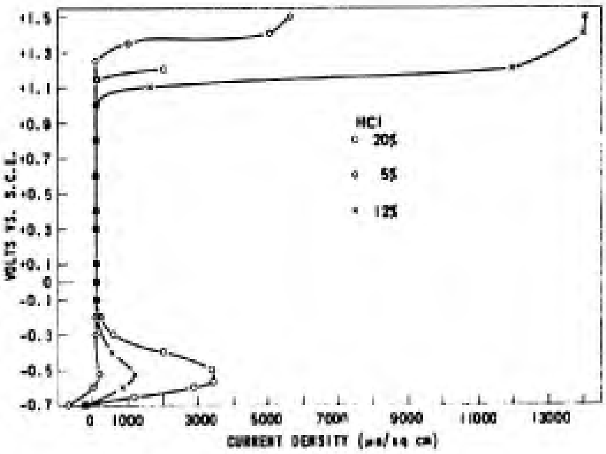

Figure 9. EFFECT OF CONCENTRATION OF HCl ON POLARIZATION OF Ti-8Al-1Mo-1V (DUPLEX ANNEALED) AT 65 C

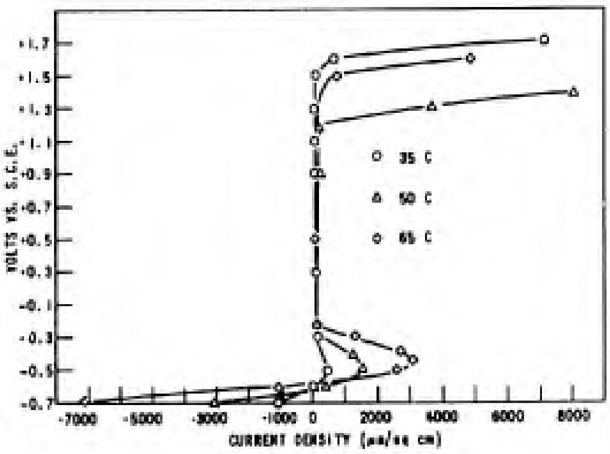

Figure 10. EFFECT OF TEMPERATURE ON POLARIZATION OF Ti-8Al-1Mo-1V (SINGLE ANNEAL, TRANSVERSE) IN 20% HCl

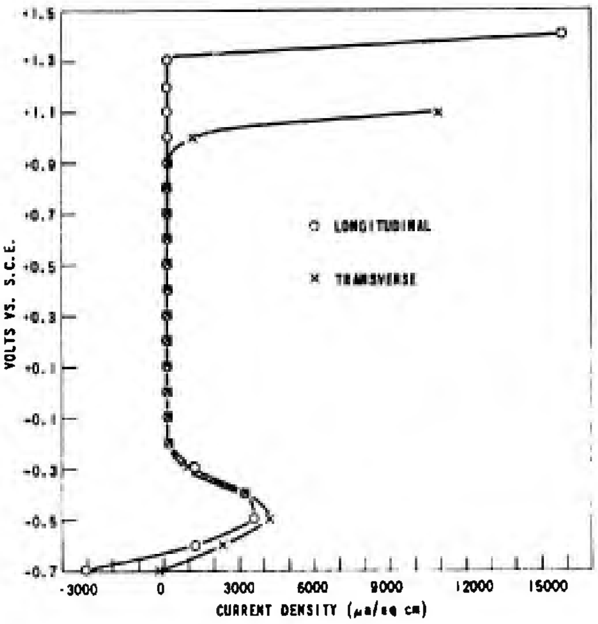

Figure 11. EFFECT OF DIRECTIONALITY ON POLARIZATION OF Ti-8Al-1Mo-1V (DUPLEX ANNEAL) IN 20% HCl AT 65 C

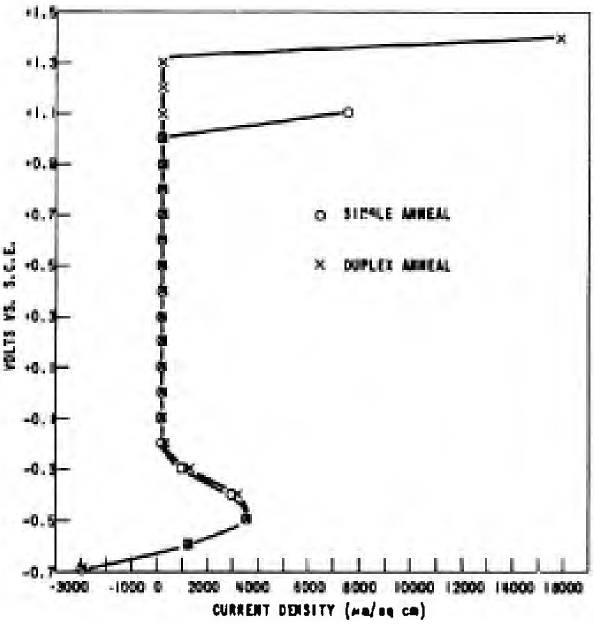

Figure 12. EFFECT OF HEAT TREATMENT ON POLARIZATION OF Ti-8Al-1Mo-1V (LONGITUDINAL) IN 20% HCl AT 65 C

increases with the increasing HCl concentration and the critical potential for passivity shifts in the more noble direction. Although not shown in the figure, above +1.0 v a secondary reaction was observed at all HCl concentrations and was characterized as transpassive behavior due to the 11 percent chromium content of the alloy.

Effect of Temperature

Figure 14 shows that the critical current for passivity increases with increasing temperature. The activation energy for the polarization process (about 20 kcal/mol) is the right order of magnitude for a reaction controlled by chemical reactivity at the surface. The alloy exhibited transpassive behavior at all temperatures at potentials above +1.0 v (not shown).

Effect of Cu^{2+}

Figure 15 shows the effect of $CuSO_4$ on the polarization of Ti-13V-11Cr-3Al in 20 percent HCl at 65 C. Corrosion is accelerated by the addition of $CuSO_4$ in concentrations of 0.005 *M* and 0.0005 *M*. The anodic reaction is markedly stifled in 0.03 *M* solution. Note the cathodic currents in the passive range which indicate that the passivity is stable.

