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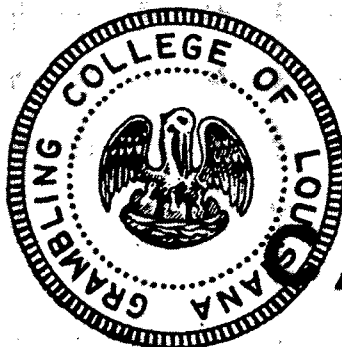
NASA Research Grant NGR 19-011-009

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

Annual Report
 for the Period
 June 1, 1971 to June 1, 1972

by
 Jethro Terrell

Grambling College Grambling, La. 71245



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 PHYSICS
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 Grambling, Louisiana**

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ABSTRACT

The effect of a constant applied stress in crack initiation of aluminum 2014-T6, 7075-T651 and titanium 6Al-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 6) and organic fluids of ethyl, methyl, and iso-propyl alcohol (reagent purity).

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, a similar observation was not noted for titanium stressed specimens.

Results of this investigation seems to suggest that aluminum 2014-T6, aluminum 7075-T651 are susceptible to stress corrosion cracking in chloride solution (NaCl); while they (both alloys) seem to resist stress corrosion cracking in methyl alcohol, ethyl alcohol, iso-propyl alcohol, and demineralized distilled water.

Titanium 6Al-4V showed some evidence of susceptibility to SCC in methanol, while no such susceptibility was exhibited in ethanol, iso-propyl alcohol and demineralized distilled water.

INTRODUCTION

The development of many high strength alloys to meet demands of various service requirements has resulted in an ever increasing need to acquire data on the stress corrosion behavior of metals in different environments. Consequently, it is within these broad demands that the objective of this research project was directed, to acquire data on the time-to-failure characteristics of Aluminum 2014-T6, Aluminum 7075-T651 and Titanium 6Al-4V in several environments.

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 relatively 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].

Stress corrosion test specimens for aluminum was c-rings and titanium was u-bend shape. The aluminum c-rings were stressed short transversely to a pre-determined stress level of 75% of its yield strength (Y.S.), while the titanium test specimen was bent until both walls were parallel. Test specimens were exposed continuously (until failure or until test was discontinued) to the following fluids (reagent purity) 3.5% NaCl, methyl alcohol, ethyl alcohol, iso-propyl alcohol and demineralized distilled water. Examination

of surface crack region was accomplished through the use of the light microscope and electron microscope.

Results of this study suggest that Aluminum 2014-T6, Aluminum 7075-T651 are susceptible to stress corrosion cracking in chloride solution (NaCl); while they (both alloys) seem to resist stress corrosion cracking in methyl alcohol, ethyl alcohol, iso-propyl alcohol and demineralized distilled water.

Titanium showed some susceptibility to SCC in methanol, while no such susceptibility was exhibited in ethanol, iso-propyl alcohol, and demineralized distilled water.

MATERIALS

Chemicals

Alloys [see table 1] used in this investigation were of three types. 2014-T6 representing the Al-Cu type, 7075-T651 representing the Al-Zn-Mg-Cu type and Titanium 6Al-4V.

Test specimens were obtained from NASA-MSFC. Aluminum c-rings (1 inch diameter) were cut from short transverse section of bar stock. Titanium test specimens were u-bend shape.

Equipment

Light microscopy was conducted with an Instrom MMU metallurgical microscope with a magnification range between 75x and 600x. An RCA Model EMU-3 was used for electron microscopy while vacuum evaporation was done with an EFFA vacuum evaporator.

EXPERIMENTAL PROCEDURE

Aluminum c-rings were tested as machined with no further surface preparation, other than degreasing and cleaning by dipping in acetone. Prior to testing, the necessary parameters (i.e. wall thickness, modulus of elasticity, mean diameter, desired stress [psi], etc.) were determined. This information was substituted into the c-ring stressing formula [2] and the c-rings were stressed (by tightening bolt) to 75% Y.S. Galvanic effects were prevented by coating each test specimen with a hot strippable plastic coating (Maskcoat #2)*.

Titanium test specimens were dipped in an etching solution (2ml HF + 10ml HNO₃ + 88ml H₂O) and subsequently flooded with an acetone jet stream to remove traces of the oxide scale.

Examination for Cracks

During periods of testing, daily observations 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. The number of visual inspections made, varied from two to four.

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)***

*Obtained from Western Coating Co. - Royal Oak, Michigan

**except on weekends

***available from Ernest F. Fullam Co. - Schenectady, N. Y.

and dissolving tape directly onto the surface of the metal with about two drops of acetone-methanol mixture. After drying, the replica was stripped from the surface of the test 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°. At this point, the replica can be prepared for electron microscopy.

RESULTS

2014-T6 and 3.5% NaCl

With c-ring stressed specimens of 2014-T6 in 3.5% NaCl at pH 6, primary cracks were observed to initiate along tension surface (region of maximum tension stress) and at base of random corrosion pit sites. Also, it is seen that cracking propagates perpendicular to the direction of stress. Microscopic examination reveal many secondary cracks running parallel to the primary (major) crack region. Typical cracks are illustrated by light micrograph in Fig. 6. Additional SCC cracks are shown by electron micrographs in Fig. 7 and 8.

In order to interpret 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 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. This method resulted in an estimated popu-

lation standard deviation of .04 days and a population average of 3.7 days. This compares to a sample standard deviation of 1.14 days and a sample average of 3.9 days [see table A].

A t-test for the population average and sample 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 ± 1.83 for 95% confidence level, which indicates statistical significance for this kind of analysis.

Fig. 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.

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. Fig. 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 noted 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—as determined by 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. This coating will be used in the continuing research project. The remaining specimens will be tested with neoprene protective coating. Therefore, because of this problem—failure of protective coating—no SCC data was successfully acquired 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 occurred 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 investigator, these black network patches are probably due to early stages of galvanic corrosion as the protective coating (Maskcoat) begin to fail by pulling away from the surface of the c-ring, causing testing to be discontinued. Consequently, no failures were recorded, even during the valid test period.

2014-T6 and Distilled Water

A nine month exposure period (273 days) in demineralized distilled water produced no failures for this alloy, however it was noted that some surface pitting occurred during this test.

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 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 [Fig. 9]. From this method the estimated population 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 5]. Application of the student t-test for the population average and sample average 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 ± 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. Fig. 10 shows a frequency distribution of crack width measurements for this alloy while Fig. 11 and 12 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 was 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 were observed.

TITANIUM 6Al-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 and 55 hours 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.

