NASA CONTRACTOR REPORT

NASA CR-217

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OPERATION OF A FORCED CIRCULATION, CROLOY 9 M, MERCURY LOOP TO STUDY CORROSION PRODUCT SEPARATION TECHNIQUES

by D. B. Cooper and E. J. Vargo

Prepared under Contract No. NAS 3-2538 *by*
THOMPSON RAMO WOOLDRIDGE, INC.
Cleveland, Ohio
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • APRIL 1965

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FOREWORD

This report is concerned with the construction, operation, and evaluation of a forced circulation mercury loop designed to investigate corrosion product separation techniques in a Croloy 9M system. The work was performed between May, 1963, and July, 1964, in support of the SNAP 8 program under NASA Contract NAS 3-2538.

The work was performed by the Materials Research and Development group of the Electromechanical Division of TRW under the supervision of E. J. Vargo and technical direction of D. B. Cooper. Other contributors to the program included R. C. Schulze, V. R. Brittain, P. D. Metz, and R. D. Shorf. The report was prepared as TRW Report ER-6123.

The program was administered by P. L. Stone and J. P. Merutka of the SNAP 8 Project Office at the NASA-Lewis Research Center.

OPERATION OF A FORCED CIRCULATION, CROLOY 9 M, MERCURY LOOP TO STUDY CORROSION PRODUCT SEPARATION TECHNIQUES

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THOMPSON RAMO WOOLDRIDGE INC.
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SUMMARY

A Croloy 9M, forced circulation, mercury loop was designed and operated for a total of 2918 hours at an average boiling temperature of 1074°F. Corrosion product separators were included in both the vapor and liquid sections of the system and were evaluated for their effectiveness in reducing problems associated with mass transfer in mercury systems. Corrosion data for this system were found to agree favorably with previously reported data for Croloy 9M. The greatest attack was found in the low vapor quality regions of the boiling section of the loop and in the superheater outlet where condensation is believed to have occurred. The dry portions of the superheater suffered negligible attack. The degree of corrosion which existed in the condenser and subcooler could not be detected metallographically because of the extremely rough surface of the as-received material.

The separators in the vapor portion of the system removed 54.3 percent of the corroded metals found in the system, while the separator in the liquid region removed 24.7 percent of the corroded metals.

Carbon diffusion was noted in the Type 316 SS - Croloy 9M composite tubing at temperatures above 1200°F. The diffusion of carbon from the Croloy 9M to the Type 316 SS resulted in grain coarsening of the Croloy 9M and a profusion of carbides in the Type 316 SS. The composite tubing also exhibited excessive creep after 2918 hours at 1405 ± 10°F, apparently a result of metallurgical changes in the Type 316 SS.

Deposition and corrosive and/or erosive attack were observed, after 448 hours of loop operation, in a Croloy 9M throttling valve located at the superheater outlet.

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INTRODUCTION

The materials problems associated with the design and operation of a mercury Rankine cycle power system are fairly well defined. With the exception of refractory metals, no known material is entirely resistant to corrosive attack by mercury at high temperatures. One of several materials which have indicated more corrosion resistance to mercury than others is a chromium alloy steel known as Croloy 9M*, which has been chosen as the SNAP 8 reference material. Capsule tests have yielded data that indicate that, for temperatures below 1200°F, the corrosion resistance of this material may be adequate for a Rankine cycle turbo-generator power system (1). However, the generation of corrosion products in a Croloy 9M system still exists, and this results in a number of secondary problems which seriously threaten the long-term reliability of the system. These problems center around the transfer of the corrosion products from one portion of the system to another, with the resultant effect that the efficiency of the system may be reduced. More serious than this is the fact that regions such as the turbine may become plugged with corrosion products. One solution to the problem is that of continuous separation of the corrosion products from the working fluid by such techniques as chemical gettering and the use of magnetic fields. Corrosion product separation work at TRW on thermal convection loop systems with flow rates of 10 to 40 pounds per hour has shown that as much as 85 percent of the corrosion products generated can be removed in such a loop (2). The operation of a forced circulation Haynes alloy No. 25 loop at TRW has also demonstrated that separation of corrosion products in a mercury system is quite feasible (3).

In a system such as SNAP 8, continuous separation of the corrosion products from the working fluid appears to be the most feasible method for avoiding mass transfer difficulties. In order to evaluate the effectiveness of corrosion product separation, a forced circulation, Croloy 9M, mercury loop was designed, constructed, and operated. The loop was designed for long-term (2500 hours) operation at conditions simulating those found in the SNAP 8 system and included three corrosion product separators. Evaluation was concerned with corrosion and mass transfer effects as well as the effectiveness of the corrosion product separation techniques employed.

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*Nominal analysis:	Chromium	8.0-10.0%
	Molybdenum	0.9-1.1%
	Carbon	0.15%

SYSTEM DESCRIPTION

A. LOOP

The design parameters for the Croloy 9M loop were chosen so as to simulate the conditions in the SNAP 8 system, with the exception of mercury flow rate. These conditions included:

Boiler outlet temperature	1075°F
Superheater outlet temperature	1250-1300°F
Condensing temperature	675-700°F
Flow rate	95-100 pounds per hour

The design flow rate was approximately 1/115 of the flow rate in the SNAP 8 system.

Croloy 9M was the basic material of construction. Type 316 SS was used in low temperature (less than 100°F) regions of the loop for instrument sensing units and valving. Because of the poor oxidation resistance and poor high temperature strength of Croloy 9M at the OD surface temperature of 1405°F encountered in the superheater, the tubing for the high temperature portions of the system was clad with Type 316 SS. Figure 1 shows a schematic of the loop and is referenced during the following description.

1. Boiler and Superheater

The boiler and superheater sections of the loop were fabricated from 1.0 inch OD x 0.095 inch wall Type 316 SS-Croloy 9M duplex tubing, the Croloy 9M wall being 0.030 inch. Electrical resistance-type heaters were used to supply energy to these sections and were designed to supply maximum overall heat fluxes of 10,000 and 5,000 Btu/hr-ft^2 in the boiler and superheater, respectively. Actual fluxes used during operation were in the order of 5500 Btu/hr-ft^2 over ten feet of heated boiler tubing and 1900 Btu/hr-ft^2 over the 8.5-foot superheater length, including the corrosion product separators. The heaters were fabricated in place by wrapping Nichrome heater ribbon around refractory tubing which surrounded the loop tubing. A solution of a refractory cement in water was spread between the loop tubing and the refractory tubing to provide better heat transfer.

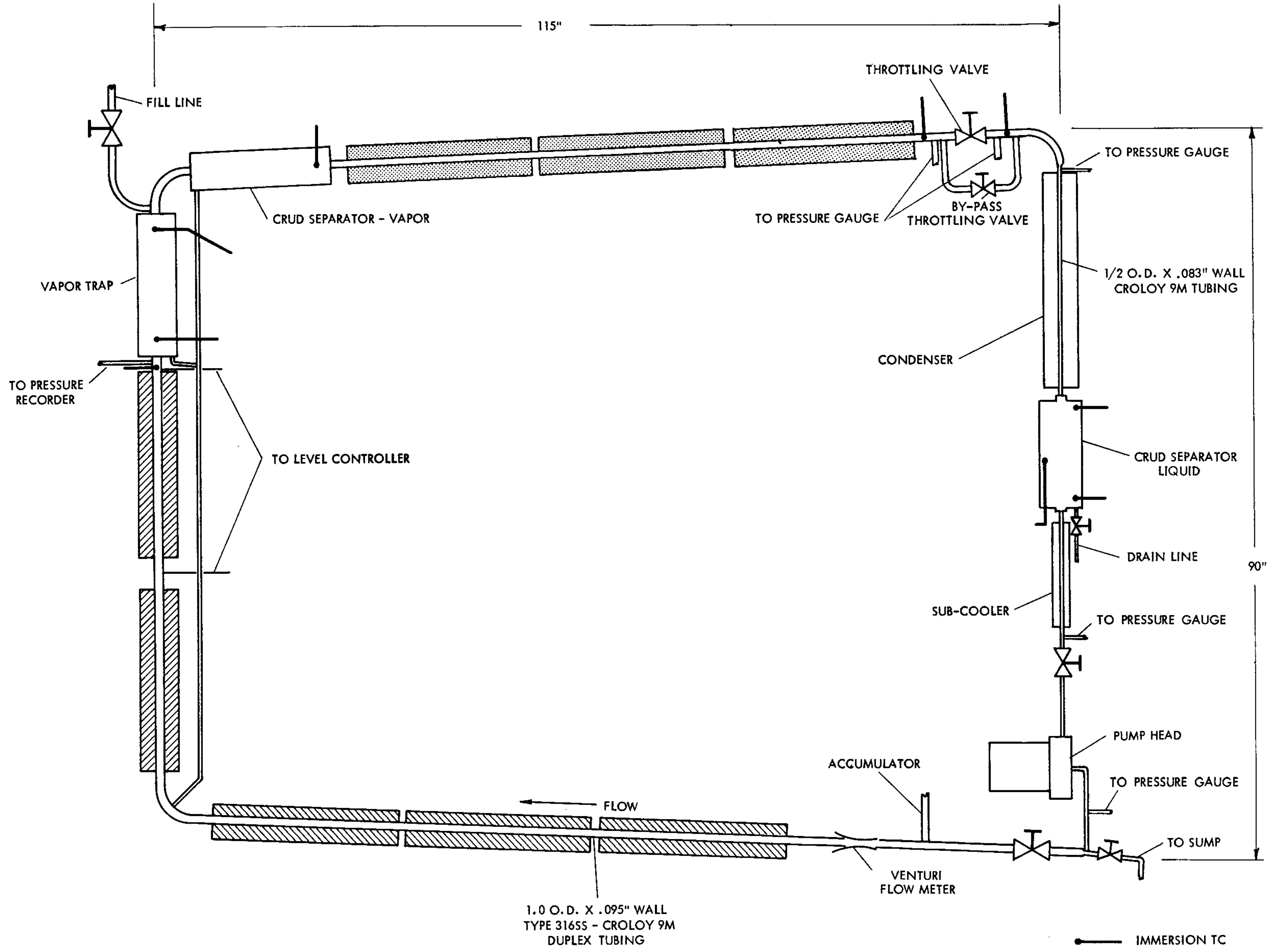


FIGURE 1. SCHEMATIC OF CROLOY 9M LOOP

The cement was also applied to the outside of the heater assembly to hold the Nichrome ribbon in place and to insulate the unit. In order to create turbulence and facilitate the heat transfer, swirl wire was used throughout the boiler and superheater. Croloy 9M wire of 0.062 inch diameter was coiled into a helix having an outside diameter of 0.810 inch and a pitch of 3.25 inches.

2. Condenser and Subcooler

The condenser and subcooler sections of the loop consisted of single Croloy 9M tubes having an outside diameter of 0.5 inch and a wall of 0.083 inch. Cooling of these sections was accomplished by both water and air. The 25.5-inch condenser was wrapped with four separate water cooling coils of equal length, and the entire unit was enclosed within a six-inch square manifold through which air was forced by a 200 CFM centrifugal blower. Cooling of the twelve-inch subcooler was effected by the same means, with the exception that only one water cooling coil was used.

3. Throttling Valves and Orifice

A bellows-sealed, manually-operated, throttling valve was located between the superheater and condenser sections and provided the required pressure differential in the system. A by-pass line was installed around the main throttling valve and contained an automatically controlled throttling valve which provided the required pressure drop should the first valve fail. The two valves were identical with the exception of control. All parts of the valves were fabricated from Croloy 9M except the bellows, which were of Type 321 SS construction. The two valves were removed after 448 hours of loop operation when leaks developed in the bellows of both valves (see "Loop Operation"). At this time the inlet side of the by-pass line was also removed from the system, while the outlet side of the by-pass line was sealed with a Croloy 9M cap. In order to provide the required pressure differential, an orifice was installed in the system. The orifice plate was fabricated from Croloy 9M and was 0.125 inch thick. The orifice was 0.052 inch in diameter. A schematic is given in Figure 2, showing the revisions in the system.

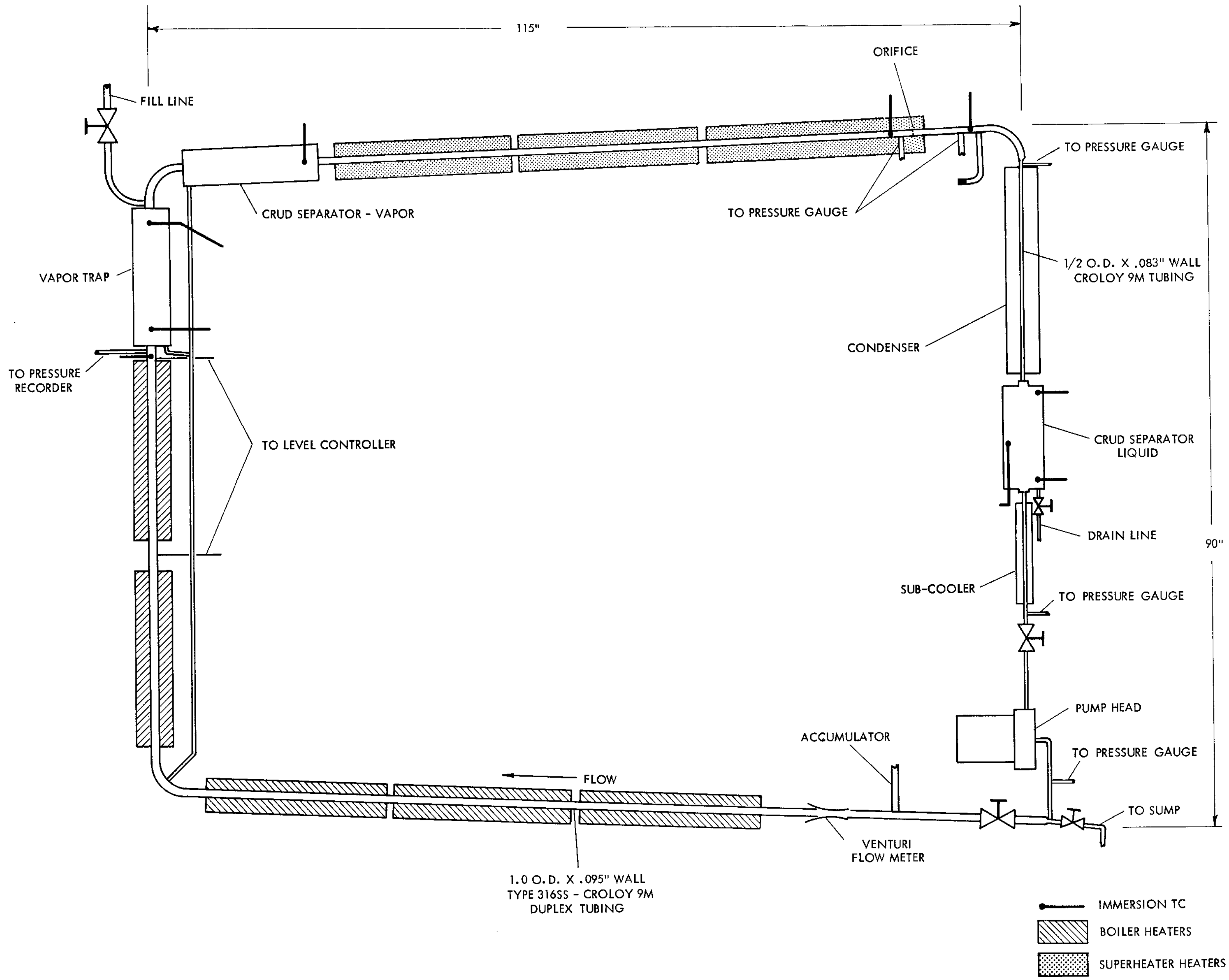


FIGURE 2. REVISED SCHEMATIC OF CROLOY 9M LOOP

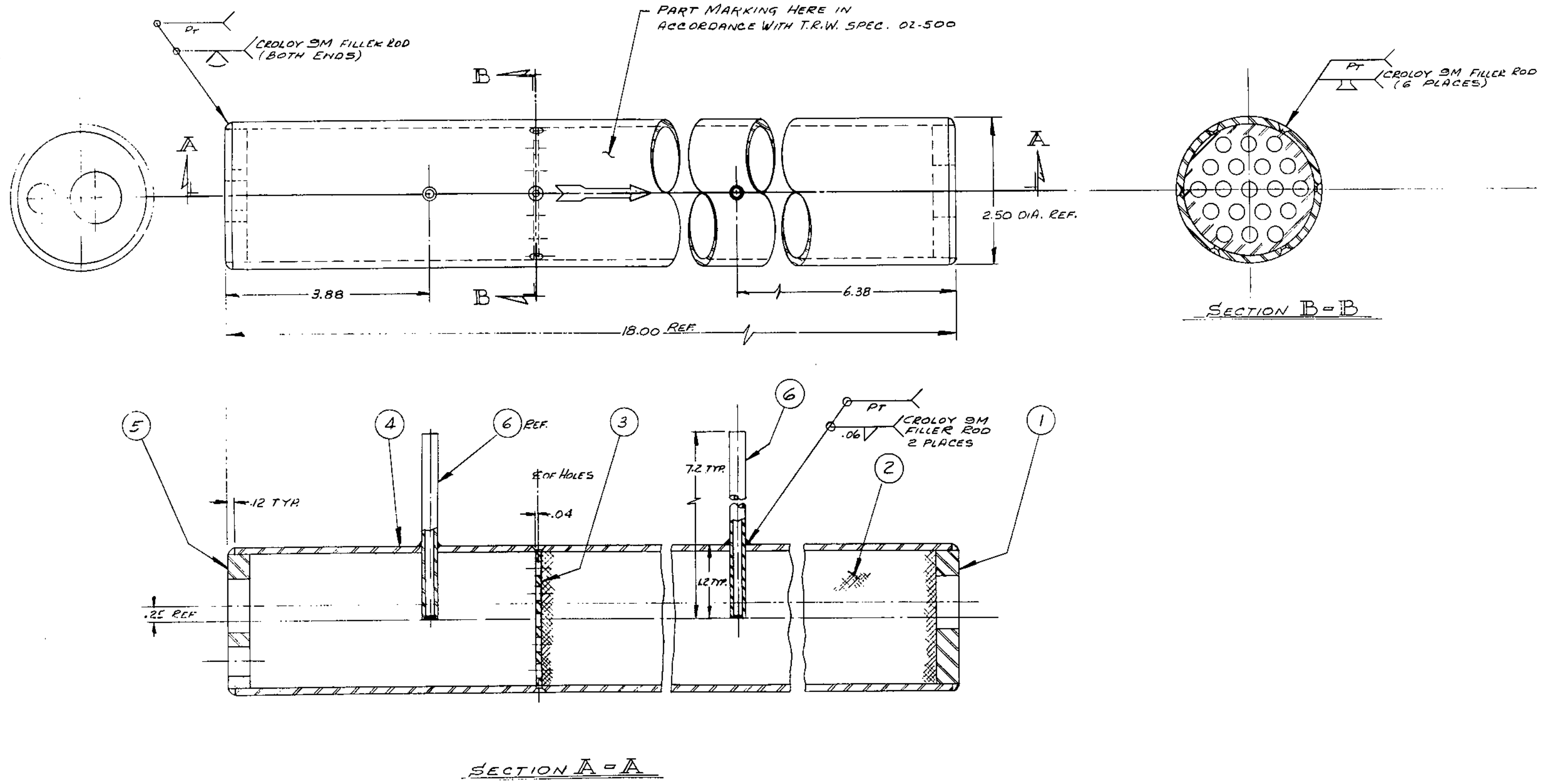
4. Corrosion Product Separators

Three corrosion product separators were included in the system. The first of these, the vapor trap, was located at the exit of the boiler and is shown in Figure 3. The liquid was separated from the vapor by gravity and was returned to the boiler through a drain line (0.5 inch OD x 0.083 inch wall Croloy 9M tubing), while the vapor passed through a tantalum wool getter before exiting from the separator. A total of 3.75 pounds of tantalum were used to separate corrosion products present in the vapor stream.

