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AEC RESEARCH AND DEVELOPMENT REPORT

# ANALYSIS OF FAILED 17-4 PH BOLTS FROM THE HWCTR

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# **ANALYSIS OF FAILED 17-4 PH BOLTS FROM THE HWCTR**

by

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September 1967

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### ABSTRACT

Analysis of five bolts of 17-4 PH stainless steel from the Heavy Water Components Test Reactor that had failed in service showed that the failures were initiated by stress corrosion. In all but one of the failed bolts, an unstable crack size was reached after very little stress corrosion causing fast crack propagation and complete failure. In one bolt, the stress corrosion crack had stopped growing. Many of the bolts, including all those that failed, were found to have been improperly heat treated to approximately the H925 condition, instead of the specified H1100 condition. The harder H925 condition is conducive to stress corrosion cracking in this alloy. No evidence for failure by fatigue was found.

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## INTRODUCTION

The Heavy Water Components Test Reactor (HWCTR) was operated for the USAEC from March 1962 to December 1964 to test fuel and mechanical components with potential applicability to power reactors moderated and cooled by liquid D<sub>2</sub>O. The reactor operation was terminated when this program was redirected toward D<sub>2</sub>O reactors cooled by organic liquids.

During an inspection of the interior of the HWCTR reactor vessel in February 1964, two 5/8-inch bolt heads from the support legs of the gas stilling baffle were found on top of the horizontal reactor shield. During removal of the gas baffle assembly for repairs, two more broken 5/8-inch bolts were found. One 3/4-inch bolt was also cracked, and it subsequently failed during removal. The gas baffle had been installed during startup tests, subsequent to original construction. This assembly contained eight 5/8-inch bolts and 34 3/4-inch bolts. All of these bolts were of 17-4 PH stainless steel\*.

This report gives the results of the detailed examinations and subsequent tests to pinpoint the causes of the bolt failures.

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\* 17-4 PH refers to a patented, hardenable stainless steel manufactured by Armco Steel Corporation.

## SUMMARY

The eight 5/8-inch 17-4 PH bolts and the failed 3/4-inch 17-4 PH bolt were much harder than specified and, thus, had not been properly heat treated.

Stress corrosion of the excessively hardened bolts initiated cracks, which grew slowly until the applied load was sufficient to cause rapid fracture of the main portion of the bolt. Electron microscopy indicated that the main fractures had occurred by ductile rupture in tension. No evidence of fatigue was found.

Although the stresses on assembly of the bolts are unknown, differential thermal expansion between the bolts and the surrounding 304L material, and intensification of the stresses at thread roots could have produced stresses of approximately 96,000 psi. Such stresses are sufficient to cause stress corrosion in the improperly heat-treated material during the 276 days of reactor operation prior to discovery of the failure.

The 17-4 PH bolts were replaced by "Inconel"\* X and AISI 4140 steel, which operated satisfactorily for the remaining 126 days of reactor operation.

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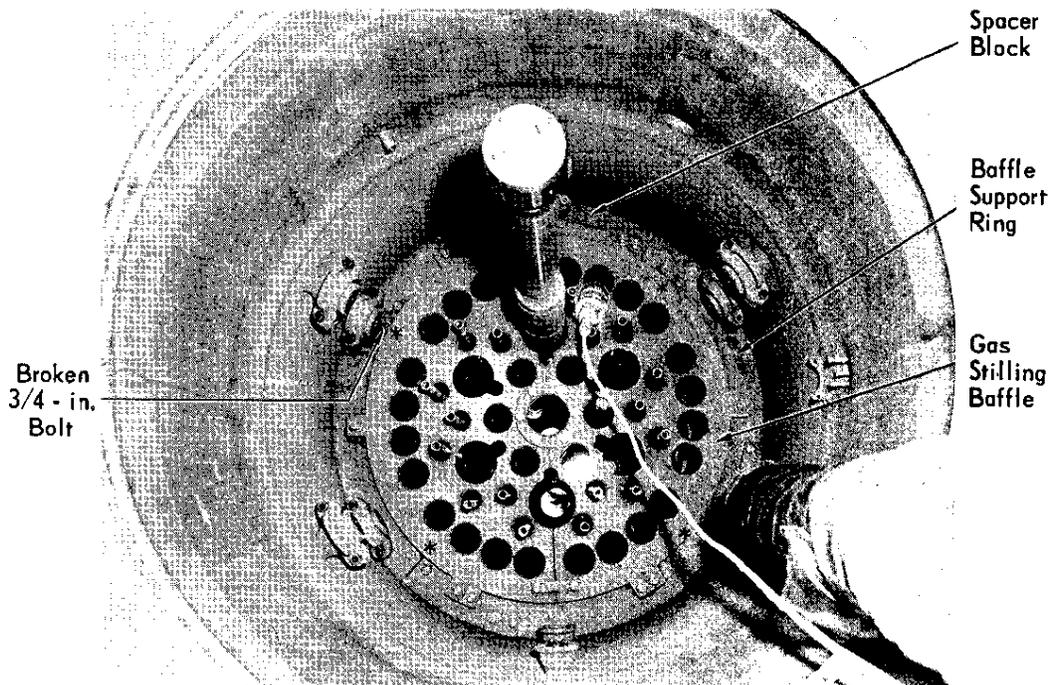
\* Trademark of the International Nickel Company