Longer exposure periods were possible through the use of Parawax as a coating. This was necessary as experience with Maskcoat coating with aluminum test specimen in organic fluids resulted in galvanic corrosion.

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 exposure 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. 7075-T651 is susceptible to SCC in 3.5% NaCl.
5. 7075-T651 appears to be resistant to SCC in methyl, ethyl, and iso-propyl alcohol.
6. Ti 6Al-4V is resistant to stress corrosion in 3.5% NaCl.
7. Ti 6Al-4V showed some susceptibility to SCC in methanol, but is resistant to stress corrosion in ethanol and iso-propyl alcohol.
8. 2014-T6, 7075-T651, and Ti 6Al-4V showed a high degree of stress corrosion resistance in demineralized distilled water.
9. During the current research project, a new protective coating (liquid neoprene) will be used for time exposure studies with aluminum c-rings in organic fluids—methyl, ethyl, and iso-propyl alcohol. This change is necessary to reduce the incidence of failure due to galvanic effects.

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APPENDICES

1. Tables

2. Figures

TABLE 1

CHEMICAL COMPOSITION
(Composition Weight %)

<u>Alloy</u>	<u>Al</u>	<u>V</u>	<u>Si</u>	<u>Fe</u>	<u>Cu</u>	<u>Mn</u>	<u>Mg</u>	<u>Cr</u>	<u>Zn</u>	<u>Ti</u>	<u>Zr</u>
Aluminum 2014-T6	--	--	.80	--	4.4	.80	.01	--	--	--	--
Aluminum 7075-T651	--	--	.12	.20	1.78	.04	2.48	2.0	5.8	--	--
Titanium 6Al-4V	6	4									

TABLE 2

MECHANICAL PROPERTIES

<u>Alloy</u>	<u>Form</u>	<u>Directionality</u>	<u>Tensile Strength</u> <u>1 x 10³ psi</u>	<u>Yield Strength</u> <u>1 x 10³ psi</u>	<u>% Elongation</u>
Aluminum 2014-T6	Bar	S.T.	84	76	15
Aluminum 7075-T651	Bar	S.T.	80	73	8
Titanium 6Al-4V	Sheet	----	138	128	12

S.T. = Short transverse direction

TABLE 3
STRESS CORROSION CRACKING TEST RESULTS

Alloy	Test Specimen	Solution	Type of Attack	Visual Examination (Film)	Stress Direction	Applied Stress (75%) 1x10 ³ psi	Y.S. 1x10 ³ psi	Failure Ratio	Days to Failure		
2014-T6	c-ring	3.5% NaCl	P + I	Gray	S.T.	51	68	15/15	2 - 5		
		Methyl Alcohol	N.A.	None	S.T.	51	68	0/6	---		
		Ethyl Alcohol	N.A.	None	S.T.	51	68	2/3*	---		
		Iso-propyl Alcohol	N.A.	None	S.T.	51	68	0/6	---		
		Distilled Water	P.	Dull	S.T.	51	68	0/2	---		
		7075-T651	c-ring	3.5% NaCl	P + I	Dark Gray	S.T.			8/12	4 - 21
				Methyl Alcohol	N.A.	None	S.T.	54	73	1/8*	---
Ethyl Alcohol	N.A.			None	S.T.	54	73	1/6*	---		
Iso-propyl Alcohol	N.A.			None	S.T.	54	73	0/3	---		
Distilled Water	P.			Dull	S.T.	54	73	0/2	---		
Ti 6Al-4V	u-bend			3.5% NaCl	N.A.	Gray	S.T.			0/3	---
				Methyl Alcohol		Gray	S.T.			4/6	1 - 2
		Ethyl Alcohol	N.A.	Gray	S.T.			0/3	---		
		Iso-propyl Alcohol	N.A.	Gray	S.T.			0/3	---		
		Distilled Water	N.A.	Gray	S.T.			0/1	---		

Notes: N.A. = No appreciable attack
I = Intergranular
* = Failure probably due to galvanic corrosion

Notes: Test Data
a. specimen size - 1 inch c-ring
b. stress method - constant tension
c. type of exposure - constant immersion

TABLE 4

STATISTICAL SCC RESULTS FOR ALUMINUM 2014-T6 in 3.5% NaCl

Alloy	No. of Test	Sample Ave. (days)	Estimated Population Average (days)	Sample Standard Deviation (days)	Estimated Population Standard Deviation (days)	Possible Error
Al 2014-T6	15	3.9	3.7	1.14	.04	.51%

The probability is .95 that the sample average failure-time is not in error by more than \pm percent shown.

TABLE 5

STATISTICAL SCC RESULTS FOR ALUMINUM 7075-T651 in 3.5% NaCl

Alloy	No. of Test	Sample Ave. (days)	Estimated Population Average (days)	Sample Standard Deviation (days)	Estimated Population Standard Deviation (days)	Possible Error
Al- 7075-T651	6	17.9	18	8.7	7.0	33.2%

The probability is .95 that the sample average failure-time is not in error by more than \pm per cent shown.

STRESS CORROSION TEST CHAMBER

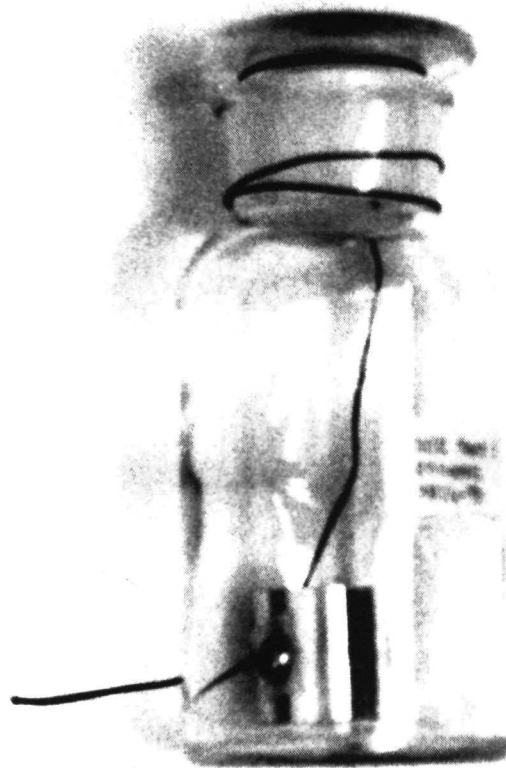
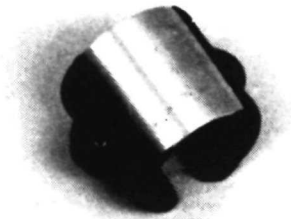


