91

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

ORNL-2673
Chemistry-General

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0351035 0

THOREX PILOT PLANT CORROSION STUDIES: 1. CORROSION OF TYPES 304L AND 309SCb STAINLESS STEEL DURING PRODUCTION AND DEVELOPMENT PERIODS

J. L. English

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this document, send in name with document and the library will arrange a loan.

OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

Printed in USA. Price $2.25 Available from the

Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

ORNL-2673

Contract No. W-7405-eng-26

REACTOR EXPERIMENTAL ENGINEERING DIVISION

THOREX PILOT PLANT CORROSION STUDIES:

I. CORROSION OF TYPES 304L AND 309SCb STAINLESS STEEL DURING PRODUCTION AND DEVELOPMENT PERIODS

J. L. English

DATE ISSUED

APR 27 1959

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
Operated by
UNION CARBIDE CORPORATION
for the
U. S. ATOMIC ENERGY COMMISSION

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0351035 0

CONTENTS

		Page No.
1.	Abstract	1
2.	Introduction	1
3.	Experimental Procedure	2
	3.1 Description of Corrosion Specimens	2
	3.2 History of Test Materials	5
	3.3 Boiling 65% HNO_3 Test Results	7
	3.4 Examination of Exposed Test Specimens	7
	3.5 Determination of Corrosion Rates	8
4.	Corrosion During Production Period	8
	4.1 Dissolver Tank	8
	4.2 Feed Adjustment Tank	19
	4.3 AP Catch Tank	26
	4.4 BT Vapor Separator Tank	30
5.	Corrosion During Development Period	38
	5.1 Feed Adjustment Tank	38
6.	Corrosion During Production-Development Period	44
	6.1 Feed Adjustment Tank	44
	6.2 Miscellaneous Information	48
7.	General Discussion	62
8.	Summary	68
9.	Acknowledgments	69
10.	References	70

1. ABSTRACT

The corrosion behavior of types 304L and 309SCb stainless steel was examined during the production and first half of the development periods of Thorex pilot plant operation between December 15, 1954, and November 10, 1955. The process vessels in which corrosion data were obtained included the batch dissolver tank, the feed adjustment tank, the AP catch tank, and the BT vapor separator. There was no significant difference between the corrosion resistance of the two alloys in any of the environments. Vapor-phase corrosion attack in some vessels was more severe than solution-phase attack in the same vessels. Dependent upon the particular process environment, corrosion rates in the vapor phase ranged from 0.1 to 46 mpy; solution-phase corrosion rates ranged from 0.1 to 28 mpy. The most severe attack in both solution and vapor phases occurred in the feed adjustment tank. There was serious corrosion in the BT vapor separator also. Corrosion was moderate (8 mpy or less) in the batch dissolver tank and was almost negligible (0.2 mpy or less) in the AP catch tank. The attack on rolled surfaces of both types of stainless steel was uniform, with the exception that shallow, intergranular penetration was experienced in the more corrosive environments. Edge-surface attack was intense in the latter environments also and introduced some question as to the validity of corrosion rates based upon total attack on specimens. Neither of the alloys, initially stressed at 35,000 psi, experienced stress-corrosion cracking in any of the environments, nor was there any evidence of a stress-induced acceleration in the rates of attack. Weld specimens of both alloys underwent preferential attack at the weld-parent metal interface in a number of the environments; the frequency of the attack was random in nature, however. Neither alloy was subject to crevice corrosion nor was accelerated corrosion at solution-vapor interfaces observed.

2. INTRODUCTION

The Thorex pilot plant was constructed for the purpose of separating and decontaminating thorium, uranium-233, and protactinium-233 in neutron-irradiated thorium metal. Nitric acid is used to dissolve thorium metal. The separations are made by a solvent extraction process using tri-n-butyl phosphate as the extractant, and aluminum nitrate and nitric acid as the aqueous salting agents. Uranium-233 is eventually isolated by an ion exchange process.

The current Thorex process flowsheet includes four major operational features:

(a) thorium metal dissolution and feed preparation,

(b) separation of thorium-232 and uranium-233 from protactinium-233 and associated fission products by extraction with tri-n-butyl phosphate solvent,

(c) partitioning of thorium-232 and uranium-233 with subsequent isolation of the two products, and

(d) solvent recovery.

The Corrosion Program. The design and the preliminary construction of the ORNL Thorex pilot plant were started in June 1953. Simultaneously, preparations were begun on a corrosion-test program for insertion of specimens in various process vessels in the pilot plant.[1] The decision for a corrosion-test program was predicated by the need for information on the corrosion behavior of stainless alloys during operation of the chemical separation and recovery plant. Such information was necessary to assist in the future selection of construction materials for a large-scale thorium processing plant.

The choice of types of stainless steel for exposure in the pilot plant was based upon the results of unpublished laboratory tests. Among the various metals and alloys examined, types 304L and 309SCb stainless steel were two of the more attractive alloys that offered promise corrosion-wise as construction materials. Type 347 stainless steel warranted some consideration, although its corrosion resistance was less satisfactory than that of the other two alloys in most of the laboratory environments.

The present report is concerned with the corrosion program that was conducted during the production period and the first half of the development period of operation of the Thorex pilot plant. As the name implies, the production period (December 15, 1954, to March 4, 1955) dealt with the separation and isolation of uranium-233 as the primary objective. The development period (March 4 to November 10, 1955) emphasized studies of the process although uranium-233 separation continued. The results of tests conducted during the second half of the development period will be discussed in a future report.

Complete details of the corrosion program for the Thorex pilot plant have been described elsewhere.[1,2] A detailed description of the environmental conditions and exposure times for specimens during the above two phases of pilot plant operation has been reported.[3,4] Information on the history, design, development, construction, and operation of the pilot plant has been included in numerous other reports.[5-11]

3. EXPERIMENTAL PROCEDURE

3.1 Description of Corrosion Specimens. A simple-beam constant-strain type of stress assembly[12,13] was selected for the corrosion specimens. The assembly consisted of (1) a specimen under applied stress, (2) a fulcrum used as a control specimen, and (3) a welded base plate. Auxiliary components included two sets of studs and nuts for applying the desired stress. All components in an assembly were machined from the same type of stainless steel to eliminate the possibility of galvanic corrosion.

A nonwelded base plate is generally used in the conventional simple-beam assembly. However, welds were incorporated in the base plates of the pilot plant stress assemblies to permit examination of weld-

corrosion behavior. Two transverse welds were made across each base plate; the roots of the welds were rotated 180 degrees from each other along the longitudinal axis of the base plate. One face of each base plate was machined to remove excess weld-metal deposit; the opposite face was left in the as-welded condition.

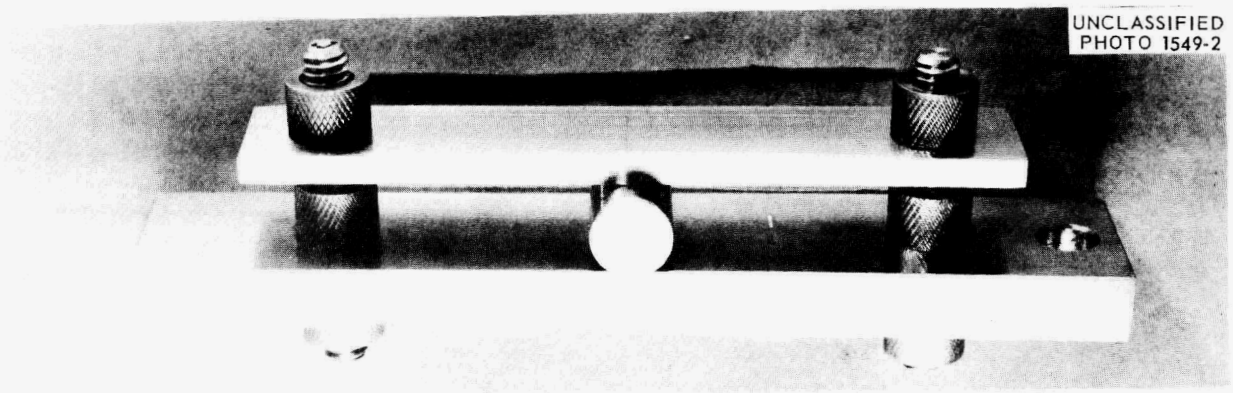
The materials used in fabrication of the assemblies were 1/8-in.-thick sheet for the stress specimen, 3/8-in.-thick plate for the base plate, 3/8-in.-dia. rod for the fulcrum and nuts, and 1/4-in.-dia. rod for the studs. The stress specimen was 1/8 x 5/8 x 3 1/2 in.; the base plate was 5/16 x 7/8 x 4 1/2 in.; and the fulcrum was 3/8 in. in diameter by 7/8 in. long. The total surface area of a complete assembly was approximately 21.7 in.^{2} (140 cm^2). The individual areas of the stress specimen, fulcrum, and base plate were 5.4 in.^{2} (34.9 cm^2), 1.3 in.^{2} (8.1 cm^2), and 12.3 in.^{2} (79.4 cm^2), respectively. Several photographs of an assembled unit appear in Fig. 1.

Uncoated type 308L stainless steel rod was used to make the Heliarc welds in the type 304L base plates; uncoated type 309SCb rod was used for the type 309SCb base plates. One base plate of type 347 stainless steel used in the tests was Heliarc welded with uncoated type 347 weld rod, and one was welded with uncoated type 308L rod.

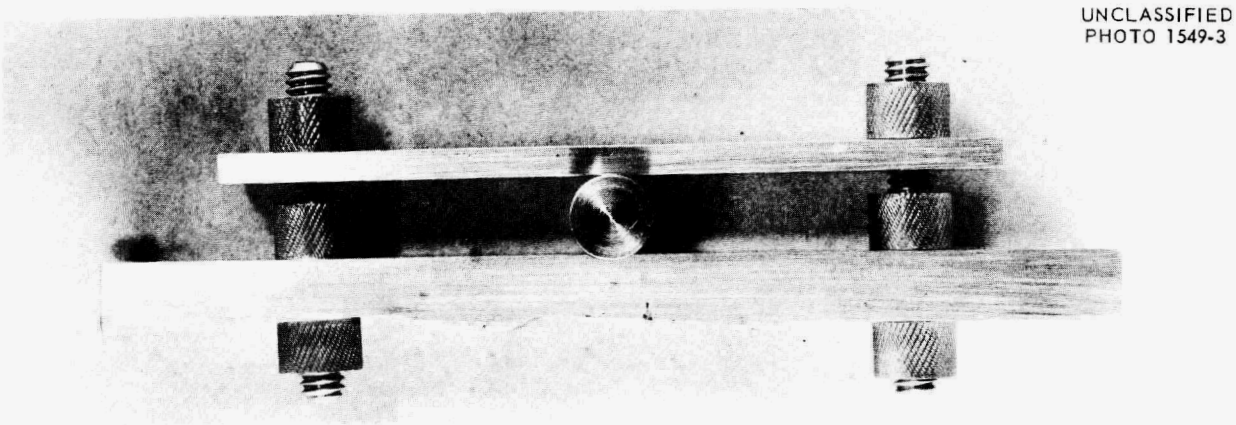
Before stressing, all components in an assembly were degreased in acetone and alcohol. The stress specimen, fulcrum, and base plate were dried at approximately 100°C and weighed to the nearest 0.1 $\pm$ 0.2 mg. The stress specimen was stressed to 35,000 psi which was approximately 85% of the annealed yield strength of each alloy. The applied stress was measured with the aid of a strain gage.

Considerable laboratory experience with the simple-beam assembly attested to its adaptability for a pilot-plant type of corrosion investigation. In addition to affording a means for evaluating stress-corrosion cracking behavior, the assembly was also suited for weld and crevice corrosion studies. For a qualitative examination of solution-vapor interface attack in the various process vessels of the pilot plant, it was planned to make use of the stainless steel rods which supported the assemblies in the vessels.

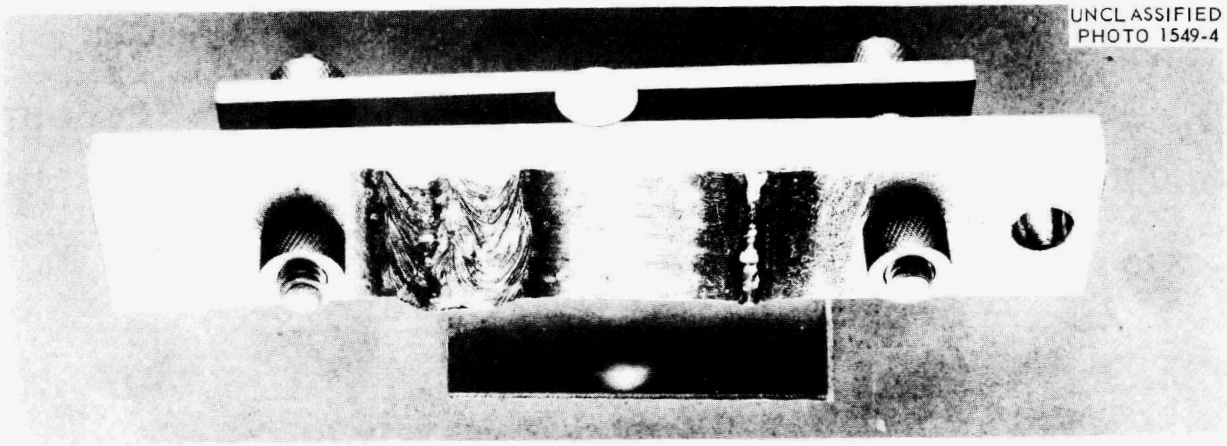
A total of 35 assemblies of the three stainless alloys was prepared for exposure in the pilot plant. The distribution of the assemblies in the solution and vapor phases of the process vessels is shown in Table 1.



(a) Top View of Assembled Stress Specimen



(b) Side View



(c) View Showing As-Welded Face of Base Plate

Fig. 1. Stainless Steel Stress Assembly Used for Corrosion Studies in Thorex Pilot Plant. 1.2X.

Table 1. Distribution of Stainless Steel Stress Assemblies in Thorex Pilot Plant

Process Vessel	Vessel Identity	Assembly Material	No. of Assemblies	
			Solution	Vapor
Batch dissolver tank	S-1	304L	1	1
		309SCb	1	1
Feed adjustment tank	S-2	304L	6	6
		309SCb	6	6
AP catch tank	N-2	304L	1	1
		309SCb	1	1
BT vapor separator	P-2	304L	0	1
		347	0	2

3.2 History of Test Materials. A summary of the size, source, and chemical composition of the stainless steels from which the specimens were fabricated appears in Table 2. All materials were within the chemical composition limits required by AISI specifications for types 304L, 309SCb, and 347 stainless steel. Information on the studs and nuts is not included in Table 2 since these materials were not considered in the quantitative evaluation of corrosion damage. Certified chemical analyses of the stud and nut materials, however, showed them to be within the required chemical composition limits for the respective alloys.

Table 2. Material History of Stainless Steels Used for Fabrication of Stress Assemblies

Material Type	Component	Form	Supplier	Heat No.	Element	Composition (wt %) Stress Specimen	Fulcrum	Base Plate
304L	Stress specimen	1/8-in. sheet	Armco Steel Company	-	C	0.02	0.02	0.02
					Mn	1.00	0.96	1.09
					Si	0.50	0.45	0.54
304L	Base plate	3/8-in. plate	Armco Steel Company	-	Ni	9.26	9.25	9.70
					Cr	18.59	18.80	18.40
					P	0.023	0.025	0.023
304L	Fulcrum	3/8-in.-dia. bar	Armco Steel Company	-	S	0.016	0.016	0.009
309SCb	Stress specimen	1/8-in. sheet	Crucible Steel Company	7999A3	C	0.06	0.06	0.05
					Mn	1.65	1.65	0.58
					Si	0.60	0.60	0.36
309SCb	Base plate	3/8-in. plate	Crucible Steel Company	7999A3	Ni	14.01	14.01	14.50
					Cr	22.92	22.92	23.50
					Cb	0.89	0.89	0.83
309SCb	Fulcrum	3/8-in.-dia. bar	Crucible Steel Company	-	P	0.011	0.011	-
					S	0.015	0.015	-
347	Stress specimen	1/8-in. sheet	Crucible Steel Company	623937	C	0.06	0.07	0.05
					Mn	1.89	1.58	1.90
					Si	0.64	0.61	0.64
347	Base plate	3/8-in. plate	Crucible Steel Company	K-6563	Ni	10.32	9.97	10.18
					Cr	18.02	18.59	18.20
					Cb + Ta	0.88	0.92	0.82
347	Fulcrum	3/8-in.-dia. bar	Crucible Steel Company	623569	P	0.025	0.013	0.021
					S	0.025	0.024	0.015

3.3 Boiling 65% HNO_3 Test Results. Prior to fabrication, specimens of the various alloy forms were subjected to the boiling 65% HNO_3 test in accordance with the recommended practice for corrosion-resistant steels, ASTM designation A262-55T.[14] Since the Thorex pilot plant was concerned to a large extent with the handling of nitric-acid-containing process solutions, the use of the boiling 65% HNO_3 test as a quality control test was considered pertinent.

The criterion used for acceptance or rejection of an austenitic stainless steel exposed to the nitric acid test is an arbitrarily chosen maximum penetration rate based upon observed weight losses on specimens during five (generally) 48-hr immersion periods. The maximum allowable rate, an average value for the five periods, may vary slightly for different stainless steels, but for types 304L, 309SCb, and 347 stainless steel in a sensitized condition, the rate is usually 2 mils/mo. (0.002 in./mo.).

Specimens were sensitized prior to test by heating 1 hr in air at 649°C (1200°F) followed by cooling in air to room temperature. All test materials except one exhibited average penetration rates of 2 mils/mo. or less after five 48-hr periods. The single exception was the 3/8-in.-thick type 309SCb stainless steel plate used for the welded base plates; the average penetration rate was 3 mils/mo. Inasmuch as the rate was not greatly in excess of the maximum allowable rate, plus the fact that microscopic examination failed to reveal any serious intergranular attack, it was decided to use the material for fabrication of the base plates.

3.4 Examination of Exposed Test Specimens. The stress assemblies as removed from the process vessels were scrubbed in soapy water to remove loosely adherent corrosion products and associated contamination. The radioactivity of most of the assemblies, measured 1 to 2 in. from the surface, was 20 to 80 mr/hr after scrubbing. Several of the assemblies measured about 600 mr/hr at the same distance. The highest surface-contact activity observed was 3000 mr/hr.

The general appearance of each assembly was noted, after which it was dismantled. As-scrubbed weights were obtained for the stress specimen, fulcrum, and base plate. These components were cathodically defilmed at 75°C in inhibited 5% H_2SO_4 solution.[15] The defilming process was repeated twice on each specimen. Attack by the defilming solution on unexposed control specimens of the three alloys was almost negligible, amounting to a weight loss of 0.03 mg/cm^2. The specimens presented no handling hazard due to radioactivity after defilming. The defilming process (one of hydrogen discharge from a metal surface) effectively reduced the radioactivity to 5 mr/hr or less, measured at contact.

3.5 Determination of Corrosion Rates. The defilmed specimens were degreased in acetone and alcohol, dried, and weighed to the nearest 0.1 ± 0.2 mg/cm^2. Corrosion rates were determined by the conventional weight-loss method.

The total residence time for solution-phase specimens in the pilot plant consisted of periods in which specimens were contacted by thermally hot process solutions, periods in which specimens were contacted by process solutions at room temperature, and periods in which specimens were exposed to air in empty vessels. Since laboratory tests at room temperature with similar stainless alloys in simulated Thorex process solutions showed that corrosion rates were less than 1 mpy,[16] the exposure time for specimens contacted by room-temperature process solutions was not considered as part of the accumulated active exposure time in the pilot plant vessels that during usage operated at elevated temperatures. Likewise, the time that specimens were contacted by air at room temperature was not considered. Thus the active exposure time for solution-phase specimens was realistically restricted to the period during which process solutions were above room temperature. With the exception of the AP catch tank which was operated around 40°C, the greater portion of the active exposure time in the process vessels was accumulated at temperatures from 110 to 155°C. The remaining portion of an active exposure period included the time required to heat the process solution to an elevated temperature, the time required to cool the solution to room temperature, and the time required for decontamination with hot chemical solutions. The active vapor-phase exposure periods were the same as for the solution-phase specimens since attack on vapor-exposed specimens by process solutions at room temperature was considered to be negligible.