## RESULTS AND DISCUSSION

### DESCRIPTION OF GAS BAFFLE ASSEMBLY

The gas stilling baffle was a circular-shaped plate 1-1/4 inches thick and 50-1/4 inches in diameter that was bolted to a 3/4-inch-thick baffle support ring, Figure 1. Both the gas baffle and the support ring were in two sections to facilitate assembly inside the HWCTR vessel, and were bolted together with metal straps. The gas-baffle assembly was supported by four T-shaped support posts that were in turn bolted to the top of the flow-distribution baffle. The gas baffle and baffle-support ring were bolted together and to the top of the support posts with thirty-four 3/4-inch-diameter bolts. The baffle-support posts were bolted to the flow-distribution baffle with eight 5/8-inch diameter bolts. Nothing is known about the initial stresses induced in the bolts during installation. A proprietary thread lubricant subsequently found to contain chlorides may have been used during the installation of the baffle. Thus, chlorides may have contributed to the failures.

All plate material in the gas baffle including the washers was 304L stainless steel; all bolts were 17-4 PH.



\* 3/4 - in. bolts holding baffle support ring to top of support posts

FIG. 1 GAS STILLING BAFFLE ASSEMBLY

## DESCRIPTION OF BOLTS

The four 5/8-inch bolts that failed were in support posts 1, 2, and 4 of the gas stilling baffle, Figure 2. The two bolts that were found first, 1A and 2B, had failed adjacent to the first or second threads from the unthreaded shank, Figure 3. Bolts 4A and 4B failed adjacent to the first engaged thread. No other cracks were found in the eight 5/8-inch bolts by low-magnification inspection.

All surfaces of the eight 5/8-inch bolts were covered by a thin, black corrosion film. This film also covered the fracture surfaces of the failed 5/8-inch bolts, and showed that the fractures had occurred some time before they were discovered, which was after about 276 days of reactor operation.

The failed 3/4-inch bolt was located in the top of post 4, position A, and held the baffle support ring to the support post. The fracture occurred adjacent to the first engaged thread, Figure 4. This bolt was also covered with the black film, but only part of the fracture surface was black. The rest of the fracture surface was light gray, denoting the area that fractured when the bolt was removed during the repair work.