The vapor from the vapor trap then passed through a second corrosion product separator which was located in the superheater section of the loop. This separator is shown in Figures 4 and 5. In this vapor corrosion product separator, the vapor passed first through a magnetic field and then through a tantalum getter before exiting into the superheater proper. The Alnico 5 magnets were machined circular segments and were enclosed with 0.015 inch thick Croloy 9M sheet so that the mercury vapor did not contact the Alnico 5. A total of 1.16 pounds of tantalum were used in the separator. A drain line was also included in this separator so that any liquid separating from the vapor would be returned to the boiler.

The third separator, shown in Figures 6 and 7, was located at the condenser exit and utilized both magnetic and chemical gettering principles. The mercury exiting from the condenser passed first through 1.88 pounds of columbium wool and then through 0.31 pound of low carbon steel wool. From this point the mercury passed upwards towards the separator entrance between the poles of a 3200-Gauss "C"-shaped Alnico 5 magnet which was enclosed with Croloy 9M sheet. After passing through the magnet, the mercury passed through 0.25 pound of low carbon steel wool and then into the outlet tube to the subcooler.

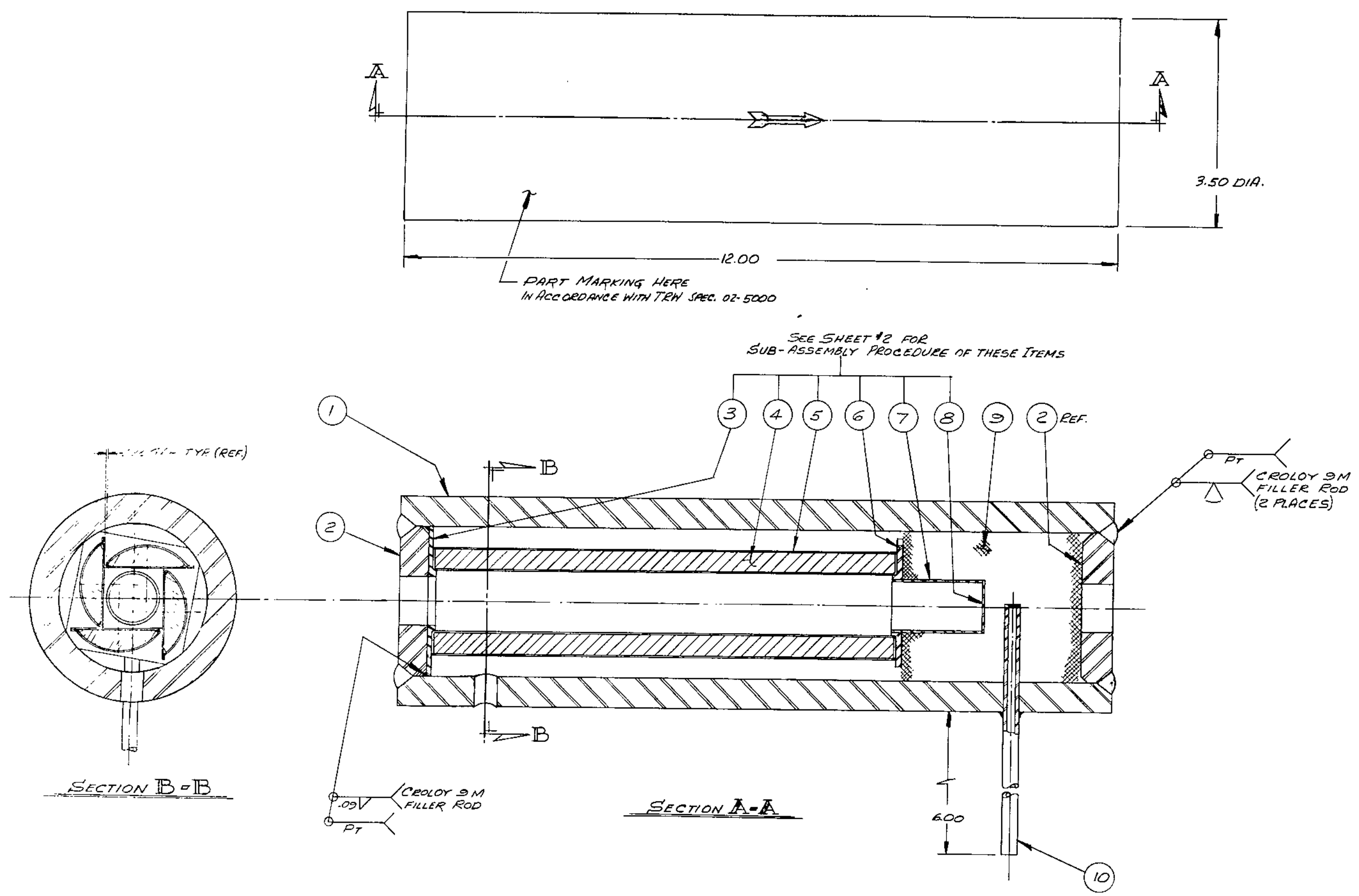
All parts of the three separators that were contacted by mercury were fabricated from Croloy 9M, with the exception of the tantalum, columbium, and low carbon steel getters.



QT'Y REQ'D	ITEM NO.	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MAT'L REFERENCE [redacted]
2	6	002819-4E	THERMOCOUPLE WELL	CROLOY 9M
1	5	002819-4D	END PLATE	CROLOY 9M
1	4	002819-4C	SHELL	CROLOY 9M
1	3	002819-4B	SEPARATION PLATE	CROLOY 9M
AR	2		GETTER	Ta WIRE
1	1	002819-4A	END PLATE	CROLOY 9M

LIST OF MATERIALS OR PARTS LIST

Figure 3. Vapor Trap Separator.



QT'Y REQ'D	ITEM NO.	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MAT'L REFERENCE OR NOTE
1	10	002819-5J	THERMOCOUPLE WELL	CROLOY 9M
A.R.	9		Ta WOOL	Ta
1	8	002819-5H	COVER, TUBE END	CROLOY 9M
1	7	002819-5G	TUBE	CROLOY 9M
1	6	002819-5F	UPPER RETAINING DISC	CROLOY 9M
4	5	002819-5E	SHELL, MAGNET COVER	CROLOY 9M
4	4	002819-5D	MAGNET	ALNICO 5
1	3	002819-5C	LOWER RETAINING DISC	CROLOY 9M
2	2	002819-5B	END PLATE	CROLOY 9M
1	1	002819-5A	SHELL	CROLOY 9M

LIST OF MATERIALS OR PARTS LIST

Figure 4. Vapor Corrosion Product Separator.

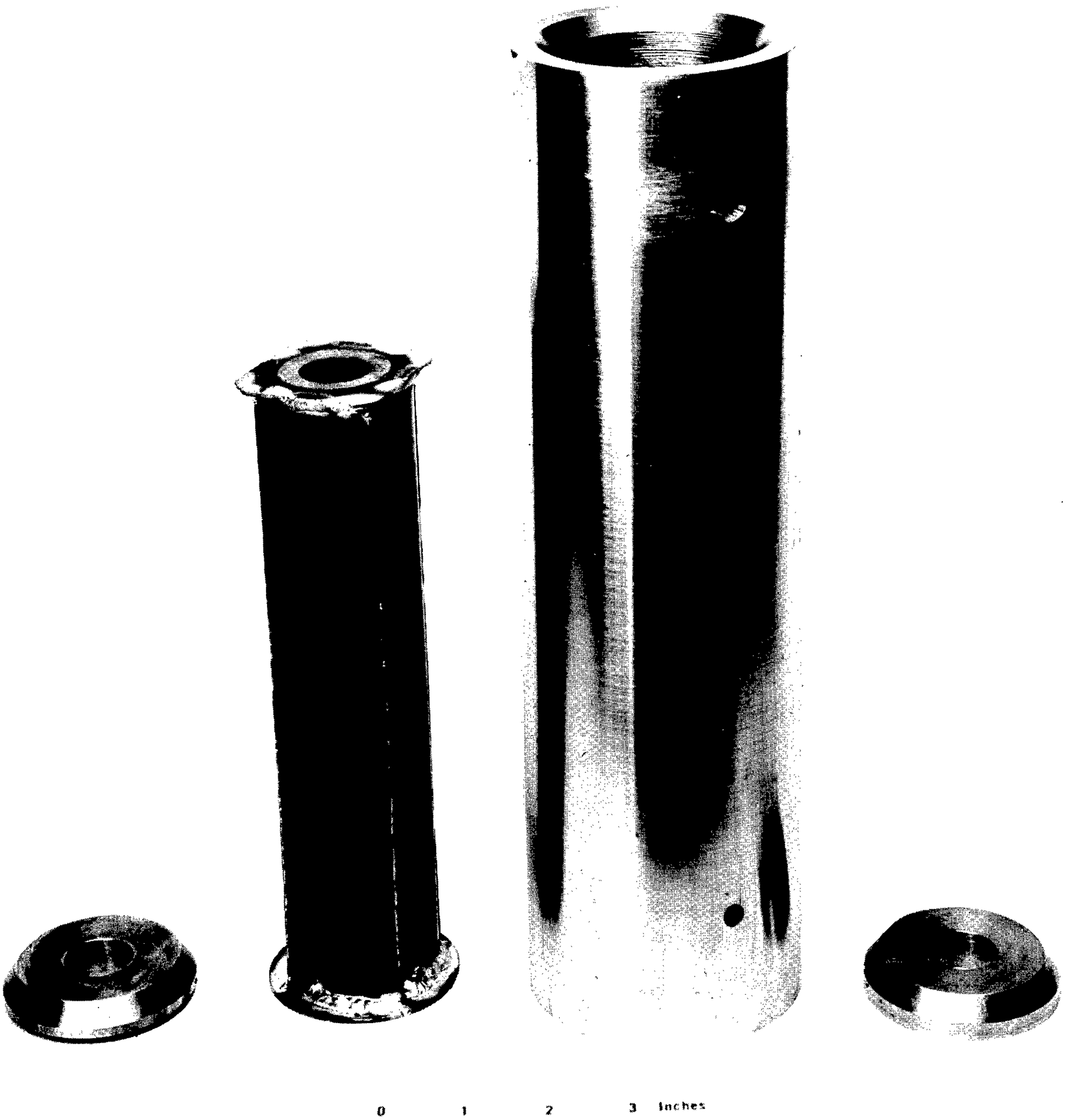
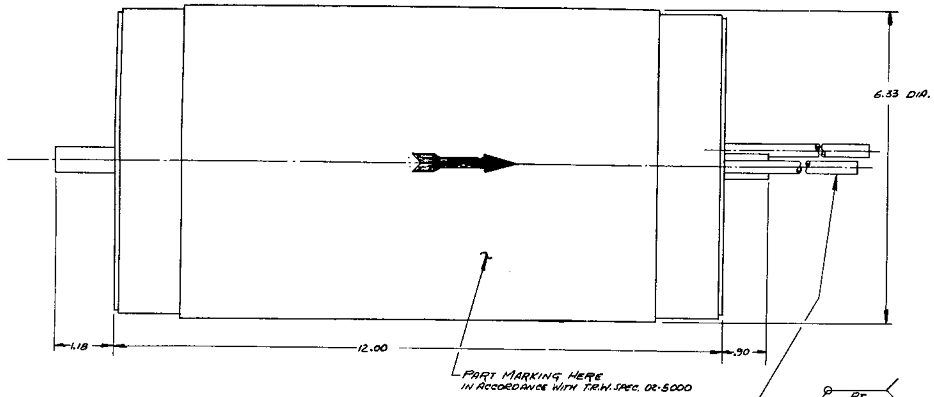


Figure 5. Photograph of Vapor Corrosion Product Separator.



1	21	002819-8U	THERMOCOUPLE WELL	CROLOY 9M
1	20	002819-8T	THERMOCOUPLE WELL	CROLOY 9M
1	19	002819-8S	THERMOCOUPLE WELL	CROLOY 9M
1	18	002819-8M	POLE PIECE, FLAT	CROLOY 9M
1	17	002819-8N	MAGNET SHIELD	CROLOY 9M
1	16	002819-8L	POLE PIECE, SHAPED	CROLOY 9M
1	15	002819-8C	END PLATE, BOTTOM	CROLOY 9M
A.R.	14		Cb TURNINGS (WOOL)	COLUMBIUM
A.R.	13		Fe TURNINGS (WOOL)	LOW CARB. STEEL
1	12	002819-8P	DRAIN TUBE	CROLOY 9M
1	11	002819-8I	SEPARATING PLATE, BOTTOM	CROLOY 9M
1	10	002819-8K	PERFORATED PLATE, BOTTOM	CROLOY 9M
1	9	002819-8J	OUTLET TUBE	CROLOY 9M
1	8	002819-8G	OUTER SHELL	CROLOY 9M
1	7	002819-8F	MAGNET	ALNICO 5
1	6	002819-8H	INNER SHELL	CROLOY 9M
1	5	002819-8D	PERFORATED PLATE, TOP	CROLOY 9M
1	4	002819-8E	SEPARATING PLATE, TOP	CROLOY 9M
2	3	002819-8R	OUTER SHELL, END PIECE	CROLOY 9M
1	2	002819-8B	END PLATE, TOP	CROLOY 9M
1	1	002819-8A	INLET TUBE	CROLOY 9M
QT'Y REQ'D	ITEM NO.	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MAT'L REFERENCE [redacted]

LIST OF MATERIALS OR PARTS LIST

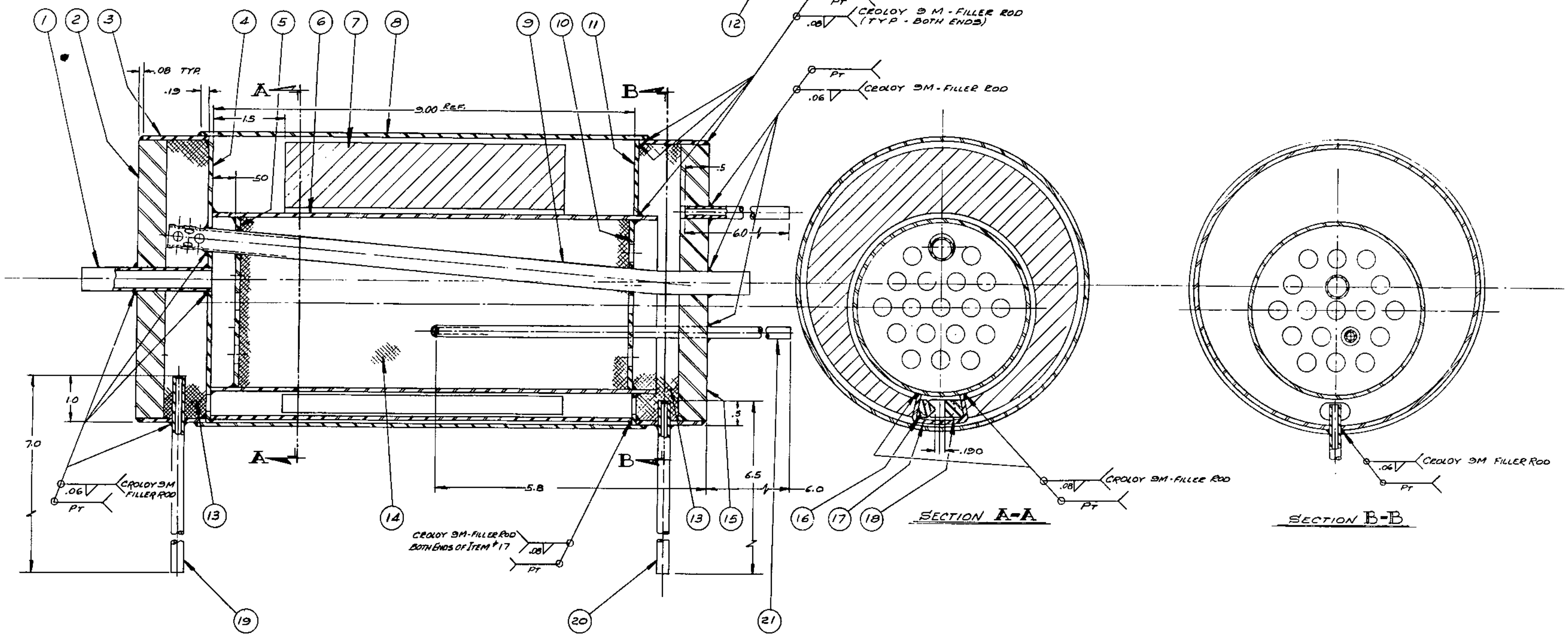


Figure 6. Liquid Corrosion Product Separator.

Figure 7. Photograph of Liquid Corrosion Product Separator.

B. CONTROLS

1. Heater Controls

Power to the heaters on the boiler, superheater, vapor trap, and vapor corrosion product separator sections of the loop was varied by the use of General Radio "Variac" autotransformers. Each heater circuit also contained a Weston ammeter. Alnor Type N-14 millivoltmeter-type temperature controllers were used to control the temperature of each heated section of the loop. Temperature indication was made by chromel-alumel thermocouples secured to the surface of the loop tubing near the exit of each heated section.

2. Condenser and Subcooler Control

The flow of cooling air for the condenser was varied by means of a Minneapolis-Honeywell motor-operated butterfly valve. The input signal to the motor was received from a Weston high-low, millivoltmeter-type temperature controller. This instrument sensed the condenser temperature as indicated by an iron-constantan thermocouple secured to the condenser wall.

The flow of cooling air for the subcooler was manually controlled by means of a butterfly valve.

The cooling water for both the condenser and subcooler sections was manually controlled.

3. Pressure Measurement

A Minneapolis-Honeywell pressure recorder with an overpressure control circuit provided a record of the boiler outlet pressure and also served as a safety device in the event of an overpressure situation. A total of five Ashcroft pressure gauges were used to measure the pressure at various locations in the system:

a) Superheater outlet pressure,

b) Throttling valve (or orifice) outlet pressure,

c) Condenser pressure,

d) Pump inlet pressure,

e) Pump outlet pressure.

All instruments were actuated by Type 316 SS diaphragm seals connected to the loop by Croloy 9M tubing. A constant head of liquid mercury was maintained in the sensing units, and all pressure readings have been corrected for this pressure head.

4. Temperature Measurement

In addition to the control thermocouples, forty-eight others were placed at various locations around the loop. Nine of these were immersion thermocouples and were located at the boiler exit, at the inlet and outlet of the vapor trap, at the outlet of the vapor corrosion product separator, at the inlet and outlet of the throttling valve (or orifice), in the center of the columbium wool in the liquid corrosion product separator, and one each in the low carbon steel wool in the top and bottom of the liquid corrosion product separator. The remaining thirty-nine were secured to the surface of the tubing. All thermocouples were chromel-alumel and were recorded on a 48-point Weston temperature recorder which was equipped with a limit switch for protection of the loop against excessive temperature.

