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SM-1 REACTOR VESSEL
CLOSURE STUD INVESTIGATION

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ABSTRACT

This report presents metallurgical analyses and corrosion tests conducted to determine causes of failure of two SM-1 reactor vessel studs due to fracture in the threaded areas, after 23 months' operation (March 1959).

Conclusions of corrosion tests are: (1) failure of the studs was caused by stress corrosion cracking, (2) contributing factors to failure of the studs were improper control of heat treatment of the studs and absence of treatment for stress relief after thread grinding operation.

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SUMMARY

This investigation covers the failure analysis of two SM-1 reactor vessel studs which fractured in the threaded areas after the reactor has operated 23 months. In addition to the two fractured studs, 12 of the remaining 14 studs were found to contain cracks of varying degrees of severity.

Results of this examination showed that all the failures were the result of stress corrosion cracking, and that the incidence of cracking was affected by the hardness of the studs. Contributing factors to failure of the studs were improper control of heat treatment of the studs and lack of treatment for stress relief after the thread grinding operation. Corrosion tests were conducted on specimens of Type 410 stainless steel taken from the actual SM-1 studs. In these tests, the cracking which occurred in the studs was duplicated and the effect of hardness substantiated.

To prevent recurrence of these failures, recommendations are made that studs be tempered to a hardness not to exceed Rockwell "C" 21 (235 B.H.N.) and that studs be stress relieved after the thread grinding operation.

1.0 INTRODUCTION

In March 1959, during inspection of the SM-1 reactor vessel, it was found that failure had occurred in two of the Type 410 stainless steel studs used to bolt the cover to the vessel body. This inspection was made after the reactor had been in operation for 23 months. The two failed studs and the remaining 14 studs were sent to the Metallurgical Laboratory in Schenectady for examination.

Alco was authorized under Item 1.2 of the Program Plan for Engineering Support and Development of Army PWR Power Plants,^{*} to conduct a failure analysis of the broken reactor vessel studs. The primary objective of this investigation was to determine the cause for failure of these studs and to recommend remedial action required to prevent recurrence of the problem. In addition, if the results of this investigation showed that Type 410 stainless steel was not suitable for this application, possible substitute alloys would be recommended. In conjunction with this program, the corrosion problems associated with the female threads in the carbon steel body would be studied.

* AP Note 286, October 10, 1960

2,0 HISTORY OF THE SM-1 REACTOR VESSEL STUDS

Type 410 stainless steel, then a non-code approved material, was selected for the reactor vessel studs. The reason for this choice is contained in APAE-10 (Volume 1), dated August 15, 1956, and is described as follows:

"A high strength rust resistant material with favorable expansion characteristics is needed to facilitate removal of the nuts when required for fuel changes, since the studs and nuts are submerged in water during reactor operation. Type 416 stainless steel, a code approved bolting material, meets all requirements except that it may suffer excessive damage from irradiation. As a free machining steel, it can contain over one-half percent of a free machining element such as sulphur. The presence of sulphur in this quantity tends to promote embrittlement under irradiation. Definite data to prove that Type 416, due to its high sulphur content, will suffer excessive radiation damage are lacking, but this phenomenon is observed in carbon and low-alloy steels, and it is reasonable to expect similar behavior for a martensitic stainless steel with high sulphur content. Type 410 stainless steel, the non-free machining equivalent of Type 416 has practically the same analysis and physical properties except that sulphur is in the range normal for quality steels. Approval for the use of 410 has been obtained."

The first lot of 18 studs was shipped by the Erie Bolt and Nut Company on June 8, 1956. These 18 studs were made from three heats of Type 410 stainless steel obtained from Industrial Steels, Incorporated, and identified on the test certificate as follows:*

HEAT NO. 55031

Order:	D-83463		
Date Shipped:	2-15-56		
Grade:	410		
Material:	1 Bar - 2-7/8" dia., 13'1" - 320#		
Condition:	H. R. H. T.		
Carbon	.098%		
Manganese	.47		
Phosphorus	.020		
Sulphur	.022		
Silicon	.26		
Chromium	12.61	Tensile Strength, psi	150,000
Nickel	.44	Yield Strength, psi	115,500
Molybdenum	.07	Elongation, %	18
copper	.14	Reduction of Area, %	55
Tin	.014	Hardness-BRINELL	302-320
Aluminum	.02	Hardenability -ROCKWELL C	43

HEAT NO. 45947

Order:	D-83463
Date Shipped:	3-26-56
Grade:	410
Material:	1 Bar - 2-7/8" dia. -325#
Condition:	H. R. H. T.

*The data given below were taken verbatim from the test certificates.

Carbon	.096%		
Manganese	.43		
Phosphorous	.017		
Sulphur	.014		
Silicon	.32		
Chromium	12.48	Tensile Strength, psi	145,000
Nickel	.19	Yield Strength, psi	114,000
Molybdenum	.06	Elongation, %	20
Copper	.10	Reduction of Area, %	59
Tin	.009	Hardness- BRINELL	269-321
Aluminum	.01	Hardenability-ROCKWELL C	41

HEAT NO. 54471

Order: D-84981
 Date Shipped: 5-7-56
 Grade: 410
 Material: 1 Bar - 3" round, 40" long - 80#
 Condition: Centerless ground - heat treated.

