1993 NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM

JOHN F. KENNEDY SPACE CENTER
UNIVERSITY OF CENTRAL FLORIDA

STRESS CORROSION CRACKING PROPERTIES OF 15-5PH STEEL

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DIVISION: Material Science

BRANCH: Failure Analysis and Materials Evaluation

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DATE: August 5, 1993

CONTRACT NUMBER: University of Central Florida
NASA-NGT-60002 Supplement: 11

ACKNOWLEDGEMENTS

I would like to express my appreciation for being selected to participate in the NASA/ASEE Summer Fellowship Program. To Dr. Ramon Hosler [University of Central Florida] and to Mr. Tom Barrom [NASA] for their capable administration of the program.

I also want to thank my NASA colleague Dr. Rupert Lee for his assistance and encouragement during this summer internship. To Peter Marciniak for his invaluable assistance whenever it was needed. My thanks go also to Ms.Kari Stiles for her dedication and invaluable help during my stay at Kennedy Space Center.

Last but not least I want to thank Mr. Scott Murray, Mr. William (Irby) Moore, Mr. Cole Bryant, and all the rest of the people in the Materials Section for making me feel at home during this summer.

ABSTRACT

Unexpected occurrences of failures,due to stress corrosion cracking (SCC) of structural components,indicate a need for improved characterization of materials and more advanced analytical procedures for reliably predicting structures performance.

Accordingly, the purpose of this study was to determine the stress corrosion susceptibility of 15-5PH steel over a wide range of applied strain rates in a highly corrosive environment. The selected environment for this investigation was a higly acidified sodium-chloride (NaCl) aqueous solution.The selected alloy for the study was a 15-5PH steel in the H900 condition. The Slow Strain Rate technique was selected to test the metals specimens.

SUMMARY

The catastrophic failure of some structural components,at Kennedy Space Center, due to environmentally assited cracking has raised questions regarding the reliability of those structures. To that effect NASA has intiated a comprehensive program to identify materials which are inmune to cracking under the above mentioned conditions,and recommend them for future applications.

The purpose of this study was to determine the behavior of some Precipitation Hardenable steels when exposed to a highly corrosive environment at different strain rates. The material selected for this study was a 15-5PH steel in the H900 condition. The environment selected consisted of a highly acidified Sodium-Chloride (3.5% NaCl) aqueous solution. The Slow Strain Rate technique was selected to test the metal's specimens. The test were programmed for strain rates between 10^{-3} to 10^{-5} inches per minutes.

The data obtained from these tests beside being useful for selection of materials on a sound engineering basis provides also for a better understanding of the Stress Corrosion Cracking phenomena.

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1.0 INTRODUCTION

1.1 BACKGROUND

The solid rocket booster used to launch the space shuttle, use ammonium perchlorate as fuel and release approximately seventeen(17) tons of hydrochloric acid into the atmosphere in each launching.This acidification of the marine atmosphere has led to severe problems and premature failure of various structural components and critical equipment [1] at the launching facilities at Kennedy Space Center(KSC).

The fact that some structural components have failed catastrophically due to environmental cracking,has raised questions regarding the reliability of those structures.Even if catastrophic failures are rarely observed in practice,when they occur,they may be more costly in terms of human life and property damage than other types of falures.To that effect NASA has been working in a comprehensive program to identify materials which are not susceptible to cracking in the launch-pad's environment when stressed,and recommend them for future applications.

1.2 STRESS CORROSION CRACKING MECHANISM

Delayed failure of structural components subjected to an aggresive environment may occur under statically applied stresses well below the yield strength of the material.Failure under these conditions is caused by stress corrosion cracking(SCC) and has long being recognized as an important failure mechanism.

Although many tests have been developed to study this mode of failure the underlying mechanism for SCC are yet to be resolved [2,3,4] and quantitative design procedures against its occurence are yet to be established.These difficulties are caused by the complex chemical,mechanical,and metallurgical interactions; the many variables that afect the behavior; the extensive data scatter [5,6];and the poor correlation between laboratory test results and service experience.

Cracking of materials may be either intergranular or transgranular and may progres at velocities between 10^{-9} to 10^{-1} mm/s.Three broad categories of stress corrosion mechanism can be identified:

1. Pre-existing path mechanism - This mechanism relates the cracking susceptibility to the chemical activity of the grain boundaries (i.e. precipitates).

2. Strain assisted active path mechanism - This mechanism is related to the rupture of a protective film at the crack tip,followed by metal dissolution by the corrosive environment.

3. Absorption mechanism - This mechanism is based on the chemisorption of an environmental specie on the crack tip which reduces the surface energy,and therefore reduces the local fracture strength of the metal lattice.

1.3 LINEAR ELASTIC FRACTURE MECHANIC

The application of Linear Elastic Fracture Mechanic (LEFM) concepts has met with considerable success in the study of SCC [7,8].Because environmentally enhanced crack growth and stress intensity factor (K) can be used to characterize the mechanical component of the driving force in SCC.