5. Pump and Liquid Level Control

The pump used in the loop was a Milton Roy positive displacement pump with a pneumatic operated U. S. "Varidrive" variable speed motor. Materials of construction in contact with the mercury included:

Liquid end	Carbon steel
Diaphragm	Teflon
Ball seats	Type 430 SS
Ball valves	Type 416 SS
Check valve housing	Type 430 SS

The pump had a capacity of 158 pounds per hour of mercury (specific gravity of 13.6) and a maximum discharge pressure of 500 psig.

A Minneapolis-Honeywell level controller was employed to maintain the proper quantity of mercury in the boiler. Indication of the level was given by a Type 316 SS bellows assembly which sensed the difference in pressure at two points in the boiler section 24 inches apart. A pneumatic signal was transmitted by the level controller to the variable speed motor on the positive displacement pump.

6. Flow Measurement

A Croloy 9M Venturi having a 0.040 inch diameter orifice was used to measure the mercury flow at the preheater entrance. The pressure drop across the Venturi was indicated by an inverted U-tube manometer of Type 316 SS construction.

7. Throttling Valve By-Pass

Control of the throttling valve by-pass valve was achieved by means of an Ashcroft pneumatic transmitter containing a Type 316 SS Bourdon tube. The transmitter was calibrated such that a decrease in the condenser pressure to less than 15 psia would result in the sending of a pneumatic signal to the by-pass valve, allowing the valve to open. The transmitter would then control the condenser pressure between 11 and 14 psia by varying the setting of the by-pass valve.

C. ENCLOSURE

The entire system, with the exception of the pump and instrument sensing units, was installed in an enclosure of two-foot square cross section fabricated from sheet metal and Unistrut framing supports. After installation of the loop, the enclosure was filled with vermiculite which served as the insulation. Photographs of the loop installed in the enclosure are shown in Figures 8, 9, and 10. The control console is pictured in Figure 11.

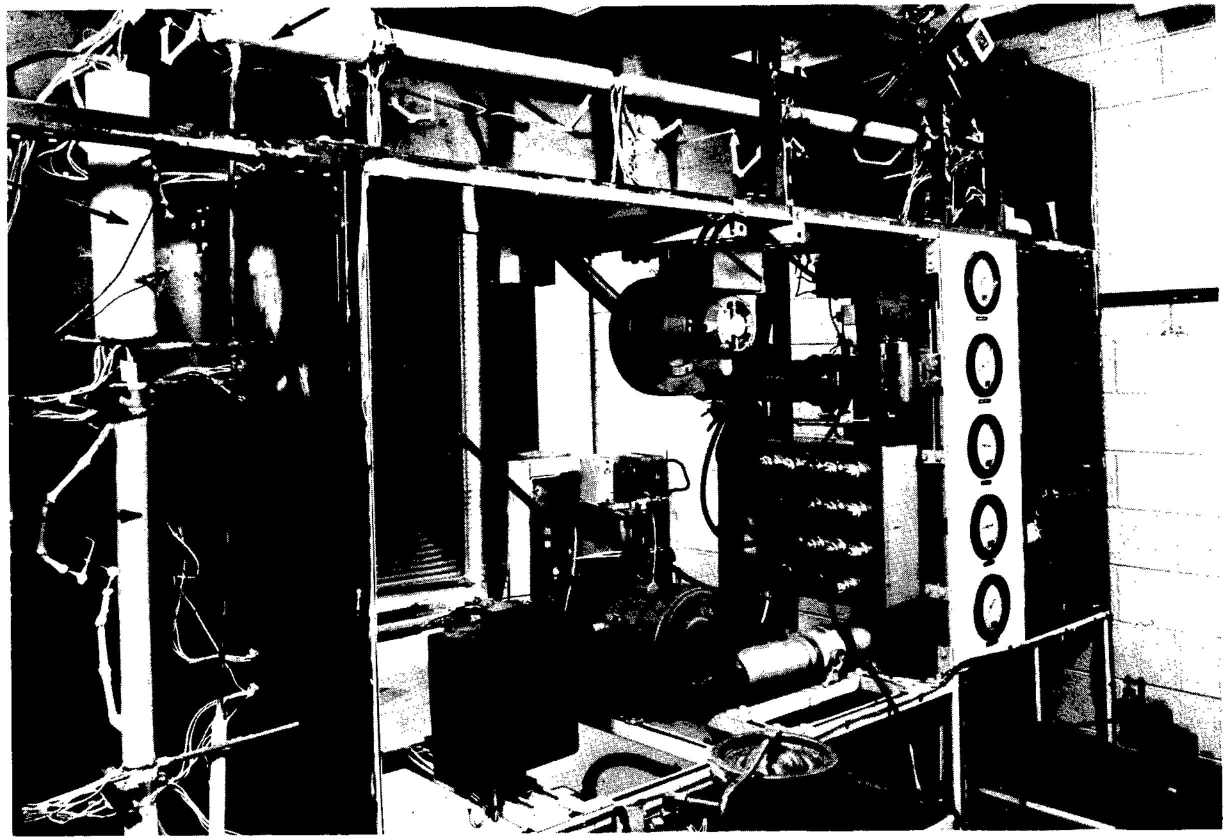


Figure 8. Croloy 9M Loop - Boiler and Superheater Sections.

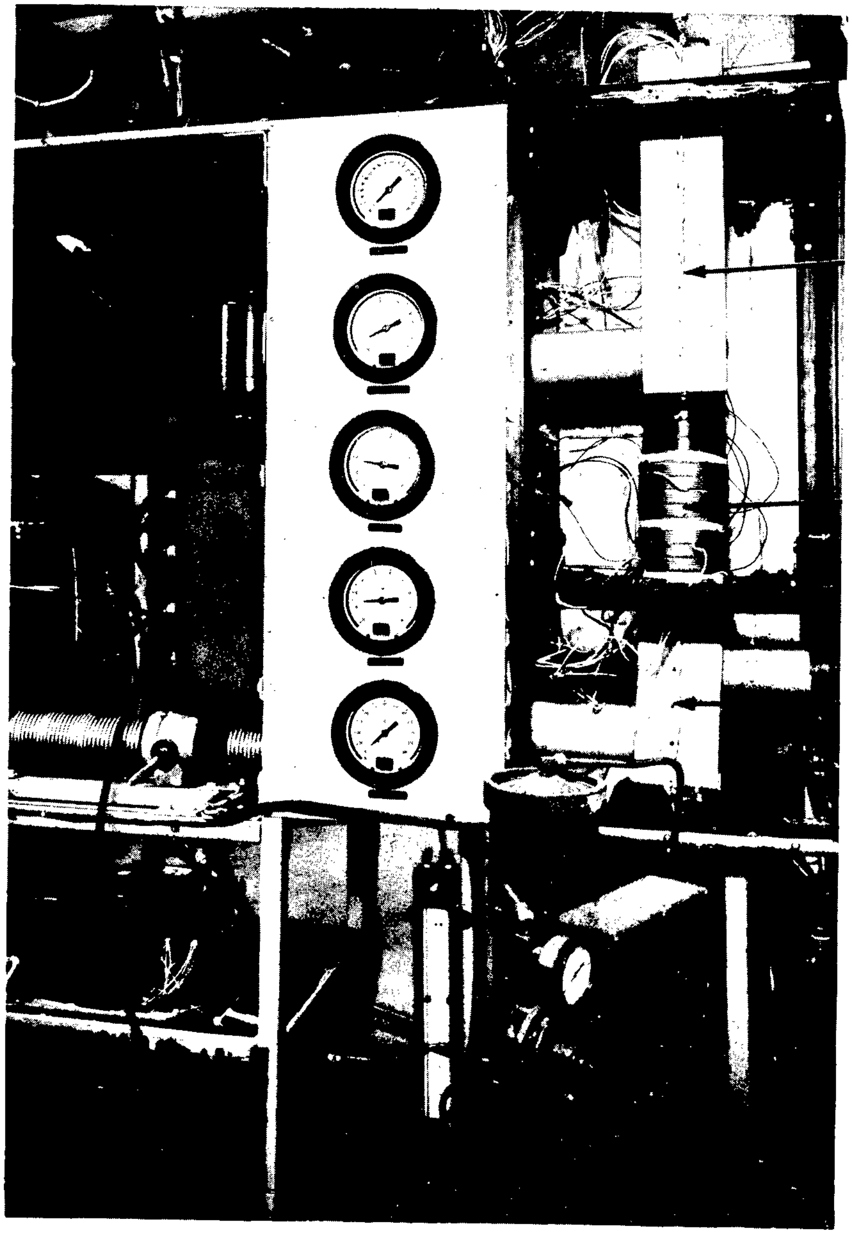


Figure 9. Croloy 9M Loop - Condenser and Subcooler Sections.

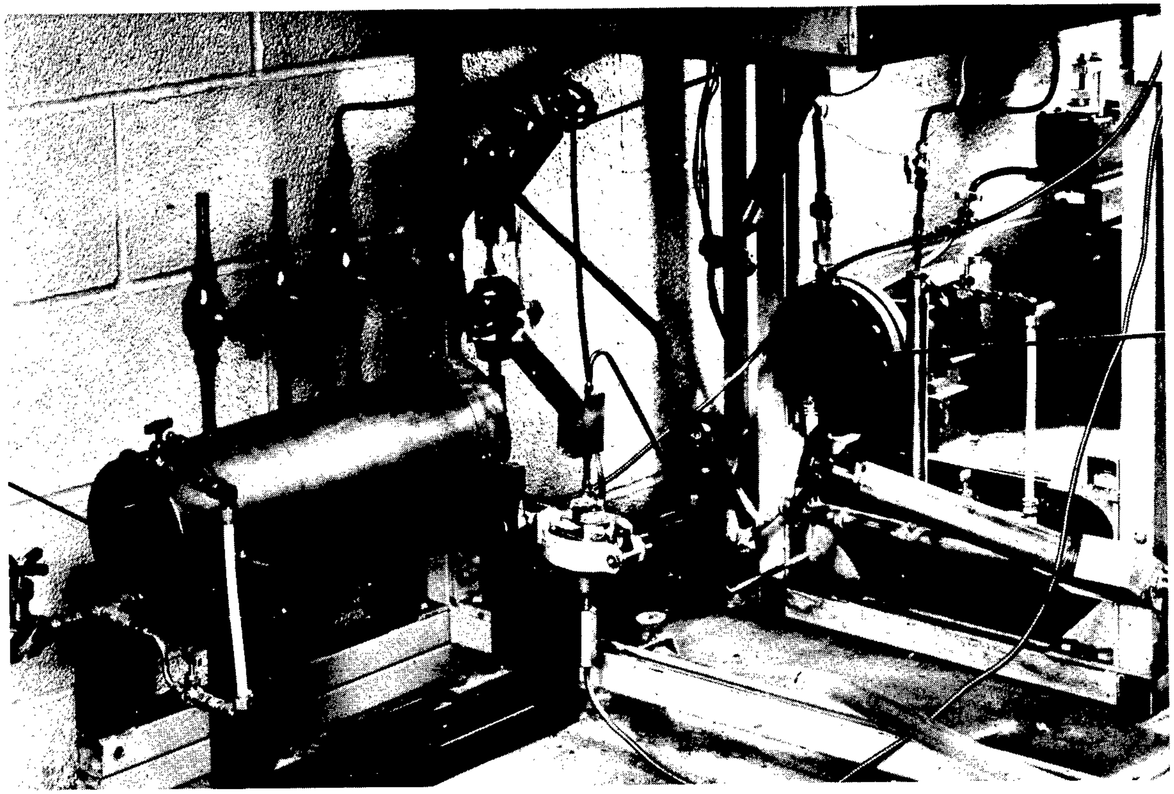


Figure 10. Croloy 9M Loop - Sump and Pump Head.

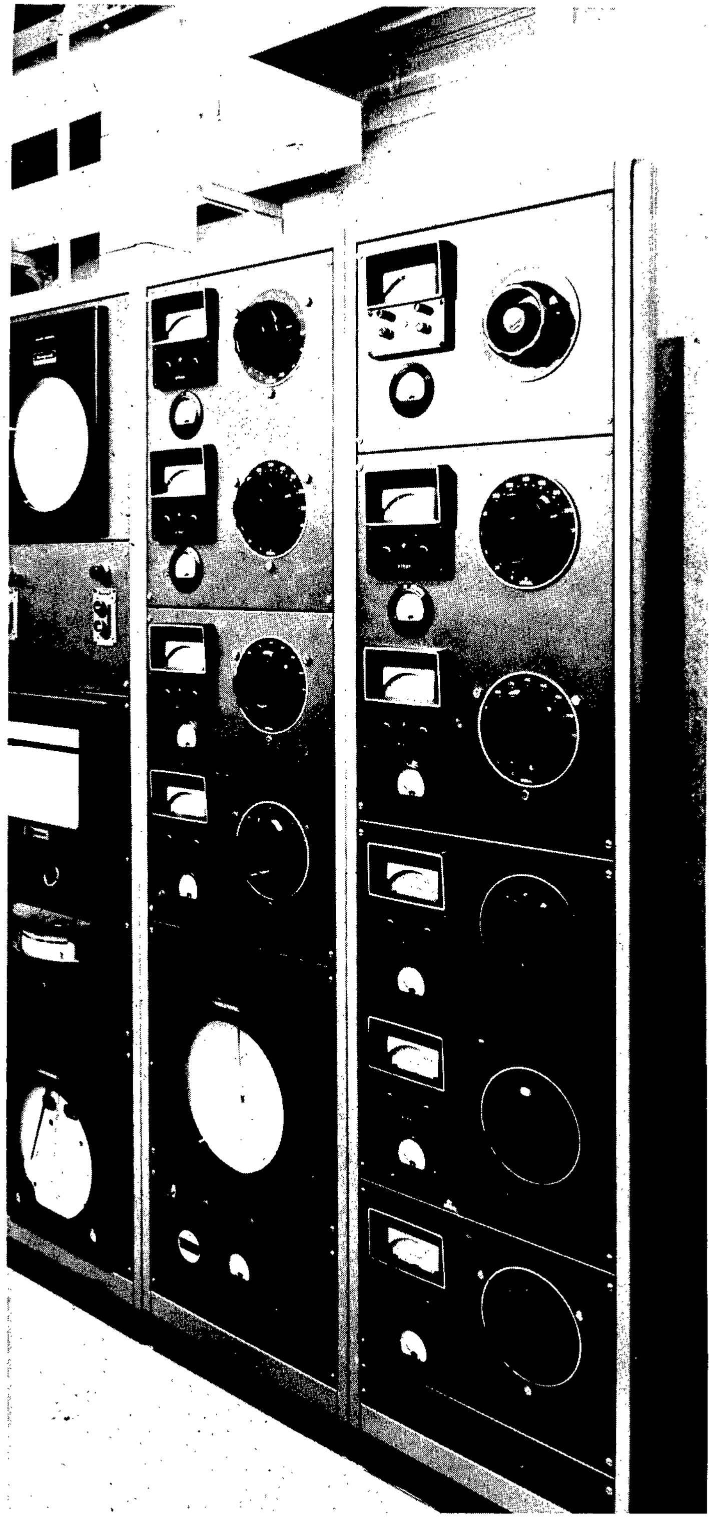


Figure 11. Control Console.

FABRICATION AND ASSEMBLY

The various components of the loop were machined and/or formed using standard shop practices. Croloy 9M sheets of 0.125 inch and 0.083 inch thickness which were required for the components of the corrosion product separators were not readily available. Therefore, forging blanks approximately 7 inches in diameter and 2.25 inches thick were obtained and rolled by the TRW Materials Technology Department. These blanks were first soaked at 2100°F for 0.5 hour and then side-forged into bars approximately 5 inches wide. These were again soaked at 2100°F for 0.5 hour, hot-forged to 0.5 inch thickness, and sand blasted when cold. The resulting pieces were trimmed, hot-rolled at 1600°F, followed by a final cold pass. Six to eight hot passes were required to reduce the thickness. The final thickness varied among pieces, ranging to approximately 0.010 inch over the nominal thickness.

All pieces that were to be formed by bending were then stress-relieved and sand blasted. Some of the components of the corrosion product separators required sheets of widths greater than those available. For these pieces, sheets were butt-welded together by the tungsten inert-gas process using Type 505 SS (Croloy 9M) filler rod. These pieces were then stress relieved at 1450°F for two hours. Hardness readings taken on the sheets that had been stress relieved showed that the final hardness ranged between $R_B 85$ and $R_B 94$. These pieces were thus sufficiently ductile that cracking would not occur during the forming operations.

The components of the corrosion product separators were then fabricated and cleaned. Several cleaning techniques were evaluated on scrap pieces, including oxidizing salt baths, hydrogen annealing, and acid baths. All methods evaluated resulted in a "bright" surface which oxidized rapidly upon exposure to air. This was as expected, since a thin oxide film is formed on all straight chromium alloys when exposed to air. The cleaning technique chosen for the components of the corrosion product separators consisted of the following steps:

1) A 10-minute dip in a 30% nitric acid, 3% hydrofluoric acid solution;

2) Thorough water rinse;

3) A 2-minute dip in a caustic permanganate solution;

4) Thorough water rinse;

5) A 20-second dip in a 10% sulfuric acid solution;

6) Thorough water rinse (180°F);

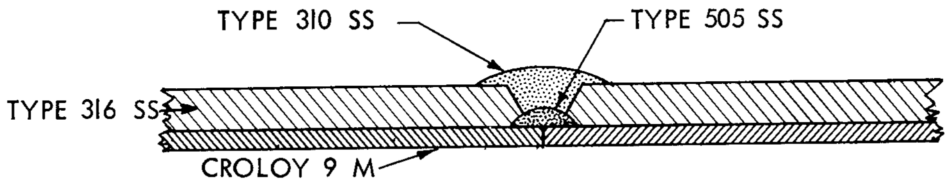
7) Rinse with analytical reagent-grade acetone.

Following the cleaning operation, the corrosion product separators were assembled by the tungsten inert-gas welding process using Type 505 SS filler rod. All parts were preheated to 400°F and interpass heated to 400°F during the welding operation.

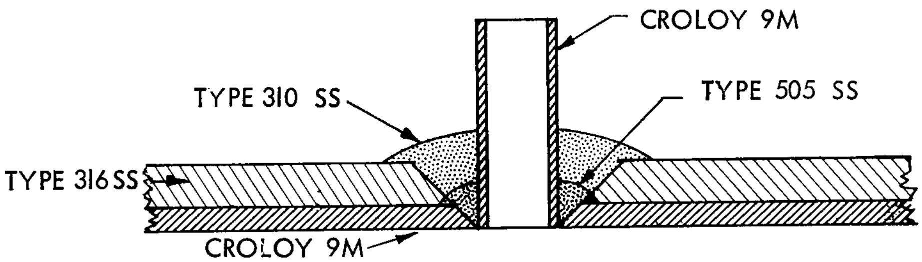
The various sections of the loop were formed and machined as required. The 0.25 and 0.5-inch Croloy 9M tubing was cleaned, following the procedure outlined above for the corrosion product separators. Since the 1.0 inch Croloy 9M-Type 316 SS tubing had been bright hydrogen annealed and protected with an oil film by the vendor, this tubing was only degreased with analytical reagent-grade acetone.

The components of the system were installed in the loop enclosure and were welded in place using the tungsten inert-gas process. During the welding, the sections of the system were preheated and interpass heated to 400°F. There were several types of weld configurations in the system, and these are shown schematically in Figures 12 and 13. In general, Type 505 SS (Croloy 9M) filler rod was used for all Croloy 9M-to-Croloy 9M joints, while Type 310 SS filler rod was used for Type 316 SS-to-Type 316 SS and Type 316 SS-to-Croloy 9M joints. Thus, the weld material in contact with the mercury throughout the system was Type 505 SS(Croloy 9M).