Carbon	.090%		
Manganese	.44		
Phosphorus	.018		
Sulphur	.017		
Silicon	.32		
Chromium	12.62		
Nickel	.18		
Molybdenum	.09		
Copper	.16		
Tin	.016	Hardness - BRINELL -	321-340
Aluminum	.01	Hardenability -ROCKWELL C	42

The test report from the Erie Bolt & Nut Company for the 18 studs made from these three heats of steel contained the following information:

Order No. - D-84415
Specification - A-276-55
Drawing No. - D-150176 Rev. B

Tensile Strength, psi 118,000
Elastic Limit, psi 101,000
Elongation, % 20.5
Reduced Section 68.1
BRINELL 262

The second lot of 16 studs was shipped by the Erie Bolt and Nut Company on October 19, 1956. These 16 studs were made from one heat of Type 410 stainless steel obtained from McInnes Steel Company, and identified on the test certificate as follows:

HEAT NO. 55484

Order No. D-86736
Date Shipped 9-4-56
Type: 410 Stainless
Size: 2-7/8" Dia. x 29' 9-1/2" -675#

Carbon .110%
Manganese .47
Phosphorus .027
Sulphur .022
Silicon .45

HEAT NO. 55484 (cont'd)

Chromium	12.08	Yield Strength, psi	81,500
Nickel	.06	Ultimate Strength, psi	102,500
Molybdenum	.02	Elongation in 2", %	25.0
Copper	.07	Reduction of Area, %	67.0
Aluminum	.011		
Tin	.013		

The test report from the Erie Bolt & Nut Company for the 16 studs made from this heat of steel contained the following information:

Order No. - D-86737
Specification - 4 C.T.R.
Drawing No. - C-150176 Rev. D

Tensile Strength, psi - 142,500
Elastic Limit, psi - 130,000
Elongation, % - 20.0
BRINELL - 270.5

Also Drawing D-150176, Rev. D, specifies the following:

"Material - 410 s.s., A-276-55, Type 410 (or Equal).

"Threads (both ends) to be milled or ground
after heat treatment.

"Austenitize 1750 - 1800 F, hold one-half hour at
temperature, oil quench, temper 262 Brinell
(1000 F min.) for 3 hours. Bright harden-
ing required.

"Physical Properties after heat treating (tempering):

Tensile Strength, psi - 100,000
Yield Point, psi - 80,000

The 16 studs from the McInnes Steel Company were indicated as "replacement studs on the Erie test report, while the 18 studs from Industrial Steels were indicated as "original" studs.

It was reported that two studs (identified as Nos 4 and 13) had been galled during application of the original set of studs and that these had been replaced by two studs from the second lot (Order Number D-86736). (These two replacement studs, which had no identifying numbers, were the two failed studs which were subsequently submitted on March 30, 1959, to the Metallurgical Laboratory for examination).

Additional information concerning the reactor studs was obtained from Mr. G. P. Brown's letter of March 30, 1959 to Dr. D. Foley:

"We have advised that all our inspection records for this order have been destroyed in a general housecleaning operation. I have talked with Mr. Gunther, who personally inspected the studs at Erie. He has advised the threads were ground in a small thread grinder and inspected on a comparator at Erie. Dimensional checks, using pitch micrometers, and visual inspection only were made by Dunkirk. In addition, on receipt of the studs, they were checked in a lathe for proper lead. In addition, our bill of material specified a one shot X-ray and magnaflux of the center unthreaded portion of the studs. this we are sure was performed satisfactorily in Dunkirk.

"From our Industrial Engineering Department, plus our job file, I find that Dunkirk broke one stud on the initial run using a test block. From this, it was concluded that Noelube must be used in the block and Molycoat on the nuts. Also all studs and nuts were match marked in sets for positive identification.

"By copy of Mr. Church's memo to Mr. Knighton, dated October 9, 1956, it was confirmed that stud and nut combinations, Nos. Part 1, 2, 3, 4, 6, 7, and 9, were made up without benefit of Molycoat lubricant and the studs and nuts galled. These studs and nuts were cleaned up and shipped with the vessel. It was also agreed that these eight stud and nut combinations would not be used in the vessel after installation.

"Our Industrial Engineering Department also advises that the bolt holes in the reactor flange were machined on a G. & L. horizontal boring mill using a rough and a finished tap with all machining done dry. We believe that the nuts were also made using the same general procedure."

2.1. WATER TREATMENT AND STRESS ANALYSIS

The reactor vessel serves the purpose of containing the active core, its supporting structure, and the thermal shielding. The basic material of construction is A-212 carbon steel plate clad with 1/4 in. Type 304 stainless steel. The upper end of the vessel is closed by a bolted and gasketed dished cover, 2-3/4 in. thick, made of A-212 Grade B carbon steel clad with Type 304 stainless steel. The 2-3/4 in. hexagonal nuts were made from Type 304 stainless steel. The bolt holes in the cover were nickel plated.

Complete details of the water treatment are described in APAE-10 (Vol. 1) issued August 15, 1956, and in APAE No. 55, issued January 13, 1960. Fort Belvoir supplied the results of an analysis of the inner shield tank water, which reportedly was made on March 9, 1959, prior to removal of the pressure vessel lid. The results of this analysis (Ref: Mr. R. V. Lichtenberger's letter of November 23, 1960 to Mr. G. A. Young), are as follows:

pH: 7.4
Cl: .28 ppm
Resistivity 100,000 ohm-cm.
Hardness: none
Crud: 1 ppm
O₂: no sample taken
Activity: 25×10^{-6} on March 2, 1959.

The original calculations for the total stress on the studs yield the following results:

Total Stress on Stud at Nut:

4,570 psi - Due to cover thermal expansion
16,500 psi - Due to torque loading
12,690 psi - Due to nut thermal expansion
33,760 psi

Total Stress on Shank of Stud:

4,570 psi - Due to cover thermal expansion
16,500 psi - Due to torque loading
21,070 psi

Total Stress on Stud at Vessel:

4,570 psi - Due to cover thermal expansion
16,500 psi - Due to torque loading
7,610 psi - Due to vessel thermal expansion
28,680 psi

At 450°F, tensile strength of 410 stainless steel - 87,500
psi maximum allowable stress - $\frac{1}{3} \times \text{Tensile Strength} = \frac{87,500}{3}$
= 29,200 psi

The original estimated temperature at various locations were as follows:

<u>Location</u>	<u>Temperature, °F</u>
At stud in vessel -	250
At nut end of stud -	150

The following information was obtained from the stress analysis investigation (4) just completed.

The stud stresses were calculated for three conditions:

A - Reactor unpressurized. Studs tightened as called for by ASME Unfired Pressure Vessel Code, paragraph UA47

$$\text{Total Load } \frac{\pi}{4} G^2 \rho + 2 \pi b G m p, \text{ for } \rho = 1200 \text{ psi.}$$

B - Pressure raised to 1200 psi. Stud setting unchanged. Unit at 125°F.

C - Steady state thermal stresses with pressurized water at 1200 psi, 420°F. Stud setting unchanged.