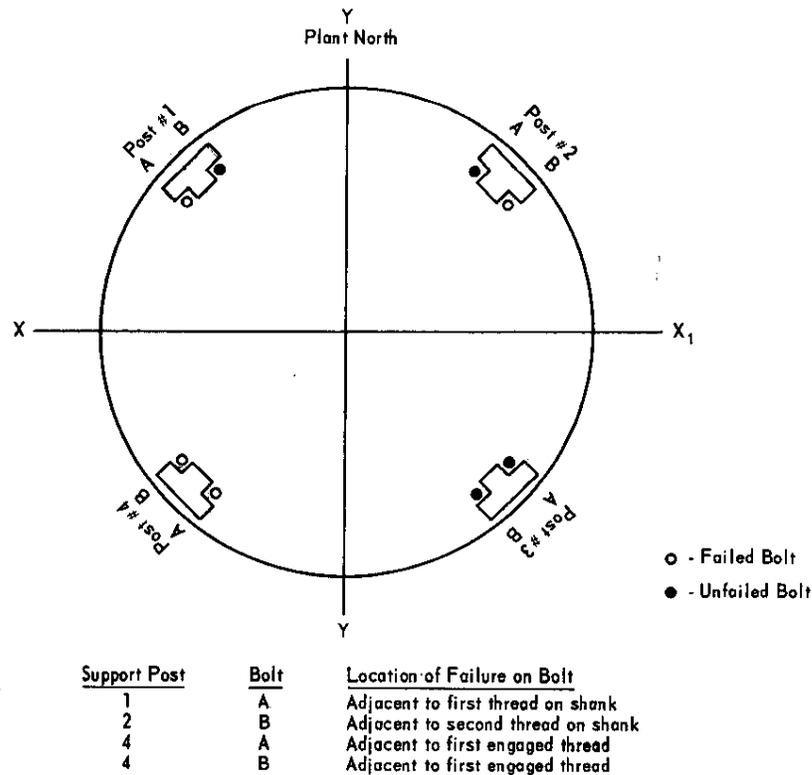


FIG. 2 POSITIONS OF BROKEN 5/8-INCH BOLTS



Bolt 1A

FAILED  
BOLTS



Bolt 2B



INTACT  
BOLTS

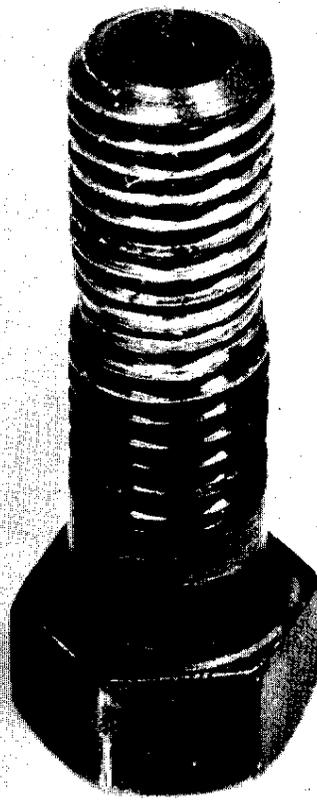


FIG. 3 IRRADIATED 5/8-INCH BOLTS

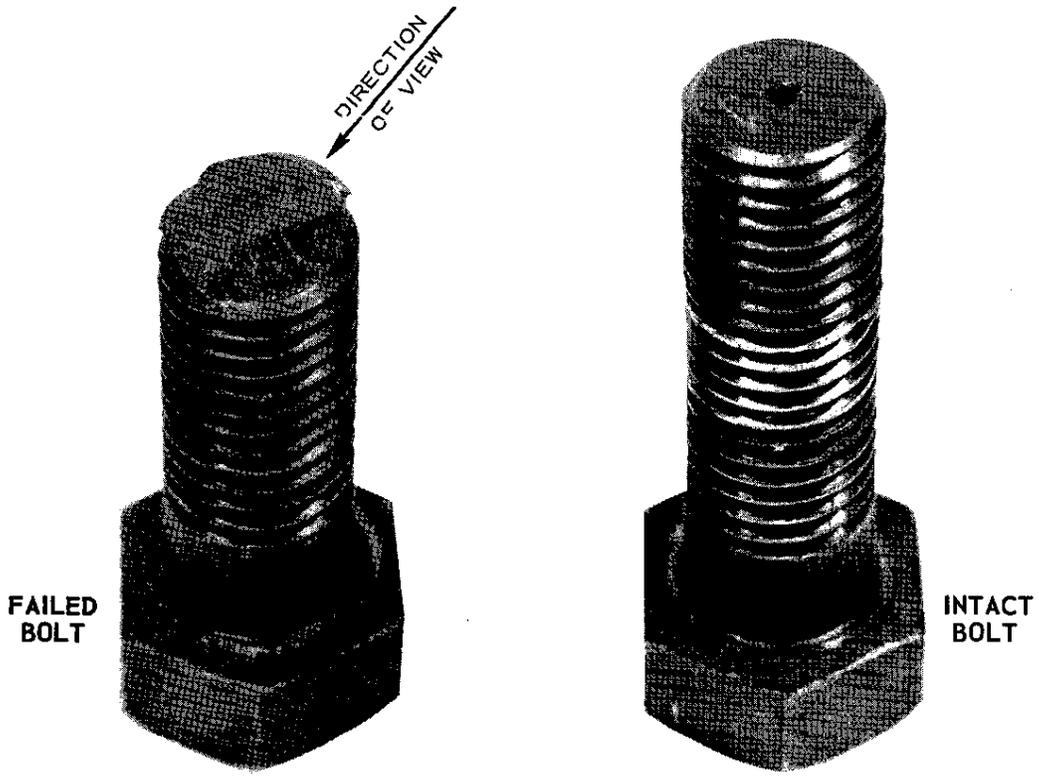
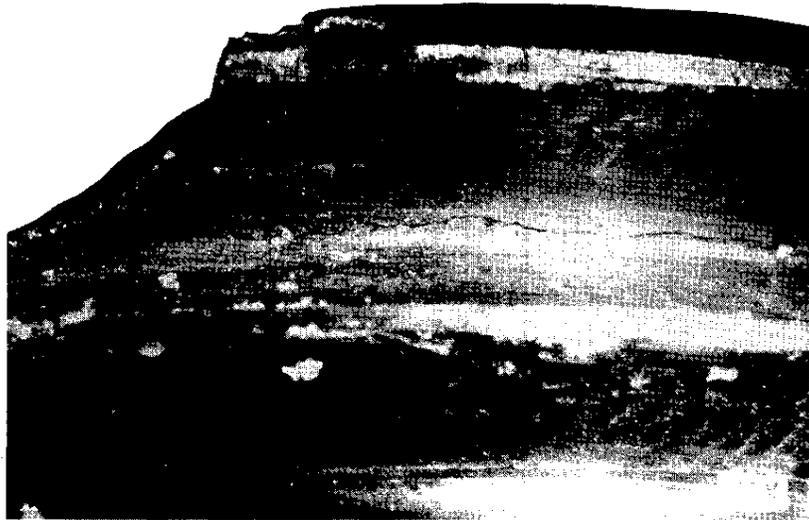