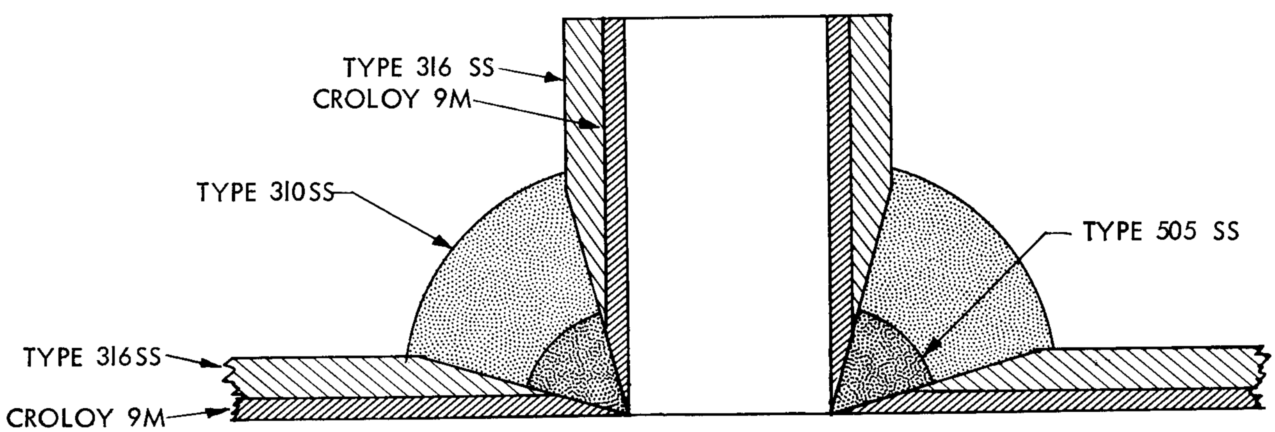
Following the welding of the system, radiographs were taken of all the final seal welds in the loop, with the exception of the tube-to-plate welds in the vapor trap and vapor corrosion product separator. Because of the complex configuration involved in these areas, fully satisfactory radiographs could not always be obtained. Nevertheless, this examination revealed porosity and lack of complete penetration in some of the welds. None of these defects were considered objectionable, and all of the welds were accepted.



a) DUPLEX TUBING - TO - DUPLEX TUBING (BUTT)

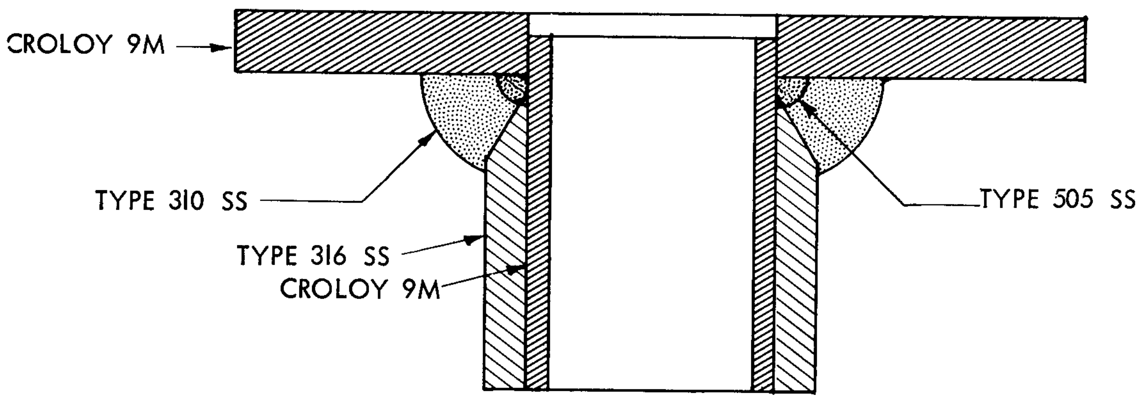


b) CROLOY 9M TUBING (0.25 OR 0.5 - INCH) - TO - DUPLEX TUBING (90°)

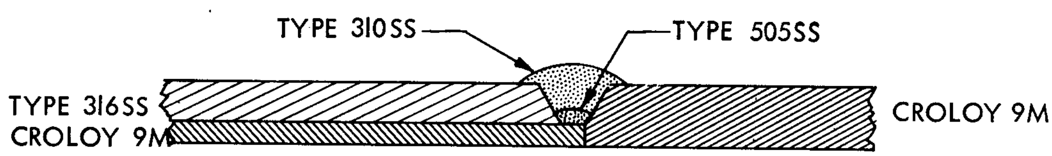


c) DUPLEX TUBING - TO - DUPLEX TUBING (90°)

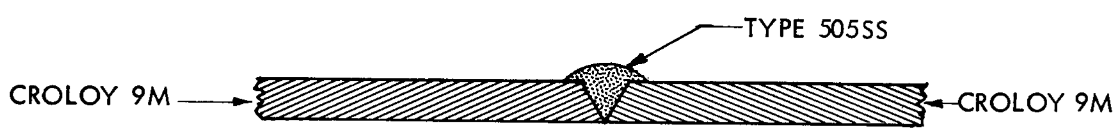
FIGURE 12. SCHEMATIC OF VARIOUS TYPES OF WELD JOINTS



a) DUPLEX TUBING-TO-CROLOY 9M PLATE (90°)



b) DUPLEX TUBING - TO - CROLOY 9M REDUCER (BUTT)



c) CROLOY 9M TUBING -TO - CROLOY 9M TUBING (BUTT)
(0.25 OR 0.5 - INCH)

FIGURE 13. SCHEMATIC OF VARIOUS TYPES OF WELD JOINTS

A dye-penetrant inspection was also conducted on all final seal welds in the system. No defects were revealed by this technique.

The system was then pressurized with argon to 350 psig (except for low pressure instrumentation) and was tested for leaks with "SNOOP" leak detector. Several small leaks were found in the 1.0 inch-to-0.5 inch reducer at the entrance of the condenser. These leaks appeared as cracks which apparently followed stringers in the bar stock from which the reducer was machined. These cracks were seal welded, the system was again tested under pressure, and no leaks were detected.

The loop was then evacuated for three days to a pressure of 0.4 micron. Over a period of 2.5 hours, a leak rate of 18 microns per hour was established. This leak rate included the entire system, with the exception of the sump. This component was not included in the vacuum leak test since it was to be valved out of the system during actual operation. The sump was tested separately and was found to be leak tight.

Thermocouples and heaters were installed, and the preheater, boiler, and superheater sections of the system were stress relieved at 1350 ± 25°F for two hours under vacuum. During the stress relief, the pressure rose to a maximum of 23 microns and then decreased to a final pressure of 3 microns when the power to the heaters was turned off.

The system was evacuated continuously during the cooling down period to a pressure of 0.3 micron. A final leak rate was determined over a period of one hour and was established at 3 microns per hour. This leak rate included all parts of the system, with the exception of the sump.

The loop enclosure was then filled with insulation in preparation for pretreatment and test operation.

QUALITY ASSURANCE

The quality assurance phases of the program were involved primarily with:

1) Inspection and verification of incoming materials and equipment;

2) Testing of materials as required;

3) Calibration of instrumentation;

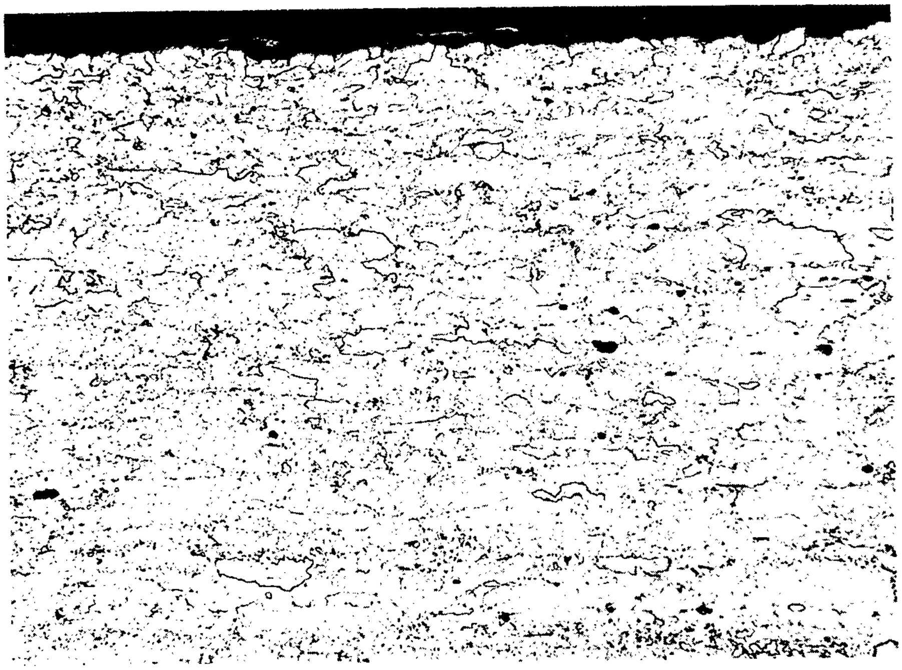
4) Weld quality.

Certified analyses were received for all of the materials of construction except the forging blanks from which the sheet stock was rolled. The certifications were reviewed as the materials were received, and all materials were found to be within specifications. Samples of the Croloy 9M sheet rolled from the forging blanks were submitted for metallographic evaluation. The results of the analysis indicated no irregularities in any of the samples. Typical photomicrographs are presented in Figures 14 and 15.

Samples of the Croloy 9M sheets were also submitted for chemical analysis. Results showed that the material was within specifications, as shown in Table I. Tensile test specimens were also prepared from the sheet. Two samples were machined from each of two sheets and were tested at room temperature. The results are presented in Table 2.

The Type 316 SS-Croloy 9M duplex tubing, as well as the 0.5 inch OD x 0.083 inch wall Croloy 9M tubing, was sampled in the as-received condition and was submitted for metallographic examination. Typical photomicrographs are presented in Figure 16.

All temperature measuring instruments were calibrated using a Leeds and Northrup millivolt potentiometer. Pressure gauges were calibrated using a dead-weight hydraulic tester. The pressure recorder was calibrated using a known Heise gauge as reference. This same procedure was employed for re-calibration of all pressure instrumentation before startup following the installation of the orifice. The Venturi flow meter was also calibrated using a known Venturi as reference.



250X
Carapella's

Figure 14. Surface of 0.083 Inch Thick Croloy 9M Sheet As Rolled.

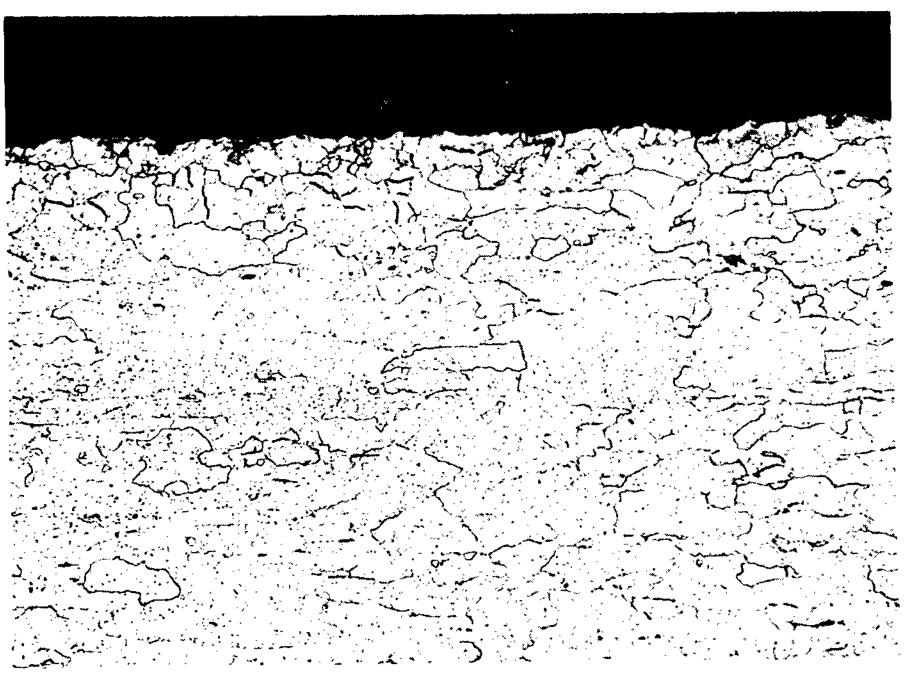


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Figure 15. Surface of 0.125 Inch Thick Croloy 9M Sheet As Rolled.

TABLE 1

CHEMICAL ANALYSIS OF CROLOY 9M SHEETS

Sample	Composition, Weight Percent				
	C	Cr	Mn	Mo	Si
Sheet 1 (0.083" thick)	0.13	8.75	0.46	1.02	0.70
Sheet 2 (0.083" thick)	0.12	8.75	0.50	1.00	0.70
Sheet 3 (0.083" thick)	0.12	8.75	0.51	1.03	0.69
Sheet 4 (0.125" thick)	0.13	8.70	0.49	1.01	0.70
Sheet 5 (0.083" thick)	0.13	8.75	0.45	1.02	0.70
Sheet 6 (0.125" thick)	0.11	8.75	0.50	1.02	0.71
Sheet 7 (0.125" thick)	0.14	8.80	0.50	0.98	0.70
Sheet 8 (0.083" thick)	0.14	8.75	0.49	1.03	0.70
Sheet 9 (0.083" thick)	0.12	8.75	0.51	1.00	0.71
Sheet 10 (0.015" thick)	0.14	8.98	0.54	1.00	0.60
Nominal Composition	0.15 max.	8.0-10.0	0.3-0.6	0.9-1.1	0.25-1.00

TABLE 2

TENSILE TEST RESULTS OF TYPICAL CROLOY 9M SHEET SPECIMENS

	0.125" Sheet		0.083" Sheet	
	A	B	A	B
Ultimate Strength, psi	124,800	108,000	89,600	90,600
0.2% Offset Yield Strength, psi	83,700	71,300	57,000	54,800
Percent Elongation in 2 Inches	27.0	18.5	35.3	34.8
Percent Reduction of Area	63.1	43.9	75.0	47.4

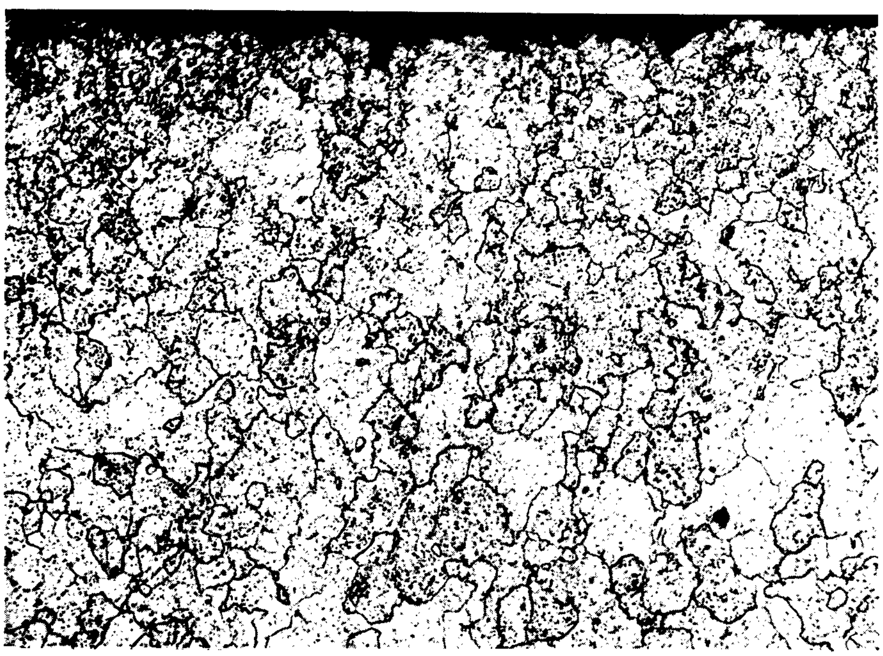
Croloy 9M

Type 316 SS

100X

a) Duplex Croloy 9M/ 316SS

Etchant-Carapella



250X

b) 0.5 Inch O.D. Croloy 9M

Etchant-Carapella

Figure 16. Microstructure of Duplex Type 316 SS-Croloy 9M and 0.5 Inch Croloy 9M Tubing As Received.

As mentioned in the "Fabrication and Assembly" section, all joining was done by tungsten inert gas welding. During the welding operations, all parts were preheated to 400°F and interpass heated to the same temperature. Subassemblies were stress relieved at 1350 ± 25°F for two hours as were the preheater, boiler, and superheater sections of the loop after final assembly.

All final seal welds in the system were inspected using radiographic and penetrant techniques. An exception here was the tube-to-plate welds in the vapor trap and vapor corrosion product separator where satisfactory radiographs could not be obtained because of the configuration. The system was tested for leak tightness under an argon pressure of 350 psig using "SNOOP" leak detector and also under a vacuum of 0.3 micron. Before startup, a leak rate of 3 microns per hour was established.

During the filling and draining operations which occurred after the initial startup, both the mercury and the loop were protected with either a vacuum or an argon cover. The only times when this was not feasible were during welding operations. Argon was flushed through the loop during welding, but complete protection could not be guaranteed.

LOOP OPERATION

A. PRETREATMENT

Prior to test operation, the loop was flushed with liquid mercury at 500-650°F as a final cleaning treatment. Triple distilled, A. C. S. -grade mercury was used. During the initial filling operation, leaks developed in two of the Croloy 9M valves. Examination of the valves indicated that the leaks resulted from poor welds in the valves themselves. The mercury was drained from the loop, and the defective valves were replaced with new Croloy 9M valves. The system was leak tested, filled with the same mercury, and the flushing operation was started. After only seven hours of operation at 500 to 600°F, a third Croloy 9M valve developed a leak at the same point as the other defective valves. Because of the uncertainty which now existed about the integrity of the Croloy 9M valves, it was felt that the third defective Croloy 9M valve should be replaced with a Type 316 SS valve. It was also observed that the Croloy 9M valve located in the line between the loop and the sump was not sealing properly. Since both this valve and the leaky one were located in very low temperature regions of the system, it was decided to replace these valves with ones of Type 316 SS construction. The two valves were installed, the loop was refilled with the same mercury, and the flushing operation was continued for an additional 69 hours.

The loop was flushed with liquid mercury for a total of 76 hours at 500-650°F. Flow rate during this treatment was 56 pounds per hour. The mercury was then drained from the loop, the system was evacuated and refilled with clean mercury (triple distilled, A. C. S. -grade), and test operation was begun.

B. TEST OPERATION

During the course of the test operation, several failures occurred in the system and changes were made to correct these failures. Because of the nature of some of the modifications, loop conditions varied during different periods of operation. For this reason, the test operation has been divided into two runs which are discussed separately. Average operating conditions for each run are given in Table 3.

