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TIME EXPOSURE STUDIES ON STRESS CORROSION CRACKING OF ALUMINUM 2014-T6, 2219-T87, 2014-T651, 7075-T651, and TITANIUM 6A1-4V

> Final Summary Report June 1, 1971 to June 1, 1973

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ABSTRACT

The effect of a constant applied stress in crack initiation of aluminum 2014-T6, 2219-T87, 2014-T651, 7075-T651 and titanium 6A1-4V has been investigated.

Aluminum c-ring specimens (1-inch diameter) and u-band titanium samples were exposed continuously to a 3.5% NaCl solution (pH 7) and organic fluids of ethyl, methyl, and iso-propyl alcohol (reagent purity), and demineralized distilled water.

Corrosive action was observed to begin during the first and second day of constant exposure as evidenced by accumulation of hydrogen bubbles on the surface of stressed aluminum samples. However, titanium stressed specimens showed no reactions to its environment.

Results of this investigation seems to suggest that aluminum 2014-T6, aluminum 7075-T651 and aluminum 2014-T651 are susceptible to stress corrosion cracking in chloride solution (NaCl), while aluminum 2219-T87 seem to resist stress corrosion cracking in sodium chloride at three levels of stress (25%, 50%, and 75% Y.S.). In organic fluids of methyl, ethyl, and iso-propyl alcohol, 2014-T6 and 7075-T651 did not fail by SCC; but 2014-T651 was susceptible to SCC in methyl alcohol, but resistant in ethyl alcohol, iso-propyl alcohol and demineralized distilled water.

Titanium 6A1-4V showed some evidence of susceptibility to SCC in methanol, while no failures were observed in ethanol, iso-propyl alcohol and demineralized distilled water.

INTRODUCTION

An investigation has been made of stress corrosion cracking of some high-strength aluminum alloys and a titanium alloy. Stress corrosion data acquired in this two-year research effort is among many studies being conducted by research workers to furnish information to help meet the demands of many service requirements for various high-strength alloys. The objective of this research project was to acquire data on the time-to-failure characteristics of Aluminum 2014-T6, Aluminum 2014-T651, Aluminum 2219-T87, Aluminum 7075-T651, and Titanium 6A1-4V in environments of 3.5% NaC1, methyl alcohol, ethyl alcohol, iso-propyl alcohol, and demineralized distilled water.

As reported in the literature Stress Corrosion Cracking (SCC), results from a spontaneous failure by cracking of a metal under the combined action of high stress and corrosion. Cracking typically occurs along the grain boundary in contrast to transgranular cracking generally associated with mechanical fracture resulting from fatigue, creep rupture, tensile overload, etc. The development of cracking occurs along localized paths, producing fissures. These preferentially corroded paths may represent strata of relative low inherent resistance to corrosion, or they may be anodic to the adjacent metal. In aluminum-base alloys, such pre-existent paths generally are associated with grain boundaries [1].

Aluminum c-rings (1 inch in diameter) and titanium u-bend specimens were used in this project. Aluminum 2014-T6 and Aluminum 7075-T651 were stressed at levels of 25%, 50%, and 75% Y.S., while Aluminum 2219-T87 and Aluminum 2014-T651 were stressed exclusively at 75% Y.S. Titanium test specimens were bent until both walls were parallel. Test specimens were exposed continuously to 3.5% solution of NaCl and organic fluids of methyl, ethyl and iso-propyl alcohol until failure was observed or testing discontinued. Specimens which failed were cut and <u>mechanically polished</u> along the crack region for purpose of examination with the light microscope. While surface replica of exposed specimens were made for study under the electron microscope.

Results of this study suggest that 2014-T6, 7075-T651 and 2014-T651 are susceptible to stress corrosion cracking in chloride solution, while 2219-T87 and Titanium resists cracking in chloride solutions.

Titanium 6A1-4V showed some susceptibility to SCC in methanol, while no failures were observed in ethyl alcohol, iso-propyl alcohol, and demineralized distilled water.

MATERIALS

Alloys:

Alloys (see Table I) used in this study were of three types. 2014-T6, 2219-T87, and 2014-T651 represents the Al-Cu type; 7075-T651 representing the Al-Zn-Mg-Cu type and Ti 6Al-4V.

