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1994 NASA/ASEE SUMMER FACULTY FELLOWSHIP PROGRAM

JOHN F. KENNEDY SPACE CENTER **UNIVERSITY OF CENTRAL FLORIDA**

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MECHANICAL BEHAVIOR OF PRECIPITATION HARDENABLE STEELS EXPOSED TO HIGHLY CORROSIVE ENVIRONMENT

PREPARED BY:

ACADEMIC RANK:

UNIVERSITY AND DEPARTMENT:

NASA/KSC

DIVISION:

BRANCH:

NASA COLLEAGUE:

DATE:

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I also want to thank my NASA colleague Dr. Rupert Lee for his invaluable assistance during this summer. To Mr. Peter Marciniak, Mr. Dean Lewis, and all the rest of the people in the Materials Section my thanks for their inconditonal help.

My gratitude go also to Ms. Karl Stiles and Ms. Maria Smith for their dedication and invaluable assistance during my stay at Kennedy Space Center.

Last but not least I want to thank Mr. Scott Murray, Mr. William (Irby) Moore, and Mr. Cole Bryant for making me feel at home during this summer.

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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 15-5 PH steel over a wide range of applied strain rates in a highly corrosive environment. The selected environment for this investigation was a 3.5 % NaCl aqueous solution. The material selected for the study was 15-5 PH steel in the H 900 condition. The Slow Strain Rate technique was used to test the metallic specimens.

SUMMARY

The catastrophic failure of some structural components, at Kennedy Space Center, due to environmentally assisted cracking has raised questions regarding the reliability of those structures. To that effect NASA has initiated 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 under different loading situations. The material used in this study was a 15-5 PH steel in the H 900 condition and the selected environment was a 3.5 % NaCl aqueous solution. The Slow Strain Rate techniques was used to test the material. Slow Strain Rate tests were done at 8×10^{-5} in/min.

Results show that the 15-5 PH steel is susceptible to stress corrosion cracking in a 3.5 % NaCl solution, and for the geometry used in these tests the stress intensity factor is of the order of 70 Ksi \sqrt{in} .

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1.0 INTRODUCTION

1.1 BACKGROUND

The solid rocket boosters 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).

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The fact that some structural components have failed catastrophically due to environmentally assisted cracking, has raised questions with regards to the reliability of those structures. Even if catastrophic failures are rarely observed in practice, when they occurs, they may be more costly in terms of life and property damage than other types of failures. 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.

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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 affect the behavior; the extensive data scatter [5,6]; and the poor correlation between laboratory test results and service experience.

Experimental procedures for SCC tests of pre-cracked specimens may be divided in two general categories. They are Time- to- Failure tests and Crack-Growth-Rate tests. The Time-to-Failure tests are similar to the conventional stress-corrosion tests for smooth or notched specimens. The Crack-Growth-Rate tests are more complex and requires more sophisticated instruments than do the Time-to-Failure tests. However data obtained by using Crack-Growth-Rate tests should provide information necessary to enhance the understanding of the kinetics of SCC and verify the threshold behavior $K_{\rm ISCC}$.

Cracking of materials may be either intergranular or transgranular and may progress at velocities between 10⁻⁹ to 10⁻¹ in/sec. 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).

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.
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 Mechanics (LEFM) concepts has met with considerable success in the study of SCC [7,8], because environmentally enhanced crack growth and the stress intensity factor 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:

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

B = specimen thickness $S_{SY} = tensile strength$

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 different 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 rates. Region I also exhibit a stress intensity factor (K_{ISCC}) below which cracks do not propagates under sustained loads for a given material - environment system. In region II crack growth rates, for many systems, is moderately dependent on the magnitude of K and for some systems, like high strength steel in gaseous hydrogen, crack growth rate is independent of K. The crack growth rates in region III increases rapidly with K and approaches the K_{IC} of the material.

