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**STRESS CORROSION CRACKING OF
AUSTENITIC STAINLESS STEEL**

STATUS REPORT - SEPTEMBER 1, 1964

M. R. Louthan, Jr.

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Savannah River Laboratory
Aiken, South Carolina

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STRESS CORROSION CRACKING OF AUSTENITIC STAINLESS STEEL
STATUS REPORT - SEPTEMBER 1, 1964

by

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February 1965

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ABSTRACT

Stress corrosion cracking of austenitic stainless steel is being investigated at Savannah River Laboratory to develop a fundamental understanding of the phenomenon. This is the second of a series of reports that present the status of studies relating specimen microstructure and dislocation substructure to the susceptibility to stress corrosion cracking. The results of this report, with the results previously reported in DP-907, show that anodic dissolution plays a major role in the nucleation and propagation of stress corrosion cracks. The susceptibilities of different heats of the same type of steel to cracking are quite different, and these differences are not attributable to differences in the composition of major alloying elements, material hardness, or dislocation substructure. It is also shown that the relative rate of preferential attack at dislocation arrays and stacking faults in thin foils of type 304 stainless steel exposed to dilute NaCl solutions increased with increasing temperature and was more rapid in heats of steel which were most susceptible to cracking in bulk tests.

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STRESS CORROSION CRACKING OF AUSTENITIC STAINLESS STEEL
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INTRODUCTION

Stress corrosion cracking has caused several failures of type 304 stainless steel reactor components and other types of equipment at the Savannah River Plant.⁽¹⁾ Therefore, a continuing investigation of transgranular stress corrosion cracking in austenitic stainless steels has been maintained at Savannah River Laboratory in an attempt to prevent further failures by developing a fundamental understanding of the phenomenon. The studies relating specimen microstructure and dislocation substructure to stress corrosion susceptibility were summarized in DP-907⁽²⁾, which presented the progress on the program to March 1964. This report presents the progress made since that date. Additional work to relate environmental conditions and susceptibility to cracking has been started and will be the topic of a subsequent report.

SUMMARY

Additional evidence was obtained that localized anodic dissolution plays a major role in the propagation of stress corrosion cracks. Several of the more important results are listed below:

- Differences were observed in the relative susceptibilities to crack nucleation and propagation for several different heats of types 304 and 316 stainless steels. These differences were not attributable to differences in the composition of major alloying elements, material hardness, or dislocation substructure. The crack propagation in each of the steels could be described by the empirical equation $D = C_1 + C_2 \log t$, where D = maximum crack depth, t = time of exposure, and C_1 and C_2 = constants characteristic of the heat of steel.
- The relative rate of preferential attack at dislocation arrays and stacking faults in thin foils of type 304 stainless steels exposed to dilute NaCl solutions increased with increasing temperature and was more rapid in heats that were most susceptible to cracking in bulk tests.
- The formation of stress corrosion cracks in type 304 stainless steel samples with various degrees of cold work and different surface preparations was inversely related to the extent of general corrosion that occurred in the sample exposed to the "crack-producing" solution. General corrosion of the samples was absent or slight when cracks formed.

DISCUSSION

EXPERIMENTAL PROCEDURES

The material used in these studies was from nine different heats, representing three types of austenitic stainless steel: five heats of type 304 and two heats each of types 304L and 316L. The chemical compositions of the major alloying elements in these steels are given in Table I.

Three general specimen designs were used for the corrosion studies:

- Square sheet samples approximately $1/2 \times 1/2 \times 0.06$ inch with various heat treatments, degrees of cold work, and surface preparations.
- U-bend samples with the general design shown in Figure 1.
- Thin foils (approximately 1000 \AA thick) prepared by wet grinding, mechanical polishing, and electrochemical thinning to the required thickness.