FIG. 1 TYPICAL CHAMBER USED FOR CONSTANT IMMERSION
STRESS CORROSION TESTING

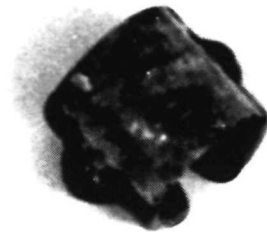
STRESS CORROSION TEST SPECIMEN

PREPARED FOR EXPOSURE



C-RING

AFTER EXPOSURE



C-RING

Fig. 2

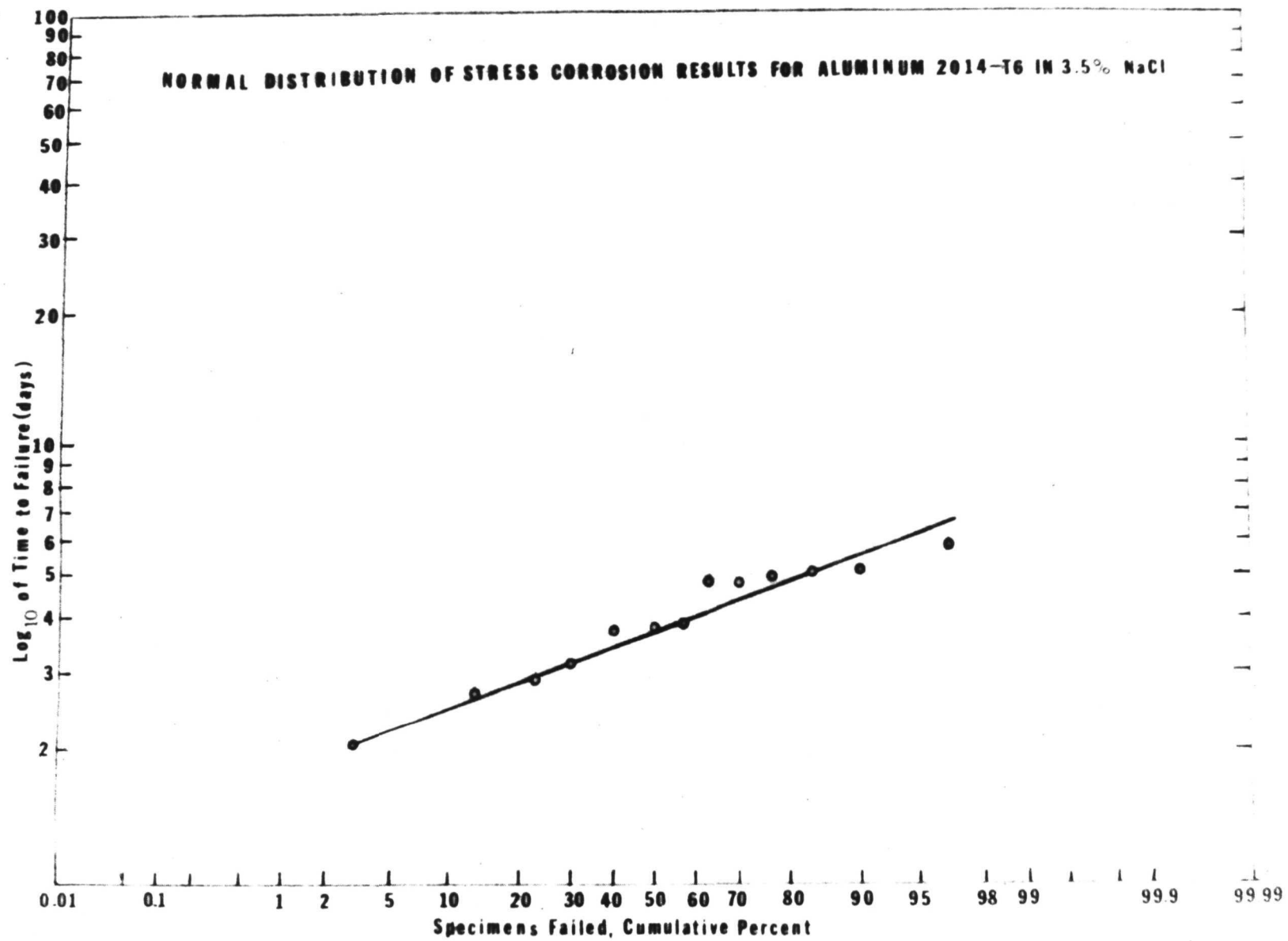


Fig. 3 Probable distribution of failure time for 2014-T6 c-ring stressed short transversely to 75% Y.S.

