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STRESS-CORROSION CRACKING OF TITANIUM AND Ti-Al ALLOYS IN METHANOL-IODINE SOLUTIONS

FIRST INTERIM REPORT
~~Special Technical Report No. 1~~

AMMRC CR 68.01/1

by

A. J. Sedriks, J.A.S. Green and P. W. Slattery

March 1968

Army Materials and Mechanics Research Center
Watertown, Massachusetts

Contract No. DAAG46-67-C-0138(X)

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ABSTRACT

Studies of the mechanical behavior of unalloyed titanium in methanol-halogen solutions have led to the suggestion that stress-corrosion cracking (SCC) in these environments occurs by localized anodic dissolution at the crack tip. The validity and generality of this model for SCC has been examined by means of comparative studies of the corrosion characteristics and susceptibility to SCC of pure titanium and Ti-Al binary alloys in methanol-iodine solutions.

Evidence is presented which indicates that two distinct SCC mechanisms may be operative, namely (i) stress accelerated intergranular corrosion involving dissolution of metal at a crack tip, and (ii) a transgranular cracking process which has the characteristics of mechanical failure. The characteristics of SCC failures of pure titanium and the Ti-2.1 w/o Al alloy appear to be consistent with the dissolution model. In the higher alloys (> 5 w/o Al), however, dissolution appears to be responsible only for the crack initiation stage, propagation involving transgranular cracking. Stress-corrosion failure in the higher alloys may therefore be prevented by inhibiting the initiation process via cathodic polarization or the introduction of passivators (i.e. water) into the testing environment.

To be presented at the International Conference on Titanium, London, May 21-24, 1968.

I. INTRODUCTION

The failure of unalloyed titanium by stress-corrosion cracking (SCC) has been observed in methanol solutions containing bromine (1), chlorine (2,3) or iodine (4), irrespective of whether the halogen was added as the sodium salt (3), acid (2), or the element in molecular form (1,4). These solutions also cause corrosion of unstressed titanium (5). Accordingly, it has been suggested that the SCC of unalloyed titanium occurs by localized anodic dissolution at the crack tip (1). If this view is correct then control of the anodic dissolution process should result in control of SCC. The purpose of the present paper is to examine the validity and generality of this dissolution model for SCC by means of comparative studies of the corrosion characteristics and susceptibility to SCC of pure titanium and Ti-Al binary alloys in methanol-iodine solutions. Evidence is presented which suggests that pure titanium and a Ti-2.09 w/o Al exhibit SCC behavior consistent with the dissolution hypothesis. Failures of alloys containing > 5 w/o Al, however, exhibit characteristics which cannot be explained solely in terms of a dissolution process, but which can be rationalized by postulating the occurrence of two separate mechanisms.

II. EXPERIMENTAL

The studies were confined to vacuum annealed pure titanium and Ti-Al binary alloys of composition and mechanical properties listed in Table I. Stress-corrosion tests were carried out using smooth (un-notched) tensile specimens (reduced section 0.5 × 0.045 × 0.090 in.) stressed under constant load. The methanol-iodine solutions were prepared using the highest purity chemicals available commercially and distilled water. The ratio of iodine to methanol was kept constant at 0.5% iodine by weight. (The water content of the methanol-iodine-water solutions is expressed as % by volume.)

Three types of electrochemical studies were conducted: (a) measurement of electrode potential of unstressed specimens, (b) galvanostatic polarization of stressed specimens during stress corrosion tests, and (c) potentiostatic polarization of unstressed materials to determine current/voltage variations. In cases (a) and (b), a high impedance Beckman pH meter was used to measure electrode potential. In the potentiostatic polarization studies the cell and electrode arrangements adopted were similar to those described by Greene (6), although the use of sheet specimen electrodes made it necessary to modify the electrode holders and to use masking materials. Polarization was carried out at a potential stepping rate of 50 mV per 2 min., using a Wenking potentiostat in conjunction with a saturated calomel electrode (SCE). For the potentiostatic polarization tests the metal surfaces were prepared by abrading the vacuum annealed material with fine emery paper, washing with water, and drying with ethanol. In all other studies, including weight loss determinations and stress-corrosion tests, the surfaces were 'as vacuum annealed'.

Table I

Composition and Mechanical Properties of the Titanium and Titanium Alloys.

