NASA TECHNICAL MEMORANDUM

NASA TM X-2404

NASA TM X-2404

CASE FILE COPY

EFFECT OF INITIAL HYDROGEN CONTENT OF A TITANIUM ALLOY ON SUSCEPTIBILITY TO HOT-SALT STRESS-CORROSION

by Hugh R. Gray

Lewis Research Center

Cleveland, Ohio 44135

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • NOVEMBER 1971

1. Report No.
NASA TM X-2404

2. Government Accession No.

3. Recipient's Catalog No.

4. Title and Subtitle
EFFECT OF INITIAL HYDROGEN CONTENT OF A TITANIUM ALLOY ON SUSCEPTIBILITY TO HOT-SALT STRESS-CORROSION

5. Report Date
November 1971

6. Performing Organization Code

7. Author(s)
Hugh R. Gray

8. Performing Organization Report No.
E-6502

9. Performing Organization Name and Address
Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio 44135

10. Work Unit No.
134-03

11. Contract or Grant No.

12. Sponsoring Agency Name and Address
National Aeronautics and Space Administration
Washington, D.C. 20546

13. Type of Report and Period Covered
Technical Memorandum

14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract
The influence of the initial hydrogen content of a titanium alloy on subsequent resistance to hot-salt stress-corrosion embrittlement and cracking was investigated. The Ti-8Al-1Mo-1V alloy was tested in four conditions: mill annealed (70 ppm H), duplex annealed (70 ppm H), vacuum annealed to an intermediate (36 ppm H) and a low (9 ppm H) hydrogen level. Material annealed at 650° C (duplex condition) exhibited resistance to hot-salt stress-corrosion superior to that exhibited by material in the mill-annealed condition. Reduction of the alloy hydrogen content from 70 to as low as 9 ppm did not influence resistance to hot-salt stress-corrosion embrittlement or cracking.

17. Key Words (Suggested by Author(s))
Titanium alloy
Hot-salt stress-corrosion
Hydrogen content
Hydrogen embrittlement

18. Distribution Statement
Unclassified - unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	8	$3.00

* For sale by the National Technical Information Service, Springfield, Virginia 22151

EFFECT OF INITIAL HYDROGEN CONTENT OF A TITANIUM ALLOY ON SUSCEPTIBILITY TO HOT-SALT STRESS-CORROSION

by Hugh R. Gray

Lewis Research Center

SUMMARY

The influence of the initial average hydrogen content of a titanium alloy on subsequent resistance to hot-salt stress-corrosion embrittlement and cracking was investigated. The Ti-8Al-1Mo-1V alloy was tested in four conditions: mill annealed (70 ppm H), duplex annealed (70 ppm H), vacuum annealed to an intermediate (36 ppm H) and a low (9 ppm H) hydrogen level. Material annealed at 650° C (duplex condition) exhibited resistance to hot-salt stress-corrosion superior to that exhibited by material in the mill-annealed condition. Reduction of the alloy hydrogen content from 70 to as low as 9 ppm did not influence resistance to hot-salt stress-corrosion embrittlement or cracking.

INTRODUCTION

A continuing research program at NASA-Lewis Research Center (refs. 1 to 3) has demonstrated that titanium alloys subjected to hot-salt stress-corrosion exposures are embrittled by the absorption of corrosion-produced hydrogen. Vacuum-fusion chemical analyses have shown that the average bulk hydrogen content of stress-corroded specimens is two to three times the as-received value. In addition, advanced analytical techniques have proven that this corrosion-produced hydrogen is segregated near the fracture surface to levels of several thousand ppm (refs. 2 and 3).