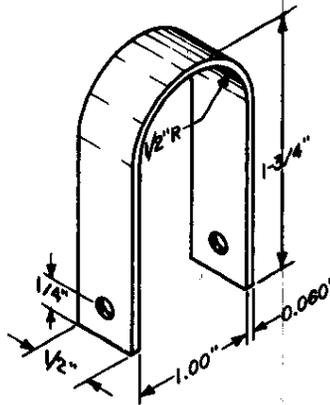


FIG. 1 DIMENSIONS OF U-BEND SPECIMEN

OBSERVATIONS

Relative Susceptibility Tests

English and Griess⁽³⁾ recently showed that although two heats of type 347 stainless steel were nearly identical in composition, specimens from one heat were consistently more susceptible to cracking in uranyl nitrate solutions than specimens from the other heat. Inspection of Savannah River Plant equipment had also indicated that

different heats of austenitic stainless steels had different cracking susceptibilities when exposed to dilute chloride solutions. Since these differences in relative susceptibility should be very important in understanding the mechanism of stress corrosion cracking, detailed investigations of the relative susceptibilities were begun.

Ten U-bend samples with surfaces wet ground with 600-grit abrasive were prepared from each of the five heats of type 304 stainless steel and the two heats of type 304L (Table I). The specimens were annealed one hour at 1010°C and then water quenched before the surface preparation. Duplicate U-bend specimens were tested in 42% MgCl₂ at 154°C for 20, 30, 40, 60, and 90 minutes. After exposure the samples were sectioned, metallographically polished and etched, and the maximum crack depth in each sample was measured.

TABLE I
Chemical Analysis of Steels Used in the Study

Type Steel	Heat Number	Composition, %							
		C	Mn	P	S	Si	Cr	Ni	Mo
304	31614	0.035	1.03	0.020	0.020	0.38	18.35	9.14	-
304 ^(a)	62232	0.044	0.80	0.021	0.016	0.58	18.68	9.13	0.26
304 ^(a)	22806	0.060	0.86	-	-	0.50	18.07	8.03	-
304	61124	0.050	0.72	0.024	0.025	0.62	18.83	8.86	-
304	41158	0.068	0.71	0.018	0.018	0.43	18.42	9.53	-
304L ^(a)	11880	0.022	1.16	0.010	0.016	0.42	18.20	9.45	-
304L ^(a)	50278	0.022	1.23	0.020	0.022	0.49	18.36	9.18	0.43
316L ^(a)	99888	0.028	1.52	0.014	0.019	0.53	17.50	13.68	2.76
316L ^(a)	32685	0.025	1.85	0.022	0.009	0.49	17.47	13.60	2.69

(a) Heats of steel used in the study of the influence of substructure.

The depth of cracking increased with increasing exposure time and a semilogarithmic plot of maximum crack depth against time showed that a relationship

$$D = C_1 + C_2 \log t \quad (1)$$

where D = maximum crack depth, t = exposure time, and C₁ and C₂ = constants, could be obtained for each heat of steel. The data were fitted to this type of equation and the empirical values of C₁ and C₂ were determined by the method of least squares. These results are

shown in Table II. The values of C_1 and C_2 were different for each heat of steel, but appeared to be related to each other in a linear fashion (Figure 2).

TABLE II

**An Empirical Analysis of Stress Corrosion Cracking
in U-Bend Samples of Stainless Steel^(a)**

Heat Number	Type Steel	Empirical Equation ^(b)	Correlation Coefficient
11880	304L	$D = 3.65 (\log t) - 3.92$	0.79
31614	304	$D = 6.19 (\log t) + 2$	0.88
22806	304	$D = 15.6 (\log t) - 11.9$	0.77
62232	304	$D = 21.87 (\log t) - 24.7$	0.85
41158	304	$D = 28.12 (\log t) - 29.2$	0.99
50278	304L	$D = 34.37 (\log t) - 38.74$	0.96
61124	304	$D = 53.12 (\log t) - 64.35$	0.98

(a) Empirical equations determined by method of least squares and relate to cracking of U-bend specimens in 42% $MgCl_2$ at 154°C. All the equations are of the form $D = C_1 + C_2 (\log t)$.