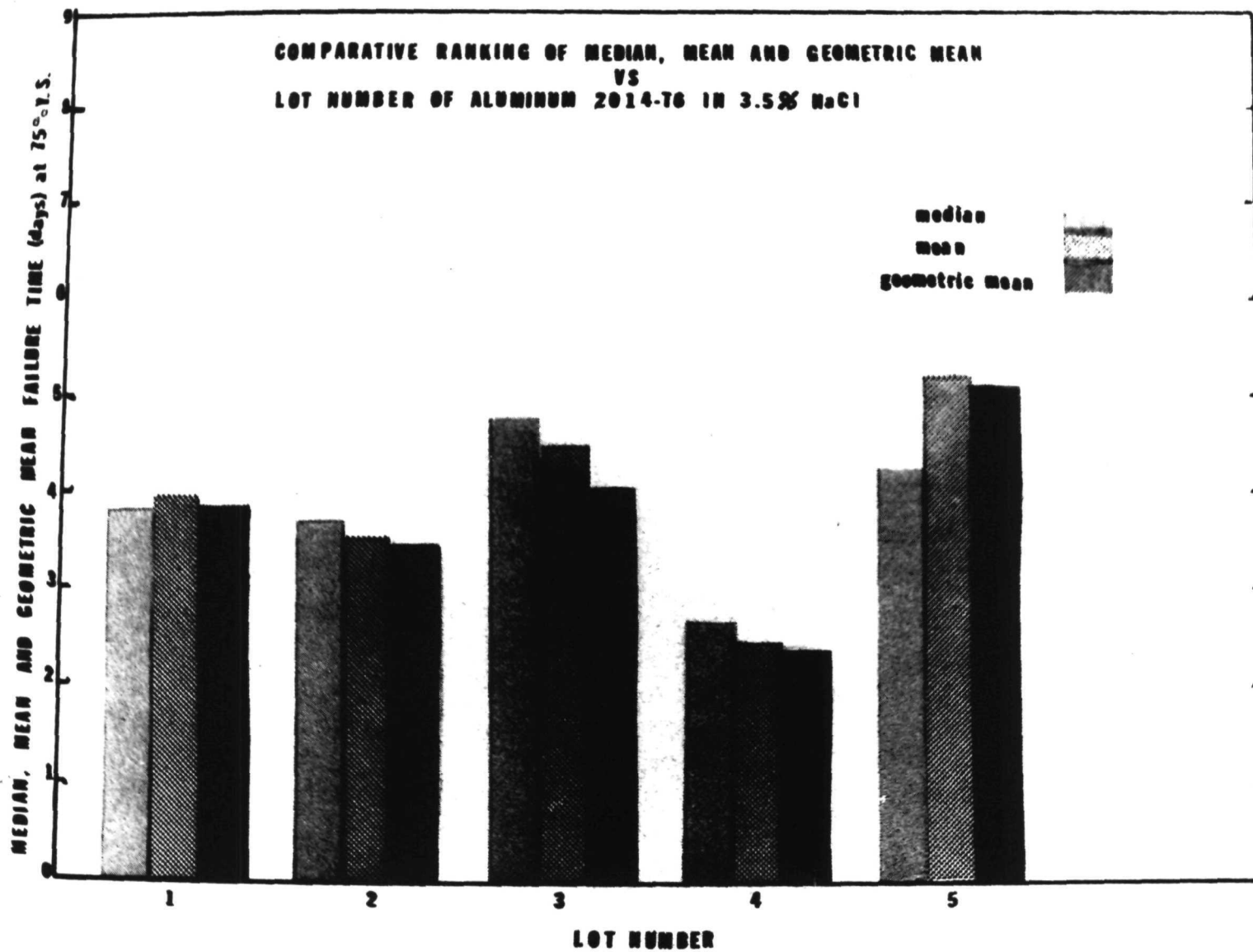


Fig.4 Comparison of median, mean, and geometric mean failure time for 5 lots(3 specimen /lot)

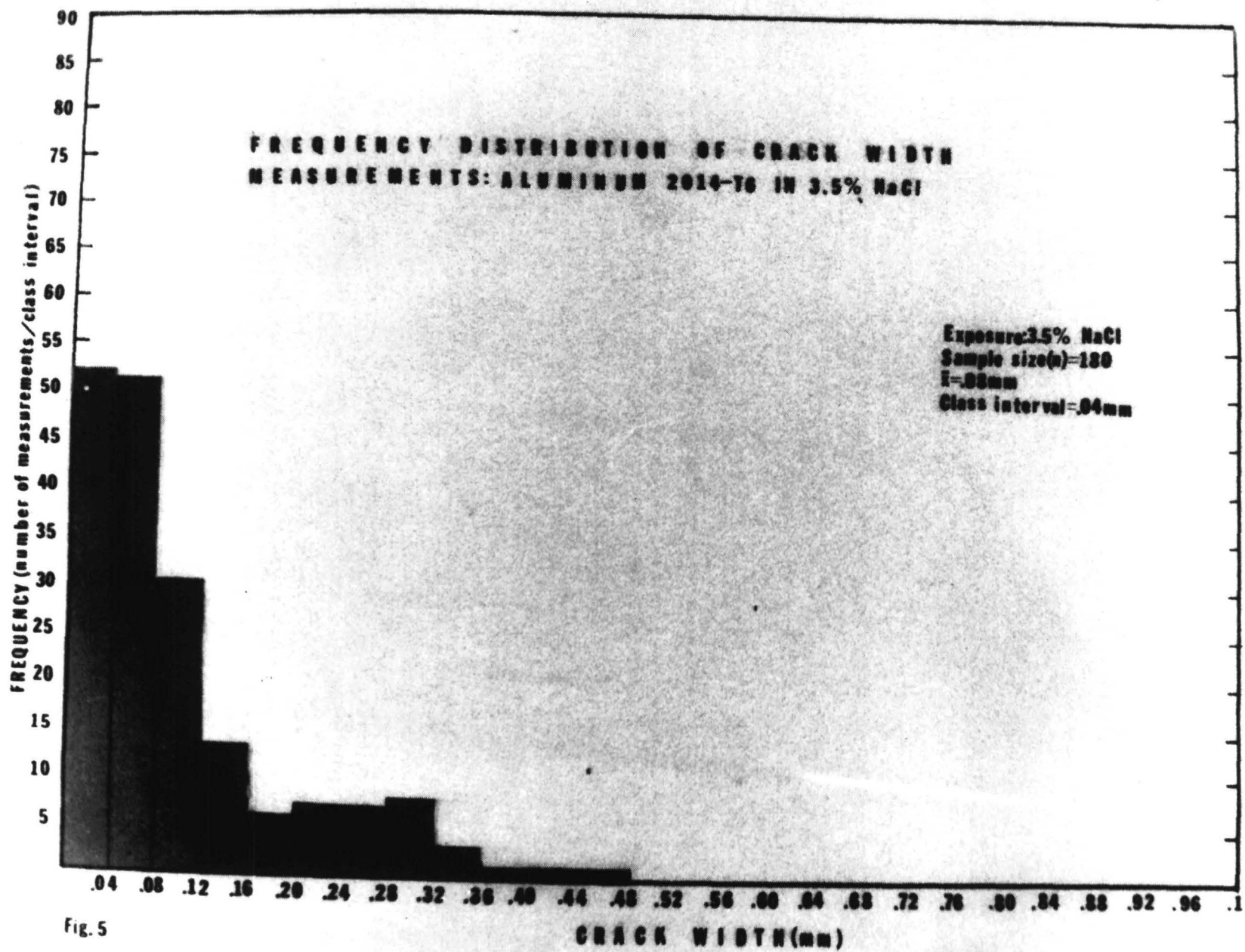
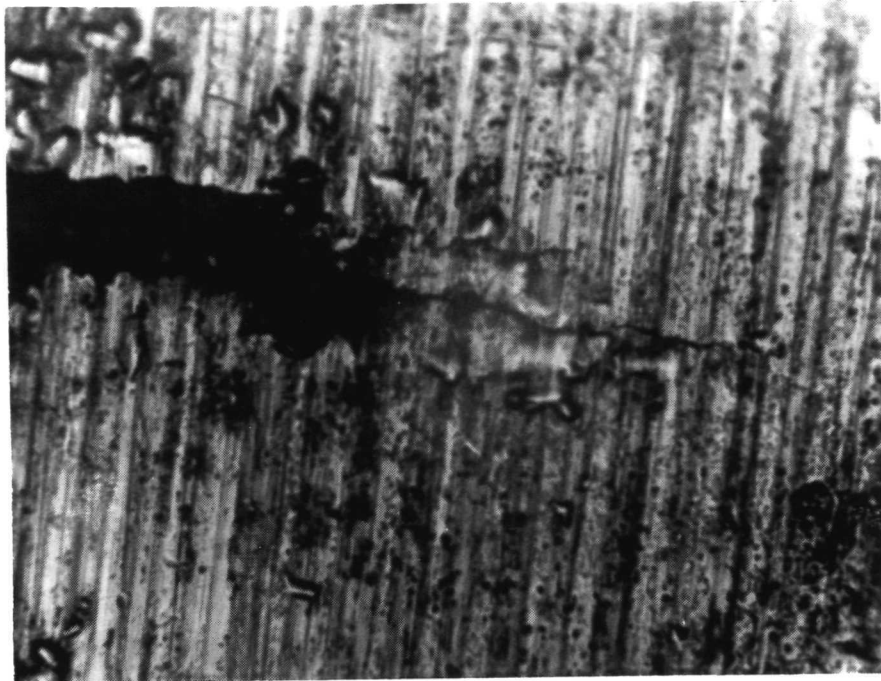