The usefullness of Fracture Mechanics for defining SCC tendencies in high 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 (See Fig. - 3). From that graph the stress intensity factor for the specific material geometry - environment (K_Q) will be calculated according to the following equation :



Specimen thickness (B)





Stress Intensity factor (K)



11.11







If the two constraints Previously discussed are not satified, then the test is not valid for determining K_{IC} or K_{ISCC} , and a new test have to be done using a thicker specimen, usually 1.5 time thicker.

The application of the Fracture Mechanics approach to design concepts relies on the definition of the boudary lines on the Stress vs Crack -Depth diagram. Figure - 4 shows the no - crack - growth, the the subcritical - crack - growth, and the 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 (S_U is the maximum tensile strength of the material).

Whether or not a component will crack, from te time of commisioning will depend on the tensile stress - crack -size combination. If the initial stress intensity associated withh 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.

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Fig. 4 Tensile stress/crack depth diagram, illustrating the regions of no crack growth, subcritical crack growth, and catastrophic failure.

1.4 SLOW STRAIN RATE TECHNIQUES

Before the early 1969'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 the Slow Strain Rate Testing of smooth specimens. These testing methods have often produced SCC in materials, where older techniques have failed to do so.

More recently the Constant Strain Rate test have become widely accepted as a quality control or screening technique quite apart from their usefulness in mechanistic studies. The prime justification for this technique is that it accelerates a known rate determining step in the cracking mechanism of ductile alloys - aqueous environment system (i..e. oxde - rupture rate). The Constant Strain Rate technique is a method to asses the susceptibility of metals and their alloys to SCC. It provides a rapid laboratory method to determine the SCC susceptibility of the material in environments in which other tests do not readily promotes SCC. The results are positive in that failure occurs in a ductile manner or prematurely in a brittle mode if SCC occurs. 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.

Several properties are used to define and compare the severity of SCC of materials and aggressiveness of environments. Generally a measure of the time to failure, or reduction in ductility in a corrosive environment is compared to the behavior of the material in an environment which does not promote that condition, 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

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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.

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 the exposed base metal, which may be too 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 15-5 PH steel in the H 900 condition. The nominal chemical composition is shown in Table - 1, and the thermal conditions are shown in Table - 2. A summary of the mechanical properties of the material is shown in Table - 3. Figure - 5 shows the distibution of precipitates in the samples.

Compact tension (CT) specimens were machined from the as received material, according to ASTM E 399 - 83 standard. Specimens were

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

Table - 1 Nominal Composition Of 15-5 PH Steel

Table - 2 Heat Treatment Of 15-5 PH Steel

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

Table - 3 Mechanical Pro	perties Of Vaccum	Melted	15-5	PH	Steel	l
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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.-500X

Fig.-5 Photomicrograph Of 15-5PH Steel In The H900 Condition Showing The Precipitate Distribution. cleaned to eliminate grease and other impurities from the machining operation and then were fatigued to produce the starting crack required in this type of test. The specimen configuration is shown in Figure - 6. The specimens were then loaded into the testing machine and strained under different conditions.

2.2 EQUIPMENT

Test were conducted in a Satec's MATS II Universal Testing Machine equipped 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 extensometer.

2.3 TESTING PROCEDURE

All the Slow - Strain - Rate K_{ISCC} tests involved in this investigation were conducted in a 0.375 - in. thick, C-T specimen in accordance with ASTM method E - 399 - 83. The specimens were fatigue precracked in air prior to loading in the corrosive environment (See Fig. - 7). Test were conducted at a strain rate of 8×10^{-5} in/min in air and in a 3.5% NaCl solution. A curve of applied load vs crack mouth opening, similar to the one shown in Figure - 8 was obtained. From the graphs the stress intensity factor (K_Q) for the material was calculated.

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Fig. - 6 Compact - Tension (CT) Type Fracture Toughness Specimen



Fig. - 7 Typical Fatigue Crack Produced in Samples Prior to Exposure to Corrosive Environment.