		Pure titanium	Ti-2.09 Al alloy	Ti-5.1 Al alloy	Ti-7.76 Al alloy
	Al	-	2.09	5.10	7.76
	Fe	0.028	0.026	0.034	0.014
	N	0.0055	0.0053	0.0051	0.0056
Composition	O	0.0386	0.0422	0.0379	0.0378
(w/o)	C	0.018	0.032	0.030	0.030
	H	0.0014	0.0015	0.0010	0.0006
Hardness (HV 20 Kg)		126	188	290	319
U.T.S. (psi)		46,000	52,000	78,500	98,500
0.2% P.S. (psi)		28,500	40,000	69,000	90,000
Tensile Elong. on 2 in. (%)		37	24	14	23.5

III. RESULTS

A. Corrosion Behavior of Unstressed Specimens

1) Pure Titanium

Exposure of unstressed specimens to the methanol-iodine solution containing no added water resulted in corrosive attack of sufficient severity to be detectable by weight loss measurements. Metallographic examination revealed that the attack was largely intergranular; there was little evidence of attack within the grains. It can be seen in Fig. 1 that the addition of water to the methanol-iodine solution progressively decreased the corrosive attack, the weight loss as measured after a 96 hr. exposure, decreasing to zero at ~ 15% water. This decrease of weight loss was accompanied by a displacement of the one-hour electrode potential in the more noble direction, Fig. 1. In order to determine whether this displacement of potential was due to the effect of water on the anodic or the cathodic* reactions, potential-current curves were determined for methanol-iodine solutions containing 5% and 30% water. These curves showed that increasing the water content significantly reduced the anodic current density at any comparable potential, while the cathodic current was little affected, and below a potential of ~ -100 mV (SCE) the cathodic current densities actually coincided for the two solutions. These observations indicate that water limits the anodic reaction, $Ti = Ti^{n+} + ne$, by acting as a passivator in the methanol-iodine-water solutions.

2) Ti-Al Alloys

As in the case of the pure metal, corrosive attack in the methanol-iodine solution was largely intergranular. Increasing the aluminum content of the alloy caused a progressive increase in weight loss and a displacement of the one-hour electrode potential in the less noble direction. It should be noted, however, that these changes are not large, Fig. 2. Thus, the addition of 7.76 w/o aluminum resulted only in an increase in weight loss of ~ 13%, and a change of the one-hour electrode potential of ~ 50 mV (SCE).

B. Stress-Corrosion Behavior

1) Pure Titanium

The effect of applied stress on time to failure (t_f) of pure titanium in the methanol-iodine solution is shown in Fig. 3, where it is seen that increasing the applied stress reduced t_f. A comparison of the behavior of specimens subjected to simultaneous stressing and exposure to the environment with that of specimens exposed in the unstressed state and then stressed in the absence of the environment revealed that stress enhances the corrosive attack. For example, specimens stressed in the environment at ~ 70% of the yield stress of unexposed material failed in ~ 10,000 sec, while specimens given this time of exposure in the unstressed state and then tested in air could sustain indefinitely stresses of ~ 95% of the yield stress of unexposed material. Increasing the load to fracture showed that the 10,000 sec exposure resulted only in ~ 5% reduction in U.T.S.

The effect of the application of small anodic and cathodic currents on the t_f of specimens tested in methanol-iodine solution is shown in Fig. 4. It can be seen that t_f in this solution is reduced by the application of small anodic currents (e.g. 0.14 mA/sq.cm ≡ + 640 mV (SCE)), while failure is inhibited

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*Possible cathodic reactions are: (a) the reduction of iodine to iodide, (b) the reduction of water, (c) the reduction of dissolved oxygen, and (d) reduction of methanol with the liberation of hydrogen.

up to times of $> 10^5$ sec by the application of small cathodic currents (e.g. 0.14 mA/sq.cm ≡ -290 mV (SCE)). A similar variation of t_f with applied anodic and cathodic currents was observed in the methanol-iodine-5% water solution, Fig. 4.

Although the addition of small amounts of water (< 1%) caused an initial decrease in t_f, Fig. 1, larger additions resulted in a progressive increase in t_f, until at water contents of ~ 15% no cracks were formed. It is important to note that the water contents at which cracking is inhibited also correspond with those at which no weight loss due to corrosion can be detected in tests on unstressed specimens, and at which the electrode potential attains a uniform value, Fig. 1.

Metallographic and fractographic examination of failed specimens indicated that cracking was entirely intergranular.

2) Ti-Al Alloys

Increasing the aluminum content of the alloys was found to produce a marked reduction of t_f for specimens tested in the methanol-iodine solution, Fig. 3. A similar reduction in t_f with aluminum content was also observed in the methanol-iodine-5% water solution. As in the case of the pure metal, t_f was reduced by the application of anodic currents and inhibited by cathodic currents, Fig. 4. Increasing the water content of the solution resulted in increasing t_f, and at high water contents (> 15%) no failures were observed. The variation of t_f with water content of the methanol-iodine solution for the Ti-7.76 w/o Al alloy is shown in Fig. 1. The minimum in the t_f/water content variation evident for pure titanium was not detected in the alloy, Fig. 1.