The critical stress intensity factor or fracture toughness (K_{IC}) represents the inherent ability of a material to withstand a given stress-field intensity at the tip of a crack and to resist progressive tensile crack extension under plane strain conditions.Plane strain conditions requires that:

$$B=2.5(K_{IC}/S_{YS})^2 ,$$ where:

B= specimen thickness
S_{YS} = tensile strengh

For materials that are susceptible to crack growth in a particular environment this threshold value is called K_{ISCC} and represent the value below which crack propagation does not occur for a given material-environment combination under plane strain conditions (See Fig.1).

Stress corrosion crack growth rates have been investigated in various material-environment combination,and the results suggest that the crack growth rate as a function of the stress intensity factor can be divided in three regions (See Fig.2).In region I the rate of crack growth is strongly dependent on the magnitude of the stress intensity factor,such that small changes in K results in large changes in crack growth rate.Region I also exhibit a stress intensity factor (K_{ISCC}) below which cracks do not propagate under sustained loads for a given material-environment system. In region II crack growth rate ,for many systems,is moderately dependent on the magnitude of K and for some systems like high strengh steels in gaseous hydrogen,crack growth rate is independent of K. The crack growth rates in region III increases rapidly with K as K approaches K_{IC} of the material.

1.4 SLOW STRAIN RATE TECHNIQUE

Before the early 1960's ,constant load and constant strain testing on smooth and notched specimens of various configurations became very popular. However the 60's produced two accelerated-test techniques based on different mechanical approaches [9,10]. One of the techniques involved testing statically loaded mechanically precracked specimens using Linear Elastic Fracture Mechanics concepts. The second involved Slow Stain Rate testing of smooth specimens. These testing methods have often produced SCC in materials,where older techniques have failed to do so.

More recently Constant Strain Rate test have become widely accepted as quality control or screening technique quite apart from their usefulness in mechanistic studies. The prime justification for this technique is that it accelerates a