The role of initial hydrogen content of titanium alloys in influencing resistance to cracking is somewhat controversial. It is important to note that all of the literature studies discussed below have considered only the average hydrogen content of the base alloy determined by vacuum-fusion chemical analyses. These investigators compared alloys in the mill-annealed (50 to 150 ppm) and vacuum-annealed (2 to 40 ppm) conditions. Decreasing the alloy hydrogen content has been reported to result in a decrease in crack propagation rates in inert environments (refs. 4 and 5). A theoretical model predicts that a decrease in alloy hydrogen content will result in decreased susceptibility to stress-corrosion cracking (ref. 6). Several investigators have demonstrated such an effect in hot-salt (refs. 7 and 8) and salt-water (ref. 9) environments. However, other investigators have reported no effect in salt water (refs. 4 and 10) and alcohols (ref. 4).

Since the effect of the initial average hydrogen content of titanium alloys was unresolved and since hydrogen is the embrittling species in the process of hot-salt stress-corrosion, an investigation was conducted to determine the influence of the initial hydrogen content of a titanium alloy on subsequent resistance to hot-salt stress-corrosion embrittlement and cracking. This was done by testing Ti-8Al-1Mo-1V alloy bar stock in four conditions: mill annealed (70 ppm H), duplex annealed (70 ppm H), vacuum annealed to an intermediate (36 ppm H) and a low (9 ppm H) hydrogen level.

PROCEDURE

The Ti-8Al-1Mo-1V alloy used in this investigation had the following composition in weight percent: 7.8 Al; 1.0 Mo; 1.0 V; 0.05 Fe; 0.011 N; 0.023 C; 0.07 O; 0.007 H. This alloy was received in the mill-annealed condition. Part of the bar stock was duplex annealed and part was vacuum annealed to reduce the hydrogen content to intermediate and low levels. All heat treatments, tensile properties, and hydrogen contents are listed in table I. Tubular specimens, with both as-machined and chemically-milled surfaces, were precoated with about 0.06 mg/cm^2 salt at 200° C. Specimens were then exposed in a Mach 0.7 dynamic air facility (ref. 11) for 96 hours at various stresses and temperatures of 320°, 370°, and 430° C. After exposure, the specimens were tensile tested at a constant, low crosshead speed of 0.01 cm/min to determine residual ductility. If the elongation was less than 15 percent and the reduction of area less than 25 percent, then

TABLE I. - HEAT TREATMENTS, TENSILE PROPERTIES, AND HYDROGEN ANALYSIS OF Ti-8Al-1Mo-1V

Condition and hydrogen content of bar stock	Heat treatment of bar stock	Tensile strength		Fracture strength		Reduction of area, percent	Elongation, percent	Average hydrogen content, ppm	
		MN/m^2	ksi	MN/m^2	ksi			Bar stock	Corroded specimens
Mill annealed; 70 ppm	[a]Mill anneal (as received)	1030	150	930	135	33	18	55 to 89	100 to 225
Duplex annealed; 70 ppm	Mill anneal; followed by heating at 650° C (1200° F) for 24 hr, and furnace cooling in air	1080	156	970	141	33	20	62,69	-----------
Vacuum annealed; 36 ppm	Mill anneal; followed by heating at 650° C (1200° F) for 24 hr, and furnace cooling in vacuum	1040	151	950	138	31	17	34,34,35,41	40,43,51,53
Vacuum annealed; 9 ppm	Mill annealed; followed by heating at 700° C (1300° F) for 24 hr, and furnace cooling in vacuum	1030	149	940	136	31	19	6, 8, 13	24,32,35

[a]Mill anneal consists of heating at 790° C (1450° F) for 1 hr, followed by air cooling.

TABLE II. - SUMMARY OF HOT-SALT STRESS-CORROSION TEST DATA FOR VACUUM-ANNEALED SPECIMENS

[See ref. 11 for data on mill- and duplex-annealed specimens.]

