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THE HOT-SALT STRESS CORROSION CRACKING OF TITANIUM ALLOYS

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16. Abstract

This report summarizes research at Savannah River on the mechanism for initiation and propagation of hot-salt cracks in titanium alloys. When a titanium alloy coated with an apparently dry halide salt is heated between 250-650°C, the hydrogen halide penetrates the protective oxide film and attacks the metal. Hydrogen is generated by the reaction of the hydrogen halide and metal and is partially absorbed by the metal surface. The hydrogen embrittles the surface, and the crack can then be initiated by residual or applied stresses. Cracks propagate by stress sorption, with hydrogen as the sorbing species.

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CONTENTS

LIST OF TABLES AND FIGURES

INTRODUCTION

Titanium-aluminum alloys will be used in supersonic aircraft that will operate at skin temperatures as high as 290°C while possibly contaminated with sea salt. Laboratory tests showed that titanium-aluminum alloys are susceptible to hot-salt stress corrosion cracking under operating conditions of supersonic aircraft. The Savannah River Laboratory (SRL), sponsored by the National Aeronautics and Space Administration, investigated the mechanism of hot-salt stress corrosion cracking of these alloys. This report is a summary of this research; a more-detailed review of this work at SRL has been given in earlier reports.[1-8]

THE PROPOSED MECHANISM

The proposed mechanism for hot-salt stress corrosion cracking involves the pyrohydrolytic formation of the hydrogen halide corresponding to the anion of the salt (e.g., HCl from Cl salts), and attack of the metal by the hydrogen halide after penetration of the protective oxide film. Hydrogen is generated by the reaction and is partially absorbed by the metal surface. The hydrogen embrittles the surface, and the crack can then be initiated by residual or applied stresses. Cracks propagate by stress sorption, with hydrogen as the sorbing species.

FORMATION OF HALOGEN ACIDS

Evidence of Pyrohydrolysis

When a titanium alloy coated with an apparently dry halide salt is heated between 250 and 650°C, a hydrogen halide is formed by pyrohydrolysis. Evidence of this reaction was shown in three different ways:

- The presence of HCl was shown by mass spectrometry, and the concentration of HCl was determined for NaCl and $SnCl_2 \cdot 2H_2O$ decomposition.[1] (HBr was also identified from bromide salts.)

- Microprobe analysis (Figure 1) showed Cl^- concentration decreased toward the crack tip, but Na^+ was not present in NaCl cracked specimens, indicating a volatile Cl species (HCl or Cl_2). The microprobe results were confirmed autoradiographically with $^{22}NaCl$ and $Na^{36}Cl$.[1]

- In three tests, air which had passed over heated NaCl-coated chips was bubbled through water, starch-iodide solution, and NaOH. The water showed only the presence of Cl^- and no metallic cation. The cation was shown to be H^+ in the starch-iodide test.[2] Ultraviolet spectroscopy of the NaOH solution showed that Cl_2 was absent.

Above 650°C, Cl_2 was identified by ultraviolet spectroscopy from the reaction of NaCl-coated titanium alloys.[2] This helped to explain the very high corrosion rates and cracking at elevated temperatures. The mechanism at this temperature was not determined because it was above the temperature of aircraft operation; both HCl and Cl_2 can crack stressed titanium alloys.

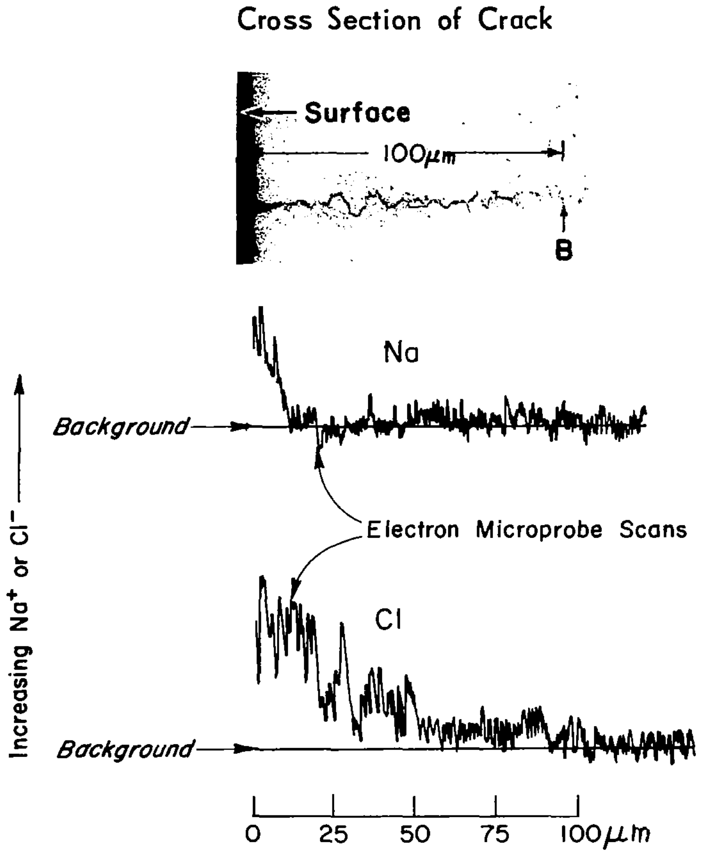


FIG. 1 VARIATIONS OF SODIUM AND CHLORIDE CONCENTRATIONS WITHIN CRACK

Requirements for Pyrohydrolysis

The formation of hydrogen halide by pyrohydrolysis requires heat and moisture; for salts which are difficult to hydrolize (e.g., NaCl), the reaction rate is increased by "accelerators," such as aluminum or vanadium oxides.

The oxide film present on Ti-Al alloys is enriched in aluminum and may also contain vanadium, if this is one of the alloying elements.[3] Both aluminum oxide and vanadium pentoxide have been used as accelerators in the pyrohydrolysis of salts difficult to hydrolyze.

PENETRATION OF OXIDE FILM

Degradation of Oxide Film by Cl^-

When a titanium alloy is immersed in a salt solution, the protective oxide film on titanium alloys is partially degraded because Cl^- is absorbed into the oxide lattice, probably in an oxygen vacancy. This absorption requires a change of cation valence to maintain charge neutrality and to avoid developing a space charge in the oxide. In NaCl tracer solutions on Pt, Ta, Ti, Ti-8Al-1Mo-1V, Zircaloy-2, and Type 304 stainless steel,[1] $^{36}Cl^-$ with little or no ^{22}Na was absorbed on the oxide film. Cl^- absorption on Ti-8Al-1Mo-1V increased to a maximum at about pH 6.