FIG. 4 IRRADIATED 3/4-INCH BOLTS

## FRAC TOGRAPHY

The macroscopic features of the fracture surfaces were examined to determine the sites of crack initiation and the areas of fast crack propagation. Previous studies of the macroscopic features<sup>(1-3)</sup> of fractures have shown that fast crack propagation generally occurs near the end of the failure sequence and produces ripples on the fracture surface. The ripples run roughly parallel to the direction of crack movement, taking a fan-shaped appearance, and point back to the area of crack initiation and slow growth. Shear lips form as the last step in fracture and, thus, are not associated with the early phases of failure.

The fracture surfaces of the 5/8-inch bolts shown in Figure 5 were almost totally covered by ripples in the fan-shaped orientation. The areas of crack initiation and slow growth, indicated by the ripples and the absence of shear lip, were small and shallow, showing that the main crack had initiated and/or grew slowly about 1/3 around the circumference, while advancing only about a thread depth into the bolt, Figure 5. The extended but shallow crack, and the roughness of the surface produced during the slow growth period, suggest that stress corrosion was responsible for crack formation early in the failures. The large fraction of the surfaces covered by the ripples and the low amplitude of the ripples indicated a high applied stress during failure.

The fracture surfaces of bolts 4A and 4B appeared very similar to the fracture surfaces of bolt 1A (Figure 5) even though the bolts fractured in different places (see p. 5).

The fracture surface of the 3/4-inch bolt was generally similar to the surfaces just described, except for the relatively larger area covered by the in-service fracture, Figure 6. The area of in-service fracture (dark area) was rough and generally featureless, suggestive of a stress-corrosion crack. The greater roughness (compare with Figure 5) and the fact that the crack had arrested long enough for the black film to form by general surface corrosion both indicate that the applied stress was low. The ripples produced during removal of the bolt are typical of fast fracture in this material in the hardened condition and compare well with similar features observed in the failed 5/8-inch bolts.

None of the areas of crack initiation exhibited any of the macroscopic features characteristic of fatigue failures. At first glance, the "half-moon" mark about midway through bolt 2B (Figure 5) may appear to be a fatigue feature. Such an interpretation is discounted, however, by the morphology and excellent

definition of the features surrounding the "half-moon" mark. For example, ripples were clearly formed and continuous across the "half-moon" mark; and the surface inside the "half-moon" contained ripples, instead of being smooth as would have been the case for a fatigue fracture. The "half-moon" mark was probably caused by a change in temperature or stress during the brief period of crack propagation.

The fracture surfaces were replicated for electron microscopic examination by the cellulose acetate-carbon technique. Electron micrographs are shown in Figure 7. Microscopically the fracture surfaces were composed largely of various sized dimples, indicating ductile rupture, and some "orange peel" and irregular microcracking, which are evidence of stress corrosion. These fractographic features showed that the fractures had occurred by tensile rupture. The low ductility suggested by some of the features, secondary cracking and discontinuous cleavage, may have been caused by the plane strain conditions set up by the notch effect of the threads and the low fracture toughness of highly hardened 17-4 PH. Detailed searches revealed no microscopic arrest lines characteristic of fatigue.

The fractures of bolts 1A and 2B and the 3/4-inch bolt were sectioned approximately parallel to the ripples and examined metallographically. The fracture surfaces were smooth and almost totally transcrystalline across the fracture surfaces of the 5/8-inch bolts and the light area of the 3/4-inch bolt, Figure 6. In contrast, the in-service part of the 3/4-inch fracture surface was much rougher and exhibited more intercrystalline fracture. Many comparatively short, branching cracks oriented approximately 90° to the surface were observed in the 5/8-inch bolts and the in-service part of the 3/4-inch bolt, Figure 6. These cracks were transcrystalline and intercrystalline, and were the same average length (depth) from one side of the bolt to the other. These secondary cracks probably formed by stress corrosion cracking from residual quenching stresses after complete failure of the bolts. These observations confirmed the results of the macrofractography, namely that most of the main fracture occurred during fast tensile fracture.