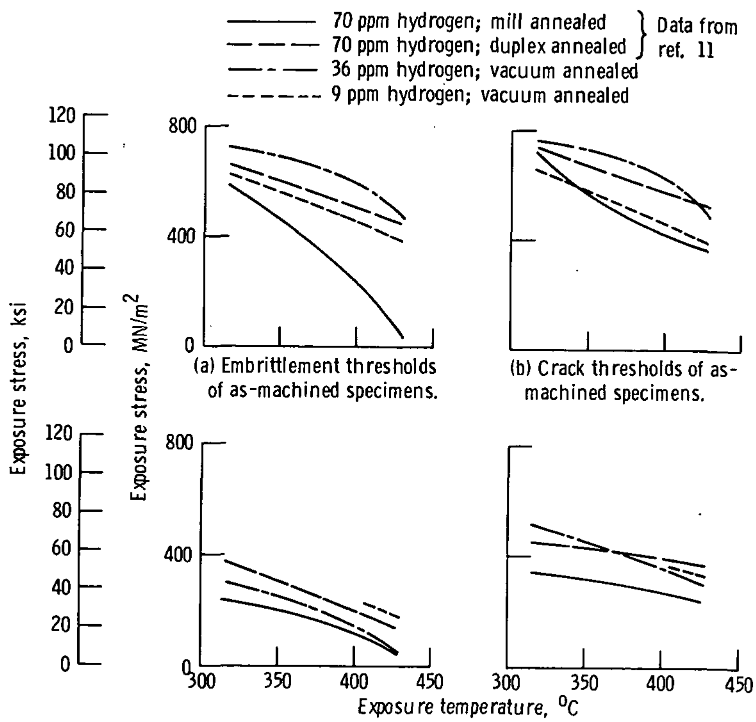
Specimen	Exposure conditions					Tensile test data						Salt-coating		Heat-treated cracks
	Temperature		Time, hr	Stress		Ultimate stress		Fracture stress		Reduction of area, percent	Elongation, percent	mg/cm^2	$mg/in.^2$	
	°C	°F		MN/m^2	ksi	MN/m^2	ksi	MN/m^2	ksi					
As-machined specimens, with 36 ppm hydrogen														
204	320	600	96	690	100	1020	148	940	136	30	16	0.06	0.38	No
211	320	600	94	760	110	1060	154	1030	150	22	12	.12	.80	No
441	320	600	97	760	110	1020	148	1020	148	5	1	.03	.19	Yes
213	370	700	95	480	70	1050	152	970	140	31	16	.06	.39	No
207	370	700	95	550	80	1040	151	950	138	30	18	.09	.55	
208	370	700	97	620	90	1030	150	980	142	25	12	.06	.41	
210	430	800	95	210	30	1060	154	1010	146	27	17	.03	.22	
215			94	280	40	1070	155	1020	148	25	16	.06	.34	
212			95	350	50	1030	149	970	141	23	15	.06	.37	
438			95	410	60	1050	152	990	144	25	15	.11	.74	↓
[a]439	↓	↓	57	550	80	--	--	--	--	6	3	.20	.13	Yes
Chemically milled specimens, with 36 ppm hydrogen														
209	320	600	96	210	30	1010	147	970	141	24	15	0.10	0.62	No
201			96	350	50	1020	148	1020	148	14	10	.08	.51	No
202			98	480	70	1050	152	1050	152	15	10	.07	.43	No
203			95	550	80	1010	146	1010	146	11	6	.03	.20	Yes
214	↓	↓	96	620	90	1010	147	1010	147	12	7	.07	.46	No
436	430	800	96	70	10	1000	145	1000	145	7	2	.07	.47	
216			96	140	20	1030	150	1020	148	18	13	.04	.26	
433			97	210	30	1060	154	1060	154	14	9	.02	.10	
206			96	280	40	1020	148	1020	148	9	5	.03	.21	↓
432	↓	↓	96	350	50	600	87	600	87	0	0	.10	.63	Yes
As-machined specimens, with 9 ppm hydrogen														
262	320	600	97	690	100	1050	152	1040	151	16	12	0.08	0.49	Yes
266	320	600	94	760	110	1050	152	1050	152	11	6	.09	.55	Yes
265	370	700	94	480	70	1010	147	930	135	32	20	.07	.43	No
269	370	700	95	550	80	1010	146	1010	146	9	5	.09	.59	Yes
263	370	700	96	620	90	990	144	990	144	9	5	.05	.34	Yes
261	430	800	96	350	50	1030	149	940	136	30	18	.05	.33	No
268	430	800	95	350	50	1030	150	970	140	25	17	.03	.19	No
267	430	800	95	410	60	480	70	480	70	5	0	.08	.52	Yes
Chemically milled specimens, with 9 ppm hydrogen														
272	430	800	97	140	20	1010	147	930	135	30	18	0.03	0.21	No
264	430	800	95	210	30	970	140	970	140	5	2	.05	.32	No
271	430	800	96	280	40	1010	146	1010	146	12	9	.05	.31	No