Effect of Film Thickness on Time to Cracking

The time to cracking consists of the time required to penetrate the protective film and the time to attack the metal and cause cracking.[3] Film thickness is a barrier to HCl diffusion through the film. Time to cracking was measured on stressed Ti-8Al-1Mo-1V specimens that had film thicknesses of <50 Å, 100-200 Å, and 400-700 Å. The time to cracking at 350°C increased with increasing oxide thickness. The reaction was, therefore, diffusion controlled.

EVOLUTION AND ABSORPTION OF HYDROGEN AND CRACK INITIATION

After the protective oxide film is penetrated by HCl, hydrogen is generated by the reaction of HCl and the metal and then is partially absorbed by the metal surface. The alloys are first corroded, and after an incubation period cracks initiate.

Identification and Location of Hydrogen

Hydrogen which is produced as a corrosion product was identified by mass spectroscopy during heating of metal, salt, and small amounts of water; its concentration increased with heating time.[4] The hydrogen did not come from a metal-water reaction, and there was insufficient hydrogen present in the metal to cause the concentrations observed, even if all the hydrogen had been released. The amount of hydrogen generated was directly proportional to the amount of water added to the salt-coated metal (Figure 2); therefore, moisture supplies the corrosion-produced hydrogen.

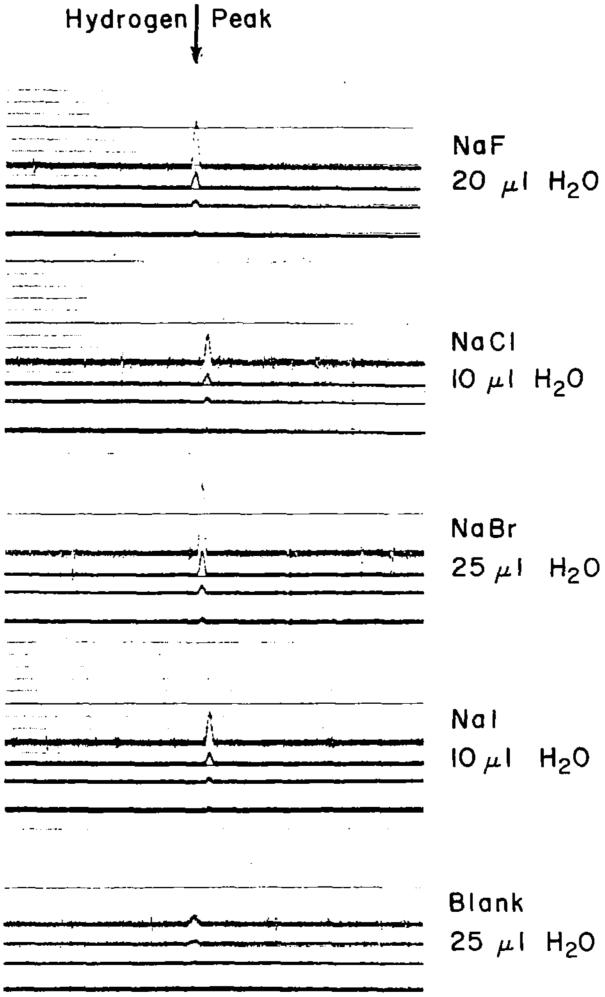


FIG. 2 HYDROGEN PRODUCTION BY SALT—TITANIUM REACTION AT 350°C

Sequence of mass spectrometer traces for each experimental condition. Note that, except for the blank, the mass of hydrogen is approximately proportional to the amount of water present.

Hydrogen was absorbed by titanium alloys during exposure to hot salts in ^{3}H tracer tests.[5] Saturated salt solutions spiked with $^{3}H_2O$ were evaporated on stressed alloy specimens, and the specimens were heated. Autoradiographs of the surfaces showed activity after the specimens had been rinsed free of water-soluble corrosion products (Figure 3).

a. Before rinsing in H_2O.

b. After H_2O rinse, before autoradiography.

c. After autoradiography (film in situ).

FIG. 3 DISTRIBUTION OF ^{3}H ON SALT-CORRODED Ti-8Al-1Mo-1V AT 343°C

The location of primary hydrogen absorption was shown by transmission electron microscopy studies of thin Ti-8Al-1Mo-1V foils.[6] Titanium hydrides formed preferentially in regions near or along α-α grain boundaries or α-β phase boundaries. The association of microscopic hydrides in grain and phase boundaries indicates these are the areas of most rapid diffusion and concentration, and therefore the most probably places of entry.

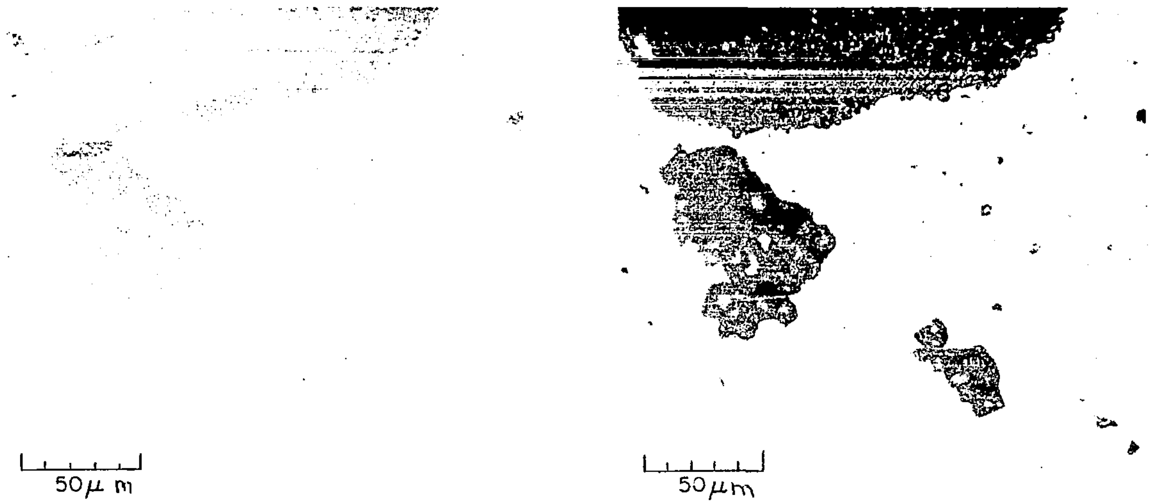
Hydrogen penetration into hot-salt corroded specimens was measured by counting β from ^{3}H.[7] After 75 hr at 343°C, the surface of specimens was removed incrementally: first by mechanical polishes, then by chemical etches. The depth of penetration was measured to be a minimum of 10 μm based on the limits of detection by the counter.