4. CORROSION DURING PRODUCTION PERIOD

Stress assemblies were removed from the batch dissolver tank, the feed adjustment tank, the AP catch tank, and the vapor separator for the BT evaporator tank at the completion of the production period. Since exposure conditions varied considerably in the four process vessels, separate discussions will be presented for each vessel. Operational information for the pilot plant during this period has been reported.[17,18]

The corrosion information presented is for the most part representative of intermittent pilot plant operation. The only process vessel operated on a continuous basis during this period was the AP catch tank, used for collection and temporary storage of protactinium-containing solution from the uranium-thorium extraction column.

4.1 Batch Dissolver Tank

Function and Description. The batch dissolver tank was used for the dissolution of irradiated aluminum-jacketed thorium metal. The tank was 4 1/2 ft in diameter with a capacity of 550 gal. The construction material was 3/8-in.-thick type 309SCb stainless steel. Heliarc welding was employed in fabrication.

The nominal dissolving solution contained 13 *M* HNO_3 with 0.0375 *M* NaF, 0.0025 *M* $Hg(NO_3)_2$, and 0.04 *M* $Al(NO_3)_3$. Mercuric nitrate and sodium fluoride were present to catalyze the aluminum-nitric acid and the thorium-nitric acid dissolution reactions, respectively. The aluminum nitrate was added to complex the fluoride ion, thereby suppressing corrosion attack by the dissolving solution on the stainless steel vessel used in room-temperature preparation and storage. Typical initial and final solution compositions for the batch dissolver tank are given in Table 3.

Table 3. Typical Initial and Final Solution Compositions in Batch Dissolver Tank

Constituent	Concentration (*M*)	
	Initial	Final
HNO_3	13.0	5.0
$Th(NO_3)_4$	0	1.0
$Al(NO_3)_3$	0.04	0.37
Fluoride	0.04	0.015
$Hg(NO_3)_2$	0.003	0.001

The dissolver tank operated at the boiling temperature of the dissolving solution, approximately 110°C. The average time required for a batch dissolution was 10 to 12 hr. When sufficient thorium was dissolved, the solution was cooled to 40°C and jetted to the feed adjustment tank.

During operation, excess nitric acid was added to the dissolver tank to expedite the dissolution of highly stable "blue" thorium oxide invariably present in neutron-irradiated thorium metal.[19] The blue thorium oxide dissolution was imperative for two reasons: (1) it represented a potential processing loss of 0.6% in both thorium and associated uranium-233, and (2) because of its high density and fine particle size, it could plug filters and small process equipment lines in the pilot plant.

Decomposition gases and vapors from the nitric acid dissolution were passed to a water-cooled shell-and-tube condenser. The non-condensable gases were discharged to the atmosphere through a 200-ft stack. The pressure in the dissolver was maintained slightly under atmospheric.

Operational History. Fifteen runs were made in the dissolver tank; twelve runs were normal dissolutions with either nonirradiated or irradiated thorium metal; and three runs were with low thorium-containing nitric acid solution. Of the twelve normal runs, five were made with nonirradiated aluminum-jacketed thorium metal slugs using 100% metal heel and 100% excess 13 *M* HNO_3. Six of the remaining seven runs were made with aluminum-jacketed slugs; the seventh run was made on undissolved thorium metal remaining in the dissolver tank. It was necessary to make three additional recovery runs with 13 *M* HNO_3 solution to remove the persistent metal heel.

The concluding phase of operation was the decontamination of the vessel. Twenty decontamination passes were made with: (1) 30% HNO_3, (2) 15% NaOH containing 1% oxalic acid, (3) 20% NaOH containing 3% tartaric acid, and (4) 20% HNO_3-3% HF solution. A decontamination pass consisted of flushing all internal surfaces (and corrosion specimens) in the vessel with the chemical reagents by means of a steam-operated jet spray system. Contact time for each pass varied between 1 and 3 hr; solution temperature of the reagents ranged from 40 to 95°C. Detailed information on the decontamination operation has been reported.[3]

Corrosion Data. A breakdown of the exposure time for the types 304L and 309SCb stainless steel stress assemblies exposed in the batch dissolver tank appears in Table 4. As discussed previously, the reported exposure times in Table 4 and following tables are representative of the periods only during which process solutions were above room temperature. The maximum gross beta-gamma activity in process solutions handled by the batch dissolver tank was 1×10^{10} counts/min/ml.

The total active exposure time for both solution- and vapor-exposed assemblies was 634 hr which included the time with process solution at operating temperature, heating and cooling times, and the time required for decontamination. Corrosion data for the individual specimens are given in Table 5. As mentioned earlier, the fulcrum in each assembly was used as a control specimen for comparison of corrosion behavior with the weld and stress specimens.

Table 4. Summary of Exposure History for Stainless Steel Stress Assemblies in Batch Dissolver Tank During Production Period

	Exposure Time (hr)	
	Solution	Vapor
Total residence time in tank	2190	2190
Five cold runs at 110°C maximum	86	86
Seven hot runs at 110°C maximum	128	128
Three recovery runs at 110°C maximum	350	350
Holding time at room temperature	466	0
Decontamination at 40 to 95°C	70	70
Total active exposure time*	634	634

* Holding time at room temperature not considered in total active exposure time.

Table 5. Corrosion of Stainless Steel Stress Assemblies in Batch Dissolver Tank During Production Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack[a]
D-16	304L	Solution at 110°C max.	634	Control	10.9	0.5	7.5	Uniform
				Stress	12.0	0.6	8.3	Uniform; no cracks
				Weld[b]	12.1	0.6	8.3	Uniform
D-65	309SCb	Solution at 110°C max.	634	Control	5.0	0.2	3.5	Uniform
				Stress	3.7	0.2	2.6	Uniform; no cracks
				Weld[c]	4.6	0.2	3.1	Uniform
D-15	304L	Vapor at 110°C max.	634	Control	4.2	0.2	2.9	Uniform
				Stress	3.8	0.2	2.6	Uniform; no cracks
				Weld	7.9	0.4	5.3	Uniform
D-64	309SCb	Vapor at 110°C max.	634	Control	3.4	0.2	2.3	Uniform
				Stress	2.6	0.1	1.8	Uniform; no cracks
				Weld	3.0	0.1	2.0	Uniform

(a) Type of attack refers to flat surfaces of specimens only.

(b) Weld specimens of type 304L were Heliarc-welded with type 308L rod.

(c) Weld specimens of type 309SCb were Heliarc-welded with type 309SCb rod.

Discussion of Data. Before dismantling, the stress assemblies were carefully examined for indications of cracking; no cracks were found on any of the solution- or vapor-phase stress specimens. As shown in Table 5, there was no acceleration in the rate of corrosion attack on specimens as a result of the applied stress. Some localized attack was observed on edge surfaces of a number of the specimens; this type of attack will hereinafter be referred to as edge-corrosion (end-grain) attack.

(1) Corrosion of Solution-Exposed Specimens. The average corrosion rate of type 309SCb stainless steel during the 634 hr was 3.1 ± 0.5 mpy as compared with 8.0 ± 0.4 mpy for type 304L stainless steel. The average rate for each alloy was determined from the individual rates for the stress specimen, fulcrum, and weld specimen. Generally, there was not sufficient variation in the corrosion behavior of the three components to preclude the use of such an average value.

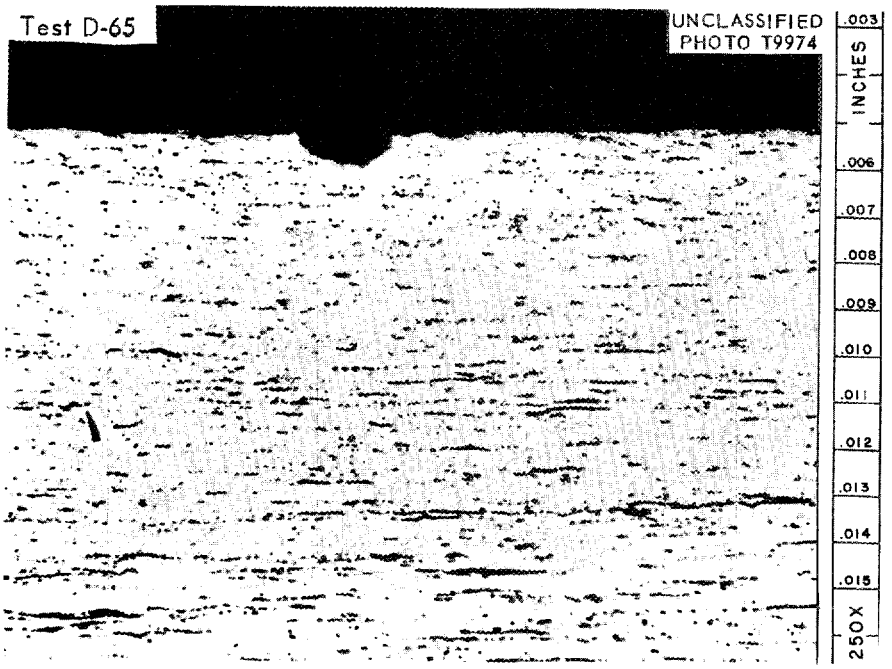
The nature of corrosion attack on flat and edge surfaces of a stress specimen of each alloy is shown in the photomicrographs of Figs. 2 and 3. Flat surfaces exhibited relatively uniform attack; some random shallow pitting (less than 1 mil in depth) was observed on both alloys. There were no evidences of intergranular corrosion. Both alloys were subject to edge corrosion as shown in Fig. 3. The pits on type 304L stainless steel were larger in diameter and of greater frequency than those observed on type 309SCb stainless steel. The maximum penetration by pitting for both alloys was 3 mils. The propagation of pits occurred in a plane parallel with the rolling direction of the stainless steel sheet as indicated in Fig. 3.

There was no unusual corrosion attack on weld specimens; examination of weld-parent metal interfaces showed complete freedom from preferential attack in the heat-affected zones. Visually, the type 308L weld metal exhibited more surface roughening than the parent metal, type 304L stainless steel. This behavior was not apparent on the type 309SCb weld metal.

No crevice corrosion was found on areas of metal-to-metal contact in assemblies of either alloy. Microscopic examination of these areas showed no difference in condition from that of the surrounding surface.

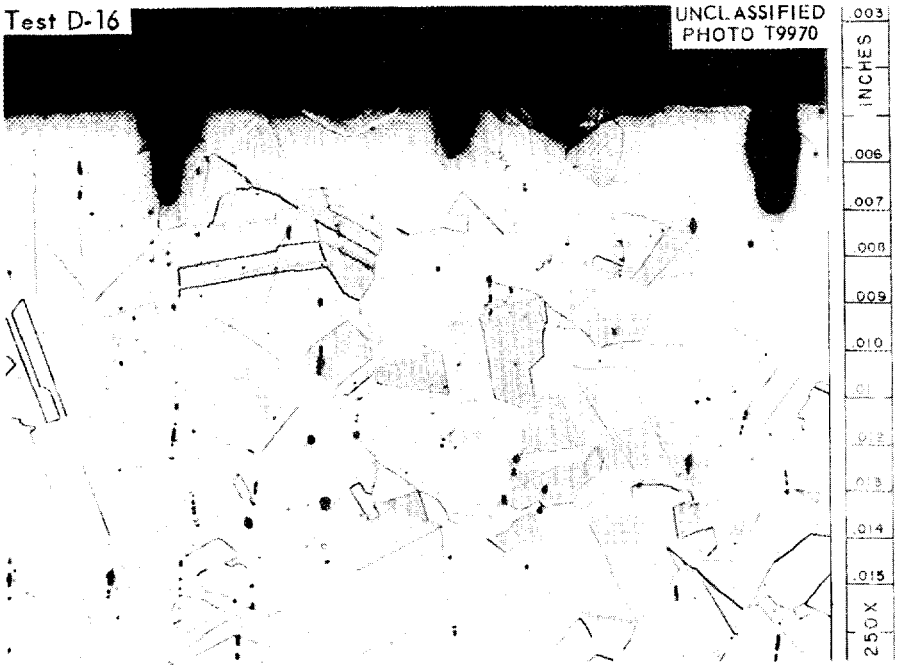
A considerable difference in grain size of stress specimens of the two alloys was evident as shown in Fig. 2. The grain size of the type 304L stainless steel was ASTM No. 4 (calculated mean grain diameter of 4 mils). Type 309SCb stainless steel had a much smaller grain size, ASTM No. 8 (calculated mean grain diameter of slightly under 1 mil).

Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 2. Surface Attack on Stress Specimens After 1100 hr Exposure (634 hr at Elevated Temperatures) to Process Solution in Batch Dissolver Tank During Production Period. Etched in aqua regia. 250X.



Type 304L Stainless Steel

Type 309SCB Stainless Steel

Fig. 3. Edge-Surface Attack on Stress Specimens After 1100 hr Exposure (634 hr at Elevated Temperatures) to Process Solution in Batch Dissolver Tank During Production Period. Etched in aqua regia. 250X.

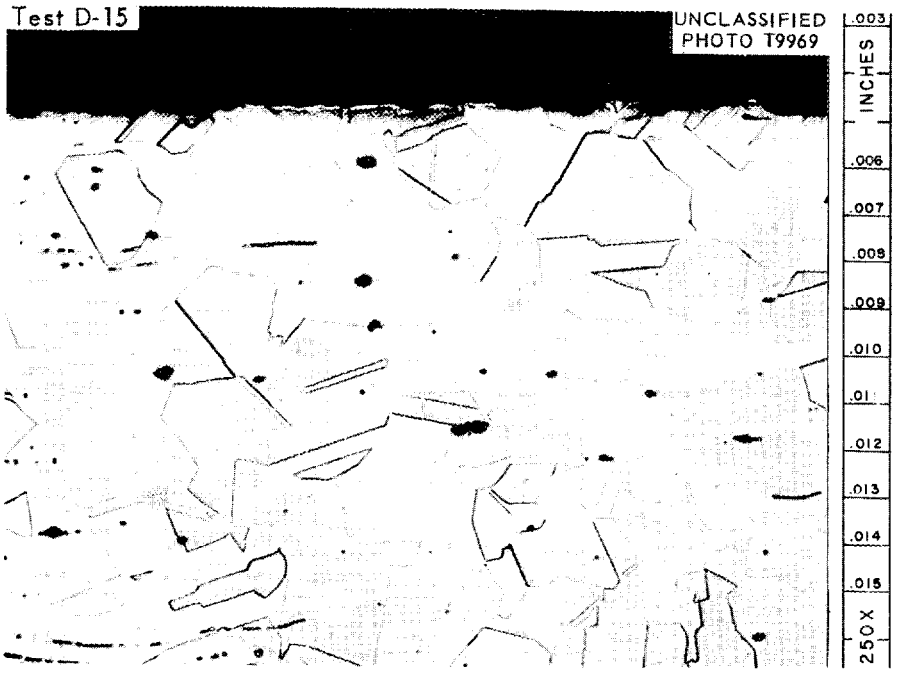
The general physical condition of assemblies upon removal from the dissolver tank was quite satisfactory. Surface staining was much in evidence on all specimens. No excessive amounts of corrosion films were present; observed film weights, represented by the difference between as-scrubbed and defilmed weights, were 0.1 mg/cm^2 or less.

(2) Corrosion of Vapor-Exposed Specimens. There was no significant difference in the corrosion behavior of the two stainless alloys exposed in the hot vapor (110°C max.) for 634 hr. Type 304L stainless steel corroded at an average rate of 3.6 ± 1.7 mpy; the average rate for type 309SCb stainless steel was 2.0 ± 0.3 mpy. The large deviation from the average rate for the type 304L was caused by a corrosion rate of 5.3 mpy for the weld specimen. The control and stress specimens in the assembly corroded at rates of 2.9 and 2.6 mpy, respectively. Surface attack on stress specimens of both alloys is shown in Fig. 4. Although some surface roughening did occur, the attack was considered as being uniform. Edge-surface attack on the two alloys was similar to that shown in Fig. 3 with pits extending up to 2 mils in depth.

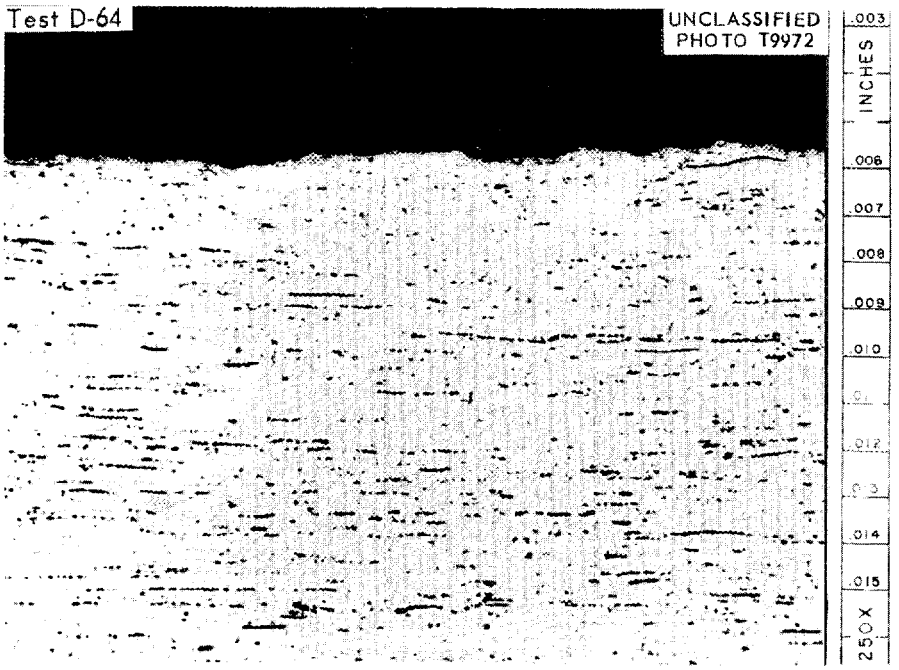
The first of a number of cases of corrosion attack peculiar to weld specimens exposed in the pilot plant was observed on a vapor-exposed specimen of type 304L stainless steel in the dissolver tank. The corrosion consisted of localized attack in the heat-affected zone between the weld metal (type 308L) and the parent metal. The attack was very similar to knife-line corrosion occasionally found in stabilized grades of stainless steel.[20] A photomicrograph showing the attack is included in Fig. 5. Actually, the depth of penetration was about 5 mils although it appears to be 10 mils in the photomicrograph. The attack extended the full width of the specimen along the root weld on the as-welded face. There was no evidence of intergranular corrosion in the attacked region as is commonly found in knife-line corrosion. The type 309SCb stainless steel weld specimen did not exhibit this type of selective attack.

There was no immediate explanation for the localized interfacial attack on the type 304L stainless steel. With a reported carbon content of 0.02% (Table 2), the alloy would be expected to be immune to carbide precipitation during welding. The fact that the attack was found only along the face weld in the same specimen would suggest the welding operation as a cause rather than some inherent characteristic of the parent metal. A mechanism has been proposed for knife-line attack in stabilized austenitic stainless steel by Holzworth and co-workers,[20] but this mechanism would not be operative in unstablized grades of stainless steel.

No crevice corrosion attack was found on specimens of either stainless alloy. The condition of surfaces contacted by other metal surfaces during exposure was the same as that of unconfined surfaces.



Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 4. Surface Attack on Stress Specimens After 634 hr of Vapor Exposure in Batch Dissolver Tank During Production Period. Etched in aqua regia. 250X.