1. Run No. 1

Throughout this run control difficulties were experienced with the throttling valve in that fine adjustment of the valve could not be obtained. This valve was later sectioned and examined, and the

TABLE 3

AVERAGE LOOP OPERATING CONDITIONS

	Thermocouple Number	Run No. 1	Run No. 2
Total Operating Time, Hours		0-448	448-2918
Elapsed Time, Hours		448	2470
Thermocouple Locations and Average Reading, °F			
Boiler Heater No. 1-Boiler Heater No. 2	2	851	834
Boiler Heater No. 2 Inlet	3	965	1002
Boiler Heater No. 2-Boiler Heater No. 3	4	1007	1059
Boiler Heater No. 3 Inlet	5	1084	1144
Boiler Heater No. 3 Outlet	6	1075	1142
Boiler Heater No. 3-Boiler Heater No. 4	7	1018	1070
Boiler Heater No. 4 Inlet	8	1074	1148
Boiler Heater No. 4-Boiler Heater No. 5	10	1056	1078
Boiler Heater No. 5 Inlet	11	1117	1128
Boiler Heater No. 5-Vapor Trap	13	1051	1069
Boiler Heater No. 5-Vapor Trap Immersion	14	1056	1074
Vapor Trap Inlet	15	1193	1132
Vapor Trap Outlet	16	1156	1133
Vapor Trap Inlet Immersion	17	1158	1160
Vapor Trap Outlet Immersion	18	1113	1129
Vapor Trap-Vapor Corrosion Product Separator	19	1051	1070
Vapor Corrosion Product Separator Inlet	20	1155	1176
Vapor Corrosion Product Separator Outlet	21	1156	1177
Vapor Corrosion Product Separator Outlet Immersion	22	1139	1166
Vapor Corrosion Product Separator-Superheater Heater No. 1	23	1118	1132
Superheater Heater No. 1 Inlet	24	1190	1214
Superheater Heater No. 1 Outlet	25	1198	-
Superheater Heater No. 1-Superheater Heater No. 2	26	1081	1150
Superheater Heater No. 2 Inlet	27	1215	1278
Superheater Heater No. 2 Outlet	28	1237	1316

TABLE 3 (Continued)

		Thermocouple Number	Run No. 1	Run No. 2
Superheater Heater No. 2-Superheater Heater No. 3		29	1193	1317
Superheater Heater No. 3 Inlet		30	1256	1409
Superheater Heater No. 3 Outlet		31	-	1405
Throttling Valve/Orifice Inlet		32	1082	1313
Throttling Valve/Orifice Inlet	Immersion	33	1090	1264
Throttling Valve/Orifice Outlet		35	803	912
Throttling Valve/Orifice Outlet	Immersion	36	758	837
Condenser Inlet		37	528	698
Condenser Mid-Point		IC	407	589
Condenser Outlet		39	338	594
Condenser-Liquid Corrosion Product Separator		40	336	483
Liquid Corrosion Product Separator Inlet		43	402	473
Liquid Corrosion Product Separator Outlet		44	374	445
Liquid Corrosion Product Separator				
Columbium Wool	Immersion	41	282	331
Steel Wool (bottom)	Immersion	45	329	405
Steel Wool (top)	Immersion	42	388	472
Subcooler Inlet		46	302	398
Subcooler Midpoint		47	193	295
Subcooler Outlet		48	144	227
Pressures and Corresponding Saturation Temperatures, psia/°F				
Boiler Outlet			221/1033	266/1066
Superheater Outlet			246/1051	269/1068
Throttling Valve/Orifice Outlet			27.8/749	29.0/744
Condenser			36.3/770	28.7/743
Flow Rate, Pounds Per Hour			70	86

results of the examination may be found in the section "Loop Disassembly, Results, and Discussion".

With the exception of the throttling valve control problem, the loop operated satisfactorily for 115 hours when a fourth Croloy 9M valve developed a leak. At this time, it was decided to replace this valve and several other low-temperature valves with Type 316 SS valves. A total of six Croloy 9M valves were replaced, in addition to the two replaced during pretreatment. All of the eight valves were located in lines where the temperature did not exceed 100°F and included:

a) Line connecting the loop and the sump,

b) Superheater pressure gauge line,

c) Throttling valve outlet pressure gauge line,

d) Condenser pressure gauge line,

e) Pump inlet pressure gauge line,

f) Pump outlet pressure gauge line,

g) Line connecting the loop and the pressure transmitter used for control of the valve in the bypass around the throttling valve,

h) Liquid corrosion product separator drain line.

Following replacement of the valves, the system was evacuated, refilled with the same mercury, and test operation was continued. Adjustments were made during operation to establish the desired operating conditions. Not all of the operating conditions agreed precisely with desired conditions. The greatest discrepancy existed in the vapor trap where a temperature drop as great as 40°F existed between the inlet and outlet. It was felt that this problem could be solved by using two separate heaters on the vapor trap so that the power inputs to the top and bottom of the vapor trap could be controlled independently.

After a total of 434 hours of operation, the loop was shut down when a leak was discovered in the region of the superheater outlet. The mercury was drained from the loop, and the system was leak tested under pressure. A leak was found in the bellows of the valve in the bypass line around the throttling valve. It was decided to remove this valve and seal the bypass line with Croloy 9M caps. It was also decided to change the heaters on the vapor trap, as described above, while the loop was shut down.

These changes were made, the system was evacuated, refilled with the same mercury, and test operation was continued. After only one day of operation, a very severe leak developed in the Croloy 9M throttling valve and the loop was again shut down. The valve was removed from the system, and a fixed orifice, fabricated from Croloy 9M, was installed. During this shutdown, the bypass inlet line was removed and a straight section of tubing was installed at the orifice outlet. A new heater was installed on this section of the loop and was extended three inches past the orifice outlet in order to minimize cooling effects that had been observed at the throttling valve inlet. All of the mercury was removed from the system, and clean mercury (triple distilled, A. C. S. -grade) was used to refill the loop in preparation for startup.

2. Run No. 2

The loop was restarted and operated very satisfactorily until a total of 960 hours of operation had been accumulated (448 of these hours were accumulated before installation of the orifice). At this time the flow increased from 65 pounds per hour to 75 pounds per hour rather abruptly. As a result of the increased flow, the boiler temperature decreased, and the temperatures on the condenser side of the loop increased. After another five hours of operation, the flow again increased abruptly, stabilizing at 105 pounds per hour, with a second decrease in boiler temperature and increase in condenser-side temperatures. At this time it was also observed that the level on the condenser side had decreased. Approximately 25 pounds of mercury were added to the loop to increase the condenser level. In order to correct the temperature profile, the power to the boiler was increased and the condenser cooling was increased to its maximum. These changes seemed to correct the situation for a short time, but the level in the condenser decreased again over a period of several hours. Approximately another 25 pounds of mercury were added to the loop, and this resulted in stable operating conditions at a flow rate of approximately 100 pounds per hour. All conditions were satisfactory with the exception of the vapor trap outlet temperature which indicated that this area contained some liquid mercury. It was decided to allow the loop to operate with this condition for several days before making any further changes.

One explanation for the abrupt changes in flow seemed to be that some restriction in the system, perhaps in the orifice, was removed with time, thus permitting a greater flow of mercury through the loop. It is also possible that the loop became "conditioned", resulting in more efficient heat transfer, and thus a greater flow rate. Since the system instrumentation was not designed for heat transfer measurements, any possible changes in the heat transfer could not be determined.

Since no mercury was lost to the atmosphere with decreases in the condenser level, and since this level eventually became stable, it was proposed that some cavity in the system had filled with mercury. It was further proposed that a crack had developed in the inner shell of the liquid corrosion product separator, allowing the magnet cavity to fill with mercury. In order to verify this and also to verify that the vapor trap outlet was wet, it was decided to take radiographs of the entire system while in operation. An iridium-192 source was employed, and the results indicated the following:

a) The vapor in the vapor trap contained some entrained liquid mercury;

b) The exit tube from the vapor trap and the vapor corrosion product separator did not contain liquid mercury;

c) A crack had developed in the inner shell of the liquid corrosion product separator, since the magnet cavity was found to contain mercury;

d) The area between the exit of the vapor trap and the liquid level in the condenser contained no liquid mercury.

The boiler power was again increased, and the liquid level in the boiler was decreased in order to provide vapor of higher quality at the inlet of the vapor trap. These changes resulted in drying of the vapor in the vapor trap, as indicated by immersion thermocouples in the unit.

Following these changes, the loop operated without incident for an additional 762 hours until a total of 1722 hours of operation had been accumulated. At this time the heater on the superheater outlet failed and the loop was shut down. The heater was replaced, and the loop was restarted without difficulty.

The loop operated satisfactorily for an additional 1196 hours until a total of 2918 hours had been accumulated. At this time, the heater on the outlet of the superheater failed for the second time and the test was terminated thirty hours shy of the planned test duration.

LOOP DISASSEMBLY, RESULTS, AND DISCUSSION

A. PROCEDURE

Following shutdown of the loop, the mercury was drained from the various loop sections and was collected in clean plastic containers. Because of the large quantity of mercury in the system, the mercury was removed in steps and the mercury from various regions of the system was isolated, processed, and analyzed separately. The regions from which the mercury was isolated and drained included:

1) Boiler,

2) Condenser,

3) Liquid corrosion product separator,

4) Pump head,

5) Fill line,

6) Vapor trap,

7) Pressure recorder sensing head line.

All of the mercury removed from the system was oxidized in a Bethlehem mercury oxifier and then filtered through a Bethlehem mercury filter (gold adhesion principle). Both the filtered mercury and the corrosion products collected on the filter were submitted for chemical analysis.

After removal of the mercury from the system, the vapor trap, vapor corrosion product separator, and the liquid corrosion product separator were cut from the system and sectioned. The chemical getters and the deposits found in the separators were submitted for chemical analysis.

The loop was then cut into sections and each section was cut longitudinally for evaluation. Specimens were removed from the various sections for metallographic, x-ray fluorescence, and electron microprobe analyses. The metallographic specimens were nickel plated prior to mounting to preserve the condition of the surface.

After the specimens were selected for analysis, all deposits in the system were collected and submitted for analysis.

B. VISUAL OBSERVATIONS

As shown in Figures 17 and 18, the Venturi was found to be relatively clean. The boiler inlet section contained some amalgam and also some deposit which appeared as a black "smudge" (see Figure 19). A photograph of the boiler midsection is shown in Figure 20. This region was found to be relatively free of deposits. The liquid level region of the boiler was found to be slightly wetted by mercury, Figure 21. As the mercury was dried and superheated, the tube wall became less discolored until it actually took on a high luster at the vapor trap inlet, Figure 22. Wetting was again observed in the inlet region of the vapor trap, Figure 23. As the vapor passed through the vapor trap and into the vapor corrosion product separator, the tubing again became discolored, as was the vapor corrosion product separator. The magnet poles in this separator were found to have the same discoloration and contained no corrosion products, as shown in Figure 24. The outlet from the separator (superheater midsection) was also discolored, Figure 25. This discoloration gradually decreased until the tubing was again relatively clean at the superheater outlet, Figure 26. This area of the loop was bulged considerably, as shown in Figure 27. The growth in the tubing undoubtedly caused the failures in the heaters on this section of the system and is discussed in detail later in this report.

The Croloy 9M orifice was found to be relatively clean and contained only a small quantity of amalgam on the outlet side, Figure 28.

The condenser and subcooler tubing is shown in Figure 29. As seen here, these sections of the loop contained small quantities of amalgam and deposit.

C. METALLOGRAPHY

Metallographic samples were prepared from all sections of the loop, i.e., the boiler, superheater, condenser, subcooler and corrosion product separators. Included with the corrosion specimens were sections cut from welded joints representing butt welds of the duplex tubing and also welds between the one-inch diameter duplex Croloy 9M/316 SS and the 1/2-inch diameter Croloy 9M tubing. The specimens were prepared for study by conventional metallographic techniques. The polished specimens were examined in both the unetched and etched condition at magnifications varying between 3 and 500 X.

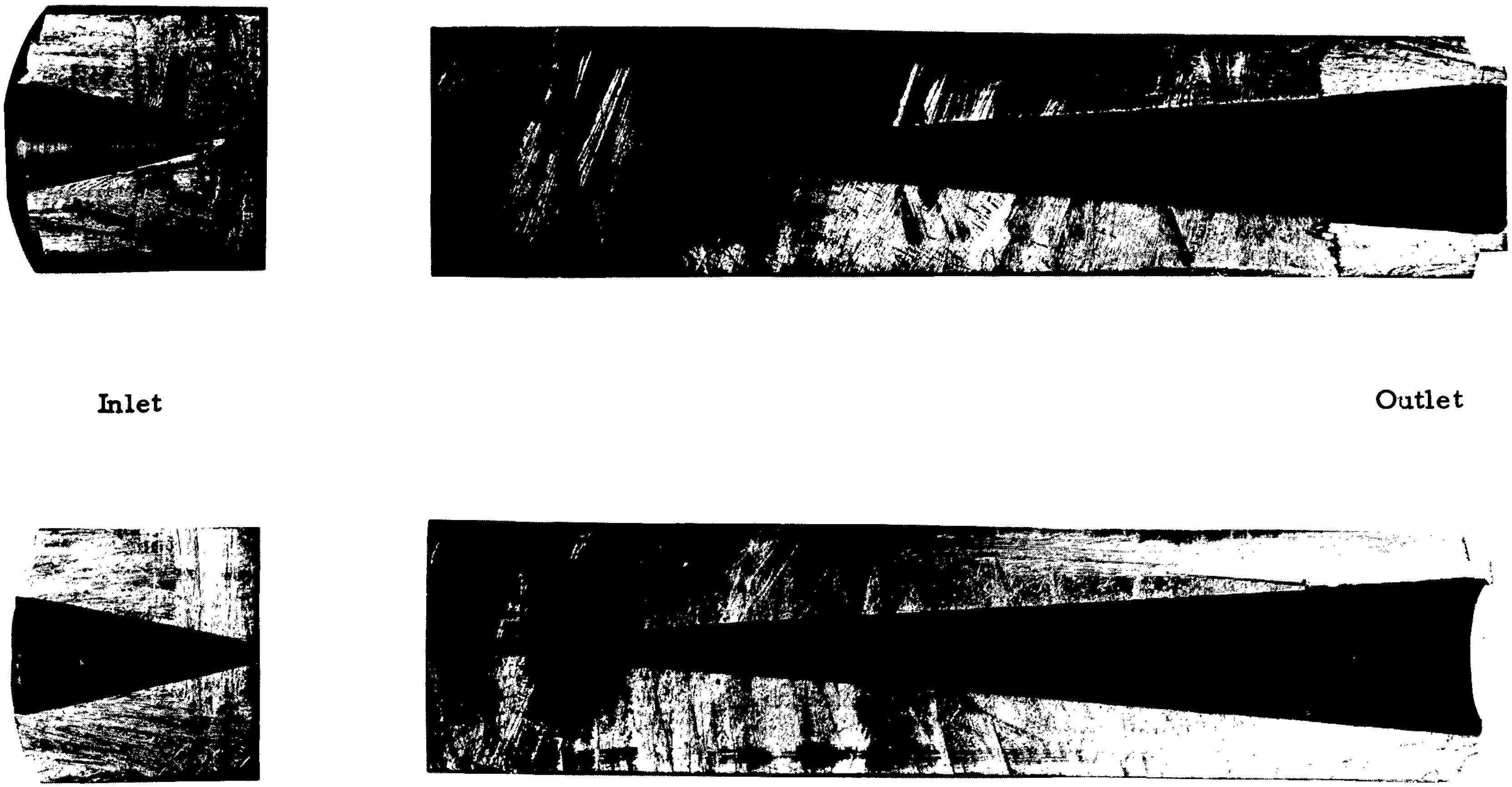
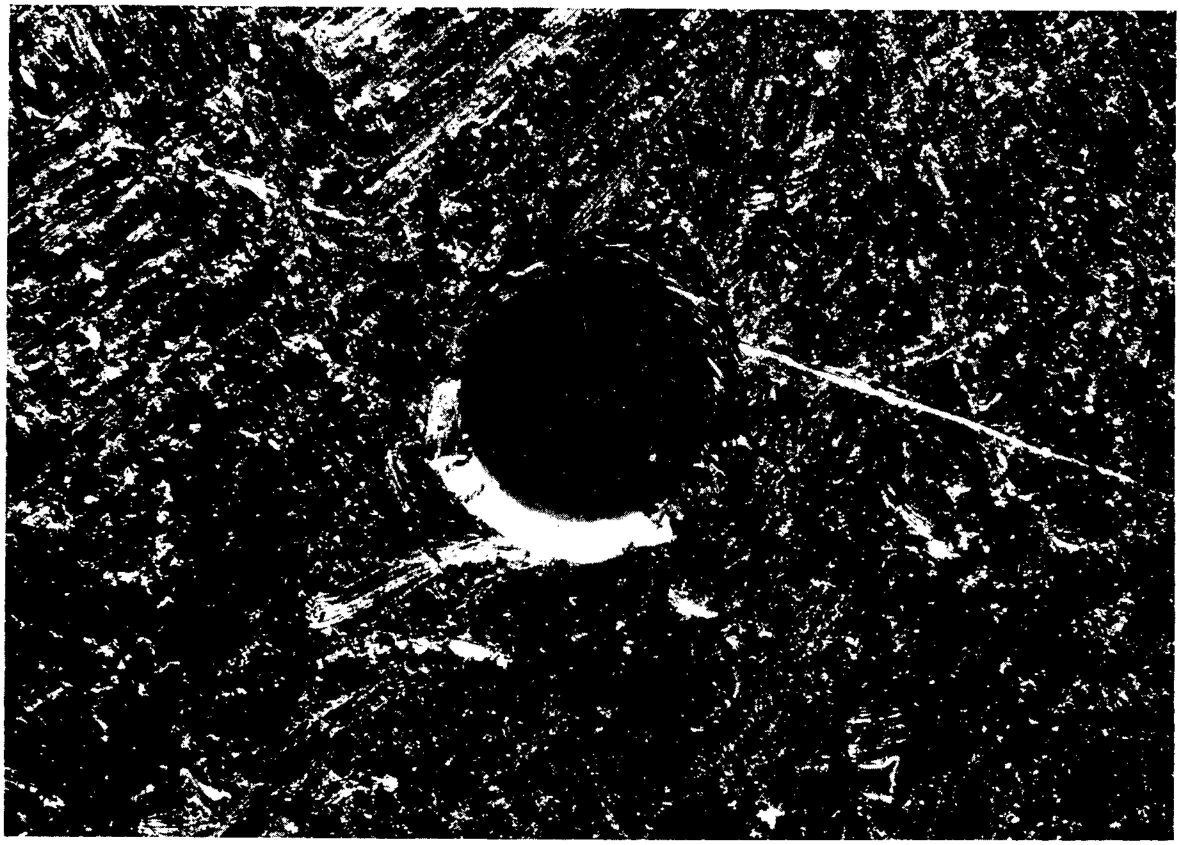
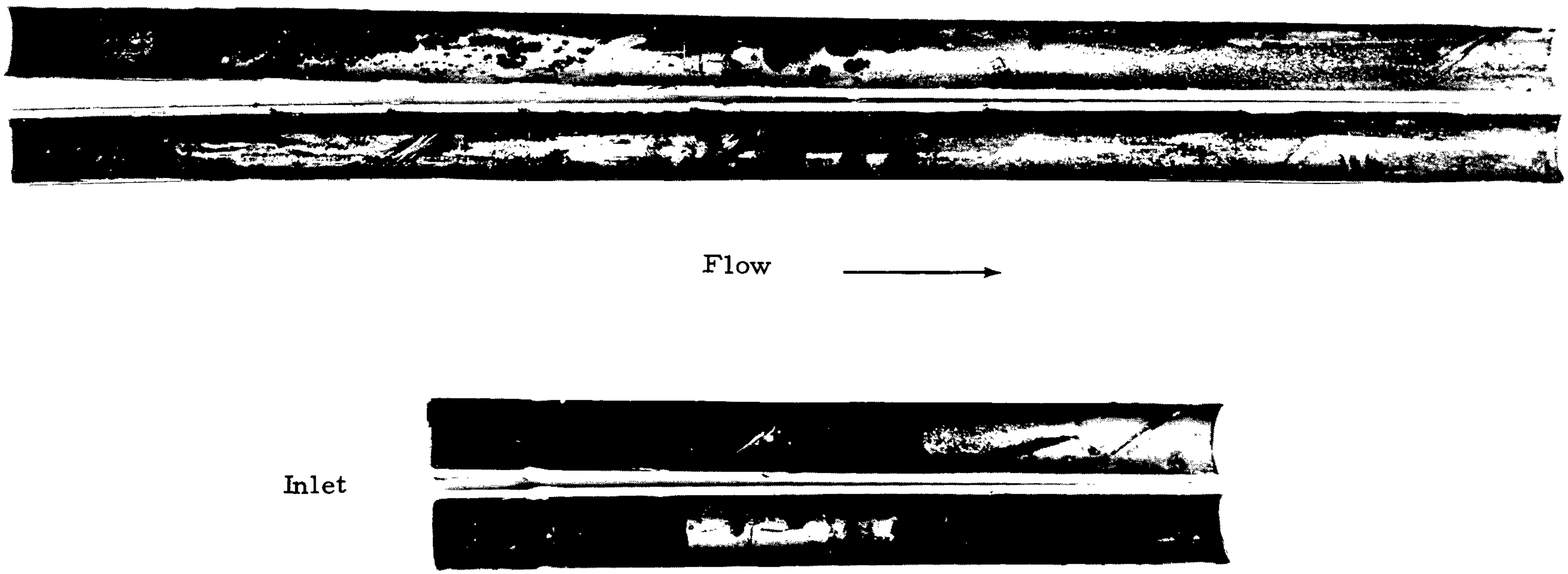


Figure 17. Appearance of the Inlet and Outlet of the Venturi Flow Meter. Approximately 1X.