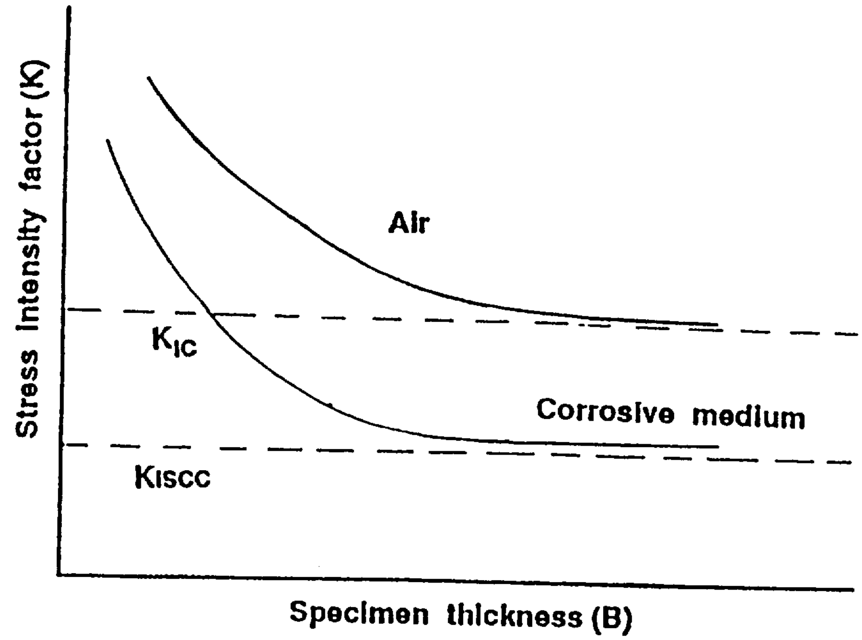


Fig. - 1 Stress Intensity Factor vs Specimen Thickness Curve

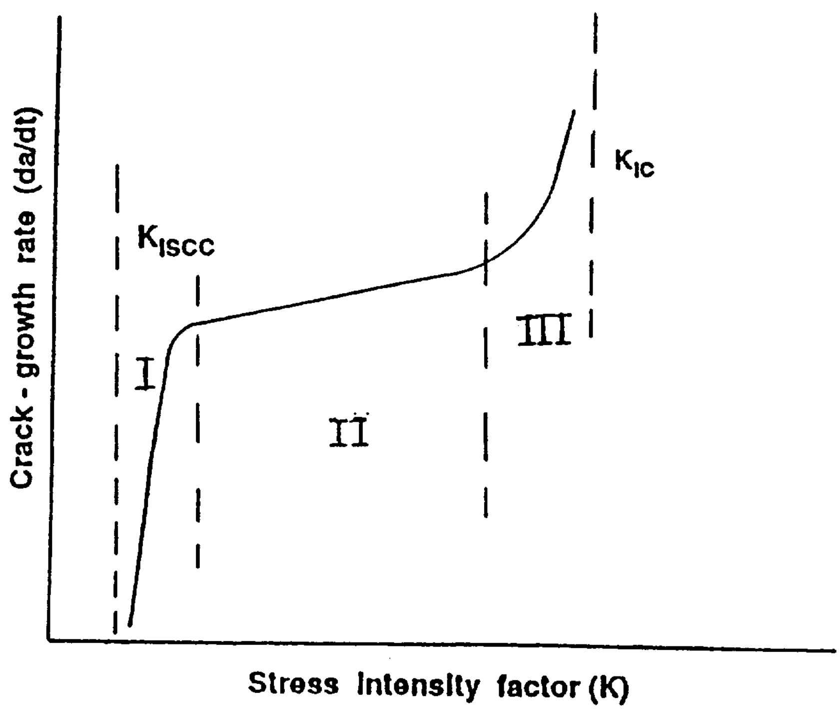


Fig. - 2 Typical Curve Of Crack-growth Rate vs Stress Intensity Factor known rate determining step in the cracking mechanism of ductile alloys-aqueous environment system (i.e. oxide-rupture rate). It is not surprising then that good correlations are observed between SCC susceptibility rated by this technique and by more protracted methods involving static loads.

Strain rate is one of the most important single parameter in evaluating SCC susceptibility of any metal or alloy in a given environment. If strain rate is too high, fracture of the material will be mostly mechanic (ductile) because the corrosion process cannot keep pace with the straining process. On the other side if the strain rate is too low,SCC may be prevented due to repassivation of te exposed base metal,which may be to fast compared to the frequency of the film rupture event. It has been observed that strain rates in the range of 10^{-5} to 10^{-1} S^{-1} tend to promote SCC in most cases.

2.0 MATERIALS AND PROCEDURES

2.1 MATERIALS

The materials used in this investigation consist of 15-5PH steel in the H900 condition. The nominal chemical composition is shown in Table 1,and the thermal condition is shown in Table 2. Table 3 is a summary of the mechanical properties of the material. A photomicrograph showing the distribution of precipitates is shown in Figure 3.

Compact tension (CT) specimens were machined from the as-received material,such that the crack growth direction was perpendicular to the material's rolling direction. Specimens were cleaned to eliminate grease and other impurities from the machining operation and then they were inmersed in a magnesium chloride solution to produce the starting crack required in this type of test. The specimen configuration is shown in Figure 4. The specimens were then loaded into the testing machine and strained until rupture occured.

2.2 EQUIPMENT

Test were conducted in a Satec's MATS II Universal Testing Machine equiped with "NuVision II" sofware package for automating the system. The minimum strain rate applied by the machine was $8x10^{-5}$ inches per minutes. The crack-mouth opening was measured with a double cantilevel beam type strain gage. A Laser based type extensometer developed at KSC was also used to measure the crack-mouth opening.

2.3 TESTING PROCEDURE

Following mounting in the testing machine,the specimens were initially tested in air at different strain rates. A curve of applied load vs crack-mouth opening similar to the one shown in Figure 5 was obtained. From that graph the crack growth rate and the stress intensity factor for the material could be evaluated.

Table - 1 Nominal Composition Of 15-5 PH Steel

Cr	Ni	Cu	Mn	Si	Cb+Ta	C
14.0- 15.0	3.5- 5.5	2.5- 4.5	1.0- Max	1.0- Max	0.15- 0.45	0.07- Max

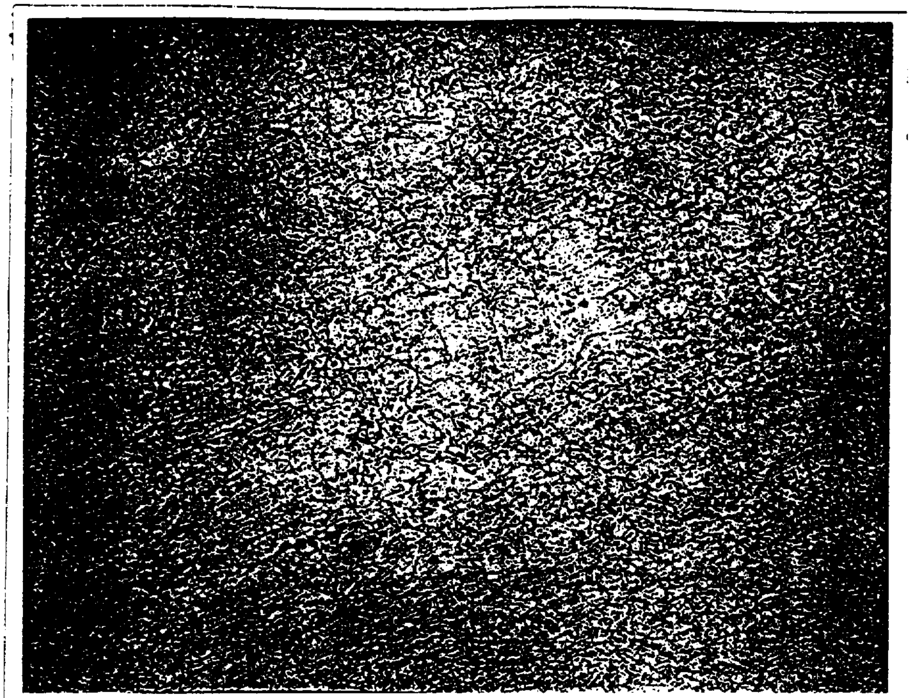
Table - 2 Heat Treatment Of 15-5 PH Steel