Corrosion Reaction

Characteristics

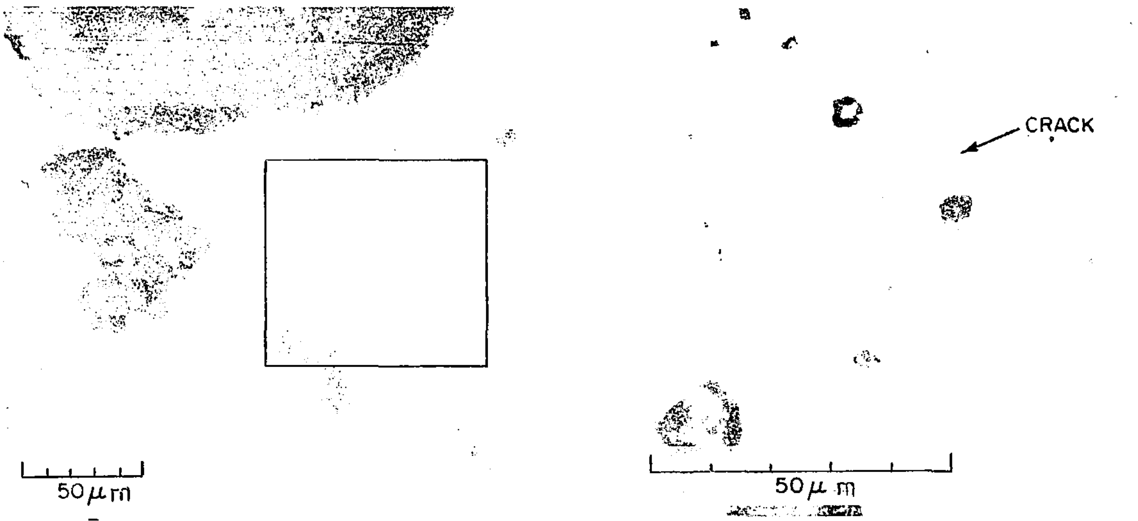
In hot stage microscopic tests at 343°C with Ti-8Al-1Mo-IV, NaCl corroded and stained the alloy after <10 minutes at that temperature, but cracking did not initiate until 75 to 90 minutes later.[8] Minute corrosion stains at the salt crystals gradually enlarged forming a fairly continuous stain around the crystals, but cracks initiated before the stain became continuous. Corrosion products did not appear to spread outward from the salt-metal contact area (Figure 4). The alloy was corroded at salt-metal contact areas, and cracks nucleated only in these regions. (Figure 5). Cracking was not related to the size of the salt crystals because cracks also initiated at very small crystals.

Although the pyrohydrolysis of NaF produces HF, NaF does not cause hot-salt stress corrosion cracking because alloys coated with NaF do not absorb hydrogen during corrosion, as shown in ^{3}H tracer tests.[2] Autoradiography and β counting could not detect tritium; however, the alloy was partially corroded. Hydrofluoric acid probably attacks the metal surface, and the hydrogen liberated by this attack and the affected titanium surface are probably removed from the system by the simultaneous formation and sublimation of TiF_4. TiF_4 might hydrolyze, forming two more volatile products, $TiOF_2$ and HF.



a. Salt Deposit at Room Temperature

b. Same as (a) After Reaching 650°F

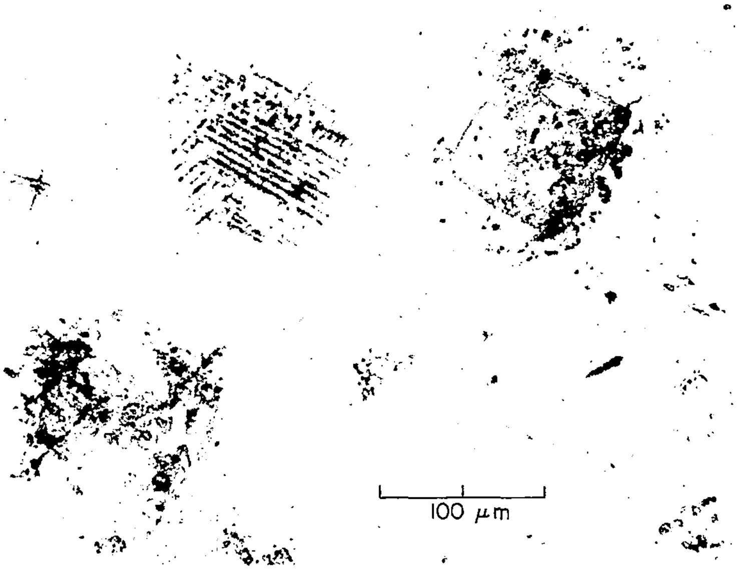


c. Same as (b) After 80 Minutes

d. Same as (c) Enlarged

FIG. 4 HOT-STAGE MICROSCOPY OF NaCl CRACKING OF Ti-8Al-1Mo-1V AT 343°C

NaCl crystal on Ti-8Al-1Mo-1V surface (polarized light). Fluid inclusions appear as dark areas and lines parallel to crystal faces.



Corrosion pattern apparently related to a crystallographic feature of the salt crystal (250X).