Aluminum 2014-T6, 7075-T651 and Titanium test specimens were obtained from NASA-MSFC, while aluminum 2219-T87 and 2014-T651 were furnished, in plate form, by Alcoa Aluminum Company. Short transverse aluminum c-rings and u-bend titanium specimens were used in testing.

Equipment:

Light microscopy was conducted with an Instrom MMU metallurgical microscope with a magnification range between 75X and 600X. Specimens were mechanically polished with a Meta-Test power grinder at 1,725 rpm.

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An RCA Model EMU-3 electron microscope was used for electron microscopy, while vacuum evaporation and shadow casting was done with one EFFA vacuum evaporator.

EXPERIMENTAL PROCEDURE

Aluminum c-rings were cleaned with acetone and tested as machined. Prior to testing, the necessary parameters-wall thickness, modulus of elasticity, mean diameter, and stress level-were determined. This information was substituted into the c-ring stressing formula [2] and c-rings were stressed (by tighten) bolt to the **desired** stress level. Two protective coatings were used in this study. Initially Maskcoat #2^{*}, a hot strippable plastic, was used as galvanic protection with 2014-T6 and 7075-T651, however, this coating exhibited poor adhesion properties on c-ring surfaces in organic fluids. Consequently, liquid neoprene was used as a replacement for Maskcoat #2. This coating show good adhesion in all invironments used in this project.

Titanium test specimens were dipped in an etching solution (2ml HF + $10ml HNO_3 + 88ml H_2O$) and subsequently washed with acetone to remove the thin oxide scale.

Examination for Cracks:

During periods of testing, daily observation for cracks were made.^{**} The presence of cracks were determined by visual inspection of the specimens. Where doubt existed, visual examinations were supplemented by examination with the light microscope.

*Obtained from Western Coating Company-Royal Oak, MI **Except on weekends

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Replication Technique:

The two-stage replicating technique was used to prepare surface replicas for this investigation. This technique involves making a surface impression (from test specimen) with replicating tape (acetyl cellulose)*** and dissolving tape directly onto the surface of the metal with a few drops of acetone-methanol mixture (1:1). After drying, the replica was stripped from surface of specimen and prepared for vacuum evaporation. This step involves evaporation of a thin film of carbon onto the surface of the specimen and subsequently evaporating a second film of metal (gold-palladium) onto the surface at an angle of 30°. After this step, the replica can be prepared for electron microscopy.

RESULTS

2014-T6 and 3.5% NaC1:

Specimens of 2014-T6 stressed in 3.5% NaCl at pH 6 exhibited cracks along region or surface of maximum tension stress and at base of random corrosion pit sites. Cracking was noted to propagate perpendicular to the direction of stress. Microscopic examination revealed numerous secondary cracks parallel to the primary (major) crack region. Typical cracks are illustrated by light micrographs in Figures 6, 7 & 8 and by electron micrographs in Figures 9 and 10.

In order to interprete the time-to-failure data for 2014-T6 and to ascertain some idea as to the spread of data and to determine whether or not the failure times are significant, 95% confident limits were determined and student t-test applied respectively to the sample average. This is the

***From Ernest F. Fullam Company-Schenectady, NY

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technique of Lewis [3], and Booth et.al. [4] which states that the logarithms of time-to-failure (or endurance) versus cumulative frequency are normally distributed.

Time-to-failure (days) versus cumulative percentage were plotted on logarithmic probability paper and the standard deviation and sample mean were determined from the graph. Using this method, resulted in an estimated <u>population</u> standard deviation of .04 days and a population average of 3.7 days. This compares to a <u>sample</u> standard deviation of 1.14 days and a sample average of 3.9 days [see Table IV).

A t-test for the <u>population</u> average and <u>sample</u> average indicated that these differences were significant for with 14 degrees of freedom, the evaluation of data resulted in t-values (.65) well within the critical value of \pm 1.83 for 95% confidence level, which indicates statistical significance for this kind of analysis.

Figure 4 shows a comparative ranking of median, mean and geometric mean failure times for 2014-T6 in 3.5% NaCl (5 lots). It can be seen that the geometric mean is consistently lower (4 out of 5 lots) than mean or median. It has been reported [2] that the geometric mean is meaningful while the arithmetic mean is not very meaningful, and the median is more reproducible. A comparison of the average geometric mean for this alloy with the estimated population mean resulted in no numerical difference.

