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ATOMIC ENERGY COMMISSION
RESEARCH AND DEVELOPMENT PROGRAM

PROGRESS REPORT

MASTER

CORRELATIONS BETWEEN SENSITIZATION AND
STRESS CORROSION CRACKING OF 300 SERIES
STAINLESS STEELS

For Period
July 1 to September 30, 1965

RELEASED FOR ANNOUNCEMENT

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Issued October 1965

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For Period
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Prepared by:

J. J. Koziol
S. S. Christopher

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W. P. Chernock
W. P. Chernock, Manager
Nuclear Laboratories

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Pittsburgh, Pennsylvania

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Pacific Northwest Laboratory
Battelle Memorial Institute
P.O. Box 999
Richland, Washington 99352

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CORRELATIONS BETWEEN SENSITIZATION AND
STRESS CORROSION CRACKING OF 300 SERIES
STAINLESS STEELS

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FOREWORD

The United States and the European Atomic Energy Community (EURATOM) on May 29 and June 18, 1958, signed an agreement which provides a basis for cooperation in programs for the advancement of the peaceful applications of atomic energy. This agreement, in part, provides for the establishment of a Joint U.S. -Euratom research and development program which is aimed at reactors to be constructed in Europe under the Joint Program.

The work described in this report represents the Joint U.S. -Euratom effort which is in keeping with the spirit of cooperation in contributing to the common good by the sharing of scientific and technical information and minimizing the duplication of effort by the limited pool of technical talent available in Western Europe and the United States.

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CORRELATIONS BETWEEN SENSITIZATION AND
STRESS CORROSION CRACKING OF 300 SERIES
STAINLESS STEELS

By

J. J. Koziol and S. S. Christopher

ABSTRACT

Effort during the present reporting period was concentrated on procurement and installation of the test equipment, procurement of specimen materials, and design and fabrication of the test specimens. Checkout of the test system and chemical sampling procedures are currently underway. Arrangements for drawing, swaging and application of a nickel surface layer to the tubing are being made.

I. SUMMARY

During this reporting period, procurement and installation of an Inconel 600 autoclave was completed. The Type 304 and 347 test specimens will be exposed to an environment containing chloride and oxygen at 316°C.

Design of the test specimens was completed and procurement of materials for their fabrication was also completed. These specimens can be internally pressurized to stress the steel sample to 75% of yield while being exposed to the autoclave solution at 316°C. A reduced internal volume of the specimen is used to minimize hazards in the event of catastrophic failure.

Surface roughness determinations were made on both types of steel and mechanical properties measurements were made at room temperature and at 316°C.

Sections of tubing were exposed to a 650 °C heat treatment for 20 hours followed by furnace cooling to promote carbide precipitation. Metallographic examinations were conducted on both the solution annealed and the sensitized materials. Grain size measurements were also made.

Specimens for the solution annealed and the sensitized conditions (pre-oxidized and unoxidized) were fabricated. Preparations are being made for swaging, drawing and application of a nickel surface layer to the stainless steel tubing. Testing of the specimens in the four surface conditions will be undertaken during the next reporting period.

II. INTRODUCTION

This program was originally aimed at demonstrating the differences in transgranular stress corrosion susceptibility which exists for stabilized and unstabilized austenitic stainless steels, namely Types 304 and 347. Various heat treatment schedules were investigated to determine optimum time and temperature relationships for carbide precipitation to occur in both the Type 304 and 347 steels. Both steels were tested in the fully annealed and heat treated conditions in which carbide precipitates were found in the grain boundaries.

One of the aims of the program was to determine the effect of pre-oxidation on the initiation of transgranular cracking. Since 304 stainless steel is not stabilized by the addition of niobium or tantalum, it was predicted that during the pre-oxidation heat treatment the grain boundaries of this material would be more severely attacked than the grain boundaries of the stabilized 347. This would result in a difference in behavior of the two steels with the Type 304 being more susceptible to transgranular cracking than the Type 347.

Since initiation of cracks at pre-oxidized grain boundaries was of particular interest rather than catastrophic failures, the U-bend configuration was selected for testing. Rectangular samples measuring 2.5 x 7.6 cm were cut from 0.76 mm sheet of each steel and wrapped at room temperature around a 1.27 cm mandrel to an angular separation of $50 \pm 20^\circ$ to form the U-bend.

Tests were performed in a static autoclave at 190°C in water containing 15 ppm dissolved oxygen and 25 ppm chloride ions. In this environment, all specimens of both steels ran in excess of 1500 hours with no evidence of transgranular or intergranular attack. It was concluded that stress relaxation of the U-bend specimens occurred during the test, and although failures would have occurred with longer times, the test environment was not severe enough to initiate transgranular cracking within a reasonable test period.