FIG. 5 ASSOCIATION OF CORROSION PATTERN WITH FLUID INCLUSIONS IN SODIUM CHLORIDE

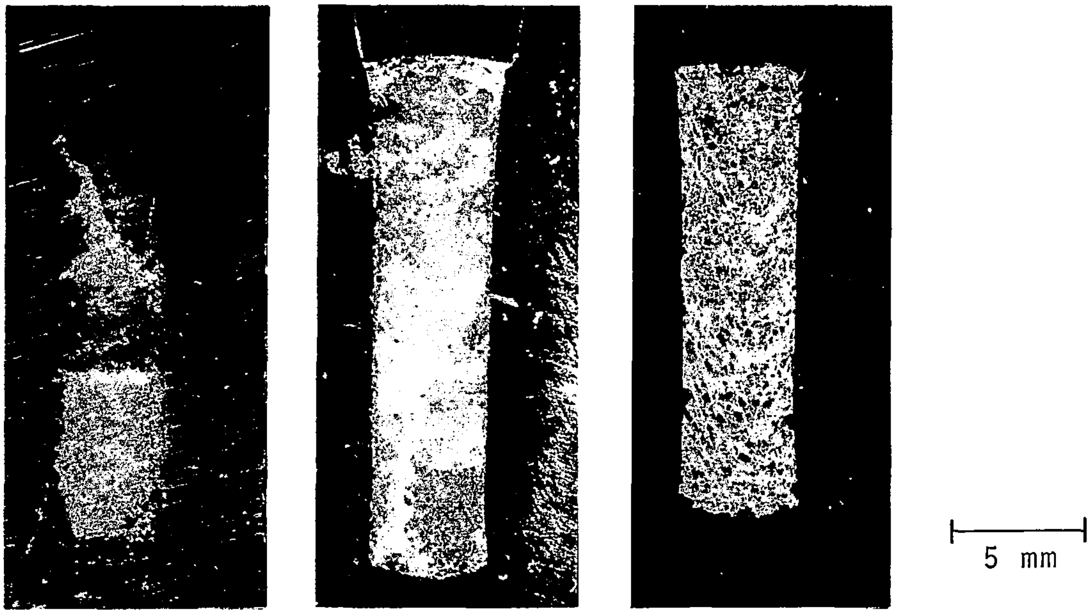
Role of Moisture

The role of moisture in the cracking process was studied because moisture supplies the corrosion-produced hydrogen and is required for pyrohydrolysis.[1] The halide salt reacts with the moisture in the presence of the protective oxide to pyrohydrolytically form a hydrogen halide. The hydrogen halide attacks the metal and generates hydrogen gas.

During corrosion of titanium alloys, water was shown to be present by mass spectrometry. The water probably came from the salt because NaCl crystallized from a saturated solution contains residual water that can be seen under polarized light as dark areas or lines generally parallel to crystal faces (Figure 5). Thus, some apparently dry crystals contain water and, therefore, can cause cracking.[5,8]

Moisture has been shown to be present in the cracking system by tests with ^3H_2O. Three possible sources of water are: the salt, moisture sorbed on metal, and atmospheric moisture. The moisture retained in salt crystals is the most important source and causes cracking over short periods of time (90 minutes at 343°C).[6] The amount of moisture present depends on the salt and the water of hydration.[7] Moisture in heated air was not sufficient to cause crack initiation in short periods of time, but might do so after prolonged exposure.

Crystals of NaCl predried at 400°C under 10^{-5} torr vacuum do not cause cracking (Figure 6). Furthermore, predried salt crystals encapsulated in dried titanium metal under anhydrous conditions did not cause corrosion when heated to the hot-salt cracking temperature range (350-480°C), and neither did mixtures of dried NaCl and dried TiO_2.[2]



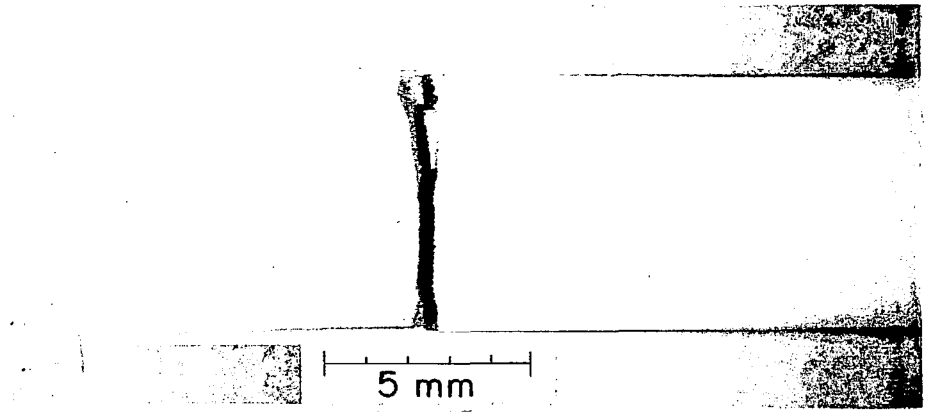
Undried NaCl | Dried NaCl | Dried TiO_2 + dried NaCl

FIG. 6 DRIED REAGENTS PREVENT HOT-SALT ATTACK AT 480°C

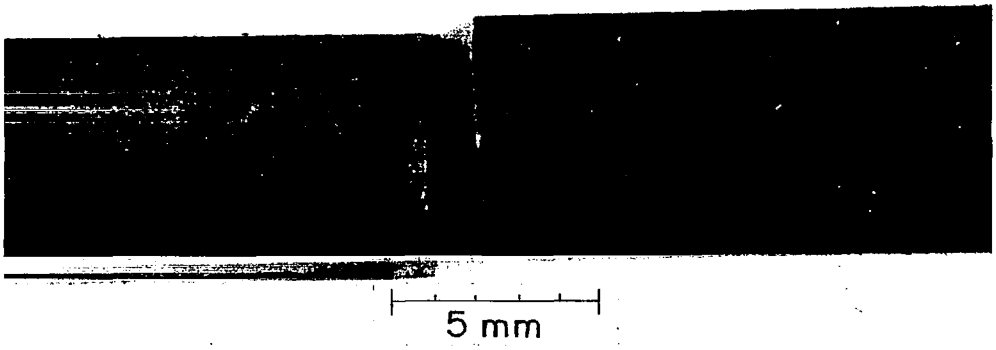
Determining Corrosion Products

Corrosion reactions of hot salts and titanium alloys were simulated by exposing Ti-8Al-1Mo-1V to anhydrous HCl and HCl with water.[3] Doubly distilled, anhydrous HCl gas reacted with stressed Ti-8Al-1Mo-1V at 350°C to form large cracks after the specimen was coated with corrosion products. The gaseous corrosion product was hydrogen; the solid corrosion products were $TiCl_2$, $TiCl_3$, $TiCl_4$, and Al_2Cl_6. These products hydrolyze ($TiCl_4$), oxidize and hydrolyze ($TiCl_2$, $TiCl_3$) or are not formed (Al_2Cl_6) in the presence of moisture and heat. These unstable corrosion products of anhydrous HCl could be considered to be the intermediates in the reaction with moist HCl. In the presence of water, the titanium chlorides decompose to TiO_2, HCl, and H_2.

