NUCLEAR MARTIN DIVISION

BALTIMORE 3, MARYLAND

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United States Atomic Energy
Commission



# SNAP I
# DYNAMIC MERCURY LOOP TESTS OF SELECTED MATERIALS

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# FOREWORD

This report has been prepared by The Martin Nuclear Division to present the mercury corrosion test loop results. The work was conducted under Contract AT(30-3)217.

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CONTENTS (continued)

## SUMMARY

Six dynamic boiling mercury loops were tested in connection with the SNAP-I program. The loops were Croloy-5 Si, Croloy-5 Ti, Carpenter 20 Cb, Types 347 and 446 stainless steel and a Type 347 stainless steel-clad niobium boiler coil inserted into the Type 347 stainless steel loop.

Operation of the Croloy-5 Si loop was discontinued after about 86 hours because the heaters burned out. Extensive general corrosion that penetrated as much as 3.5 mils occurred during this time. The boiler outlet leg of the Croloy-5 Ti loop ruptured after 166 hours operation. Again, extensive general corrosion occurred with up to 5 mils of penetration. The Carpenter 20 Cb loop was never operated at design conditions. It failed after a short time when arcing from the heater to the coil burned through the tubing. The Type 347 stainless steel loop failed after 326 hours when the outlet leg of the boiler coil ruptured. Extensive depletion of nickel with penetrations up to 20 mils was found in the condenser. The Type 446 stainless steel loop did not fail. Its operation was discontinued after 141 hours because of multiple leaks. Numerous transgranular cracks were found in the boiler coil but the origin of these cracks was problematical. The remainder of this loop was apparently unaffected. The niobium coil ruptured after 50 hours of operation. The failure was probably caused by atmospheric oxygen, which contacted the niobium when the Type 347 stainless steel cladding failed.

Successful operating experiences involving two-phase mercury flow and totalling more than 500 hours were obtained using Type 316 stainless steel tubing. Between October 1958 and June 1959, this material was used without failure in the boiler outlet and condenser tubing lines associated with the mercury boiler test rig. Operating conditions for the mercury working fluid at the inlet of this tubing were 200 psia and 1100 to 1350° F.

# I. INTRODUCTION

The successful operation of the SNAP-I Mercury Vapor, Turbine-Driven Power Conversion System was dependent upon finding a material which would withstand prolonged exposure to boiling mercury. The problem of mass transfer was precluded by the loop design. The hot and cold portions were separated by a vapor space. When the mercury in the boiler became saturated with the containment material, dissolution would cease. Some slight entrainment of the dissolved constituents in the mercury vapor could transport limited amounts of contaminants to the condenser; however, the respective volumes of condensed mercury and entrained particles in the condenser favored a solution well under saturation at the temperatures of the condenser. Therefore, no mass transfer was anticipated. It was recognized that failures would probably occur in the boilers rather than any other part of the loop. Obviously, any imperfections in the boiler, particularly at the liquid-vapor interface, would be attacked most rapidly and pave the way for eventual failure.

The dynamic loops were designed to test the operating conditions of the actual service unit, i.e., boil the mercury at 1025 to 1050° F, superheat the vapor to 1350° F, and condense it at 1000° F in three of the loops and at 350° F in three other loops. Other tests, for example, static tests and a simple rocking static test designed to approximate a dynamic system, resulted in conflicting data that were necessarily discounted.

The selection of test materials was based mainly on the literature and reported good results of certain materials in operational mercury cycle power plants. Although magnesium and titanium inhibitors are normally used in these systems, it was decided that none would be used in the loop tests because the normal products of reaction, a very fine powder of iron and magnesium oxides, could not easily be removed. The elimination of corrosion inhibitors, combined with the much higher operating temperature, eliminated the materials normally used in mercury cycle power plants from consideration. It must be remembered that the SNAP-I device was designed for 60 days operation; therefore, loop tests with these materials were justified.

Five unimetallic loops and one dissimilar metal loop were tested during this program. The first two loops tested were designed and fabricated to Martin Nuclear specifications by the Findlay Science Engineering Company. These were the Croloy-5 Si and the Croloy-5 Ti loops. The third loop, Carpenter 20 Cb, was fabricated by Martin Nuclear to a modified design of the first two loops. The fourth loop, Type 347 stainless steel, and the fifth loop, Type 446 stainless steel, were new designs. They were designed and fabricated by Martin Nuclear. The sixth loop was used to test a Type 347 stainless steel-clad niobium-tube boiler coil in the Type 347 stainless steel loop.

# II. TECHNICAL DISCUSSION

## A. MERCURY PURIFICATION

Since the purity of the mercury was of considerable importance, a large central supply was prepared. The starting material was triple distilled. The mercury was first filtered through a gold adhesion filter. Following this, filtered air was bubbled through the mercury for about 30 minutes while it was covered with a blanket of 25% $HNO_3$. Finally, the mercury was washed with distilled water and vacuum distilled into argon purged polyethylene-lined storage drums. Thereafter, argon was the only gas in contact with the mercury. A spectrographic analysis of the mercury showed no discernible lines of contaminants.

## B. CROLOY-5 Si AND CROLOY-5 Ti LOOPS

The original configuration of the Croloy-5 Si and the Croloy-5 Ti loops is shown in Fig. 1. Figure 2 shows one of these loops during operation. The composition of the two materials is as follows: Croloy-5 Si--0.075% C, 0.01% S, 0.015% P, 0.47% Mn, 1.42% Si, 4.58% Cr, 0.49% Mo and the balance Fe; Croloy-5 Ti--0.09% C, 0.02% S, 0.015% P, 0.43% Mn, 0.37% Si, 4.69% Cr, 0.45% Mo, 0.39% Ti and the balance Fe. The desired temperature controls were to boil the mercury at 1025° F (210 psi), super-heat the vapor to 1350° F in the leg between the boiler and the condenser, condense the vapor at 1000° F and return the mercury to the boiler at 1000° F. The design flow rate was 113 pounds per hour. It was planned to operate all loops to failure or for 60 days, whichever came first.

The loops were chemically cleaned, passivated with $HNO_3$ and rinsed with deionized water. They were then evacuated, filled with argon, re-evacuated, etc., for three full cycles and finally filled with the degassed mercury.

After a short run of eight hours, it was discovered that the design parameters could not be reached in either loop. Even at very low flow rates, it was impossible to reach the desired boiler temperature. Consequently, the superheat temperature could not be reached. Excessive heat losses in the upper superheat section caused refluxing of the mercury vapor. It was also found that the condenser cooling system did not provide adequate temperature control. The cooling system consisted of the condenser grid (the upper loop components shown in Fig. 1), a water-cooled flat plate which was blackened on the side parallel to the

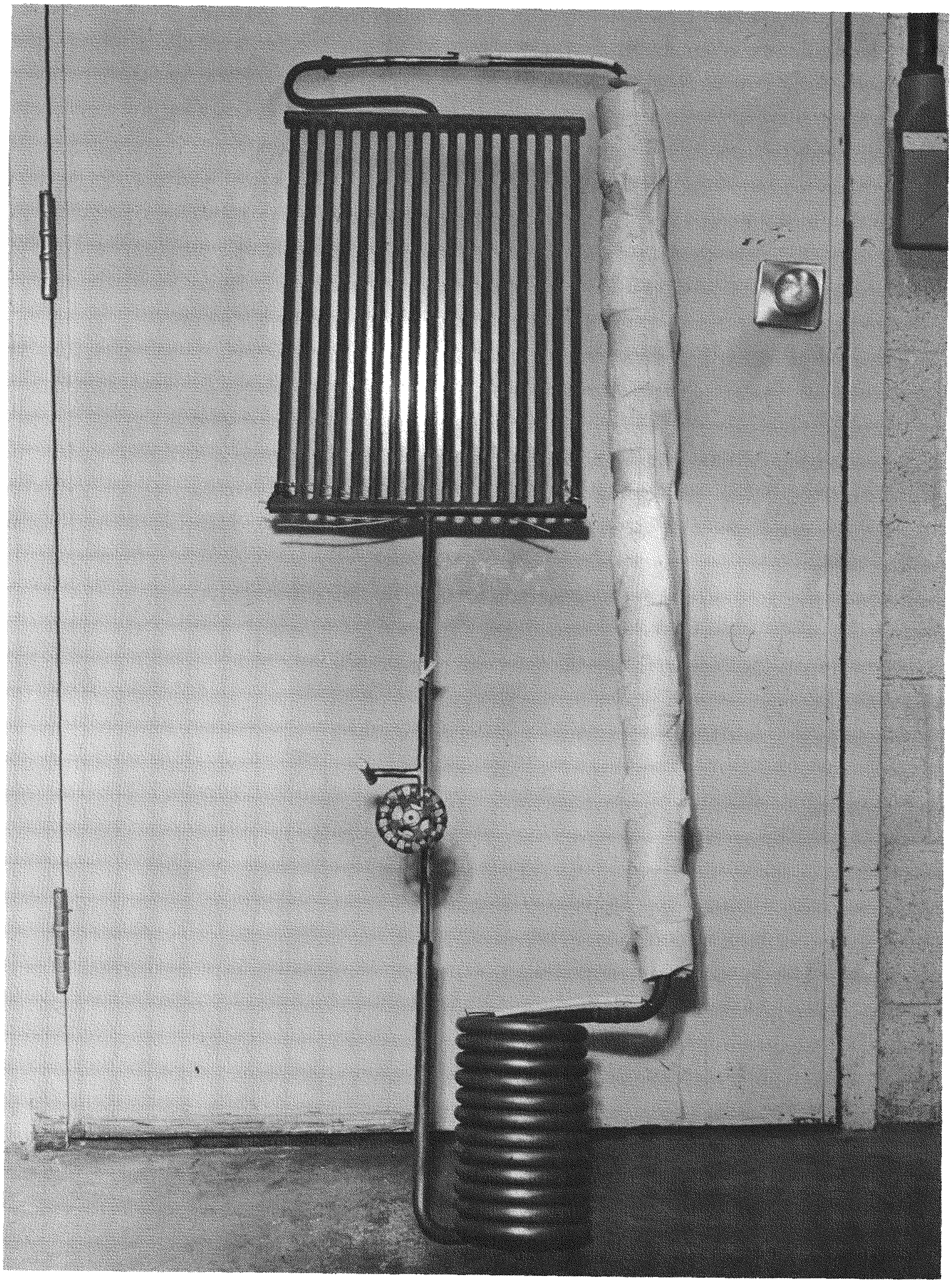


Fig. 1. Croloy-5 Si Loop Before Test

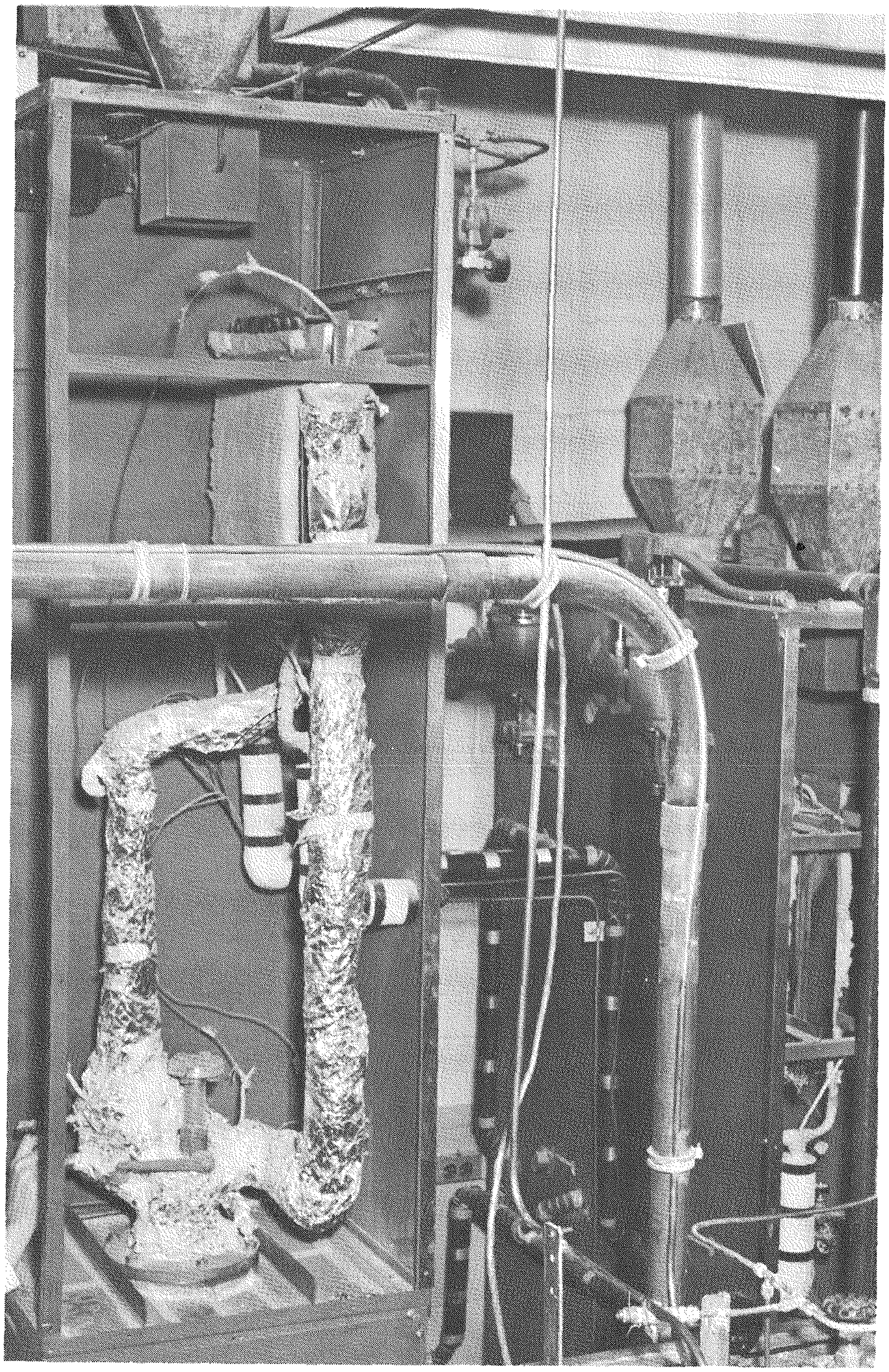


Fig. 2. Croloy-5 Si Loop During Operation

condenser grid, and a half-moon disc, located between the grid and the plate. The disc covered about one-third of the radiating area and was actuated by an automatically controlled air motor, exposing the supplemental cooling surface. Shaft slippage and shutter binding due to disc warpage contributed to the poor temperature control. During an attempt to reach and maintain the design temperatures, the boiler heaters burned out.

The boilers were rewound with higher output heater wire and the thermal shutters modified to prevent slippage and ensure smoother operation. Sections of the shutters were riveted together to decrease high temperature warpage. The superheater section itself was decreased in length by rewinding the heater elements in a shorter space to concentrate the heat output. Heater wire was wound over the entire length of tubing from the boiler to the condenser in an effort to prevent refluxing in the superheat section. Finally, the valve was moved from the original position shown in Fig. 1 to that shown in Fig. 3, which shows one of the loops after test. All of these changes were made on both loops. Both loops were again chemically cleaned before startup.

After another short run of eight hours, it was evident that further modifications were necessary. The excessive heat generated within the steel cabinet melted the insulation on some of the wiring and the pitch in the transformer. Both units were rewired with high temperature insulation wire and the transformers were relocated outside the cabinet.

Both loops were again placed in operation. The results are discussed in Chapter III of this report.

## C. CARPENTER 20 Cb LOOP

This loop was fabricated by Martin Nuclear, using the Findlay design, so that it could be tested with the Findlay equipment. The unit was constructed with Carpenter 20 Cb of the following composition: 0.045% C, 0.69% Mn, 0.46% Si, 28.40% Ni, 19.30% Cr, 2.34% Mo, 0.90% Cb, 3.58% Cu and the balance Fe.