Approximately 30X

Figure 18. Appearance of the Orifice in the Venturi Flow Meter.



Approximately 0.5X

Figure 19. Appearance of Boiler Inlet Section.

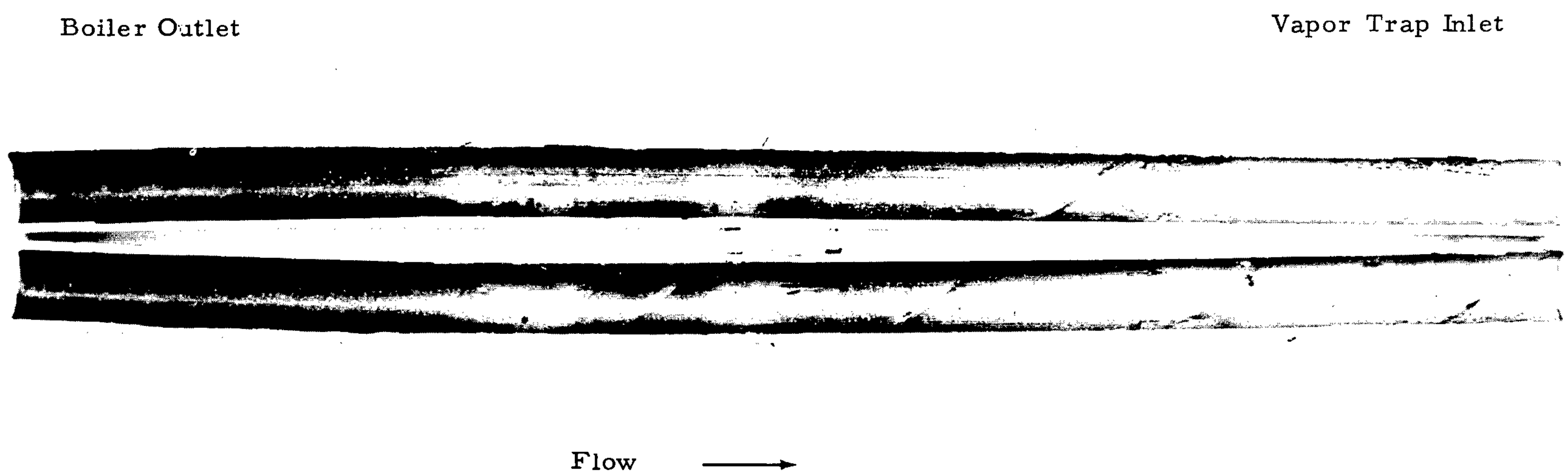
Flow ⟶

Approximately 0.5X

Figure 20. Appearance of Boiler Mid-Section.

Approximately 0.5X

Figure 21. Appearance of Liquid Level Region of Boiler Section.

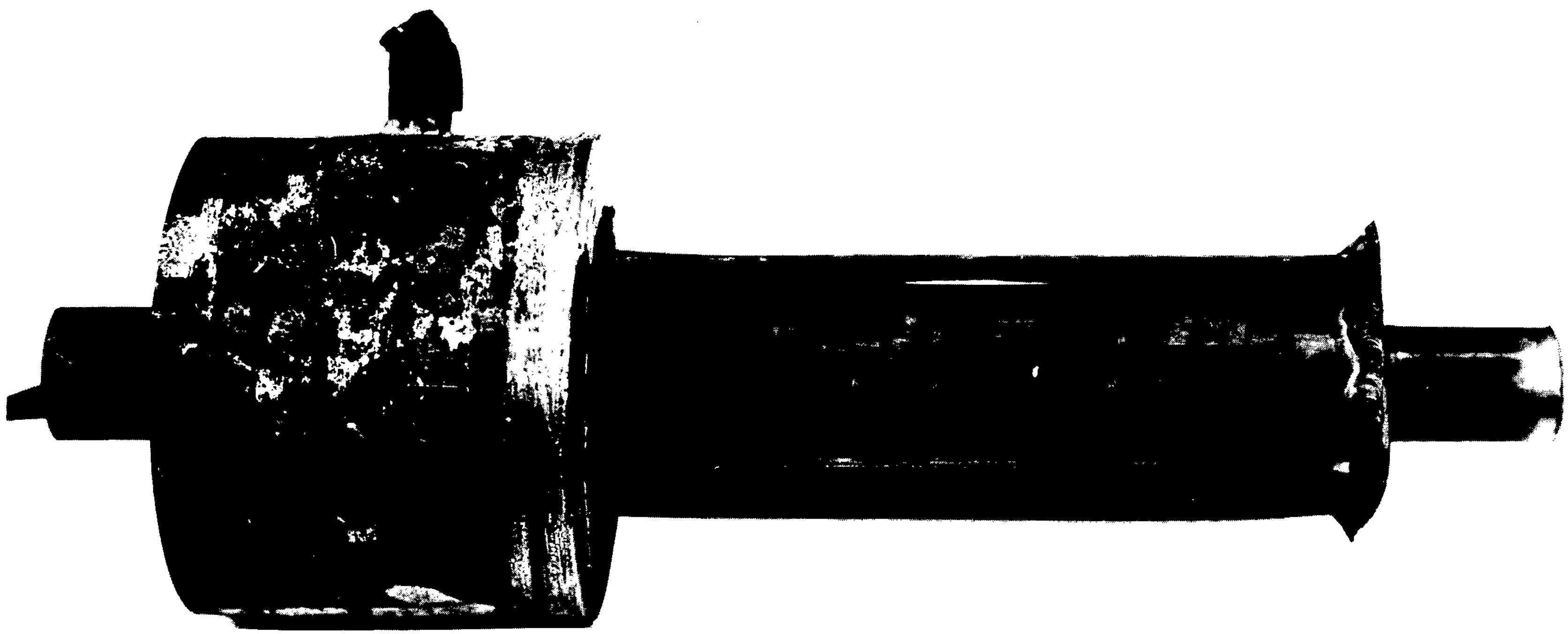


Approximately 0.5X

Figure 22. Appearance of Boiler Outlet and Vapor Trap Inlet Sections.

Approximately 0. 5X

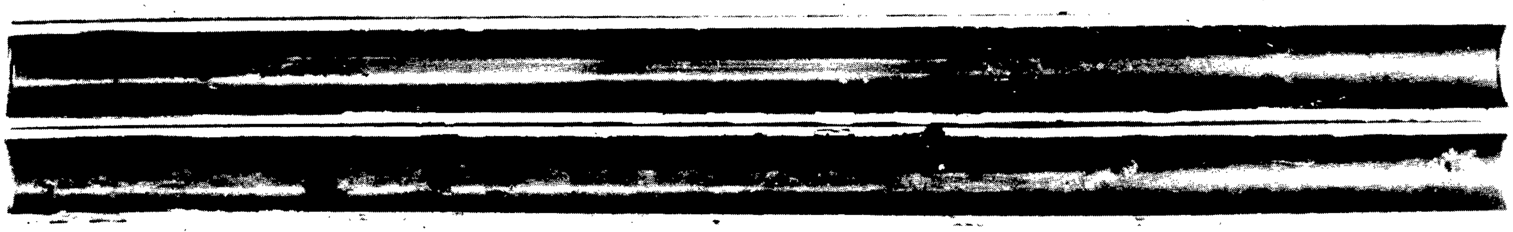
Figure 23. Appearance of Vapor Trap Inlet Section.



Flow ⟶

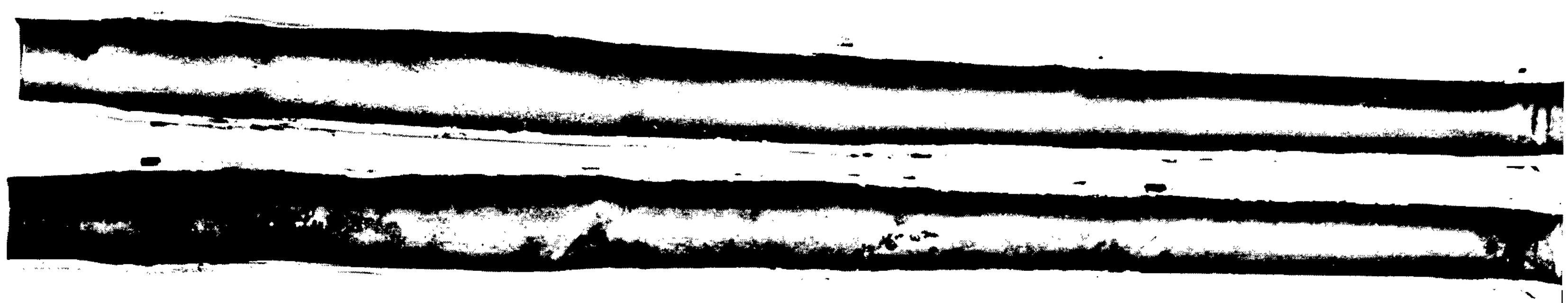
Approximately 0.8X

Figure 24. Appearance of the Magnet Poles in the Vapor Corrosion Product Separator.



Flow ⟶ Approximately 0.5X

Figure 25. Appearance of Superheater Mid-Section.



Flow ⟶ Approximately 0.5X

Figure 26. Appearance of Superheater Outlet Section.

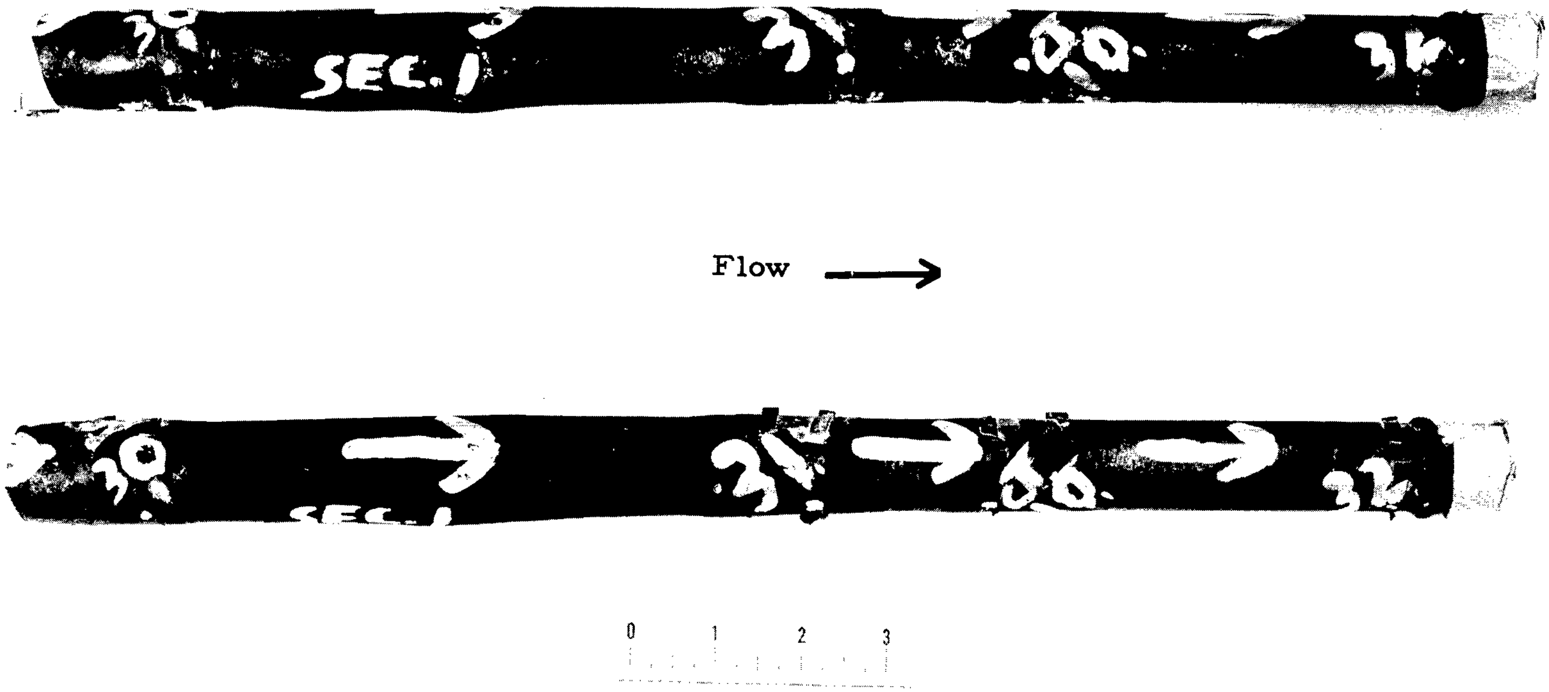
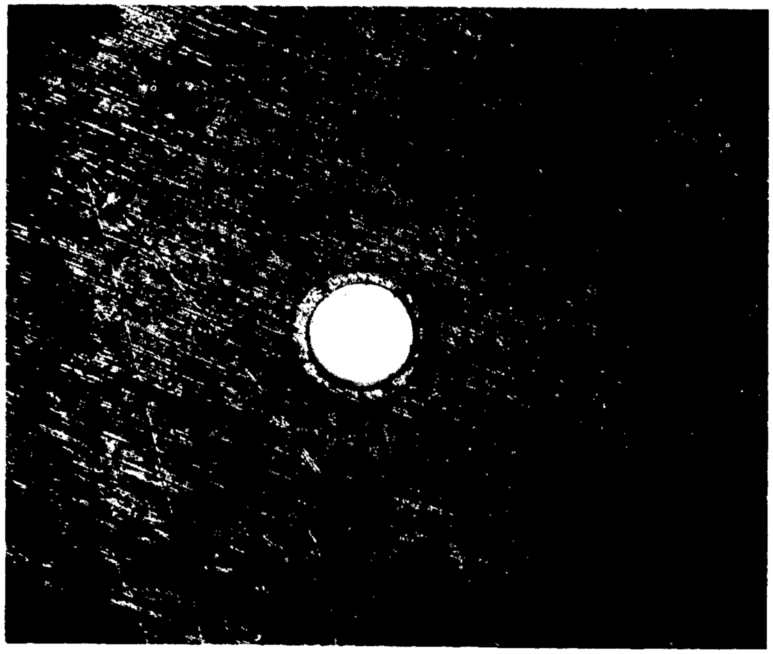
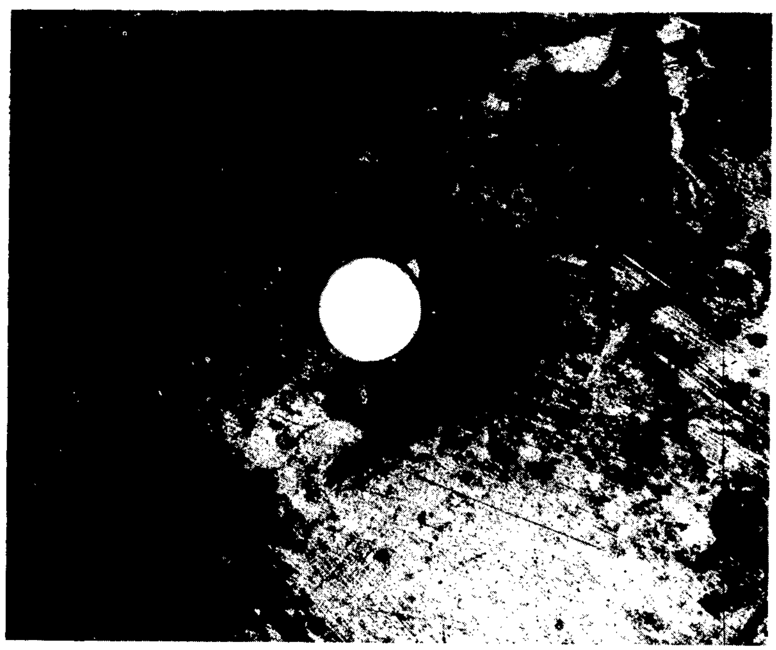


Figure 27 Appearance of the Superheater Outlet Section Showing the Deformation which Occurred.

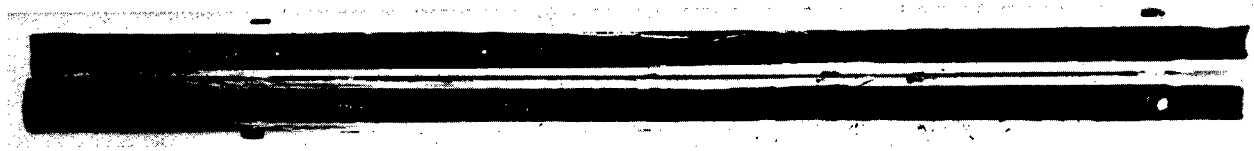


a) Inlet Approximately 10X



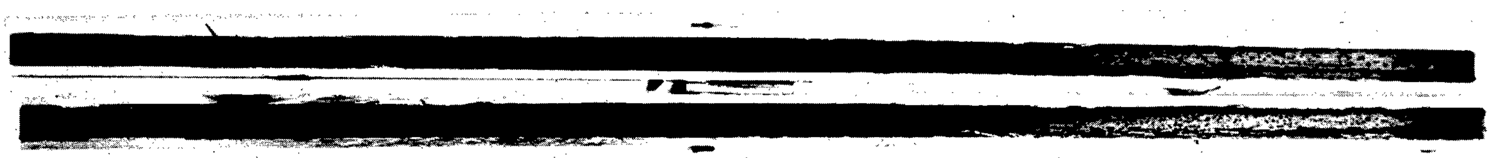
b) Outlet Approximately 10X

Figure 28. Appearance of Croloy 9M Orifice.



a) Condenser

Flow ⟶



b) Subcooler

Approximately 0.5X

Figure 29. Appearance of Condenser and Subcooler Sections.