With 5 to 6 mole % H_2O in the HCl gas, titanium chlorides were not observed as corrosion products.[7] A protective oxide formed over the specimens, and they remained bright although they cracked as badly as when exposed to anhydrous HCl (Figure 7). Hydrogen was a corrosion product.



a. HCl containing 5 - 6 mole % H_2O



b. Anhydrous HCl

FIG. 7 STRESS CORROSION CRACKING OF Ti-8Al-1Mo-1V BY HCl

Crack Initiation Time

Effect of Temperature

The incubation time for cracking at yield stresses decreased by a factor of about 100 as the temperature increased from 245° to 370°C. Cyclic heating and cooling should cause little or no cracking if the heating time is less than the crack initiation time at that temperature; however, alloys might crack after prolonged cyclic heating.

Effect of pH

The time required for a salt-coated alloy to crack was directly related to the pH of the solution from which the salt was crystallized.[1] The time to initiate cracking in stressed Ti-8Al-1Mo-1V coated with NaCl, NaBr, and NaI decreased greatly if the saturated solution used to crystallize each salt were acidified with the corresponding halogen acid. If a highly acidic salt such as $SnCl_2 \cdot 2H_2O$ (pH of saturated solution = 0.2) were partially neutralized, the time to initiate cracking increased (Table I).

TABLE I

Effect of pH on Time to Cracking

Salt	pH of Saturated Solution	Temp, °C	Time to Cracking, min	pH of Adjusted Solution	Time to Cracking, min
NaCl	4.1	343	80	0.5	40
NaBr	4.7	343	150	0.5	75
NaI	8.9	400	150	0.5	75
NaF	8.4	400	>15,000[a]	5.2	>240[a]
$SnCl_2 \cdot 2H_2O$	0.2	343	10	4.1	30
$SnCl_2 \cdot 2H_2O$	0.2	343	10	0.5	15
CuCl	3.4	343	60	0.5	15

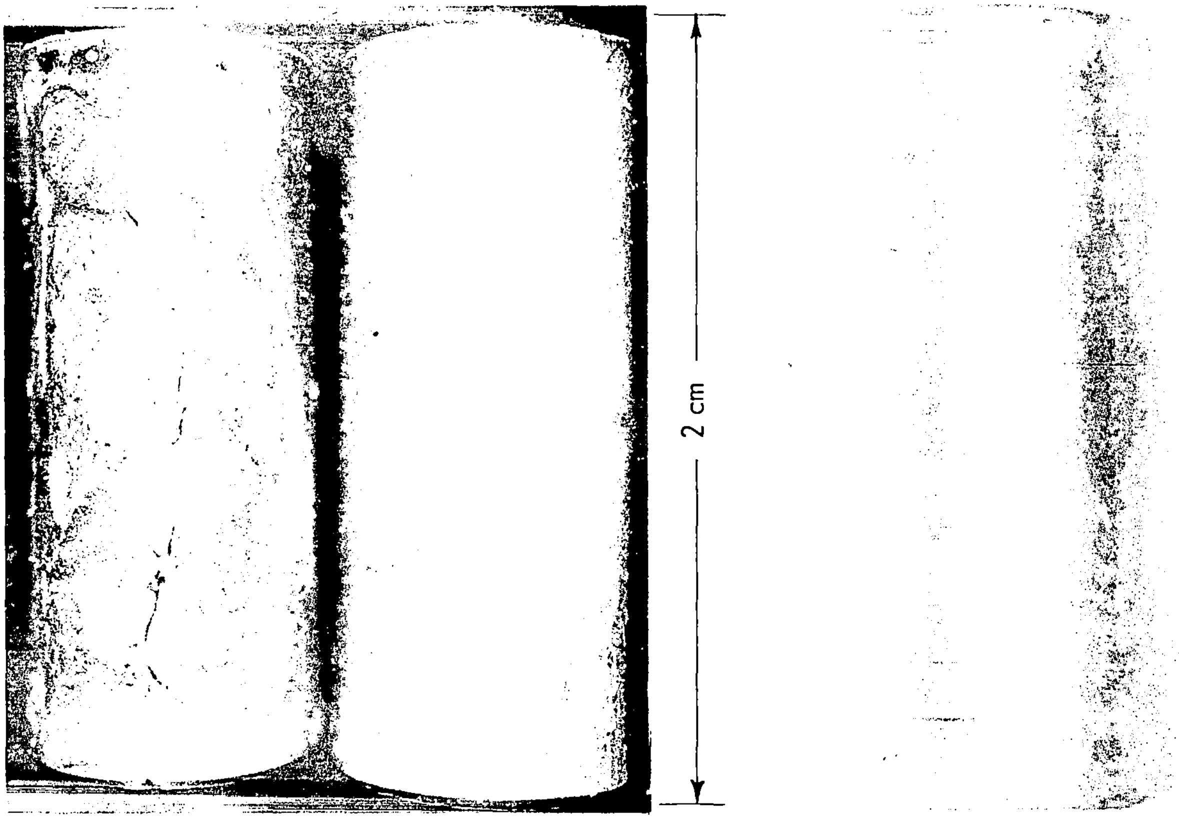
[a] Specimen did not crack; pH could not be made highly acidic because of HF association.

Effect of Salt Composition

The time to initiate cracking and the extent of cracking are determined by the ease with which the salt hydrolyzes and the time required for the hydrolysis products to penetrate the protective film. The time to initiate cracks in Ti-8Al-1Mo-1V coated with $SnCl_2 \cdot 2H_2O$ was 1/10 the time for Ti-8Al-1Mo-1V coated with NaCl. $SnCl_2 \cdot 2H_2O$ produces HCl by hydrolysis much more rapidly than does NaCl at the temperatures studied.

Deposits of NaCl, NaBr, NaI, natural sea salt, $SnCl_2 \cdot 2H_2O$, $CaCl_2 \cdot 2H_2O$, and $MgCl_2 \cdot 2H_2O$ caused cracking of Ti-8Al-1Mo-1V at 343°C with the metal at yield stresses. Of the anhydrous salts, NaCl and KCl caused the most extensive cracking; of the hydrated salts, cracking by $SnCl_2 \cdot 2H_2O$ was most severe.

Stressed Ti-8Mn at 343°C exposed to $SnCl_2 \cdot 2H_2O$ did not crack although corrosion was severe after 91 hours[6]. The metal was embrittled, however, because the metal cracked during bending at room temperature after the residual salt was washed off (Figure 8). The cracking was not caused by binding or heating.