Stud tension and stresses for these three conditions are tabulated below:

TABLE 1. STUD TENSION AND STRESSES FOR CASES A, B AND C

STUD TENSION

	<u>lb/Bolt</u>	<u>lb/Inch of Stud Circle</u>
A	63,500	9,244
B	68,200	9,920
C	100,000	14,560

TABLE 1 (cont'd)

STUD STRESSES PSI IN SECTION AT POINT "E" (REF. FIG. 1)
 (TOP THREADED PORTION)

Loading	Direct	Bending	Left	Combined at	
				Left	Right
Case A	12590T	2,800	9,790T		15,390 T
Case B	13500T	2,040	11,460T		15,540 T
Case C	19800T	2,760	22,560T		17,040 T

STUD STRESSES PSI IN SECTION AT POINT "F" (REF. FIG. 1)
 (BOTTOM THREADED PORTION)

Loading	Direct	Bending	Left	Combined at	
				Left	Right
Case A	12590T	6,450	19,040 T		6,140 T
Case B	13500T	4,570	18,070 T		8,930 T
Case C	19800T	7,380	12,420 T		27,180 T

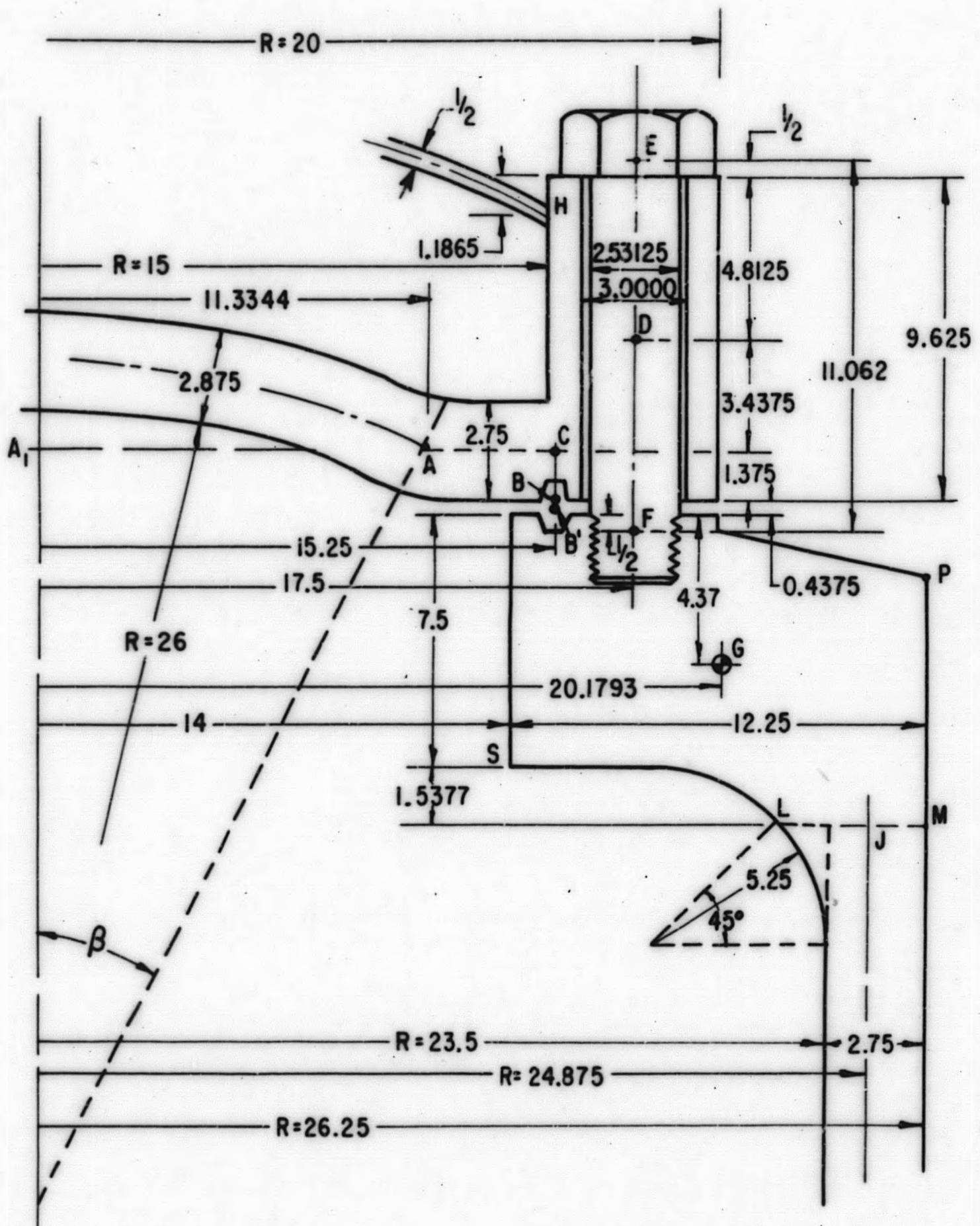


Figure 1. SM-1 Reactor Vessel Cover, Flange and Stud Region

3.0 CHEMICAL ANALYSIS

3.1 STUD MATERIAL

A total of 18 studs from the SM-1 were submitted to the Metallurgical Laboratory for examination. Two of these studs, numbered 4 and 13, had been galled during application of the original set and were replaced by two others, which subsequently failed. The two failed studs had no identifying numbers and so were labeled as Laboratory Samples A and B. The remaining 14 studs were numbered, 0, 1, 2, 3, 5, 6, 7, 8, 9, 10, 11, 12, 14, and 15.

Chemical analyses for samples 4, 13, and A were determined; the results are as follows:

	<u>Stud 4</u>	<u>Stud 13</u>	<u>Sample A</u>	<u>AISI 410</u>
Carbon	.12%	.10%	.12%	.15% Max.
Manganese	.45	.50	.65	1.00 "
Phosphorus	.02	.02	.01	.04 "
Sulphur	.02	.02	.02	.03 "
Silicon	.30	.31	.55	1.00 "
Chromium	12.34	12.75	12.12	11.50-13.50

The three studs conformed satisfactorily to the chemical composition limits for AISI 410 stainless steel.

