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PRELIMINARY MEASUREMENTS OF THE ENVIRONMENTAL CRACKING BEHAVIOUR OF TITANIUM ALLOY 721

G. J. BIEFER AND J. G. GARRISON

PHYSICAL METALLURGY DIVISION

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by

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

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ABSTRACT

In order to test the functioning of equipment, and to gain some initial experience concerning the environmental cracking susceptibility of Ti-7%Al-2%Nb-1%Ta (Ti-721) alloy, some tests were carried out on notched, precracked specimens loaded as cantilevers.

In tests performed in 3.5% NaCl solution, it was confirmed that, subsequent to a sensitizing heat treatment designed to produce Ti_3Al (8 hr at 650°C (1202°F)), alloy Ti-721 is extremely susceptible to environmental cracking. It is somewhat more resistant in the as-rolled condition.

Environmental cracking tests in which specimens were broken under a gradually rising load were found to give very nearly the same results as did time-to-failure tests under a constant load. It appeared, therefore, that the rising-load method was sufficiently accurate for screening tests of alloys resembling Ti-721, though constant load tests might sometimes be needed for a more precise determination of the critical stress-intensity threshold for cracking, K_{ISCC}.

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*Head, Corrosion Section, and **Technician, Physical Metallurgy Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

Direction des mines

Bulletin technique TB 132

MESURES PRÉLIMINAIRES DU COMPORTEMENT DE L'ALLIAGE DE TITANE 721 À LA FISSURATION DUE À L'ENVIRONNEMENT

par

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

Résumé

En vue d'essayer le fonctionnement du materiel et d'acquérir une certaine expérience initiale concernant la susceptibilité à la fissuration due à l'environnement que présente l'alliage Ti-7%Al-2%Nb-1%Ta (Ti-721), les auteurs ont procédé à des essais sur des éprouvettes entaillées, présentant une amorce de fissure et soumises à une charge en porte-à-faux.

Les essais effectués dans une solution à 3.5% de NaCl ont confirmé que, après un traitement thermique de sensibilisation destiné à former extrêmement susceptible à la fissuration due à l'environnement. À l'état brut, sortie de laminoir, sa résistance est quelque peu supérieure.

Les essais de fissuration due à l'environnement, au cours desquels les éprouvettes ont été amenées à la rupture par augmentation progressive de la charge, ont donné sensiblement les mêmes résultats que ceux où l'on a mesuré le temps écoulé jusq'à la rupture d'une éprouvette soumise à une charge constante. Il semblait donc que la méthode par augmentation progressive de la charge était suffisamment précise pour des essais de réception d'alliages ressemblant au Ti-721; il peut cependant s'avérer nécessaire de recourir, dans certaines circonstances, à des essais sous charge constante, si l'on veut obtenir une détermination plus précise du coéfficient d'intensité de contrainte critique relatif à la fissuration, K_{ISCC}.

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* Chef, Section de la corrosion, et Technicien, Division de la métallurgie physique, Direction des mines, ministère de l'Énergie, des Mines et des Ressources, Ottawa, Canada.

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INTRODUCTION

Titanium alloys have some obvious advantages for use in sea-water - e.g., excellent corrosion resistance in both stagnant and rapidly flowing sea-water, good resistance to damaging cavitating conditions, good corrosion fatigue properties, and high strength/weight ratios. However, as first demonstrated by Brown[1], some high-strength titanium alloys are extremely susceptible to environmental cracking* in sea-water or in neutral salt solutions if the test specimens contain pre-existing flaws, such as sharp notches or fatigue cracks. In susceptible alloys, the cracking propagates rapidly from the flaw, provided the stress intensity is sufficient.

It is realistic to assume that any complex structure, no matter how carefully fabricated and inspected, will either contain flaws before use or will develop them during service. This susceptibility to environmental cracking is, therefore, serious because it has helped to eliminate some high-strength titanium alloys from consideration for demanding marine applications, e.g., in deep-diving submarines and in hydrofoil craft.

Obviously, if the susceptibility of high-strength titanium alloys to environmental cracking could be eliminated - and the other good properties retained - this would represent an important technological advance. It appears, in fact, that some resistant high-strength alloys are now being produced[2] - e.g., Ti-6%Al-2%Nb-1%Ta-1%Mo (Ti-6211), a replacement of the highly sensitive Ti-7%Al-2%Nb-1%Ta (Ti-721).