Metallographic studies showed that the path of cracking was also influenced by the aluminum content. As noted previously, the stress-corrosion crack path in pure titanium was intergranular, and this was also the case in the Ti-2.09 w/o Al alloy. In the Ti-5.1 w/o Al alloy and the Ti-7.76 w/o Al alloy only the initial part of the crack was intergranular; the part adjacent to the final ductile fracture was "mixed" (i.e. both intergranular and transgranular) in the Ti-5.1 w/o Al alloy and transgranular in the Ti-7.76 w/o Al alloy, Fig. 5(a).

Replicas of the stress-corrosion fracture surface of the Ti-7.76 w/o alloy taken from the initial part of the stress-corrosion crack showed intergranular facets, indistinguishable from those produced by intergranular corrosion in the absence of stress Fig. 5(b). Replicas of the transgranular regions of the stress-corrosion crack exhibited markings similar to those found on surfaces of materials failed by cleavage, Fig. 5(c), A.

Examination of thin foils of pure titanium and the alloys by transmission electron microscopy confirmed the earlier observations by Blackburn and Beck (7) that increasing the aluminum content favored the tendency for dislocations to form co-planar arrays on deformation. In pure titanium and the Ti-2.09 w/o Al alloy the dislocation distribution was "tangled" or cellular after ~ 5% strain; in alloys containing higher aluminum contents the dislocations formed planar arrays, Fig. 5(d). The formation of such arrays is indicative of reduced propensity for cross-slip, and has been attributed to the presence of short-range order (7).

A comparison of the metallographic, fractographic and thin foil transmission data revealed that the aluminum contents at which transgranular crack propagation could be detected coincided with those at which the dislocations exhibited a pronounced tendency to form co-planar arrays on deformation.

IV. DISCUSSION

The present studies using pure titanium and methanol-iodine solutions have shown that: (i) anodic currents reduce t_f while cathodic currents inhibit failure, (ii) introduction of large amounts of water inhibit both corrosion and SCC, and (iii) both corrosive attack in unstressed specimens and SCC are intergranular. All of these observations support the view that the SCC of pure titanium in methanol-iodine solutions involves preferential anodic dissolution at the grain boundaries. This conclusion is also valid for the Ti-2.09 w/o Al alloy.

Stress has been observed to increase the rate of intergranular attack. However, it is premature to attempt to explain this increase until the mechanism of the intergranular corrosion process in the unstressed specimens is better understood. In particular, it would be important to know whether (a) at the tip of the intergranular corrosion crevice there exists a tendency for passive films to form which may reduce the extent of intergranular corrosion, or (b) the crevice tip is completely free of films and the environment is in contact with the bare metal. Clearly stress would have somewhat different roles in cases (a) and (b). Thus, for case (a), localized plastic flow would be expected to rupture any films that may form thereby providing a greater access of the environment to the metal. For case (b) it could be argued that plastic flow may increase the surface density of sites favorable for dissolution (8). In both cases the net effect would be an increase in the rate of dissolution.

With regard to the higher alloys, there is little doubt that the process by which stress-corrosion cracks initiate also involves preferential anodic dissolution. Thus, the initial part of the crack is intergranular, applied anodic currents reduce t_f (Fig. 4), and the introduction of large amounts of water inhibit SCC (Fig. 1). However, it is difficult to reconcile the features of the transgranular part of the failure with the anodic dissolution hypothesis since the presence of cleavage markings on the transgranular fracture surfaces indicates that such failures are mechanical. Thus, if it is accepted that the intergranular cracks result from anodic dissolution, then the presence of the cleavage markings on the transgranular surfaces indicates that a change of mechanism occurs. End

Similar transgranular failures have been observed in titanium alloys tested in aqueous chloride solutions (9,10) and in methanol-2% water solutions (11) saturated with sodium chloride. In these cases, failure requires the presence of a notch; failure does not occur in "smooth" specimens. Furthermore, Cavallaro (12) has shown that this type of cracking occurs in Ti-Al binary alloys in aqueous chloride only when the aluminum content is greater than ~ 5 w/o Al. Thus in the present system, it is possible that the initial stage of intergranular SCC in the higher alloys provides a notch of sufficient depth and acuity to cause the onset of the transgranular failure. If it is assumed that the transgranular process is more rapid than the intergranular one, then this will provide an explanation for the low values of t_f for the higher alloys, which cannot be accounted for by the slightly lowered corrosion resistance due to alloying.

The mechanism by which transgranular cracking occurs remains to be established, although thin foil studies of the deformation characteristics suggest that its occurrence is associated with a reduced propensity for cross slip.