3.0 RESULTS AND DISCUSSION

3.1 GENERAL

In order to standardize the technique used to establish the point of deviation on the load - crack opening displacement curve associated with the onset of subcritical crack growth, a 5 % secant offset procedure similar to that used for KIC testing (ASTM method 399-83) was employed. Specifically a line with a slope 5 % less than the slope of the linear load - displacement record is used to define the load (P_Q) and the displacement at the onset of crack growth. Fig. - 8 represent a load - mouth opening displacement record of the Constant Strain Rate test involved in this investigation. In air the first deviation from linearity occured at about 4270 lb. and the load continued to increase up to 5599 lb. before fracture occured. In the 3.5 % NaCl solution, deviation from linearity started at about 3060 lb. and load increased to 5131 lb. before development of rapid unstable crack growth. Because of high K values associated with these tests, deviation from linearity, may be the result of either subcritical crack growth, plastic yielding, or a combination of both mechanism. In SEM studies of the fracture surface, (Fig. - 9), no subcritical cracks were observed, but and increase in the number of voids formed, can be observed in the stress corrosion region as compared to the fast fracture region.

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Fig. - 9 SEM of Fracture Surface of SCC Specimen . a) Fast Fracture Region , b) Stress Corrosion Region .

Structural materials whose toughness levels are such that they exceed the plane - strain limits, exhibit elastic - plastic fractures wih varying amounts of yielding prior to fracture. The tolerable flaw sizes at fracture vary considerable but can be fairly large. Fracture is usually preceded by the formation of large plastic zones ahead of the crack. Fig. - 10 is typical of this type of behavior where failure occurs mainly by general yielding. The direction of the crack propagation can be observed in Fig. - 11 and confirm also, the previews observations.

Table - 4 is a summary of the SCC properties of 15 - 5 PH steel in air and in a 3.5 % NaCl solution tested at a strain rate of $8x10^{-5}$ in/min. In Figs. - 12 & 13 the changes in stress intensity factors as a function of initial crack length can be observed. For the samples exposed to the corrosive environment the value of K_Q, (K_{ISCC} apparent), is aproximately 70 Ksi In and is independent of initial crack length. For the samples tested in air there is a big decrease in the K value as a function of increasing initial crack length.

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Fig. - 10 Plastic Fracture Surface of 15 - 5 PH Steel Exposed to a 3.5 % NaCl Solution, Showing Regions of Fatigue, Stress Corrosion, and Fast Fracture.



Fig. - 11 Fracture Propagation in C-T Specimens of 15 - 5 PH steel Exposed to a 3.5 % NaCl Solution at a Constant Strain Rate of 8×10^{-5} inches / min.

Sample	Solution	a in .	P _Q Ib .	P _{max} Ib .	K _Q ksi in	K _{max} ksi in	
A - 1	Air	0.910	2780	3351	72.1	86.9	
C - C	Air	0.780	4270	5599	95.7	125.4	
G - G	3.5 %NaCl	0.800	2920	4810	68.2	112.4	
н -н	3.5 %NaCl	0.780	3060	5131	68.6	115.0	
1-1	3.5 %NaCl	0.753	3380	5639	71.1	118.6	

Table - 4 SCC Properties of 15 - 5 PH Steel



Fig. - 12 Stress Intensity as a Function of Initial Crack Length for Dry and Wet Specimens.

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Fig. - 13 Maximun Stress Intensity as a Function of Initial Crack Length for Dry and Wet Specimens.

4.0 CONCLUDING REMARKS

The primary design criterion for most large structures is still based or strength and stability requirements such that nominal elastic behavior is obtained under conditions of maximun load. Usually the strength and stability criteria are achieved by limiting the maximun design stress to some percent of the yield strength. In many cases fracture toughness is also an important design criterion, and yet specifying a notch toughness criterion is much more difficult.