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*In this report, "environmental cracking" is used as a general term, without any implications as to mechanism, to describe the brittle cracking which occurs in susceptible materials under tensile stress in the presence of a suitable corrodent. The term, therefore, includes the phenomena sometimes referred to as "stress-corrosion cracking" and "hydrogen-embrittlement cracking".

However, it has not been explained satisfactorily why alloys such as Ti-721 are susceptible to environmental cracking. Because increased knowledge could lead to the development of new and improved high-strength titanium alloys, it has been decided to initiate research in this area at the Physical Metallurgy Division (PMD) of the Mines Branch at Ottawa.

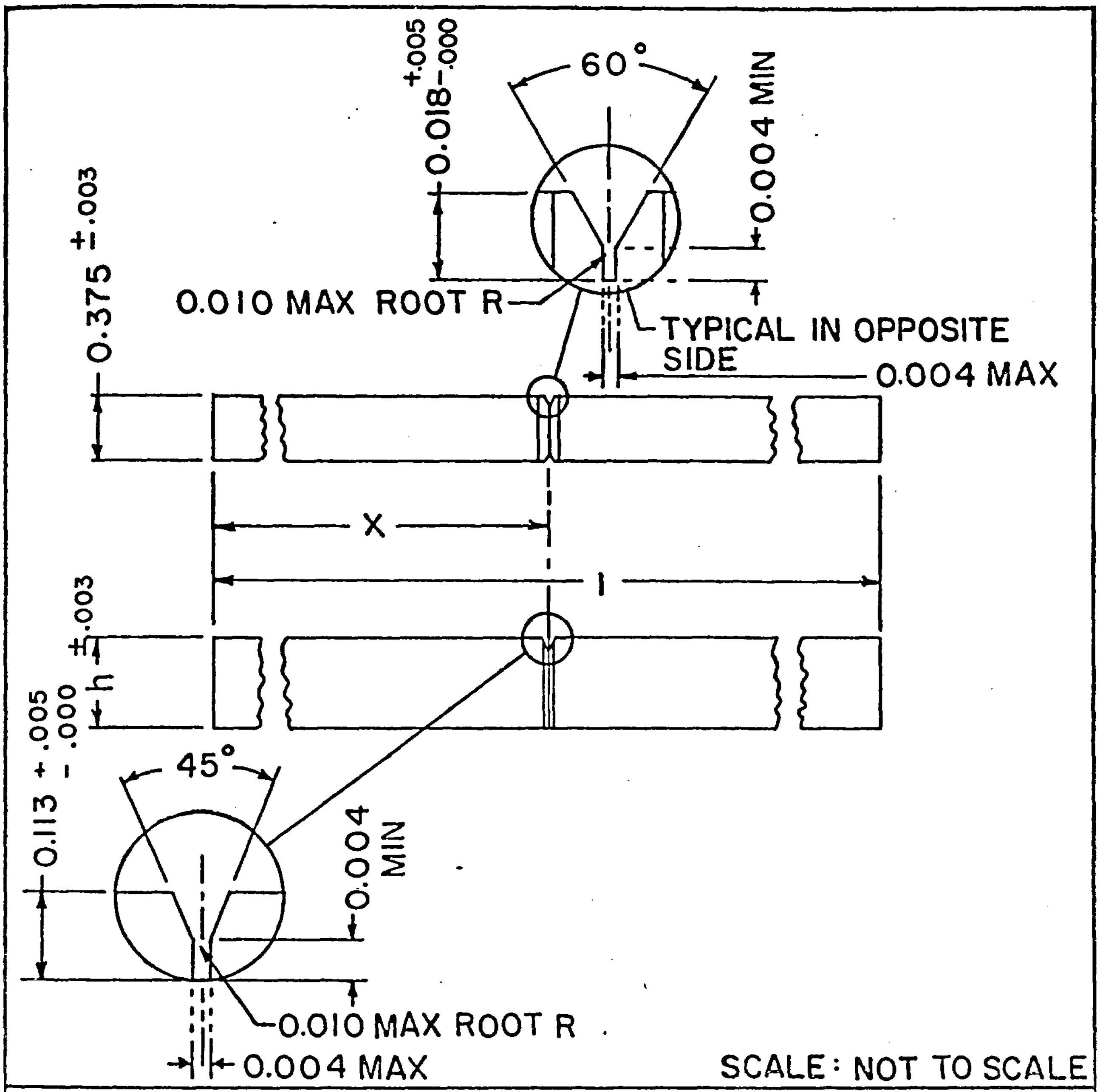
This report presents the first results obtained in developing suitable methods for measuring the susceptibility of titanium alloys to environmental cracking, using the crack-sensitive Ti-721 alloy. These methods will then be available to assess a number of experimental alloys having compositions resembling that of Ti-721.