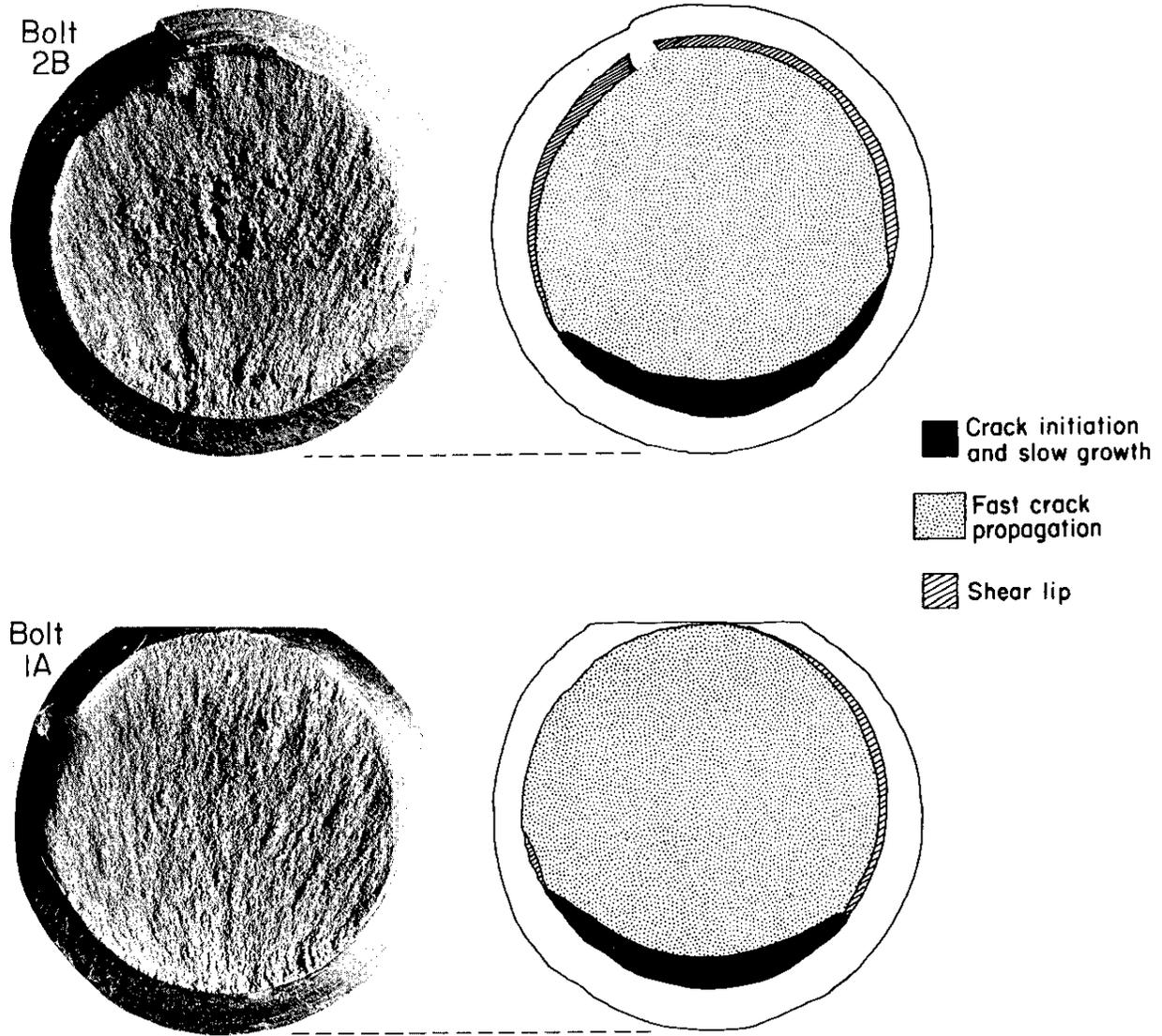
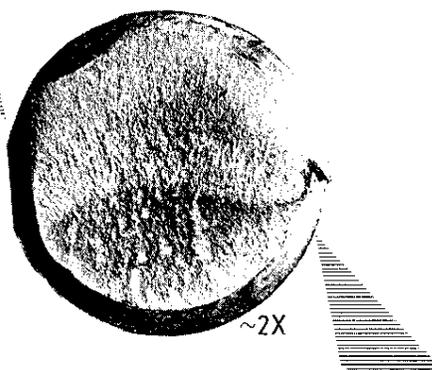