Since 2014-T6 seems to be highly susceptible to SCC at 75% Y.S. stress level,attempts were made to establish a lower or minimum level below which failure would not occur. Consequently, c-rings were stressed at 25%, 50% and 75% Y.S. Failures were recorded at each stress level and averaged 16.2 days, 6 days and 3.9 days respectively. This data probably suggest that 2014-T6 is susceptible to SCC at low levels of stress during service exposure.

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To get some idea of the variation of the surface crack widths, 180 random measurements were made from 5 lots of specimens tested. Measurements were made with the light microscope. Figure 5 shows how these measurements are distributed. It is seen from the frequency distribution that a large number of SCC range between .02 mm and .08 mm with a sample mean of .08 mm.

2014-T6 and Methyl Alcohol:

No evidence of stress corrosion failures were observed during an exposure period of 3 days during which the testing were considered valid. During the fourth day of constant immersion, examination revealed that the Maskcoat had started to degrade--extensive decoloration of fluid--resulting in the protective coating failing to prevent galvanic effects. Later it was found that liquid neoprene can be used as a suitable protective coating for organic fluids. No SCC failures were recorded for c-rings exposed continuously to methyl alcohol.

2014-T6 and Ethyl Alcohol:

Exposure of 2014-T6 (3 specimens) in ethyl alcohol produced no failures that could be attributed to stress corrosion cracking. It was noted, however, that 2 failures occured during 185 days of exposure. These failures were interpreted as galvanic failure, as protective coating had started to fail by pulling away from contact points between bolt and c-ring test specimen.

2014-T6 and Iso-propyl Alcohol:

During an exposure period of 77 days, no SCC developed for 2 lots (3 specimens each) of 2014-T6 in iso-propyl alcohol, although it was observed that a black network or web-like patches developed on the surface of one test specimen near the end of the test period. In the opinion of the inves-

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tigator, these black network patches are probably due to early stages of galvanic corrosion as the protective coating (Maskcoat) exhibited poor adhesion by pulling away from the surface of the c-ring, resulting in discontinuation of testing.

2014-T6 and Distilled Water:

An exposure period (353 days) in demineralized distilled water produced no failures for this alloy, however it was noted that some surface pitting occurred during this test.

2219-T87 and 3.5% NaCl:

Attempts were made to establish an indication of a threshold stress level for 2219-T87 by exposing three lots (4 specimen each) at stress levels of 25%, 50% and 75% yield strength. Each lot were exposed continuously for a period of 79 days in 3.5% NaCl at pH 7. Some pitting was observed on the surface of stressed c-rings, however, no stress corrosion failures were recorded [see Figure 12].

A comparison of the probable survival rate of 2219-T87 and 7075-T651 in chloride solutions at 75% Y.S. is illustrated by Figure 11. It is seen that 2219-T87 has a 100% rate of survival for twelve test specimens (4 specimens per data point) while 7075-T651 rate of survival is less (66%). This comparison was made because of the high SCC resistance displayed by these alloys in tests conducted in this study.

2014-T651 and 3.5% NaCl:

This alloy (10 specimens) showed some susceptibility to SCC. Cracks occurred mainly on the compressive side (inner surface) of c-ring with *p* few cracks initiating on tension surface of test specimen. The direction of cracks were perpendicular to direction of stress. Failures were recorded between 11 and 14 days.

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2014-T651 in Ethyl and Iso-propyl Alcohol:

During 108 days of constant immersion at 75% Y.S. stress, no failures were observed, nor did the organic fluids cause any general corrosion or pitting. Three c-rings were exposed to ethyl alcohol and two specimens were tested in iso-propyl alcohol.

2014-T651 and Methyl Alcohol:

Four lots (10 specimens) were exposed continuously to methyl alcohol, with some corrosive attacks occurring after 24 hours exposure. A black deposit of corrosion product typically accumulated at site of pitting and cracking. Small cross-section samples were observed under the light microscope to verify the nature of cracking. It was concluded that the small surface cracks were intergranular.