The loop was charged with mercury by the same method used to charge the Croloy loops. The Lewis wire used for heating the loop proved unsatisfactory due to excessive power requirements. At high temperature, the insulation powdered and the wires burned in a number of places. The loop was rewired with nichrome and insulated with ceramic sleeves. Four separate temperature controllers were installed in the superheater area. The loop operating time exceeded 80 hours, but at no time was it possible to run more than four hours continuously. Inconsistencies in temperature readings led to a complete recalibration and replacement of some instrumentation. Following this, a trial run revealed a deficiency in the superheat capacity.

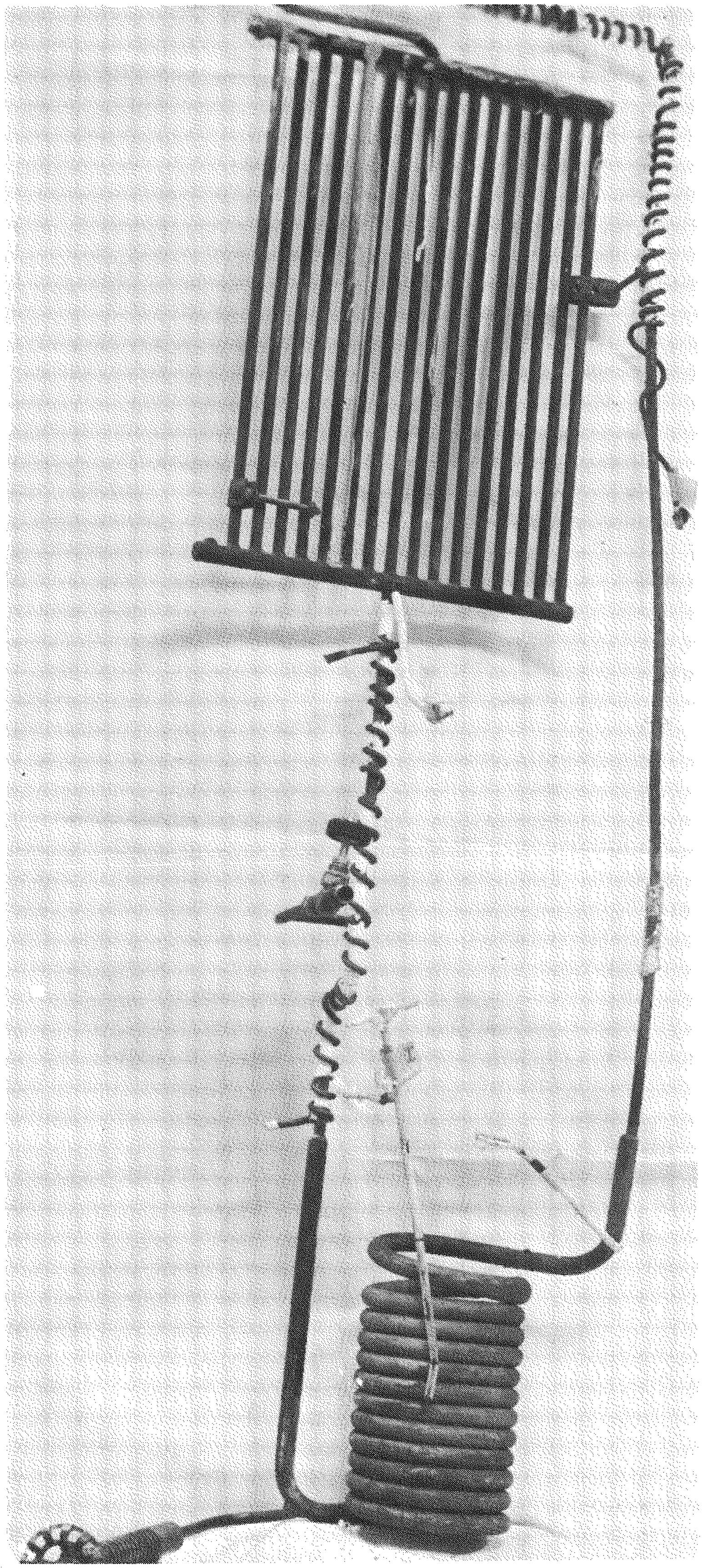


Fig. 3. Croloy-5 Si Loop After Test

The superheater section was reworked by installing specially wound alundum cores with sufficient power density to maintain superheat. The loop was again tested and sections of the superheat section broke down. It was found that the wire in the superheater was arcing to the coil with subsequent melting at these points.

In an effort to reach design conditions with the Findlay loops, the wire-wound superheater was replaced with a tubular Globar heater, and the condenser was modified from the grid-type to a spiral coil configuration. This improved loop was never fully tested because a concurrent re-evaluation of past experience indicated that only a complete redesign of the entire loop concept would permit prolonged operation at design conditions.

## D. TYPE 347 AND TYPE 446 LOOPS

Much design information was gained from operating the described loops. The major changes included lengthening the vertical legs and increasing the temperature gradient of 1000 to 1350° F to 330 to 1350° F, using a water-cooled condenser coil instead of the air-cooled multiple straight tube condenser formerly used. A major change in operational philosophy was adopted, i.e., the mercury was boiled at about 1050° F and superheated to 1350° F within the boiler and the superheat temperature was maintained in the vertical and horizontal legs between the boiler and condenser. This contrasts sharply with the former practice of boiling and exiting the mercury from the boiler at about 1050° F and then heating it to 1350° F in the superheater--the factor which contributed most to the failure of the previous loops. Additionally, Globar heated furnaces were designed and built to provide heat for the boiler. These replaced the lead pot-type furnaces used on the Carpenter 20 Cb loop. A continuous argon purge through the furnace was provided to reduce the air oxidation of the boiler coils.

The design specifications for the new loops were outlined only after due consideration of experience and data from the first three loops. Figure 4 shows the new design. The loops were designed so they could be easily changed in case of failure. Either the entire loop could be changed if the system was to be kept unimetallic, or just the coil replaced. The new loops were fabricated of 0.065-inch wall tubing with a 3/8-inch outside diameter. The boiler consisted of 28 feet of tubing coiled on a 2-1/4-inch radius. This resulted in 20 coils. Since a natural convection loop has no pump, the tubing lengths had to be greater than those in the actual power unit. The height of the condenser (liquid level) was governed by the head required to overcome the pressure drop of the boiler coil. The condenser was 11 feet above the coil. The tube

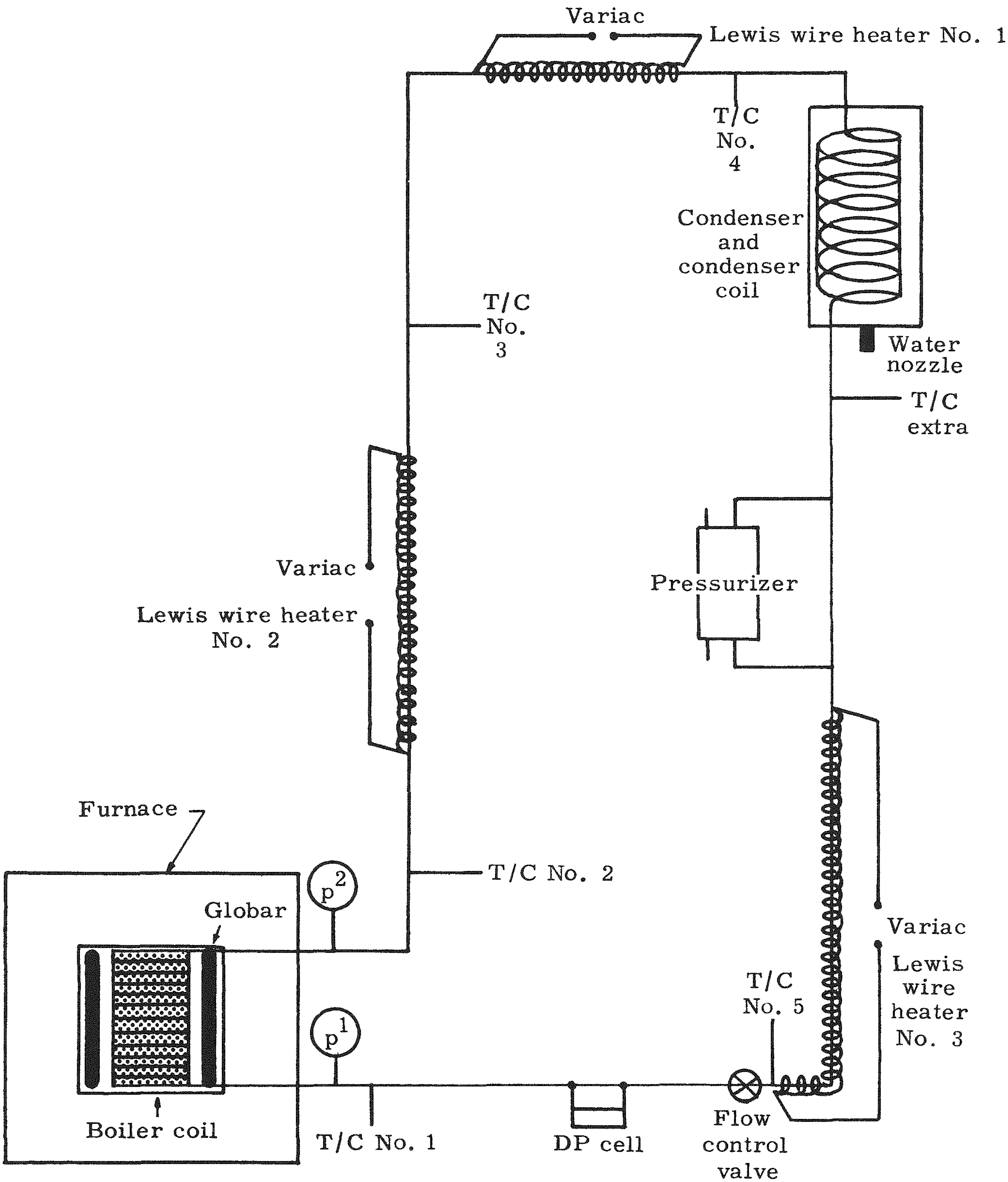
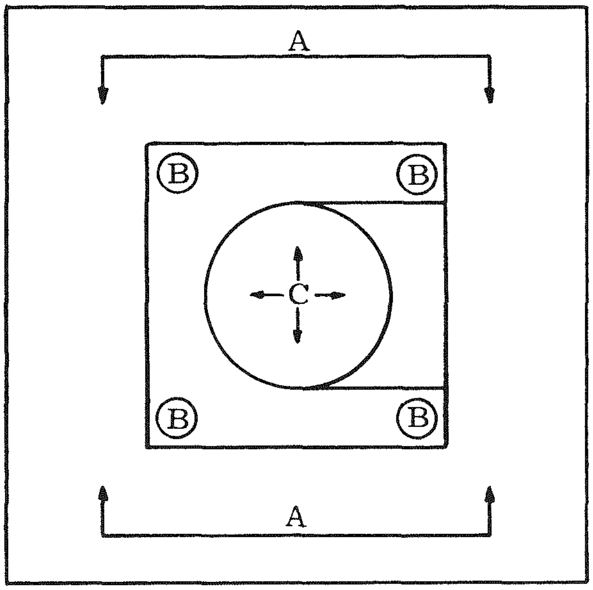



Fig. 4. Schematic of Design for Types 347 and 446 Stainless Steel Loops

from the coil to the condenser was constructed in one piece. The entire length was wound with two Lewis wire heaters. These provided the 1.5 kilowatts of power needed to maintain temperature and prevent the dry mercury vapor from condensing before it reached the condenser. When the rest of the loop was functioning properly, this proved to be a very effective means of controlling heat loss. Heat resistant electrical insulating tape was wound onto the tubing under all of the Lewis wire heaters. Three inches of Super-X insulation were applied to the full length of the hot leg of the loop. The condenser consisted of 6 feet of tubing coiled on a 2-1/4-inch radius which resulted in 5 coils. The condenser coil was placed in an aluminum chamber 8 inches in diameter and 26 inches long. A water spray nozzle was located in the bottom of the chamber. The water spray cooled the mercury below 330° F; therefore, a 0.4 kilowatt Lewis wire heater was provided to assure the correct boiler feed temperature of 330° F. One inch of Super-X insulation was used on the cold leg of the loop. Between the condenser and the inlet leg of the boiler coil were two tees, one for filling and evacuating the loop and the other for the pressurizer which also served as the mercury reservoir; a pneumatically operated needle valve to control the flow; and a differential pressure cell to measure the flow rate and actuate the control valve. Five thermocouples and two pressure tees were located as shown in Fig. 4. A sixth thermocouple was provided, but not used, at the condenser exit. All hot leg joints were welded. The cold leg joints consisted of Swagelok fittings.

The furnaces were a radiant heat type (Globar) with a 20-kilowatt capacity. Although they were designed to operate with eight heaters, four proved ample. The Globars were wired two in series with each pair connected to a saturable core reactor. Control was effected by a Martin-made d-c power supply regulated by a Wheelco on-off type controller. The entire package was wired such that in the "off" position the d-c power was at an intermediate position rather than off, thus supplying some heat to the furnace to avoid excessive cycling of the Globars. In the "on" position, the power level could be preset to supply the heat required to "drive" the boiler coil. Figure 5 shows the furnace design. The boiler coil was supported in the furnace chamber by fire bricks and surrounded by four Globar heaters. The furnace door was made in two sections with a slot in each for the inlet and outlet legs. After the coil had been positioned, the doors were closed and the slots packed with insulation to prevent excessive heat loss.

A re-evaluation of the test materials resulted in choosing several metals for testing. Three of these metals, Types 347, 446 and 316 stainless steel, were chosen on the basis of their compatibility with the other system components, particularly the turbine. Actually, only three of these metals, Types 347 and 446 stainless steel and niobium, were tested during the remainder of the program.




A--Insulation

B--Globar heaters

C--Furnace coil

Fig. 5. Furnace--Cross Section of Overhead View

The Types 347 and 446 stainless steel loops were fabricated entirely by Martin with the exception of the boiler coils. Each loop was housed in a separate cubicle operated under negative pressure to prevent gross contamination of the entire work area in the event of a rupture.

After installation of the Type 347 stainless steel loop was completed, it became the guinea pig used to test the new design. The loop was cleaned according to the following schedule:

(1) Ten ounces per gallon Oakite 90 was pumped through the loop for 10 minutes.

(2) The system was drained, flushed and filled with 1-1 $HNO_3$ and allowed to stand for 30 minutes.

(3) The system was drained and flushed with distilled water until an acidity test was negative.

(4) The system was filled with acetone, drained and purged with argon gas while applying some heat to the loop.

(5) The loop was pressure checked at 300 psi before and after cleaning.

The purging of the loop with argon while simultaneously applying heat to the loop with the Lewis wire heaters is considered a better means of eliminating the air than evacuating, filling with argon, re-evacuating, etc., as done with the first three loops. After one hour of purging, the loop and reservoir were evacuated and he reservoir was filled with deaerated mercury. To further assure cleanliness of the loop, the first charge was removed after an eight-hour operational run and recharged with clean deaerated mercury. The mercury from the first cleaning run showed no contamination.

The Type 446 stainless steel loop was cleaned and charged with mercury according to the schedule used on the Type 347 loop. After operating for the first eight hours, the mercury was found to contain a dark sediment so the loop was again cleaned and recharged with mercury. A second test run was made; this time the mercury drained from the loop was not contaminated. After recharging the Type 446 stainless steel loop with clean deaerated mercury, both loops were ready for endurance runs.