EXPERIMENTAL

Methods

It was decided to evaluate susceptibility to cracking in salt solution using the Cantilever Stress-Corrosion Cracking Test developed by Brown at the U.S.N, Research Laboratory(1). This test had been used previously at the PMD to study the environmental cracking behaviour of some high-strength steels(3), and the same equipment and methods could be used for the titanium alloys with only minor changes.

The specimens used in the Cantilever Test are bars, usually about 6 in. long, cut from plate. These specimens are cut so that the length runs in the rolling direction of the plate. The specimen depth ("h" in Figure 1) corresponds to the plate thickness, which was 1/2 in. and 1 in. in this work. The bars are notched on their top surfaces, and also on each of their side surfaces, as shown in Figure 1. It will be noted that the side notches are shallower than those in specimens used previously(3); this conforms with Brown's more recent recommendations(4). However, some of the results presented in this report were obtained on specimens made according to the drawing used previously.



Ti ENVIRONMENTAL CRACKING TEST BAR

REMARKS:
DIMENSIONS h, l & X TO BE SPECIFIED.
GENERAL FINISH 63 MICROINCHES
NO SURFACE TO BE GROUND
NOTCH CENTRE-LINES ON EACH FACE TO BE WITHIN 0.001 OF EACH OTHER.

P.M.D. 568 BOOTH ST. OTTAWA

DRAWN BY: A.KAM
CHECKED BY: H.J.OWEN

MATERIAL:
AS STATED ON WORK ORDER

DWG NO. 122

MODIFIED: A.J.WILLIAMS

Figure 1. Cantilever test bar, revised in accordance with recommendations of Reference 4.

Prior to testing, precracking of the bars at the root of the top notch was done by fatiguing each specimen in a Krouse Plate Fatigue Testing Machine at a nominal stress of 20,000 to 30,000 psi for 10,000 to 30,000 cycles. The most usual conditions were 20,000 psi stress for 30,000 cycles, i.e., 17 minutes on the Krouse tester.

In the Cantilever Stress-Corrosion Cracking Test, the bar specimen is clamped at one end to a vertical post and at the other end to a cantilever beam, to the end of which a weight may be suspended. This arrangement applies tensile stress to the upper (notched and precracked) surface of the specimen. The notched area may be surrounded, when desired, by a plastic vessel containing a corrodent. In our tests, 3.5% NaCl solution was used and replenished at the rate of 4 litres per day. Also, if desired, specimens may be polarized to some preselected potential. In our work, this was done by immersing a sacrificial anode of zinc in the plastic vessel and connecting it externally to the test specimen.

Figure 2 shows the apparatus used in this work. In general, this is the same equipment as used previously[3] except that provision has been made for gradually increasing the load on the specimen by adding water at a constant rate to a plastic container hanging from the end of the cantilever beam. When the load has become sufficient to break the specimen, the weight of the fallen loading vessel operates a micro-switch which shuts off the dripping water and also stops a timer. One of the tests was carried out in a similar apparatus which was equipped so that both specimen potential and cantilever beam deflection could be recorded continuously. Additional details of the experimental methods used appear in Reference 5.

"Initial" stress intensity values were calculated, as before, using the same equation as B. F. Brown,

$$K_{Ii} = \left(\frac{B}{B_N}\right)^{\frac{1}{2}} \frac{\beta M}{BD^{3/2}}, \quad \ldots \quad (1)$$

where K_{Ii} is the nominal stress intensity parameter, based on the initial crack geometry; and β is the function of a/D, as shown below, where a is the crack depth, including the notch, and D is the specimen depth.