Temper	Heat Schedule
Solution Treated	1900 F for half (.5) hour Oil quench
H 900	900 F for one (1) hour Air cooled

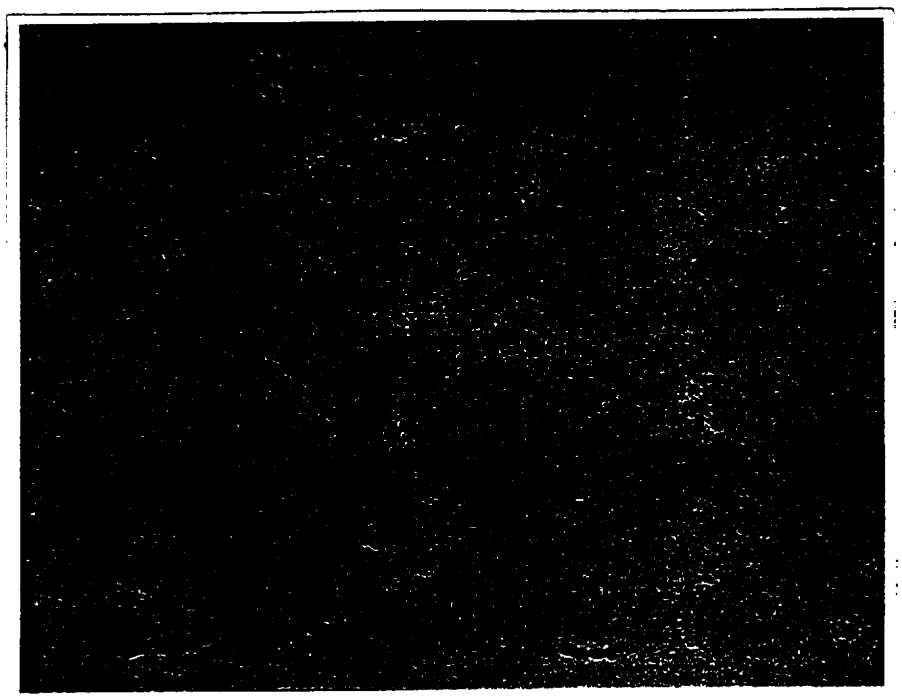
Table - 3 Mechanical Properties Of Vaccum Melted 15-5 PH Steel

Temper	Su	Sys	% Elong	% R.A.	Hardness
H 900	199.6	178.9	17	61	HB 401

Su = Tensile Strength (KSI)
Sys = 0.2% offset yield strength (KSI)
R.A. = Reduction in area
HB = Brinell hardness



Mag.-100X



Mag.-500X

Fig.-3 Photomicrograph Of 15-5PH Steel In The H900 Condition Showing The Precipitate Distribution.