## E. NIOBIUM BOILER TEST

A niobium boiler coil, clad with Type 347 stainless steel, was inserted into the Type 347 stainless steel loop after the Type 347 stainless steel boiler coil had ruptured. The niobium exceeded 99.9% purity. The loop was cleaned and charged according to the procedures outlined previously and testing was started.

## F. TANTALUM BOILER TEST

A tantalum boiler coil clad with Type 347 stainless steel was inserted into the Type 347 stainless steel loop after the niobium coil failed. The tantalum exceeded 99.9% purity. The effort was terminated before testing could be started.

## III. RESULTS AND EVALUATION*

### A. CROLOY-5 Si

The Croloy-5 Si loop operated continuously for 70 hours at design conditions before the boiler heaters again burned out. Testing was discontinued. The mercury was drained from the loop and found to contain a considerable amount of finely divided black powder. A quantitative measurement was not made; however, X-ray diffraction of the powder showed that it was iron oxide with traces of chromium and silicon oxides. The loop was removed from the cabinet for visual, optical and metallographic examination.

The entire exterior of the loop was covered with a dark adherent oxide film as shown in Fig. 3. The boiler coil and superheat area in particular were severely oxidized as was expected because they were in contact with atmospheric oxygen during the test. Thirty-eight metallographic samples, including one from each condenser tube and one from each turn of the coil, were prepared for examination. The corrosion consisted chiefly of pitting in the various areas and the depth of penetration was tabulated from measurements of these pits with the micrometer eyepiece of a metallograph. Eighteen specimens from the condenser averaged penetration depths of 2.0 mils, 5 specimens from the superheater averaged 3.2 mils, and 14 specimens from the boiler averaged 2.4 mils. The greatest depth found was 3.5 mils which was in the superheater. The evidence indicates that the superheater exhibited the greatest amount of corrosion. This was particularly true of the area just after the boiler which contained "wet" mercury vapor and was one of the hottest sections of the loop. The attack was of a general pitting type with precipitation of the metallic oxides throughout the loop and an eventual return of these oxides to the boiler by entrainment of the oxide particles in the vapor or liquid mercury.

The magnitude of the oxides present was quite surprising as considerable care was used in filling the loops. It was evident that considerable oxygen remained in the system. Some of this oxygen was in the tubing itself; however, the mean free path of the molecules limited the efficiency that could be attained in removing air from the loop even with the use of argon. This no doubt accounts for the major portion of the oxygen present. A modified technique, used on some of the following loops, provided a much more efficient way of removing atmospheric gases.

*J. W. Smith

The boiler was heated by two sources: The outer heater was a tightly wrapped insulated heater in contact with the tube wall, while the smaller inside Calrod heater did not contact the coils. This variation in temperature, combined with the low flow rate of less than 100 pounds per hour at 1000° F, suggests that a vapor phase may have occurred on the interior of the outer walls of the coil. The higher temperature on the outer walls, combined with decreased heat transfer caused by precipitated metallic oxides and the vapor blanket, led to overheating of the outer heater. The external side of the boiler coil showed an accelerated corrosive attack with scaling which again was caused by the outside heater overheating and the ready access to atmospheric oxygen.

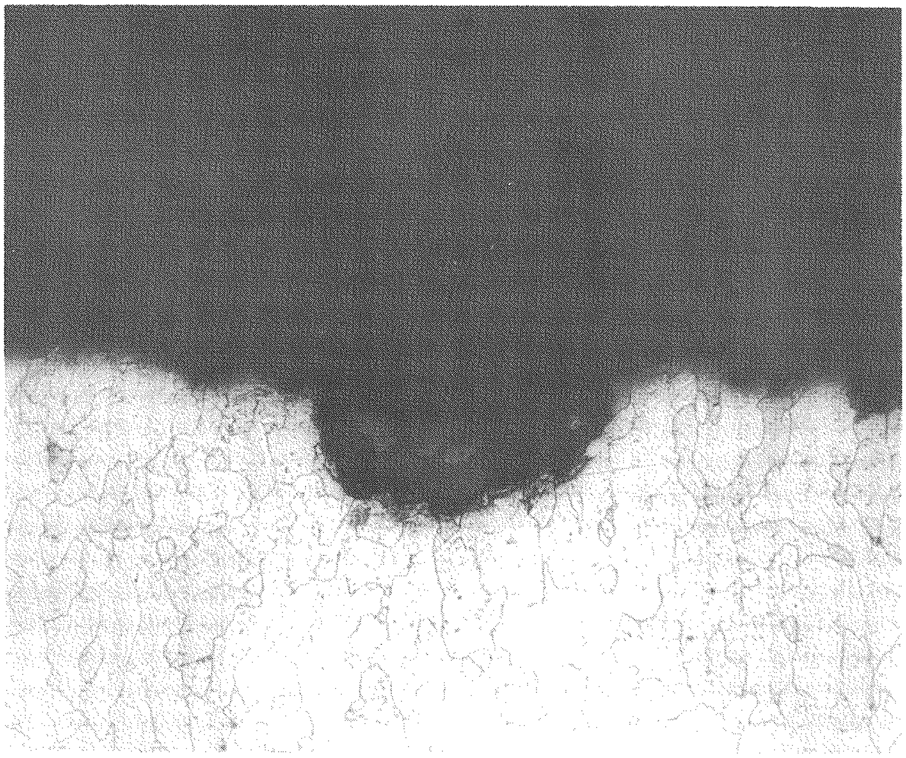
The condenser showed various degrees of pitting which averaged 2.0 mils in depth. Several specimens had indications of concentrated attack in single large pits and general corrosion elsewhere. These are shown in Figs. 6, 7 and 8.

The liquid return leg showed an average penetration of 1.5 mils. The attack in this area was general but limited. This section was the coolest portion of the loop and the area where presumably most of the oxide precipitation occurred. The mercury at this point was probably saturated with dissolved metals and metallic oxides and was essentially inactive corrosionwise after the first few hours of operation.

## B. CROLOY-5 Ti LOOP

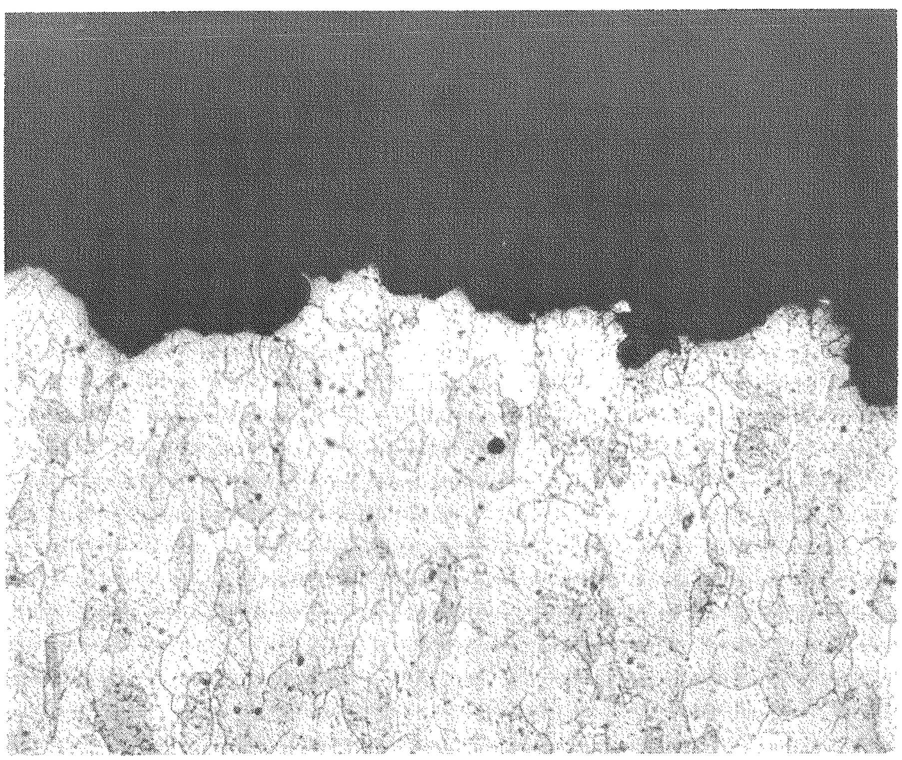
This loop operated continuously for 150 hours at design conditions until a rupture occurred in the boiler outlet. The loop was then drained and disassembled for visual, optical and metallographic examination. The same trend corrosionwise as that found in the Croloy-5 Si was noted in this loop. All samples taken from the areas subjected to the higher temperatures showed a greater depth of penetration than the specimens removed from the relatively cooler areas. The external appearance of this loop duplicated that noted with the Croloy-5 Si loop.

Seventeen specimens from the condenser averaged 2.3 mils in depth of penetration while 5 specimens from the superheater area averaged 3.1 mils and 16 specimens from the boiler averaged 2.4 mils in depth. The greatest penetration found was 5 mils in the condenser. The discussion concerning overheating of the outer heaters with consequent scaling-type oxidation on the Croloy-5 Si loop is also pertinent for the Croloy-5 Ti loop. Generally, the results were common to both loops; however, one prominent difference occurred--the Croloy-5 Ti loop perforated in the general vicinity of the vapor-liquid interface. Figure 9 shows the perforation. It should be noted that this same general area is also exposed



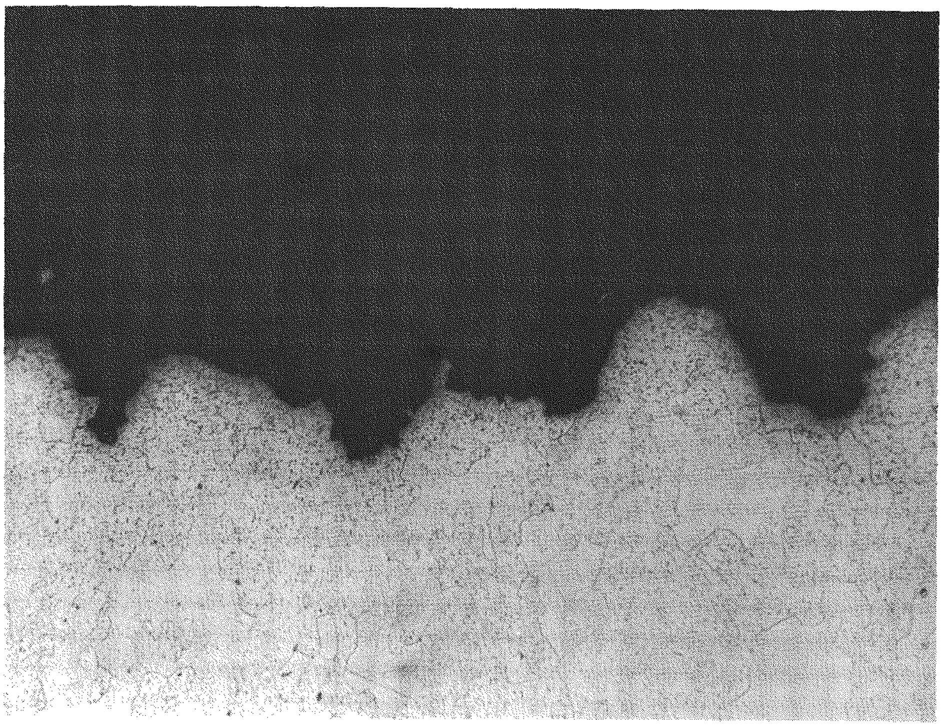
(Exposed to mercury at 1000° F) 250X

Fig. 6. Croloy-5 Si Loop--Pitting in the Condenser Tube No. 2



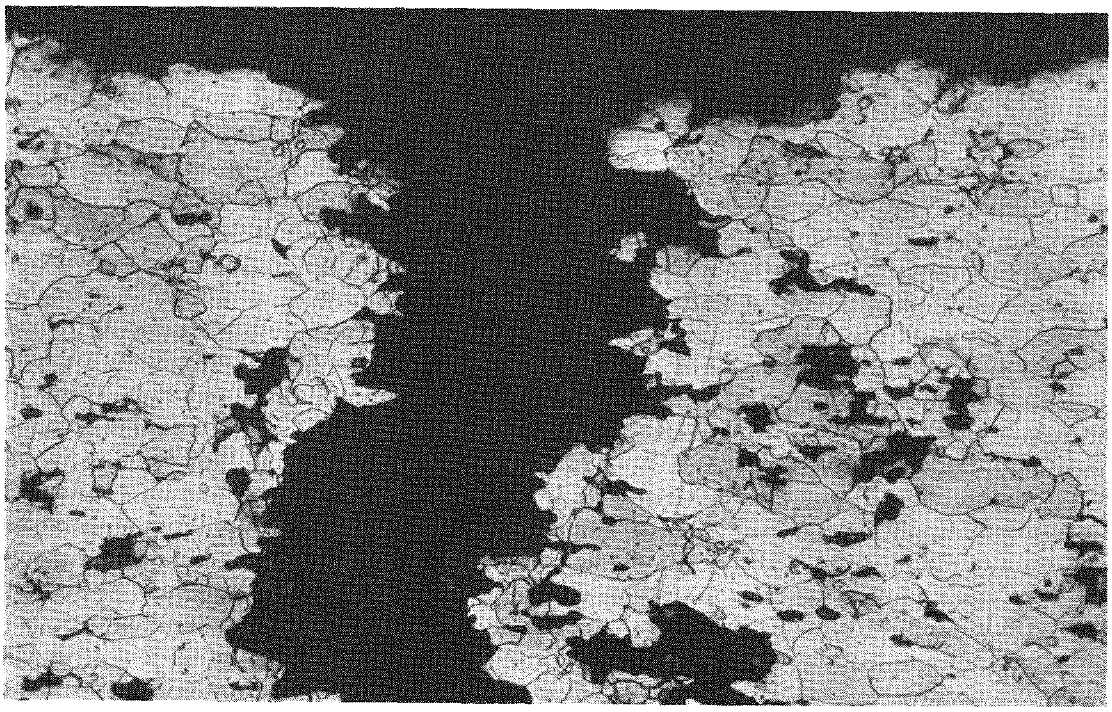
(Exposed to mercury at 1000° F) 250X

Fig. 7. Croloy-5 Si Loop--Pitting in Condenser Tube No. 7



(Exposed to mercury at 1000° F) 250X

Fig. 8. Croloy-5 Si Loop--Pitting in Condenser Tube No. 12.



(Exposed to mercury at 1050° F) 250X

Fig. 9. Croloy-5 Ti Loop--Boiler Coil Perforation

to yet another unfavorable action. Nucleate boiling of the mercury below the liquid surface at startup with subsequent collapse of the vapor bubble may have produced a fatigued area which, when combined with the interface phenomenon, resulted in perforation at that point.

Figures 10 through 13 are photomicrographs from selected loop areas.

## C. CARPENTER 20 Cb LOOP

The Carpenter 20 Cb loop did not reach design conditions at any time during operation. However, it brought to light many of the shortcomings of the design. The loop operation was continuously plagued by insulation and heating difficulties which culminated in the superheater wire arcing to the coil and melting through the tubing in this area. Figure 14 shows the structure adjacent to the melted area and Fig. 15 shows the structure in the melted area. Since the design conditions were never attained and no continuous run was made, it was not considered necessary to do an extensive evaluation of the loop.

