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SUBJECT: Metallurgical Examination of HRT
Leak Detector Tubing And Flanges

TO: Distribution

FROM: G. M. Adamson, J. P. Hammond, T. M. Kegley, J. K. White

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After several failures had occurred in the HRT leak detector system, several lengths of this tubing were removed for metallurgical examination. The tubing was of type 304 stainless steel and was 1/4" in diameter with a 0.065" wall. The tubing had been purchased as three different lots, the first in 45 ft. lengths and the other two as standard lengths. Tubing from the first lot was used primarily for the shield penetration and, therefore, sections of it are present in all lines of the system.

In the original design and during the preliminary testing, the leak detector system was filled with water under a static pressure. During the preliminary testing and before the reactor system had been heated, it was discovered that the leak detector system had become contaminated with chlorides. One analysis of the water drained from it reported a concentration of over 1000 ppm of chloride. At this time, an effort was made to remove the chlorides by flushing with water, but it was not completely successful. The first section of tubing was examined during this interval. When testing of the reactor was resumed, the leak detector system was vacuum dried and then filled with high pressure oxygen. A hot test was undertaken during this part of the program, giving temperatures in the leak detector system over the entire range from room temperature to almost 300°C. It was during this stage of testing that the failures occurred. Because of the mechanics of the system, it proved to be impossible to keep all the leak detector tubes dry during testing.

While no samples of "as-received" tubing were available, several lines had been welded into the shield and were then blanked off to be used as spare lines; they had not even been filled with water. These lines were removed and over 20 sections were examined by one means or another. Visually, a greenish colored scale was observed on the inner surface, while the outer surface was bright. Fig. 1 shows a typical I.D. surface of the poor tubing from this lot. The surface was quite rough and a second phase was visible after etching. At a higher magnification, Fig. 2, it is evident that the irregularities are narrow and of a worm-hole nature, varying in depth from 1 to 2 mils. In 9 out of 17 sections, from three of five lengths examined microscopically, conditions were as bad or worse than those shown. Conditions representative of the other 8 sections, from two lengths, are shown in Fig. 3. While these surfaces are rough, they are not much worse than those found in tubing from other manufacturers, Fig. 4, and do not show the precipitate.

Fig. 5 shows, at high magnification, two different sections of tubing etched to determine the nature of the precipitate. The deposit appears to be a carbide and extends to a maximum depth varying from 3 to 5 mils. A thin layer is present on the surface in which the carbides are no longer present.

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Distribution

-3-

January 31, 1957

A section of the outer surface from the same tubing is shown in Fig. 6 and appears to be free from carburization. J. L. English ran some Huey tests on sections from another length of this tubing and obtained average rates of 6.0 MPM in the unsensitized condition. The ID surface of the tubing after Huey testing is shown in Fig. 7 and helps to confirm that carburization had occurred.

The surface of a section of tubing removed from the shield wall after the second shut-down is shown in Fig. 8. This length had been used during both the contaminated water and the oxygen runs, but was not subjected to elevated temperatures. The surface compares to that shown in Fig. 3, indicating that the general surface condition was not greatly affected by the operation. This comparison is not accurate enough to say that no corrosion has occurred.

Figures 9 to 11 show the types of cracks found in the tubing during the second shut-down, which followed the hot operation. These are fine, branching, transgranular cracks. No evidence of mechanical failure as shown by opening or misalignment of the cracks or by the presence of a deformed structure near any crack may be noted. The base metal has a non-deformed equiaxed grain structure typical of the annealed condition which had been specified. Figs. 9 and 10 were cut from a straight section of line A113 and were not located near a weld or a bend; Fig. 11 was from another line, 243. The only stress on this tubing appears to have been that which resulted from the applied gas pressure of about 2,000 psi. All cracks occurred in the sections of the tube near the flanges, where the temperatures would be highest.

While cracks were present in these lengths of tubing, it may be noted in all three figures that the inner surface is relatively smooth. No evidence of carburization was found. The surfaces of these tubes are not comparable to those used in the shield penetration. It appears that the cracking in this area was caused by the higher temperature and the chlorides carried there by the water.

Since cracking had developed in the tubing at a considerable distance from the most likely source of contamination, a recommendation was made that some of the flanges from the main system be examined. Three sets of flanges, small flanges A-111 and A-213 and large flange A-101, were therefore removed from the system and turned over to Metallurgy for examination.