FIG. 5 FRACTURE SURFACES OF FAILED 5/8-INCH BOLTS  
Ripples point to the area of crack initiation

PROFILE OF FRACTURE PRODUCED DURING REMOVAL OF BOLT



PROFILE OF FRACTURE PRODUCED BY STRESS CORROSION DURING HWCTR OPERATION

FIG. 6 FRACTURE SURFACES OF FAILED 3/4-INCH BOLT

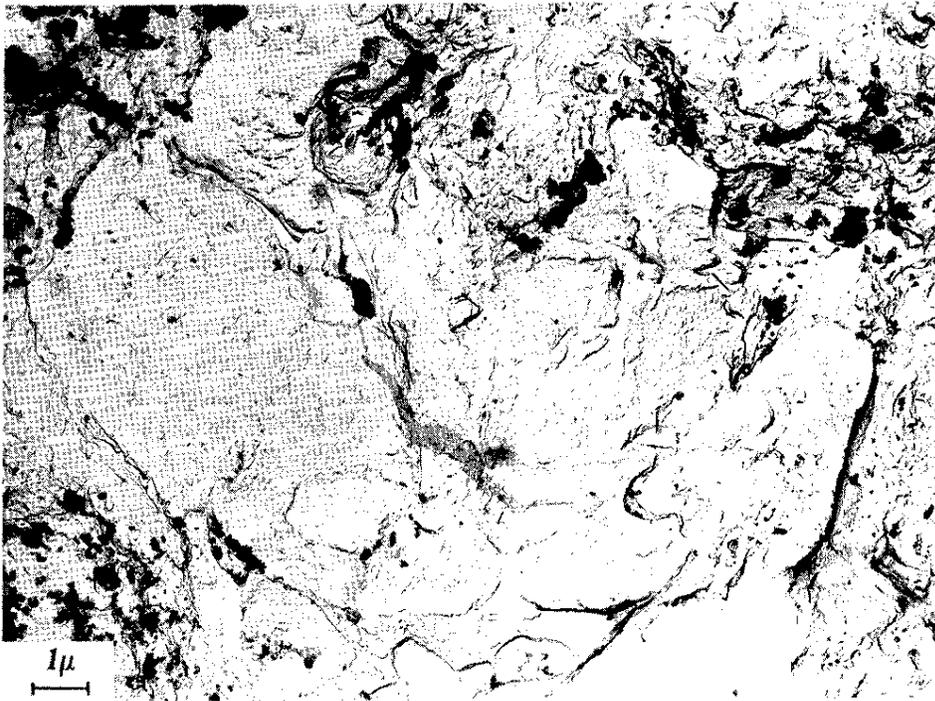
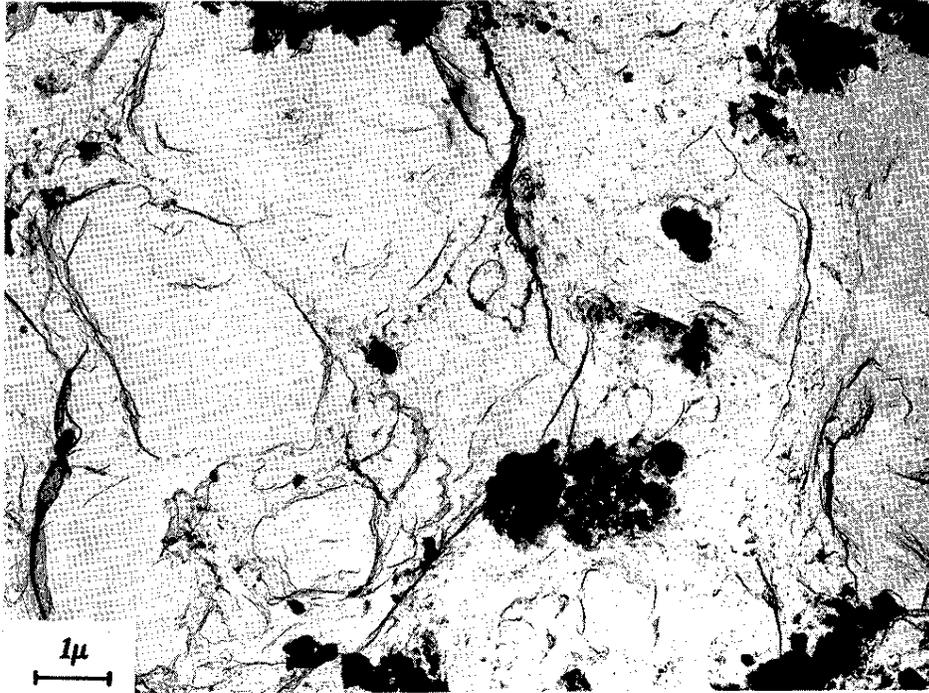