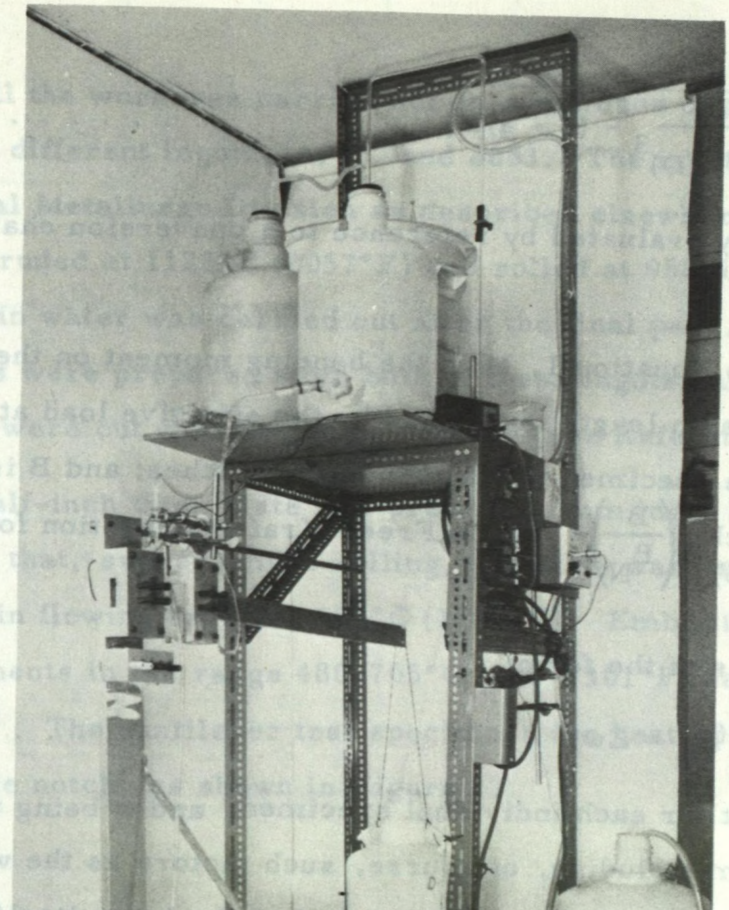


Figure 2. Cantilever test equipment used in the rising-load tests.

$$\beta = 4.12\sqrt{\frac{1}{(1 - a/D)^3} - (1 - a/D)^3} \qquad \ldots\ldots\ldots \quad (2)$$

β may be conveniently evaluated by reference to a conversion chart in Reference 5.

Returning to Equation 1, M is the bending moment on the notch, i.e., the cantilever arm length multiplied by the effective load at the end of the arm; B_N is the specimen width at the side notches; and B is the width at other than the notch. $\left(\frac{B}{B_N}\right)^{\frac{1}{2}}$ is the Freed-Krafft correction for the effect of the side notching[6].

Equation 1 is of the form:

$$K_{Ii} = Zw, \qquad \ldots\ldots\ldots \quad (3)$$

with Z being constant for each individual specimen, and w being the total weight at the end of the beam including, of course, such factors as the weight of the empty container, of its support straps, and of the beam itself.

In the present work, following Reference 5, we have used the symbol K^*_{Ii} to represent nominal stress intensities, at specimen fracture, obtained using a steadily increasing load, to differentiate them from the K_{Ii} values calculated in constant load tests.

In the rising-load tests we have also calculated the rate at which the stress intensity on a specimen is increased, using the formula:

$$\text{Average Loading Rate} = \frac{K^*_{Ii} - K_{Ii}}{t} \qquad \ldots\ldots \quad (4)$$

Here, t is the time in minutes during which the rising load is applied, while K_{Ii} is the stress intensity just prior to the application of the increasing load, and results from such factors as beam weight, empty container weight, etc.

Materials

All the work was carried out on specimens of alloy Ti-721 cut from three different ingots, A, B, and 2053. The ingots were prepared at the Physical Metallurgy Division as described elsewhere[7]. Heats A and B were extruded at 1125°C (2057°F) and rolled at 950-1000°C (1742-1832°F). Quenching in water was carried out after the final pass. Half-inch and 1-in. thick plates were prepared from both of these ingots, and the cantilever specimens were cut with deep side notches (see Reference 3).