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These flanges were first studied using a post-emulsified fluorescent penetrant with dry powder developer. This method was selected over other penetrants because of its greater sensitivity. Care was used to insure that all surfaces to be examined were clean and dry. One flange showed no significant indications, one was considered questionable, and the third was reported as probably containing cracks. The questionable results were caused by our inability to distinguish between deep pits, machining marks, and small cracks. Both halves of flange A-101 contained areas giving indefinite indications but other areas, Fig. 12, did show small cracks. A visual and hand lens examination of these flanges revealed only severe pitting and in flange A-101 what appeared to be a fine heat check pattern. The sides of the grooves had been deformed by the high loading applied to the seating area, Fig. 13.

For the metallographic examination, 6 sections were removed from each half of the small flanges and 8 from each half of the large one. Sections were deliberately cut through suspicious areas as revealed by the penetrant inspection. One section from each flange was also checked by macroetching.

Fig. 14 shows a macroetched section from the bottom of the groove of flange A-101. While cracks were present in all areas, the heaviest concentrations were found near the corners which are at the top and bottom of the photograph. All cracks were located within the groove. The cracks do not seem to have any preferred direction. While the figure shown was obtained with hydrochloric acid etching, similar results were obtained with sulphuric acid. Cracks were found on both halves of this flange pair, but not on any of the others.

The results obtained during the microscopic examination confirmed those previously obtained. Cracks were found in flange A-101 but not in the other two. Figs. 15 to 18 show typical cracks found in this flange. These illustrations show cracks from all surfaces of the groove and from both the top and bottom flange. In no section were any cracks found above the seating area of the ring or on the face of the flange. While cracks were found on all surfaces, they were both more numerous and deeper at the corners. While quite a few cracks were found with depths between 0.020" and 0.025", none were found deeper than 0.025". In Fig. 16, are shown cracks which progress to a certain depth and then spread out below the surface. This type of crack was quite common.

Fig. 19 shows typical areas at the corners of flanges A-111 and A-213 with no evidence of cracking. As shown by the figures, flange A-111 has a sharp corner similar to flange A-101, while A-213 has a rounded corner.

UNCLASSIFIED

Distribution

-5-

January 31, 1957

Pitting was found in all flanges, even those without cracks. While most of these pits were only a few mils deep, occasional ones to 20 mils were found. No special effort was made to locate such pits or to determine their maximum depths. Fig. 20 shows what was reported by penetrant inspection to be a possible crack in flange A-111 and the resulting deep pit found by sectioning. Two concentrated areas of deep pits in flange A-101 are shown in Fig. 21. The pitting was worse in this flange and the incidence of pitting decreased with distance from the leak detector hole. Similar pits have been reported in chloride stress-corrosion work carried out by G. E. Moore.

Another type of attack that was present on all flanges is shown in Fig. 22. This attack is not much more than a surface roughening but does extend over considerable areas. It is usually found adjacent to the seating area of the "O" ring. In Fig. 22 may be noted one of the few areas where it extended well down into the groove. No mechanism has been offered to explain this type of attack.

While the reactor system was shut down the second time, the large top flange of the reactor pressure vessel dome, B-1, was opened. This flange is one of the few in the system that cannot be replaced easily. While open, both halves of this flange were penetrant inspected. The top half showed a few suspected areas but nothing that could definitely be called a crack. All these indications were easily removed by machining. The bottom half, which was the part attached to the reactor, showed several suspicious areas and on the side of the groove above the leak detector inlet hole one fairly definite crack. This groove was lapped in place and after the first lapping, the original indication had disappeared, but another was found, and, after the second lapping, another new one appeared. Alternate abrasion and examination finally removed all indications. It was not possible to say if these indications were shallow cracks or deep scratches.

Another component that showed cracking when examined was the "O" ring gaskets. Gaskets from flanges A-101 and B-1 were examined and cracks were found in both. Fig. 23 shows one of the penetrant indications and a microsection from the gasket from flange B-1. Cracks were present on both the inside and outside surface but were more prevalent on the inside. As would be expected from the high stress, these cracks are more open and do not have as many branches.

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Distribution

-6-

January 31, 1957

Conclusions:

From these data, it seems that the chloride contamination entered the system in a portion of the first lot of tubing used for the shield penetration. It is not possible to determine how the bad tubing was distributed, whether it was complete tubes or only certain areas in the tubes. It was spread through the system by the water, and cracking occurred in the high temperature areas. In the leak detector system, cracks were found that extended entirely through the tube wall. While these cracks are typical chloride stress-corrosion cracks, they formed under very low stress levels. A complete replacing of this tubing is necessary.