(b) D = maximum crack depth, mils
 t = exposure time, minutes

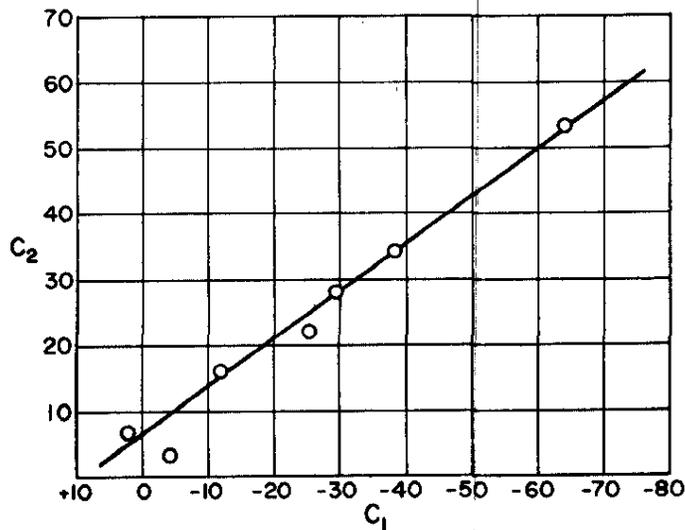


FIG. 2 CORRELATION OF EMPIRICAL CONSTANTS DETERMINED FOR DIFFERENT HEATS OF STAINLESS STEEL

The empirical relationship between crack depth and exposure time allows the calculation of the time for crack nucleation, t_0 , from the equation

$$t_0 = e^{-C_1/C_2} \quad (2)$$

and the rate of propagation from

$$\frac{dD}{dt} = \frac{0.4342 C_2}{t} \quad (3)$$

No apparent correlation between time for crack nucleation and the propagation rate was found, which suggests that different factors influence the two processes. Additionally, no correlations were found between t_0 or C_2 and either the chemical composition or the hardness of the heats. The effect of trace impurities has yet to be examined.

Influence of Metal Substructure on Relative Susceptibility

Previous work⁽⁴⁻¹⁰⁾ has shown that lattice defects have an important influence on stress corrosion cracking. A low stacking fault energy and the existence of coplanar arrays of dislocations are associated with increased susceptibility to cracking, whereas alloys that exhibit cellular arrangements of dislocation tangles show low susceptibility. These results indicated that the dislocation substructures might contribute to the differences in susceptibilities observed in the different heats of stainless steel. Experiments were therefore begun to characterize the substructure of different heats of stainless steel with the same nominal alloy compositions. Corrosion tests of two heats each of types 304, 304L, and 316L stainless steel (see Table I for compositions) were made, and the substructures examined by transmission electron microscopy.

All six heats of steel contained coplanar arrays of dislocations (Figure 3), and stacking faults were frequently observed in the 304 and 304L steels (Figure 4) and occasionally observed in the 316L steel; extended dislocation nodes were also seen in several of the 304 and 304L steels (Figure 5). These types of dislocation arrangements are indicative of low stacking fault energy.⁽⁸⁾

The cracking susceptibilities of the different heats of steel in both the as-quenched and cold-rolled conditions were determined by exposing U-bend samples from each heat of steel to boiling solutions of deionized water and 42% $MgCl_2$ at 154°C. The number of cracks that were visible along the edges of each sample were counted and the deepest crack was measured. These values were considered a measure of



NEG. 1786B

10,000X

a. Heat 31614 (304). Dislocation pileups at grain boundary.



NEG. 1769E

15,800X

b. Heat 99888 (316L). Dislocations beginning to form cell structure.

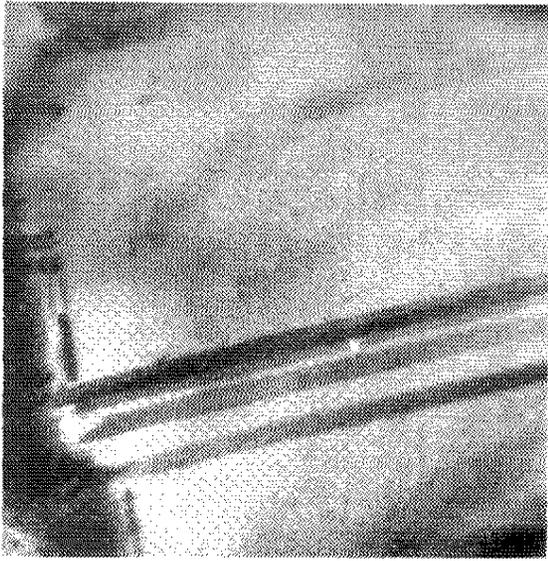


NEG. 1810C

21,000X

c. Heat 11880 (304L). Dislocation arrays and start of stacking fault formation.