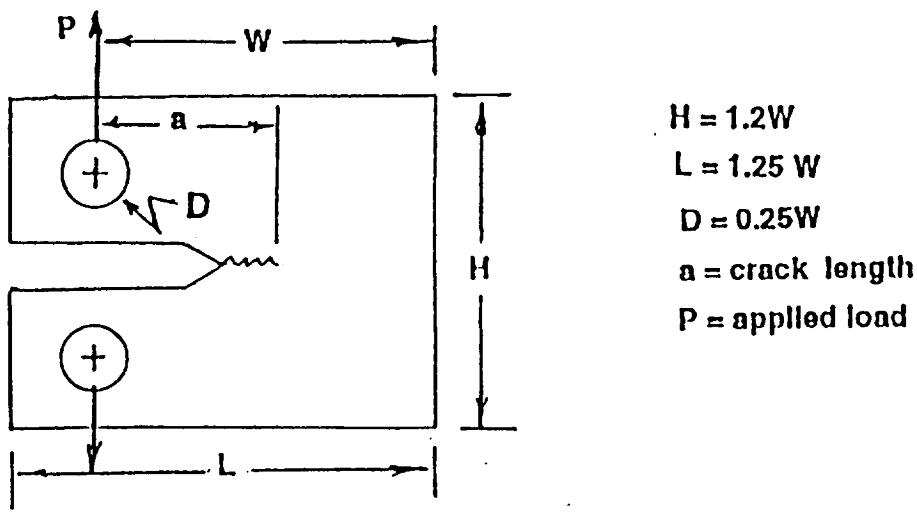


Fig. - 4 Compact - Tension (CT) Type Fracture Toughness Specimen

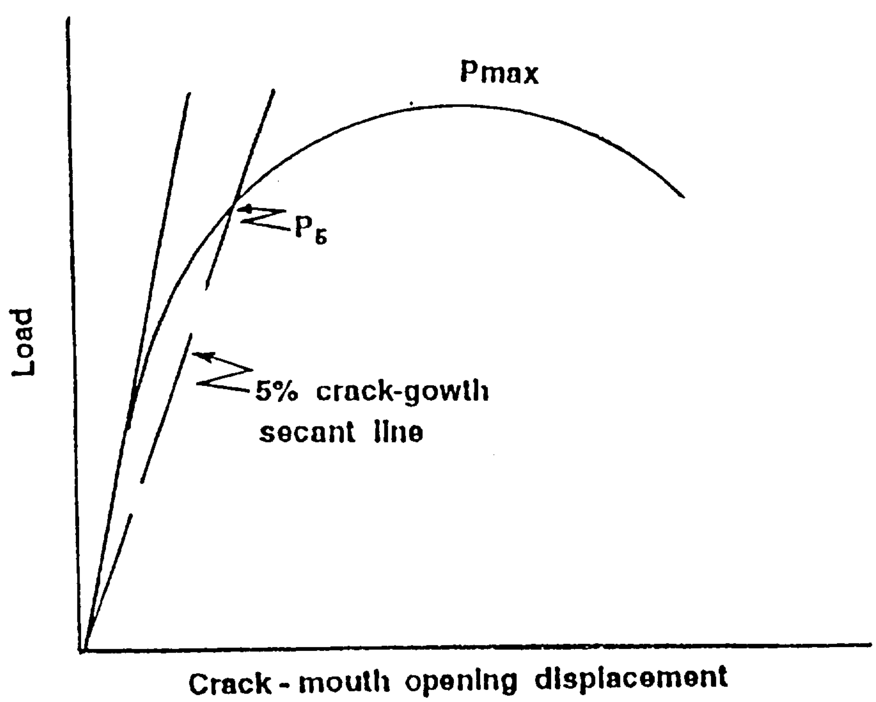


Fig. - 5 Load vs Crack-mouth Opening Displacement Curve

A similar set of tests in a corrosive solution of 3.5% NaCl acidified to 1N.HCl was also scheduled. From the data obtained from those tests the crack growth rate and the critical stress intensity factor under corrosive conditions were to be evaluated.

3.0 RESULTS AND DISCUSSION

3.1 GENERAL

The constant strain rate technique is a method to asses susceptibility of metals and alloys to SCC. It provides a rapid laboratory method to determine the SCC susceptibility of materials in environments in which other tests do not readily promotes SCC. The results are positive in that failure occurs either in a ductile manner or prematurely in a brittle mode if SCC occurs.

Several properties are used to define and compare the severity of SCC of materials and aggresiveness of environments. Generally a measure of the time to failure,or reduction in ductility in a corrosive environment is compared to the behavior in an environment which does not promote SCC,for example air.Increased severity of SCC is indicated by shorter times to failure or reduced ductility,as measured by reduction in area or reduction in elongation. The presence or absence of SCC on the fractured specimen can be unequivocally determined only by metallographic examination. Results of those examinations can be presented quantitatively by comparing the number or length of secondary stress corrosion cracks.

The usefulness of Fracture Mechanics for defining SCC tendencies in hig strength metals is derived from the ability to use the parameter K_{ISCC} for calculating the stress-flaw size combination necessary for the initiation of cracks growth. The value of K_{ISCC} is calculated from the load vs crack-mouth opening displacement curve. From that graph the stress intensity factor for the specific material-sample geometry-environment (K_Q) will be calculated according to:

$$K_Q = [P_Q / B\sqrt{W}].f(a/W) \quad \text{where:}$$

B= specimen thickness

W= specimen width

a= crack length, and

$$f(a/W) = [(2+a/W)(.886+464a/W-13.32a^2/W^2+14.72a^3/W^3-5.6a^4/W^4)]/[1-a/W]^{3/2}$$

$P_Q=P_5$ if the load at every point in the graph preceded P_5 (See Fig.5)
$P_Q=P_{max}$ if there is a maximun load preceding P_5 in the graph (Fig.5)

The value of K_Q will be equivalent to K_{IC} or K_{ISCC} if:

$P_{max}/P_5 \geq 1.10$ and

B and $a \geq 2.5(K_Q/S_{ys})$

if the above two constraints are not satisfied then the test is not valid for determining K_{IC} and K_{ISCC},and a new test have to be done using a thicker specimen,usually 1.5 times thicker.
With values of K_{IC} and K_{ISCC} obtained from the slow-strain rate test and ultimate and yield strength of the material,a curve similar to that in Fig.6 can be obtain,that will permit the designer a better way of predicting the life of the structure for a given design stress-crack length combination.