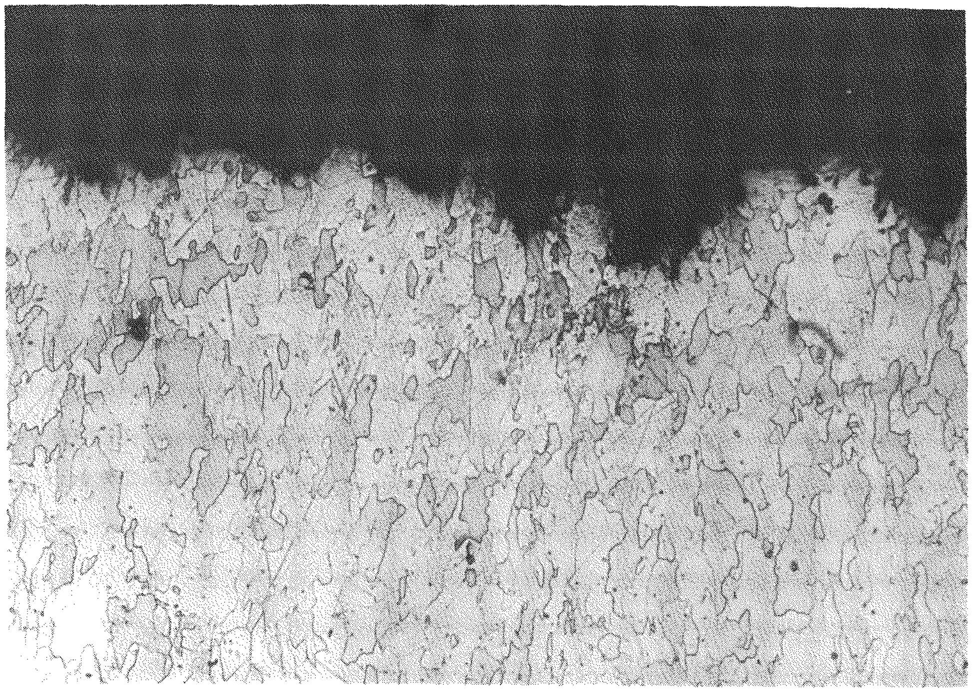
## D. TYPE 347 STAINLESS STEEL BOILER COIL

The checkout work on the Types 347 and 446 stainless steel loops consisted chiefly of eliminating leaks in the Swagelok fittings of the cold leg and in the pipe fittings on the pressurizer. It was not possible to stop the leaks on the pressurizer of the Type 446 loop without rebuilding it. As a result testing of the loop was eventually discontinued.

The arrangement of the filling tee and pressurizer was cumbersome during operation. The pressurizer should have been designed to allow it to be used for evacuating and filling. The differential pressure, cell leaked continuously and was eventually removed, after the flow was noted.

Before the furnace was turned on, the Lewis wire heaters were preheated to 1000° F and 200 psi argon pressure was applied at the pressurizer. The argon furnace purge was also started. This provided a protective blanket around the boiler coil. The furnace was brought to temperature in 200° F increments. Operating the furnace at 1625° F resulted in the design temperature of 1350° F at the outlet leg.

When boiling started during the startup, the mercury appeared to "slug," causing violent fluctuations in the gage readings. This usually lasted only a short time and then the loops settled down to a quiet steady operation. It is interesting to note that once the operating parameters.



(Exposed to mercury at 1000° F) 250X

Fig. 10. Croloy-5 Ti Loop--Pitting in Condenser Tube No. 7

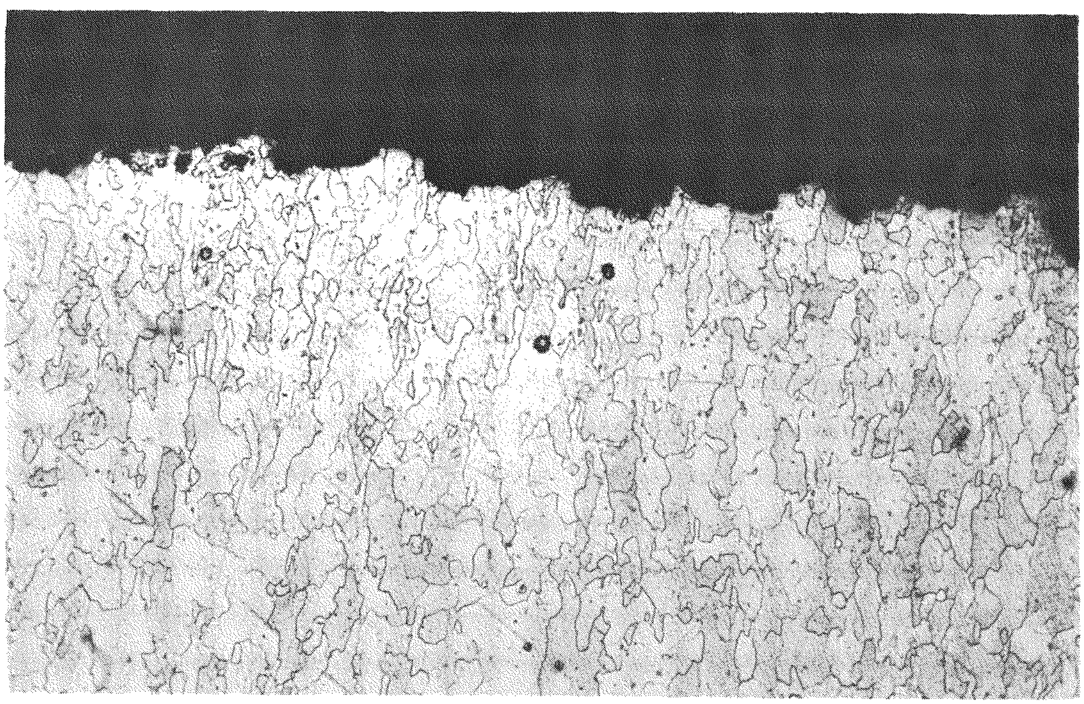
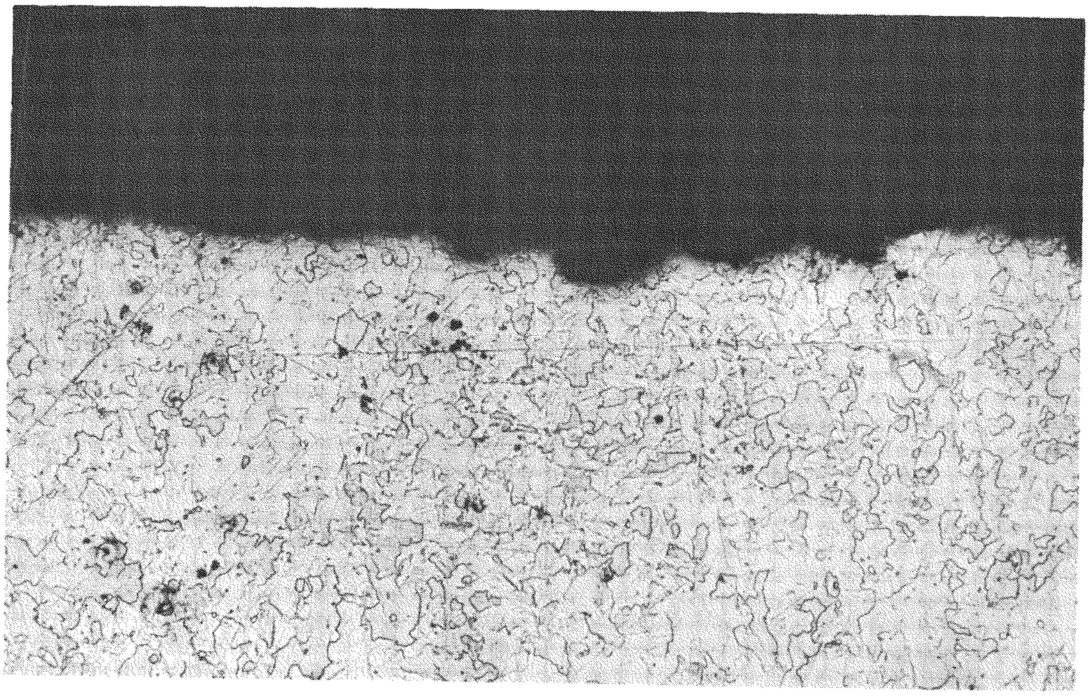
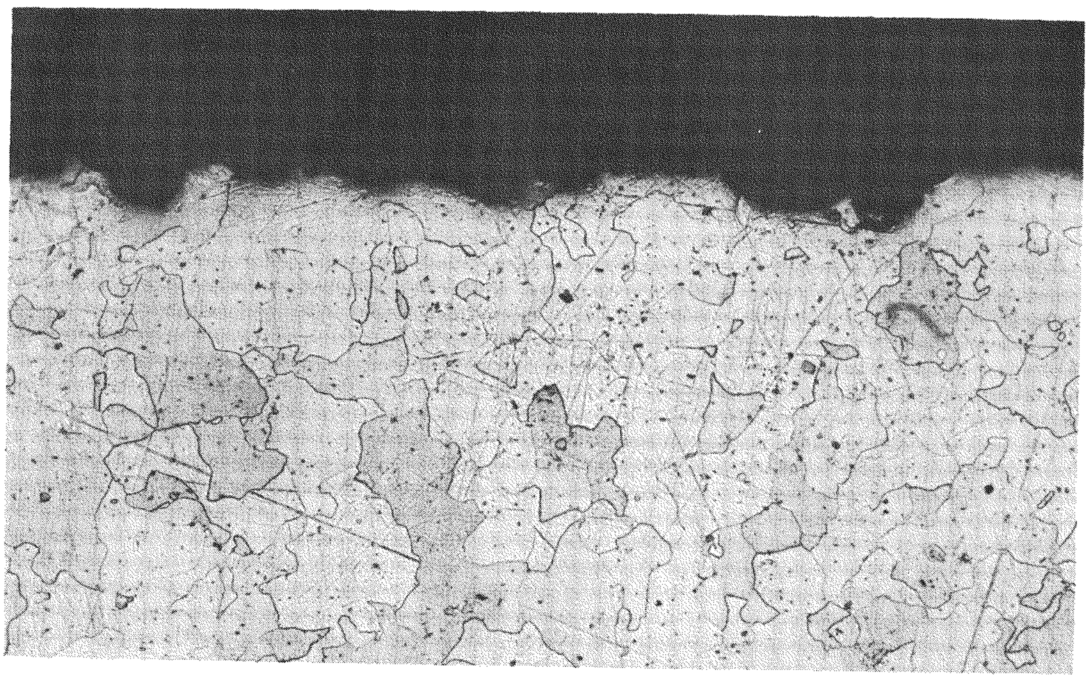


Fig. 11. Croloy-5 Ti Loop--Pitting in Condenser Tube No. 11



(Exposed to mercury at 1000° F) 250X

Fig. 12. Croloy-5 Ti Loop--Section Through the Cold Leg Return to the Boiler



(Exposed to mercury at about 1025° F)

Fig. 13. Croloy-5 Ti Loop--Section Through the Middle Coil of the Boiler

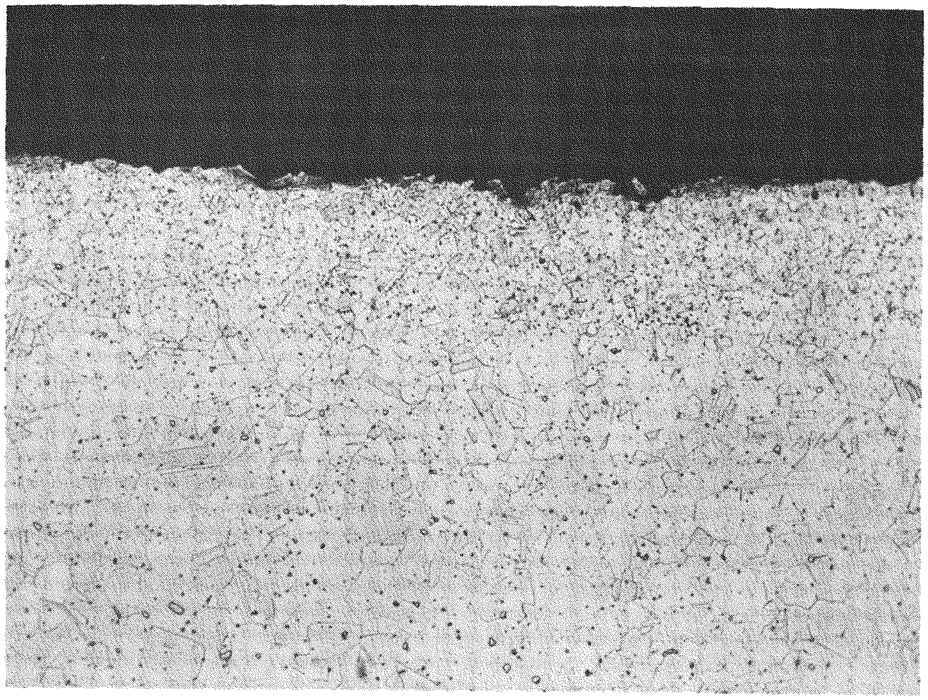


Fig. 14. Carpenter 20 Cb Loop--Tube Structure Adjacent to the Melted Area

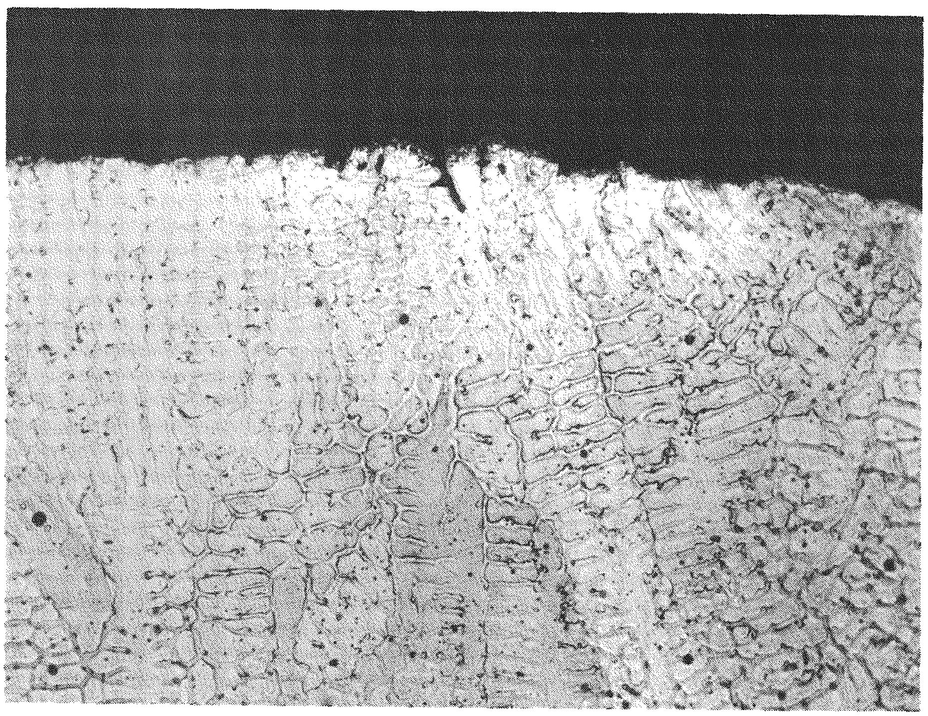


Fig. 15. Carpenter 20 Cb Loop--Melted Area of Tube Where Arcing Occurred

had been established, the loop could be restarted and design conditions met with little or no adjustments necessary. This was only true in the operational period prior to saturation of the mercury with dissolved solids. After some experience had been gained in the operation and behavior of the loops, one could accurately predict the effect of changing the loop variables. Both of the long time runs on the Type 347 loop (135 and 175 hours) were terminated by power failures. The first was plant wide, the second was due to the diodes burning out in the d-c power supply. The latter occurred so frequently that it prevented any long time runs. The problem was not solved until one week before the work was terminated.

When the loop shut itself down during the power outage, it could not be brought back to design parameters. The valve was removed and a light gray coating was found plated on the valve stem. An analysis of this coating showed it to contain 78% Fe, 21.5% Cr and traces of Ni, Nb and Mn. A spectrographic analysis of the mercury drained from the loop showed a strong Cr trace with other constituents on the borderline of spectrographic sensitivity. The scum from the mercury analyzed 73% Fe, 18.5% Cr, 7% Ni and traces of Nb, Mn and Si.