Polarization of Ti-6Al-4V

Effect of Temperature

The temperature dependence of the anodic polarization process is shown in Figure 16. The critical current for passivity increases with increasing temperature. Again the activation energy is the right order of magnitude for a chemical-controlled reaction rather than diffusion-controlled. Paralleling the effect of temperature, increasing the HCl concentration increased the critical current for passivity.

Comparison of Polarization Behavior of Titanium and Alloys

The anodic behaviors of commercially pure titanium and several alloys in 20 percent HCl at 65 C are compared in Figure 17. Based on maximum dissolution current, alpha-beta Ti-6Al-6V-2Sn shows the least corrosion resistance followed by beta Ti-13V-11Cr-3Al, alpha-beta Ti-6Al-4V, alpha-beta 8Al-1Mo-1V (10% beta), and alpha Ti-75A, in order of increasing corrosion resistance. Since the anodic polarization process was shown to be controlled by reactions occurring at the surface, metal composition and structure should achieve their greatest effect. Composition gradients in a metal create galvanic cells causing accelerated attack of anodic areas. A pure metal or homogeneous single-phase alloy, therefore, is usually more corrosion-resistannt than an impure or multiphase alloy, which appears to be the case here. The critical potential for passivity is essentially the same for the metal and the alloys, which means that the alloying does not significantly alter the critical potential for passivity. The commercially pure Ti-75A remains passive throughout the potential range (from -0.2 to +2.3 v). All the alloys exhibit secondary reactions above potentials of +1.0 v. The secondary