Interpretation of time-to-failure data was made by plotting on logarithmic probability paper, time-to-failure (days) versus cumulative percentage. Again the technique of Lewis [3] and Booth et.al. [4] was used. This method resulted in an estimated <u>population</u> standard deviation of 3.18 days and a popululation average of 5.8 days. This compares to a <u>sample</u> standard deviation of 2.76 days and a <u>sample</u> average of 6.3 days [see Table VI]. Applying the student t-test for both population and sample average indicated that these differences were significant. With 7 degrees of freedom, evaluation of the time-to-failure data resulted in a t-value (.48) falling within the critical value of \pm 1.90 which is interpreted for 95% confidence interval as statistical significant [see Figure 13].

7075-T651 and 3.5% NaCl:

The stress corrosion performance for this alloy was conducted by exposing continuously, 4 lots (3 specimens each) to a 3.5% NaCl solution. The

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surface appearance of 7075-T651 during exposure, can be described by a heavy build-up of dark corrosion product that progressively increases with time. This problem causes difficulty in making visual examination for cracks.

In contrast to 2014-T6, it was noted that after crack initiation no additional secondary cracks occurred. This was probably due to the change in stress level to the extent that no additional parallel cracks developed.

Time-to-failure data for 7075-T651 was treated statistically on the basis of a normal distribution plot where the time-to-failure (or endurance) was plotted against the cumulative percentage on logarithmic probability paper.[Figure 14]. From this method the estimated <u>population</u> standard deviation (as determined from graph) was 7.0 days, whereas, the population average was 18 days. On comparing the same kind of data for the sample, the standard deviation was 8.7 days and the sample average was 17.9 days [Table V]. Application of the student t-test for the <u>population</u> average and <u>sample average</u> indicated that these differences were significant. With 5 degrees of freedom, evaluation of the time-to-failure data resulted in a t-value (.03) falling within the critical value of \pm 2.13 which is interpreted for 95% confidence interval as statistical significant.

To determine a representative value of the stress corrosion crack width, 80 measurements were made at random points on the surface of 6 specimens which failed in NaCl solution. It was found that the average crack width was .12 mm. Figure 14 shows a frequency distribution of crack width measurements for this alloy while Figure 16 and 17 illustrates electron micrographs of the replicated surface along crack regions.

7075-T651 and Methyl Alcohol:

One failure was recorded after an exposure period of 103 days, however, it is doubtful that this failure is due to stress corrosion. Galvanic effects

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were probably the cause of failure, as plastic protective coating showed evidence of failure.

7075-T651 in Ethyl Alcohol, Iso-propyl Alcohol:

An exposure period of 244 days for three test specimens in ethyl alcohol produced no failures, while two failures occurred in 87 and 140 days respectively, for 7075-T651 in iso-propyl alcohol. These failures are interpreted as being caused by galvanic corrosion and not stress corrosion mainly because the Maskcoat failed.

7075-T651 and Distilled Water:

No failures were recorded for an exposure period of nine months (273 days) in demineralized distilled water although some pitting corrosion was observed.

TITANIUM 6A1-4V

3.5% NaCl, Methyl Alcohol, Iso-propyl Alcohol, Ethyl Alcohol, and Distilled Water:

No titanium failures were observed for a constant immersion period of 310 days to a 3.5% NaCl solution. However, it was observed that two failures occurred (two lots) for continuous exposure to methyl alcohol. These failures were observed at 15 hours (.6 days) and 55 hours (2.2 days) respectively.

During the same time period (310 days), no failures were observed for titanium in fluids of ethyl alcohol, iso-propyl alcohol and distilled water.