It must be noted that care should be taken to prevent the boiler coil from shifting and thereby touching the Globar heating elements. This results in arcing through the tubing.

Failure of the Type 347 stainless steel loop occurred when the outlet leg of the boiler ruptured. A four-inch long section of the tube just outside the top coil was expanded around the point of rupture. The loop had operated over periods of 8, 8, 135 and 175 hours for a total of 326 hours before the failure. Metallographic sections were taken from the top, middle and bottom loops of the coil as well as from the ruptured area. There was no attack at the bottom of the boiler coil as shown in Figs. 16 and 17. About half way up the coil where the temperature was some 700 or 800° F higher and liquid mercury was converted to vapor, some corrosion occurred as shown in Figs. 18 and 19. Farther up the tube near the top, where the temperature was about 1350° F and dryer mercury vapor was in contact with the tube, the general attack noted in the middle coil is not present. However, there is a continuous crack running longitudinally along the tube as shown in Figs. 20 and 21. Figure 22 is an enlargement of the crack shown in Fig. 21.

The ruptured area is shown in Fig. 23. The numerous cracks that appear in the photograph are attributed to the overall expansion of the tubing just prior to its rupturing. Figure 24 shows a transverse section through the rupture. The continuous crack in the upper boiler area was attributed to the severe cold work of the tubing. The crack was located along the outer wall of the tube where the greatest amount of cold work occurred. It appears that this area which was cold worked extensively was susceptible to mercury corrosion.

(Exposed to mercury at 350° F) 75X

Fig. 16. Type 347 Stainless Steel Boiler Two Coils from Bottom (cold end about 350° F)

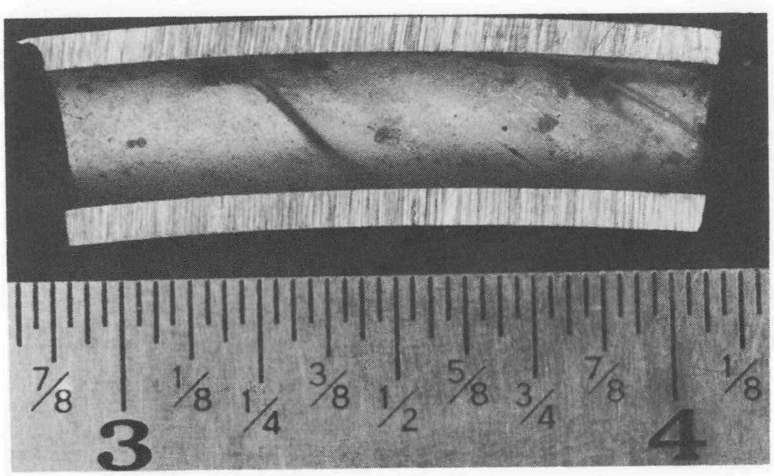



Fig. 17. Type 347 Stainless Steel Inside Surface of Tube At Same Locations as Fig. 16

(Exposed to mercury at about 1050° F) 75X

Fig. 18. Type 347 Stainless Steel Boiler--Middle Coil

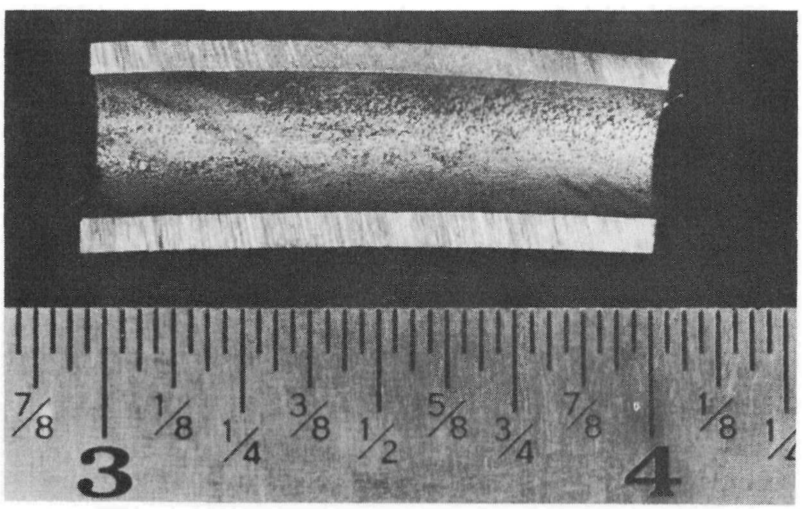
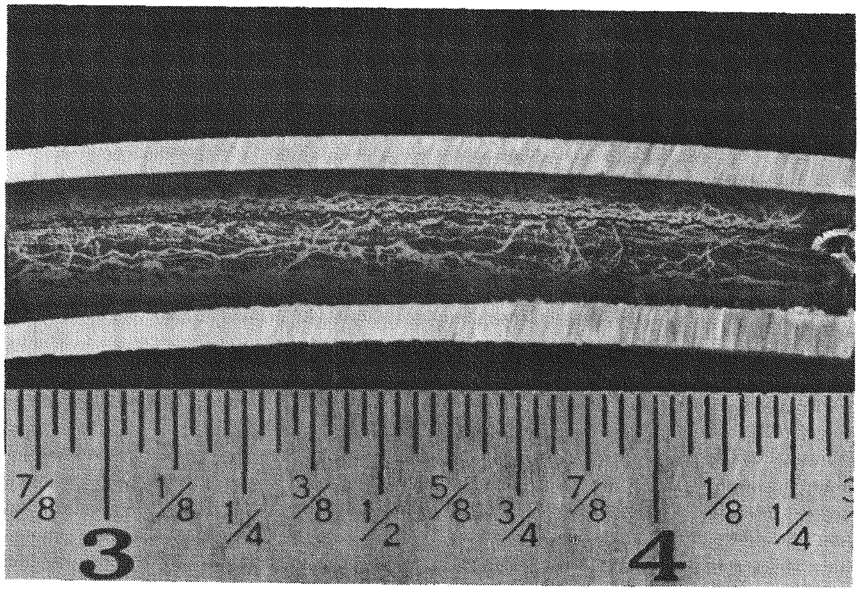



Fig. 19. Type 347 Stainless Steel--Inside Surface of Stainless Steel Tube at Same Location as on Fig. 18




(Note longitudinal crack; exposed to mercury vapor at 1350° F)

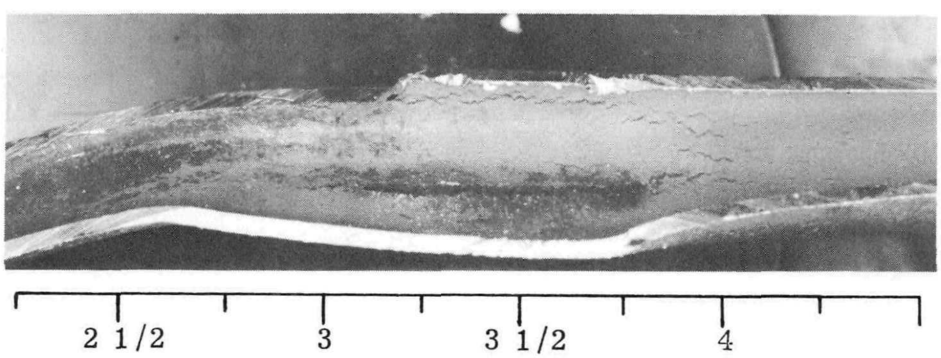
Fig. 20. Type 347 Stainless Steel Boiler--Two Coils from Top (hot) End--Inside Surface of Stainless Tube

75X

Fig. 21. Type 347 Stainless Steel--Transverse Section Through Longitudinal Crack Shown in Fig. 20

250X

Fig. 22. Type 347 Stainless Steel--Enlargement of Area Shown in Fig. 21




(Exposed to mercury vapor at 1350° F)

Fig. 23. Type 347 Stainless Steel--Inside Surface of Tube at Rupture on the Exit Leg

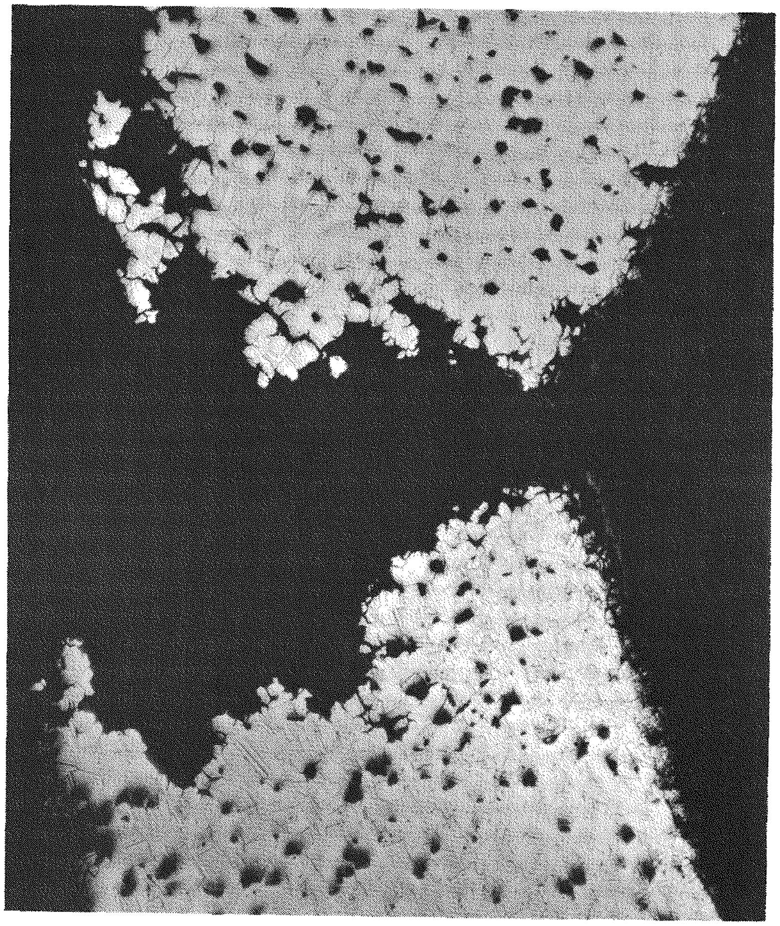
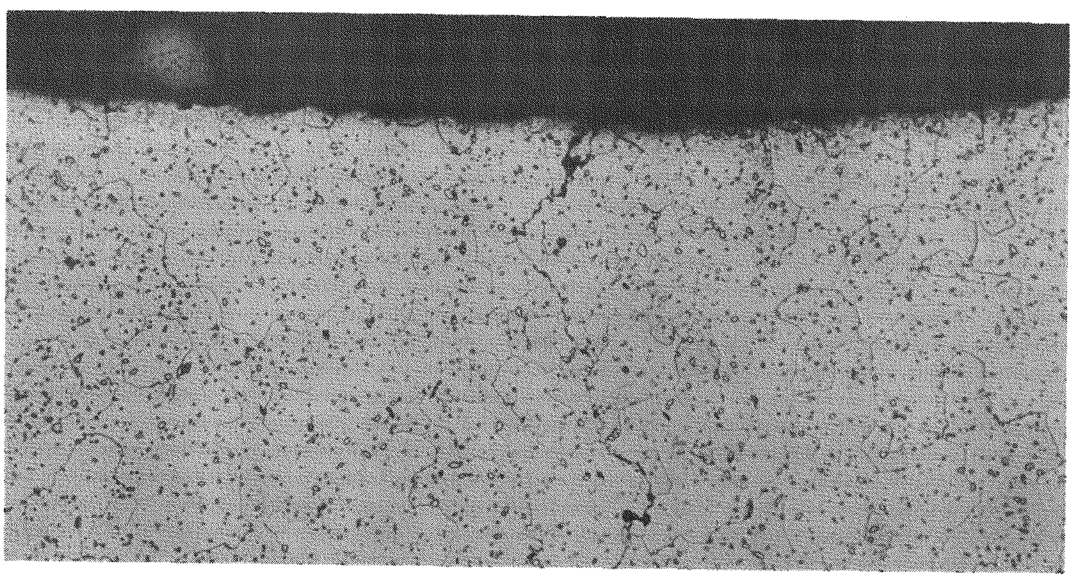


Fig. 24. Type 347 Stainless Steel Boiler--Transverse Section Through One End of Rupture on Exit Leg



(Note partial sealing of crack)

Fig. 25. Type 446 Stainless Steel Boiler, (exposed to Mercury at 1050° F)

## E. TYPE 446 STAINLESS STEEL BOILER COIL

Much the same problems that were encountered with the Type 347 stainless steel loop were encountered with the Type 446 stainless steel loop. The discussion will not be repeated.

This loop did not fail. It was shut down after operating periods of 8, 8 and 125 hours due to multiple leaks in the Swagelok fittings which could not be controlled. Examination of the boiler coil after disassembly revealed the transgranular cracks shown in Figs. 25 and 26. Some of the cracks, which occurred only on the inner wall of the tubing, appear to be partially healed. This indicates that the cracks probably originated during fabrication of the tubing. The subsequent bending of the tubing, aided by the embrittlement due to the sigma phase, may have reopened some of these cracks.

Metallographic sections of the unbent portions of tubing from the same lot showed sigma phase, but no cracks were found (see Fig. 27). The fact that cracks were not found is not conclusive proof that none were there. It is difficult to say what role, if any, mercury played in producing or enlarging these cracks. However, it is notable that, in general, the mercury did not attack the metal.

## F. NIOBIUM BOILER TEST

The niobium boiler ruptured after 50 hours, two coils below the top. Figure 28 shows the outside of the Type 347 stainless steel clad at the rupture point. Figure 29 shows the perforated niobium tube with the stainless steel clad removed. A transverse section of the rupture area, Fig. 30, shows some of the remaining corrosion products between the tubes. The probable cause of failure was atmospheric oxidation from outside the tubing--not mercury corrosion. The argon blanket normally maintained in the furnace was somehow reduced in depth, exposing the upper loops of the boiler coil to atmospheric oxygen. Figure 28 shows that considerable external oxidation occurred. An imperfection in the stainless steel clad perforated, allowing oxygen to contact the niobium with resultant oxidation and the deposition of the reaction products. Examination of the as-received material showed that the stainless steel clad was weld-drawn tubing rather than seamless tubing. The failure occurred along this seam.

During the period of this test (50 hours), mercury had little effect corrosionwise on the niobium. Figures 31 and 32 show this. The dark areas in the photomicrographs are the mounting resin. The bimetal tubes separated when the metallographic sections were cut. The dark trace just inside the surface of the niobium which contacted mercury was made by the etchant.