The maximun allowable flaw size in a member has been shown to be related to the notch toughness and yield strength of a material as follow :

 $a = c (critical K / yield strength)^2$

Thus the critical K and yield strength, becomes a good index for measuring the relative toughness of structural materials. It is desirable that the structure tolerate large flaws without fracturing, therefore the use of materials with high K/S_{ys} ratio is a desirable condition.

Constant Strain Rate techniques provides a rapid laboratory method to determine the stress intensity factor (K), and the susceptibility of materials to stress corrosion cracking. Absolute results are obtained because failure occurs either in a ductile manner or prematurely by a

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brittle mode when SCC is present. A decrease in the value of (K) is indicative of SCC susceptibility to a given environment if all other parameters are keep constant. The absence of SCC assures that the material can be used safely under the specific conditions examined. If SCC is observed, judgement is necessary, because the material still can provide sufficient useful life in service. The data obtained from these tests beside being useful for materials selection provides also for a better understanding of the SCC mechanism.

5.0 RECOMMENDATIONS

Due to the broad scope of this project the proposed testing program could not be finished during the summer. So it is suggested that this investigation be continued and if possible be expanded to cover other areas like :

-Testing at other temperatures

-Testing at other heat treatment conditions

-Testing at atmospheric conditions under static loads Other sample geometries should be tested to determine the effect of geometry on the test's results.

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

6.0 APPENDICES

6.1 APPENDIX A

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

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15-5 FH SEMI FINISH	BILLET STHLS >	(M-12 V/	AC CE	FQ OA FO	R FOF	GE			
AMS 2300G * AMS 231 COND A HDNS WVD * A CFBLTY ONLY * ASTM ONLY * ASTM A705-89 MIL-I-45208A * MIL- ARMCO UTP 7 REV 4 *	5C EX ONE TEST SME SA705 89ED A564-89 TYPE XM TYPE XM-12 COM STD-2154 (SUPER	SAMPLE TYPE XI 1-12 CON 1D H900 RCEDES N	>6" (M-12 (ND H90 T ANAL 1IL-I-	NK * AMS COND H900 NGT ANAL & MECH (8950) TY	56596)T ANA & MEC FROP FROP	F TYPE 1 E AL & MECH CH PROF CF CPBLTY OF CONTACT	EX FRC BLT NLY F) F)F Y * 'ER	
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MULT: TYPE ANAL: TOP C: 0.047 MN: NI: 4.70 MO: TA: 0.008	5. 0.350 P: 0 0.070 CU: 3	MULT X HEAT 0.024 3.430	X C: Q NO: 4 S: 7 CB:	9.235 90837 * 0.004 0.360	SI: CO:	0.390 (0.043	CR: N:	0.36 14.8 0,0	8) 6 124
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824 S. CE	NTRAL AVE			824 S. CEN	TRAL	AVE			
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PRODUCT N	UMBER	TEST LOT I	D	SALES ORDE	r no.		PURCHA	SE ORDER NO	D .
32280-1815	7	4083-7-81262	AØ1	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.

ASAF certifies conformance to NRC 10CFR PT 21, and 10CFR PT 50 AFF B. This material was manufactured and tested in accordance with the noted specifications, and is in conformance with those specification requirements ASAF certifies that this material is manufactured free from mercury,

radium, alpha source and low melting metal or alloy contamination. This material was produced in accordance with the Ruality Assurance

Frogram, 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 ASAF. 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, CERTIFICATION CLERK

METALLURGICAL **RELEASE**

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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

T	ensile Strength, psi	199,600
Y.	ield Strength, psi (.2% offset)	178,900
¥	Elongation in 2"	17
\$	Reduction of Area	61

Hardness Testing

Hardness, HB

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

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Respectfully Submitted,

Metallurgical Services Inc.

David 🕼 Hoffman

415 Senior Metallurgical Engineer

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DGH/mj



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

BUBJECT

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. Abffman

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