[a]Failed during exposure.

the specimen was classified as embrittled. An embrittlement threshold curve was then drawn below the data points meeting this criterion. The fracture surfaces of all specimens were examined optically at 30X to detect stress-corrosion cracks. Cracks as small as 0.02-mm deep could be detected because they were covered with corrosion products (ref. 11). A crack threshold curve was drawn below the data points for specimens on which cracks were detected. A complete summary of all test data is contained in reference 11 and table II.

RESULTS AND DISCUSSION

Both embrittlement and crack threshold curves for as-machined and chemically-milled specimens were determined for the Ti-8Al-1Mo-1V alloy in four conditions: mill annealed, duplex annealed, and vacuum annealed to intermediate (36 ppm H) and low (9 ppm H) hydrogen contents. A comparison of these threshold curves can be made from figure 1. Both the intermediate- and low-hydrogen alloys (36 and 9 ppm H, respectively) exhibit superior resistance to embrittlement and cracking when compared with the mill-



(c) Embrittlement thresholds of chemically milled specimens.

(d) Crack thresholds of chemically milled specimens.

Figure 1. - Hot-salt stress-corrosion threshold curves for Ti-8Al-1Mo-1V alloy with various hydrogen contents.

annealed condition. However, since the material in the duplex-annealed condition was annealed in air at a temperature (650° C) similar to those used for the vacuum-annealing treatments (650° and 700° C), the duplex-annealed condition is a more suitable base line for comparative purposes than is the mill-annealed condition. The hot-salt stress-corrosion resistance of the alloy in the vacuum-annealed conditions is approximately equivalent to that of the duplex-annealed alloy. Therefore, it appears that the lowering of the hydrogen content of the base alloy has an insignificant effect when compared with the effect of annealing at temperatures near 650° C.

The beneficial effect of this stabilization, or aging, anneal is not fully understood. It is possible that duplex annealing results in the decomposition of some metastable beta phase and/or the transformation of metastable martensitic phases. However, no significant microstructural variations between the mill- and duplex-annealed conditions were detected in a previous investigation (ref. 11). This beneficial effect is obviously not related to the embrittling effect of Ti_3Al which commonly results from slow cooling through or extended annealing in the range of 500° to 600° C (ref. 11).

Vacuum-fusion chemical analyses of selected stress-corroded specimens revealed increases in hydrogen content of up to four times the as-received hydrogen content (table I). These average bulk values undoubtedly reflect local concentrations of several thousand ppm (ref. 3).

CONCLUDING REMARKS

The results of this investigation indicate that resistance to hot-salt stress-corrosion embrittlement and cracking is not affected by reductions in the initial average hydrogen content of the base alloy. This conclusion might have been anticipated from the findings of a previous investigation by the author (ref. 3) which demonstrated that corrosion-produced hydrogen segregated to the fracture surface of hot-salt stress-corroded specimens to levels of several thousand ppm. On that basis, a reduction of the initial average hydrogen content of the alloy from 70 to as low as 9 ppm would be considered insignificant when compared with the several thousand ppm actually measured in stress-corroded titanium alloy specimens.