FIG. 3 COPLANAR ARRAYS OF DISLOCATIONS IN SEVERAL HEATS OF STAINLESS STEEL



NEG. 1786D

33,000X

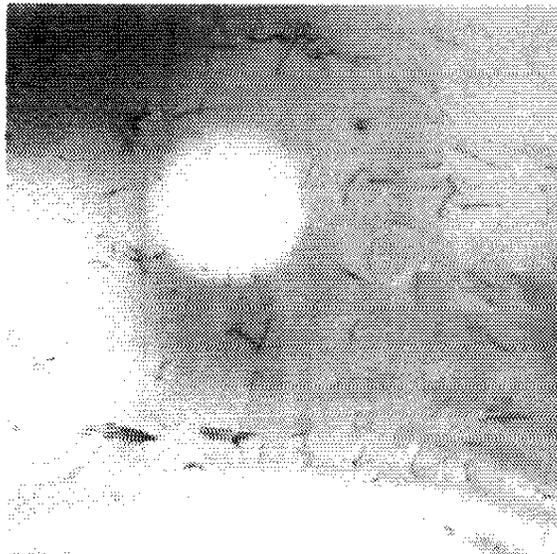
a. Heat 31614 (304).



NEG. 1861

7,800X

b. Heat 11880 (304L).

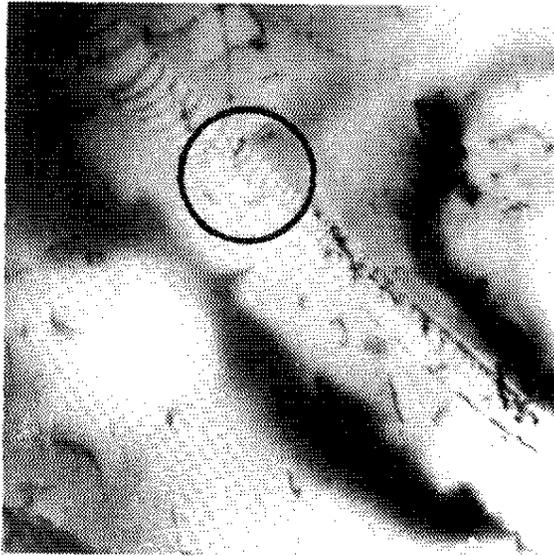


NEG. 1794B

21,000X

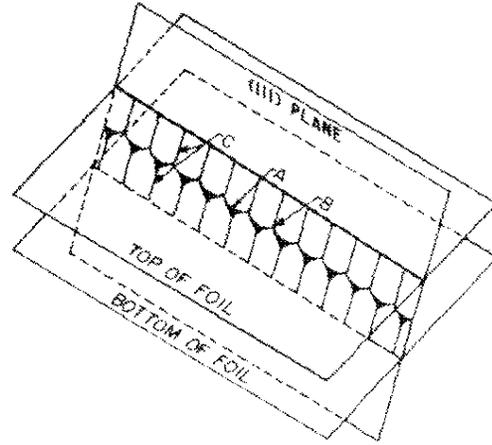
c. Heat 62232 (304).

FIG. 4 STACKING FAULTS IN SEVERAL HEATS OF STAINLESS STEEL



NEG. 1759C

50,000X



a. Dislocation network containing alternate extended and contracted dislocation nodes.

b. Sketch showing region circled in Figure 5a. Arrow A points to an extended dislocation node, B to a contracted node, and C to dislocations.

FIG. 5 EXTENDED DISLOCATION NODES IN TYPE 304 STAINLESS STEEL

the susceptibility to stress corrosion and are shown in Table III. (These data should not be compared to those reported in Table II since these data concern surface cracks while the data in Table II concern cracks in the interior of the sample.)