Another aspect that requires clarification is the significance of the minimum in the plot of t_f versus water content variation at lower water contents for specimens of pure titanium, Fig. 1. Similar minima have been reported by Haney et al. (3) for various commercial titanium alloys tested in methanol-water solutions containing small amounts of NaCl or HCl. A possible explanation is that the factors giving rise to the minimum are factors which lead to localization of the corrosive attack. There is evidence that increasing the water content not only reduces the extent of overall corrosive attack but also localizes the attack. For instance, Tomashov et al. (5) have shown that in methanol-2% bromine-water solutions the tendency toward intergranular corrosion of unalloyed titanium is maximized at 5% water, and that this maximum is not reflected in the overall weight loss variation.

V. CONCLUSIONS

The observations reported in this paper indicate that two distinct SCC mechanisms may be operative in Ti-Al alloys in methanol-iodine solutions, namely (i) stress accelerated intergranular corrosion involving dissolution of metal at a crack tip, and (ii) a transgranular cracking process which has the characteristics of mechanical failure. The characteristics of SCC failures of pure titanium and the Ti-2.09 w/o Al alloy appear to be consistent with the occurrence of the dissolution mechanism. In the higher alloys (> 5 w/o Al) the dissolution process may be responsible only for the crack initiation stage, propagation involving transgranular cracking. Nevertheless, failures in the higher alloys may be prevented by inhibiting the initiation process by cathodic polarization or the introduction of passivators (i.e. water) in the solution.

ACKNOWLEDGEMENTS

The authors wish to thank Dr. E. N. Pugh and Dr. A.R.C. Westwood for helpful and stimulating discussions. The financial support received from the U. S. Army Materials and Mechanics Research Center, Watertown under Contract DAAG46-67-C-0138(X) is gratefully acknowledged.

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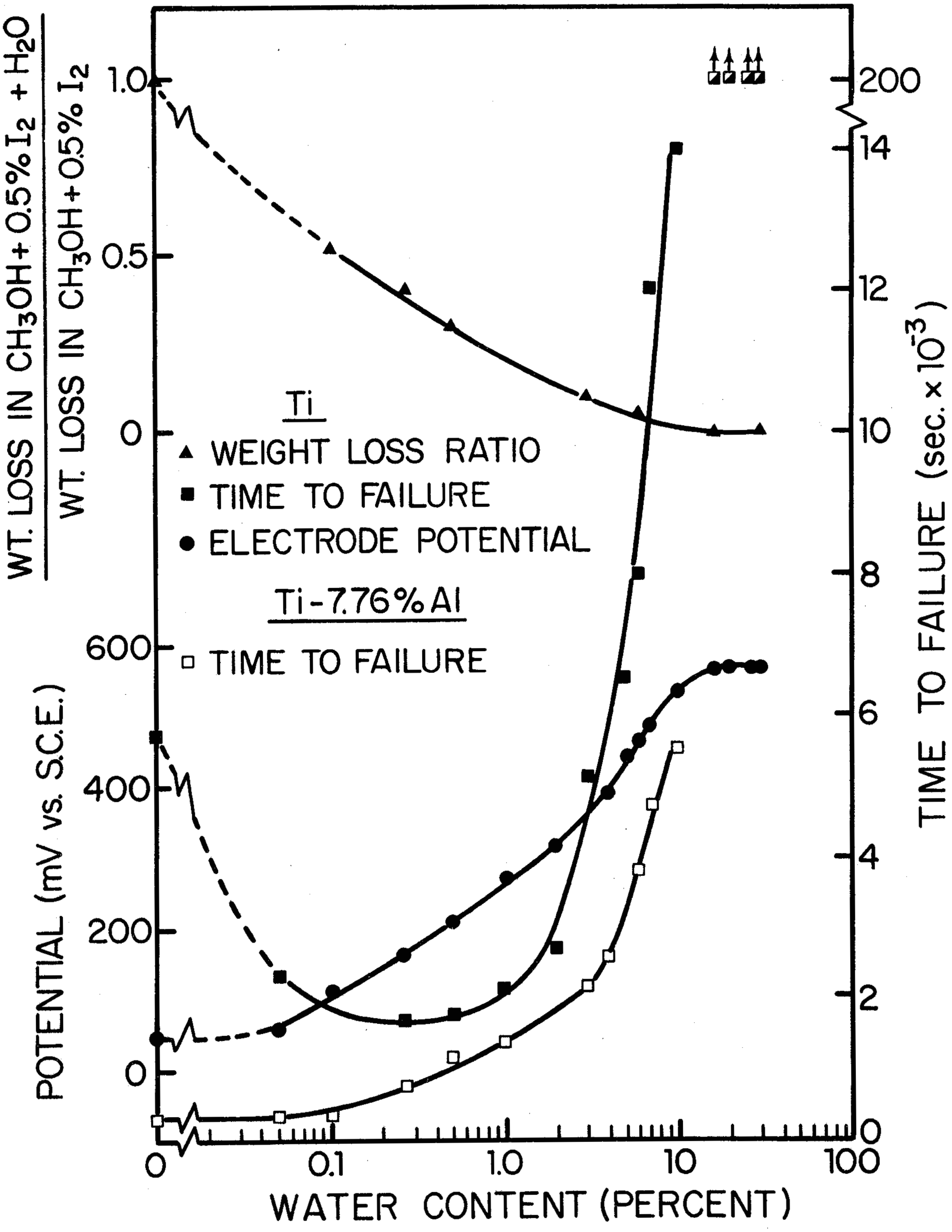