Half-inch thick plate was prepared from ingot 2053 in the same way except that, subsequent to rolling, the plate was "sensitized" by heating it for 8 hr in flowing argon at 650°C (1202°F). Embrittlement of Ti-721 by heat treatments in the range 480-705°C (896-1301°F) has previously been reported[8]. The cantilever test specimens for heat 2053 were cut with a shallow side notch, as shown in Figure 1.

RESULTS

Specimens from the as-rolled plate of heats A and B were subjected to rising-load tests, mostly in the dry condition. The results obtained are shown in Table 1. It is of interest that specimens from 1/2-in. plate give consistently lower K^*_{Ii} values than do specimens from 1-in. plate. It is also noteworthy that a considerable lowering of K^*_{Ii} occurs when the specimen is broken in contact with 3.5% NaCl solution.

In the single rising-load test in which specimen potential and deflection were monitored continuously (Specimen B-9), it was observed that the potential became more negative and more irregular just prior to a noticeable increase in beam deflection rate; also, that potentials became even more negative just prior to break, reaching a minimum of -0.85 volt S.C.E. (Figure 3). These results supported the suggestion that titanium alloys exhibit environmental cracking because of a loss of passivity in a

TABLE 1

Results of Rising-Load Cantilever Tests on As-Rolled Ti-721

Heat	Specimen No.	Nominal Depth, in.	Environment	Geometrical Factors, Z, psi $\sqrt{in.}$ P^{-1}	Initial Stress Intensity, K^o_{Ii}, kpsi$\sqrt{in.}$	Time to Failure, t	Average Loading Rate, psi$\sqrt{in.}$ min^{-1}	Stress Intensity at Failure, K^*_{Ii} kpsi$\sqrt{in.}$
A	1	1	Dry	427	13.1	30.5 min	2580	92
	2	1	Dry	439	13.45	31.4 min	2820	102
	5	$\frac{1}{2}$	Dry	1900	21.2	21.7 min	2730	80.5
	6	$\frac{1}{2}$	Dry	1835	21.6	26.05 min	2540	87.8
B	3	1	Dry	446	13.7	28.0 min	2700	89.2
	4	1	Dry	428	13.17	32.1 min	2520	94
	7	$\frac{1}{2}$	Dry	3310	38.8	7.95 min	4450	74.2
	8	$\frac{1}{2}$	3.5% NaCl	3310	21.1	42.3 hr	7.8	40.9
	9	$\frac{1}{2}$	3.5% NaCl	3070	18.5	17.8 hr	33.4	35.6