TABLE III

Relative Susceptibilities of U-Bend Samples to Stress Corrosion in 42% MgCl₂ Solutions

Heat Number	Type Steel	As-Quenched from 1010°C ^(a)		30% Cold-Rolled ^(b)	
		No. of Cracks	Maximum Depth, mils	No. of Cracks	Maximum Depth, mils
50278	304L	77	33	88	48
11880	304L	21	26	18	24
32625	316L	22	10	10	8
99888	316L	16	5	3	2
62232	304	74	32	46	53
22806	304	27	14	-	-

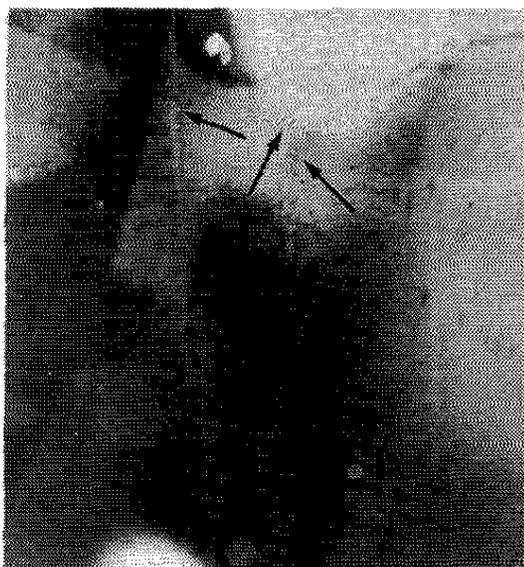
(a) Exposed 40 minutes

(b) Exposed 55 minutes

All six heats of steel were susceptible to transgranular stress corrosion cracking, as would be predicted from the substructure observations. Furthermore, the type 316L steels, which apparently had the highest stacking fault energies, showed the greatest resistance to cracking. However, samples from one heat of each type of steel were more resistant to cracking than were samples from the other heat of the same type of steel. This difference was apparent in both metallurgical conditions and could not be attributed to differences in stacking fault energy or dislocation substructure. Thus some other variable(s), in addition to the dislocation substructure, influences the susceptibility to transgranular stress corrosion cracking.

Thin Foil Exposures

Previous results had shown that preferential attack at crystallographic defects occurred in type 304 stainless steel foils exposed to dilute chloride solutions and that corrosion product formation accompanied that attack.⁽²⁾ This preferential attack produced crack-like voids in the foil and the extent of attack increased with increasing exposure time. These results indicated that crack propagation occurred by anodic dissolution at sites provided by crystallographic defects.



NEG. 1738B

11,100X

FIG. 6 LIMITED PREFERENTIAL ATTACK IN SPECIMENS EXPOSED 20 MINUTES TO DILUTE CHLORIDE SOLUTIONS AT 20°C (HEAT 31614). Arrows point to attacked regions.

In further tests, to illustrate the effects of increasing exposure temperature, thin foils of type 304 stainless steel as-quenched from 1010°C were examined as-thinned, after 20 minutes exposure to a solution of deionized H₂O-100 ppm Cl⁻ (as NaCl) at 20°C, and after 5 minutes exposure to the same chloride solution at 100°C. The as-thinned foils (from heat 31614, which had shown a very short crack initiation time in bulk samples) contained coplanar arrays of dislocations, stacking faults, and extended dislocation nodes, and exposure of the foils for 20 minutes at 20°C had little effect on the appearance of the foil; however some regions of preferential attack were observed at the foil edges (Figure 6). Exposure of the foils for 5 minutes in the chloride solutions at 100°C caused



NEG. 1862

7800X

FIG. 8 LIMITED PREFERENTIAL ATTACK IN FOIL EXPOSED 30 MINUTES IN DILUTE CHLORIDE SOLUTION AT 100°C (Heat 11880)

Relationship between Cracking and Corrosion

Corrosion of metal in an aqueous media should involve the formation of metal ions and the liberation of hydrogen or formation of hydroxyl ions, thereby changing the pH of the medium. Thus, some indication of the amount of corrosion should be provided by changes in pH of the environment.

Eight samples of type 304L stainless steel were quenched from 1010°C then tested in various solutions to determine whether the susceptibility to stress corrosion cracking could be related to changes in the pH of the solutions. These samples were exposed as U-bend specimens with dry ground surfaces for approximately 200 hours in boiling deionized water to which was added NaCl, LiCl, NaI, MgCl₂, or NiCl₂. The pH of each solution was measured at room temperature before and after test, and each sample was examined metallographically to determine whether cracking occurred. The results of this test (Table IV) show that, in general, the magnitude of the change in pH was related inversely to the extent of stress corrosion cracking that occurred in the sample.