Fig. 5

ALUMINUM 2014-T6

↑ Stress ↓



↑ Stress ↓

Fig. 6a Light Micrograph of 2014-T6 SCC. Dark round features are corrosion pits and slightly vertical ridges are machining grooves. 150x

↑ Stress ↓



↑ Stress ↓

Fig. 6b Light Micrograph of 2014-T6. Crack region perpendicular to direction of stress. 150x

ALUMINUM 2014-T6

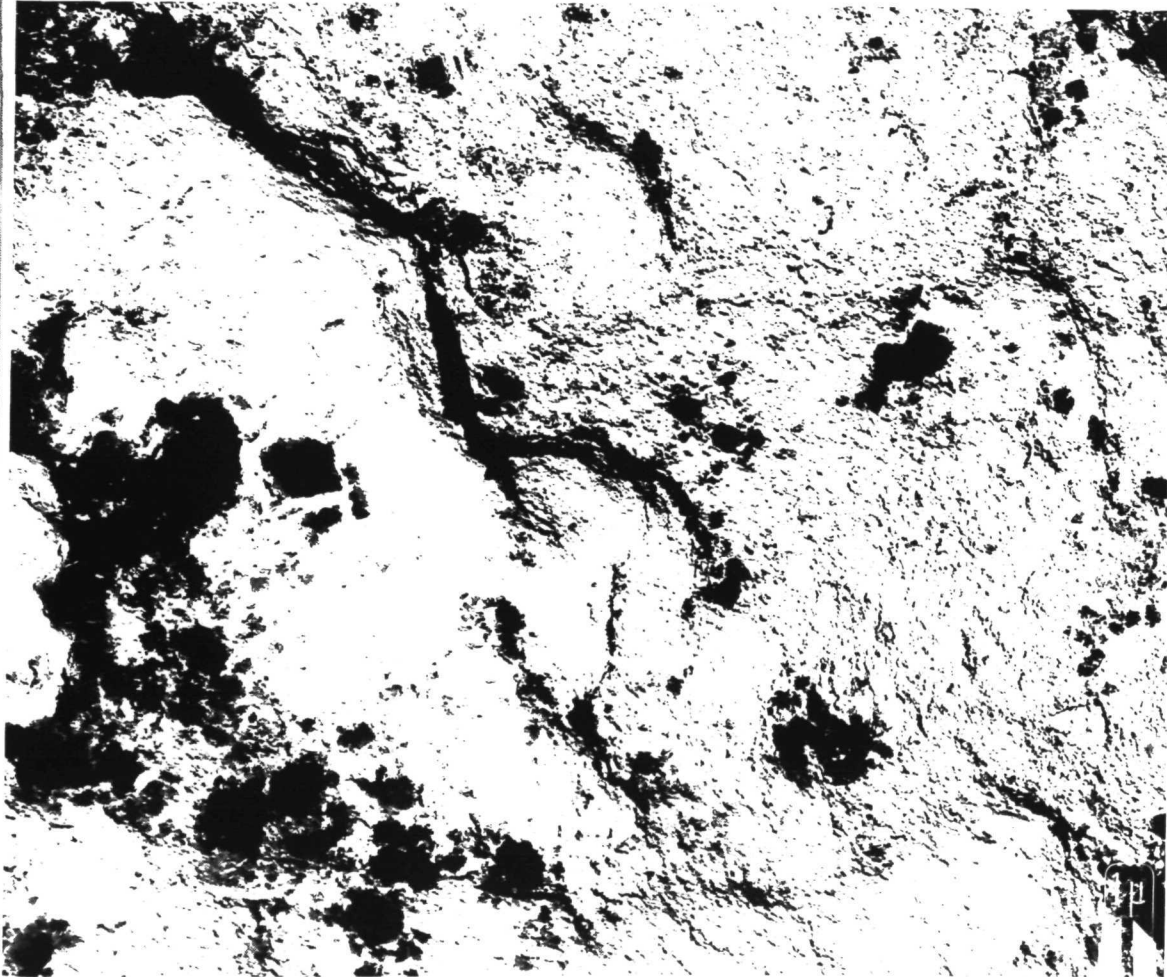


Fig. 7 Electron micrograph of surface replica of 2014-T6 showing stress corrosion cracks. Extensive pitting sites can be seen. 3000x