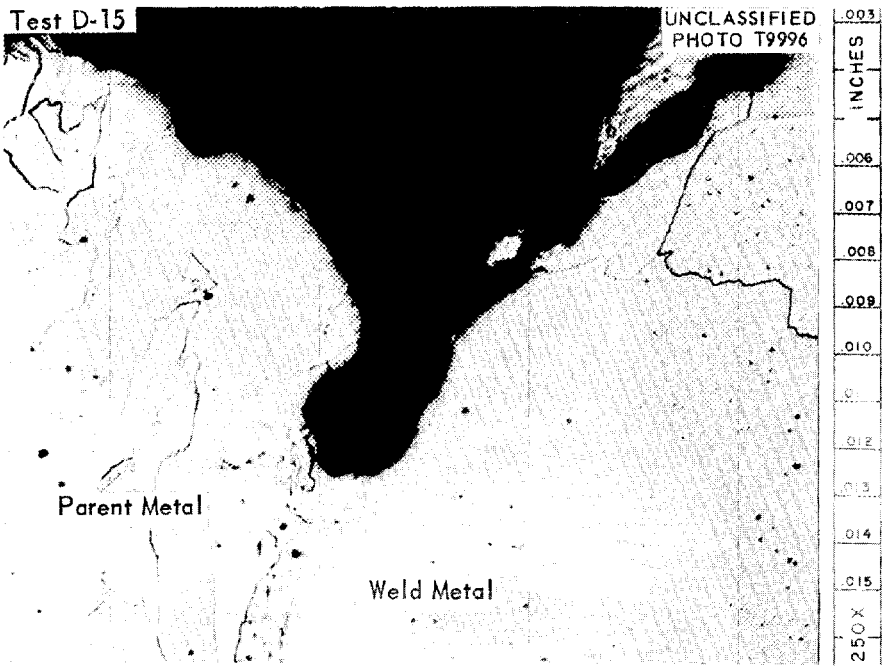


Fig. 5. Preferential Corrosion Attack at Weld-Parent Metal Interface on Type 304L Stainless Steel Weld Specimen After 634 hr of Vapor Exposure in Batch Dissolver Tank During Production Period. Etched in aqua regia. 250X.

The as-removed condition of the assemblies was little changed from the original appearance except for random dark-brown stains. Corrosion films were thin; observed film weights were less than 0.1 mg/cm^2.

(3) Solution-Vapor Interface Corrosion. Although it had been planned to use the stress assembly stainless steel support rods for qualitative evaluation of solution-vapor interface corrosion, it was found upon examination that the solution level in the dissolver tank fluctuated so extensively that it was impossible to detect any solution-vapor interface. The surfaces of the rods were etched in appearance, and the attack appeared uniform, however.

4.2 Feed Adjustment Tank

Function and Description. The feed adjustment tank and auxiliary equipment were used to provide a nitric acid-deficient feed solution for the uranium-thorium solvent extraction process. The welded, 1/2-in.-thick type 309SCb stainless steel tank was 4 ft in diameter, with a total capacity of 450 gal.

Excess nitric acid in process solution received from the dissolver tank was first removed in the feed adjustment tank by an evaporation process at 110°C. In addition, the evaporation process also provided solution and temperature conditions which promoted the formation of stable silicas that were not objectionable in the subsequent extraction cycle.[21] The silicas, which originate in the aluminum and thorium metals, also serve as carriers of inordinate amounts and levels of radiation involving similar physical hydrous species as protactinium and fission product activities. The nitric acid-deficient aluminum and thorium nitrate solution was then digested at 150 to 155°C to destroy the tributyl phosphate extractable fractions of the ruthenium fission product. After digestion, the solution was diluted with water to the composition specified for feed solution to the thorium-uranium extraction column. Typical initial and final solution compositions for the feed adjustment tank are shown in Table 6.

Table 6. Typical Initial and Final Solution Compositions in Feed Adjustment Tank

Constituent	Concentration (*M*)	
	Initial	Final*
HNO_3	5.0	0.5**
$Th(NO_3)_4$	1.0	4.0
$Al(NO_3)_3$	0.37	1.6
$Hg(NO_3)_2$	0.001	0.004
Fluoride	0.015	0.047

* Solution composition before dilution with water to meet feed specifications for extraction column.

** Nitric acid deficient.

Operational History. Fifteen runs with both irradiated and nonirradiated thorium metal were made. The operation of the tank was much the same for both types of process solution. During evaporation cycles, the initial nitric acid content of the condensed vapors leaving the tank was 1 *M*. The concentration reached an equilibrium value of 8 *M* HNO_3 as the evaporation and digestion processes continued.

The fifteen runs consisted of five runs with nonirradiated metal, seven runs with irradiated metal, and three recovery runs. Seven evaporations were made during the three recovery runs with solution that contained relatively little thorium. The temperature during the recovery runs was 133°C.

Decontamination at completion of the feed adjustment tank operation was similar to that described for the batch dissolver tank. A total of ten alternate passes with dilute nitric acid and alkaline oxalate or tartrate solutions was made at solution temperatures from 40 to 95°C.

Corrosion Data. A summary of the exposure history for the stress assemblies during feed adjustment tank operation appears in Table 7. The reported exposure hours, in addition to time at elevated temperatures (110 to 155°C), include heating and cooling times for the process solutions and also 20 hr for decontamination with solutions at 40 to 95°C.

Table 7. Summary of Exposure History for Stainless Steel Stress Assemblies in Feed Adjustment Tank During Production Period

	Exposure Time (hr)	
	Solution	Vapor
Total residence time in tank	1880	1880
Five cold runs at 155°C max.	85	85
Seven hot runs at 155°C max.	100	100
Three recovery runs at 133°C max.	350	350
Holding time at room temperature	625	0
Decontamination at 40 to 95°C	20	20
Total active exposure time*	555	555

* Holding time at room temperature not considered in total active exposure time.

Eight stress assemblies were removed from the feed adjustment tank. Duplicate sets of types 304L and 309SCb stainless steel were exposed to the solution and vapor phases. Other assemblies installed originally with the eight assemblies were left in the tank for additional exposure time during the development period of operation which followed. A summary of the corrosion data for the assemblies removed from the feed adjustment tank is given in Table 8.

Table 8. Corrosion of Stainless Steel Stress Assemblies in Feed Adjustment Tank during Production Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack[a]
D-19	304L	Solution at 155°C max.	555	Control	5.3	0.3	4.0	Uniform
				Stress	5.2	0.3	4.0	Uniform; no cracks
				Weld[b]	6.1	0.3	4.7	Uniform
D-20	304L	Solution at 155°C max.	555	Control	5.0	0.2	4.0	Uniform
				Stress	5.3	0.3	4.0	Uniform; no cracks
				Weld	6.0	0.3	4.7	Uniform
D-68	309SCb	Solution at 155°C max.	555	Control	4.5	0.2	3.6	Uniform
				Stress	10.9	0.5	8.5	Uniform; no cracks
				Weld[c]	8.5	0.4	6.6	Uniform
D-69	309SCb	Solution at 155°C max.	555	Control	4.7	0.2	3.6	Uniform
				Stress	9.2	0.4	7.2	Uniform; no cracks
				Weld	9.2	0.4	7.2	Uniform
D-17	304L	Vapor at 155°C max.	555	Control	24.4	1.2	19.1	Mild acid-etch
				Stress	25.9	1.3	20.2	Mild acid-etch; no cracks
				Weld	26.8	1.3	21.0	Mild acid-etch
D-18	304L	Vapor at 155°C	555	Control	29.6	1.4	23.2	Mild acid-etch
				Stress	28.5	1.4	22.3	Mild acid-etch; no cracks
				Weld	26.3	1.3	20.6	Mild acid-etch
D-66	309SCb	Vapor at 155°C	555	Control	34.0	1.7	26.6	Mild acid-etch
				Stress	52.3	2.6	40.9	Severe acid-etch; no cracks
				Weld	46.3	2.3	36.2	Severe acid-etch
D-67	309SCb	Vapor at 155°C	555	Control	34.5	1.7	27.0	Mild acid-etch
				Stress	54.7	2.7	42.7	Severe acid-etch; no cracks
				Weld	47.1	2.3	36.9	Severe acid-etch

(a) Type of attack refers to flat surfaces of specimens only.
(b) Weld specimens of type 304L were Heliarc-welded with type 308L rod.
(c) Weld specimens of type 309SCb were Heliarc-welded with type 309SCb rod.

Discussion of Data. None of the stress specimens in the eight assemblies exhibited any signs of cracks prior to dismantling. After defilming, the tension surfaces were re-examined microscopically; no cracks were found. The as-removed appearance of solution- and vapor-exposed assemblies was characterized by mottled gray- and brown-colored stains and slight surface roughening. Film weights varied between 0.1 and 0.2 mg/cm^2.

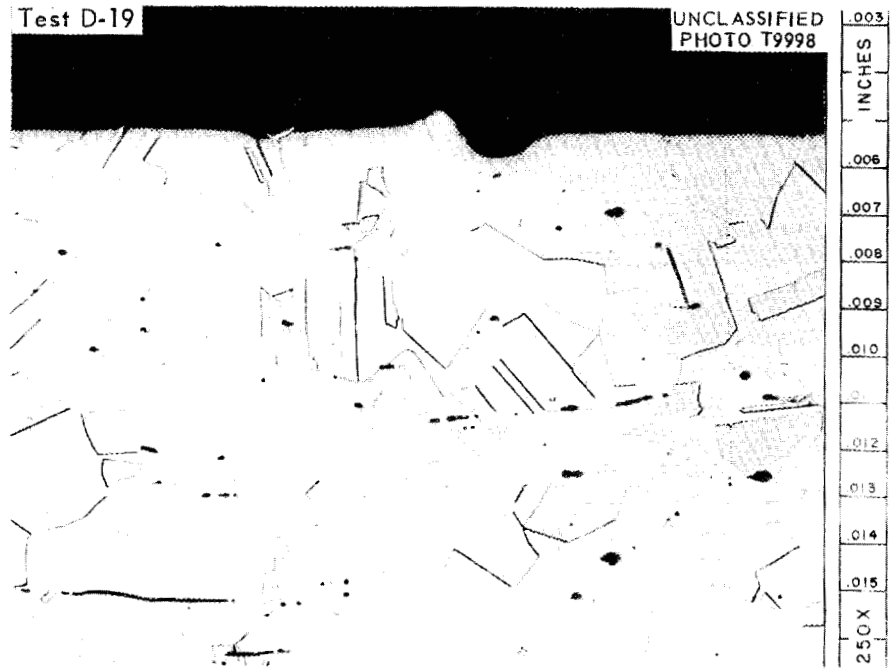
(1) Corrosion of Solution-Exposed Specimens. Observed over-all corrosion rates for both types 304L and 309SCb stainless steel control, stress, and weld specimens were not in excess of 8.5 mpy during the active exposure period of 555 hr at temperatures up to 155°C. The average rate for type 304L stainless steel was 4.2 $\pm$ 0.4 mpy as compared with an average rate of 6.0 $\pm$ 2.5 mpy for type 309SCb stainless steel. The maximum corrosion rate of 8.5 mpy was on a stress specimen of type 309SCb stainless steel (test D-68 in Table 8).

Transverse sections of the flat surface of stress specimens of the two alloys are shown in Fig. 6. Some surface roughening was evident in both cases, but neither pitting nor intergranular attack was observed. The attack on edge surfaces was intergranular and highly localized in numerous areas. Pitting attack ranged in depth from 2 to 5 mils in both alloys.

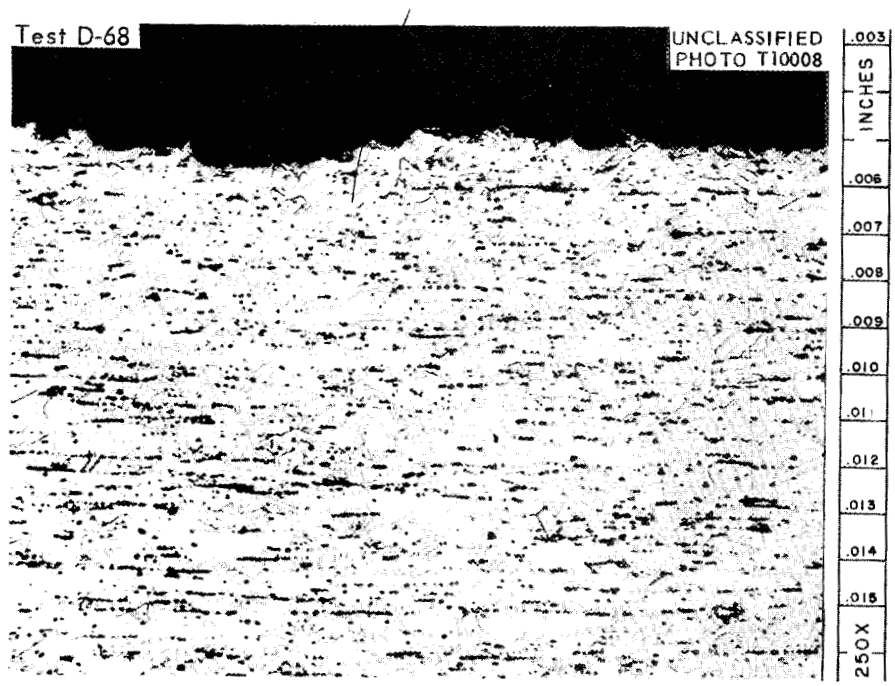
Knife-line attack was observed in the heat-affected zones along the root and face welds on the as-welded surface of one of the type 309SCb stainless steel specimens as shown in Fig. 7. The depth of the attack was 5 mils. The companion weld specimen was not subject to knife-line attack. No weld-parent metal interface attack was noted on the type 304L weld specimens. Visually, the weld metal deposits in both alloys appeared as resistant to the environment as the respective parent metals.

No signs of crevice corrosion were observed on any of the components in the four stress assemblies.

(2) Corrosion of Vapor-Exposed Specimen. Corrosion attack on vapor-exposed specimens was appreciably more severe than that on solution-exposed specimens. During the 555-hr exposure, most of which was accumulated at temperatures between 110 and 155°C, type 304L stainless steel corroded at an average rate of 21.1 $\pm$ 2.0 mpy, about five times the rate obtained for the same alloy during a comparable exposure time in the solution phase. Type 309SCb stainless steel corroded at an average rate of 35.0 $\pm$ 8.5 mpy in the vapor. This rate was about six times greater than the rate obtained for the alloy in the solution phase.



Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 6. Surface Attack on Stress Specimens After 1180 hr Exposure (555 hr at Elevated Temperatures) to Process Solution in Feed Adjustment Tank During Production Period. Etched in aqua regia. 250X.

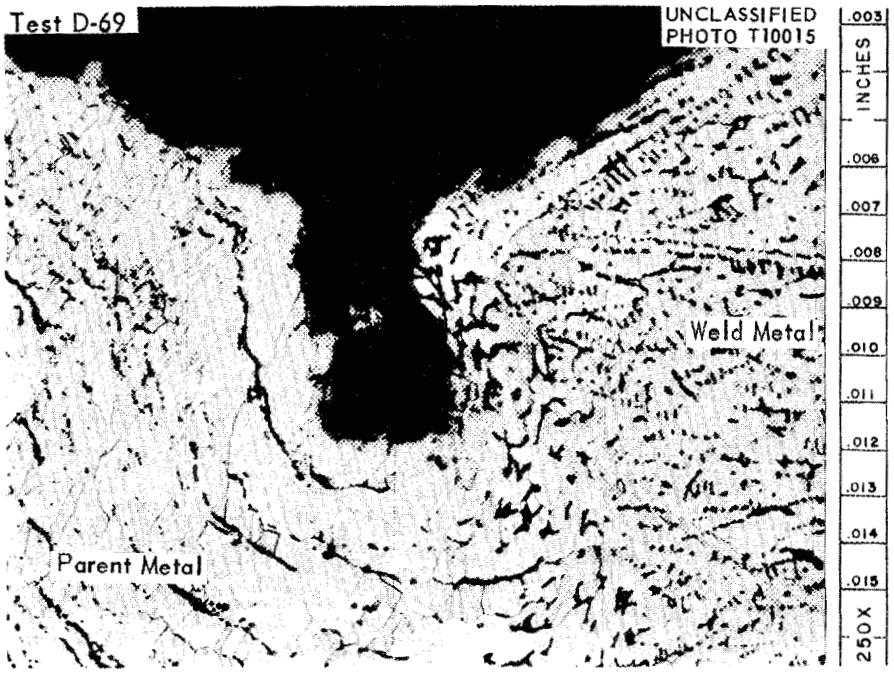


Fig. 7. Knife-Line Corrosion Attack on Type 309SCB Stainless Steel Weld Specimen After 1180 hr of Solution Exposure in Feed Adjustment Tank During Production Period. Etched in aqua regia. 250X.

Corrosion on flat surfaces of stress specimens of the two alloys was characterized by a mild, preferential grain-boundary attack; photomicrographs of transverse sections of the specimens appear in Fig. 8. The penetration on type 304L was 1 mil or less, whereas it was less than 0.5 mil on the type 309SCb stainless steel. Both alloys exhibited substantial surface roughening.

The severity of edge-corrosion attack on the alloys during the 555-hr exposure is illustrated in Fig. 9. Basically, the attack was intergranular, culminating in intense pitting. Penetration by pitting was 12 mils or less in type 304L and 6 mils or less in type 309SCb stainless steel.

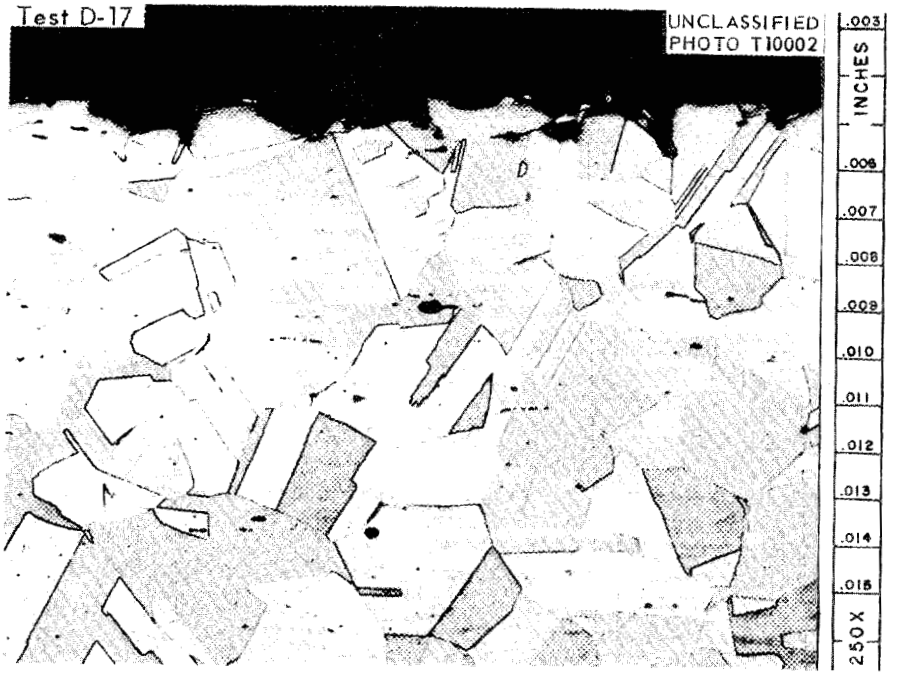
Knife-line corrosion attack was found in the heat-affected zones adjacent to welds on the as-welded face of one of the type 309SCb stainless steel weld specimens but not on the companion specimen. The type 304L weld specimens were free from preferential attack in heat-affected zones. The weld metal deposits - type 308L in 304L stainless steel and type 309SCb in 309SCb stainless steel - exhibited a "grooving" type of attack. Based upon visual examination, it was concluded, however, that the over-all attack on the weld deposits was no more severe than on the respective parent metals.

No crevice corrosion was found on any of the specimens.