FIG. 7 DIMPLED RUPTURE AND INTERGRANULAR FRACTURE IN 5/8-INCH BOLTS  
Large areas of the fracture surfaces were covered by corrosion products (black spots)  
and small pits from general surface corrosion after failure

## METALLURGICAL CONDITION OF BOLTS

The chemical composition of bolts 1A and 2B was determined spectrographically and found to meet Armco Steel specifications for the major elements except possibly chromium. Results are given in the accompanying Table I. The indicated chromium content of each bolt was marginally lower than the specified value, but this factor is not believed to be significant. According to vacuum fusion analyses, the hydrogen contents were below 1/2 ppm.

TABLE I  
Chemical Composition of 5/8-inch Bolts

Element	Composition, weight percent		
	Armco Specification <sup>(4)</sup>	Spectrographic Determination	
		Bolt 2B	Bolt 1A
Copper	3 - 5	4	4
Chromium	15.5 - 17.5	13	14
Nickel	3 - 5	4	4
Manganese	1.0 max	-	-
Iron	-	78	77
Carbon, phosphorus, sulfur, silicon	1.14 max	-	-
Columbium and tantalum	0.15 - 0.45	-	-
		Total	99

The hardness of the bolts was measured on cross sections near the bolt head and near the threads. The hardness of both failed and unfailed 5/8-inch bolts and the failed 3/4-inch bolt was greater than the specified level of Rockwell C 34, ( $R_C$  34) and varied between  $R_C$  39 and 44, Table II. The hardness of an unfailed 3/4-inch bolt was  $R_C$  33, a typical hardness for bolts heat treated to the H1100 condition as required in the HWCTR specifications. These data show that all the 5/8-inch bolts and at least the failed 3/4-inch bolt had been annealed for precipitation hardening at about 925°F, rather than at 1100°F. As a result, the bolts in the H925 condition were stronger than specified, but were also highly susceptible to stress corrosion cracking.<sup>(5)</sup>

TABLE II  
Hardness of Bolts

Size	Hardness, R <sub>c</sub> (a)								
5/8 in., four unfailed	43 - 44								
5/8 in., failed	<table style="display: inline-table; vertical-align: middle;"> <tr><td style="border-right: 1px solid black; padding-right: 5px;">1A</td><td style="padding-left: 5px;">41</td></tr> <tr><td style="border-right: 1px solid black; padding-right: 5px;">2B</td><td style="padding-left: 5px;">39</td></tr> <tr><td style="border-right: 1px solid black; padding-right: 5px;">4A</td><td style="padding-left: 5px;">41</td></tr> <tr><td style="border-right: 1px solid black; padding-right: 5px;">4B</td><td style="padding-left: 5px;">43</td></tr> </table>	1A	41	2B	39	4A	41	4B	43
1A	41								
2B	39								
4A	41								
4B	43								
3/4 in., one unfailed	33								
3/4 in., one failed	41								

(a) Armco specified<sup>(4)</sup> hardness for H1100 is R<sub>c</sub> 34; H1025, 38; and H925, 42.

Subsequent evaluation of stressed corrosion specimens from HWCTR have confirmed the high susceptibility of the fully hard 17-4 PH alloy to cracking in the HWCTR environment.<sup>(6)</sup> Out of ten stressed bend specimens of 17-4 PH alloy with different treatments, the three specimens with hardness above R<sub>c</sub> 40 failed during exposure in HWCTR. The comparison of hardness with failure experience is presented in Table III.

TABLE III  
Behavior of Stressed 17-4 PH Stainless Samples in HWCTR

(Exposure: D<sub>2</sub>O maintained at pD of 10.7 ± 0.5 with LiOH, temperature during operation about 240°C, temperature during shut-down about 25°C)

Condition When Placed in Test	Hardness After Test, R <sub>c</sub>	Behavior in the HWCTR
Cold worked 48%, annealed 8 hours at 1100°F	30	OK
	30	OK
Annealed 8 hours at 1100°F	26	OK
	25	OK
Cold worked 48%, annealed 1 hour at 900°F	42	Failed <sup>(a)</sup>
	39	OK
Annealed 1 hour at 900°F	32	OK
	30	OK
	43	Failed <sup>(b)</sup>
	(Presumed >40)	Failed <sup>(c)</sup>

- (a) Failure discovered after total exposure of 1000 days to D<sub>2</sub>O atmosphere in reactor gas space, of which 400 days were at operating temperature.
- (b) Failure discovered after total exposure of 65 days to H<sub>2</sub>O atmosphere plus 1000 days in D<sub>2</sub>O atmosphere in reactor gas space, of which 400 days were at operating temperature.
- (c) Failure discovered after exposure of 65 days in H<sub>2</sub>O plus 535 days in D<sub>2</sub>O of which 276 days were at operating temperature.