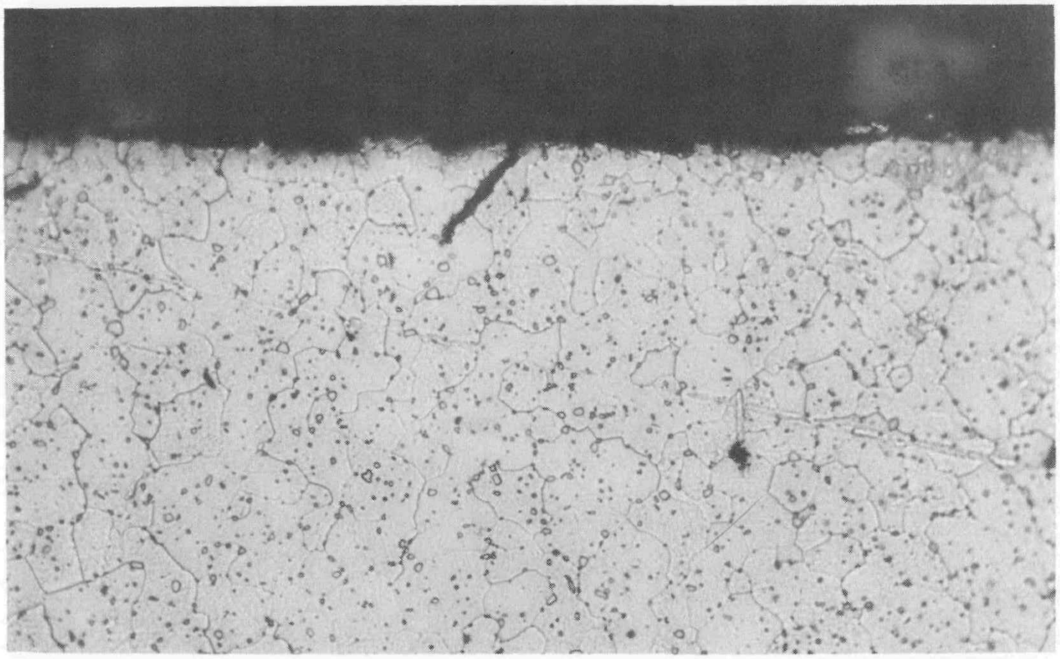
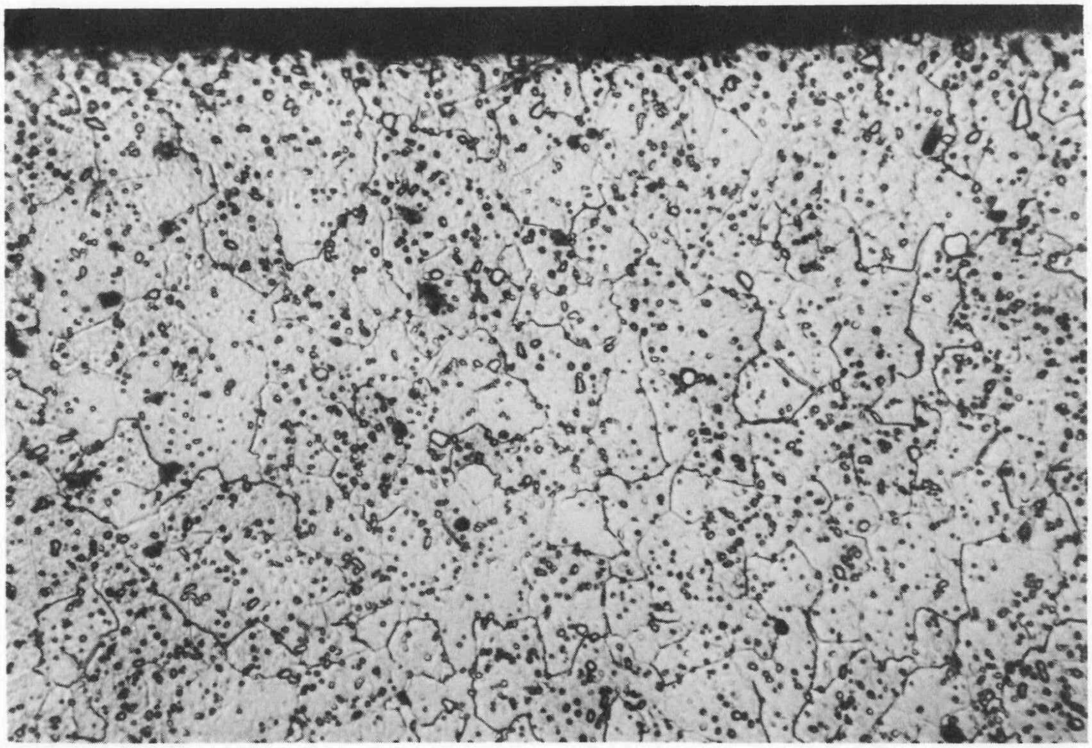
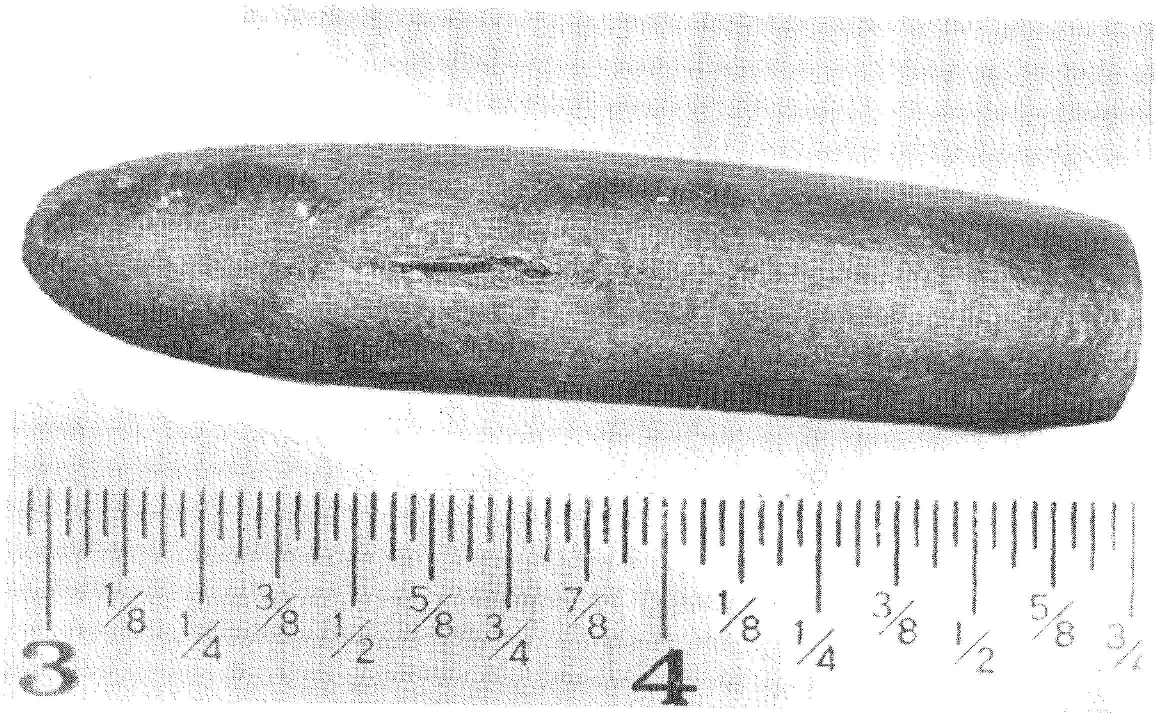


Fig. 26. Type 446 Stainless Steel Boil in Same Area as Shown in Fig. 25



(Note presence of sigma phase)

Fig. 27. Type 446 Stainless Steel--Unused Tubing from Same Lot as Loop



(Exposed to mercury vapor at 1350° F)

Fig. 28. Type 347 Stainless Steel Clad Niobium Boiler--Point of Rupture Two Coils from Top (Hot) End

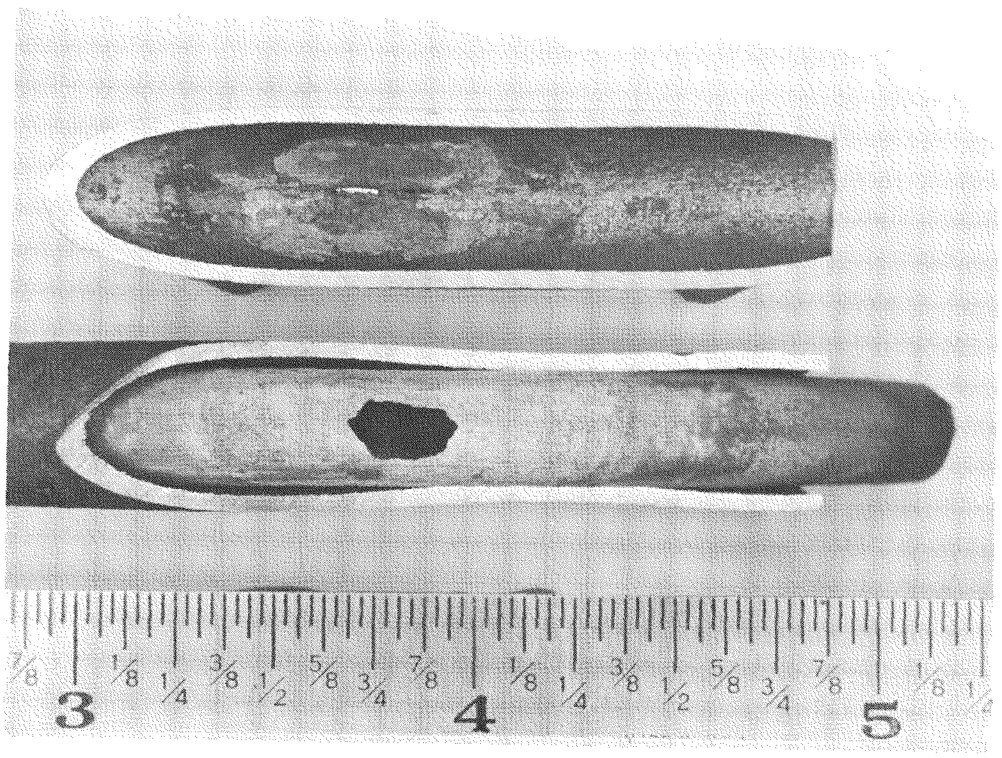
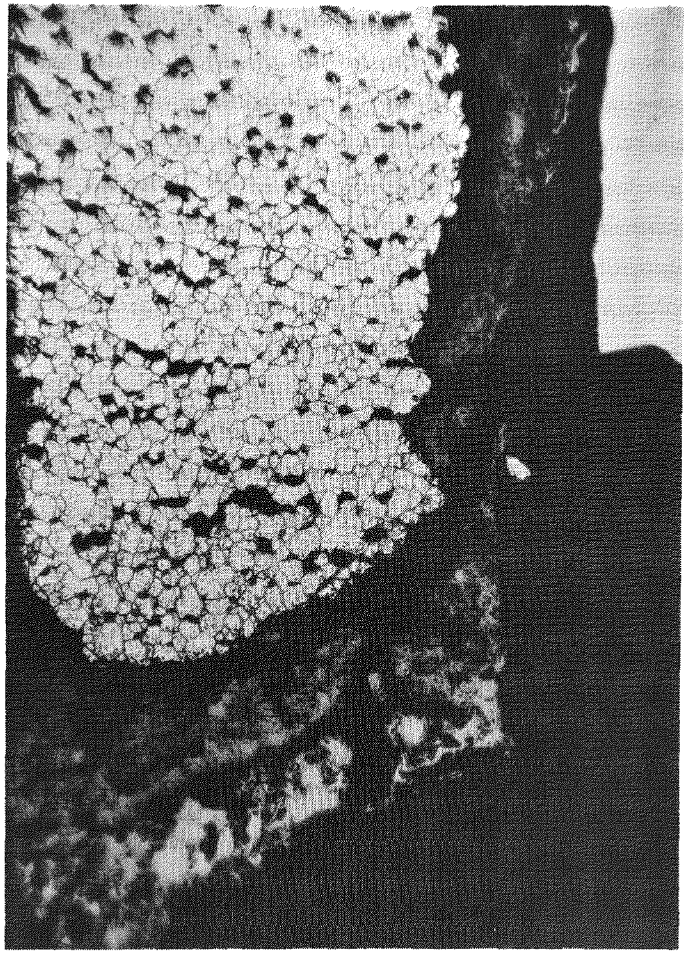
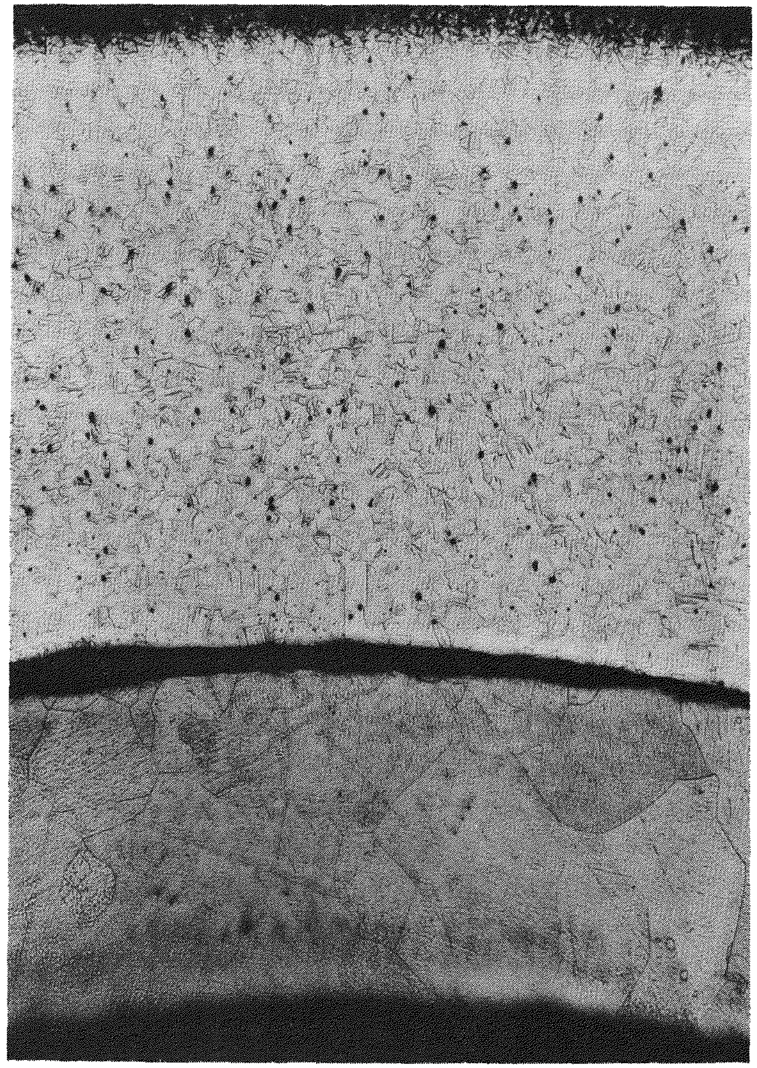


Fig. 29. Niobium Liner with Type 347 Stainless Steel Clad at Point of Rupture



(Exposed to mercury vapor at approximately 1350° F)

Fig. 30. Niobium Liner Clad with Type 347 Stainless Steel--Sample Taken Two Turns from Top of Coil at Point of Rupture



(Media-mercury at 350° F)

Fig. 31. Niobium Liner Clad with Type 347 Stainless Steel--Sample Taken Two Turns from Bottom of Coil