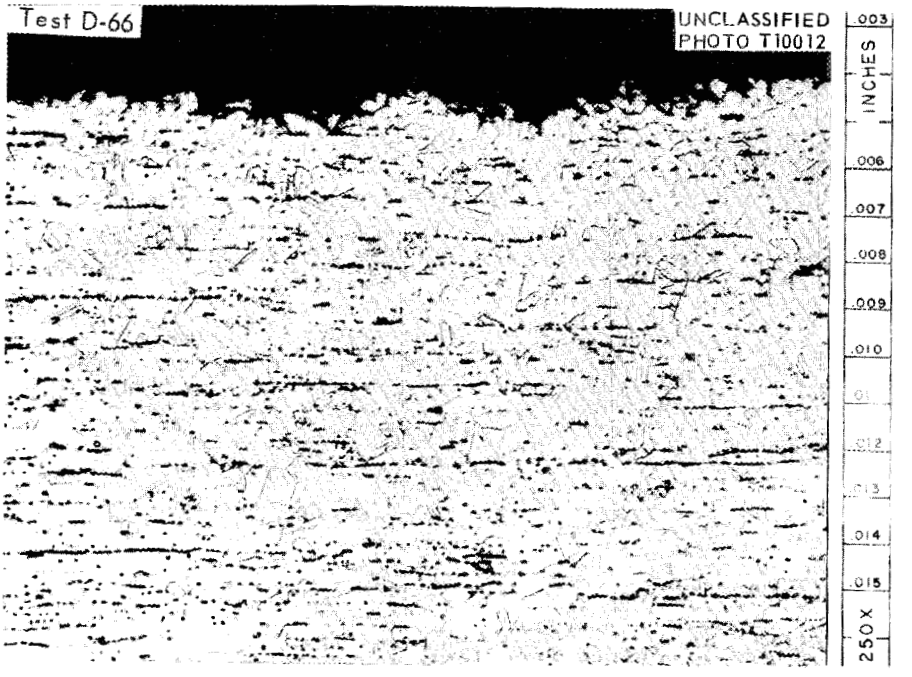
(3) Solution-Vapor Interface Corrosion. As was experienced in the batch dissolver, the solution level in the feed adjustment tank underwent such fluctuation that there was no indication of a sharp solution-vapor interface on the types 304L and 309SCb stainless steel rods used to support the stress assemblies. It was apparent, however, that the upper portions of the rods were corroded more than the lower (solution-exposed) portions. The surfaces of the former were much rougher and darker in appearance. Several 1/8-in.-dia. holes drilled in the vapor-exposed end of a type 304L rod were enlarged to approximately 1/4 in. due to corrosion attack. The interior surfaces of the holes were badly pitted with pit growth occurring along a plane parallel with the rolling direction of the bar.

4.3 AP Catch Tank

Function and Description. The AP catch tank was used for collection and temporary storage of the aqueous protactinium-containing process solution from the uranium-thorium extraction column. The nominal composition of the solution was 0.6 *M* $Al(NO_3)_3$ and 0.03 *M* HNO_3 (acid-deficient) with traces of protactinium and fission products. Mercuric and fluoride ions were usually present also.



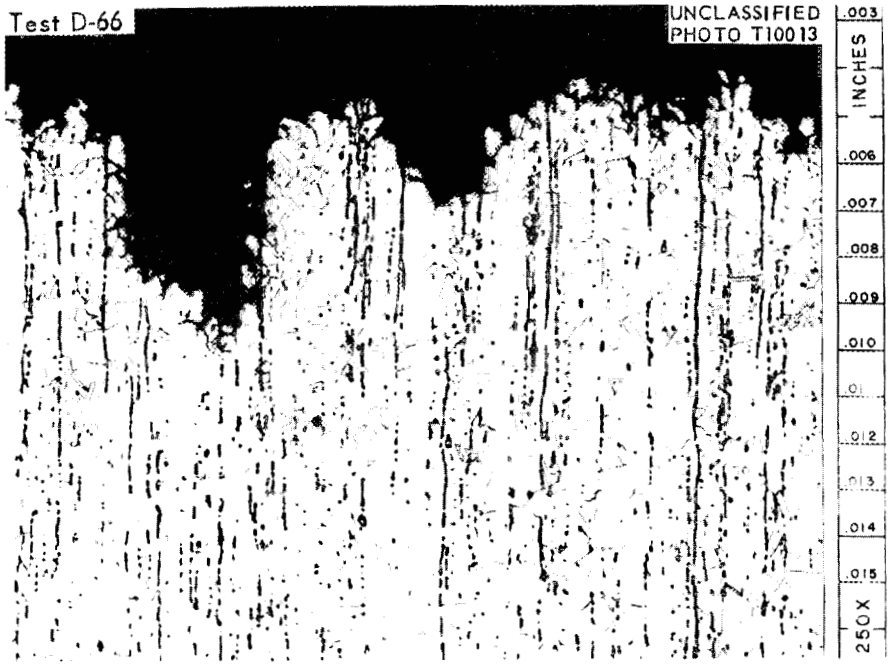
Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 8. Surface Attack on Stress Specimens After 555 hr of Vapor Exposure in Feed Adjustment Tank During Production Period. Etched in aqua regia. 250X.

Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 9. Edge-Surface Attack on Stress Specimens After 555 hr of Vapor Exposure in Feed Adjustment Tank During Production Period. Etched in aqua regia. 250X.

Table 9 shows the approximate chemical composition of the solution in the AP catch tank. The presence of phosphate and ferrous ions was due to the following: phosphate was added to the scrub solution entering the uranium extraction column to aid in the decontamination from protactinium, and iron, as ferrous sulfate, was added to the same scrub solution to prevent extraction of an oxidized chromium compound from stainless steel equipment corrosion during the head-end treatment in the feed adjustment tank.

Table 9. Approximate Chemical Composition of Process Solution in AP Catch Tank

Constituent	Concentration (M)
$Al(NO_3)_3$	0.6
HNO_3	0.3*
$Hg(NO_3)_2$	0.01
Fluoride	0.02
$Fe_2(SO_4)_3$	0.001
H_3PO_4	0.002
Fission products	$\sim 10^{-3}$ mg/ml
pH	2.5 to 3.0

* Nitric acid deficient.

The solution from the extraction column flowed continuously into the catch tank. After the tank was filled, if chemical analysis showed insignificant amounts of thorium-232 and uranium-233 to be present, the solution was discharged to the X-10 area waste-disposal system. Solution temperature in the tank remained fairly constant at 40°C. The tank had a capacity of 290 gal and was of welded type 309SCb stainless steel construction.

Operational History. As mentioned, the AP catch tank was used continuously during the production period. The maximum gross beta-gamma activity in the process solution handled by the tank was 1×10^{10} counts/min/ml.

Decontamination of the tank consisted of treatment with eight alternate passes of alkaline tartrate and nitric acid solutions at temperatures between 20 and 80°C.

Corrosion Data. Single stress assemblies of both types 304L and 309SCb stainless steel were removed from the solution and vapor phases of the catch tank after a 910-hr exposure which included 10 hr for decontamination. A summary of the corrosion data is given in Table 10.

Discussion of Data. No cracks were observed in stress specimens of either alloy exposed to the solution and vapor phases in the tank. The final appearance of specimens was unchanged from the original appearance. All surfaces were metallic and lustrous; there was no visible film formation.

(1) Corrosion of Solution-Exposed Specimens. The average corrosion rates for both stainless alloys, types 304L and 309SCb, during the 910-hr solution exposure were slightly in excess of 0.1 mpy. The attack was of a very light, uniform nature on both flat and edge surfaces of the specimens. There were no indications of preferential attack at welded areas nor were there any signs of crevice corrosion.

(2) Corrosion of Vapor-Exposed Specimens. As was the case for solution-exposed specimens, the attack on the vapor-phase specimens was extremely mild during the 910-hr exposure. Observed average corrosion rates for both alloys were slightly in excess of 0.1 mpy. There were no evidences of localized corrosion attack.

4.4 BT Vapor Separator

Function and Description. The vapor separator tank, a component in the BT evaporator system, was used to concentrate thorium product from the uranium-thorium partition column. The aqueous thorium-containing stream from the column was passed to the BT stripper where the last traces of organic solvent were removed. The stream then passed to the BT evaporator system consisting of a recirculation loop and a heat exchanger. The vapor separator was located above the solution reboiler. The vapor passing through the separator was condensed and discharged as hot waste. The condensate contained approximately 0.05 *M* HNO_3.

The separator consisted of a welded 2-ft-dia tank with a height of 2 ft. The body of the tank was type 347 stainless steel; the dished-type top and bottom heads were type 304L stainless steel.

Table 10. Corrosion of Stainless Steel Stress Assemblies in AP Catch Tank during Production Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack
D-23	304L	Solution at 40°C max.	910	Control	0.1	0.01	0.05	Light uniform
				Stress	0.1	0.01	0.05	Light uniform; no cracks
				Weld[a]	0.2	0.01	0.10	Light uniform
D-71	309SCb	Solution at 40°C max.	910	Control	0.3	0.02	0.15	Light uniform
				Stress	0.1	0.01	0.05	Light uniform; no cracks
				Weld[b]	0.2	0.01	0.10	Light uniform
D-22	304L	Vapor at 40°C max.	910	Stress	0.3	0.02	0.15	Light uniform
				Control	0.1	0.01	0.05	Light uniform; no cracks
				Weld	0.3	0.02	0.15	Light uniform
D-70	309SCb	Vapor at 40°C max.	910	Stress	0.3	0.02	0.15	Light uniform
				Control	0.1	0.01	0.05	Light uniform; no cracks
				Weld	0.3	0.02	0.15	Light uniform

(a) Weld specimens of type 304L were Heliarc-welded with type 308L rod.

(b) Weld specimens of type 309SCb were Heliarc-welded with type 309SCb rod.

Operational History. The vapor separator was in use for 910 hr, mostly at a temperature between 110 and 115°C. The accumulated time consisted of (1) 410 hr with nonirradiated solution, (2) 345 hr with irradiated solution, (3) 145 hr with recovery-run solution, and (4) 10 hr with decontamination solutions. The temperature of the latter varied from 35 to 90°C. The first 330 hr of operation were made with no fluoride or mercury present in the process solution; the remaining 570 hr were made with solution containing approximately 0.004 *M* fluoride and small amounts of mercury. When fluoride was present, the condensate from the separator usually contained about 3 ppm of fluoride. The nominal initial and final process solution compositions for the BT evaporator system appear in Table 11.

Table 11. Nominal Initial and Final Compositions of Process Solution in BT Evaporator System

Constituent	Concentration (*M*)	
	Initial	Final
$Th(NO_3)_4$	0.025	2.0
$UO_2(NO_3)_2$	Trace	0.001
HNO_3	0.2	1.5
Fluoride*	0.004	0.009

* Fluoride was present for 570 hr of the 900-hr operating period.

The maximum gross beta-gamma activity of the process solution was 1×10^4 counts/min/ml. Decontamination of the separator was accomplished by the use of alternate passes with nitric acid and with alkaline tartrate solutions.

Corrosion Data. Since the vapor separator was fabricated from both types 304L and 347 stainless steel, stress assemblies of the two alloys were exposed in the vapor region. The corrosion data are summarized in Table 12 for a single type 304L assembly and for duplicate type 347 assemblies.

Table 12. Corrosion of Stainless Steel Stress Assemblies in BT Vapor Separator during Production Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack[a]
D-24	304L	Vapor at 115°C max.	910	Control	29.2	1.5	14.0	Moderate acid-etch
				Stress	14.2	0.7	6.8	Moderate acid-etch; no cracks
				Weld[b]	22.3	1.1	10.7	Moderate acid-etch
D-41	347	Vapor at 115°C max.	910	Control	40.5	2.0	19.4	Moderate acid-etch
				Stress	87.8	4.4	42.0	Severe acid-etch; no cracks
				Weld[c]	85.3	4.3	42.8	Severe acid-etch
D-50	347	Vapor at 115°C	910	Control	39.0	1.9	18.7	Moderate acid-etch
				Stress	90.7	4.5	45.3	Severe acid-etch; no cracks
				Weld[d]	73.5	3.6	35.1	Severe acid-etch

(a) Type of attack refers to flat surfaces of specimens only.

(b) Weld specimen of type 304L was Heliarc-welded with type 308L rod.

(c) Specimen Heliarc-welded with type 347 rod.

(d) Specimen Heliarc-welded with type 308L rod.

Discussion of Data. The assemblies were examined carefully before dismantling for signs of cracking; no cracks were found. The surfaces were dark brown as removed and were very grainy. Attack on type 347 stainless steel appeared much more severe than on type 304L stainless steel.

The average defilmed corrosion rate for type 304L was found to be 10.5 ± 3.7 mpy for the 910-hr exposure. The average rate for type 347 stainless steel was 33.2 ± 13.8 mpy. Poor agreement in rates among the individual components in the duplicate assemblies accounted for the large deviation from the average corrosion rate for type 347 stainless steel.

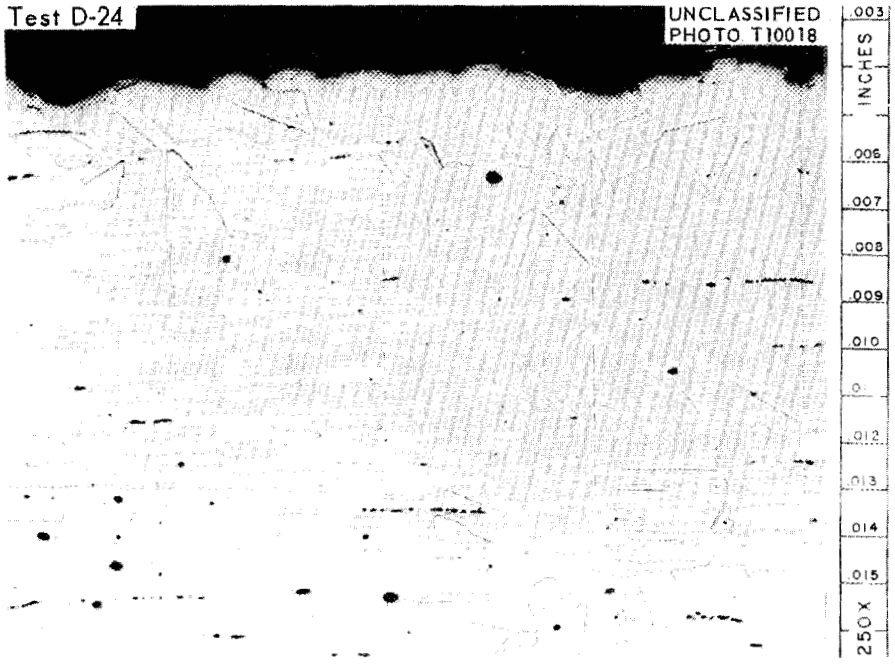
Metallographic examination of the flat surfaces of the stress specimens showed the attack as an acid-etch type; photomicrographs of transverse sections appear in Fig. 10. The attack was more pronounced on type 347 stainless steel. In both alloys, however, the preferential attack at the grain boundaries was less than 0.5 mil.

Edge corrosion, typified by intensive pitting, was observed on both alloys. Type 347 stainless steel was more prone to this type of attack than type 304L stainless steel, as shown by the low-magnification photographs in Fig. 11. The maximum pit depth on the former was 15 mils as compared with about 7 mils on the type 304L. The metal surrounding the pitted areas in the type 347 stainless steel exhibited some mild, intergranular corrosion. This was not the case in type 304L, however.

Knife-line corrosion attack occurred in the heat-affected zones adjacent to welds in both type 347 stainless steel specimens. A typical example appears in Fig. 12. The depth of the attack was approximately 10 mils. No weld-parent metal interface attack was found on the type 304L stainless steel weld specimen.

The two types of weld deposits (308L and 347) on type 347 stainless steel were attacked severely, resulting in extreme surface roughness and deep grooves. The type 308L deposits in the 304L stainless steel specimen, however, were attacked uniformly and appeared equally as corrosion resistant as the parent metal.

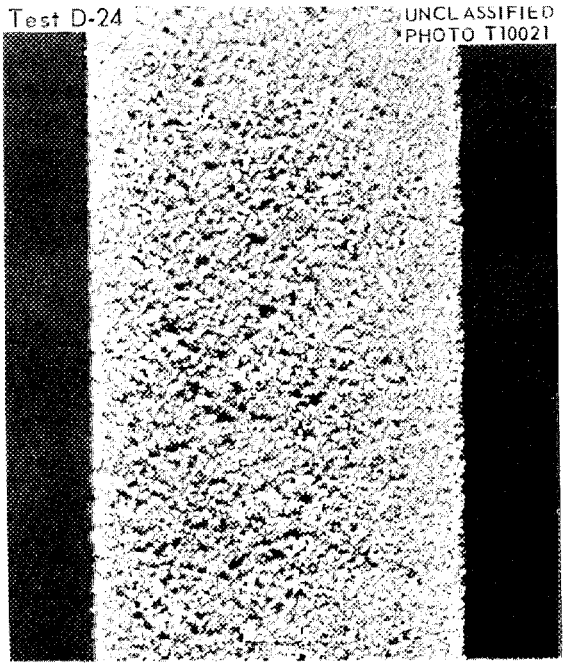
Localized corrosion attack in crevice areas was observed on the type 347 stainless steel weld specimens. The attack, consisting primarily of numerous 2- to 3-mil deep pits, occurred beneath the fulcrum on which the stress specimen was mounted. Other areas of metal-to-metal contact on the specimens were not subject to crevice corrosion.



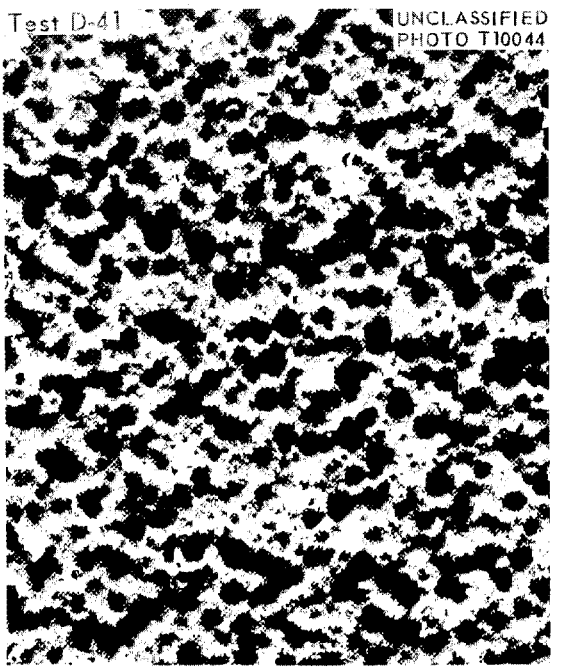
Type 304L Stainless Steel

Type 347 Stainless Steel

Fig. 10. Surface Attack on Stress Specimens After 910 hr of Vapor Exposure in BT Vapor Separator During Production Period. Etched in aqua regia. 250X.



Type 304L Stainless Steel
(Stress Specimen)



Type 347 Stainless Steel
(Base Plate)

Fig. 11. Edge-Surface Attack on Specimen After 910 hr of Vapor Exposure in BT Vapor Separator During Production Period. 20X.

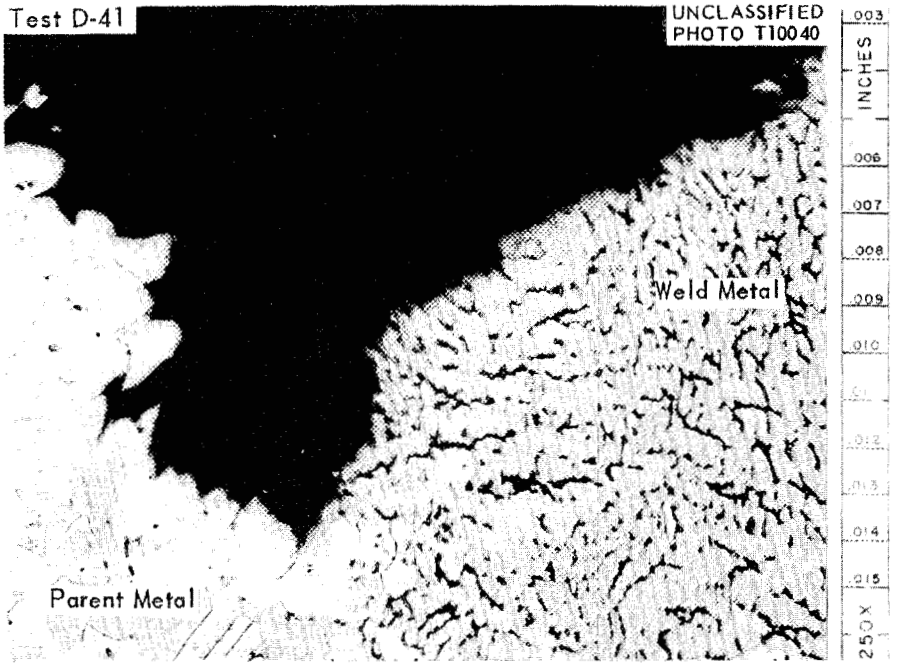


Fig. 12. Knife-Line Corrosion Attack on Welded Type 347 Stainless Steel Specimen (Type 347 Weld Metal) After 910 hr of Exposure in BT Vapor Separator During Production Period. Etched in aqua regia. 250X.

5. CORROSION DURING DEVELOPMENT PERIOD

Following the production period, the Thorex pilot plant entered the development period of operation. Stress assemblies were placed in the feed adjustment tank at the start of the development period and were removed midway through the operation. The exposure period occurred between March 4 and November 10, 1955. Based upon the corrosion data obtained during the production period, the feed adjustment tank was considered to be one of the more serious corrosion environments associated with the pilot plant. Corrosion specimens in other vessels in the pilot plant were not examined during the first half of the development period.