## ANALYSIS OF APPLIED STRESSES

No record could be found that torque limits were specified or used in the installation of the 17-4 PH bolts. Thus, the initial stresses are unknown.

Calculations have shown that during HWCTR operation significant thermal stresses were generated in the bolts because the thermal expansion of the 304 stainless steel plates and washers was greater than that of the 17-4 PH bolts. At 250°C the additional tensile stress that would be generated in the threaded part of the 5/8-inch bolts by thermal expansion was calculated to be 24,000 psi. The total stress would be further intensified by the notch effect of the threads, particularly at the first thread on the bolt shank. Peterson<sup>(7)</sup> reported a stress concentration factor of over 4 as determined by photoelastic measurements on a Whitworth (English) thread, which is very similar in thread design to the UNC thread on the 5/8-inch bolts. Considering only thermal stresses, therefore, the minimum stresses in the bolts were approximately 96,000 psi. This stress alone is enough to initiate stress-corrosion cracking in improperly heat-treated material.<sup>(5)</sup>

## CONCLUSIONS

1. The stress-corrosion failure of (four 5/8-inch and one 3/4-inch) improperly heat-treated 17-4 PH bolts in a total of 42 bolts after 276 days<sup>(8)</sup> of operation in the gas stilling baffle of the HWCTR indicated the necessity of proper heat treatment of 17-4 PH for service in water-cooled reactors operating at temperatures of about 240°C.
2. The detailed metallographic examination of the excessively hard failed bolts confirmed that stress-corrosion cracking had initiated the failures observed in the HWCTR.
3. The hardness of the failed bolts ranged from Rockwell C 39 to C 43, which indicated that they had been improperly heat treated. The bolts corresponded to the H 925 condition (aged at 925°F) rather than the specified H 1100 condition (aged at 1100°F).
4. The excessively hard 17-4 PH alloy is known from its performance in other reactors to be susceptible to stress-corrosion cracking<sup>(5)</sup>. This behavior was confirmed in a subsequent test of stressed test specimens in the HWCTR environment, which showed that specimens harder than Rockwell C-40 failed after about 400 days of operation.

5. At the time that corrective measures were taken to replace the bolts in the stilling baffle - before the results of this investigation were available - the 5/8-inch bolts were replaced with "Inconel" X (AMS-5667-F Heat Treatment) containing rolled threads, and the 3/4-inch bolts were replaced with AISI 4140 steel (ASTM No. 193-62T). In the following eight months of operation before the HWCTR was shut down, during which the reactor accumulated 126 operating days, the replacement bolts operated satisfactorily. Reference 9 gives details of the corrective measures.

## ACKNOWLEDGMENT

The author wishes to thank M. R. Louthan, Jr. for the electron microscopic examination.

## REFERENCES

1. F. R. Larson and F. L. Carr. "How Failures Occur ... Topography of Fracture Surfaces." Metal Progress 85, No. 2, 74 (1964).
2. F. R. Larson and F. L. Carr. "How Failures Occur ... Experiences in the Field." Metal Progress 85, No. 3, 109 (1964).
3. R. M. N. Pelloux. "The Analysis of Fracture Surfaces by Electron Microscopy." Met. Eng. Qtrly. 5, 26 (Nov. 1965).
4. "Armco Precipitation Hardening Stainless Steels," Product Data Bulletin, Armco Steel Corp., November 3, 1958.
5. M. C. Rowland and W. R. Smith, Sr. "Precipitation-Hardening Stainless Steels in Water-Cooled Reactors." Nucl. Eng. 7, 14 (1962).
6. J. M. McKibben. Corrosion of Equipment in the Heavy Water Components Test Reactor, USAEC Report DP-964, E. I. du Pont de Nemours & Co., Aiken, S. C. (1965).
7. R. E. Peterson. Stress Concentration Design Factors. John Wiley and Sons, Inc., New York. (1953).
8. E. O. Kiger. Operational Summary of the Heavy Water Components Test Reactor USAEC Report DP-991, E. I. du Pont de Nemours & Co., Aiken, S. C. (1966).
9. P. L. Gray. "In-Situ Repairs in the Heavy Water Components Test Reactor." Nucl. Appl. 2, 213 (1966).