ALUMINUM 2014-T6

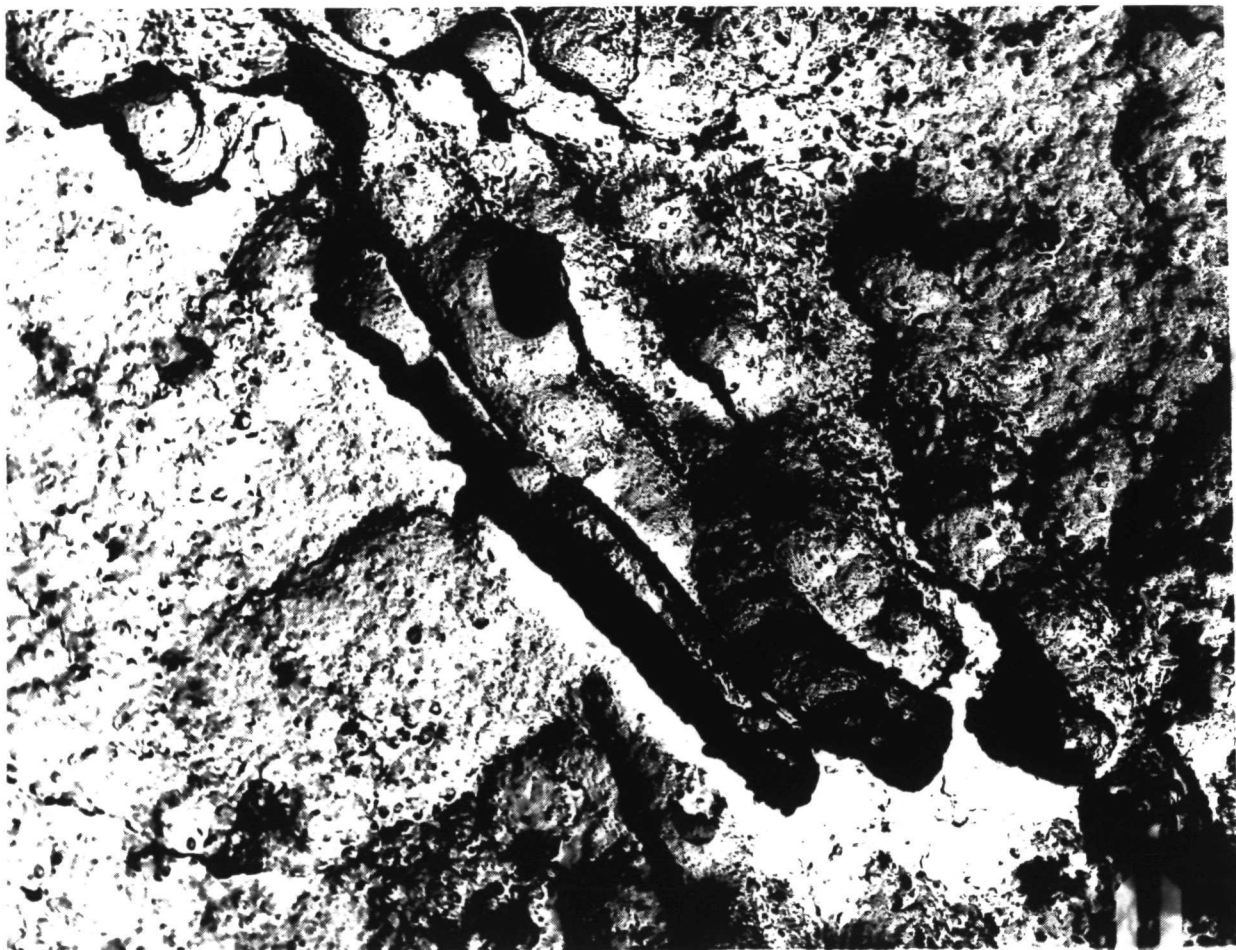


Fig. 8 Electron micrograph of replicated surface of aluminum 2014-T6 showing an intergranular crack (diagonal across center). 3000x

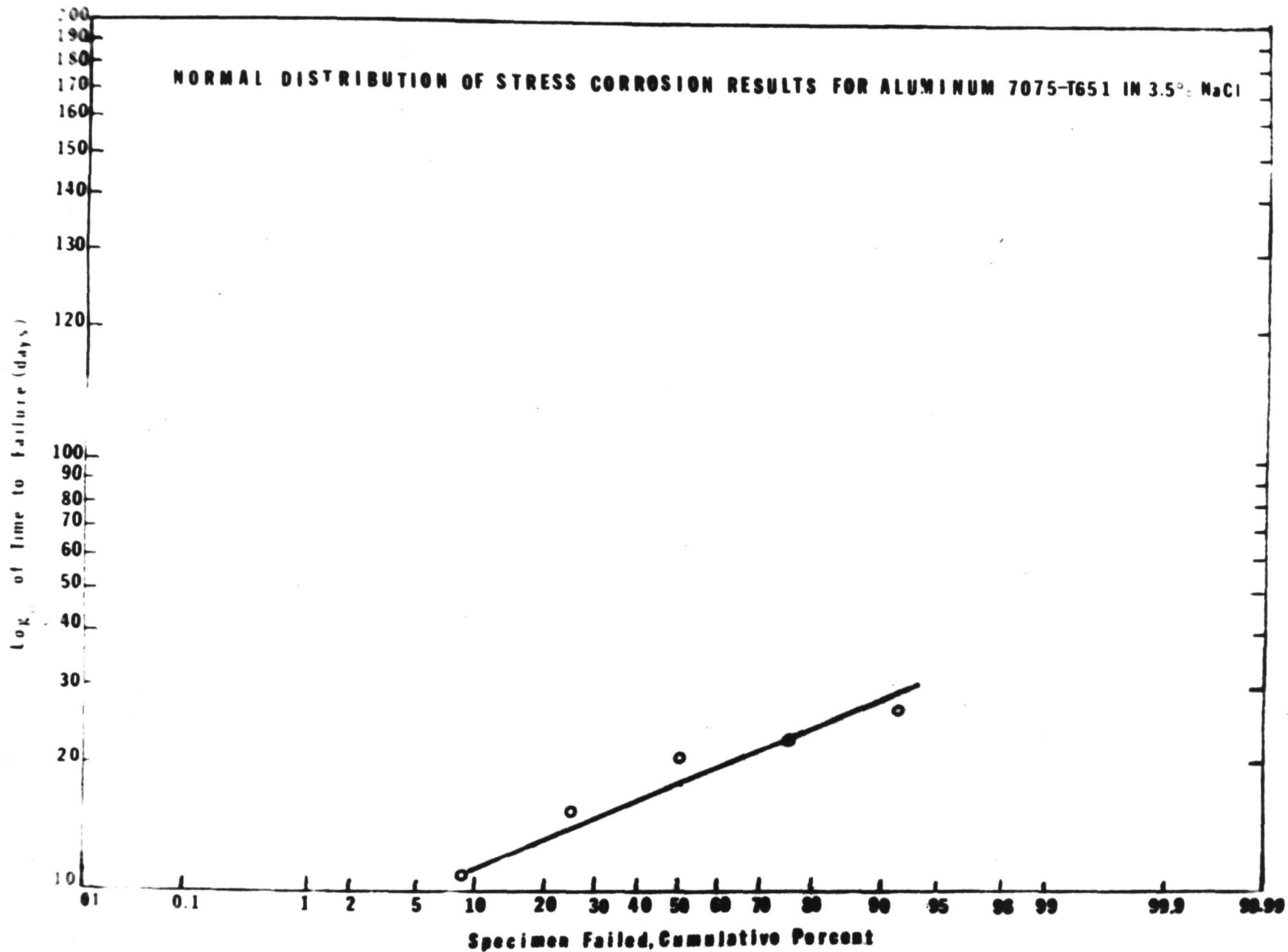


Fig 9 Probable distribution of failure time for 7075-T651 c-ring stressed short transversely to 75% Y.S.