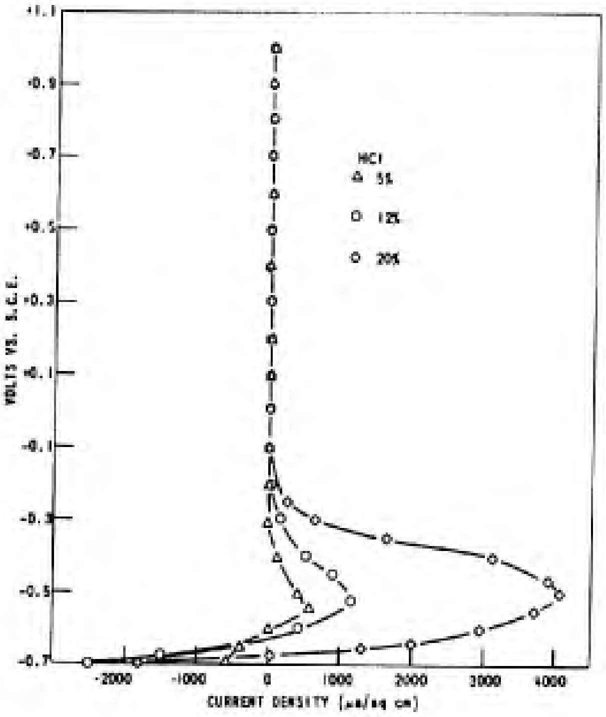

Figure 13. EFFECT OF HCl CONCENTRATION ON POLARIZATION OF Ti-13V-11Cr-3Al AT 65 C

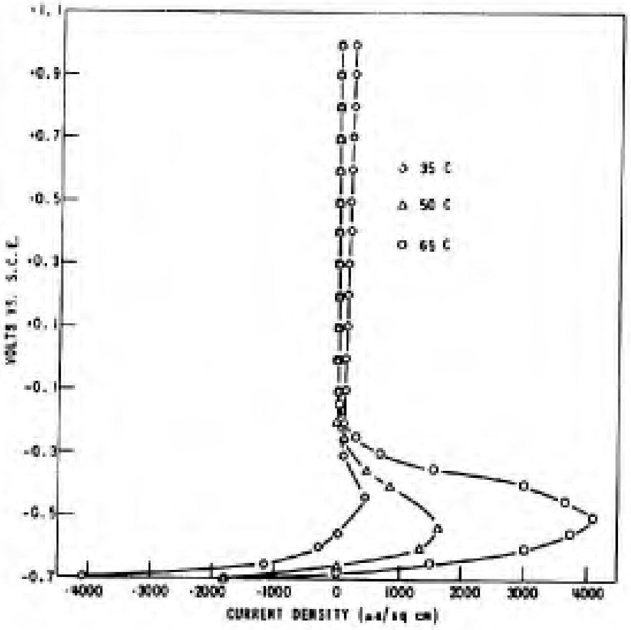

Figure 14. EFFECT OF TEMPERATURE ON POLARIZATION OF Ti-13V-11Cr-3Al IN 20% HCl

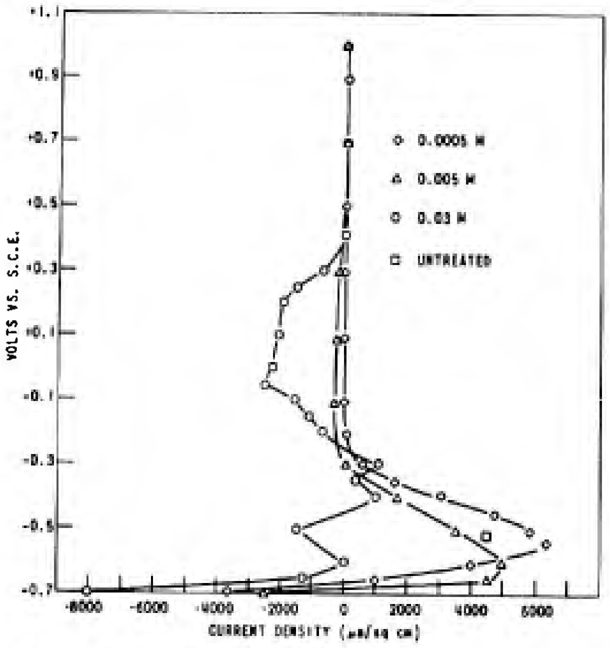

Figure 15. EFFECT OF $CuSO_4$ CONCENTRATION ON POLARIZATION OF Ti-13V-11Cr-3Al AT 65 C

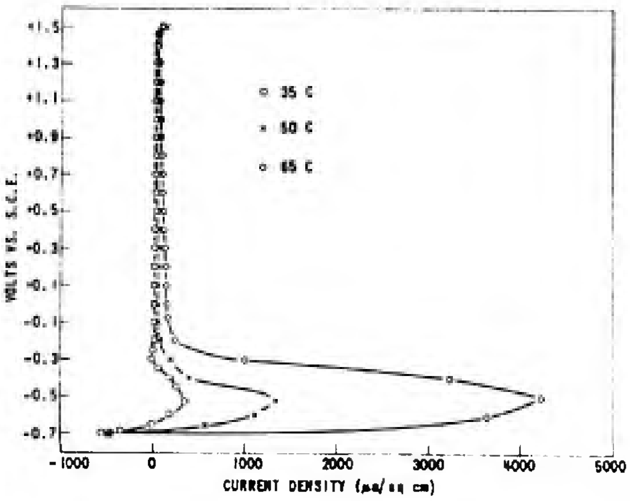

Figure 16. EFFECT OF TEMPERATURE ON POLARIZATION OF Ti-6Al-4V IN 20% HCl

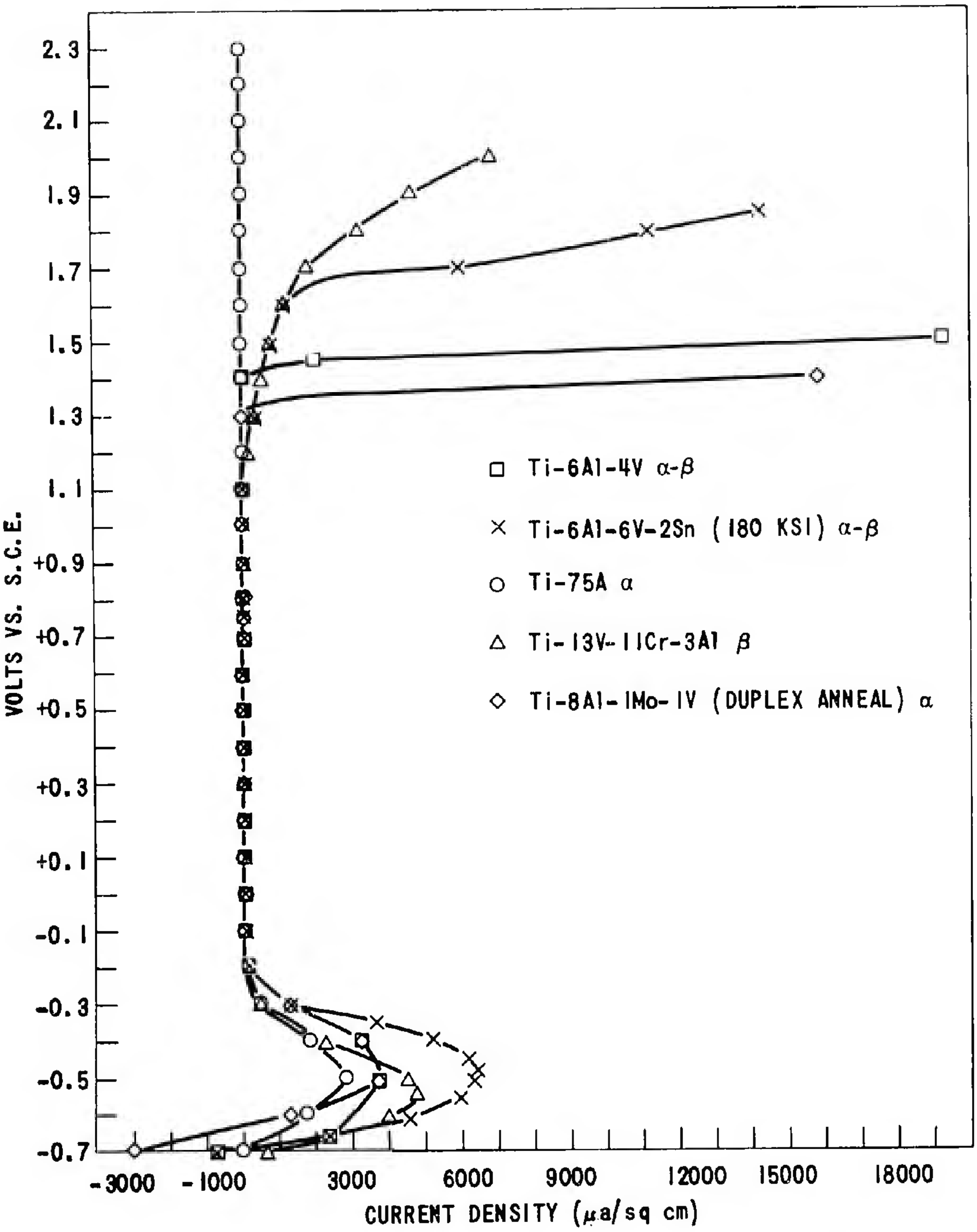

Figure 17. COMPARISON OF POLARIZATION BEHAVIOR OF TITANIUM AND ALLOYS IN 20% HCl AT 65 C

reaction for the Ti-13V-11Cr-3Al alloy (+1.1 v) is attributed to transpassivity. In this region of potential, chromium becomes oxidized to its hexavalent soluble state. The secondary current increase associated with the remaining alloys (Ti-6Al-6V-2Sn, Ti-6Al-4V, Ti-8Al-1Mo-1V) is attributed to localized corrosion or pitting. Comparing the maximum dissolution current densities (i_c) in HCl and H_2SO_4 shown in Table I, it is apparent that dissolution is doubled in HCl. It should be noted here that the pitting observed in HCl solutions was not observed in H_2SO_4 solutions.

Table I. COMPARISON OF MAXIMUM DISSOLUTION CURRENT DENSITIES IN H_2SO_4 AND HCl

Metal	i_c H_2SO_4	i_c HCl
6Al-6V-2Sn(180)	3220	6325
6Al-4V(L)	2170	4255
6Al-4V(T)	2040	3575
8Al-1Mo-1V(DL)	1940	3645
8Al-1Mo-1V(SL)	1910	3580
8Al-1Mo-1V(DT)	1830	4300
8Al-1Mo-1V(ST)	1850	3100
13V-11Cr-3Al	1795	4772
Comm. pure	1430	2863

SUMMARY