The application of the Fracture Mechanic approach to design concepts relies on the definition of the boundary lines on the stress vs crack-depth diagram. Figure 6 shows the no-crack growth,subcritical-crack growth,and catastrophic failure regions. The boundaries are experimentally definable,with limits imposed by S_{YS} and S_U for smooth specimens and K_{IC} and K_{ISCC} for pre-cracked samples.Where S_{YS} is the material yield strength and S_U is the maximum tensile strength.

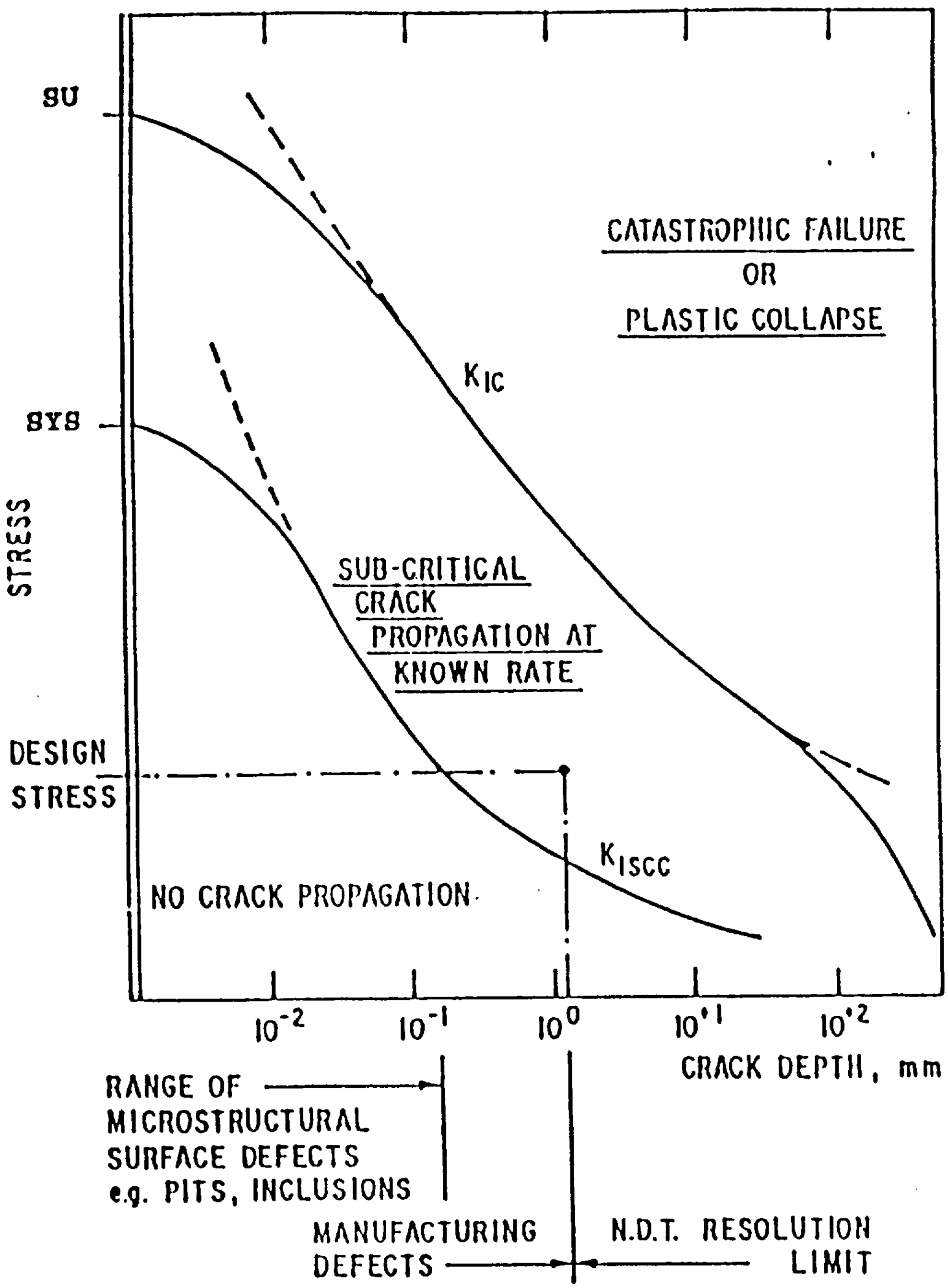


Fig.-6 Tensile Strength vs Crack Length Diagram Illustrating The Regions Of No-crack, Subcritical Crack Growth, And Catastrophic Failure.

Whether or not a component will crack from the time of commisioning will depend on the tensile stress-crack size combination. If the initial stress intensity associated with this combination is greater than K_{ISCC}, its lifetime or inspection periodicity may be determined by integration of the appropriate crack-velocity vs stress intensity curve.