Fig. 1. Variation of weight loss ratio, electrode potential and t_f (95% Y.S.) of pure titanium, and t_f (90% Y.S.) of Ti-7.76 w/o Al, with increasing water content of methanol-iodine solution.

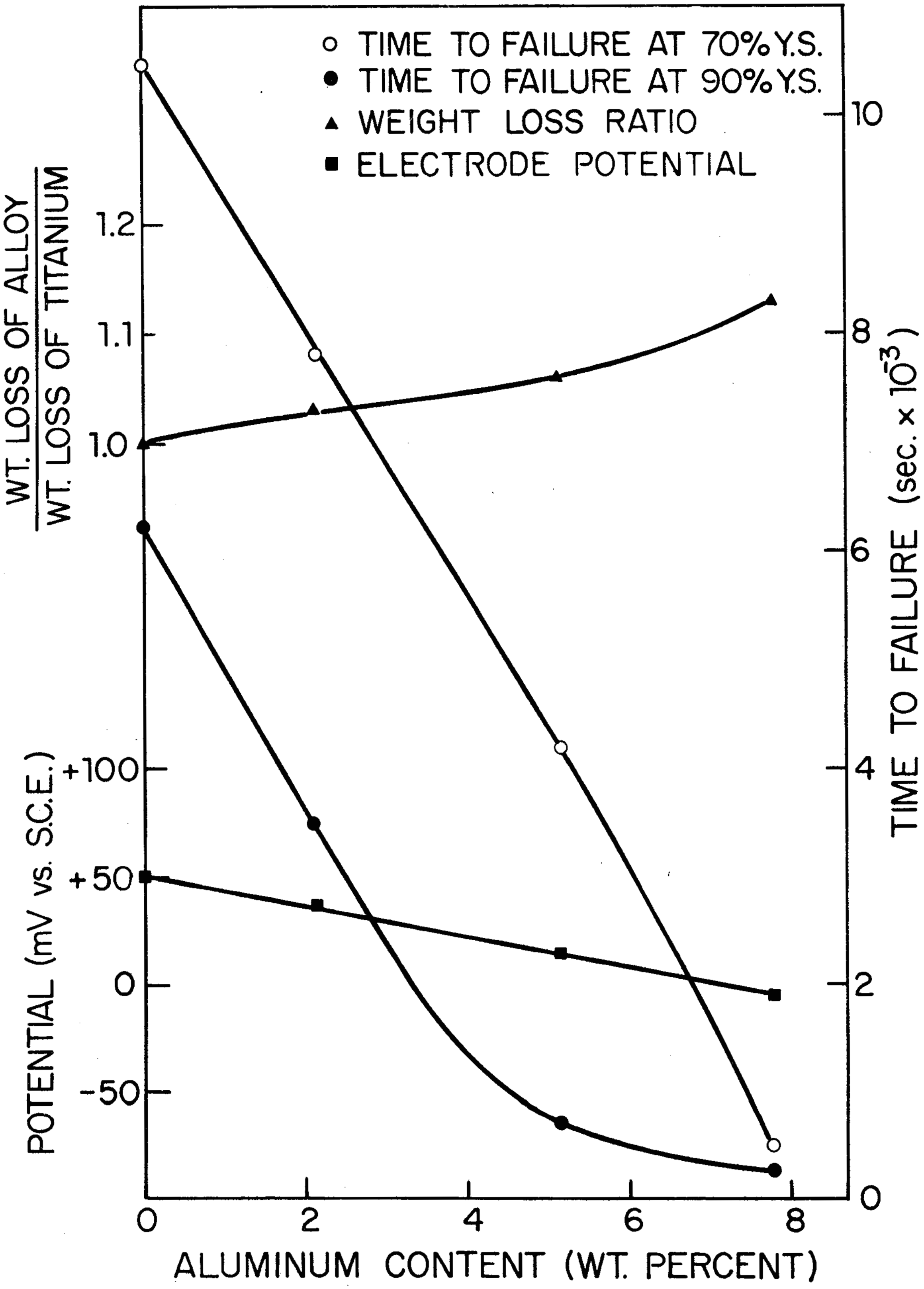

Fig. 2. Effect of aluminum content on weight loss (96 hr. exposure), electrode potential, and t_f in methanol-iodine solution containing no added water.