3.2 CORROSION PRODUCT

Chemical analyses were made of the deposit which was found in the threaded areas of the studs removed from the reactor vessel. Since the deposit on the body end (threaded area of the stud which engaged the vessel body) was different in appearance from the deposit at the nut end, separate analyses were performed on each type. The results of these analyses are as follows:

Body End Deposit

Ferric Oxide (Fe_2O_3)	- 13%
Nickel Oxide (NiO)	- 30%
Manganese Oxide (MnO)	- 1%
Calcium Oxide (CaO)	- 5.5%
Silica (SiO_2)	- 5%
Zinc Oxide (ZnO)	- 10%
Chromic Oxide (Cr_2O_3)	- 0.5%
Aluminum Oxide (Al_2O_3)	- 0.3%
Copper Oxide (CuO)	- 0.9%
Sodium, Potassium & Lithium	Nil
Chlorides (Cl)	- Nil
Sulfates (SO_4)	- Nil
Borates (BO_4)	- Nil
Phosphorus Pentoxide (P_2O_5)	0.5%
Carbonates (CO_3)	- trace
Ignition loss	- 22%

(The ignition loss includes moisture and carbon graphite).

Nut End Deposit

Ferric Oxide (Fe_2O_3)	- 82.5%
Silica (SiO_2)	- .5%
Chromium Oxide (Cr_2O_3)	- 5.4%
Manganese Oxide (MnO)	- .5%
Chlorides (Cl)	- Nil
Sulfates (SO_4)	- Nil

4.0 MAGNETIC PARTICLE INSPECTION

Of the 16 original SM-1 reactor studs submitted to the metallurgical Laboratory for examination, two had fractured transversely in the threaded areas. These two studs were not numbered and so were identified as Laboratory Samples A and B. Sample A had fractured at the nut end (the end which engaged the Type 304 stainless steel nut) close to the last engaged thread. Sample B fractured at the body end (the end which engaged the vessel body) close to the last engaged thread. The fracture surfaces of both studs were coated with a corrosion product, as were the threaded areas of the studs. Numerous longitudinal cracks, ranging from 1/2 to 2 in. in length and up to 2 in. in depth were found in the threads of both the failed studs. In addition, circumferential cracks were found in the roots of the threads. All cracks were confined to the threaded areas; there was no evidence of cracking in the stud bodies.

Magnetic particle inspection of the remaining 14 studs revealed that all contained longitudinal indications to some extent. These indications were restricted to areas on the thread flanks, except for studs 7 and 10, where several longitudinal indications continued over the crown of one thread. The majority of longitudinal indications were located in areas at (and adjacent to) the last engaged thread in both the body end and nut end of the studs.

Twelve of the 14 studs also contained circumferential cracks, which were located, in most cases, at (or adjacent to) the last engaged thread in the body end and/or nut end of the stud, where the longitudinal indications predominated. Studs 0 and 1 were the two which showed no evidence of circumferential indications, but did contain slight longitudinal indications. To determine if

these indications were actually cracks, Stud 1 was sectioned and examined metallographically. The results of this examination revealed that the indications were not cracks, but rather were a pitting-type corrosive attack of the thread flank surface.

A magnetic particle inspection of the two galled studs, numbered 4 and 13, did not reveal any evidence of cracking, but did reveal indications of short stringer-type non-metallic inclusions in the shank areas.

Detailed results (at specific hardness values) of the magnetic particle inspection of 14 of the original SM-1 reactor studs are shown in Table 2.

TABLE 2

RESULTS OF MAGNETIC PARTICLE INSPECTION OF 14 ORIGINAL
SM-1 REACTOR STUDS

<u>Stud*</u>	<u>Hardness Rockwell "C"</u>	<u>Indications</u>
8	32	Heavy longitudinal indications on thread flanks. Circumferential cracks on thread flanks.
5	30	Heavy longitudinal indications on thread flanks. Circumferential cracks on thread roots.
7	31	Heavy longitudinal indications on thread flanks. Circumferential cracks in roots of threads. Short longitudinal inclusions in shank and threaded areas.
3	27	Heavy longitudinal indications on thread flanks. Circumferential cracks in roots of threads. Short longitudinal inclusions in shank and threaded areas.
11	32	Heavy longitudinal indications on thread flanks. Circumferential cracks at run-out of thread area on shank. Short longitudinal inclusions in threaded area.
2	27	Longitudinal indications on thread flanks. Circumferential cracks in roots of threads. Short longitudinal inclusions in shank.
12	27	Longitudinal indications on thread flanks. Circumferential cracks in roots of threads. Short longitudinal inclusions in shank.
10	31	Longitudinal indications on thread flanks. Circumferential cracks in thread roots and run-out of thread area on shank.

TABLE 2 (cont'd)

<u>Stud*</u>	<u>Hardness Rockwell "C"</u>	<u>Indications</u>
14	32	Longitudinal indications on thread flanks. Circumferential cracks in thread roots. Short longitudinal inclusions in shank and threaded areas.
15	30	Longitudinal indications on thread flanks. Circumferential cracks in thread roots.
9	26	Light longitudinal indications on thread flanks. Circumferential cracks in roots of threads.
6	27	Light longitudinal indications on thread flanks. Circumferential cracks in thread roots.
1	22	Very light indications on thread flanks.
0	21	Very light indications on thread flanks.

* Studs are listed in decreasing order of severity of cracking or indications.

5.0 MECHANICAL PROPERTIES

Tensile tests, using standard 0.505 in. diam specimens were conducted on one of the original galled studs (Stud 4), on the two failed studs (A and B), and on the stud which contained the fewest indications on the magnetic particle inspection (Stud 0).

The results of the tensile tests are as follows:

	<u>Stud 4</u>	<u>Stud A</u>	<u>Stud B</u>	<u>Stud 0</u>
Tensile Strength,psi	139,000	170,500	165,000	95,000
Yield Point,psi	92,500	148,500	152,500	87,000
Elongation, %	20.0	18.5	17.5	20.5
Reduction of Area, %	62.1	65.6	66.1	69.5
Hardness Rockwell "C"	27	38	38	21

As noted earlier under Section 2.0, Alco Dwg. D-150176, Rev. D, specified a hardness of 262 B.H.N. (approximately equivalent to 26.5 Rockwell "C").