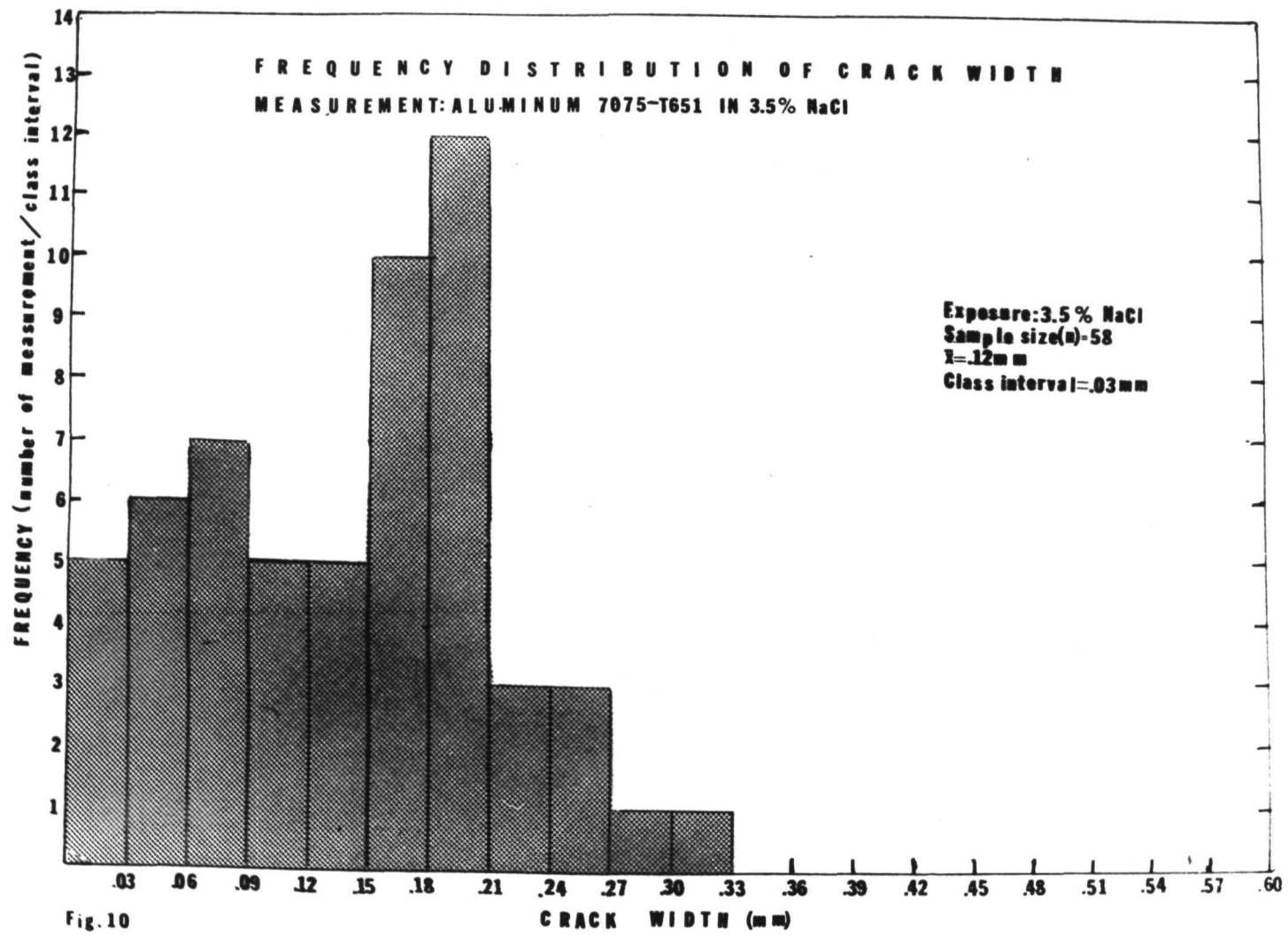
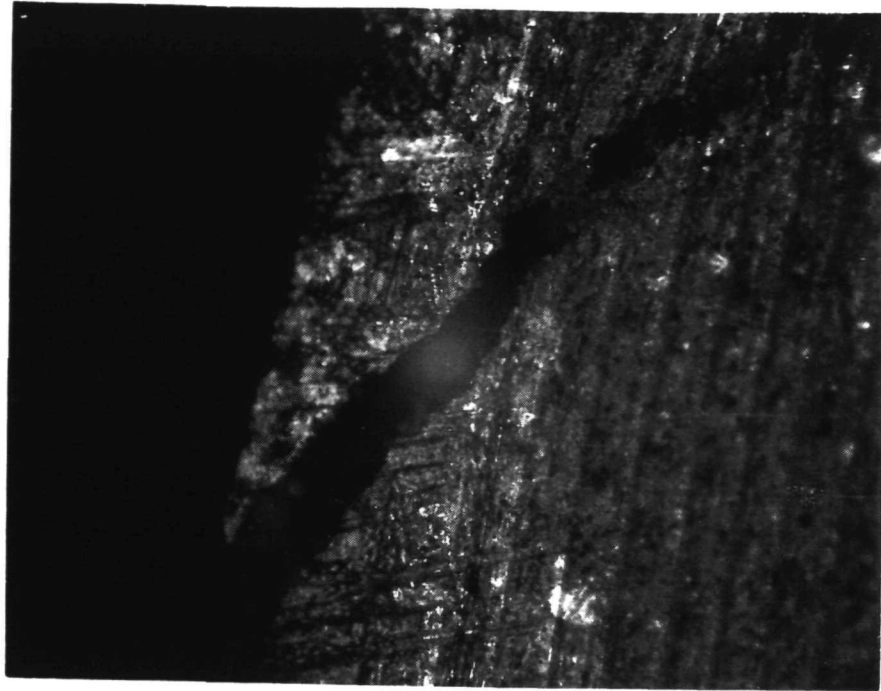