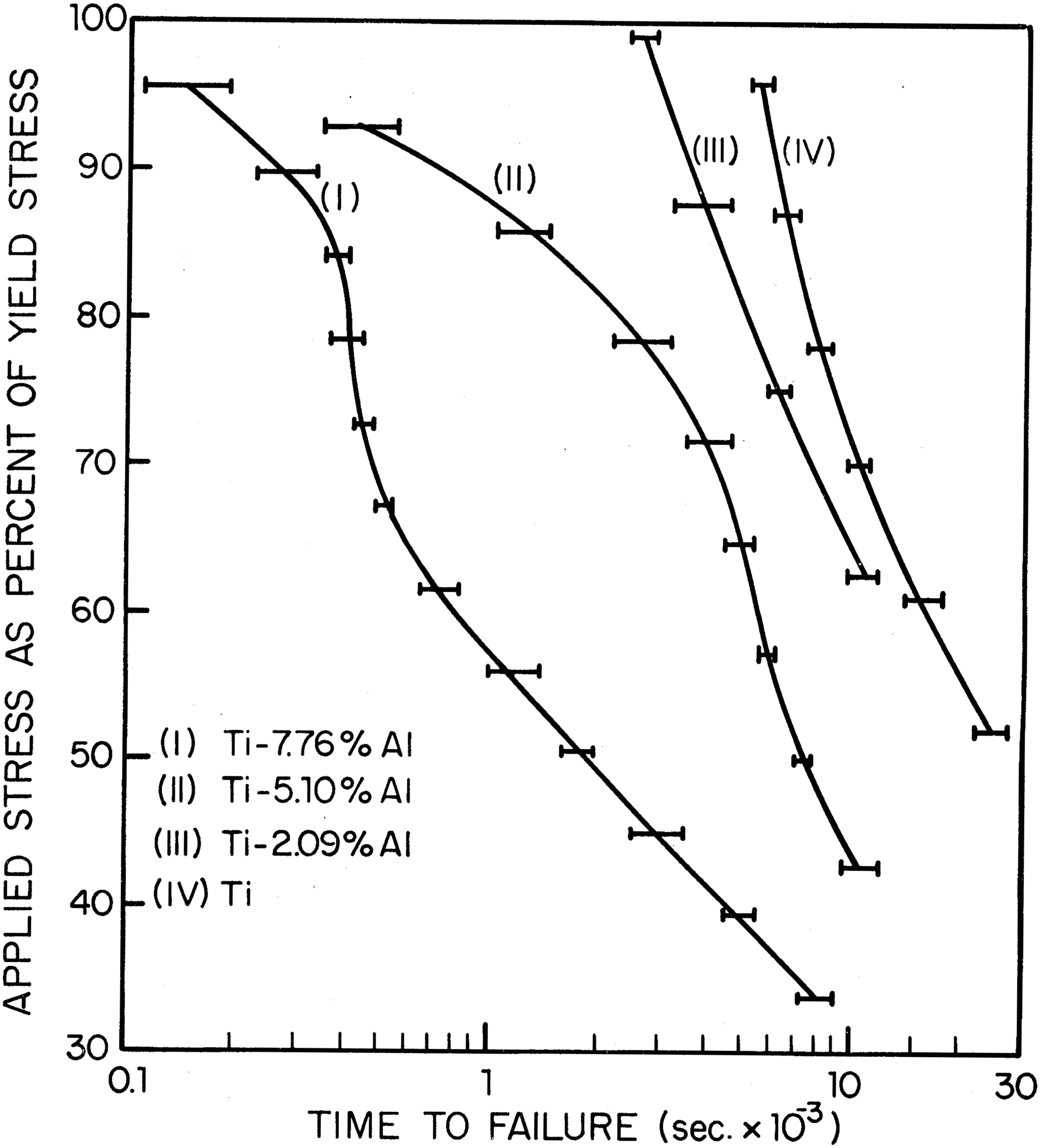

Fig. 3. Effect of applied stress (engineering) on t_f of titanium and Ti-Al binary alloys in methanol-iodine solution containing no added water.