CONCLUSION

- 1. Stress-corrosion cracks in 2014-T6, 7075-T651 c-rings initiates primarily along top surface (region of maximum tension stress) and perpendicular to direction of stress.
- 2. Stress corrosion crack widths for 2014-T6 exposed to NaCl was smaller and more numerous than 7075-T651 in the same environment.
- 3. 2014-T6 is susceptible to SCC in chloride environments, but appears to be resistant in methyl alcohol, ethyl alcohol, iso-propyl alcohol, and demineralized distilled water.
- 4. 2219-T87 resists cracking in 3.5% NaCl.
- 5. 2014-T651 is susceptible to SCC in sodium chloride solution, but is highly resistant to cracking in ethyl and iso-propyl alcohol.
- 6. 7075-T651 is susceptible to SCC in 3.5% NaCl.
- 7. 7075-T651 appears to be resistant to SCC in methyl, ethyl, and isopropyl alcohols.
- 8. Ti 6A1-4V is resistant to stress corrosion in 3.5% NaCl.
- 9. Ti 6A1-4V showed some susceptibility to SCC in methanol, but is resistant to stress corrosion in ethanol and iso-propyl alcohol.
- 10. 2014-T6, 2014-T651, 7075-T651, 2219-T87, and Ti 6A1-4V showed a high degree of stress corrosion resistance in demineralized distilled water.

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APPENDICES

- 1. Tables
- 2. Figures

TABLE I

CHEMICAL COMPOSITION (Composition Weight %)

Alloy	A1	>	Si	Fe	8	Ę	or M	ઠા	R N	ц.	ĪN	Zr
Aluminum 2014-T6	ľ		• 80	Í L	t1° t1	• 80	1 0.		₽ ₽ ₽	I I I	8	1
Aluminum 7075-T651	t L	\$ 1. 1	.12	•20	1.78	•0•	2.48	2.0	5 . 8	ł	1	ļ
Aluminum 2219-T87	ł	.10	60 *	• 22	6.24	.26	• 00	• 00	• 06	•06	10.	.15
Aluminum 2014-T651	ł	1 1 1	• 92	3 ° 8	tı • tı	•74	•57	• 03	.18	•03	00.	
Titanium 6Al-4V	9	4	1	1			1	ľ	8	8 9 1	8	

TABLE II

MECHANICAL PROPERTIES

Alloy	Form	Directionality	Tensile Strength 1 x 10 ³ psi	Yield Strength 1 x 10 ³ psi	Percent Elongation
Aluminum 2014-T6	Bar	S.T	84	76	15
Alumînum 7075-T651	Bar	S.T	80	73	œ
Aluminum 2219-T87	Plate	S.T	71.8	60	JO
Aluminum 2014-T651	Plate	S.T	70.8	63.8	7.3
Titanium 6Al-4V	Sheet	1	138	128	12

111	+++
TARTE	

STRESS CORROSION TEST RESULTS

Days re to o Failure	5 2-5	8	1		8	2 4-21		-4x	i i i		1 5 1		ie.				-rings tension ant immersion
Failu Rati	15/1:	0/6	2/3	0/6	0/2	8/1	1/8	1/6	0/3	0/2	4/0	×	×	×		0/2	l inch c- constant e-consta
Yield Strength 1 x 10 ³ psi	68	68	68	68	68	73	73	73	73	73	58	×	×	×		58	Test Data Specimen size-1 Stress method-o Type of exposur
Applied Stress (75%) 1 x 10 ³ psi	51	51	51	51	51	54	2th C	54	54	54	45	×	×	×		45	Notes: a. prrosion b.
Stress Direction	S.T	S.T	S.T	S . T	S.T	S . T	S.T	S.T	S.T	S.T	S.T	Xř	×	×		S.T	o galvanic cc
Visual Examination (Film)	Gray	None	None	None	Dull	Dark Gray	None	None	None	Dull	Gray	X	×	×	Dark	Gray	iable attack ular robably due t
Type of Attack	I+d	N.A	N.A	N.A	۵.	I+d	N.A	N.A	N.A	Ч	ρ.,	×	×	×		ዋ	No apprec Intergran Failure p Pitting
Solution	3.5% NaCl	Methyl Alcohol	Ethy1 Alcohol	Iso-propyl Alcohol	Distilled Water	3.5% NaCl	Methy1 Alcoho1	Ethyl Alcohol	Iso-propyl Alcohol	Distilled Water	3.5% NaCl	Methyl Alcohol	Ethyl Alcohol	Iso-propýl Alcohol	Distilled	Water	Notes: N.A.A = I = * = P =
Test Specimen	c-ring					c-ring					c-ring						
Alloy	2014-T6					7075-T651					2219-T87						