6.0 METALLOGRAPHIC EXAMINATION

A metallographic examination was made on cross sections of both failed studs (A and B), and on Studs 1 and 6. The magnetic particle inspection of Stud 1 revealed only very light longitudinal indications on the thread flanks. Inspection of Stud 6 showed light longitudinal indications on the thread flanks and circumferential cracks in the thread roots. Of the studs which contained circumferential cracks, the indications were the least severe in Stud 6 (see Table 2).

All the samples examined revealed a microstructure of tempered martensite. Studs A and B were examined metallographically to determine the nature of the longitudinal and circumferential indications. In all the areas observed, the indications were found to be intergranular cracks. A typical circumferential is shown in Fig. 2 at 250 x magnification.

An area of Stud 1 which showed slight longitudinal indications in the magnetic particle inspection, was also sectioned to determine whether the indications were actually cracks in the thread flanks. The examination revealed that the indications were not cracks but were evidence of a pitting-type corrosive attack of the thread flank surface.

The threaded area of Stud 6, which included a circumferential indication, was sectioned for examination. The indication proved to be an intergranular crack, similar to that found in the failed studs.

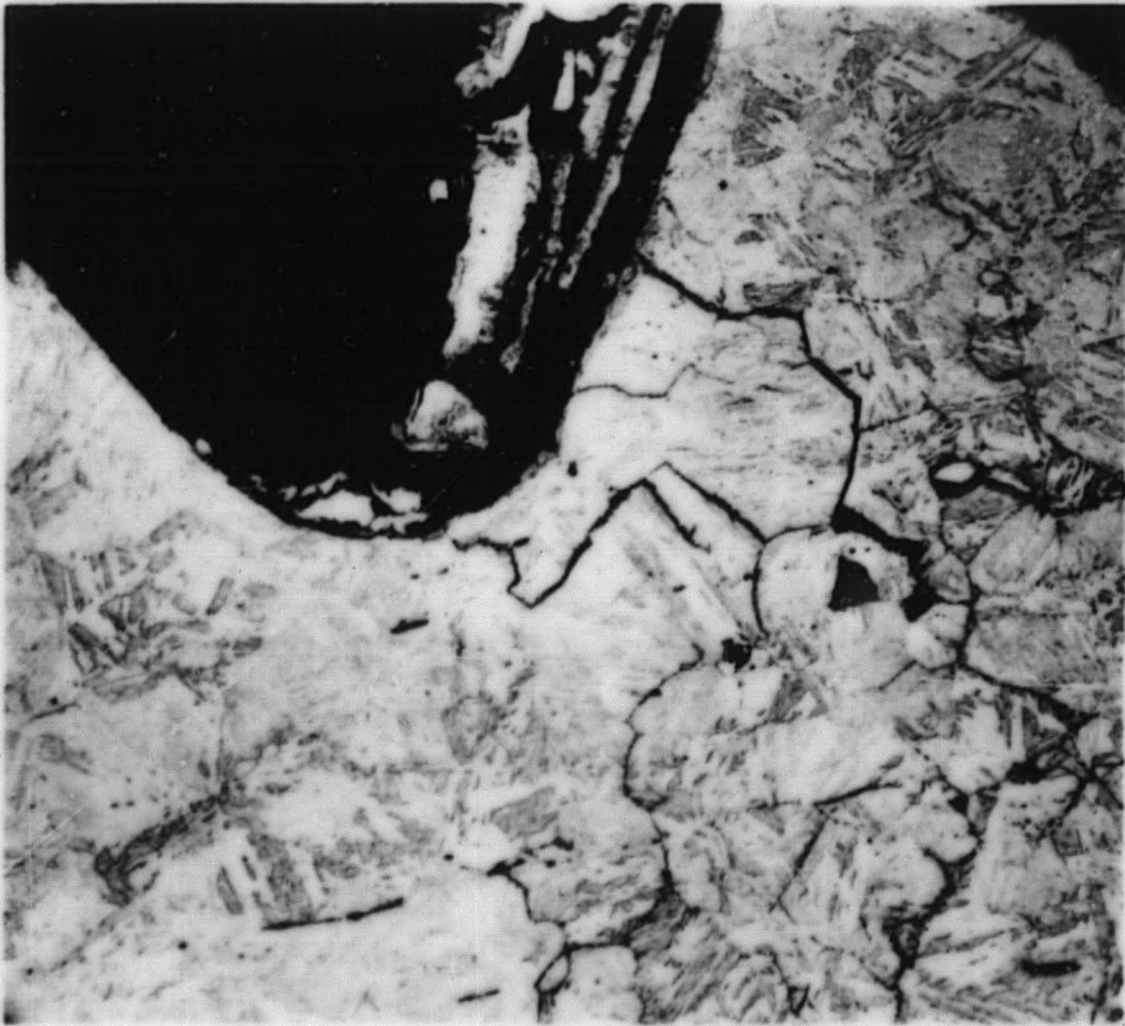


Figure 2. Intergranular Cracking in Thread of Failed SM-1 Reactor Vessel Stud "A". Rockwell Hardness C-38

7.0 CORROSION TESTING

7.1 CORROSION TEST PROCEDURE

A corrosion test apparatus (Fig. 3) was designed and assembled in an attempt to duplicate in the laboratory the type of failure experienced by the reactor studs.