Additional tests were then conducted in a more aggressive environment. The test configuration was changed from the U-bend specimens to tubular specimens which were internally pressurized to 75% of their yield strength. The test environment was changed to steam containing 25 ppm chloride and 8 ppm oxygen. In this series of tests, failure occurred in less than 24 hours and no definitive conclusions were made.

In addition to the effect of pre-oxidation on the initiation of transgranular cracking, the surface condition is also an important factor in the life expectancy of stainless steel in a typical water reactor environment. VBWR⁽¹⁾ studies have demonstrated large differences in the performance of swaged stainless steel tubing and conventional tubing fabricated by drawing. Work by Neilson⁽²⁾ has shown that pile-ups of dislocations at particles or oxide films can lead to localized attack which in turn can contribute transgranular crack formation and propagation.

These observations were incorporated into the present program and the scope of work was expanded to include a study of a number of surface conditions which will permit an evaluation of methods for preventing stress corrosion cracking during the operating life of a reactor element.

III. EXPERIMENTAL PROGRAM

A. Scope of Work

The objective of this program is to evaluate the effects of surface conditions on the susceptibility of Type 304 and Type 347 stainless steels to transgranular cracking. Investigation of the effect of four surface conditions on the susceptibility to transgranular cracking shall be conducted in order to permit an evaluation of methods of preventing stress corrosion cracking of a fuel element cladding during its operating lifetime. The conditions to be evaluated are:

1. Pre-oxidized tubing
2. As-drawn tubing
3. Swaged tubing
4. Tubing with a surface diffusion layer of nickel

1. Pre-Oxidized Tubing

Pre-oxidized specimens will be evaluated to determine the effect of grain boundary oxidation on the initiation of transgranular cracks. This phase of study, which is a continuation of the earlier phase of the program, will utilize specimens pre-oxidized in 316°C high purity water and tested at a stress level of 75% of the tensile yield strength at 316°C.

2. As-Drawn Tubing

As-drawn tubing with approximately 10% cold work, will be evaluated in order to provide a baseline for comparison with swaged and annealed tubing.

3. Swaged Tubing

Swaged tubing with 10 and 15% cold work will be used to evaluate the effects of this process on the susceptibility of Type 304 and 347 stainless steel to failure by transgranular cracking. These levels of cold work will be sufficient

to put the surface of the tubing in compression, thus higher than normal tensile stresses must be generated to initiate a crack. Specimens will be stressed in a corrosive environment to 75% of the tensile yield strength as determined by elevated temperature tensile tests.

4. Tubing with a surface diffusion layer of nickel

Irradiation experience in VBWR has demonstrated an improved corrosion resistance of a high nickel content alloy (Incoloy 800) with respect to Type 304 stainless steel. However, one of the disadvantages is the alloys high nickel and chromium content which results in a relatively high neutron cross section.

One method of obtaining the corrosion resistance demonstrated by the high nickel content alloys, without substantially increasing the neutron cross section, would be to coat the surface of stainless steel with a thin coating of nickel which would subsequently be diffused into the surface of the tubing. Thus the corrosion resistance is obtained on the outside diameter where it is needed and the neutron cross section of the alloy would not be changed to an appreciable extent.

The tests will be performed in a static autoclave with 316°C pressurized water containing 1000 ppm Cl as NaCl. Pure oxygen will be introduced into the unit by bubbling oxygen through the autoclave. The internal pressure will be raised to 100 psi before the inlet is closed.

After the oxygen is introduced, the specimens will be internally pressurized with argon and the water temperature will be raised to 316°C. The oxygen and chloride concentration at room temperature will be analyzed and will serve as a control of conditions at 316°C.

B. Program

A program to accomplish the objectives of the investigation has been established. Basically, test specimens fabricated using tubular samples with specific surface conditions will be exposed to a corrosive environment in an autoclave until failure occurs. Table I lists the exposure conditions which will be investigated.

The solution to which the samples are exposed will be analyzed for both oxygen and chloride. Oxygen analyses using the Winkler method for concentrations above 0.05 ppm and the carmine indigo method for lower oxygen concentrations will be used. Chloride analysis will be made titrimetrically using mercuric nitrate and diphenyl carbazone indicator.

Post-test evaluations of the steels will be primarily through metallographic examinations of the samples. An attempt to establish a correlation between surface condition and mode of failure will be made.

C. Equipment

The Type 304 and 347 stainless steel samples will be exposed to the corrosive environment at 316°C in a 5.08 cm (2 in.) ID x 45.7 cm (18 in.) long Inconel 600 autoclave rated at 5000 psig at 370°C. This rating is well above the test conditions of 316°C and 1530 psig. A view of the overall test system is presented in Figure 1 and a schematic is in Figure 2. A closeup of the autoclave head and the tubing arrangement is presented in Figure 3.