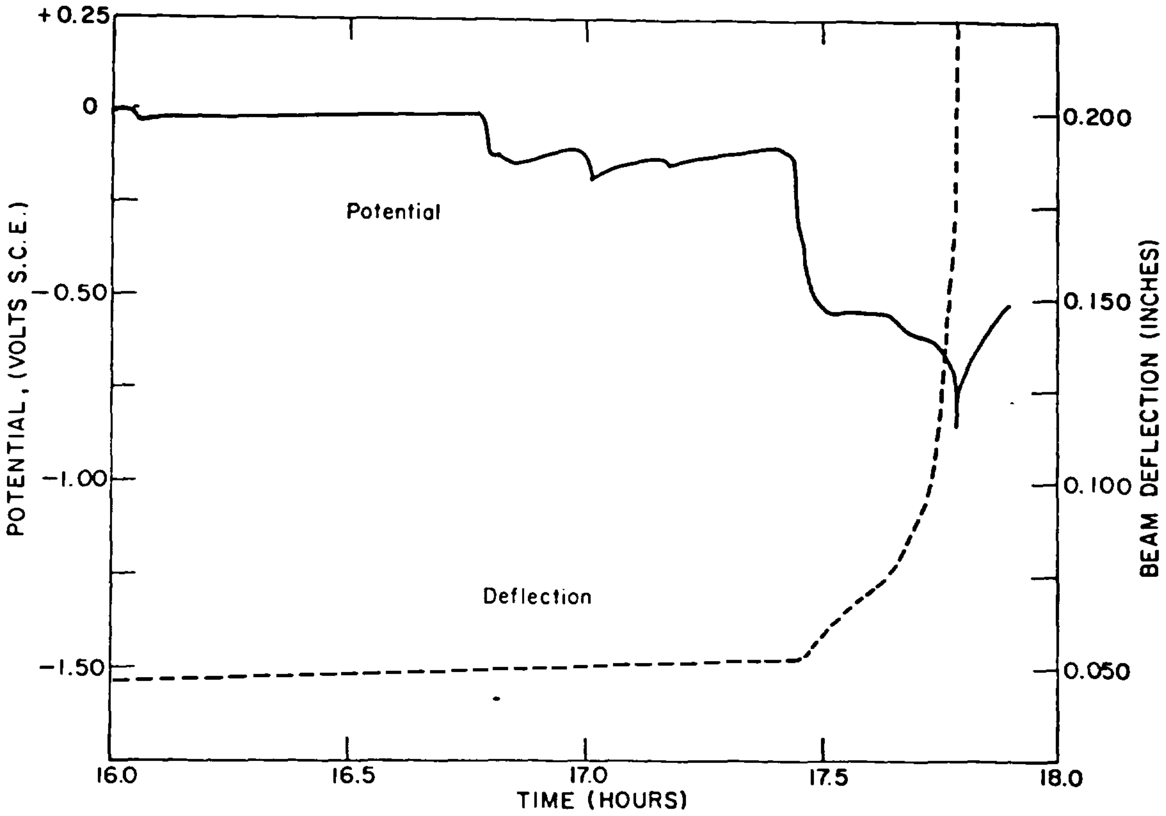


Figure 3. Changes of potential and beam deflection upon failure of Specimen B-9.

TABLE 2

Results of Rising-Load and Constant-Load Cantilever Tests on Sensitized Ti-721

Heat	Specimen No.	Environment	Geometrical Factors, Z, psi$\sqrt{in.}$ p^{-1}	Initial Stress Intensity, K_{Ii}^{o}, kpsi$\sqrt{in.}$	Time to Failure, t	Average Loading Rate, psi$\sqrt{in.}$ min^{-1}	Stress Intensity at Failure, K_{Ii}^{*}, kpsi $\sqrt{in.}$
2053	1-1	Dry	1536	17.27	22.3 min	2610	75.5
	1-2	Dry	1560	17.52	21.1 min	2660	73.7
	1-3	3.5% NaCl	1605	18.03	21.5 hr	8.8	29.2
	4-11	3.5% NaCl	1732	11.24	32.05 hr	7.11	24.9
	4-10	3.5% NaCl coupled to zinc	1645	10.65	88.4 hr	6.55	45.5
	2-4	3.5% NaCl	1611	32.6	6.7 min	0	32.5
	2-5	3.5% NaCl	1695	27.4	>168 hr	0	27.4*
	2-6	3.5% NaCl	1665	29.8	32 min	0	29.8

protective film. They showed the same trends as were observed by S. Tudor for Ti-721 alloy in tests similar to ours[9].

The results obtained for the sensitized specimens from heat 2053, in both rising-load and constant-load tests, are presented in Table 2. In these tests, the specimens broken in air show values of K_{Ii}^{*} which tend to be slightly lower than those for the 1/2-in. plate of heat B. When tests were carried out with the specimen notch area enclosed by 3.5% NaCl, there was a considerable decrease of K_{Ii}^{*}, to values lower than those obtained for the as-rolled 1/2-in. plate of heat B in 3.5% NaCl. Polarization to the potential of zinc caused an increase of K_{Ii}^{*}, though not to the dry level. This beneficial effect of cathodic polarization has been reported previously[10,11], and it appears that polarization to still more negative potentials would have brought about an additional increase in resistance to environmental crack propagation, i.e., still higher K_{Ii}^{*} values.

The results of three constant-load tests are also presented in Table 2. It is seen that two specimens broke very rapidly, whereas the third, at a slightly lower K_{Ii}, had not broken after one week. A slight increase in load then caused this specimen to fracture in a few minutes.

An examination of the fracture faces of the specimens broken under constant load (Figure 4) shows that cracking must have proceeded very rapidly. For example, specimen 2-4, which cracked after only 6.7 min on test, shows a roughened band of environmental cracking about 5 mm wide, i.e., cracking proceeded at at least 0.75 mm/min.