5.1 Feed Adjustment Tank

Function and Description. The function and description of the feed adjustment tank are discussed in Section 4.2.

Operational History. Twenty-two runs were made with solution from irradiated metal dissolution under operating conditions similar to those described in Section 4.2. A number of recovery runs with 0.8 *M* $Th(NO_3)_4$ solution that previously had been processed in the pilot plant followed these runs. Nitric acid and aluminum nitrate were present in the recovery-run solution also.

After the recovery runs, a number of concentration runs starting with 0.4 *M* $Th(NO_3)_4$ and 0.3 *M* HNO_3 solution were made. The solution was boiled until the desired thorium concentration (~4 *M*) was obtained. Fluoride and mercury were not present in this solution. However, these ions were present in the dissolver and recovery-run solutions. The nominal initial and final chemical compositions of process solution in the feed adjustment tank were similar to those in Table 6 for the production period.

The tank was decontaminated with alternate passes of 30% HNO_3 and 20% NaOH-2% sodium tartrate solutions at the completion of operation. The maximum solution temperature was 85°C.

Corrosion Data. The total contact time by process solutions on the stainless steel stress assemblies was 4701 hr. Of this total, 2142 hr (including 54 hr for decontamination) were accumulated at temperatures not in excess of 155°C. The 2142-hr period was taken as the active exposure time for reasons discussed previously. Similarly, the active exposure period for vapor-exposed assemblies was 2142 hr. The holding time at room temperature which was not considered in determination of corrosion rates was 2559 hr. A summary of the exposure history is given in Table 13.

Table 13. Summary of Exposure History for Stainless Steel Stress Assemblies in Feed Adjustment Tank During First Half of Development Period

	Exposure Time (hr)	
	Solution	Vapor
Total residence time in tank	5976	5976
Twenty-two hot runs at 155°C max.	1230	1230
Recovery runs at 133°C max.	858	858
Holding time at room temp.*	2559	0
Decontamination at 65 to 85°C	54	54
Total active exposure time**	2142	2142

* Includes 2195 hr with dissolver and recovery solutions and 364 hr with decontamination solutions.

** Holding time at room temperature not considered in total active exposure time.

Corrosion data for the duplicate assemblies of types 304L and 304SCb stainless steel exposed in the solution and vapor phases of the feed adjustment tank are shown in Table 14. The maximum gross beta-gamma activity of process solution handled in the tank during the development period was 4 to 5 x 10^{10} counts/min/ml.

Table 14. Corrosion of Stainless Steel Stress Assemblies in Feed Adjustment Tank during First Half of Development Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack[a]
D-30	304L	Solution at 155°C max.	2142[b]	Control	112.0	5.5	22.6	Severe acid-etch
				Stress	88.2	4.4	17.8	Severe acid-etch; no cracks
				Weld[c]	97.7	4.8	19.7	Severe acid-etch
D-31	304L	Solution at 155°C max.	2142	Control	105.6	5.2	21.3	Severe acid-etch
				Stress	83.3	4.1	16.9	Severe acid-etch; no cracks
				Weld	96.0	4.7	19.3	Severe acid-etch
D-85	309SCb	Solution at 155°C max.	2142	Control	-	-	-	Specimen lost
				Stress	133.9	6.6	27.0	Severe acid-etch; no cracks
				Weld[d]	122.1	6.0	24.6	Severe acid-etch; knife-line attack
D-86	309SCb	Solution at 155°C max.	2142	Control	94.5	4.7	19.1	Severe acid-etch
				Stress	137.6	6.8	27.9	Severe acid-etch; no cracks
				Weld	127.1	6.3	25.7	Severe acid-etch; knife-line attack
D-28	304L	Vapor at 155°C max.	2142[e]	Control	138.5	6.9	28.0	Severe acid-etch
				Stress	177.2	8.8	35.8	Severe acid-etch; no cracks
				Weld	176.4	8.7	35.6	Severe acid-etch; weld-parent metal interface attack
D-29	304L	Vapor at 155°C max.	2142	Control	-	-	-	Specimen lost
				Stress	126.2	6.2	25.5	Severe acid-etch; no cracks
				Weld	116.2	5.8	23.5	Severe acid-etch; weld-parent metal interface attack
D-74	309SCb	Vapor at 155°C max.	2142	Control	200.6	9.9	40.5	Severe acid-etch
				Stress	212.6	10.5	43.0	Severe acid-etch; no cracks
				Weld	210.4	10.4	42.5	Severe acid-etch; knife-line attack
D-84	309SCb	Vapor at 155°C max.	2142	Control	-	-	-	Specimen lost
				Stress	189.5	9.4	38.3	Severe acid-etch; no cracks
				Weld	173.4	8.6	35.0	Severe acid-etch; knife-line attack

(a) Type of attack refers to flat surfaces of specimens only.
(b) Solution-exposure time includes 765 hr for heating and cooling and 54 hr for decontamination.
(c) Weld specimens of type 304L were Heliarc-welded with type 308L rod.
(d) Weld specimens of type 309SCb were Heliarc-welded with type 309SCb rod.
(e) Vapor-exposure time includes 765 hr in which process solutions were heated and cooled and 54 hr for decontamination.

Discussion of Data. Three of the eight assemblies removed from the tank were in an unstressed condition due to severe corrosion attack which caused the fulcrum in each to slip from position. The three assemblies were removed from the vapor phase. The remaining five assemblies were judged to be at stresses considerably below the original applied stress of 35,000 psi as a result of corrosion attack on the components. Careful examination of the tension face of the eight stress specimens disclosed no cracks.

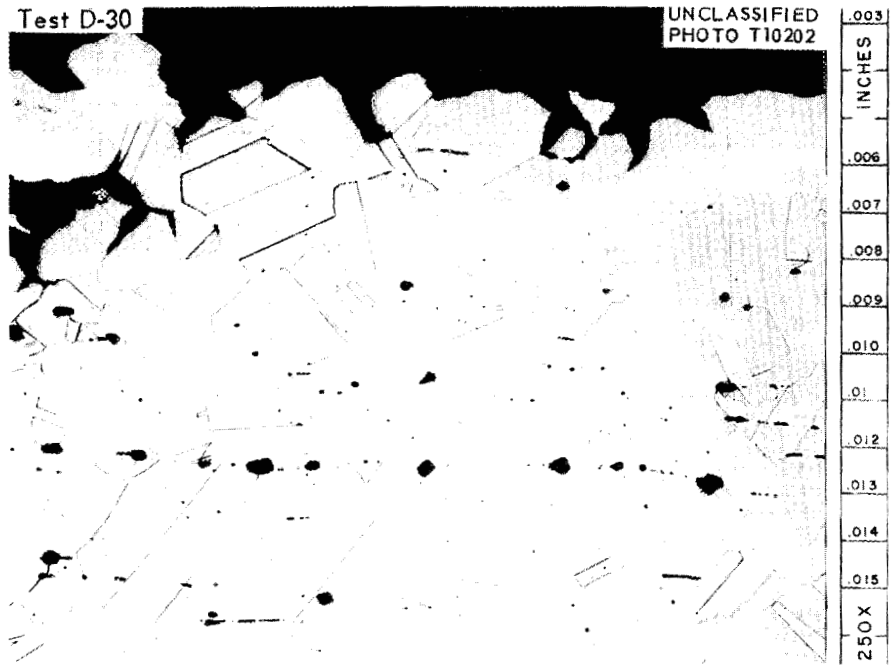
The physical condition of the assemblies was characterized by deep-brown colored mottled stains. All surfaces were grainy in appearance, particularly cross-sectional surfaces. The general condition of vapor-phase assemblies was appreciably worse than that of the solution-phase assemblies. No voluminous corrosion-product deposition was observed; the film weights on the various components ranged between 0.2 and 1.8 mg/cm^2.

(1) Corrosion of Solution-Exposed Specimens. The average corrosion rates for the two solution-exposed stainless alloys were very similar. After 2142 hr at temperatures not in excess of 155°C, type 304L stainless steel corroded at a rate of 19.6 ± 2.8 mpy; the rate for type 309SCb stainless steel was 24.9 ± 3.0 mpy. The nature of the attack on flat surfaces of the stress specimens is shown in Fig. 13. Selective attack at grain boundaries was evident on type 304L stainless steel. At a number of areas, the attack was of sufficient depth (~5 to 6 mils) to be classified as intergranular corrosion. The attack on type 309SCb was of a similar but much milder type. The depth of penetration at grain boundaries was 0.5 mil or less.

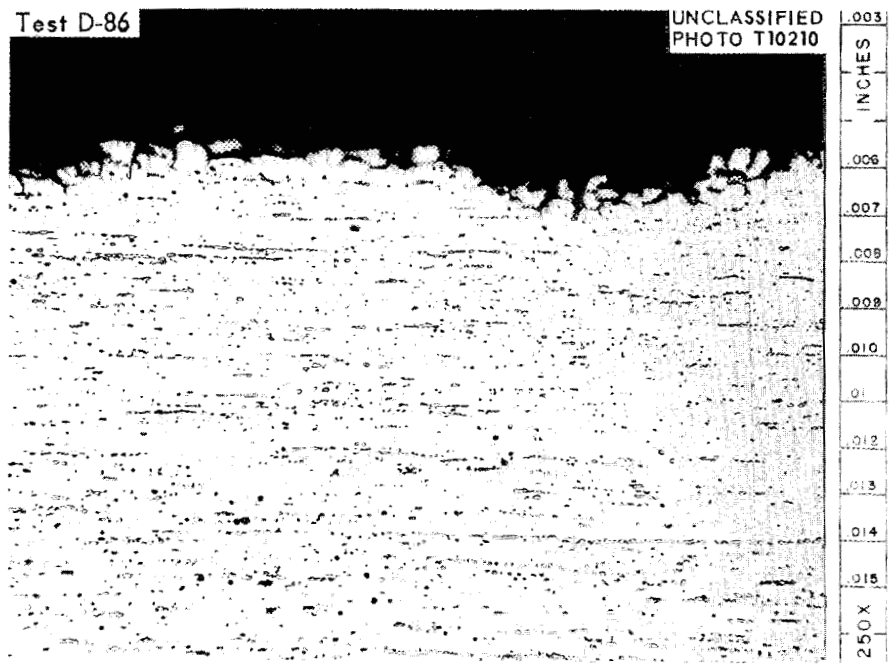
All specimens exhibited quite severe edge-corrosion typified by preferential grain-boundary attack. Pitting was excessive, and pit depths of 15 mils were not uncommon.

An example of weld-metal corrosion behavior in the two stainless steels is shown in Fig. 14. The type 304L deposit in the 304L stainless steel was very grainy and spongy in appearance; indications were that it underwent more severe attack than the parent metal. The type 309SCb deposit exhibited a heavy, grooving-type of attack which was not observed on the parent metal. Knife-line attack was found on the as-welded face of both type 309SCb stainless steel weld specimens. No preferential corrosion in heat-affected zones was observed on the type 304L weld specimens.

None of the specimens of either alloy gave evidence of crevice corrosion.

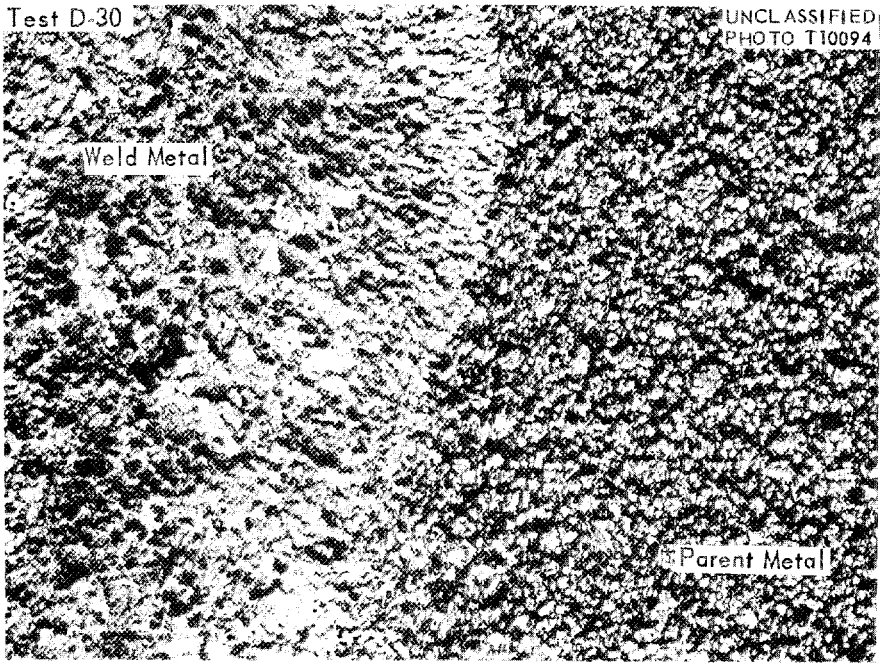


Type 304L Stainless Steel

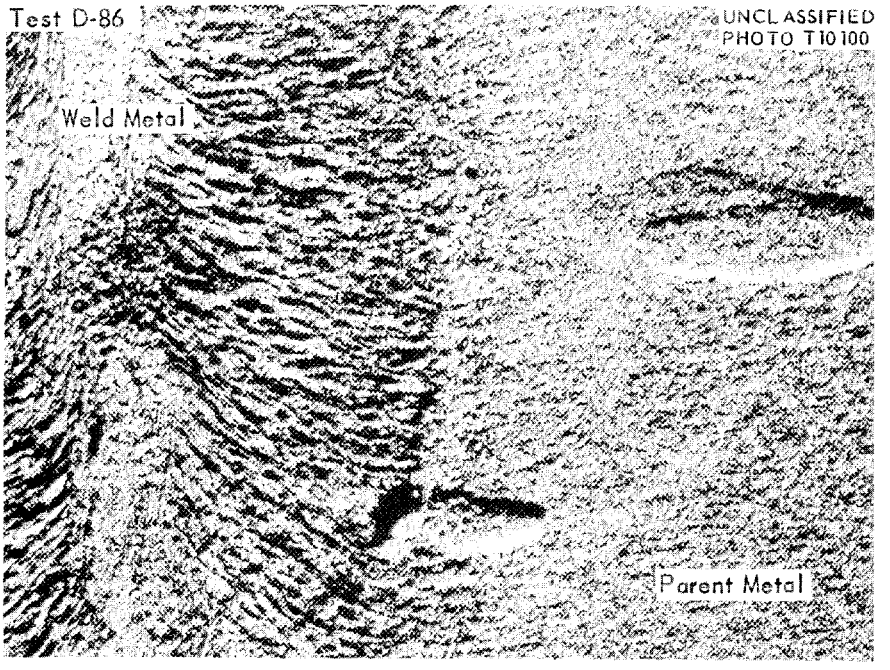


Type 309SCB Stainless Steel

Fig. 13. Surface Attack on Stress Specimens After 4701 hr Exposure (2142 hr at Elevated Temperatures) to Process Solution in Feed Adjustment Tank During Production Period. Etched in aqua regia. 250X.



Type 304L Stainless Steel



Type 309SCB Stainless Steel

Fig. 14. Appearance of Weld-Parent Metal Interface on Machined Face of Weld Specimens After 4701 hr Exposure (2142 hr at Elevated Temperatures) to Process Solution in Feed Adjustment Tank During Development Period. Etched in aqua regia. 20X.

(2) Corrosion of Vapor-Exposed Specimens. The active contact time by vapor on the stress assemblies was 2142 hr at a maximum temperature of 155°C. As mentioned earlier, three of the four assemblies were removed in an unstressed condition due to the severity of the corrosion attack. High corrosion rates were displayed by both alloys; the rates were slightly less than double the rates obtained for a similar exposure period in the solution phase. Type 304L stainless steel corroded at an average rate of 29.7 ± 6.1 mpy while type 309SCb stainless steel corroded at a somewhat higher rate, 39.9 ± 4.0 mpy.

Flat surfaces of specimens of the two alloys were subject to a mild preferential grain boundary attack similar to that shown in Fig. 13. In some areas, removal of complete grains was evident. Edge-corrosion attack was intense and was characterized by intergranular penetration and massive pits.

Weld specimens of both alloys underwent preferential corrosion in the heat-affected zones adjacent to deposits on as-welded surfaces. The depth of the penetration in the attacked areas was between 5 and 8 mils. The condition of weld metal deposits was very much like that shown in Fig. 14. There were no indications of accelerated attack at areas of metal-to-metal contact on any of the specimens.

6. CORROSION DURING PRODUCTION-DEVELOPMENT PERIOD

Stress assemblies in the feed adjustment tank were removed after having undergone continuous exposure during the production period and the first half of the development period (December 15, 1954, to November 10, 1955). No specimens from other process vessels were examined at the end of this period. However, corrosion data for assemblies that were exposed to the complete production-development period of operation will be treated in a subsequent report.

6.1 Feed Adjustment Tank

Function and Description. The operational history of the feed adjustment tank during the production period and the first half of the development period has been reported previously (Sections 4.2 and 5.1). The nominal initial and final solution compositions for feed adjustment tank operation have been reported in Table 6.

Corrosion Data. Table 15 presents a summary of the exposure history for stress assemblies exposed in the tank. Corrosion data for the assemblies exposed in the solution and vapor phases appear in Table 16. The total contact time for the solution-exposed assemblies was

5881 hr. Of this period, 3184 hr were accumulated with process solution at room temperature, leaving an active exposure period of 2697 hr which was accumulated at temperatures above room temperature but not in excess of 155°C. Similarly, the active exposure period of 2697 hr for the vapor-phase assemblies was based upon the hours in which process solutions were above room temperature.

Table 15. Summary of Exposure History for Stainless Steel Stress Assemblies in Feed Adjustment Tank During the Production-Development Periods

	Exposure Time (hr)	
	Solution	Vapor
Total residence time in tank	7856	7856
Five cold runs at 155°C max.	85	85
Twenty-nine hot runs at 155°C max.	1330	1330
Recovery runs at 133°C max.	1208	1208
Holding time at room temp.*	3184	0
Decontamination at 40 to 95°C	74	74
Total active exposure time	2697	2697

* Includes 2820 hr with dissolver and recovery solutions and 364 hr with decontamination solutions.

** Holding time at room temperature not considered in total active exposure time.

Table 16. Corrosion of Stainless Steel Stress Assemblies in Feed Adjustment Tank During Production-Development Period

Test No.	Alloy Type	Environment	Total Time (hr)	Specimen	Wt. Loss (mg/cm^2)	Penetration (mils)	Penetration (mpy)	Type of Attack[a]
D-8	304L	Solution at 155°C max.	2697[b]	Control	125.8	6.2	20.1	Severe acid-etch
				Stress	97.4	4.8	15.5	Severe acid-etch; no cracks
				Weld[c]	115.2	5.6	18.3	Severe acid-etch
D-9	304L	Solution at 155°C max.	2697	Control	124.9	6.1	19.8	Severe acid-etch
				Stress	103.1	5.0	16.3	Severe acid-etch; no cracks
				Weld	118.0	5.8	18.7	Severe acid-etch
D-60	309SCb	Solution at 155°C max.	2697	Control	133.2	6.5	21.1	Severe acid-etch
				Stress	161.7	7.4	25.7	Severe acid-etch; no cracks
				Weld[d]	145.5	7.1	23.1	Severe acid-etch; knife-line attack
D-61	309SCb	Solution at 155°C max.	2697	Control	133.5	6.6	21.4	Severe acid-etch
				Stress	166.1	8.1	26.4	Severe acid-etch; no cracks
				Weld	146.7	7.2	23.3	Severe acid-etch; knife-line attack
D-6	304L	Vapor at 155°C max.	2697[e]	Control	-	-	-	Specimen lost
				Stress	233.8	11.5	37.4	Mild intergranular; no cracks
				Weld	255.6	12.6	41.0	Mild intergranular
D-7	304L	Vapor at 155°C max.	2697	Control	-	-	-	Specimen lost
				Stress	223.7	11.0	35.8	Mild intergranular; no cracks
				Weld	231.7	11.4	37.1	Mild intergranular
D-58	309SCb	Vapor at 155°C max.	2697	Control	-	-	-	Specimen lost
				Stress	286.3	14.1	45.8	Severe acid-etch; no cracks
				Weld	277.6	13.7	44.4	Severe acid-etch; knife-line attack
D-59	309SCb	Vapor at 155°C max.	2697	Control	-	-	-	Specimen lost
				Stress	286.5	14.1	45.8	Severe acid-etch; no cracks
				Weld	277.3	13.7	44.4	Severe acid-etch; knife-line attack

(a) Type of attack refers to flat surfaces of specimens only.
(b) Solution-exposure time includes 800 hr for heating and cooling and 74 hr for decontamination.
(c) Weld specimens of type 304L were Heliarc-welded with type 308L rod.
(d) Weld specimens of type 309SCb were Heliarc-welded with type 309SCb rod.
(e) Vapor-exposure time includes 800 hr in which process solutions were heated and cooled 74 hr for decontamination.