Salt Exposed-Heated Unexposed-Unheated Unexposed-Heated

FIG. 8 CORROSION-EMBRITTLEMENT OF Ti-8Mn ALLOY

Effect of Alloy Composition

The time to initiate cracks with NaCl at 343°C increased with decreasing aluminum content as shown in Figure 9. The most resistant titanium-aluminum alloy (Ti-4Al-3Mo-1V) cracked after 28 hours exposure, but Ti-8Mn had not cracked after 48 hours.[8]

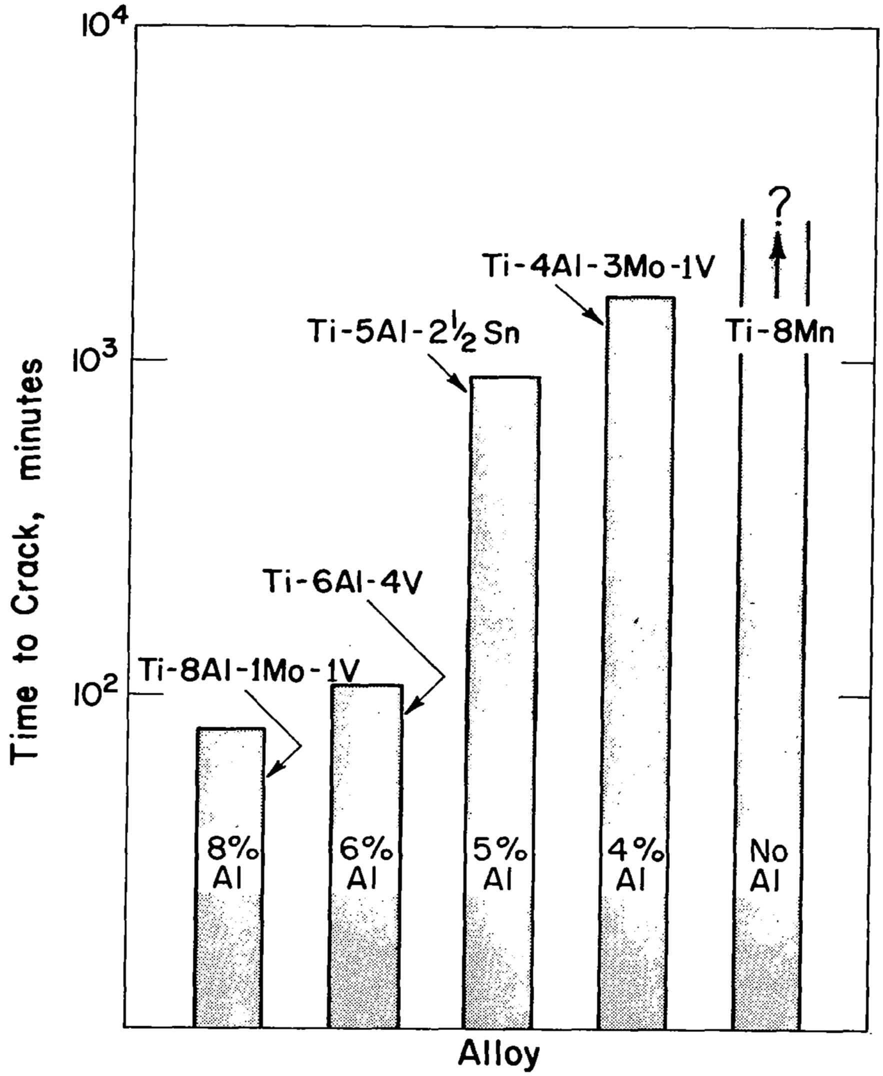
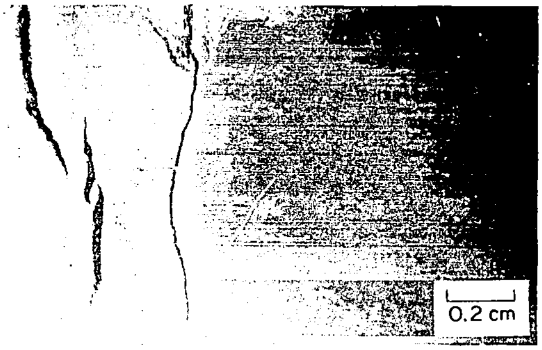


FIG. 9 EFFECT OF ALLOY COMPOSITION ON TIME TO INITIATE CRACKING BY NaCl AT 343°C

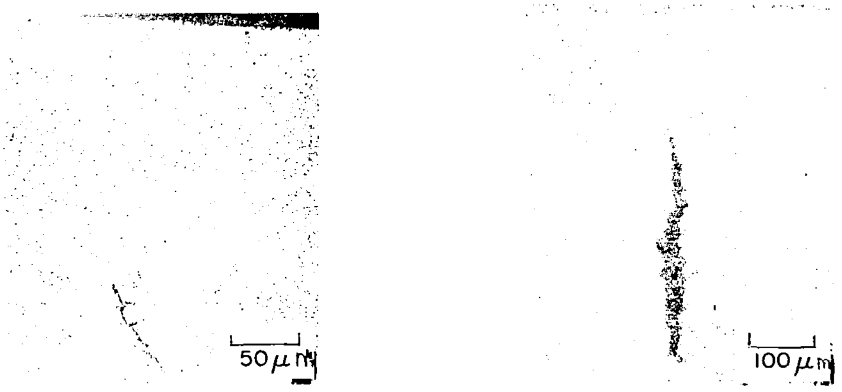
CRACK PROPAGATION BY STRESS SORPTION OF HYDROGEN

The hydrogen, generated by the reaction of HCl and the alloy, is absorbed by the metal and causes localized surface embrittlement which leads to crack nucleation. Nucleation of a crack produces a sharp notch (the crack tip), and sorption of hydrogen at the crack tip lowers the surface energy of the metal such that the existing stresses can cause rupture and crack propagation.[4] Cracks propagate by mechanical rupture; the propagation rate is highest where a continuous supply of halogen acid and, therefore, hydrogen is produced near the crack tip.

Hot-salt stress cracks appear abruptly, propagate rapidly for short distances, stop, and then again propagate for short distances. The cracks often extend beneath the surface of the metal.[6] Rippling and necking of the metal surface above the crack showed that plastic deformation accompanies fracture (Figure 10). These results were verified by fractographic studies.