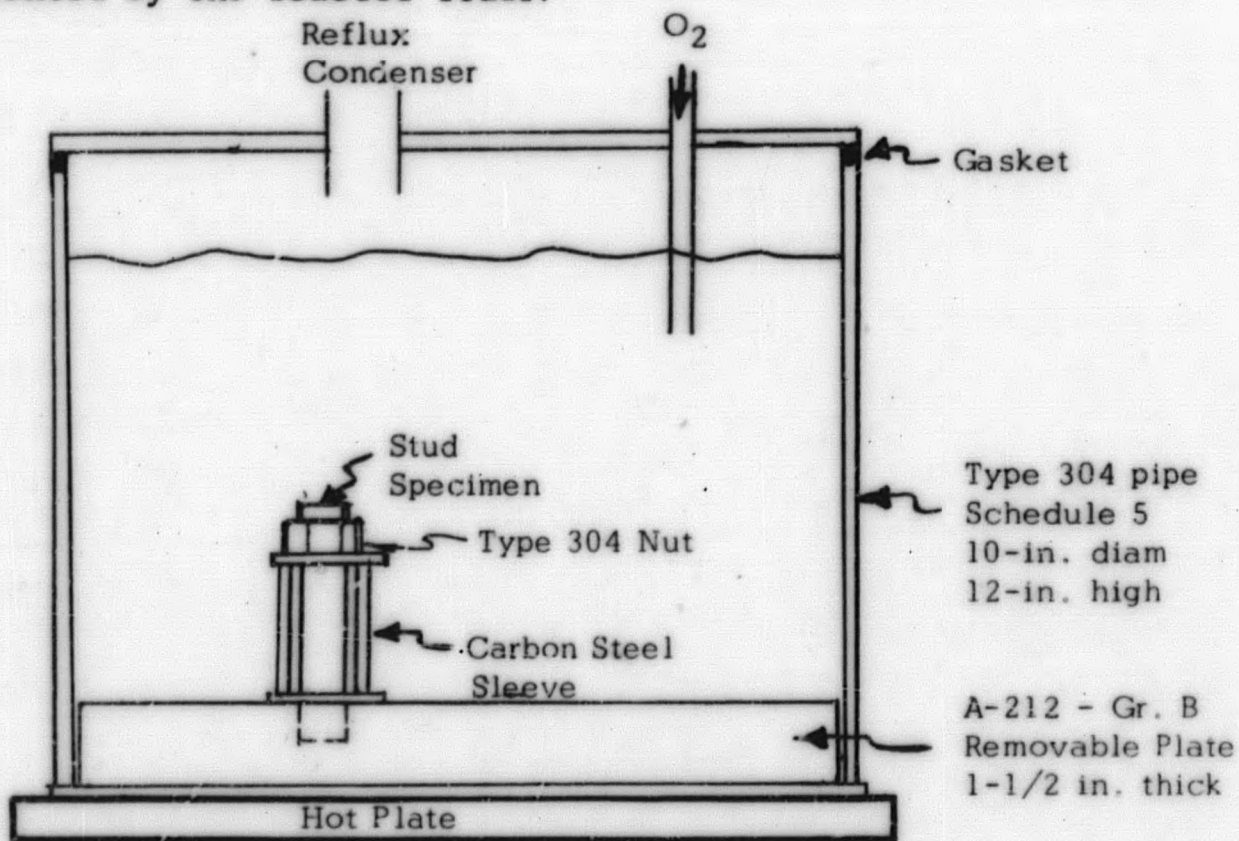


Figure 3 - CORROSION TEST APPARATUS

The carbon steel plate contained 13 tapped holes to accommodate the corrosion test specimens, which were miniature replicas of the reactor studs, scaled down to one-fourth of actual size. The dimensions of the specimens were as follows;

Length of Specimen	4.625 in.
Diameter of Specimen	0.6447 in.
Length of shank portion	2.3125 in.
Thread	3/4 by 16

By employing a carbon steel sleeve and a Type 304 stainless steel nut, it was possible to stress the specimens to any designated level. The stress was calculated by measuring with a height gauge the distance the stud specimen elongated.

The carbon steel plate was removable so that corrosion in the female threads could be studied. With the water at boiling temperature, the threaded areas in the carbon steel plate would be a somewhat higher temperature, duplicating more closely the conditions in the reactor vessel body.

The specimens were immersed in water having the following conditions:

1. Chloride Content 0.3 to 0.6 ppm
2. Resistivity 80,000 to 100,000 ohm-cm.
3. pH 6.5 to 7.5
4. Oxygen Saturated
5. Temperature of 212 F (boiling)

In order to determine what percentage of oxygen was present in the water at the test temperature, an apparatus was designed for obtaining a sample of the boiling water for oxygen analysis. The apparatus functions briefly as follows: Water is pumped from the vessel through a cooler and into a collecting flask. Ten volumes of water must pass through the flask before a representative sample can be taken. The water therefore must be delivered back to the vessel, in order to keep a constant volume of water in the vessel. To maintain boiling, the water which returns to the vessel is circulated through a tube which is resistance heated. A thermocouple attached to this tube, close to the point of re-entry into the vessel, records the temperature of the water. By adjusting the water flow rate, the temperature of the water returned to the vessel can be controlled.

Non-Referee Method A, described in A.S.T.M. Designation D-888, was used for the oxygen determination. While the first sample was being taken for analysis, the temperature of the water in the vessel varied from 208°F to 212°F. The results of this analysis showed a dissolved oxygen content of 2.5 ppm. An additional sample was taken where the temperature of the water in the vessel varied from 200°F to 208°F. The results of this analysis showed a dissolved oxygen content of 3.8 ppm.

The intention of the corrosion test was to evaluate three variables; hardness, stress level, and effect of thread fit. The four hardness levels were, Rockwell "C" 20, 26, 31, and 40, selected to bracket the range of hardnesses found in SM-1 studs.

The stress levels were 25,000, 30,000 and 58,000 psi. To determine the effect of thread fit, three of the specimens were threaded to a Class 2 (loose) fit, while the remaining specimens were threaded to a Class 3 fit.

7.2 INITIAL CORROSION TEST

The Type 410 stainless steel specimens were cut from the actual reactor studs. The 13 specimens used for the initial phase of the corrosion test were identified as follows:

<u>20 R_c</u>	<u>26 R_c</u>	<u>31 R_c</u>	<u>40 R_c</u>
1A	9E	11A	16C
1D	9G	11C	16E
1F		11E	16L
1L		11L	

Specimen 1A and 11A were stressed to 25,000 psi; all the remaining specimens were stressed to 30,000 psi. The specimens were threaded to a Class 3 fit, except for 1L, 11L, and 16L, which were threaded to a Class 2 (loose) fit. Specimen identified as Nos. 1, 9, and 11, were from studs having those numbers, whereas specimens identified as 16 were from Stud A, the fractured stud.

The specimens were machined and the threads ground with no additional heat treatment. The threaded areas of all the specimens were dipped in Neolube prior to stressing.