Discussion of Data. Eight stress assemblies of types 304L and 309SCb stainless steel were removed from the solution and vapor phases of the feed adjustment tank. Duplicate assemblies of each alloy were exposed in each phase. Excessive corrosion attack on the four vapor-exposed assemblies resulted in complete relieving of the initially applied stress of 35,000 psi. The solution-exposed assemblies were intact, but the magnitude of the stress remaining on the specimens was estimated visually to be appreciably below the initial stress of 35,000 psi. Examination of the stress specimens disclosed no cracking in either alloy.

The physical condition of the assemblies as removed was generally poor, particularly in the case of the vapor phase. The components of all assemblies were dark gray-brown, and surfaces exhibited a heavily pickled appearance which was more pronounced on the vapor-phase specimens. Film weights varied between 0.3 and 1.7 mg/cm^2 with the heavier weights encountered on the vapor-phase specimens.

(1) Corrosion of Solution-Exposed Specimens. The active exposure time at temperatures up to 155°C maximum was 2697 hr which included 74 hr for decontamination with hot solutions. Corrosion behavior of the two alloys was very similar. Type 304L stainless steel corroded at an average rate of 18.1 ± 2.1 mpy, whereas type 309SCb stainless steel corroded at an average rate of 23.5 ± 2.7 mpy. Flat surfaces of specimens of both alloys were considerably roughened; metallographic examination showed the attack as a severe acid-etch with mild preferential corrosion at grain boundaries, similar to that shown in Fig. 13. In a few cases, areas were found in both alloys where grains were encircled completely by the preferential attack. Edge-corrosion was intense; severe intergranular attack and pits up to 15 mils in depth characterized the attack.

Weld-metal corrosion in the two alloys was similar to that shown in Fig. 14. The type 308L deposit appeared more resistant than the type 304L stainless steel parent metal. The type 309SCb weld deposit was subject to a severe grooving-type of attack and appeared less resistant than the parent metal. Knife-line corrosion attack was observed on the as-welded face of the duplicate type 309SCb weld specimens. The depth of the attack was approximately 5 mils. No preferential attack was found in heat-affected zones in the type 304L weld specimens.

None of the specimens of either alloy were prone to an accelerated rate of corrosion attack at areas of metal-to-metal contact.

(2) Corrosion of Vapor-Exposed Specimens. During an active exposure period of 2697 hr which included 74 hr for decontamination, the average corrosion rate for vapor-exposed type 304L stainless steel was 37.8 ± 2.0 mpy as compared with an average rate of 45.1 ± 0.7 mpy for type 309SCb stainless steel. Flat surfaces of specimens of both alloys were very grainy-appearing. Metallographic examination of the surfaces disclosed the attack to be of a mildly intergranular form extending to a depth of 4 mils in type 304L stainless steel and to a depth of 0.5 mil in the smaller-grained type 309SCb stainless steel.

Edge corrosion was experienced by specimens of both alloys. The attack was similar to that shown in Fig. 9 except that it was more intense. Prolific pitting was present with pits ranging up to 15 mils in depth.

Weld metal attack was much the same as depicted in Fig. 14. The type 309SCb deposit was pitted and spongy-appearing; the general condition was worse than the base metal. The type 308L weld metal deposit was channeled extensively.

Knife-line attack occurred along the welds on the as-welded face of the two type 309SCb stainless steel specimens; the depth of the attack was approximately 5 mils. The type 304L stainless steel weld specimens were immune to preferential attack in heat-affected zones.

No crevice corrosion was found at areas of metal-to-metal contact on specimens of either alloy.

6.2 Miscellaneous Information

This section of the report discusses: (a) qualitative examination of auxiliary equipment components removed from the pilot plant upon completion of the production and development periods, and (b) results of a laboratory corrosion study to determine the contribution of edge-surface attack to the total attack on stainless steel specimens in boiling aluminum nitrate-nitric acid solution. The edge-surface attack results are presented because many of the specimens removed from the pilot plant were highly subject to this type of attack.

(1) Corrosion of Spray Nozzles. Two type 316 stainless steel spray nozzles used for jetting decontamination solutions over the internal surfaces of the feed adjustment tank were removed for examination upon completion of the production and development periods. The nozzles were a solid cone type and were located

in the vapor phase of the tank. The exposure time was 2697 hr at a maximum temperature of 155°C. Seventy-four hours in this period constituted the time required for decontamination with dilute nitric acid and alkaline tartrate and oxalate solutions at 40 to 95°C.

The condition of the spray nozzles removed from the tank is shown in Fig. 15. Extremely severe end-grain attack occurred which resulted in pitting oriented along an axis parallel with the rolling direction of the bar from which the nozzles were machined. Many of the pits, estimated at 250 to 300 mils in depth, completely penetrated some areas on the nozzles, as is evident in the end-view photograph in Fig. 15. One of the nozzles was sectioned for metallographic examination; a photomicrograph of a transverse section appears in Fig. 16. The attack was heavily intergranular in nature. Exclusive of the pitted areas, penetration by intergranular attack was as great as 15 mils.

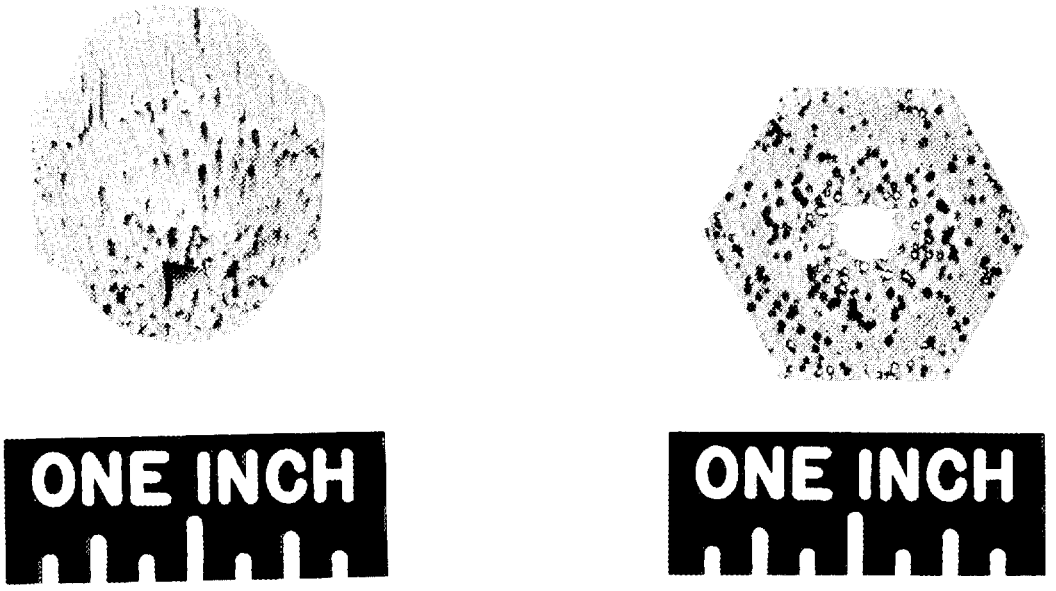
(2) Corrosion of Agitator Shaft. A second item examined was a 1-in.-dia. type 309SCb stainless steel agitator shaft that was also removed from the feed adjustment tank at the end of the production and development periods. The shaft was part of a motor-driven agitator used for stirring process solution in the tank during the evaporation and digestion cycles. The shaft extended nearly the full depth of the tank. The active exposure time at temperatures of 155°C maximum in both solution and vapor phases was 2697 hr.

A 5-in.-dia. type 316 stainless steel agitator blade was originally tack-welded to the bottom of the shaft. During operation, the agitator broke loose from the shaft as the result of either a mechanical failure or excessive corrosion attack on or around welded areas. When the tank was finally opened for inspection, no trace of the blade was found. It was presumed to have undergone complete dissolution.

Five-inch sections were cut from the top and bottom ends of the shaft for metallographic examination. The two sections were representative of solution and vapor conditions of exposure. Photographs of the sections are included in Fig. 17. The solution-exposed section still had weld metal-deposit on the bottom where the agitator blade was attached. End-grain attack was more severe in the solution than in the vapor phase, as shown by the longitudinal sections in Fig. 18. Photomicrographs of the longitudinal sections, shown in Fig. 19, disclosed intergranular corrosion attack with extensive pitting. The maximum penetration by pitting was 15 mils.

UNCLASSIFIED
PHOTO T10421

UNCLASSIFIED
PHOTO T10420



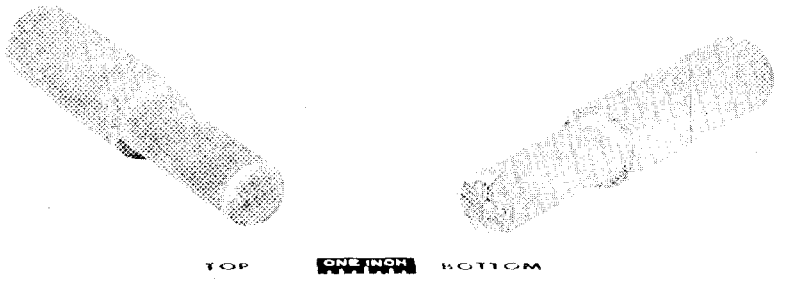
Side View

End View

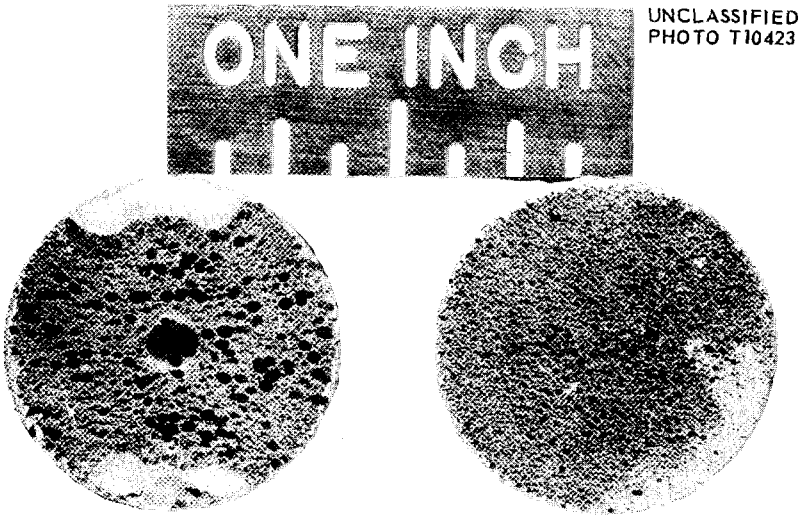
Fig. 15. Type 316 Stainless Steel Spray Nozzles After 2697 hr of Vapor Exposure in Feed Adjustment Tank During Production-Development Period of Thorex Pilot Plant Operation.

Fig. 16. Intergranular Attack on Type 316 Stainless Steel Spray Nozzles After 2697 hr of Vapor Exposure in Feed Adjustment Tank During Production-Development Period of Thorex Pilot Plant Operation. Etched in aqua regia. 250X.

UNCLASSIFIED
PHOTO T10422



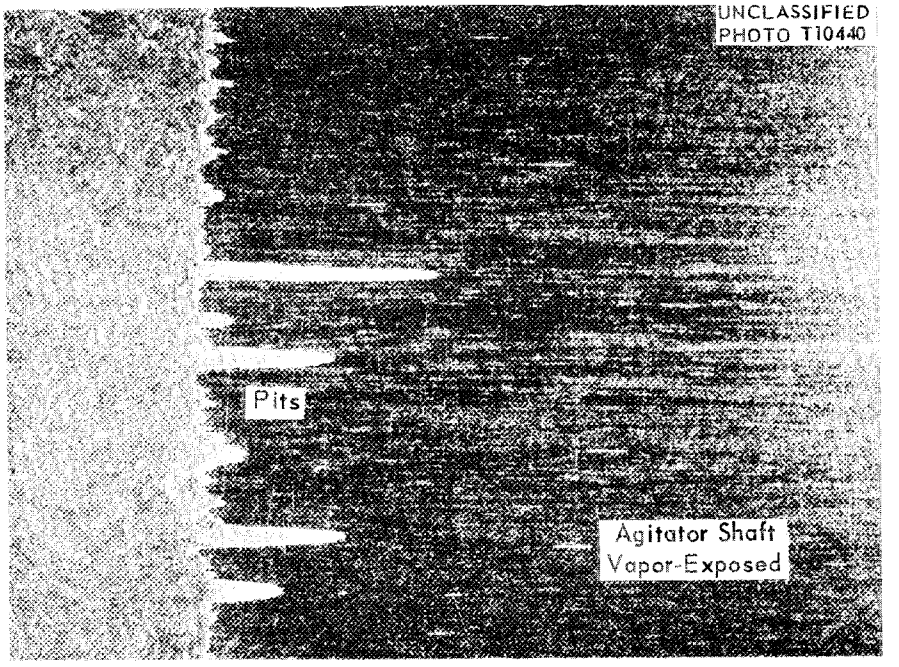
UNCLASSIFIED
PHOTO T10423



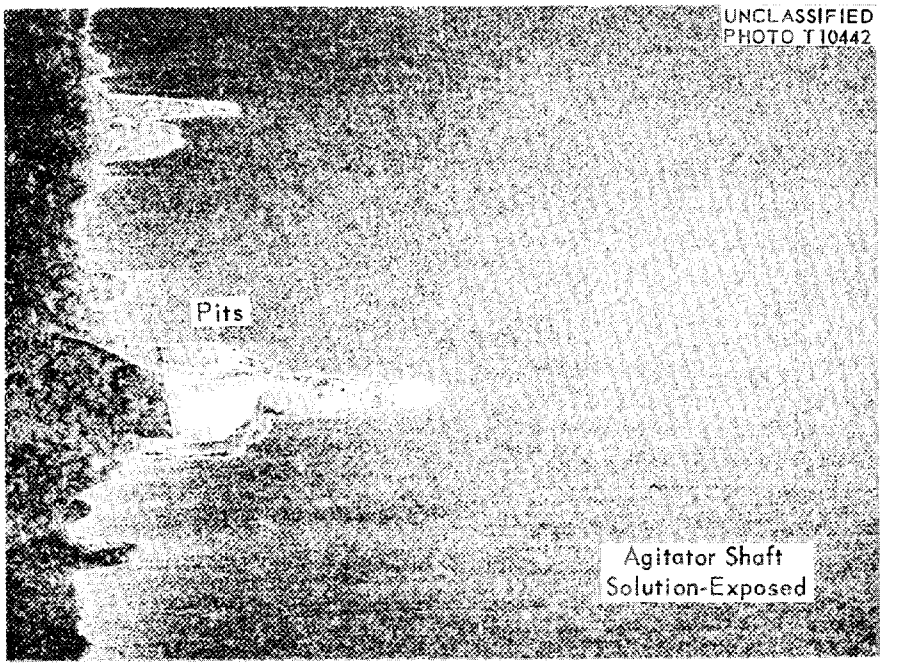
Bottom Section
Solution-Exposed

Top Section
Vapor-Exposed

Fig. 17. Condition of Type 309SCB Stainless Steel Agitator Shaft Sections After Solution and Vapor Exposure in Feed Adjustment Tank During Production-Development Period of Thorex Pilot Plant Operation.

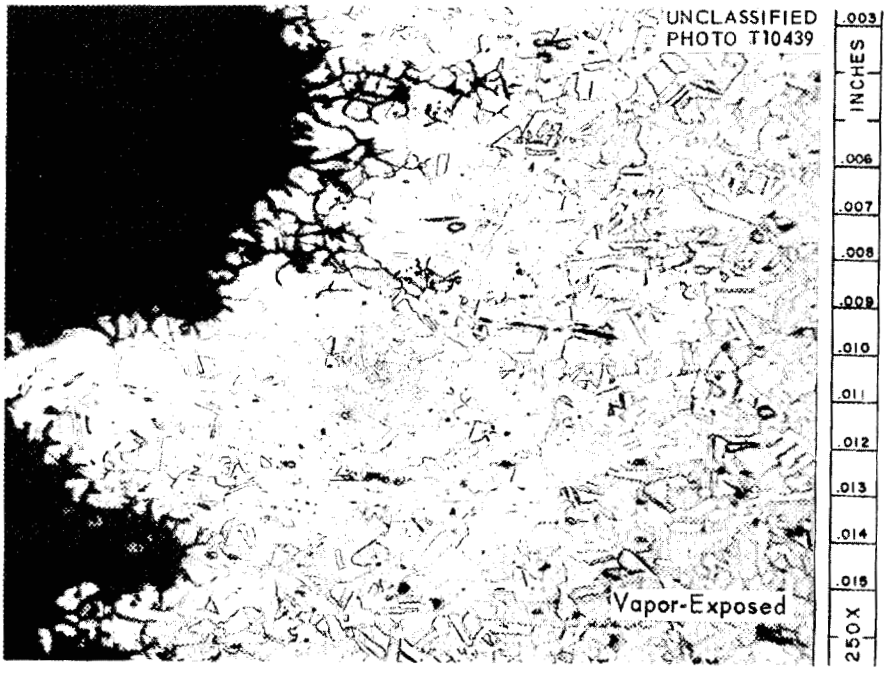


Top Section

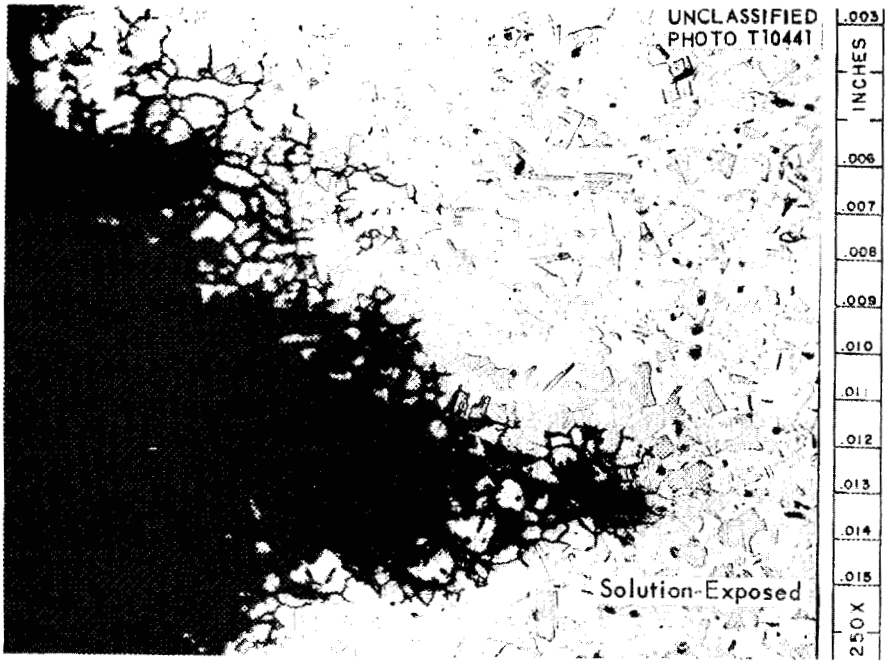


Bottom Section

Fig. 18. Pitting Attack on Sections of Type 309SCB Stainless Steel Agitator Shaft After Solution and Vapor Exposure in Feed Adjustment Tank During Production-Development Period of Thorex Pilot Plant Operation. 7X.



Top Section



Bottom Section

Fig. 19. Intergranular Attack on End Sections of Type 309SCB Stainless Steel Agitator Shaft During Solution and Vapor Exposure in Feed Adjustment Tank During Production-Development Period of Thorex Pilot Plant Operation. Etched in aqua regia. 250X.