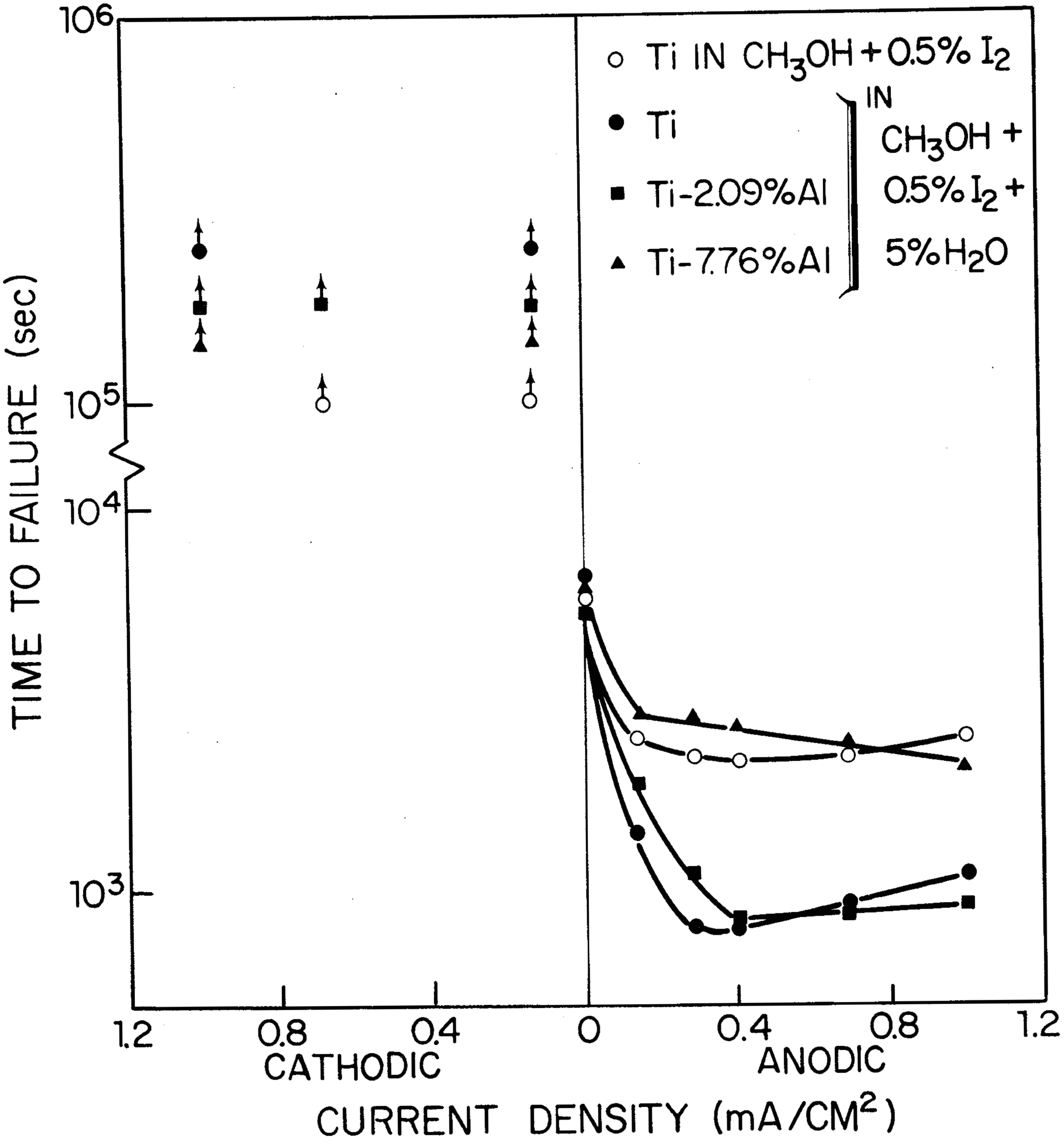

Fig. 4. Effect of applied anodic and cathodic currents on t_f of titanium and Ti-Al binary alloys in methanol-iodine and methanol-iodine-water solutions. Applied stress:- Ti ≡ 95% Y.S., Ti-2.09 w/o Al ≡ 90% Y.S., Ti-7.76 w/o Al ≡ 85% Y.S.