After the initial corrosion test had preceded a total of 1000 hr, the carbon steel plate was removed from the vessel. The elongation of each stud was measured to determine of the stresses which had been applied to studs at the start of the test had been maintained. The results of these measurements showed that all studs had remained at their initial applied stress level.

The female threads in the carbon steel plate were examined visually revealing that corrosive attack was negligible in all cases. No effect of thread fit on the corrosion of the female threads was evident.

Magnetic particle inspection of all stud specimens revealed no evidence of cracking. However, subsequent metallographic examination showed intergranular micro-cracks in specimen 16C (Rock C-40, 30,000 psi). Therefore it is presumed that specimens 16E and 16L were also cracked at this time.

7.3 FINAL CORROSION TEST

Thirteen stud specimens were again installed in the carbon steel plate for the second phase of the corrosion test. Specimen 1E from the first test was severely galled during removal from the carbon steel plate, and was therefore discarded. This galled specimen was replaced by a spare stud having the same hardness (Specimen 1C). Two other stud specimens (16C and 11E) were also replaced with new specimens (16F and 11B), having the same hardness. These three new studs were stressed to 58,000 psi., in an attempt to accelerate the test. Three of the original studs (11C, 16E and 1D), were also stressed to 58,000 psi. The remaining studs were stressed to their original levels, viz., 25,000 and 30,000 psi.

The identity of the specimens used for the second test were as follows:

<u>20 R_c</u>	<u>26 R_c</u>	<u>31 R_c</u>	<u>40 R_c</u>
1A (25,000 psi)	9E (30,000 psi)	11A (25,000 psi)	16E (58,000 psi)
1C (58,000 psi)	9G (30,000 psi)	11B (58,000 psi)	16F (58,000 psi)
1D (58,000 psi)		11C (58,000 psi)	16L (30,000 psi)
1L (30,000 psi)		11L (30,000 psi)	

When the second phase of the corrosion test had proceeded for 1000 hr, the carbon steel plate was removed from the vessel. Stud elongations were again measured and showed that all the specimens had remained at their initial applied stress level.

Visual examination of the female threads in the carbon steel plate again revealed no significant amount of corrosive attack. The variance in thread fit had no observable effect on the corrosion of the female threads.

The 13 stud specimens were magnetic particle inspected; no cracks were found. In order to verify the results of the magnetic particle inspection, a metallographic examination was made of stud 16E. Specimen 16E was selected for examination, since it was the stud most likely to be cracked, by virtue of its high hardness (40 R_c) and high stress level (1000 hrs. at 30,000 psi. plus 1000 hrs. at 58,000 psi). Longitudinal and transverse areas of both the nut end and the plate end of this specimen were examined. The results of the metallographic examination revealed that intergranular cracking had occurred circumferentially at the last engaged thread of the nut end.

Since magnetic particle inspection was apparently not suitable for detecting the presence of circumferential micro-cracks at the thread roots of the small diameter laboratory specimens, additional metallographic examination was conducted to determine which specimens had failed and the extent of the crack.

The results of the metallographic examination revealed that cracking had occurred in three of the following specimens:

Specimen No.	Rockwell "C" Hardness	Depth of Crack, in.	Testing Time hrs	psi	Total psi-hrs
16E	40	.080	1000 hr at &1000 " "	30,000 psi 58,000 "	8.8×10^7
16L	40	.048	2000 " "	30,000 "	6.0×10^7
16C	40	.030	1000 " "	30,000 "	3.0×10^7
11C	31	none	1000 " " &1000 " "	30,000 " 58,000 "	8.8×10^7
9E	26	none	2000 " "	58,000 "	6.0×10^7

All the cracks were intergranular and all were found at the last engaged thread on the nut end of the specimens. It can be seen from the above data, that the depth of cracking is roughly proportional to the time multiplied by the stress, at the Rockwell "C" hardness level.

Typical circumferential cracking similar to that observed in the reactor vessel studs is shown in Fig. 4.



Figure 4. Intergranular Cracking in Thread of Laboratory Specimens 16E
After 1000 Hr at 30000 psi and 1000 Hr at 58,000 psi.
Rockwell Hardness C-40

8.0 DISCUSSION OF RESULTS

The results of this investigation revealed that failure of the two SM-1 reactor vessel studs was caused by stress corrosion cracking. In addition, the cracks found in 12 of the 14 remaining studs removed from the reactor vessel were attributed to stress corrosion cracking.

The effect of hardness on the stress corrosion cracking susceptibility of the subject studs is clearly demonstrated in Table 2 (Results of the Magnetic Particle Inspection). Note that all studs having a hardness value of 26 or higher were cracked to some extent. The two studs (A and B) having the highest hardness values had fractured. Studs 0 and 1, which had the lowest hardness values, did not contain cracks. The relationship between hardness and the occurrence of cracking found for the SM-1 studs indicates that, for this application, Type 410 stainless steel is susceptible to stress corrosion cracking at hardness values of Rockwell "C" 26 (260 B.H.N.), and higher. On the other hand, Type 410 stainless steel studs having hardness values of Rockwell "C" 21 (235 B.H.N.) should be expected to perform satisfactorily.

The wide variation in hardness values found in the SM-1 studs indicated improper control of the heat treatment operation. The attainment of a specific hardness is admittedly difficult to achieve in Type 410 stainless steel due to the fact that, in tempering, small differences in temperature produce large changes in hardness. An application of the Holloman-Jaffe Parameter to the tempering of Type 410 stainless steel has been developed, which may be useful for choosing a suitable time-temperature cycle for obtaining a required hardness. ⁽¹⁾ Reference to this tempering chart indicates that Type 410 stainless steel should be tempered

for 3 hr at 1200°F to obtain a hardness of Rockwell "C" 22. The tempering temperature for the SM-1 reactor studs were reportedly 1000°F.

An investigation into the effect of tempering temperatures on the stress corrosion cracking of martensitic stainless steel was conducted by Lillys and Nehrenberg.⁽²⁾ Their results showed that, in an atmosphere of 5% NaCl, Type 410 stainless steel was susceptible to stress corrosion cracking when tempered in the range of 800 to 1000°F. Specimens which had been tempered at 900°F were found to be most susceptible.