All the specimens broken while freely corroding (potential of about -0.03 volt, S.C.E.) showed one or more cracks proceeding into the specimen at an angle to the plane of the notches, somewhat similar to the angled cracks that were observed previously in the cantilever tests on specimens of 18% Ni (250) maraging steel[5]. Interestingly, such angled cracks were not apparent in the single specimen that was broken while coupled to zinc (potential about -1.06 volts, S.C.E.).

DISCUSSION

According to B. F. Brown, there is for every high-strength metal a characteristic critical stress-intensity level, which he has termed K_{ISCC}, above which environmental cracking can be definitely expected to occur. However, at or below K_{ISCC}, it cannot be established positively that cracking will not occur. The K_{ISCC} level, then, resembles the fatigue limit in fatigue testing.

In the present series of tests on freely corroding specimens of sensitized Ti-721 in 3.5% NaCl solution, the rising-load tests indicated that K_{ISCC} lay somewhat below the K_{Ii}^{*} values of 29.2 and 24.9 kpsi$\sqrt{in.}$ The constant-load tests, on the other hand, indicated that K_{ISCC} lay above 27.4 but below 29.8 kpsi$\sqrt{in.}$.

Agreement between the above two methods as applied to Ti-721 is considered good and indicates that the more rapid and economical rising-load method can be employed for screening similar alloys having somewhat different composition or for investigating the effects of various heat-treatments. Constant-load tests can be employed, if time is available and there are sufficient specimens, to obtain more precise determination of K_{ISCC}.

It is of interest, finally, to consider whether the specimen sizes were suitable for plane-strain conditions and, hence, accurate K_{ISCC} values. According to an ASTM recommended practice[11], specimen thickness B must fulfil the requirement that

$$B \geq 2.5\left(\frac{K_{ISCC}}{\sigma_{y.s.}}\right)^2 \quad \ldots\ldots \quad (4)$$

If we assume that K_{ISCC} is equal to 27 kpsi$\sqrt{in.}$ for freely corroding specimens of sensitized Ti-721 and that the yield strength, $\sigma_{y.s.}$, is 100 kpsi, then a specimen thickness of 0.18 in. would be sufficient and the specimen thickness (B) of 0.375 in. employed in this work would be satisfactory.

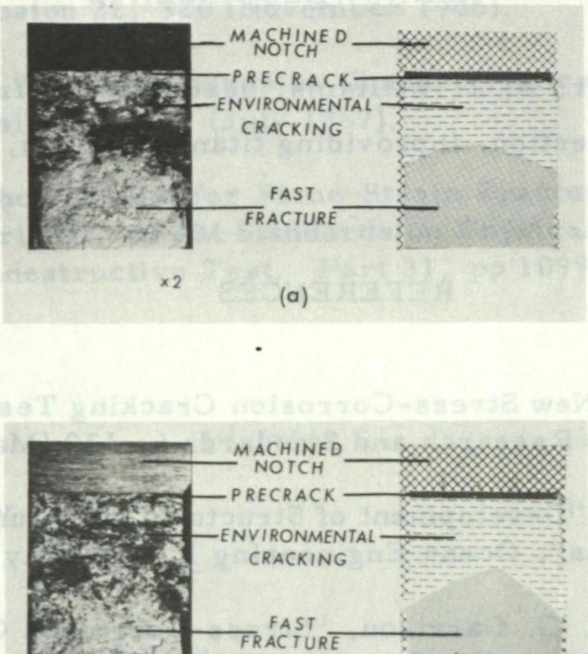


Figure 4. Fracture faces of specimens of sensitized Ti-721 broken in 3.5% NaCl solution under a static load. (a) Specimen 2-4; (b) specimen 2-6.

However, for specimens of the same alloy broken under dry conditions, and assuming that the fracture toughness K_{Ic} is equal to 75, the thickness required by the above equation would be about 1.4 in. Therefore, the dry K_{Ii}^* values obtained in this work cannot be considered as yielding precise information on the fracture toughness of Ti-721. The consistently higher dry K_{Ii}^* values obtained for 1-in. thick plate than for 1/2-in. thick plate (Table 1) are possibly related to the fact that, for our specimen size, the significance of these values is only qualitative.

ACKNOWLEDGEMENTS

The help of Dr. A. J. Williams, head of the Refractory Metals and Engineering Physics Section, inproviding titanium alloys, is much appreciated.

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