A summary of the corrosion observations made by metallographic examination is listed in Tables 4 and 5. Corrosive attack was classified according to the chart presented in Figure 30 on the basis of microstructural examination. Representative photomicrographs illustrating the typical appearance of the loop sections are presented and will be specifically referred to in the following portions of this report. For comparison, reference is made to the microstructure of the as-received material illustrated in Figure 16.

Corrosion of the boiler inlet section of the loop was not severe and appeared to be limited to a pitting type of attack of 0.3 mil penetration. The attack of the swirl wire was of the same level of intensity but was intergranular. The average wall temperature measured in this section was 834°F.

An aggressive corrosion attack, significantly limited to the midsection of the boiler, where liquid mercury was at its highest temperature, was observed. Intergranular penetrations ranging up to 3.3 mils (Table 4) were measured in the Croloy 9M tube wall. Typical views of this corrosive attack are illustrated in the photomicrographs of Figures 31 and 32. The observed attack of the swirl wire in these sections corroborated the observed deterioration of the tube wall. An intergranular and layer attack of 2.0 mils depth was observed and measured on the swirl wire. This attack is illustrated in Figure 33b. The wall temperatures in this area of the loop varied from 1060 to 1142°F.

The intensity of the corrosive attack diminished near the boiler outlet. The tube wall exhibited a penetration of 1.1 mils (Figure 34). Metallographic examination of the swirl wire taken from the liquid/vapor interface revealed an intergranular type of corrosion of 0.6 mil penetration (Figure 33c). A similar intensity of attack was revealed in the inlet and midsection portions of the superheater. Intergranular corrosion (Figure 35 and Table 4) up to 1.7 mils penetration was measured. The swirl wire from these sections revealed 0.5 mil depth layer-type and 1.0 mil intergranular-type of corrosion at the superheater inlet and midsection, respectively, as shown in Figures 33d and 33e.

An increase in mercury corrosion of the Croloy 9M was noted in the outlet portion of the superheater. Intergranular corrosion to a depth varying between 1.2 and 2.5 mils (Figures 36 and 37) was observed. The swirl wire in this region exhibited 0.5 mil crevice attack, Figure 33f.

This corrosive attack at the superheater outlet indicates that high temperature condensation of the mercury vapor probably took place

TABLE 4

SUMMARY OF THE METALLOGRAPHIC EVALUATION OF THE MERCURY CORROSION ON VARIOUS SECTIONS OF THE LOOP

Location	Thermocouple Number	Average Temperature °F (*)	Maximum Corrosion (**) Penetration-Mils	Type
Boiler Inlet	2	834	0.3	P
Boiler Midsection	3	1050	2.75	I
Boiler Midsection	4	1060	2.2	I
Boiler Midsection	6	1142	3.3	I
Boiler Outlet	10	1078	1.1	I
Boiler Outlet	11	1128	1.0	I
Superheater Inlet	14	1074 Immersion	1.0	I
Superheater Inlet	17	1160	1.7	I
Superheater Midsection	24	1214	0.5	P
Superheater Outlet	30	1409	2.5	I
Superheater Outlet	31	1405	1.2	I
Condenser Inlet	37	698	-	-
Condenser Midsection	39	594	-	-
Subcooler	46	398	-	-

(*) Wall temperature unless otherwise indicated.

(**) P - pitting
I - intergranular

TABLE 5

METALLOGRAPHIC EVALUATION OF SWIRL WIRE FROM VARIOUS SECTIONS OF THE LOOP

Location	Average(*) Temperature °F	Maximum Corrosion Mils	Maximum Corrosion Type(**)
Boiler Inlet	800	0.3	I
Boiler Midsection	1142	2.0	I, L
Boiler, Liquid Vapor Interface	1128	0.6	I
Superheater Inlet	1074 Immersion	0.5	L
Superheater Midsection	1214	1.0	I
Superheater Outlet	1400	0.5	C

(*) Wall temperature unless otherwise indicated.

(**) I - intergranular

L - layer

C - crevice

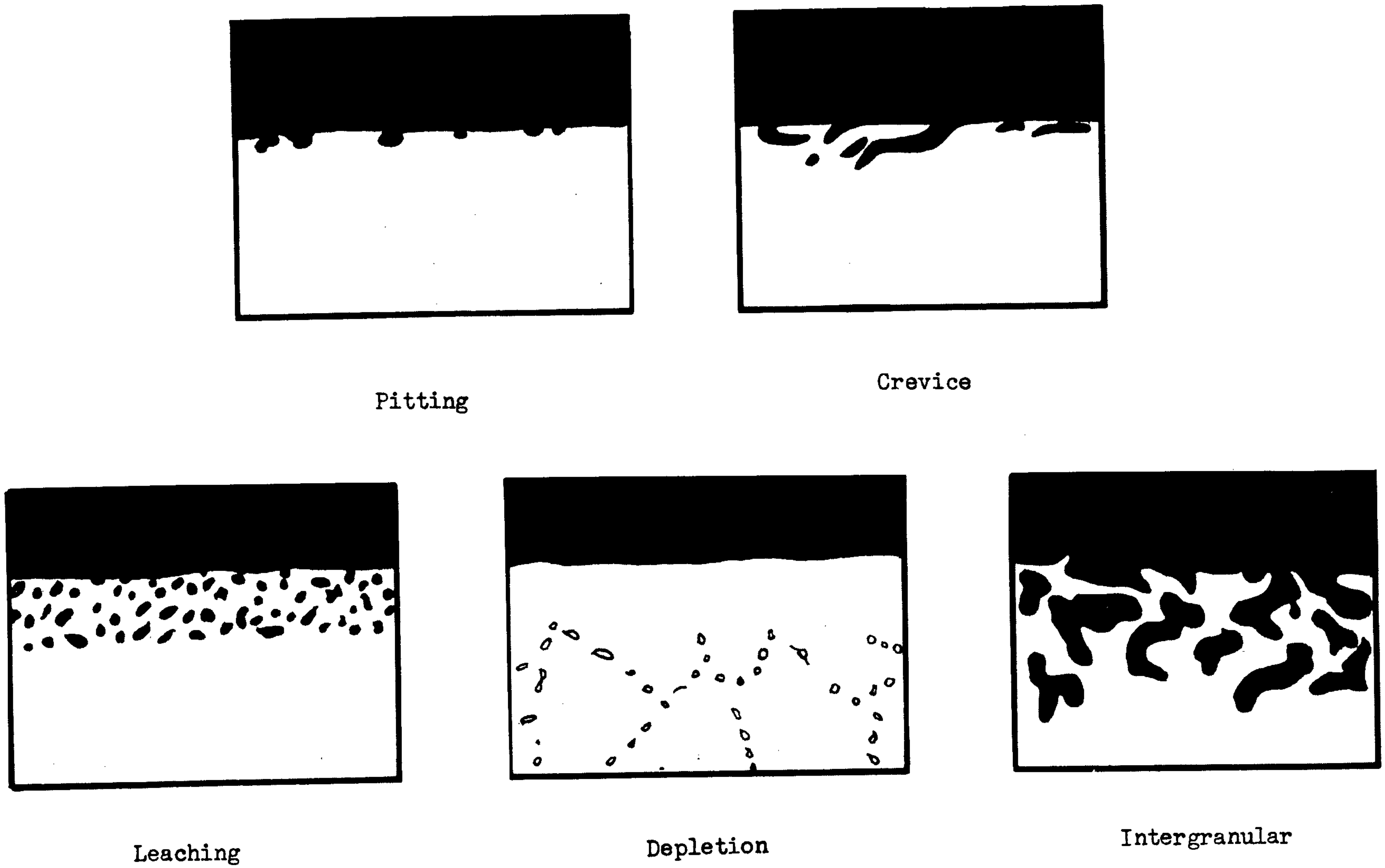
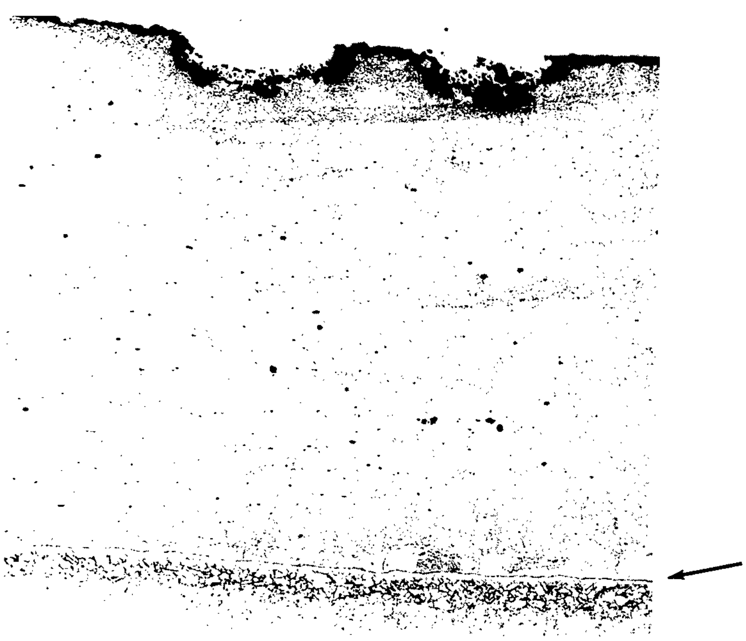


Figure 30. Examples Illustrating Types of Corrosive Attack.

250X

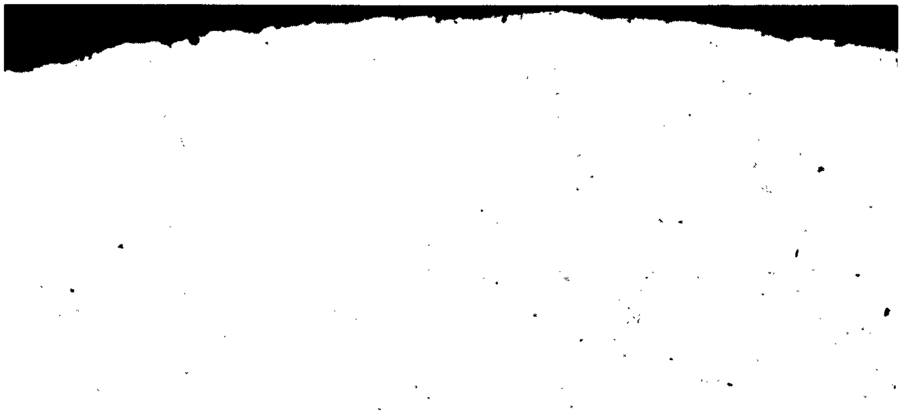
Figure 31. Intergranular Corrosion of Boiler Midsection.
Unetched



100 x

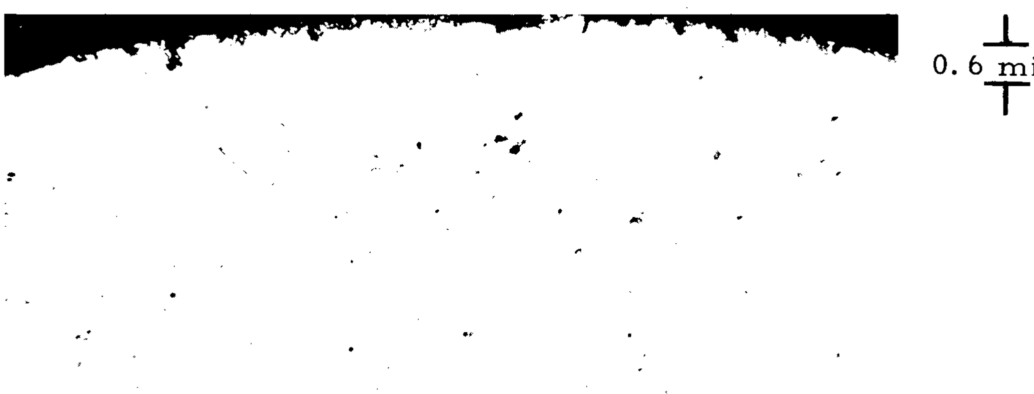
Figure 32. Intergranular Corrosion of Boiler Midsection.

Etchant - Carapella



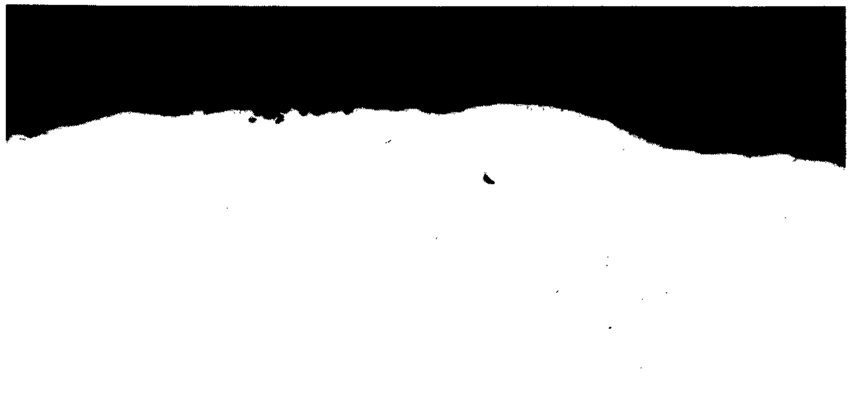
a) Intergranular Corrosion of Swirl Wire at Boiler Inlet

b) Intergranular Corrosion of Swirl Wire at Boiler Mid-Section



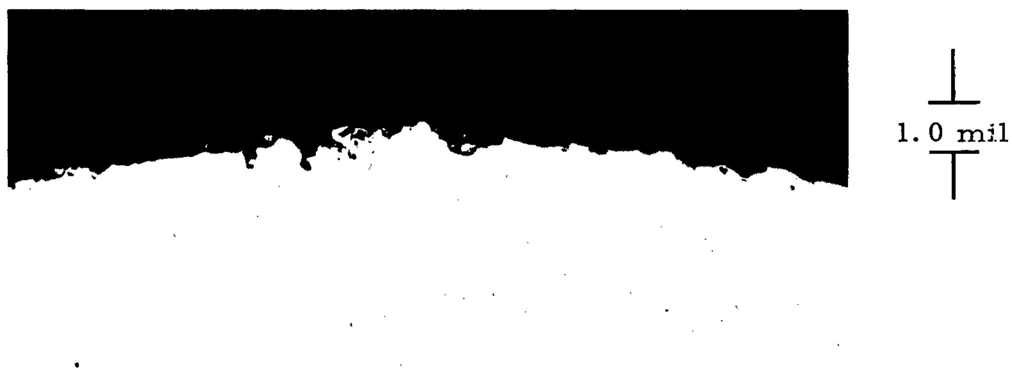
c) Intergranular Corrosion of Swirl Wire at Boiler Liquid/Vapor Interface

Figure 33. Corrosion of Croloy 9M Swirl Wire. Unetched 250X



250X

d) Layer Corrosion of Swirl Wire at Superheater Inlet



250X

e) Intergranular Corrosion of Swirl Wire at Superheater Mid-Section

250X

f) Crevice Corrosion of Swirl Wire at Superheater Outlet

Figure 33. (Continued).

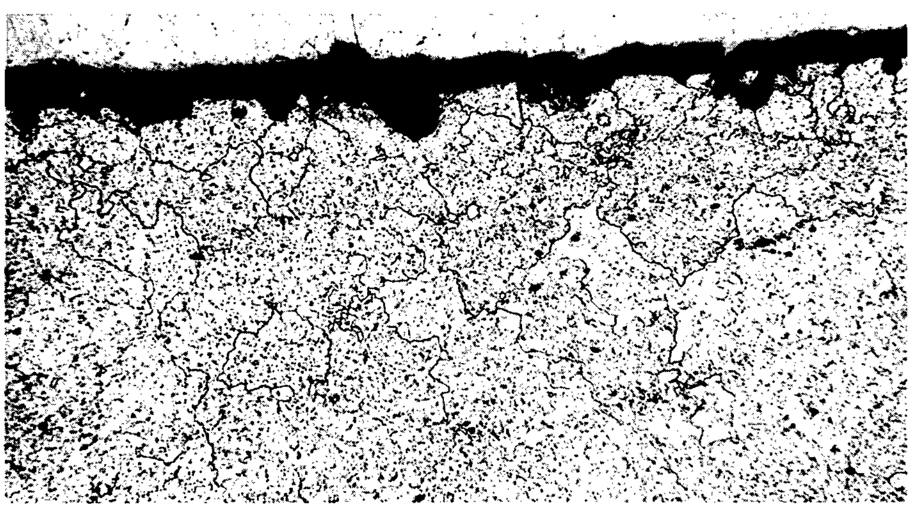
1. 1 mils

Croloy 9M

250X

Figure 34. Intergranular Corrosive Attack of Boiler Wall Near Outlet.

Etchant - Carapella



1. 0 mil

Croloy 9M

250X

Figure 35. Typical Intergranular Corrosion of Croloy 9M at Inlet to Superheater.

Etchant - Carapella

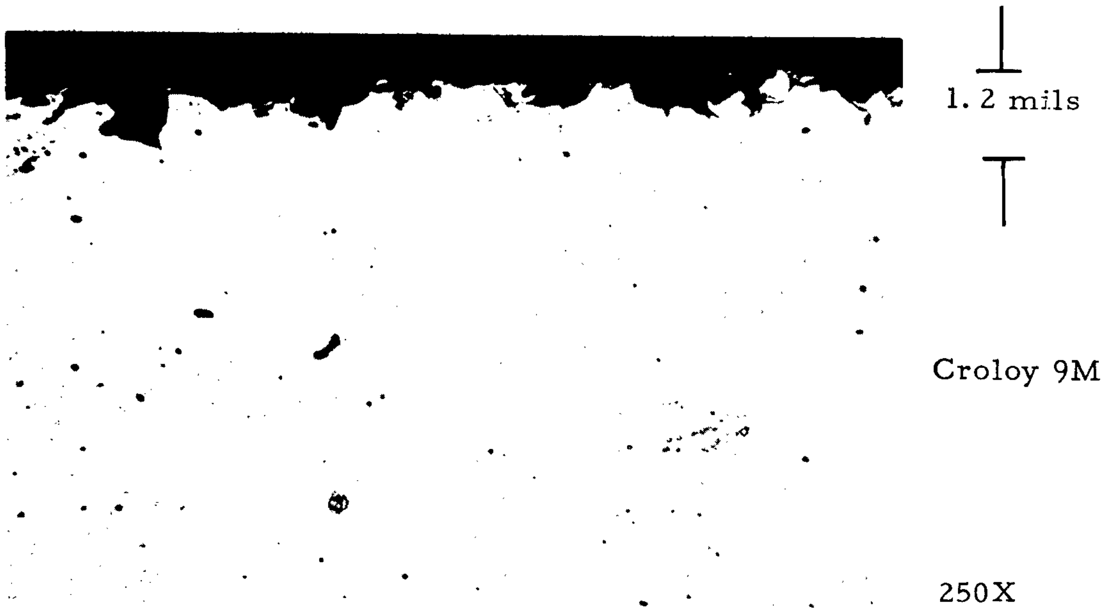
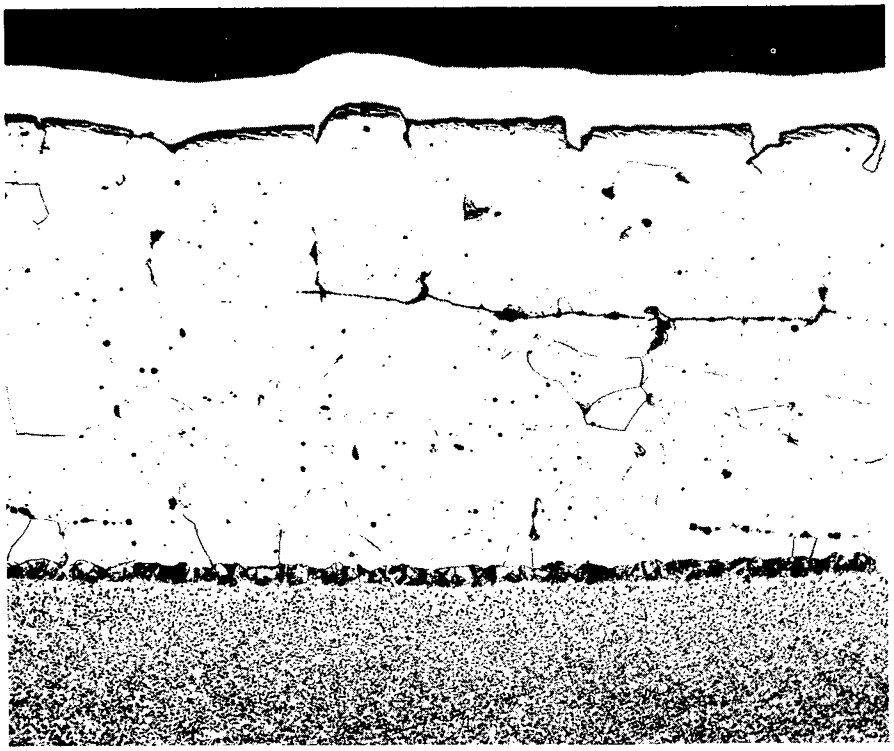


Figure 36. Intergranular Corrosion of Croloy 9M at Superheater Outlet.