The autoclave instrumentation is composed of a pressure recorder (PRC-3) which controls the autoclave heaters in addition to recording the autoclave pressure. A temperature indicator (TIC) provides overpressure protection through a high temperature heater cutout as well as indicating the autoclave temperature. Should both these controllers fail, overpressure protection is provided by a rupture disc. An expansion loop in the discharge piping from the safety head is provided to prevent loading of the piping should the disc rupture.

OVERALL VIEW OF TEST SYSTEM

FIGURE 1

SCHEMATIC DIAGRAM OF AUTOCLAVE PIPING

VIEW OF AUTOCLAVE HEAD AND PIPING

FIGURE 3

TABLE I

PRE - TEST PROGRAM

<u>Surface Condition</u>	Type 304		Type 347		<u>Mechanical Tests (304 and 347)</u>	Metallography (304 and 347)	
	<u>Pre-Ox</u>	<u>Un-Ox</u>	<u>Pre-Ox</u>	<u>Un-Ox</u>		<u>Pre-Test</u>	<u>Post-Test</u>
As-Drawn	X	X	X	X	20°C and 316°C	X	X
Solution Annealed	X	X	X	X	20°C and 316°C	X	X
Solution Annealed and Sensitized	X	X	X	X	---	X	X
Swaged - 10%	X	X	X	X	20°C and 316°C	X	X
- 15%	X	X	X	X	20°C and 316°C	X	X
Nickel Surface		X		X	---	X	X

Each test specimen is provided with an isolation valve and a connection for internal pressurization with argon using a gas booster pump. Instrumentation consists of a pressure recorder (PRC 1 and 2) connected to each of the specimens. These recorders are equipped with pressure switches wired so that the autoclave heaters are automatically de-energized when both specimens have failed. Failure of one specimen will not affect the normal automatic operation of the heaters.

The piping and fittings in the system are either Type 304 or 316 stainless steel except for the fill and sample line at the bottom of the autoclave. Since the line is exposed, at least at one end, to the full autoclave conditions, it was felt that this line should be Inconel 600 to avoid the dangers of failure during operation. All other lines will be replaced with each test or operate at such a low stress level that difficulties are not anticipated.

The electrical control of this system is shown schematically in Figure 4. A switch-on pressure recorder, PRC-3, cycles the autoclave heaters as necessary to maintain the desired pressure. Upon the failure of both specimens (indicated by the loss of specimen internal pressure,) this circuit will be broken by the opening of the switches on PRC-1 and 2. Over-pressure protection is provided by a contact on the autoclave temperature indicator (TIC) which opens when the upper temperature limit is reached. Normal cycling of the autoclave heaters is done by contactor HC-1. When the contact on TIC opens, a second contactor HC-2 is de-energized as well. These two contactors are wired in series; should an over temperature condition occur, the contacts on HC-2 serve as a backup to contact on HC-1.

SCHEMATIC DIAGRAM OF ELECTRICAL SYSTEM

FIGURE 4

D. Test Specimens

1. Materials

The Type 304 and 347 stainless steel tubing used for the samples was purchased in the solution annealed condition. The chemical composition and mechanical properties are presented in Tables II and III. Surface finish measurements made with a Brush Instrument Company "Surfindicator", Model No. BL-110, indicated a finish of 22 microinches on the Type 304 tubing and 20 microinches on the Type 347 tubing.

Metallographic examination of the as-received tubing in the solution annealed condition showed a normal carbide distribution for this condition and an ASTM grain size of 5 in the Type 304 material. The Type 347 material was essentially free of carbides and exhibited an ASTM grain size of 8 or finer. Figures 5 and 6 show the microstructures of the Type 304 and 347 steels in the solution annealed condition.

The Type 304 and 347 materials were exposed to a sensitizing heat treatment to promote carbide precipitation. Tubular samples were heat treated in vacuum at 650 °C for 20 hours followed by a slow furnace cool. Metallographic examination of these specimens showed essentially no increase in grain size. The Type 347 material showed a slight increase in the amount of carbide precipitation as can be seen in Figure 7. The Type 304 material on the other hand showed a substantial amount of carbide precipitate in the grain boundaries, as can be seen in Figure 8.

Tubular specimens in the as-drawn, swaged, and nickel diffusion surface are currently being prepared and evaluations of these samples are incomplete.