(3) Laboratory Studies on Edge-Surface Corrosion. Examination of the control, stress, and weld specimens removed from the pilot plant disclosed a highly selective corrosion attack on cross-sectional or edge surfaces. The intensity of the attack was related directly to the corrosivity of the environment. In environments where the observed corrosion rates based upon total attack were moderately low, edge-corrosion effects were not pronounced. However, when the rates were 10 mpy or greater, edge-surface attack assumed significant proportions. The occurrence of the attack in the more corrosive environments raised the question as to its influence on observed corrosion rates since the latter were based upon total attack on the specimens. The possibility existed that corrosion rates in the more corrosive environments were pessimistically high because of significant edge-surface attack. As applied to pilot plant operation, the observed rates would be misleading, since process solution contacted flat, as-rolled surfaces principally and not edge surfaces of the stainless steel vessels. It is conceivable, however, that any edge surfaces subject to exposure could be eliminated in future component designs.

The problem of an edge-corrosion attack was encountered during a laboratory corrosion study with stainless steel specimens in a simulated Thorex process waste solution. The stainless steel was type 309SCb; the solution consisted of 2 *M* $Al(NO_3)_3$ and 1 *M* HNO_3. The latter was added to prevent hydrolytic precipitation of the aluminum ion at the test temperature, approximately 110°C. Wafer-type specimens, 3/16-in.-thick, were used. The cross-sectional areas underwent intense end-grain attack; complete penetration of the wafer thickness occurred during 5000 hr of test.

In order to evaluate the effect of end-grain corrosion on total attack, three types of specimens were prepared as follows:

(a) 0% edge surface. Coupons 1/2 in. wide x 1 3/4 in. long were machined from 1/8-in.-thick stainless steel sheet. Edge surfaces were eliminated by Heliarc welding with type 309SCb stainless steel rod. Total surface area per coupon was 15.2 cm^2.

(b) 15% edge surface. Coupons 1/2 in. wide by 1 3/4 in. long were machined from the same type 309SCb sheet used for the 0% edge-surface coupons. Total surface area per coupon was 15.2 cm^2, of which 2.3 cm^2 was edge surface.

(c) 71% edge surface. Wafers 3/16 in. thick were machined from 1 1/4-in.-dia. type 309SCb stainless steel bar. Total surface area per wafer was 19.2 cm^2, of which 13.6 cm^2 was edge surface.

The materials used for preparing the different types of specimens were found to conform with the chemical composition limits specified for AISI type 309SCb stainless steel. The chemical compositions are listed in Table 17.

Table 17. Chemical Compositions of Type 309SCb Stainless Steel for Edge-Corrosion Study

Element	Chemical Composition (wt %) Edge-Surface Area		
	0%	15%	71%
C	0.04	0.04	0.07
Mn	1.55	1.55	1.21
Si	0.31	0.31	0.32
Cr	23.0	23.0	22.5
Ni	15.0	15.0	14.6
Cb	0.57	0.57	0.76
Fe	Balance	Balance	Balance
Cb/C	14.2	14.2	10.9

Specimens were exposed in aerated and boiling 2 *M* $Al(NO_3)_3$-1 *M* HNO_3 solution contained in glass flasks equipped with water-cooled condensers. Three or four specimens were used in each test. The specimens were weighed periodically to the nearest 0.1 ± 0.2 mg. In all cases, the deviation in individual weight loss values was not greater than 10% of the average value in a particular test.

Figure 20 presents a plot of observed average weight losses as a function of exposure time for the three types of test specimens. There was no significant effect of edge-surface area on total attack during the first 4500 hr; weight losses of 70.1, 54.1, and 68.0 mg/cm^2 were observed for specimens with 0, 15, and 71% edge-surface area, respectively. However, starting in the 3500- to 4500-hr exposure interval, the rate of attack on the 71% edge-surface specimens started to increase much more rapidly than were the observed rates on the other specimens. By the end of 5000 hr, the average weight loss was 100 mg/cm^2, and was still increasing rapidly as compared with losses of 87 mg/cm^2 and 68 mg/cm^2, respectively, for the 0 and 15% edge-surface specimens. Thus for extended periods of exposure (> 4500 hr), there was an indication that a high percentage of edge-surface area on specimens would result in an appreciable contribution to the total corrosion attack. Although the boiling 2 *M* $Al(NO_3)_3$ - 1 *M* HNO_3 solution did not constitute an exact duplication of any Thorex pilot plant environment, the similarity between the edge-corrosion effects noted on laboratory specimens and edge corrosion on pilot plant specimens suggested that the ability of the environments to produce this type of corrosion was analogous.

The marked increase in the rate of attack on all specimens that started during the 2000- to 3000-hr exposure interval (Fig. 20) was attributed to the presence of hexavalent chromium in solution. The Cr(VI), produced by attack on the stainless steel, increased to between 30 and 50 ppm after 3000 hr. A similar effect of Cr(VI) on the corrosion behavior of stainless steels in nitric acid solutions has been observed.[22,23] Acceleration in the rate of attack due to the increase in Cr(VI) content required considerably shorter exposure periods in the boiling, concentrated nitric acid solutions that were used[22,23] than were required in the present boiling aluminum nitrate-nitric acid system, however. The influence of Cr(VI) on the corrosion of type 309SCb and other stainless steels in simulated Thorex process solutions will be treated in a report to be issued at a later date.

The attack experienced by a type 309SCb stainless steel wafer specimen with 71% edge-surface area in the boiling aluminum nitrate-nitric acid solution is shown in Fig. 21. Approximately 90% of the cross-sectional areas was very grainy in appearance and contained many pits. The light-colored areas near the periphery of the specimen are "islands" of the relatively unattacked original surface. Corrosion attack generally initiated around the hole in the center of the specimen and gradually propagated to the outer edge with increased exposure time. Pit depths up to 80 mils were measured microscopically after 4345 hr. Perforation due to pitting occurred by the end of 5180 hr. The specimen was approximately 150 mils thick at this time. A typical stringer-type pit is shown in Fig. 22. Pitting and intergranular corrosion attack on the specimen are also shown in Fig. 23.

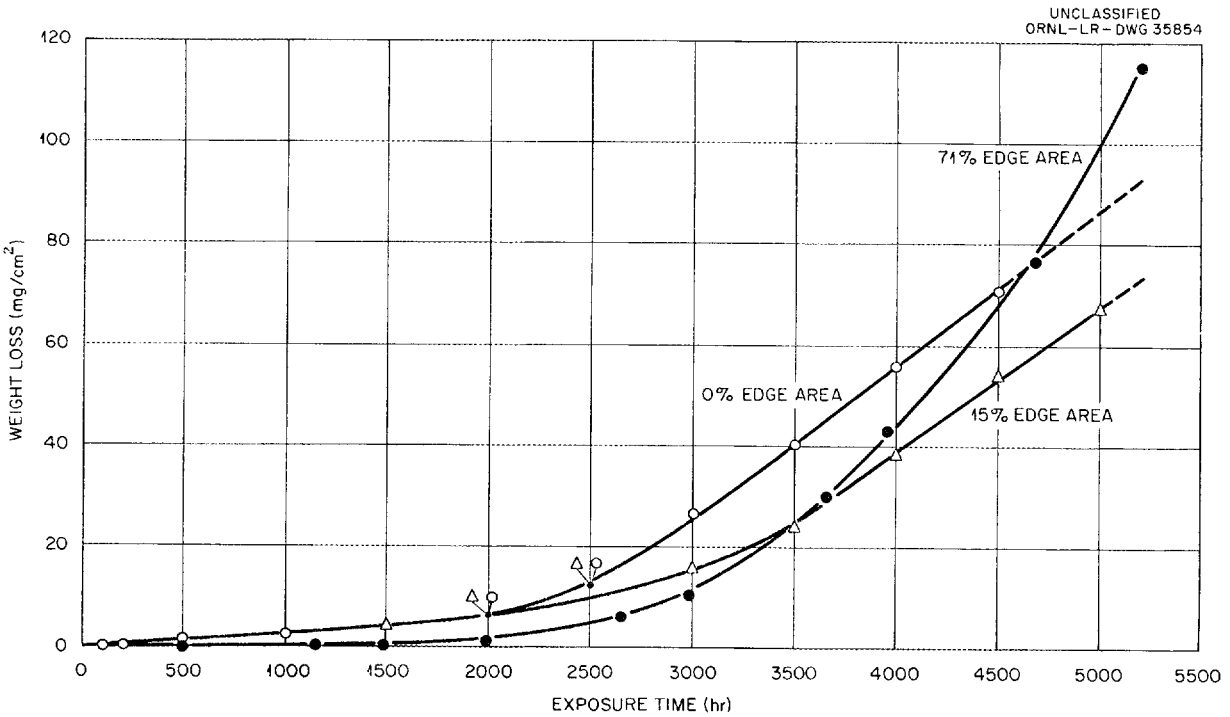


Fig. 20. The Effect of Edge-Surface Area on Total Attack on Type 309SCB Stainless Steel by Boiling and Aerated 2 M $Al(NO_3)_3$ - 1 M HNO_3 Solution.

Test 673

UNCLASSIFIED
PHOTO 1141-2

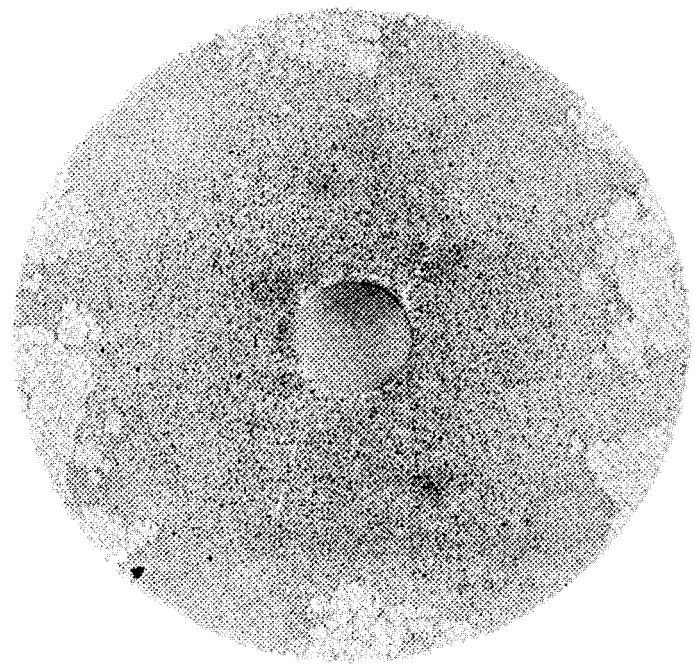


Fig. 21. Condition of Type 309SCB Stainless Steel Test Specimen (71% Edge Area) After 4345 hr in Boiling and Aerated 2 M $Al(NO_3)_3$ - 1 M HNO_3 Solution. 3X.

Fig. 22. Stringer-Type Pitting Attack on Type 309SCB Stainless Steel Specimen (71% Edge Area) After 5540 hr in Boiling and Aerated 2 M $Al(NO_3)_3$ - 1 M HNO_3 Solution. Measured depth of pit was 78 mils. Etched in aqua regia. 75X.

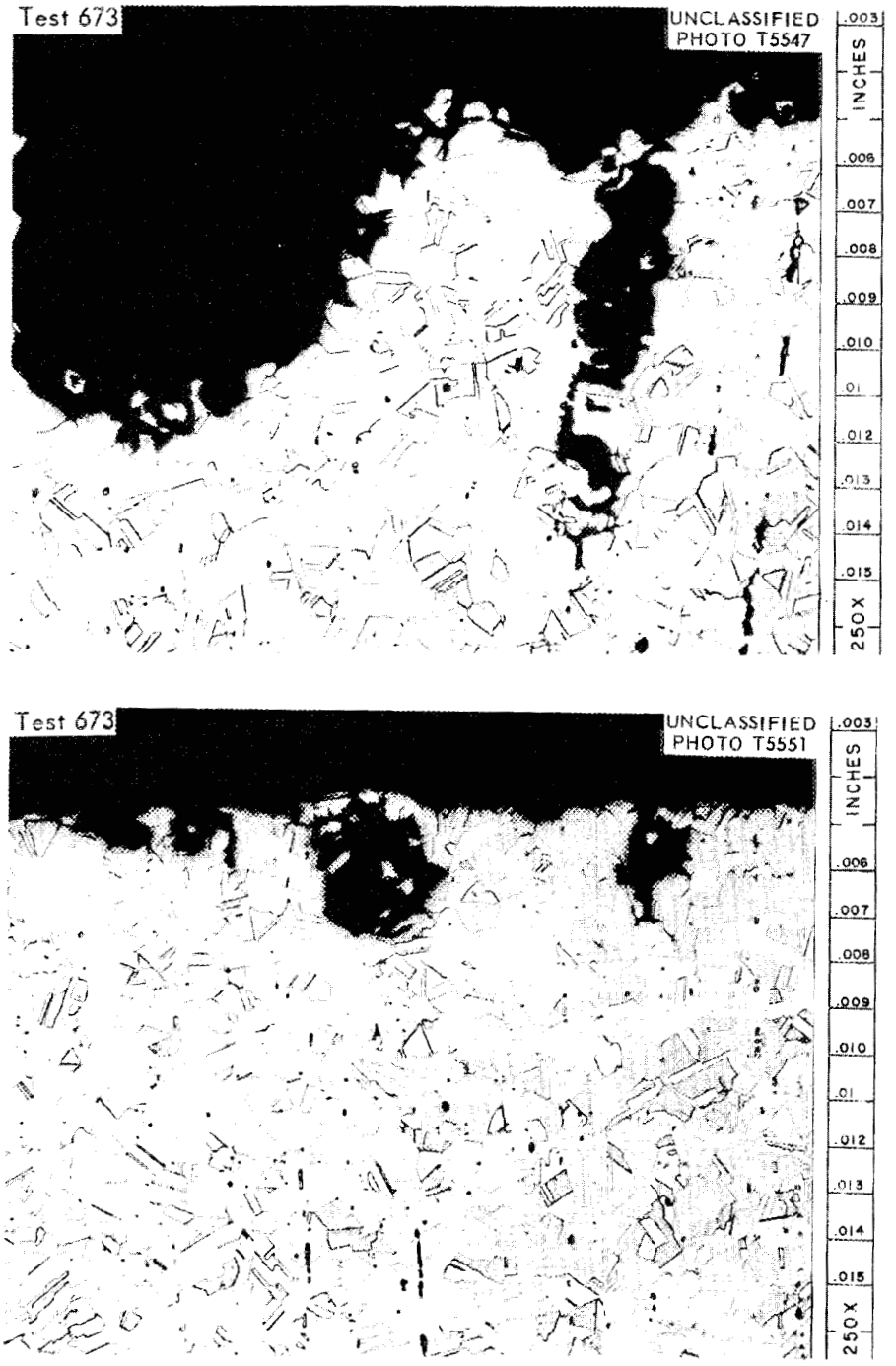


Fig. 23. Intergranular and Pitting Attack on Type 309SCB Stainless Steel Test Specimen (71% Edge Area) After 5540 hr in Boiling and Aerated 2 M $Al(NO_3)_3$ - 1 M HNO_3 Solution. Etched in aqua regia. 250X.

Edge-corrosion attack on the 15% edge-surface specimens of type 309SCb stainless steel was identical in appearance to that observed on edge surfaces of a number of stainless steel specimens removed from the Thorex pilot plant. The attack, in both cases, was characterized by pitting and intergranular corrosion.

In relating the results of the laboratory studies on edge-surface corrosion to interpretation of the corrosion rates experienced by pilot plant specimens in the most corrosive environments, the edge-surface areas of the latter must be considered, of course. The edge-surface areas of the control, stress, and weld specimens were 15, 17, and 35%, respectively, of the total surface areas. Thus two of the specimen types (the control and the stress) had edge-surface areas equal or close to the 15% edge-surface area specimens used in the laboratory study. The weld specimen, with 35% edge-surface area, had an edge-surface area only one-half that of the wafers used in laboratory tests.

The laboratory tests showed that for exposure periods up to 5000 hr in boiling 2 *M* $Al(NO_3)_3$ - 1 *M* HNO_3 solution, specimens with 15% edge-surface area were not subject to any greater attack than that experienced by the 0% edge-surface area specimens. Actually, the total attack was somewhat greater on the latter. After 5000 hr, the attack on the 71% edge-surface specimens became 15 and 50% greater, respectively, than on 0 and 15% edge-surface specimens. Thus, if an edge-surface corrosion effect were operative on pilot plant specimens, it would be more likely to occur on the weld specimens with 35% edge-surface area. However, a review of weight loss data for control, stress, and weld specimens in Tables 5, 8, 10, 12, 14, and 16 shows that total attack on weld specimens, with very few exceptions, was no greater than the attack on either control or stress specimens. In numerous cases, the attack on weld specimens was less than that on the control and stress specimens.

In consideration of the laboratory data, it was concluded that corrosion rates on pilot plant specimens were representative of the corrosion experienced by the various process vessels and that the attack on edge surfaces of the specimens did not significantly affect the total attack. Tests are being run with suitable specimens in the pilot plant to examine edge-corrosion behavior under actual operating conditions. The results of these tests will be reported.

7. GENERAL DISCUSSION

A summary of the average corrosion rates for types 304L and 309SCb stainless steel exposed to solution- and vapor-phase environments in the Thorex

pilot plant during the production period and the first half of the development period is shown in Table 18. These two periods of operation occurred between December 15, 1954, and November 10, 1955. The reported rates for the solution-phase specimens were based upon actual contact time when process solutions were above room temperature. The maximum operating temperature was 155°C. The vapor-exposure times were determined in the same manner as solution-exposure times.

Neither of the alloys, initially stressed at 35,000 psi, was subject to stress-corrosion cracking in any of the environments. Similarly, there was no evidence of an acceleration in the rate of attack of the two alloys as a result of the applied stress.

Corrosion During Production Period. Two aggressive environments were disclosed during the production period, namely, the vapor phases in the feed adjustment tank and the BT vapor separator. In the former, operated at a maximum temperature of 155°C, type 304L stainless steel corroded at a rate of 21.1 ± 2.0 mpy as compared with 35.0 ± 8.5 mpy for type 309SCb stainless steel. The maximum corrosion rate for either alloy exposed in the solution phase of the feed adjustment tank for a similar exposure period, 555 hr, was 8.5 mpy for type 309SCb stainless steel. Generally, the rates for the alloys ranged between 4 and 6 mpy in the solution phase. The second environment of aggressive attack was the BT vapor separator, operated between 110 and 115°C. Type 347 stainless steel was attacked at a rate of 33.2 ± 13.8 mpy; type 304L stainless steel corroded at a substantially lower rate of 10.5 ± 3.7 mpy.

Other process vessels experienced mild or minor corrosion attack during the production period. Corrosion rates for types 304L and 309SCb stainless steel in the solution and vapor phases of the batch dissolver tank did not exceed 8.4 mpy in the solution phase. In this particular vessel, corrosion attack in the solution phase was approximately two times as great as it was in the vapor phase for comparable exposure periods. Corrosion rates for the two stainless alloys in the AP catch tank, which operated at about 40°C, were 0.2 mpy or less for both solution- and vapor-phase exposure.

Corrosion During Production and Production-Development Periods. Extended operational time in the feed adjustment tank during the production-development periods at temperatures above room temperature and not in excess of 155°C resulted in a marked increase in the rate of corrosion attack on the stainless alloys, particularly in the solution phase. For an exposure period of 2697 hr, solution-phase corrosion rates for types 304L and 309SCb stainless steel were 18.1 ± 2.1 and 23.5 ± 2.7 mpy, respectively. The vapor-phase corrosion rates for the same exposure period, 2697 hr, were nearly double the rates for the solution-exposed alloys. For example, in the vapor, type 304L stainless steel corroded at a rate of 37.8 ± 2.0 mpy and type 309SCb stainless steel corroded at a rate of 45.1 ± 0.7 mpy.