These findings were substantiated by H. Suss in an investigation to determine the susceptibility of Type 410 stainless steel to stress corrosion cracking in high temperature, high purity water.⁽³⁾ The results of this investigation revealed that temperature above 1050°F tended to provide specimens of Type 410 with immunity to stress corrosion cracking. This immunity was attributed to changes in metallurgical structure, relief of residual stresses, and the nature of pit formation (increase in pit radii with increasing tempering temperature).

The cracking found in the SM-1 studs was identified as stress corrosion cracks after metallographic examination of the failed areas. The intergranular path and the discontinuous nature of the crack propagation were typical of stress corrosion cracking in martensitic stainless steels.

The term "stress corrosion cracking" refers to the spontaneous, brittle cracking that may result from the combined effects of stress corrosion. Surface or sub-surface tensile stresses, residual or applied, are required for cracking to occur. In general, stress corrosion cracking will proceed transverse to the resultant tensile stresses acting on the material. In the case of SM-1 studs, a complex system resulted from the applied bolting stress, thermal

stresses, residual grinding stresses, and heat treating stresses not entirely relieved by tempering. In addition, the stress components resulting from torsion or the multi-axial stresses in the root areas of the threads further complicate the stress pattern and, in turn, the expected crack pattern. Such, then is the complexity of the stress distribution in the threaded areas of the studs that, while only circumferential cracking would be expected, longitudinal cracking may have been caused by components of tensile transverse to the major applied stress.

With regard to the residual stresses present in the studs, it should be noted that no heat treatment for stress relief was conducted after the thread grinding operation. The stresses developed during grinding can be high enough to exceed the tensile strength of the material. To minimize the possibility of stress corrosion cracking therefore, one source of residual stress could be controlled by employing a stress relief heat treatment on the studs after the grinding operation.

to provide added assurance of trouble-free operation of the SM-1, the possibility of removing the oxygen from the shielded tank water was considered in detail. It would be possible to design and operate a demineralizer system to achieve oxygen concentration in the shield tank water as low as 0.1 ppm. However, the practical attainment of this low oxygen condition would increase considerably the difficulty of operating the Ft. Belvoir reactor, and there would be no guarantee that a high oxygen incidence could not occur. Furthermore, corrosion tests ⁽³⁾ have shown that the susceptibility of Type 410 stainless steel to stress corrosion cracking may be greater in low-oxygen water (0.5 to 2 ppm) than in

high-oxygen water (30 to 40 ppm). Because of these factors, no additional effort will be made with respect to processing the shield tank water beyond present standards.

The results of the laboratory corrosion tests, which were conducted on specimens of Type 410 stainless steel taken from the actual SM-1 studs, demonstrated how susceptibility to stress corrosion cracking is affected by hardness level of the material. Specimens having hardnesses of Rockwell "C" 20, 26, 31, and 40 were tested; only Rockwell "C" 40 specimens were found to be cracked after 2000 hr of testing. For these tests, the hardness proved to be more critical than the stress level or the time of testing. This is evidenced by the fact that the Rockwell "C" 40 specimen 16C (1000 hr at 30,000 psi or 3×10^7 psi-hours) contained cracks, while the Rockwell "C" 31 specimen 11C (1000 hr at 30,000 psi and 1000 hr at 58,000 psi or 8.8×10^7 psi-hours) did not crack.

The corrosion tests served the purpose of duplicating the cracking which occurred in the original studs under comparable conditions and environment, and substantiating that the susceptibility to cracking increases with increased hardness in Type 410 stainless steel. Extending the testing time would undoubtedly have increased the severity of the cracking in the Rockwell "C" 40 specimens, and introduced cracks into some of the specimens at the Rockwell "C" 31 hardness level. Time and task funding did not permit the extension of the investigation to determine the threshold of cracking as a function of hardness level. However, results indicated that the susceptibility of the Rockwell "C" 40 material is at least three times that of the "C" 31 material, since the former showed cracking at 3.0×10^7 psi-hours, whereas the "C" 31 material was untouched after 8.8×10^7 psi-hours. It is also probable that cracking was initiated in the two failed reactor vessel studs in the first 1000 hr operation at full pressure (2.7×10^7 psi-hours).

In addition to evaluating the variables (hardness and stress level) for which it was designed, the corrosion test used in this investigation would be applicable for evaluating the relative susceptibilities of various alloys to stress corrosion cracking for use as bolting material in a reactor vessel environment.

9.0 CONCLUSIONS

1. Failure of the two Type 410 stainless steel reactor vessel studs was caused by stress corrosion cracking.
2. The cracks found in 12 of the 14 remaining studs removed from the reactor vessel were caused by stress corrosion cracking.
3. For this application, Type 410 stainless steel is susceptible to stress corrosion cracking at hardness values of Rockwell "C" 26 (260 B.H.N.) and higher. Studs having hardness values of Rockwell "C" 21 (235 B.H.N.) should be expected to perform satisfactorily.
4. Improper control of the heat treatment of the studs and the absence of stress relief treatment after the thread grinding operation were contributing factors to the failure of the SM-1 studs.

10. RECOMMENDATIONS

1. To minimize the possibility of stress corrosion cracking, studs should be tempered to a hardness not exceeding Rockwell "C" 21 (235 B.H.N.).
2. The studs should be given a stress relief heat treatment after the final thread grinding operation.

11.0 BIBLIOGRAPHY

1. Lewis, C. F., "A Practical Application of the Holloman-Jaffe Parameter to the Tempering of Type 410 Stainless Steel." High Strength Steels for Aircraft, American Society for Metals, (1958)
2. Lillys, P., Nehrenberg, A. E., " Effects of Tempering Temperature on Stress Corrosion Cracking and Hydrogen Embrittlement of Martensitic Stainless Steels," Transactions American Society for Metals, Vol. 48 (1955).
3. Suss, H., "Susceptibility of A.I.S.I. 410 to Stress Corrosion Cracking in High Temperature High Purity Water." KAPL Memo HOS-6, (1959).
4. Sayre, M. F., "SM-1 Reactor Vessel Cover and Flange Stress Analysis." APAE Memo No. 302, February 19, 1962.

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