Top Surface of Specimen



Edge Section

Edge Section

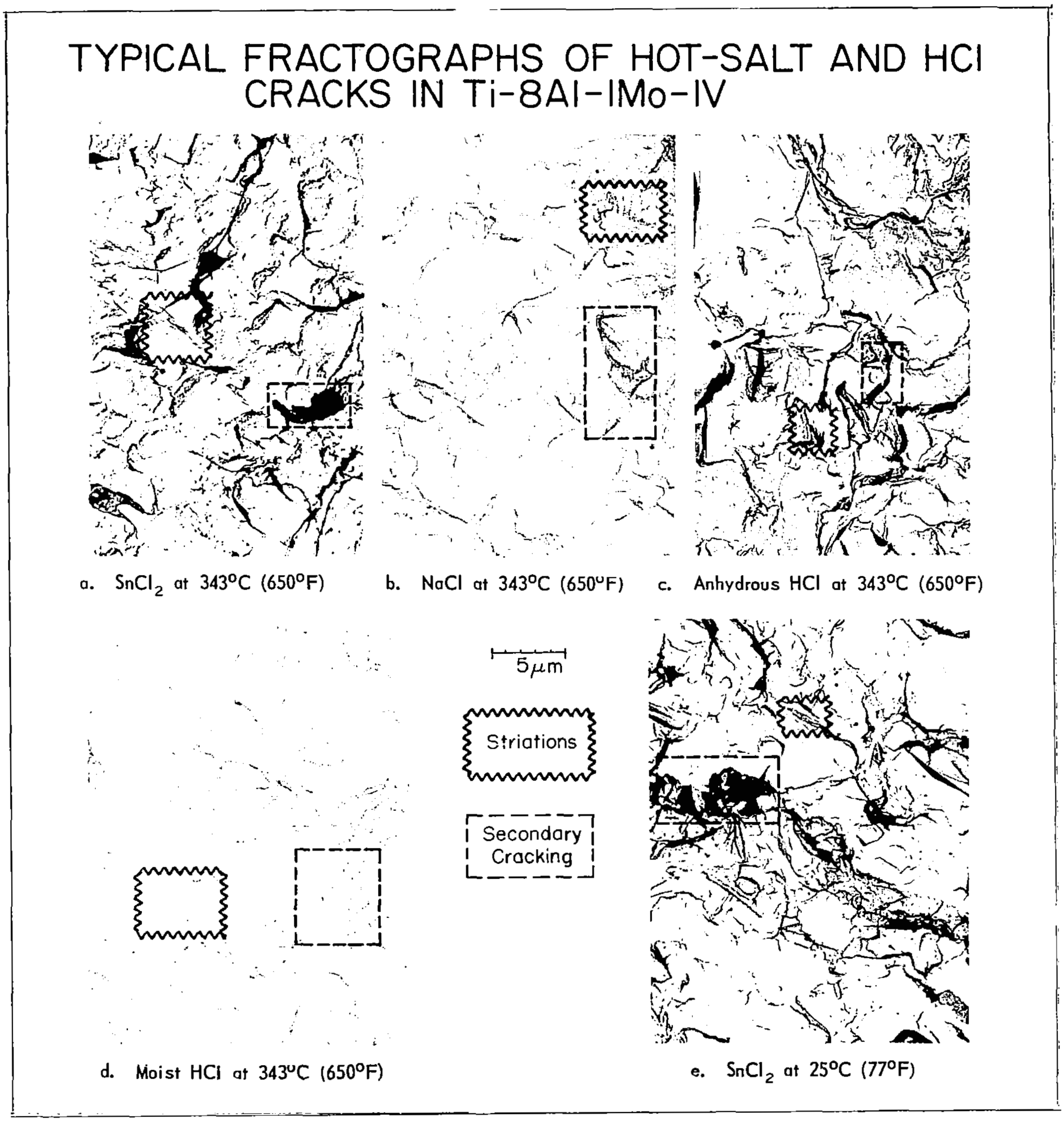
FIG. 10 "TUNNELING" OF $SnCl_2$ - INDUCED CRACKS IN Ti-8Al-1Mo-1V

Studies of Fracture Surfaces

Studies of fracture surfaces eliminated chemical attack by HCl and hydrogen embrittlement by hydrogen platelets as a means of crack propagation. A mechanical fracture would result if: 1) some species were absorbed on the specimen surface and lowered the surface energy to the extent that existing stresses promoted fracture, or 2) some species diffusing in front of the crack promoted brittle fracture at the specimen stress level. The high propagation rates seen in some specimens (0.1 in./min at room temperature) preclude diffusion in advance of the crack. Therefore, stress sorption is proposed as the mechanism of crack propagation.

Cracking with HCl and Hot Salts

Fractographs of alloys produced in a variety of environments were surprisingly similar. Cracks propagated in Ti-8Al-1Mo-1V at 343°C with NaCl, $SnCl_2 \cdot 2H_2O$, anhydrous HCl, and HCl with 5 to 6 mole % H_2O all showed intergranular fractures with little evidence of ductile failure.[3] Secondary cracking and rough grain surfaces with areas of regular striations or folds were common features. These areas, probably a result of serpentine glide, glide plane decohesion, and stretching, are interpreted as evidence of dislocation movement (Figure 11). Typical river patterns, characteristic of transgranular cleavage, were also found in small regions. Some of the fracture faces had severe surface roughening, probably resulting from corrosion after cracking, but other faces were not rough.



a. $SnCl_2$ at 343°C (650°F)

b. NaCl at 343°C (650°F)

c. Anhydrous HCl at 343°C (650°F)

d. Moist HCl at 343°C (650°F)

e. $SnCl_2$ at 25°C (77°F)

FIG. 11 TYPICAL FRACTOGRAPHS OF HOT-SALT AND HCl CRACKS IN Ti-8Al-1Mo-1V

The similarity between hot-salt and HCl cracking is shown in Table II.

