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TECHNICAL REPORT

CONTROL OF STRESS CORROSION - INTERIM REPORT

By

R. H. Wolff

Department of the Army Project No. 1A024401A11005

AMC Code No. 5025.11.84205

Report No. 63-3890 Copy No. _____

IEL No. 1-9-100-2 Date 26 November 1963

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

This study of the control of stress corrosion cracking susceptibility of steels by application of protective coatings was designed to use abrasive blasted specimens to simulate more nearly the surfaces and conditions of a manufacturing operation. Initial work was conducted using specimens of aircraft quality 4130 alloy sheet steel, heat treated to 200,000 psi yield strength. Zinc was chosen as protective coating and was applied to abrasive blasted bent beam specimens in three forms: by zinc electroplating, zinc phosphatizing, and by zinc dust dispersion. Coated bent beam specimens, tensile loaded at 75% of yield strength were placed in high humidity and in semi-industrial outdoor exposure. Control specimens in "as heat treated" condition have failed in both atmospheres. Tests are continuing.

RECOMMENDATIONS

Insufficient data has been collected at this stage to allow the formation of recommendations except that the work appears promising and should be continued along the line of approach taken.

CONTROL OF STRESS CORROSION - INTERIM REPORT

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CONTROL OF STRESS CORROSION - INTERIM REPORT

OBJECT

To study the reduction of susceptibility to stress corrosion cracking of high strength steels by use of protective coatings.

INTRODUCTION

Major advances in design concepts have almost reached the operational limits of many engineering materials. These advances are shown in the trends toward higher strength levels. Increased strength and decreased safety factors require greater reliability in service. Unfortunately, these materials show a tendency toward increased susceptibility to stress corrosion cracking as the strength levels rise.

Stress corrosion, by currently accepted definition, is the acceleration of the rate of corrosion damage by static stress⁽¹⁾. Stress corrosion cracking is the spontaneous cracking that may result from combined effects of stress and corrosion^(2,3).

Many factors are believed to influence the susceptibility of a material to stress corrosion cracking. These factors include chemical composition, chemical reactivity, metallurgical treatment, residual stresses, crack sensitivity and environment. Investigations are being conducted in these phases to provide clarification of the mechanisms of stress corrosion cracking in terms of specific environments and materials.

Stress corrosion cracking experiments are performed with a variety of specimen types and in various environments. Example specimens are wire, rods, strips of sheet material and pieces formed similar to a tuning fork. For the most precise knowledge of stresses accomplished by a loading technique, the wire, rod or strip for the bent beam⁽⁴⁾ are most effective. Tuning forks (whose tines are drawn together to apply the load) or "U" bend specimens are subject to less precise determination of stress unless strain gages are used. The "U" bend specimen is stressed beyond the elastic limit of the material.

Studies of stress corrosion have been largely devoted to the evaluation of materials under variations in metallurgical state, in chemical composition and in environmental exposure. In these studies the specimens are carefully prepared under the most exacting conditions. Bent beam

specimens are carefully heat treated, and then ground on all surfaces to remove layers of material that may be pre-disposed to minor imperfections.

Among the applications of high strength steels where reliability is vital, are the materials for use in forming rocket motor cases. Work in progress under contract is currently involved with stress corrosion tests of motor case steels using bent beam specimens. Environments have been: immersion of specimens in fluids, including distilled water, oil and a variety of chemical salt solutions. The determination of stress corrosion susceptibility depends upon use of the bent beam test procedure as outlined by Phelps and Loginow⁽⁷⁾. Procedures have been initiated recently for study of propagation of artificially produced cracks. These tests are conducted with surface ground specimens prepared under laboratory conditions.

As a preliminary part of this work, a survey was made of a number of missile contractors, soliciting their procedures of manufacture of rocket motor cases. The use of abrasive blasting as a descaling operation after heat treatment and as a pretreatment for finishing was significantly common.

In a discussion of abrasive blasting of bent beam specimens with Dr. E. H. Phelps,⁽⁸⁾ it was suggested that the tuning fork test specimen might be used effectively. The suggested specimen was less precise in manner of load application unless a strain gage is used. It appeared that the use of abrasive blasted specimens in the bent beam test would be no less precise than the tuning fork method.

Since the bent beam test procedure is widely used and accepted as providing a precise method of loading the specimens, it was selected for use in this work. It is acknowledged that blasting of the surfaces will modify the precision of the method. However, it is felt that the inaccuracies of applied load will not exceed that to be expected with tuning fork or other specimen types available that do not resort to strain gage application.

Accelerated tests are desirable in this work since the initiation of stress corrosion cracks and their propagation to failure is often of long duration. Immersion of bent beam specimens in chemical solutions is an accelerated test that is widely used because solutions can be chosen that will bring about failure in relatively short periods. However, the test solution is usually specific to the material, and simulation to normal exposure and use is unrealistic.

It was proposed in this work to study stress corrosion cracking susceptibility of steels by application of protective coatings to abrasive blasted bent beam specimens. Exposure conditions were high humidity and outdoor exposure.