In addition, it is suggested that similar conclusions can be made for the stress corrosion of titanium alloys in salt water and alcohols. Specifically, it is possible that localized concentrations of corrosion-produced hydrogen of the order of several thousand ppm are responsible for stress-corrosion cracking in these media. Therefore, it is unlikely that vacuum-annealing treatments would significantly influence resistance to stress-corrosion cracking.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 9, 1971,
134-03.

REFERENCES

1. Gray, H. R.: Hot Salt Stress Corrosion of a Titanium Alloy: Generation of Hydrogen and Its Embrittling Effect. Corrosion, vol. 25, no. 8, Aug. 1969, pp. 337-341.
2. Gray, Hugh R.: Role of Hydrogen in Hot-Salt Stress-Corrosion of a Titanium Alloy. NASA TN D-6188, 1971.
3. Gray, Hugh R.: Ion and Laser Microprobes Applied to the Measurement of Corrosion-Produced Hydrogen on a Microscopic Scale. NASA TN D-6521, 1971.
4. Sandoz, George: Subcritical Crack Propagation in Ti-8Al-1Mo-1V Alloy in Organic Environments, Salt Water, and Inert Environments. Fundamental Aspects of Stress-Corrosion Cracking. R. W. Staehle, A. J. Forty and D. van Rooyen, eds., Nat. Assoc. Corrosion Eng., 1969, pp. 684-690.
5. Blackburn, M. J.; and Williams, J. C.: Metallurgical Aspects of the Stress Corrosion Cracking of Titanium Alloys. Fundamental Aspects of Stress-Corrosion Cracking. R. W. Staehle, A. J. Forty and D. van Rooyen, eds., Nat. Assoc. Corrosion Eng., 1969, pp. 620-637.
6. Powell, D. T.; and Scully, J. C.: Stress Corrosion Cracking of Alpha Titanium Alloys at Room Temperature. Corrosion, vol. 24, no. 6, June 1968, pp. 151-158.
7. Lingwall, R. G.; and Ripling, E. J.: Elevated Temperature Stress Corrosion of High Strength Sheet Materials in the Presence of Stress Concentrators. Material Research Lab., Inc. (NASA CR-88979), Aug. 1967.
8. Kochka, E. L.; and Petersen, V. C.: The Salt Corrosion of Titanium Alloys at Elevated Temperatures. Final Rep., Crucible Steel Co. of America, Jan. 15, 1961. (Work under Contract NOa(s)-60-6004-c.)

9. Howe, D. G.; and Goode, R. J.: Effects of Heat Treating Environmental Conditions on the Stress-Corrosion Cracking Resistance of Several Titanium Alloys. Applications Related Phenomena in Titanium Alloys. Spec. Tech. Publ. No. 432, ASTM, 1968, pp. 189-201.

10. Mackay, T. L.: Stress Corrosion Cracking of Titanium Alloys at Ambient Temperature in Aqueous Solutions. NASA CR-1464, 1969.

11. Gray, Hugh R.: Relative Susceptibility of Titanium Alloys to Hot-Salt Stress-Corrosion. NASA TN D-6498, 1971.

NATIONAL AERONAUTICS AND SPACE ADMISTRATION
WASHINGTON, D.C. 20546

OFFICIAL BUSINESS
PENALTY FOR PRIVATE USE $300

FIRST CLASS MAIL

POSTAGE AND FEES PAID
NATIONAL AERONAUTICS AND
SPACE ADMINISTRATION

U.S.MAIL

POSTMASTER: If Undeliverable (Section 158
Postal Manual) Do Not Retur

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classification, or other reasons.

CONTRACTOR REPORTS: Scientific and technical information generated under a NASA contract or grant and considered an important contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION PUBLICATIONS: Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION OFFICE

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546