TABLE II

Characteristics of Hot-Salt and HCl Cracking

Characteristics	Hot-Salt Cracks	HCl Cracks
Fracture path	Predominantly intergranular	Predominantly intergranular
Fracture mode	Mechanical	Mechanical
Crack threshold temperature	232°C	245°C
Effect of oxide film	Delays crack initiation	Delays crack initiation
Crack propagation rate[a]	Slow	Rapid

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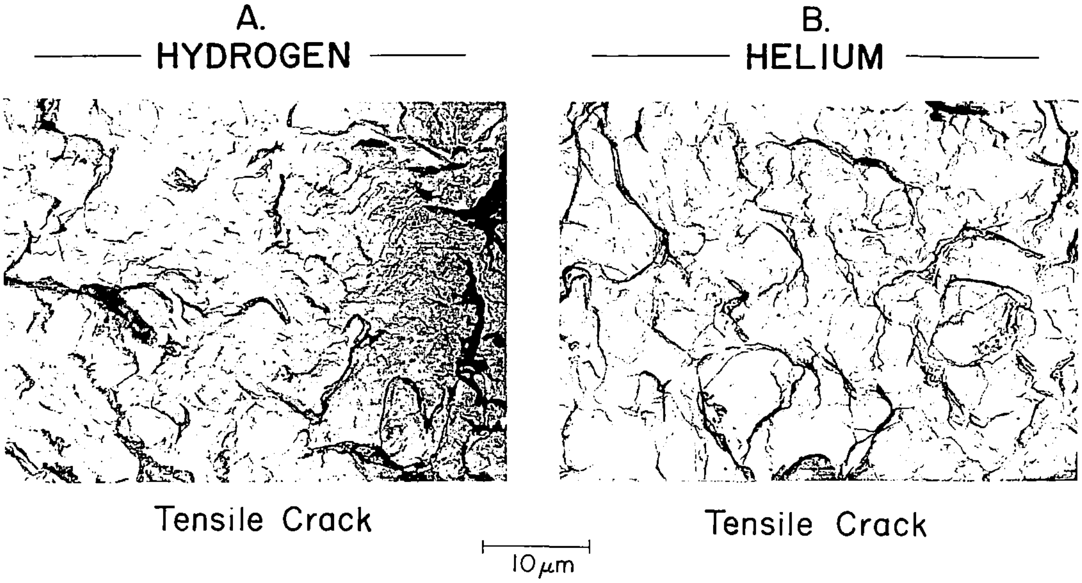
[a] Difference is due to the availability of HCl to react.

Effect of Hydrogen on Fracture Surfaces

Fracture surfaces of specimens charged with 1400 and 4000 ppm hydrogen and fractured mechanically at room temperature did not resemble hot-salt cracks.[3] At 1400 ppm, the metal failed by both ductile and brittle fracture, but mainly by ductile mechanisms. At 4000 ppm hydrogen, failure was brittle; the fracture was along an almost continuous network of hydrides lying along the α-α and α-β grain and phase boundaries.

The effect of hydrogen gas on the fracture surfaces of Ti-8Al-Mo-1V and Ti-5Al-2.5Sn was evaluated.[4] Notched, precracked tensile specimens were tested to failure in 10^4 psi (He and H_2 gas). Replicas of the fracture faces produced in helium showed the typical dimpled structure of ductile rupture. Replicas of fracture surfaces produced in hydrogen were much more brittle, showing that hydrogen did affect failure (Figure 12). Fracture surfaces with hydrogen were compared to fracture surfaces produced by hot-salt and HCl cracking. The topographies were quite similar, both showing brittle failure by quasi-cleavage and some secondary cracking.


A. HYDROGEN

B. HELIUM

Tensile Crack

Tensile Crack

FIG. 12 CRACK SURFACES OF Ti-8Al-1Mo-1V PRODUCED IN 10^4 psi HYDROGEN AND HELIUM

Hydrogen Concentration Necessary to Initiate and Propagate Cracks

Method of Measurement

The amount of hydrogen required to initiate and propagate cracks in stressed Ti-5Al-2.55Sn and Ti-8Al-1Mo-1V was measured.[4] The stressed alloys were bombarded with accelerated H^+ ions, and the concentration of hydrogen was calculated at the surfaces with no cracks (initiation) and at crack tips (propagation).

A neutron generator at Savannah River was modified to accept stressed, water-cooled titanium targets and adjusted to produce and accelerate $^1H^+$ rather than $^2H^+$. Maximum acceleration was 150 kev, equivalent to a penetration of about 1 μm into the alloy. The beam diameter was measured directly on the target, and the proton flux was determined by beam current, and the number of ions per unit area was calculated. Strain gages were used to determine stress on the outer surfaces of the titanium specimens.

Results

At a total fluence of 3.2×10^{19} H^+/cm^2, cracks were observed in Ti-5Al-2.5Sn; but cracks in Ti-8Al-1Mo-1V were observed after 2.0×10^{19} H^+/cm^2. Bombardment by a fluence lower by a factor of five did not cause cracking in either alloy.

The fluence data were corrected for hydrogen outgassing under vacuum and inward diffusion of hydrogen.[4] Hydrogen concentrations were calculated at the tip of the crack for values required to produce propagation and at the surface of uncracked specimens for values that must be exceeded to initiate cracking at 10^5 psi (Table III).

Table III shows that to initiate a crack in Ti-5Al-2.5Sn at 10^5 psi the H/Ti atom ratio must be >1/7 (3.0×10^2 cm^3H_2/cm^3Ti) but <1/1.5 (1.5×10^3 cm^3H_2/cm^3Ti) and must be 1/300 (7 cm^3H_2/cm^3Ti) to propagate. Similarly with Ti-8Al-1Mo-1V at 10^5 psi, the H/Ti atom ratio must be >1/16 but <1/3 to initiate cracks and 1/350 to propagate cracks.

TABLE III

Hydrogen Concentration to Initiate and Propagate Cracks

Alloy	H_2 Concentration at Crack Tip, cm^3 H_2/cm^3 Ti	Average H_2 Concentration at Surface, cm^3 H_2/cm^3 Ti
Ti-5Al-2.5Sn	7[a]	1.5×10^3
Ti-5Al-2.5Sn	No cracks	3.0×10^2
Ti-8Al-1Mo-1V	6[b]	9.6×10^2
Ti-8Al-1Mo-1V	No cracks	1.9×10^2

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[a] At 10 µm.

[b] At 30 µm.

Hot-salt crack initiation time is much longer for Ti-5Al-2.5Sn than for Ti-8Al-1Mo-1V (10^3 vs 10^2 min) at 343°C under 10^5 psi outer fiber stress. The amount of H^+ required to initiate cracks is also much higher for Ti-5Al-2.5Sn than for Ti-8Al-1Mo-1V under 10^5 psi outer fiber stress. As previously reported,[4] the hydrogen concentration is higher in the metal near a hot-salt crack.

ACKNOWLEDGMENT

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