4.0 CONCLUDING REMARKS

Constant strain rate techniques provide a rapid laboratory method to determine the susceptibility of metals to stress corrosion cracking. Absolute results are obtained because failure occurs either in a ductile manner or prematurely by a brittle mode when stress corrosion cracking is present. The absence of stress corrosion cracking assures that the material can be used safely under the specific condition examined. If stress corrosion cracking is observed,judgement is necessary,because the material still can provide sufficient useful life in service. The data obtained from those tests beside being useful for materials selection on a sound engineering basis provides also for a better understanding of the stress corrosion cracking mechanism.

5.0 RECOMMENDATIONS

Due to the short time available and some unexpected delays part of the testing program scheduled for the summer could not be finished. So it is suggested that this study be continued and expanded. The areas where expansion is possible include;

1- Testing at different temperatures,
2- Testing at other heat treatment conditions, and
3- Testing in real conditions (exposure to real atmosphere under static load).

A similar set of experiments can be also done using other types of Precipitation Hardenable steels.

6.0 APPENDICES

6.1 APPENDIX A

Manufacturer certification of properties and quality of 15-5 PH steel.

ARMCO STAINLESS & ALLOY PRODUCTS
A DIVISION OF ARMCO INC.

CERTIFICATION

DATE: JAN 15 1993

5123823 CUST. NO.

BILL TO: 2110 286

BILL TO	SHIP TO
ANDERSON SHUMAKER CO	ANDERSON SHUMAKER CO
824 S. CENTRAL AVE	824 S. CENTRAL AVE
CHICAGO IL 60644	CHICAGO IL 60644

PRODUCT NUMBER	TEST LOT ID	SALES ORDER NO.	PURCHASE ORDER NO.
32280-18157	4083-7-B1262A01	23689	735

SHIP LOT ID: 4083-7-B1262A01 MET LOT NO.: B1262A

15-5 PH SEMI FINISH BILLET STNLS XM-12 VAC CE FQ OA FOR FORGE

AMS 2300G * AMS 2315C EX ONE TEST SAMPLE >6" OK * AMS 5659G TYPE 1 EX COND A HDNS WVD * ASME SA705 89ED TYPE XM-12 COND H900T ANAL & MECH PROP CPBLTY ONLY * ASTM A564-89 TYPE XM-12 COND H900T ANAL & MECH PROP CPBLTY ONLY * ASTM A705-89 TYPE XM-12 COND H900T ANAL & MECH PROP CPBLTY ONLY * MIL-I-45208A * MIL-STD-2154 (SUPERCEDES MIL-I-8950) TYPE II (CONTACT) PER ARMCO UTP 7 REV 4 *

LINE:	SIZE:	SHAPE:	LENGTH:	WEIGHT:
1	8.00000"	RCS	16' 0" TO 20' 0" MIN/MAX	4,420#

TYPE ANAL: REGULAR PARENT HEAT NO: 626233
C: 0.050 MN: 0.540 P: 0.020 S: 0.004 SI: 0.380 CR: 14.91
NI: 4.57 MO: 0.080 CU: 3.580 CB: 0.310 SN: 0.009 W: 0.003
CO: 0.040 AL: 0.004 N: 0.034 TI: 0.004 V: 0.080 TA: 0.008
B: 0.00130 MULT: 5. MULT X C: 0.250
CB+TA LOWLIM: 0.250 CB+TA: 0.318

TYPE ANAL: PRODUCT ANALYSIS HEAT NO: 4083-7 *
C: 0.047 MN: 0.350 P: 0.024 S: 0.004 SI: 0.390 CR: 14.86
NI: 4.70 MO: 0.070 CU: 3.430 CB: 0.360 CO: 0.043 N: 0.024
TA: 0.008
MULT: 5. MULT X C: 0.235 CB+TA: 0.368

TYPE ANAL: TOP HEAT NO: 4083-7 *
C: 0.047 MN: 0.350 P: 0.024 S: 0.004 SI: 0.390 CR: 14.86
NI: 4.70 MO: 0.070 CU: 3.430 CB: 0.360 CO: 0.043 N: 0.024
TA: 0.008
MULT: 5. MULT X C: 0.235 CB+TA: 0.368
HTL: 2-920199

AS SHIP TEST: OVERAGE
B HRDNS: 293. B HRDNS (P/F): OK TEMP: 1300.
HOURS: 2. MINS: 0. QUENCH: AIR

CAPABILITY TEST: H900T
B HRDNS: 401. B HRDNS (P/F): OK TEMP: 900.
HOURS: 1. MINS: 0. QUENCH: AIR
UTS KSI: 198.7 .2% YLD STR KSI: 180.9
OFFSET %: 0.20
% EL 2" OR EQ: 8.0 % RED OF AREA: 20.60

PHYSICAL:
FERRITE PCT: <1 FERRITE (P/F): OK MACRO 604 CL 1: A
MACRO 604 CL 2: A MACRO 604 CL 3: A MACRO 604 CL 4: A
MACRO 604 (P/F): OK MFLUX FREQUENCY: 0.00 MFLUX SEVERITY: 0.00
MAGNAFLUX (P/F): OK UT: OK

METALLURGICAL RELEASE [signature] 1/30/93

ʘ STAINLESS & ALLOY PRODUCTS CERTIFICATION
..VISION OF ARMCO INC.