Other samples of type 304 stainless steel, with various amounts of cold work and various surface preparations, were exposed for 24 hours in 42% MgCl₂ at 154°C. These specimens were 1/2 x 1/2 x 0.06 inch in size and were exposed without externally applied stress. The results (Table V) showed that whenever stress corrosion cracks were formed, little or no general corrosion of the sample occurred and when generalized corrosion occurred no cracks were formed.

TABLE IV

Correlation of pH Changes and Formation
of Stress Corrosion Cracks

<u>Chloride Content of Solution</u>	<u>Salt Added to Solution</u>	<u>pH of Solution</u>		<u>pH Change</u>	<u>Cracking Observed</u>
		<u>Before Test</u>	<u>After Test</u>		
100 ppm	NaCl	5.7	8.9	3.2	No
100 ppm	LiCl	5.6	8.6	3.0	No
1,000 ppm	NaCl	5.8	8.7	2.9	No
10,000 ppm	NaCl	6.1	8.8	2.7	No
100 ppm (I ⁻)	NaI	6.0	8.5	2.5	No
10% MgCl ₂	MgCl ₂	6.0	8.2	2.2	Yes
100 ppm	NiCl ₂	5.7	7.7	2.0	Yes
42% MgCl ₂	MgCl ₂	6.3	7.2	0.9	Yes

TABLE V

Correlation of Extent of Corrosion and
Formation of Stress Corrosion Cracks

<u>Sample Deformation History (% cold rolled)</u>	<u>Sample Surface Condition</u>	<u>Generalized Corrosion^(a)</u>	<u>Cracks in Surface</u>
Annealed	As-polished	Yes	No
5	"	"	"
10	"	"	"
15	"	"	"
20	"	"	"
30	"	"	"
Annealed	Wet-ground	No	Yes
5	"	"	"
10	"	Yes	No
15	"	Slight	Yes
20	"	No	"
30	"	Yes	No
Annealed	Dry-ground	No	Yes
5	"	Slight	"
10	"	No	"
15	"	"	"
20	"	"	"
30	"	"	"

(a) Determined by optical microscopy. If oxide deposits were observable, generalized corrosion was noted to have occurred.

These results indicate that when general corrosion occurs the anodic regions in the sample are relatively large and pits are produced, and that when little or no general corrosion occurs the anodic regions are highly localized and corrosion in these regions produces crack-like voids in the metal. The inverse relationships between pH changes and extent of cracking can be explained if one assumes that the quantity of metal corroded decreases with decreasing size of the anodic region. Thus if the anodic regions were highly localized, the quantity of corrosion was low, the pH change was small, and the corrosion caused the production of crack-like voids in the metal, whereas when the anodic regions were larger, more generalized corrosion occurred, the pH changes were larger, and pits were produced in the metal.

The results to date do not offer any explanation of the effect the salt in solution on the susceptibility to stress corrosion cracking; however, they do show that such an effect exists (samples cracked in 100 ppm Cl^- solutions when NiCl_2 was the chloride-bearing salt but did not crack when the chloride was added as NaCl). Work along these lines is therefore continuing to determine the role of the anion in stress corrosion cracking.

Stress-Aging Tests

In 1963, Swann and Pickering⁽¹¹⁾ reported that "studies of the stress-aging yield phenomenon in alloys susceptible to transgranular stress corrosion cracking indicate that segregation of substitutional atoms to dislocations occurs during a stress corrosion cracking test." They studied stress aging in several alloys and showed that "there is a coincidence between the temperature ranges corresponding to the rapid decrease in cracking time and the rapid increase in the stress-aging effect." Since cathodic charging experiments at SRL had previously indicated a possible relationship between hydrogen pickup and stress corrosion cracking,⁽²⁾ experiments were initiated to determine whether cathodic charging had any effect on the stress-aging phenomena.