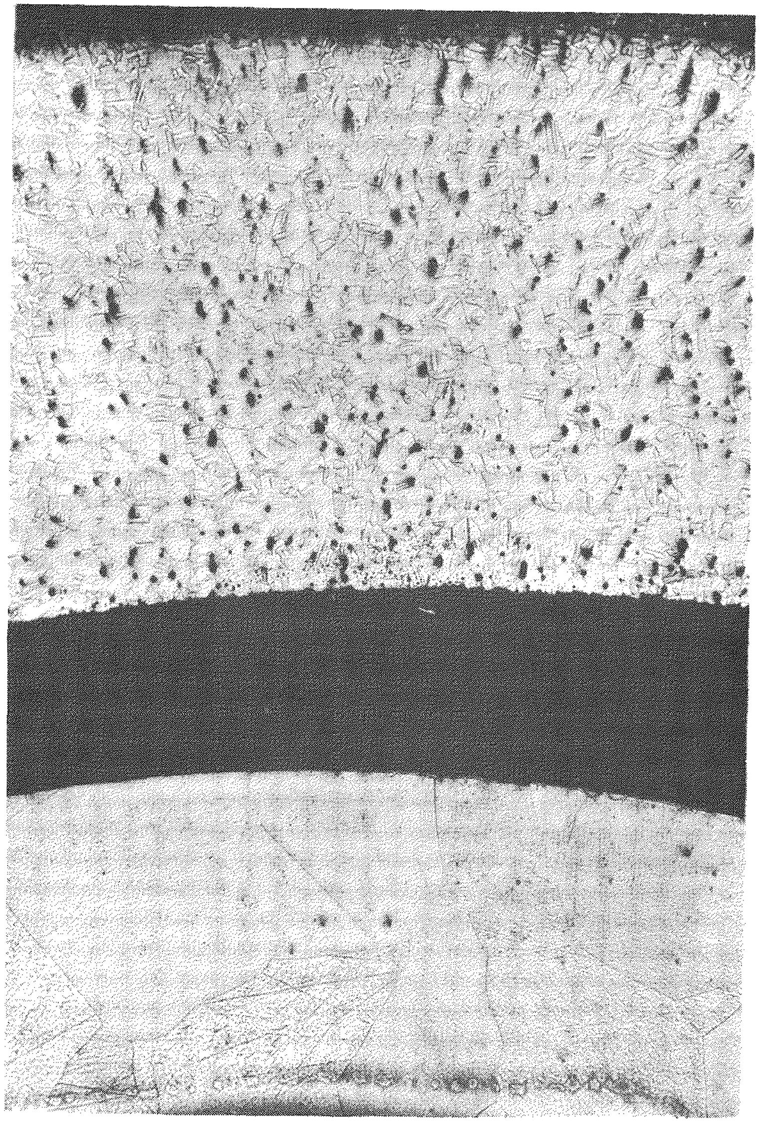


Fig. 32. Niobium Liner Clad with Type 347 Stainless Steel--Sample Taken at Middle of Coil

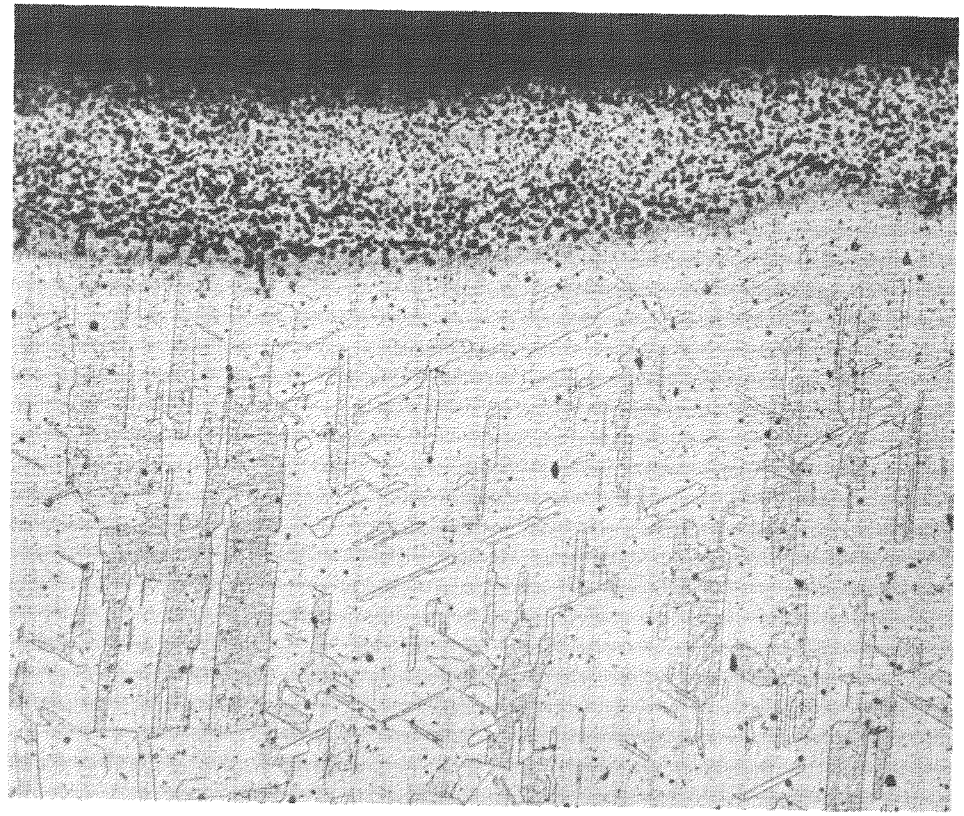


Fig. 33. Type 347 Stainless Steel Loop--Hot Leg Immediately Beyond Boiler Outlet, Temperature 1350° F

## G. EXAMINATION OF TYPES 347 AND 446 STAINLESS STEEL LOOPS, EXCLUSIVE OF BOILER COILS*

After completing the niobium boiler coil test, the Type 347 stainless steel loop was disassembled for examination. Metallographic sections were taken from the hot and cold legs and the condenser. Figure 33 is a cross section of the tubing in the hot leg just beyond the boiler coil rupture. It is obvious that a reaction has occurred. The mercury has penetrated the tubing to an average depth of six to seven mils with a maximum penetration of about 20 mils in isolated areas. A spectrographic analysis of the corroded area shows a depletion of nickel. The entire hot leg through the first loop in the condenser coil suffered the same penetration. A short section of the hot leg was cut longitudinally and flattened in a vise. Mercury droplets were squeezed from the penetrated layer as shown in Fig. 34. The entire corroded area was permeated with mercury.

The condenser coil is shown in Fig. 35. A metallographic section from the top condenser coil, Fig. 36, shows the continuation of the attack noted in Fig. 33. Farther down the coil the metal is unattacked. Figure 37 shows the middle of the coil, and Fig. 38 pictures the bottom of the coil where the temperature was about 350° F. The remainder of the cold leg was also unaffected.

No corrosion was expected in the hot leg because the tubing was exposed to dry mercury vapor, i.e., there were, supposedly, no condensed droplets present. Generally, it was postulated that the most severe corrosion would occur in the boiler in the area of the interface. The area of the rupture did not show a general attack like that just beyond the rupture. This indicates that the failure was probably due to an imperfection in the tubing.

The entire Type 446 stainless steel loop was apparently unaffected by the mercury. The top, middle and bottom of the condenser coil, shown in Figs. 39, 40 and 41, show no signs of attack, as was previously noted in the discussion of the Type 446 boiler coil. It should be noted that the material was very brittle. A longitudinally cut section shattered when an attempt was made to flatten it in a vise.

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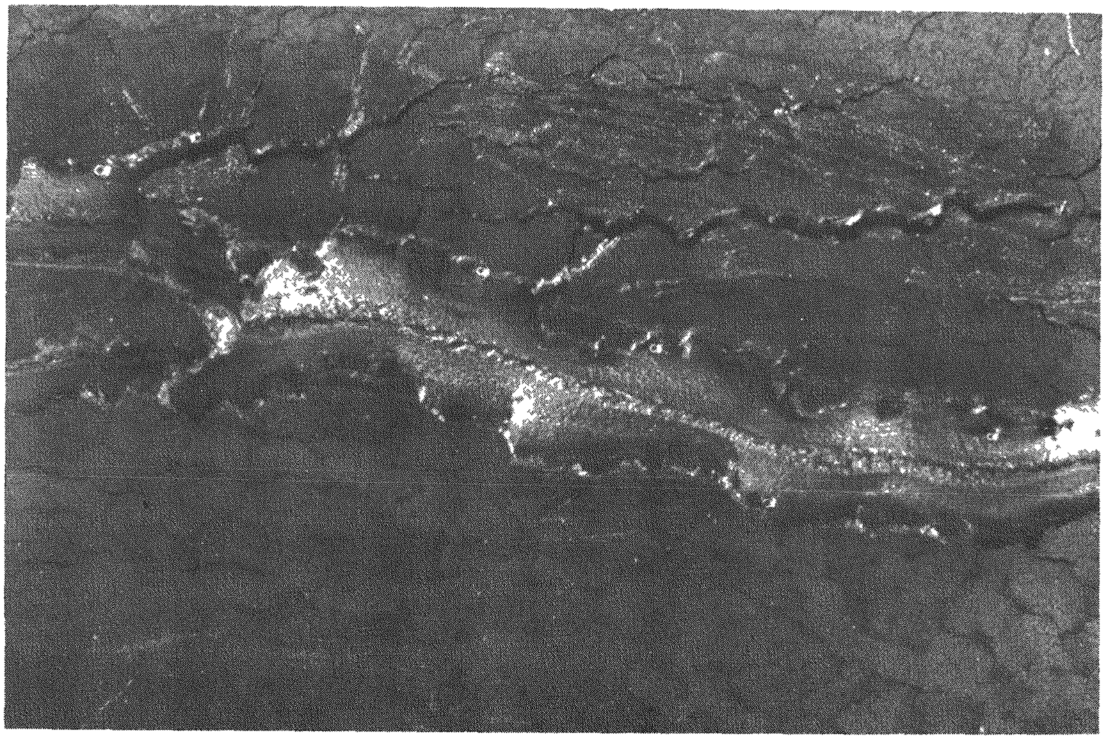
*J. Neri



10X

Fig. 34. Type 347 Stainless Steel Condenser--Top (hot) Coil, (exposed to mercury vapor at 1350° F)

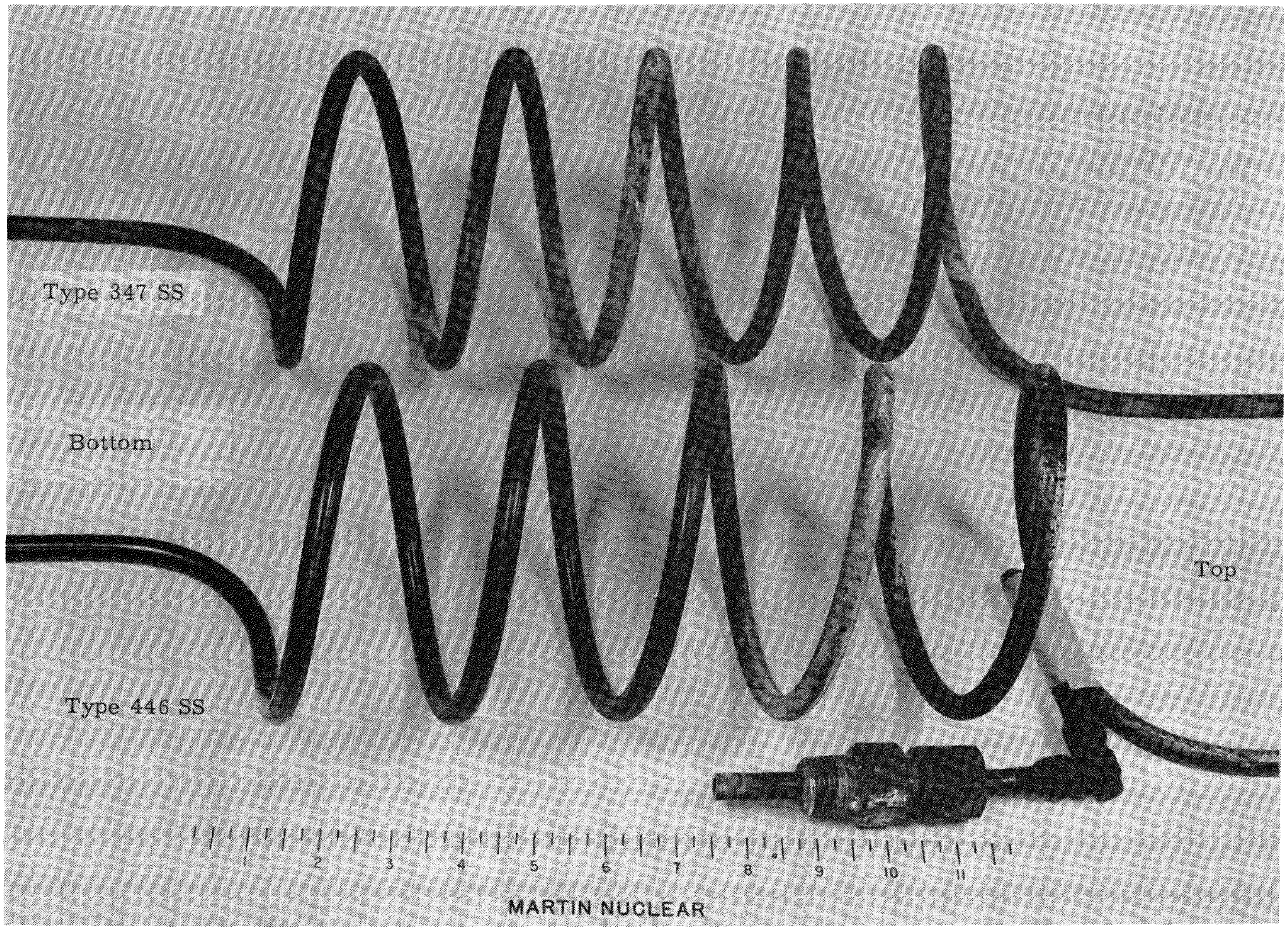
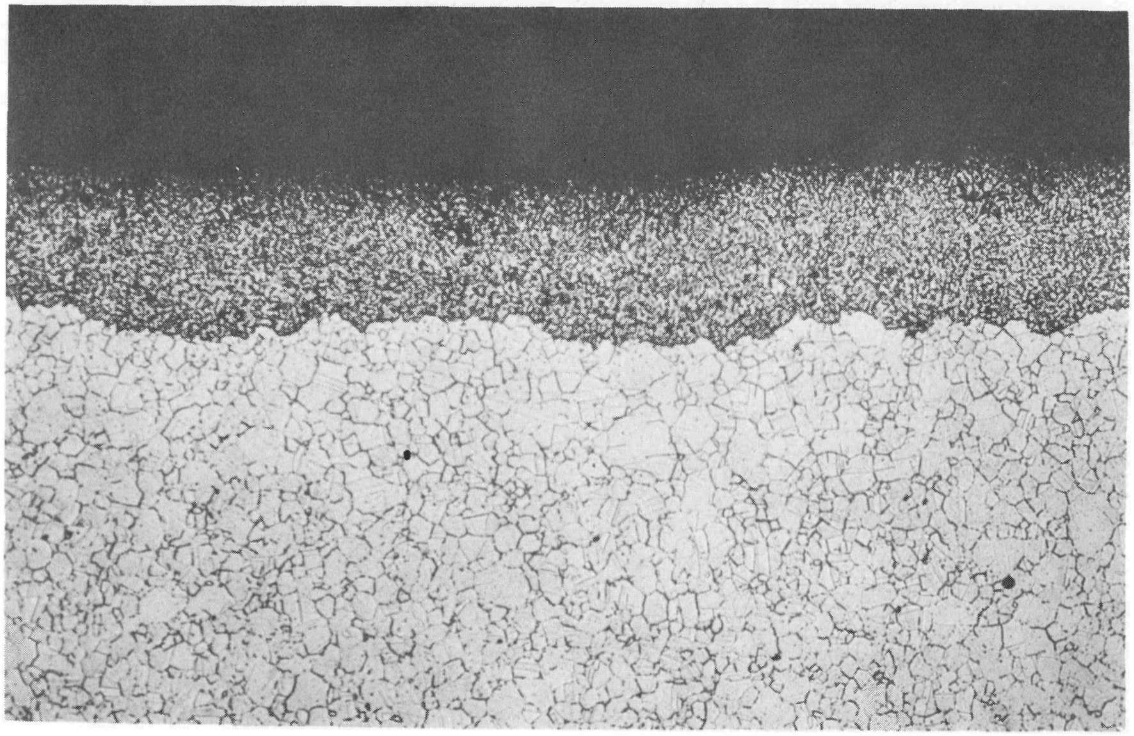



Fig. 35. Condenser Coils



(Note penetration of the tubing by mercury)