Fig. 10

ALUMINUM 7075-T651

↑
Stress
↓



↑
Stress
↓

Fig. 11a. Light micrograph of SCC across thickness dimension of c-ring sample failed in 3.5% NaCl. Dark ridges are machining grooves. 600x

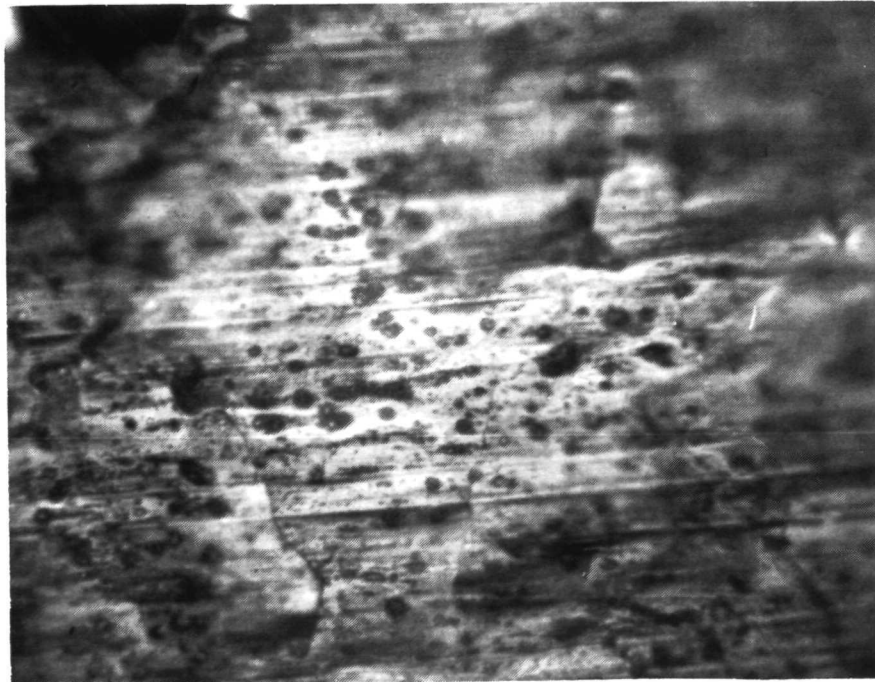


Fig. 11b. Light micrograph of corroded surface of 7075-T651 (3.5% NaCl) showing grain boundary and numerous corrosion pits. Keller's Etch. 600x

ALUMINUM 7075-T651



Fig. 12 Electron micrograph of replicated surface of 7075-T651 showing stress corrosion crack running diagonally (continuous dark paths). 3000x

ALUMINUM 7075-T651

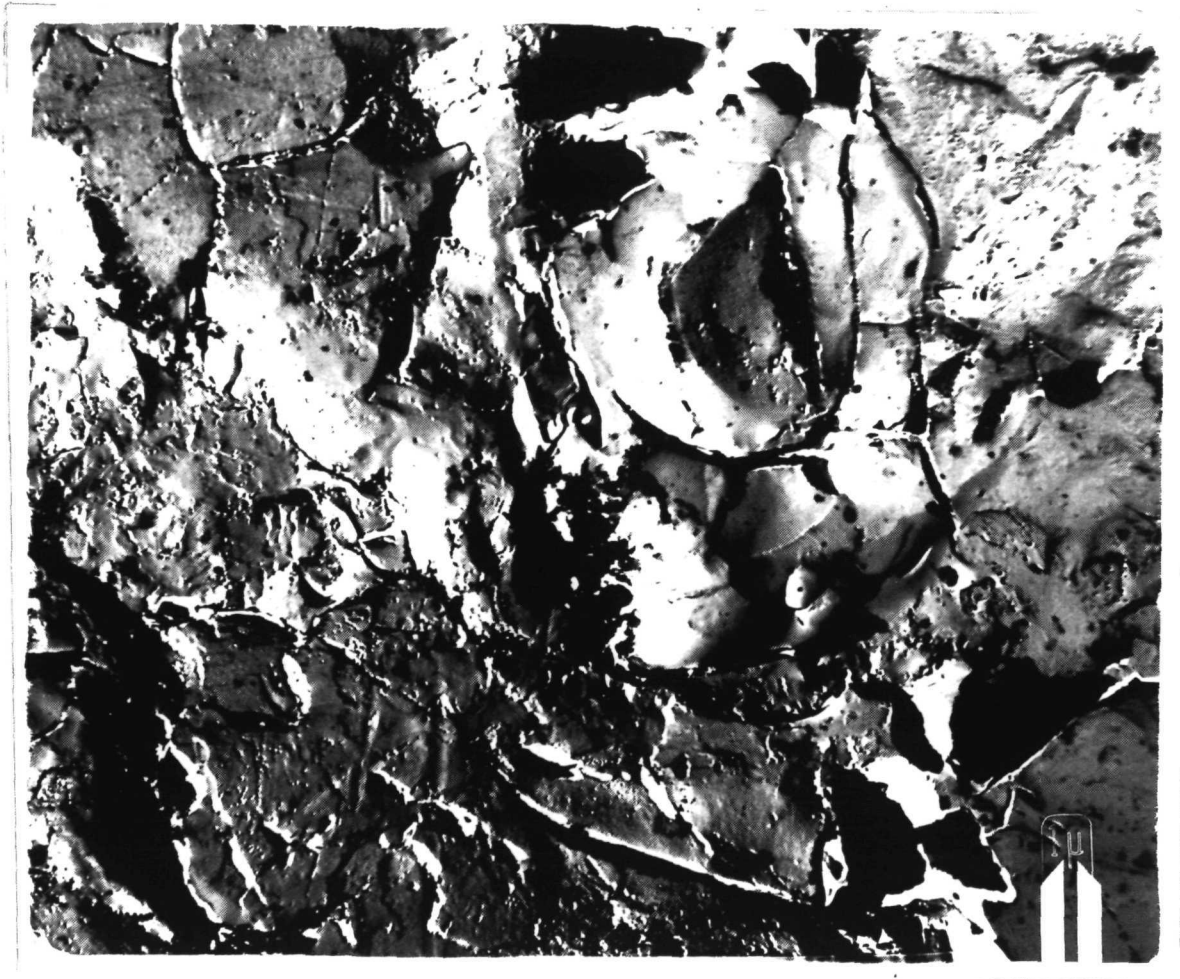


Fig. 13 Electron micrograph of replicated surface of 7075-T651 showing numerous cracks and associated pitting. 1500x

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