Commercially pure titanium and the alloys studied exhibited active-to-passive transitions in HCl. Increasing the acid concentration increased the critical current for passivity and shifted the critical potential for passivity in the noble direction. Increasing the temperature served only to increase the critical current for passivity. Activation energies for the anodic polarization process were the right order of magnitude for a reaction controlled by reactivity at the metal surface. For the alpha-beta alloy 6Al-6V-2Sn the critical current for passivity increased as the ratio of amounts of beta-to-alpha phase decreased with increasing strength level. The dissolution is accelerated, probably by the galvanic effects and the unfavorable area ratio. Ti-75A, a commercially pure metal, had a smaller critical current for passivity value than did the alloys, which was expected. Secondary current density increases were observed for all the alloys at potentials above +1.0 v. For the 13V-11Cr-3Al alloy such behavior was attributed to transpassivity. For the other alloys the phenomena was attributed to pitting or localized corrosion. The commercially pure metal remained passive in this potential range. The addition of Fe^{3+} and Cu^{2+} (0.03 *M*) to the HCl facilitated passivation of the metal and its alloys. The maximum dissolution current densities for the metal and alloys in HCl were double those in H_2SO_4. Pitting of several of the alloys occurred in HCl but not in H_2SO_4.

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13. ABSTRACT

Commercially pure titanium and the alloys studied exhibited active to passive transitions in HCl. Increasing the acid concentration increased the critical current for passivity and shifted the critical potential for passivity in the noble direction. Increasing the temperature served only to increase the critical current for passivity. Activation energies for the anodic polarization process were the right order of magnitude for a reaction controlled by reactivity at the metal surface. For the alpha-beta alloy 6Al-6V-2Sn the critical current for passivity increased as the ratio of amounts of beta-to-alpha phase decreased with increasing strength level. The dissolution is accelerated, probably by the galvanic effects and the unfavorable area ratio. Ti-75A, a commercially pure metal, had a smaller critical current for passivity value than did the alloys, which was expected. Secondary current density increases were observed for all the alloys at potentials above +1.0 v. For the 13V-11Cr-3Al alloy such behavior was attributed to transpassivity. For the other alloys the phenomena was attributed to pitting or localized corrosion. The commercially pure metal remained passive in this potential range. The addition of Fe^{3+} and Cu^{2+} (0.03 *M*) to the HCl facilitated passivation of the metal and its alloys. The maximum dissolution current densities for the metal and alloys in HCl were double those in H_2SO_4. Pitting of several of the alloys occurred in HCl but not in H_2SO_4. (Authors)

14 KEY WORDS	LINK A		LINK B		LINK C	
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13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS)*, *(S)*, *(C)*, or *(U)*.

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