Cracks were found in two out of four high pressure flanges examined. In both cases, cracking was in the larger flanges. The cracks are not deep but are present in the area of highest stress. While in a normal industrial application, the system would be operated with such cracks present, in this case we recommend replacement of the flanges. Once the reactor has gone critical, a failure in many of these flanges would cause termination, and we feel the risk is too great. Even if the stress-corrosion conditions are eliminated, the present cracks will act as stress raisers and could propagate through the flange. No nondestructive test method is available at present which will enable us to definitely determine the presence of such cracks or to determine their depth.

The exact source of the chloride cannot be determined, but, after considering these results and visiting the manufacturer's plant, the following hypothesis appears to be the most likely one: (1) Because of the unusual lengths after rod drawing with a chlorinated hydrocarbon lubricated rod, the inside of the tube, or portions of it, was not degreased (2) during annealing, the drawing compound decomposed and carburized the inner surface (3) the carbides were partially leached out during a subsequent nitric acid pickle (4) the cycle was repeated with a rough surface to make it even worse. Two other possible sources of contamination are a hydrochloric acid etch, which the manufacturer denies, or from trichlorethylene degreasing compound trapped in the tube and decomposed.

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T-11835

Glyceregia etched

T-11903

Unetched

Fig. 1. Inner Surface of Defective "As-Received" Leak
Detector Tubing.

250X

T-10799

Unetched

Fig. 2. Surface of Leak Detector Tubing Surface at High Magnification.
750X

T-11901

Unetched

T-11902

Fig. 3. Better Areas on Inner Surface of Defective Leak Detector
Tubing.

Glyceric acid etched

250X

T-11862

Glyceregia etch

Fig. 4. "As-Received" Inner Surface of Leak Detector Tubing
From Another Lot. 250X

T-11858

Glyceregia etched

T-10823

Vilella's etch

Fig. 5. Inner Surface of Defective Leak Detector Tubing
Etched to Show Carbides.

750X

T-11904 Glyceregia etched
 Fig. 6. Outside Surface of Defective Leak Detector Tubing
 Showing Absence of Carbides. 250X.

T-11423 Glyceregia etched
 Fig. 7. Inner Surface of Defective Leak Detector Tubing
 After Huey Testing. 250X

T-11894

Glyceregia etched

Fig. 8. Section of Leak Detector System From Line FCPNW
Removed After Testing. 250X

T-11488

50X

T-11489

250X

Fig. 9. Transgranular Cracks in Transverse Sections of Leak Detector Line A-113.

T-11503
 Fig. 10. Transgranular Crack in Longitudinal Section of
 Leak Detector Line A-213. 50X

T-11718
 Fig. 11. Cracks in Transverse Section of Leak Detector
 Line 243. 50X.

T-11853 Black Light
Fig. 12. Penetrant Indications of Cracks in Groove of Flange A-101.

T-11542
Fig. 13. Macro Section of Groove in Flange A-213 Showing Deformation. 9X

T-11543

Fig. 14. Hydrochloric Acid Macro-etched Section of Bottom of
Groove From Flange A-101. 15X

T-11620

Outside Corner

T-11623

Inside Corner

Fig. 15. Typical Cracks in Corners of Groove of Bottom Flange A-101. 100X

T-11667

Bearing Area

T-11619

Side

Fig. 16. Typical Cracks From Side of Groove From Lower Flange A-101. 100X

T-11715

Fig. 17. Cracks in Bottom of Groove From Lower Flange A-101.
100X

T-11621

Fig. 18. Cracks in Inside Corner of Top Flange A-101.
100X

T-11716

Flange

A-111

T-11717

Flange A-213

Fig. 19. Typical Corners of Grooves From Two Small Flanges Showing Absence of Cracking.

T-11516

Surface Appearance

16X

T-11618

Micro Section

250X

Fig. 20. Apparent Crack and Resulting Pit in Flange A-111.

T-11548

Bottom of Groove

T-11545

Side of Groove

Fig. 21. Areas of Deep Pits in Groove of Lower Flange A-101.
15X

T-11546

10X

Fig. 22. Example of Roughening Attack Found in All Flanges.
Flange A-101.

T-11852

Black Light
Penetrant Indication

T-11653

100X

Fig. 23. One of the Circumferential Cracks Found in "O"
Ring From Flange B-1.