2. Assembly

The test specimens were designed to permit the tubular steel sample, possessing the desired surface condition, to be internally stressed while

3527

AQUA REGIA ETCH

250X

MICROSTRUCTURE OF SOLUTION ANNEALED TYPE 304 STAINLESS STEEL

FIGURE 5

3532

250X

MICROSTRUCTURE OF SOLUTION ANNEALED TYPE 347 STAINLESS STEEL

FIGURE 6

3607

ELECTROLYTIC ETCH

750X

MICROSTRUCTURE OF TYPE 347 STAINLESS STEEL AFTER SENSITIZING HEAT TREATMENT

FIGURE 7

3594

ELECTROLYTIC ETCH

750X

MICROSTRUCTURE OF TYPE 304 STAINLESS STEEL AFTER SENSITIZING HEAT TREATMENT

TABLE II

CHEMICAL COMPOSITION

<u>Type of Steel</u>	<u>Chemical Composition, wt%</u>						<u>Cr</u>	<u>Nb + Ta</u>
	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Ni</u>		
304	0.05	1.71	0.011	0.015	0.43	10.45	18.62	--
347	0.06	1.74	0.016	0.011	0.60	11.21	17.99	0.88

TABLE III

MECHANICAL PROPERTIES

<u>Material</u>	<u>Temperature</u>	<u>Tensile (psi)</u>	<u>Yield (psi)</u>	<u>Elongation (%)</u>	<u>R.A. (%)</u>
304 (Annealed)	R.T.	97,500	52,900	60	50
		97,000	52,300	60	52.1
	316 °C	77,700	44,300	30	21.8
		78,000	43,000	30	19.1
347 (Annealed)	R.T.	103,700	53,900	43	43.5
		104,000	54,600	42	43.7
	316°C	74,400	42,000	24	20.4
		72,700	42,300	23	22.2

being externally exposed to an elevated temperature corrosive environment. A diagram of the test specimen is shown in Figure 9 and the dimensions of the test specimen components are listed in Table IV. The finished components as well as an assembled specimen are shown in Figure 10.

Standard TIG welding techniques were used for specimen assembly. The pressure tube was welded to the top end cap which was then welded to the Type 304 or 347 stainless steel sample. After the insert plug was placed within the sample, the bottom end plug was welded to the tubing. Figure 11 shows the assembly procedure for the pressure tube to top end cap. Figure 12 and 13 show a similar procedure for the top and bottom end caps and Figure 14 presents a closeup view of a typical end cap to tubing weld.

The test specimens were then installed in the autoclave head. Figure 15 shows the appearance of two test specimens (304 and 347) in the autoclave head.

E. Present Status

During this reporting period, the major effort was placed in procurement and installation of the test equipment, design of the test specimens and procurement of the specimen materials.

The Inconel 600 autoclave has been installed and checkout of the system is in progress. Type 304 and 347 stainless steel tubing was procured. Mechanical tests at elevated and ambient temperatures have been performed and metallographic evaluations are being conducted. A portion of the tubing has been exposed to a sensitizing heat treatment and metallographic evaluations of these structures are underway.

TEST SPECIMEN ASSEMBLY

FIGURE 9

3542

VIEW OF SPECIMEN COMPONENTS AND ASSEMBLED TEST SPECIMEN

3536

PRESSURE TUBE TO TOP END CAP ASSEMBLY

FIGURE 11

3538

TOP END CAP TO TUBE ASSEMBLY

3540

BOTTOM END CAP TO TUBE ASSEMBLY

FIGURE 13

3541

TYPICAL END CAP TO TUBE WELD

3583

TEST SPECIMENS INSTALLED IN AUTOCLAVE HEAD

TABLE IV

DIMENSIONS OF TEST SPECIMEN COMPONENTS

<u>Component</u>	<u>Material</u>	<u>OD</u>		<u>Dimensions</u> <u>ID</u>		<u>Length</u>	
		<u>cm</u>	<u>in.</u>	<u>cm</u>	<u>in.</u>	<u>cm</u>	<u>in.</u>
Specimen Tube	304 347	1.27	0.5	1.168	0.460	12.7	5.0
Top End Plug	304	1.168	0.460	0.318	0.125	1.905	0.750
Bottom End Plug	304	1.168	0.460	--	--	1.905	0.750
Insert Plug	304	1.067	0.420	--	--	8.763	3.450
Pressure Tube	304	0.318	0.125	0.159	0.0625	50.8	20.0

Test specimens have been fabricated for all of the solution annealed and solution annealed and sensitized conditions.

Arrangements are currently being made for drawing and swaging the necessary tubing for the specimens to complete those phases of the program. Preparations are also underway for application of the nickel layer to the tubing.

IV. FUTURE EFFORT

Upon completion of the autoclave system checkout, testing of the specimens in the solution annealed and in the solution annealed and sensitized conditions will be started. During this time, the as-drawn, swaged, and nickel surface layer specimens will be fabricated and tested. The testing phase of the program should be completed during the next reporting period.

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2. Neilson, N. A., "The Nature of Initial Corrosion of Stressed Austenitic Stainless Steel by Chloride Ions, Platinum Decoration of Active Sites," Second International Congress on Metallic Corrosion, New York, March 1963.