Fig. 36. Type 347 Stainless Steel Condenser at Area Shown in Fig. 34

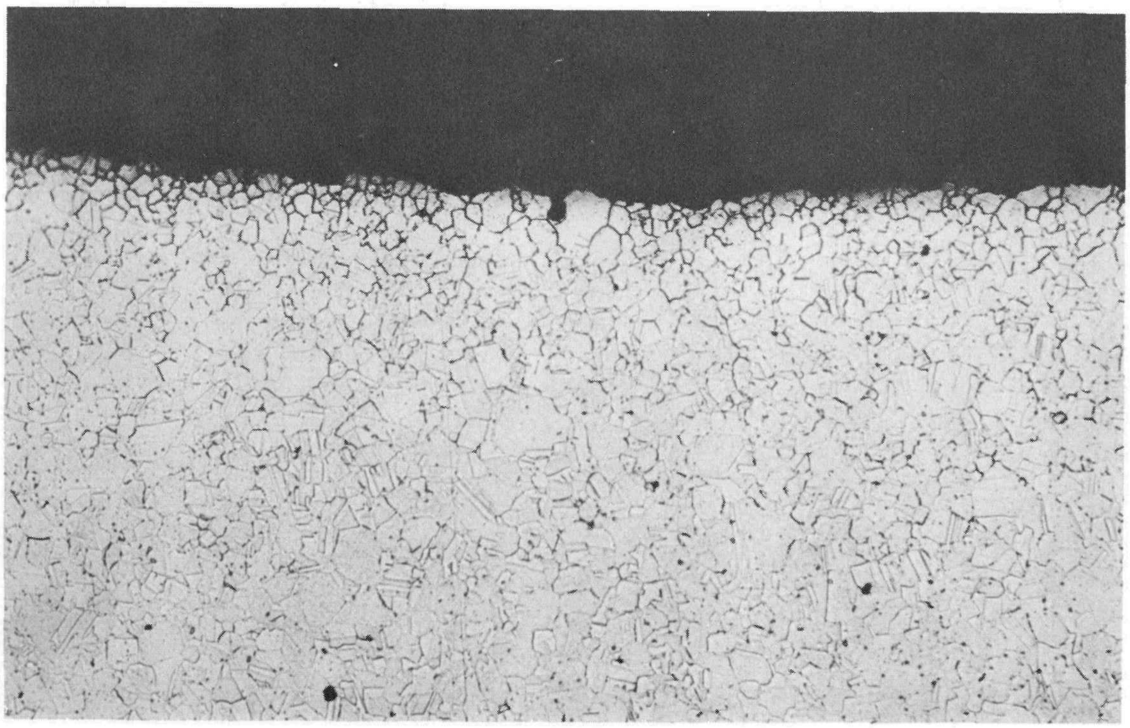
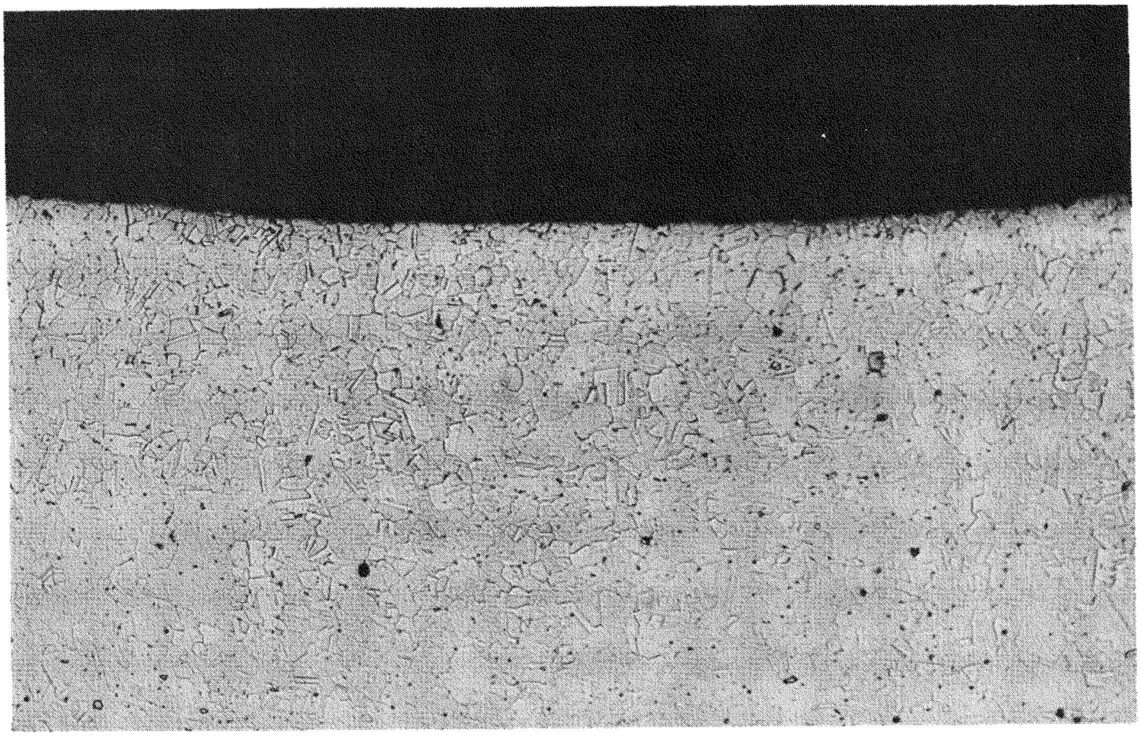
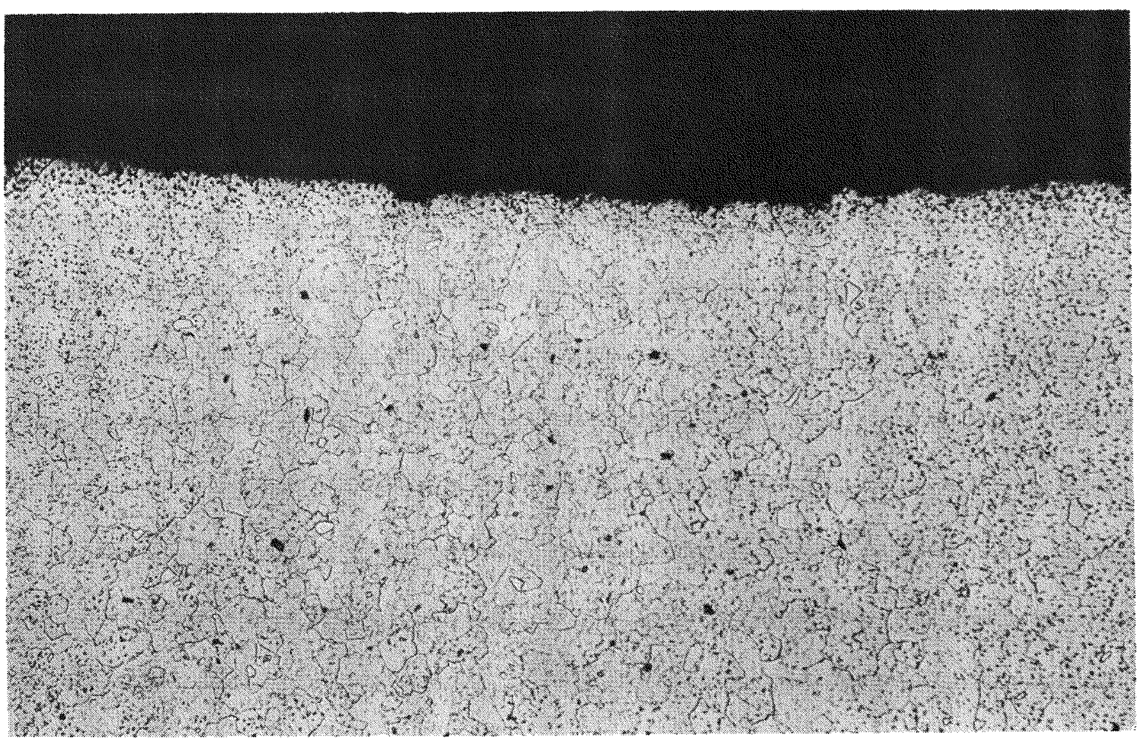


Fig. 37. Type 347 Stainless Steel Condenser--Middle Coil, Exposed to Mercury Droplets and Mercury Vapor in 500° to 700° F Range 250X



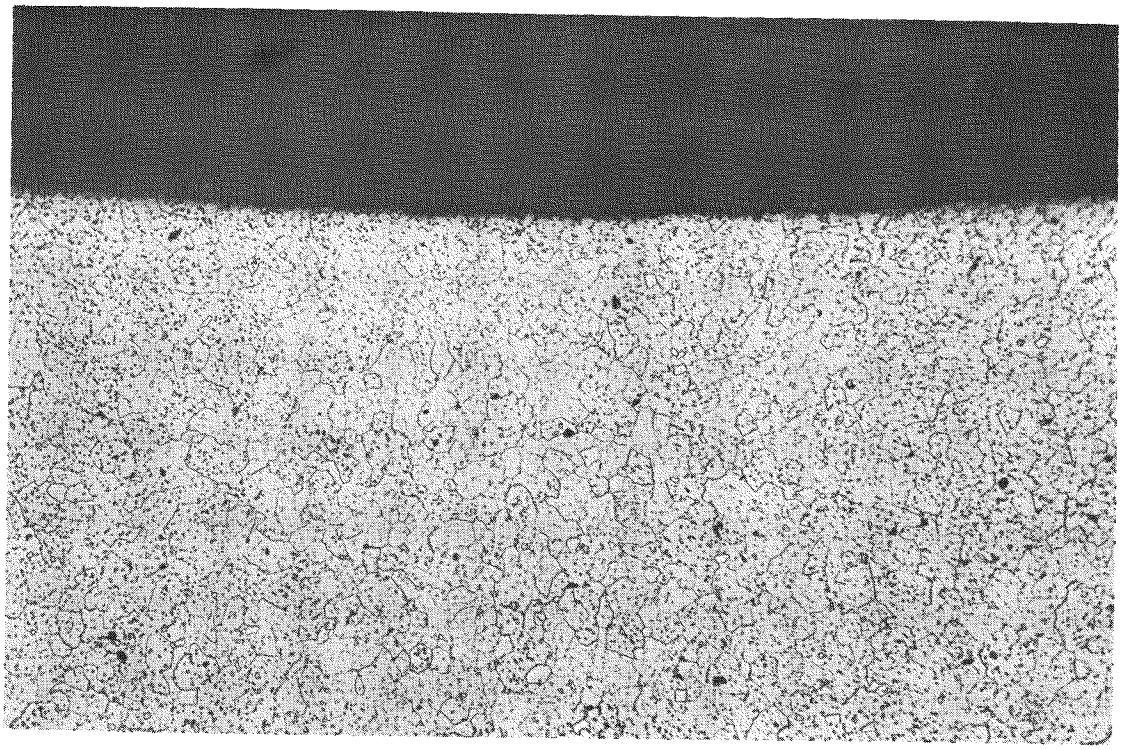
250X

Fig. 38. Type 347 Stainless Steel Condenser--Bottom Coil, Exposed to Mercury at About 300° to 350° F



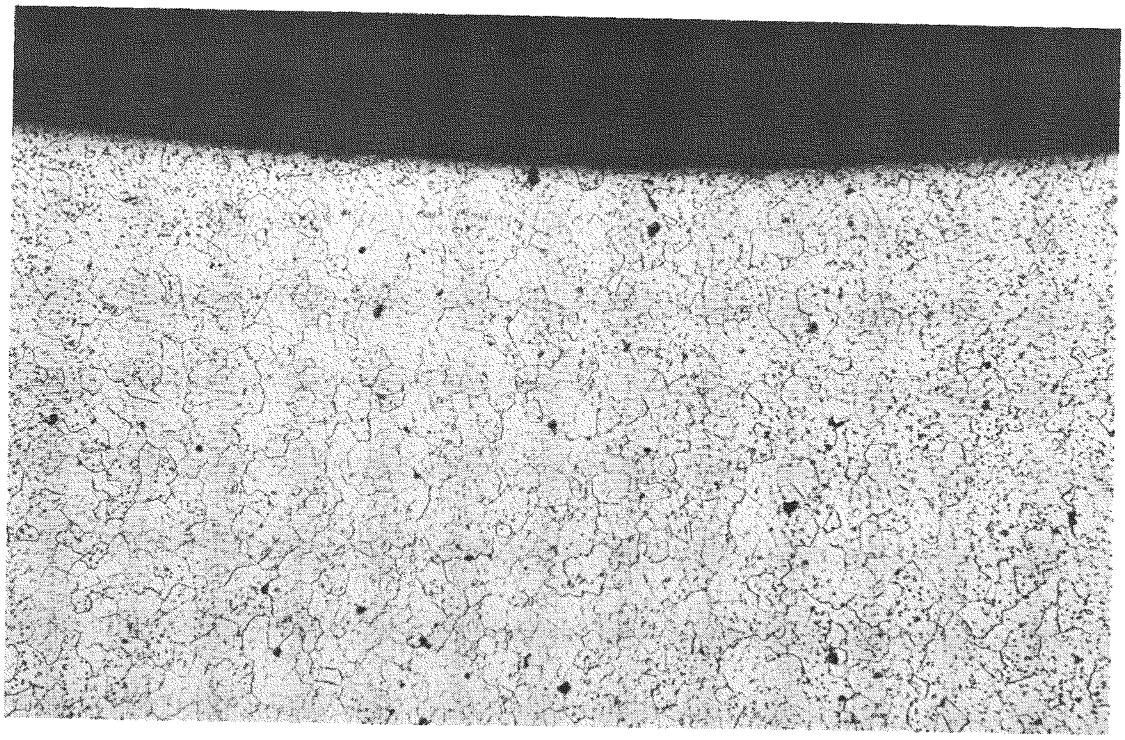
250X

Fig. 39. Type 446 Stainless Steel Condenser--Top Coil, Exposed to Mercury Vapor at About 1350° F



250X

Fig. 40. Type 446 Stainless Steel Condenser--Middle Coil, Exposed to Mercury Droplets and Vapor in the Temperature Range of 500° to 700° F



250X

Fig. 41. Type 446 Stainless Steel Condenser--Bottom Coil, Exposed to Mercury at 300° to 350° F

## IV. CONCLUSIONS

The experience with the Croloy alloys 5-Si and 5-Ti eliminated them from further consideration. The rate of attack was much greater than anticipated from reports in the literature and the static tests. Little practical data were gained from the Carpenter 20 Cb loop. The attack which did occur was not severe; however, the exposure time was short and never at design temperatures.

The results of the Type 347 stainless steel loop tests show that its ability to contain mercury at high temperatures is doubtful. Disregarding the rupture, which was attributed to a material imperfection, the severe attack along the entire hot leg eliminated Type 347 from consideration.

The Type 446 stainless steel loop contained fine cracks throughout which were attributed to the sigma phase formed during fabrication. These cracks might have caused eventual failure of the loop but the material itself appeared to be unaffected by the hot dynamic mercury.

The niobium coil failed before any significant evaluation of its ability to resist mercury corrosion could be obtained. Considering the oxidation properties of niobium at elevated temperatures, it is obvious that an effective means of providing absolute exclusion of oxygen is required.

It was unfortunate that this effort was discontinued. It was considered that the design, after considerable evolution, was sound and had proved itself on at least four occasions. Unfortunate material failures and power outages were the only things which caused loop shutdown. A few changes in the design would permit smoother operation, i.e., weld all connections in the cold leg as well as the hot leg, redesign the pressurizer to permit charging of mercury directly to the pressurizer and, finally, provide a positive maintenance of the argon blanket over the boiler coil. It is very important to be sure that all of the materials to be tested are properly fabricated and sound, i.e., the sigma phase in the Type 446 and the severe cold work in the Type 347 should be avoided.

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