Stress-aging tests were performed using annealed and quenched tensile specimens (Figure 9) machined from 304 stainless steel heat 31614. Tests were conducted at room temperature, 100, 125, 150, 175, and 200°C. The specimens were strained several percent before straining

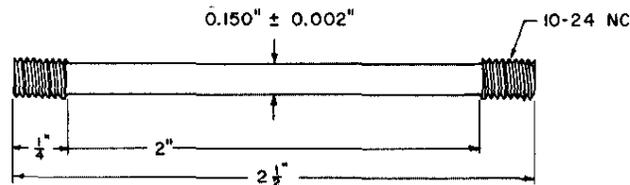


FIG. 9 DIMENSIONS OF TENSILE SPECIMEN

was discontinued, and the specimen was held at load (stress-aged) for 10 minutes. After aging, the specimens were strained several percent more. This procedure produced a stress-aging peak in the load-elongation curve (Figure 10). This peak was due to the increase in stress (load) required to re-initiate plastic flow. The height of this peak was a function of test temperature (Figure 11). Similar specimens were tested immediately after a cathodic charging treatment of 2 hours in a 5% H₂SO₄ solution at a current density of 0.25 amp/cm². No stress aging effect was observed in four of the five cathodic-charged specimens, and the magnitude of the effect in the remaining sample was less than in uncharged samples tested at the same temperature.

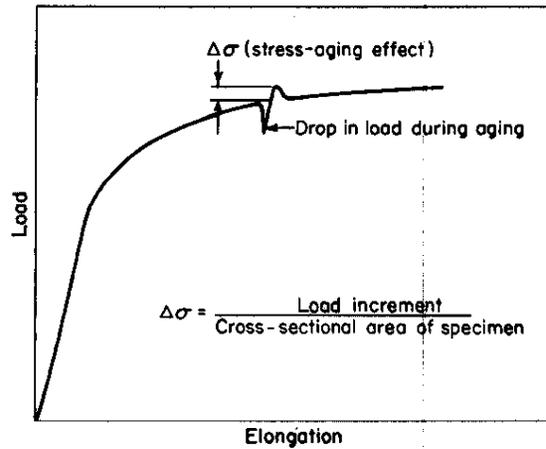


FIG. 10 SHAPE OF TYPICAL LOAD-ELONGATION CURVE SHOWING STRESS-AGING PEAK

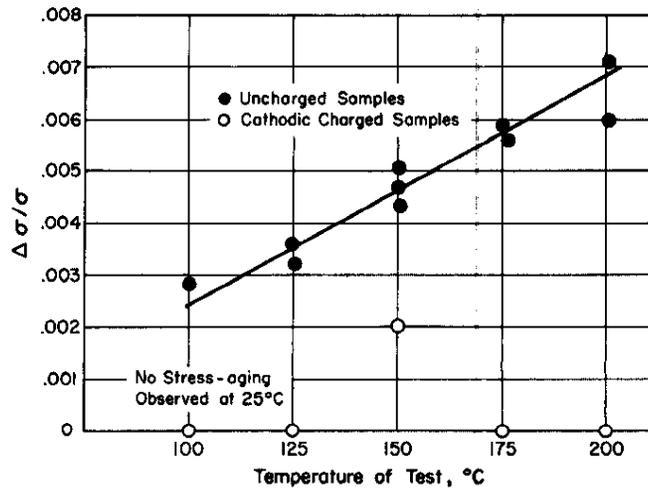


FIG. 11 RELATIONSHIP BETWEEN STRESS-AGING EFFECT AND TEST TEMPERATURE

These results indicate that the introduction of hydrogen decreased the response to stress aging in much the same way as Swann and Pickering⁽¹¹⁾ had observed for nitrogen. Nitrogen has also been shown to increase the susceptibility to stress corrosion cracking,⁽¹²⁾ however, this increase in susceptibility has been rationalized as being caused by the effect of nitrogen on the degree of local order,⁽⁹⁾ not because of nitrogen segregation to dislocations. Additional experiments are necessary to clarify the role of hydrogen and nitrogen in the stress-aging and stress corrosion processes.

PROGRAM

The observations to date have indicated that anodic dissolution plays a major role in the stress corrosion processes although no definitive experiment has yet been derived that illustrates the exact mechanism. The role hydrogen plays in the cracking processes is not yet clear. An extensive program is currently underway to determine the relative cracking susceptibility for several heats of types 304, 304L, 316, 316L, 321, and 347 stainless steels. These relative susceptibilities will be compared to the mechanical properties, anodic polarization curves, and impurity content of each heat of steel to determine if any correlations exist.

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