EXPERIMENTAL PROCEDURE AND RESULTS

A series of specimens 1 x 9 inches were sheared from 63 mil thick, 4130 sheet steel (MIL-S-18729B, Steel, Plate, Sheet and Strip, Alloy 4130 Aircraft Quality). Both longitudinal and transverse directions were represented. These specimens were heat treated in a neutral, controlled atmosphere furnace at a carbon potential of 0.30, oil quenched from 1650°F and tempered to an approximate yield strength of 200,000 psi. Yield strength* and modulus of elasticity determinations were conducted on tensile specimens included with the specimens during heat treatment. (See Table)

* Tensile specimens were steel grit blasted and the 0.2% offset method used to determine yield strength.

TENSILE TEST OF BLASTED AND UNBLASTED 4130 ALLOY SPECIMENS

Test	Yield Strength x 1000 psi.	Ultimate Strength x 1000 psi.	Modulus of Elasticity x 10 ⁶
1	205	251.	28.9
2	206	252.5	30.0
3	204	252.5	30.2
4	203	252.	29.7
5	206	252.5	-
6	201	252.	29.7
Average	204	252	29.7

Hardness: Rockwell C scale 51

Specimens in tests 3 through 6 were steel grit blasted.

Using this data and the method of Phelps⁽⁷⁾ the specimen length necessary to produce a tensile load of 75% of yield strength was calculated.

After cutting to length, all specimens, except those for "as heat treated" control, were steel grit blasted to a uniform appearance. Protective finishes applied for the first series of tests were based on zinc because of the general availability, low cost and desirable galvanic relationship of zinc with iron.

Zinc was electrodeposited from an alkaline cyanide solution to a thickness of 0.5 mil on one group of specimens. A second group was zinc phosphatized and a third group was coated with a proprietary zinc dust dispersion applied with a brush. The dry film is claimed to be 97% zinc and the particles in contact such that electrical conductivity is possible.

The test fixtures were designed to hold six specimens and were made of type 416 stainless steel. The holder span was seven inches.

After mounting, the specimens were exposed in two sets. One set was placed directly in semi-industrial outdoor exposure about 300 feet from the Mississippi River. The second group was exposed to 100% relative humidity at 100°F for one week, and then transferred to the outdoor exposure site. During the week of exposure to high humidity, two of the three "as heat treated" control specimens broke. (See Figure 1). In the outdoor exposure the "as heat treated" controls also failed. Two were broken after seven weeks and the third at ten weeks. The blasted uncoated controls show general rusting, but have not failed. The phosphatized specimens show scattered general corrosion, not as heavy as the controls. The zinc plated and zinc dispersion coated specimens are in good condition.

Tests are continuing.

DISCUSSION

As indicated in the introduction, the use of abrasive blasting on bent beam specimens was not a normal procedure. It was therefore desirable to experiment with a group of specimens to establish that the procedure did not have any unusual drawback. Concurrent tests of blasted and unblasted tensile specimens did not show any significant difference in yield and ultimate strengths other than the normal variation found among specimens within the group. From this result it was believed that the blasting would not weaken the specimen nor would it appreciably strengthen the specimen as a result of a cold working effect such as expected with shot peening.



BENT BEAM TEST FIXTURE WITH "AS HEAT TREATED" SPECIMENS
BROKEN IN ONE WEEK OF HIGH HUMIDITY EXPOSURE

FIGURE 1

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The parallel failures of unblasted heat treated specimens in humidity cabinet and outdoor indicates a possible correlation for accelerated testing. It was the intention of this work to combine shortterm humidity cabinet and salt spray exposures followed with outdoor exposure. In this manner it was considered that the crack initiation, the first step in stress corrosion cracking, would be promoted in the accelerated test, and crack propagation would be advanced in the outdoor test. These tests are believed to be more realistic than immersion tests.

Work will continue with bent beam specimens of 4130 alloy steel and also with 4340 alloy and maraging steels. Yield strength levels will be maintained at 200,000 psi for attempts at correlation of tests. Lower levels will be considered for some tests, although stress corrosion cracking susceptibility generally decreases at lower strengths.

LITERATURE REFERENCES

1. "Corrosion Handbook" edited by H. H. Uhlig, John Wiley & Sons, New York, 1948.
2. "Stress Corrosion Cracking and Embrittlement" edited by W. D. Robertson, John Wiley & Sons, New York 1956.
3. "Physical Metallurgy of Stress Corrosion Fracture" edited by T. N. Rhodin, Interscience Publishers, New York 1959.
4. "Tests for Determining Susceptibility to Stress-Corrosion Cracking," G. F. Sager, R. H. Brown and R. B. Mears, Symposium on Stress Corrosion Cracking of Metals, ASTM-AIME 1944, p. 258.
5. "Stress Corrosion Cracking of High Strength Steels," Aerojet General Corp., Azusa, California, Contract No. DA-04-495-ORD-3069 (Frankford Arsenal).
6. "Stress Corrosion Cracking of High Strength Steels and Alloys - Artificial Environments," Mellon Institute Contract No. DA-36-034-ORD-3277RD (Frankford Arsenal)
7. "Stress Corrosion of Steels for Aircraft and Missiles," E. H. Phelps and A. W. Loginow, Corrosion, Vol. 16 No. 7 (1960) p. 97.
8. Private communication with Dr. E. H. Phelps, A. W. Loginow and J. Bates, U.S. Steel Applied Research Laboratory, Monroeville, Pennsylvania, 20 Dec. 1962.

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