P = Pitting
S.T = Short transvense dinantian

TABLE III^a

STRESS CORROSION TEST RESULTS

Alloy	Test Specimen	Solution	Type of Attack	Visual Examination (Film)	Stress Direction	Applied Stress (75%) 1 x 10 ³ psi	Yield Strength I x 10 ³ psi	Failure Ratio	Days of Failure
2014-T651	c-ring	3.5% NaCl	I+d	Dark Gray	S.T	48	63.8	7/10	10-12
)	Methyl Alcohol	I+d	None	S.T	84	63.8	01/01	3-10
		Ethyl Alcohol	N.A	None	S.T	48	63.8	0/3**	
		Iso-propyl Alcohol	N.A	None	S.T	48	63.8	0/2**	
		Distilled Water	ቤ	Dull	S.T	48	63.8	0/2	
		÷.							
Titanium	u-bend	3.5% NaCl	N.A	Gray	S.T			6/3	
		Methyl Alcohol	н	Gray	S.T			5/9	1-121
		Ethyl Alcohol	N.A	Gray	S.T			0/6	1
		Iso-propyl Alcohol	N.A	Gray	S.T			0/6	
		Distilled			8				
		Water	N.A	VEY.E)	с У			7/0	1171

**Test determined after 108 day exposure.

		STATISTIC	AL SCC RESULTS P	OR ALUMINUM 2014-T6	in 3.5% NaCl	
Alloy	No. of Tests	Sample Avg (days)	Estimated Population Average(days)	Sample Standard Deviation(days)	Estimated Population Stand ar d Deviation(days)	Possib le Error
A1 2014-T6	15	3°0	3.7	1.14	+0 -	.51%
	The probat shown.	ility is .95	that the sample	average failure-time	is not in error by more	than <u>+</u> percent
		STATISTICA	L SCC RESULTS FO	TABLE V R ALUMINUM 7075-T651	in 3.5% NaCl	
Alloy	No. of Tests	Sample Avg (days)	Estimated Population Average(days)	Sample Standard Deviation(days)	Estimated Population Standard Deviation(days)	Possible Error
A1 7075-T651	9	17.9	18	8.7	7.0	33. 2%
-	The probat shown.	ility is .95	that the sample	average failure-time	is not in error by more	than <u>+</u> percent
			F	ABLE VI		
		STATISTICA	L SCC RESULTS FO	R ALUMINUM 2014-T651	in Methanol	
Alloy	No. of Tests	Sample Avg (ḋays)	Estimated Population Average(days)	Sample Standard Deviation(days)	EstimatedPopulation Standard Deviation(days)	Possible Error
A1 2014-T651	60	6 • 3	5.8	2.76	3.18	17.9%
	The probab	ility is .95 1	that the sample a	avg. failure-time is	not in error by more that	an + percent shown.

TABLE IV





IMMERSION TYPICAL CHAMBER USED FOR CONSTANT CORROSION TESTING STRESS F1G. 1

NANOS NANO



S N N N N AFTER EXPOSURE

C-RING

F1G.2









Fig.6a Typical machining grooves on c-ring surface of 2014-T6

before exposure.

150x



Fig. 6b Light micrograph along crack region of 2014-T6

in NaCl.



Fig.7 Microstructure of 2014-T6 showing stress corrosion cracking. Keller's Etch 15

150x

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Fig. 8Microstructure of 2014 - T6 showing stress corrosion, cracks,
and inclusions. Keller's Etch600x

ALUMINUM 2014-T6



Fig. 9. Electron micrograph of surface replica of 2014-T6 showing stress corrosion cracks. Extensive pitting sites can be seen. 3000x



Fig.10b

Fig.10(a,b) Electron micrograph of surface replica of 2014 -T6 showing pitting and machining grooves. Keller's Etch 1000x













Stress

Stress

Fig.16a.. Light micrograph of SCC across thickness dimension of c-ring sample failed in 3.5% NaCl. Dark ridges are machining grooves. 600x



Fig. 16b. Light micrograph of corroded surface of 7075-T651 (3.5% NaCl) showing grain boundary and numerous corrosion pits. Keller's Etch. 600x

ALUMINUM 7075-T651



Fig.17. Electron micrograph of replicated surface of 7075-T651 showing numerous cracks and associated pitting. 1500x