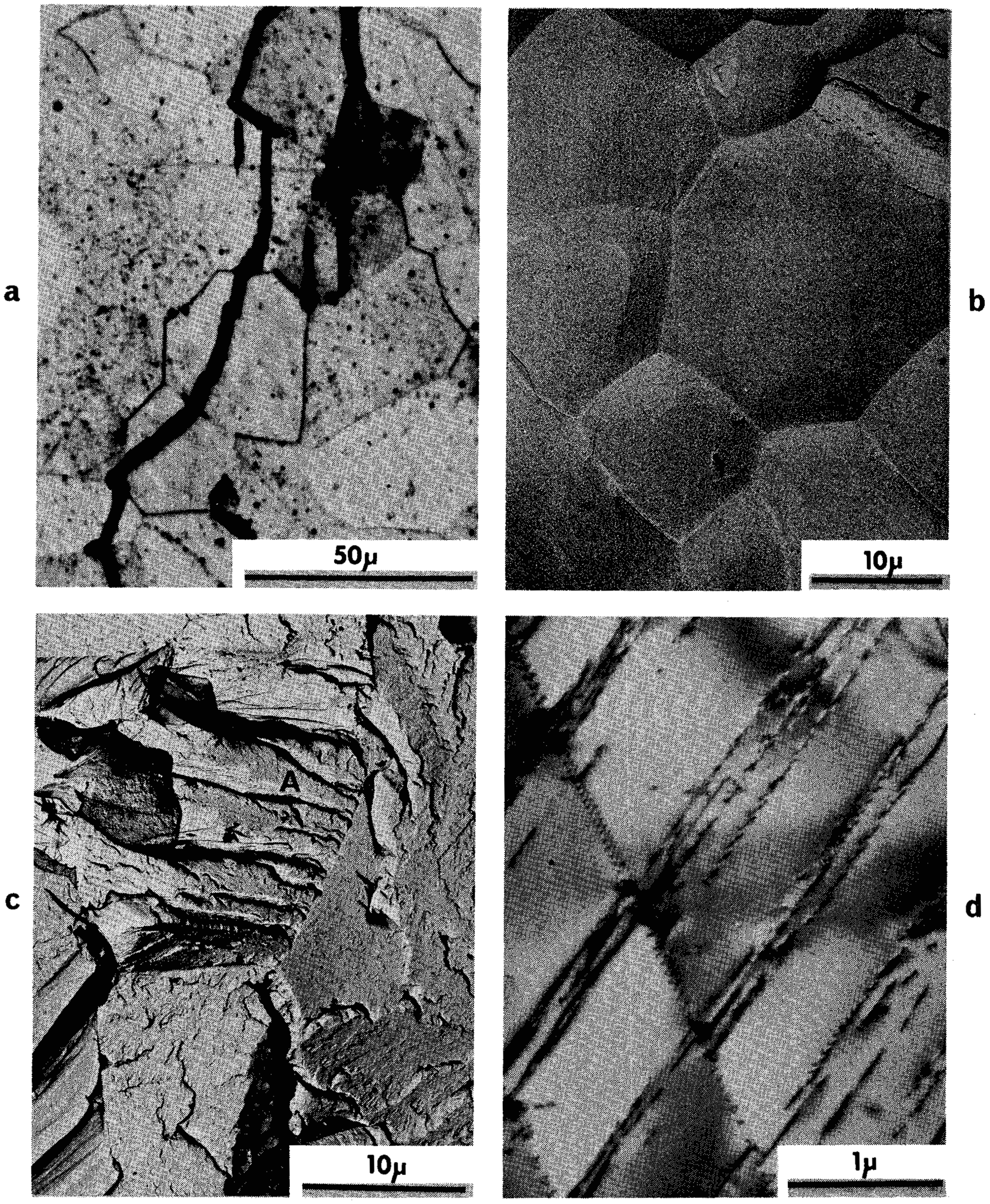

Fig. 5. Ti-7.76 w/o Al alloy. (a) section of transgranular crack, (b) replica of intergranular facets produced by corrosion in the absence of stress, (c) replica of surface of transgranular part of stress corrosion crack, (d) transmission electron micrograph of alloy after $\sim$ 5% deformation.

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Research Institute for Advanced Studies (RIAS)
Martin Marietta Corporation
1450 S. Rolling Road, Baltimore, Md. 21227

2a. REPORT SECURITY CLASSIFICATION
UNCLASSIFIED

2b. GROUP
N/A

3. REPORT TITLE
Stress-Corrosion Cracking of Titanium and Ti-Al Alloys in Methanol-Iodine Solutions.

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Technical Report No. 1, March 1968

5. AUTHOR(S) *(First name, middle initial, last name)*
A. J. Sedriks, J.A.S. Green and P. W. Slattery

6. REPORT DATE
March 1968

7a. TOTAL NO. OF PAGES
24

7b. NO. OF REFS
12

8a. CONTRACT OR GRANT NO.
DAAG-46-67-C-0138(X)

b. PROJECT NO.

c.

d.

9a. ORIGINATOR'S REPORT NUMBER(S)
RIAS TR-68-4c

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

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13. ABSTRACT

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2. Stress-corrosion cracking						
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