DATE JAN 1 5 1993

PAGE NO. 2 of 2

5123823	CUST. NO.	BILL TO	2110	286	SHIP TO

ᴱRSON SHUMAKER CO
824 S. CENTRAL AVE
CHICAGO IL
60644

ANDERSON SHUMAKER CO
824 S. CENTRAL AVE
CHICAGO IL
60644

PRODUCT NUMBER	TEST LOT ID	SALES ORDER NO.	PURCHASE ORDER NO.
32280-18157	4083-7-B1262A01	23689	735

MATERIAL OVERAGED 1300 DEG F 2 HRS @TEMP 12 HRS T.T. AIR COOLED

Material Ultrasonic tested per MIL-STD-2154 (SIZES 0"-10"INCL - CL A, OVER 10" - CL B) TYPE II (supercedes MIL-I-8950) per ARMCO UTP 7 REV 4 (SIZES 0"-10" INCL - CL A, SIZES OVER 10" - CL B) (CONTACT) and was found

Heat treated at TIME, TEMP and QUENCH indicated above.

Material electric furnace melted, AOD refined, VAC ARC remelted.

Melted and manufactured in the U.S.A.

No welding or weld repair performed on this material.

ASAP certifies conformance to NRC 10CFR PT 21, and 10CFR PT 50 APP B.

This material was manufactured and tested in accordance with the noted specifications, and is in conformance with those specification requirements

ASAP certifies that this material is manufactured free from mercury, r um, alpha source and low melting metal or alloy contamination.

This material was produced in accordance with the Quality Assurance Program, Quality Assurance Manual, Issue 5 Rev 0 dtd 6/1/92.

All testing procedures were conducted in accordance with the latest ASTM standards or applicable specifications.

The recording of false, fictitious or fraudulent statements or entries on this document may be punished as a felony under federal statutes including Federal Law, Title 18, Chapter 47. This certified test report has been delivered to a consignee of material purchased from ASAP. To avoid the possibility of its misuse on the redelivery of this report to a third party it must be recertified by and under the name of such consignee.

The chemical analyses and physical or mechanical test report are correct as contained in the records of the Corporation.

E Famularo E. FAMULARO, CERTIFICATION CLERK

METALLURGICAL RELEASE [signature] 1/20/93

MSi Metallurgical Services Inc.
1201 S. Ninth Avenue
Maywood, Illinois 60153
708•343•3444

Anderson Shumaker Company
824 S. Central Ave.
Chicago, IL 60644

Report No.: 9274-1
Date: 2-5-93
Order No.: 7401

Attention: Mr. Steven Tribble

SUBJECT

Tensile and hardness testing of one (1) test bar identified as 15-5 PHVAC, Condition H900, Heat #4083-7, Job #10553, AMS 5659.

TEST RESULTS*

Tensile Testing

Tensile Strength, psi	199,600
Yield Strength, psi (.2% offset)	178,900
% Elongation in 2"	17
% Reduction of Area	61

Hardness Testing

Hardness, HB	401

* Testing performed in accordance with ASTM E8-91 and E10-84.

Respectfully Submitted,

Metallurgical Services Inc.

David G. Hoffman
Senior Metallurgical Engineer

DGH/mj

MSi Metallurgical Services Inc.
1201 S. Ninth Avenue
Maywood, Illinois 60153
708•343•3444

Anderson Shumaker Company
824 S. Central Ave.
Chicago, IL 60644

Report No.: 9274-2
Date: 2-5-93
Order No.: 7401

Attention: Mr. Steven Tribble

SUBJECT

Free ferrite examination of one (1) test bar identified as 15-5 PHVAC, Condition H900, Job #10553, Heat #4083-7, AMS 5659.

TEST RESULTS*

Free Ferrite Examination

One (1) 15-5 PHVAC stainless steel sample was examined for percentage of free ferrite in accordance with Aerospace Material Specification (AMS) 2315A. The sample was sectioned perpendicular to the direction of rolling as identified in paragraph 3.1.6. The sample was metallographically prepared in accordance with ASTM E3-80 and examined in the etched condition at a magnification of 250X.

The microstructure was rated for percentage of free ferrite in accordance with the occupied squares method as outlined in paragraph 3.2.1. The worst field determined by metallographic examination was photographed at 250X and rated using a transparent grid overlay. A total of fifteen squares were used in the free ferrite calculation. The percentage of free ferrite was determined in accordance with paragraph 3.2.1.1. The results are as follows:

Percentage Free Ferrite

.92

Respectfully Submitted,

Metallurgical Services Inc.

David G. Hoffman
Senior Metallurgical Engineer

DGH/mj

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