Table 18. Summary of Corrosion Rates for Types 304L and 309SCb Stainless Steel Exposed in ORNL Thorex Pilot Plant During the Period from December 15, 1954, to November 10, 1955

Period of Operation	Vessel Designation and Number	Specimen Location	Active Exposure (hr)*	Corrosion Rate (mpy) 304L	Corrosion Rate (mpy) 309SCb
Production	Batch dissolver tank (S-1)	Solution	634	8.0 ± 0.4	3.1 ± 0.5
		Vapor	634	3.6 ± 1.7	2.0 ± 0.3
Production	Feed adjustment tank (S-2)	Solution	555	4.2 ± 0.4	6.1 ± 2.5
		Vapor	555	21.1 ± 2.0	35.0 ± 8.5
Production	AP catch tank (N-2)	Solution	910	0.1 ± < 0.1	0.1 ± < 0.1
		Vapor	910	0.1 ± < 0.1	0.1 ± < 0.1
Production	BT vapor separator (P-2)	Vapor	910	10.5 ± 3.7	-
		Vapor**	910	33.2 ± 13.8	-
Development	Feed adjustment tank (S-2)	Solution	2142	19.6 ± 2.8	24.9 ± 3.0
		Vapor	2142	29.7 ± 6.1	39.9 ± 4.0
Production-Development	Feed adjustment tank (S-2)	Solution	2697	18.1 ± 2.1	23.5 ± 2.7
		Vapor	2697	37.8 ± 2.0	45.1 ± 0.7

* Holding time for process solution at room temperature not included.

** Reported corrosion rate for type 347 stainless steel; no type 309SCb stainless steel specimens exposed in BT vapor separator.

Several possibilities existed for the higher corrosion rates experienced in the vapor phase as compared with solution-phase rates in the feed adjustment tank. First, the corrosion of austenitic stainless steels in nitric acid is rapidly accelerated by an increase in temperature above the boiling point for a specific acid concentration and also by an increase in nitric acid concentration at temperatures above the boiling point.[24,25] The entering solution to the feed adjustment tank was 5 *M* in nitric acid; the final solution was 0.5 *M* nitric acid-deficient. At the start of the evaporation-digestion cycle in the tank at 110°C, the exiting vapor was 1 *M* in HNO_3.[3] As boiling continued, the vapor increased to a maximum of 12 *M* in HNO_3, and then decreased to an equilibrium value of 8 *M* HNO_3 as water of crystallization was driven from the process solution at a temperature of approximately 155°C. Therefore, the combination of increased temperature and increased nitric acid concentration could have been responsible for the higher corrosion rates observed in the vapor phase.

A second possibility for the high vapor-phase corrosion rates was the presence of uncomplexed fluoride in the nitric acid-rich vapors. Laboratory tests with simulated Thorex solutions have shown that when fluoride is complexed by aluminum and/or thorium, corrosion attack on stainless steels is negligible.[16] However, corrosion increased considerably when aluminum and thorium were not present to complex the fluoride. Other experimental data obtained during evaporation of a simulated Thorex dissolver solution showed that the fluoride content in four cuts of condensate averaged 4 ppm.[26] The first three cuts were low in fluoride. However, the fourth cut, taken near the completion of the evaporation, showed a fluoride content of 11.5 ppm. It has been reported that fluoride-contaminated boiling 65% HNO_3 solution produced a significant increase in the rate of attack on austenitic stainless steels.[27]

A graph showing average weight losses for solution- and vapor-exposed specimens of types 304L and 309SCb stainless steel in the feed adjustment tank during the production-development periods of operation is shown in Fig. 24. The active exposure time refers to the period during which process solution was above room temperature and not in excess of 155°C. The relationship between weight loss and time was linear between 600 and 2700 hr for the solution-exposed specimens. Slight deviation from a linear relationship was obtained for specimens of both alloys exposed in the vapor during the entire exposure period of approximately 2700 hr. The steady-state corrosion rates for solution-exposed types 304L and 309SCb stainless steel between the 600- to 2700-hr interval were approximately 24 and 29 mpy, respectively. The average rates for the entire 2700 hr were 18 ± 2.1 mpy for type 304L and 23.5 ± 2.7 mpy for type 309SCb stainless steel. The average corrosion rates for the vapor-exposed alloys during the 2700-hr exposure were 37.8 ± 2.0 and 45.1 ± 0.7 mpy, respectively, for types 304L and 309SCb stainless steel.

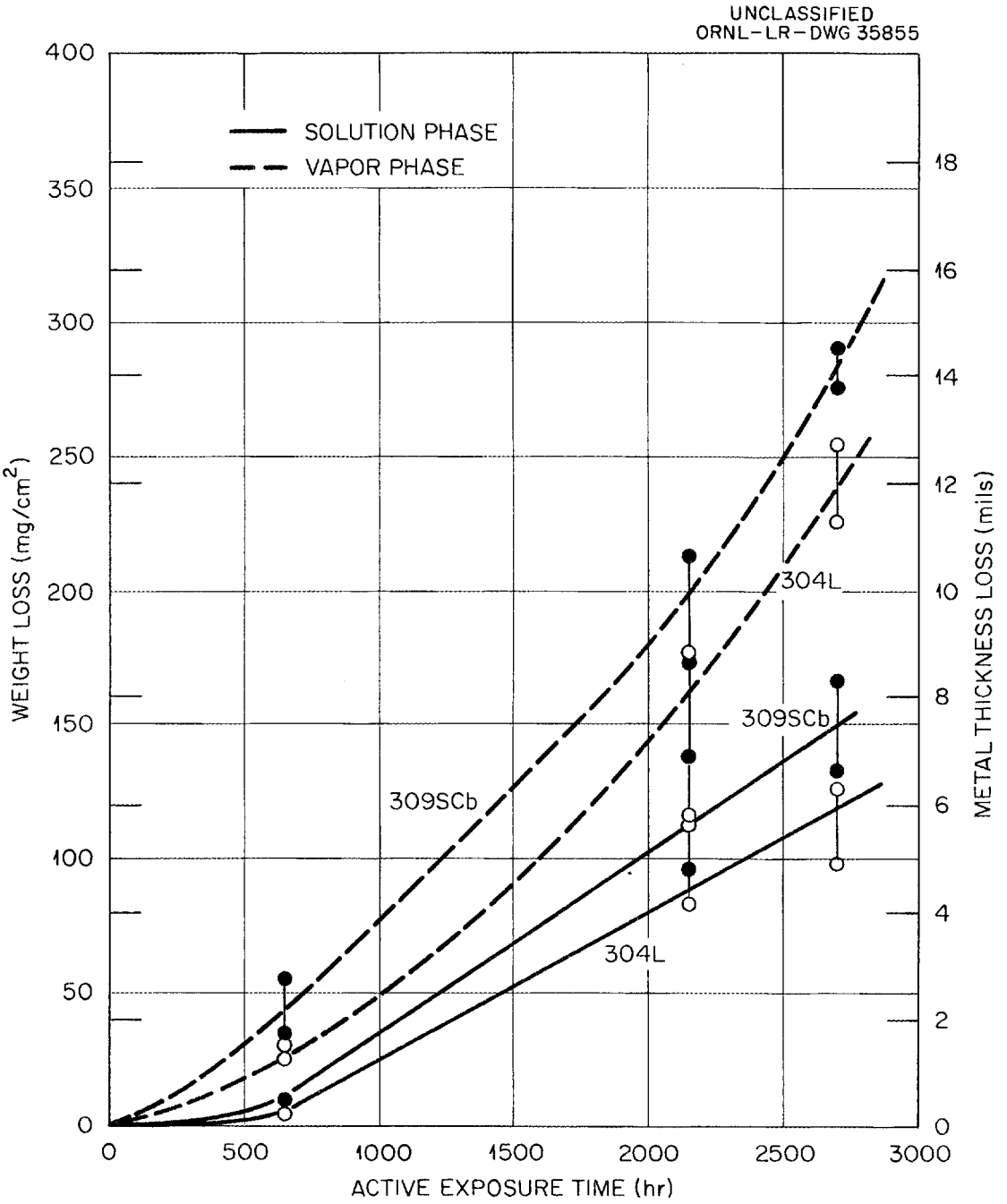


Fig. 24. Corrosion of Types 304L and 309SCB Stainless Steels in Feed Adjustment Tank During Thorex Pilot Plant Operation Between December 15, 1954 and November 10, 1955.

Nature of the Attack. Corrosion damage on the two stainless alloys resulted from metal reactions with acidic process solutions or with acid-rich vapors above the process solutions. In most cases, the attack on flat surfaces of specimens of the alloys was characterized by uniform metal removal. Some preferential grain boundary attack was experienced in the more corrosive environments; the depth of penetration was usually between 1 to 2 mils. However, in a few cases, grain boundary attack up to 5 mils in depth was observed. Edge surfaces of the stainless steel specimens underwent pitting and intergranular attack, the severity of which was related to the corrosivity of the particular environment.

No quantitative corrosion data were obtained for weld-metal deposits in the Thorex pilot plant. Generally, visual examination disclosed the condition of weld metal to be equal to or better than that of the respective parent metal. In the more severely corrosive environments, however, weld-metal deposits of both types 308L and 309SCb stainless steel were subject to a deep, channeling type of attack that was not characteristic of the attack observed on the respective parent metals.

Frequent reference has been made in this report to a preferential corrosion attack in the heat-affected zone adjacent to weld metal in both alloys. This type of attack, when associated with stabilized grades of austenitic stainless steels, is called knife-line corrosion because the attack is confined to a very narrow band of metal in the heat-affected zone. Knife-line corrosion as such is not common to the unstabilized grades of austenitic stainless steels. However, in the present investigation, random observations of a preferential attack in the heat-affected zone immediately adjacent to weld metal deposits in type 304L stainless steel were made. The attack was not greater than 5 to 10 mils in depth. Holzworth and co-workers have proposed a mechanism for knife-line attack in stabilized austenitic stainless steels[20] but no mechanism has been proposed for an analagous form of the attack in unstabilized austenitic stainless steels. However, in view of the random nature of the attack that was observed on the type 304L stainless steel weld specimens, it is conceivable that faulty welding procedures were responsible for its occurrence.

It has been stated that corrosion rates on the order of 25 mpy would not be considered untenable in the present Thorex pilot plant.[28] The present investigation has demonstrated that several environments in the Thorex pilot plant are capable of incurring attack near to or in excess of this rate. For example, maximum corrosion rates observed in the solution phase of the feed adjustment tank were 22 and 28 mpy, respectively, for types 304L and 309SCb stainless steel. A maximum rate of 47 mpy was obtained for type 347 stainless steel in the BT vapor separator. And, finally, maximum vapor-phase corrosion rates of 40 mpy for type 304L stainless steel and 46 mpy for type 309SCb stainless steel were experienced in the feed adjustment tank.

The feed adjustment tank in the pilot plant is fabricated from 1/2-in.-thick type 309SCb stainless steel plate. For a maximum corrosion rate of 46 mpy during a 2697-hr exposure period, and assuming uniform corrosion, the estimated half-life of the tank would be five years. This time is made shorter, however, by consideration of the localized forms of corrosion observed on specimens. Allowing an extra total penetration of 10 mpy for intergranular corrosion and preferential attack at weld-parent metal interfaces, the half-life would be reduced to four years of operation.

Since the issuance of the present report, additional stainless steel assemblies have been removed from the pilot plant for evaluation of corrosion damage. The results of this evaluation will be summarized in a supplementary report.

8. SUMMARY

Certain conclusions were suggested by analysis of corrosion data obtained during the production period and the first half of the development period of Thorex pilot plant operation. Since the data are representative of only the preliminary phases of the test program, subsequent information from other phases of the program may alter the present conclusions. The conclusions are as follows:

(a) Types 304L and 309SCb stainless steel generally exhibited comparable corrosion resistance in all environments. In those environments where one alloy was more corrosion-resistant, the difference was not pronounced.

(b) Neither type 304L nor type 309SCb stainless steel, initially stressed to 35,000 psi, were subject to stress-corrosion cracking in any of the environments.

(c) The area of most severe corrosion attack was the vapor phase of the feed adjustment tank operated at 155°C maximum. Types 304L and 309SCb stainless steel corroded at rates of 37.8 ± 2.0 and 45.1 ± 0.7 mpy, respectively.

(d) The second most serious area of attack was the BT vapor separator at 115°C maximum. The corrosion rates for types 304L and 347 stainless steel were 10.5 ± 3.7 and 33.2 ± 13.8 mpy, respectively.

(e) The highest solution corrosion rates, observed in the feed adjustment tank during the development period, were 19.6 ± 2.8 and 24.9 ± 3.0 mpy, respectively, for types 304L and 309SCb stainless steel.

(f) Observed corrosion rates in the batch dissolver tank were 8 mpy or less for types 304L and 309SCb stainless steel during the production period. Corrosion attack on the two alloys in the AP catch tank was almost negligible during this period.

(g) Attack on flat surfaces of types 304L and 309SCb was characterized for the most part by uniform metal removal. In the most corrosive environments, mild intergranular attack was observed.

(h) Edge-corrosion attack, typified by intergranular penetration and pitting, was experienced by both alloys. The intensity of the attack was dependent on the corrosivity of the environment. The results of laboratory studies suggested that edge corrosion did not contribute significantly to the total attack on pilot plant specimens.

(i) Preferential corrosion in heat-affected zones adjacent to weld deposits occurred randomly on both types 304L and 309SCb stainless steel in a number of the environments. The maximum penetration by the attack was 10 mils.

(j) Both types 304L and 309SCb stainless steel were insensitive to crevice corrosion attack in all environments. Some crevice corrosion was experienced by type 347 stainless steel exposed in the BT vapor separator.

(k) Suggested reasons for high vapor-phase corrosion rates as compared with the solution-phase rates in the feed adjustment tank were higher nitric acid concentration and/or uncomplexed fluorides in the vapor phase.

(l) Based upon the highest corrosion rate observed in the vapor phase of the feed adjustment tank, 46 mpy, and allowing 10 mpy excess for preferential forms of corrosion, the operating half-life of the 1/2-in.-thick type 309SCb stainless steel vessel was estimated to be four years.

9. ACKNOWLEDGMENTS

The author wishes to express his appreciation to J. W. Landry, G. S. Sadowski, and E. M. Shank of the Chemical Technology Division for their assistance in installation and removal of corrosion specimens from the pilot plant and for their maintaining complete exposure histories for specimens.

In addition, appreciation is due C. M. Boyd of the ORNL Metallography Group for metallographic assistance and to L. L. Fairchild and H. L. Barker for assistance in the evaluation of corrosion damage on specimens.

10. REFERENCES

1. A. C. Jealous, Meetings on Pilot Plant Samples, Corrosion Program and Provisions for Decontamination, ORNL CF-53-6-113, June 13, 1953.

2. J. W. Landry, Mounting of Corrosion Specimens in Thorex Tanks, memorandum to W. K. Jackson, September 15, 1954.

3. J. W. Landry, Corrosion Specimens Exposed in Thorex Pilot Plant for Production Runs, memorandum to J. L. English, August 18, 1955.

4. J. W. Landry, Thorex S-2 Corrosion Specimens, memorandum to J. L. English, November 17, 1955.

5. A. T. Gresky et al., Laboratory Development of the Thorex Process, ORNL-1367, January 2, 1953.

6. C. D. Hylton et al., Separation of U^{233} in the ORNL Pilot Plant, ORNL-1425, December 30, 1952.

7. A. T. Gresky et al., Laboratory Development of the Thorex Process, ORNL-1518, January 31, 1953.

8. A. C. Jealous, Thorex Information Meeting Summary, ORNL CF-54-11-145, November 26, 1954.

9. W. T. McDuffee et al., Thorex Pilot Plant Run HD-8 Summary, ORNL CF-55-8-180, August 5, 1955.

10. A. J. Pecquet et al., Thorex Pilot Plant: Evaluation of Equipment Performance for the Period October, 1954 through March, 1955, ORNL CF-54-11-145, November 14, 1955.

11. A. T. Gresky, Thorex Process Summary, ORNL CF-56-2-157, April 25, 1956.

12. F. A. Champion, Corrosion Testing Procedures, John Wiley and Sons, Inc., New York, 1952, pp 147-149.

13. M. A. Scheil, Some Observations on Stress-Corrosion Cracking in Austenitic Stainless Alloys, Symposium on Stress-Corrosion Cracking of Metals, ASTM, 1945, pp 396-398.

14. Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels, ASTM Designation: A262-55T, ASTM Standards, Part I, Ferrous Metals, 1955, pp 1108-1118.

15. H. H. Uhlig, Corrosion Handbook, John Wiley and Sons, Inc., New York, 1948, pp 1079-1080.

16. J. L. English, Corrosion Studies for the Thorex Process, ORNL CF-53-3-246, March 27, 1953.

17. R. P. Wischow, Thorium Slug Dissolution and Feed Preparation, ORNL CF-54-11-145, November 26, 1954, pp 18-21.

18. R. H. Winget, Thorex Pilot Plant: Production of U^{233} from Hanford Irradiated Thorium Slugs, ORNL CF-55-4-76, April 13, 1955.

19. Reference 8, p 21.

20. M. L. Holzworth et al., "The Mechanism of Knife-Line Attack in Welded Type 347 Stainless Steel," Corrosion, 7, December, 1951, pp 441-449.

21. Reference 8, pp 25, 65.

22. J. A. Truman, "Factors Affecting the Testing of Stainless Steels in Boiling Concentrated Nitric Acid," Journal of Applied Chemistry, May 4, 1954, pp 273-283.

23. W. B. DeLong, Testing Multiple Specimens of Stainless Steels in a Modified Boiling Nitric Acid Test Apparatus, Symposium on Evaluation Tests for Stainless Steels, ASTM Publication No. 93, 1950, pp 211-214.

24. F. H. Beck and M. G. Fontana, "Corrosion by Aqueous Solutions at Elevated Temperatures and Pressures," Corrosion, 9, August, 1953, pp 287-293.

25. R. F. Miller et al., "Corrosion by Acids at High Temperatures," Corrosion, 10, January, 1954, pp 7-12.

26. Reference 11, p 70.

27. R. J. Bendurc, "Contamination of Nitric Acid as a Source of Error in the Huey Boiling Nitric Acid Test," Corrosion, 10, March, 1954, p 1.

28. Reference 8, p 66.

ORNL-2673
Chemistry-General
TID-4500 (14th Ed.)

INTERNAL DISTRIBUTION

1. D. S. Billington
2. F. F. Blankenship
3. E. P. Blizard
4. A. L. Boch
5. C. J. Borkowski
6. G. E. Boyd
7. E. J. Breeding
8. R. B. Briggs
9. C. E. Center (K-25)
10. R. A. Charpie
11. F. L. Culler
12. L. B. Emlet (K-25)
13. D. E. Ferguson
14. A. P. Fraas
15. J. H. Frye, Jr.
16. W. R. Grimes
17. E. Guth
18. C. S. Harrill
19. H. W. Hoffman
20. A. Hollaender
21. A. S. Householder
22. W. H. Jordan
23. G. W. Keilholtz
24. M. T. Kelley
25. J. A. Lane
26. R. S. Livingston
27. H. G. MacPherson
28. W. D. Manly
29. J. R. McNally
30. K. Z. Morgan
31. J. P. Murray (Y-12)
32. M. L. Nelson
33. A. M. Perry
34. H. W. Savage
35. A. W. Savolainen
36. H. E. Seagren
37. E. D. Shipley
38. M. J. Skinner
39. A. H. Snell
40. J. A. Swartout
41. E. H. Taylor
42. A. M. Weinberg
43. C. E. Winters
44. R. E. Blanco
45. E. G. Bohlmann
46. J. C. Bresee
47. F. R. Bruce
48. W. E. Clark
49. E. L. Compere
50. W. K. Eister
51-55. J. L. English
56. L. L. Fairchild
57. H. E. Goeller
58. A. T. Gresky
59. J. C. Griess
60. D. N. Hess
61. J. W. Landry
62. W. H. Lewis
63. R. B. Lindauer
64. R. N. Lyon
65. R. J. McNamee
66. P. D. Neumann
67. A. R. Olsen
68. R. H. Rainey
69. G. W. Sadowski
70-82. E. M. Shank
83. E. S. Snavely, Jr.
84. W. E. Unger
85. C. D. Watson
86. W. R. Winsbro
87. Biology Library
88. Health Physics Library
89. Reactor Experimental Engineering Library
90-91. Central Research Library
92-109. Laboratory Records Department
110. Laboratory Records, ORNL R.C.
111. ORNL - Y-12 Technical Library Document Reference Section
112. C. P. Keim

EXTERNAL DISTRIBUTION

113. D. H. Groelsema, AEC, Washington
114. Division of Research and Development, AEC, ORO
115-676. Given distribution as shown in TID-4500 (14th ed.) under Chemistry-General category (75 copies - OTS)