Unetched



100X

Figure 37. Intergranular Corrosion of Superheater Wall.

Etchant - Carapella

during operation. A review of the loop operation data indicated that this may have occurred during the initial run of 448 hours. Geometrical problems caused by the throttling valve used during this initial operation of the loop prevented direct heating of the last several inches of the superheat section. The resultant drop in temperature caused the partial condensation of mercury. Typical before and after temperature readings for this area are as follows:

With Throttling Valve	With Orifice Plate
1082°F	1313°F

Corrosion by mercury under similar conditions has been previously noted as in the Sunflower CSUI-3 turboalternator.(4) However, if this high temperature condensation condition did cause the attack noted in this area, it is interesting to note the relatively short time of exposure, 448 hours, to induce this depth of penetration.

The degree of corrosion which existed in the condenser and subcooler portion of the loop could not be detected by metallography. These sections of the loop were constructed of 1/2-inch diameter Croloy 9M which unfortunately had an extremely rough surface in the as-received condition as illustrated in the photomicrograph of Figure 16b. Thus, any corrosion which may have occurred during operation of the loop could not be detected and measured with confidence. However, a deposit was present on the walls of both the colder portion of the condenser and the entire subcooler. X-ray diffraction has identified the deposit as principally α iron, with no indication of carbides. A chemical analysis for carbon of a specimen machined from the first 0.005 inch layer of the I.D. surface of the Croloy 9M condenser revealed a high carbon content (see Figure 53 and Table 10, to be discussed later in greater detail). Since metallographic examination (including microhardness) and x-ray diffraction did not disclose any carburization of the Croloy 9M tube wall in this section, it is indicated that the deposit contained a substantial quantity of carbon, in addition to α iron. The implication is that corrosion of the dissolution type probably took place in the high-temperature regions of the condenser. The corrosion products thus generated were subsequently either separated in the liquid corrosion product separator and/or deposited in the lower temperature regions of the condenser and subcooler. However, there is little reason to expect that catastrophic corrosion will occur in this section at the relatively low temperatures, 698 and 227°F in the condenser and subcooler, respectively. Cooper(3) reported less than 1.0 mil penetration in the condenser and subcooler sections of a Haynes alloy No. 25 forced circulation loop operated under very similar conditions for 5200 hours.

An electron beam microanalysis of samples of tubing from the boiler midsection, where the greatest attack was found, the superheater outlet, and the condenser midsection showed that slight depletion gradients were present at the I. D. Croloy 9M surface in all of the samples. No selective leaching of elements was apparent. This would seem to indicate that the attack experienced in the loop was of the general dissolution type.

Fabrication of the loop necessitated the joining by welding of duplex tubing (Croloy 9M/316 SS) to other duplex or to Croloy 9M tubing. In order to preserve the integrity of the Croloy 9M interface with mercury, the welds were performed in two steps. First the Croloy 9M was welded (TIG) using Type 505 SS (Croloy 9M) filler rod. This was followed by an overlay of Type 310 SS weld.

Metallographic study of the welds revealed that in some cases the Type 310 SS weld filler was exposed to mercury during loop operation. Such exposure resulted in isolated severe attack of the Type 310 SS weld. The cause of the exposure was due to either the inadvertent "burn through" of the backup 310 SS through the initial Croloy 9M weld or failure to completely join the Croloy 9M sections prior to welding the backup 310 SS. An example of the latter case is illustrated in the photomicrograph of Figure 38. The slight fissure between the two Croloy 9M components permitted mercury exposure to the Type 310 SS weld at a temperature of 1070°F. A leached layer attack of the Type 310 SS of 16.0 mils depth resulted. Corrosion of the Croloy 9M at the fissure was also noted which may appear excessively severe because of a bimetallic effect.

The corrosion capsule test results of other investigators[1] indicate that the corrosive attack by mercury of Croloy 9M is of the order of 0.8 mil penetration after 1000 hours at 1100°F. Thermal convection loops operated for 1000 hours at a boiling and condensing temperature of 1075°F (7.2 lbs/hr flow rate) revealed corrosive penetration of Croloy 9M to a depth of 1.2 mils[5]. A comparison of the corrosion data obtained from the operation of this loop with the above referenced results was made. The data compare favorably, as shown in Figure 39, which is a plot of these data assuming diffusion control[8].

D. CORROSION PRODUCTS

The material corroded from the loop components is termed corrosion products. These may be present as deposits in the loop or as solute or suspensoids in the mercury. In this report the weights of the corrosion products generated and their compositions are presented only as elemental metal and not as compounds.

Figure 38. Leached Layer Corrosion of Type 310 SS Weld Metal and Croloy 9M at Weldment of 1/2 Inch Diameter Croloy 9M Tube to 1.0 Inch Diameter Duplex Tube (Croloy 9M/316 SS) in Boiler Section. Operating Temperature—1070°F. Etchant - Carapella.

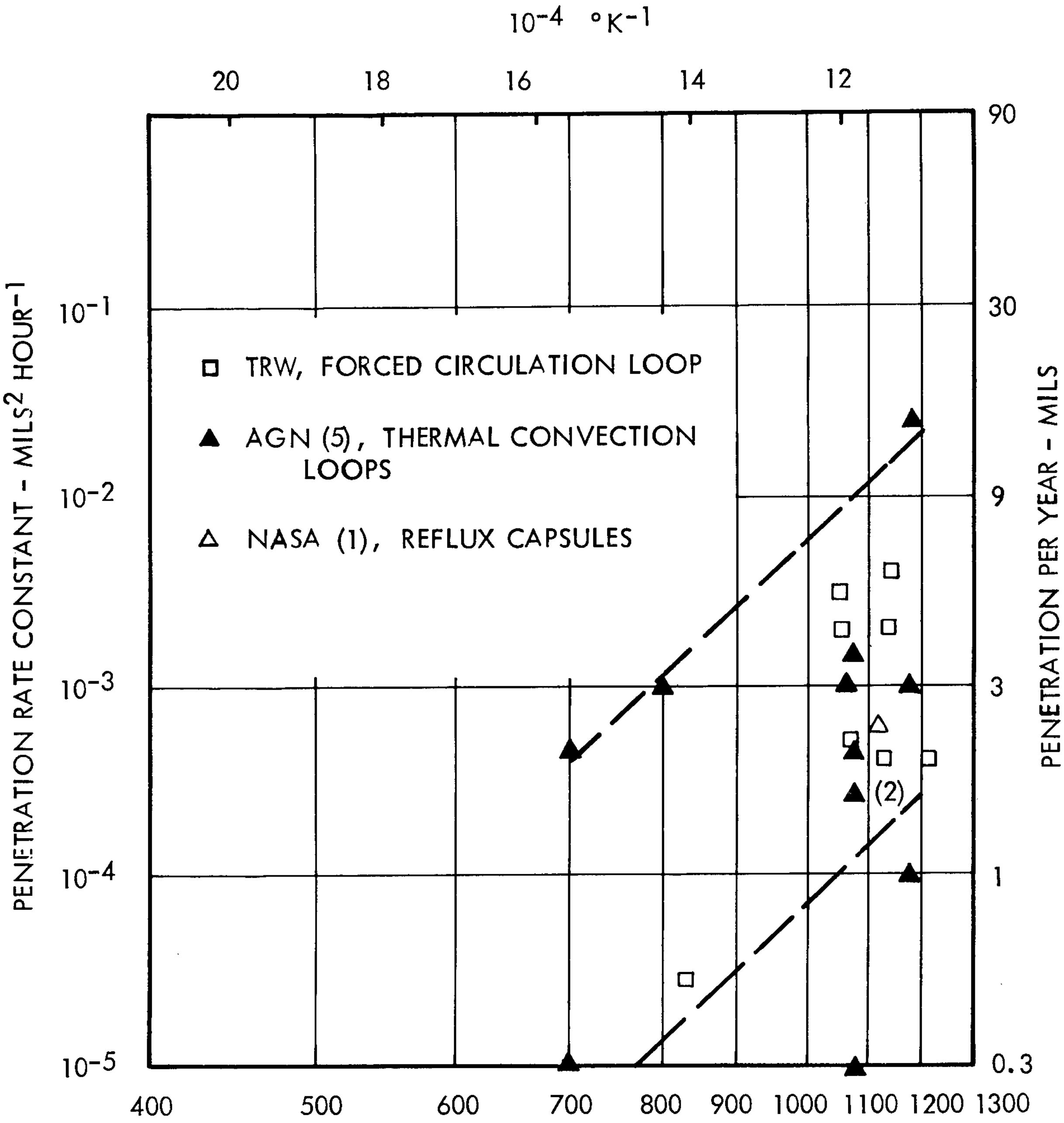


Figure 39. Plot of Corrosion Data for Croloy 9M Assuming Diffusion Control.

A summary of the corroded metal determination and composition is presented in Table 6. A total of 2.4677 gms of corroded metal (reported as elemental metal) was accounted for, of which 1.9543 gms were removed from the mercury by the separators. This figure indicates a separation efficiency of 79 percent, i.e., 54.3 percent for the vapor corrosion product separators and 24.7 percent for the liquid separator. This efficiency compares favorably with the 85 and 51 percent separation reported for the thermal convection and forced circulation loops employing the use of similar corrosion product separators.[2, 3] The vapor trap, vapor separator, and their common return line are considered as one corrosion product separation unit in this analysis (see Figure 1). The magnets were found to be demagnetized at the end of the test and, thus, were not effective. Consequently, of the three types of separators used in this loop, it is indicated that only chemical getters and centrifugal-type separators should be considered for corrosion product separation in the vapor region of mercury systems.

The corrosion product separation accomplished by the "C" magnet in the liquid separator was very low, 0.3%. A similar low level of separation by the liquid corrosion product separator magnets was also reported for the Sunflower Haynes alloy No. 25 forced circulation loop.[3]

Despite the poor performance of the magnets, the overall efficiency (79 percent) of the separators demonstrates the feasibility of incorporating corrosion product separators in forced circulation, two phase, mercury systems. The balance of the corroded, untrapped metal was accounted for as deposits on the walls of the boiler and condenser and as elemental metal in solution or as suspensoids in the mercury, 10.1 and 10.7 percent, respectively.

The results of the chemical analyses made of all of the corrosion products and mercury show that iron was the principal element removed by the mercury from the container metal. In all specimens except one, the iron content of the corroded metal was greater than 74 percent. The chromium content varied between zero and 31.2 percent. The molybdenum content of each specimen was low and never exceeded 6.4 percent. Nickel, manganese, and silicon were found in very small quantities in two of the deposits and in two of the mercury samples.

TABLE 6

CORRODED METAL SUMMARY AND COMPOSITION

Sample	Temperature °F	Weight of Corroded Metals gms	Percent of Total Corroded Metals	Composition of Corroded Metal Deposits, Percent Fe	Cr	Mo	Other
Loop Deposit							
a. Boiler Horizontal Leg	100-1144	0.1491	6.0	83.1	12.6	3.7	-
b. Boiler Vertical Leg	1070	0.0783	3.2	83.4	14.1	2.0	-
c. Condenser and Subcooler	698-227	0.0225	0.9	79.1	14.4	5.8	-
	Sub-Total	0.2499	10.1				
Liquid Corrosion Product Separator							
a. Magnet Pole Pieces	400-475	0.0083	0.3	88.2	9.4	1.2	-
b. Cb Wool	331	0.0896	3.6	91.2	5.4	2.0	-
c. Fe Wool (Top)	472	0.0308	1.2	95.2	0.0	0.0	-
d. Fe Wool (Bottom)	405	0.4850	19.6	96.0	0.0	0.0	-
	Sub-Total	0.6137	24.7				
Vapor Corrosion Product Separators							
a. Return Line	900	1.0423	42.2	91.7	4.5	0.7	1.9 Mn
b. Ta Wool (Trap)	1129	0.2440	9.9	83.3	13.3	1.1	-
c. Ta Wool (Separator)	1166	0.0313	1.3	82.5	12.6	4.2	-
d. Vapor Trap Wall Deposits	1129-1160	0.0230	0.9	79.7	12.3	1.8	3.2 Ni
	Sub-Total	1.3406	54.3				
Solute or Suspensoids in Hg From:							
a. Boiler	100-1144	0.0007	0.03	85.8	7.1	Nil	-
b. Condenser	698-227	0.0008	0.03	74.0	23.5	Nil	-
c. Liquid Separator	331-472	0.0355	1.44	82.8	8.7	1.1	4.5 Ni
d. Fill Line	1000	0.1327	5.37	80.1	12.2	6.4	-
e. Pump Head	100	0.0030	.12	93.5	4.6	Nil	-
f. Vapor Trap	1130-1160	0.0513	2.08	78.8	16.7	2.2	-
g. Pressure Recorder	1000	0.0395	1.60	47.8	31.2	0.5	0.8 Mn 5.8 Si 11.9 Ni
	Sub-Total	0.2635	10.7				
	Total	2.4677					

A comparison between the overall composition of the corroded metals with that of the loop tubing is shown below:

		Composition, Weight Percent	
Element	Corroded Metals	1" Duplex Tubing	1/2" Tubing
Fe	88.8	88.5	89.2
Cr	6.7	9.02	8.78
Mo	1.3	1.01	0.97
Mn	0.8	0.5	0.49
Ni	0.3	0.10	—
Si	0.1	0.56	0.46

The composition of corroded metals removed from the loop is essentially the same as that of the container materials, indicating that attack was of the general dissolution type.

E. X-RAY FLUORESCENT ANALYSIS

The ID surface of the loop was analyzed by x-ray fluorescent techniques, the results of which are semiquantitative. The values obtained for iron and chromium were plotted in an effort to determine any trends (Figures 40 and 41). The interpretation of these results is somewhat conjectural. Nevertheless, the plotted scatter-band indicates that the mean values for these elements are lower than the reported values of the Croloy 9M tube material given below:

	1" Duplex	1/2"
Fe	88.5	89.2
Cr	9.02	8.78

This trend would indicate that corrosion of the general dissolution type does take place rather than selective leaching. The random high iron or chromium content of the deposits and several of the mercury samples also reflect this indication.

Two sections of the loop are indicated by this plot to have a depletion of chromium, the last half of the boiler, and the condenser and subcooler. The metallographic examination of the boiler revealed the severest corrosive attack by mercury and thus may substantiate this low chromium content. However, the low chromium content of

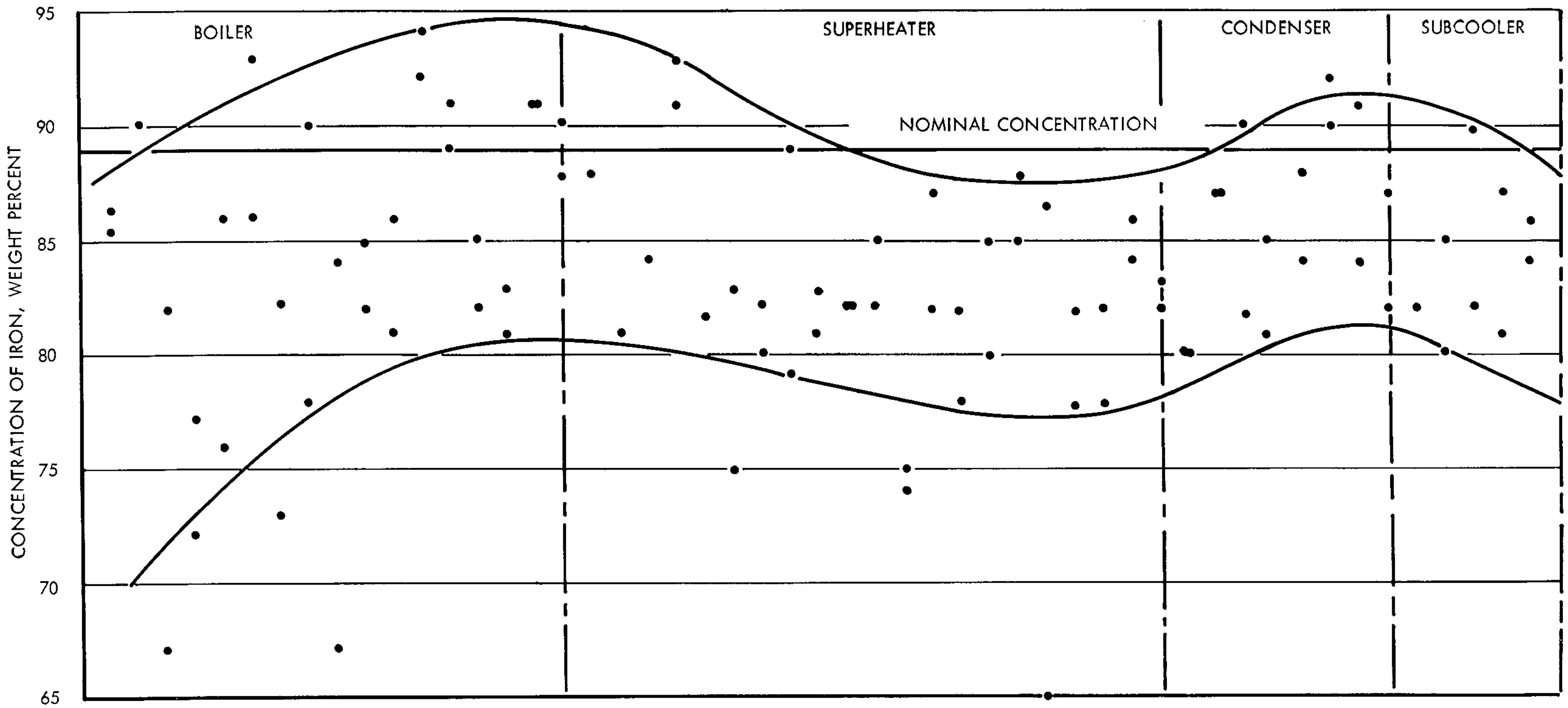


Figure 40. Variation of Iron Content of I. D. Surface with Loop Section.

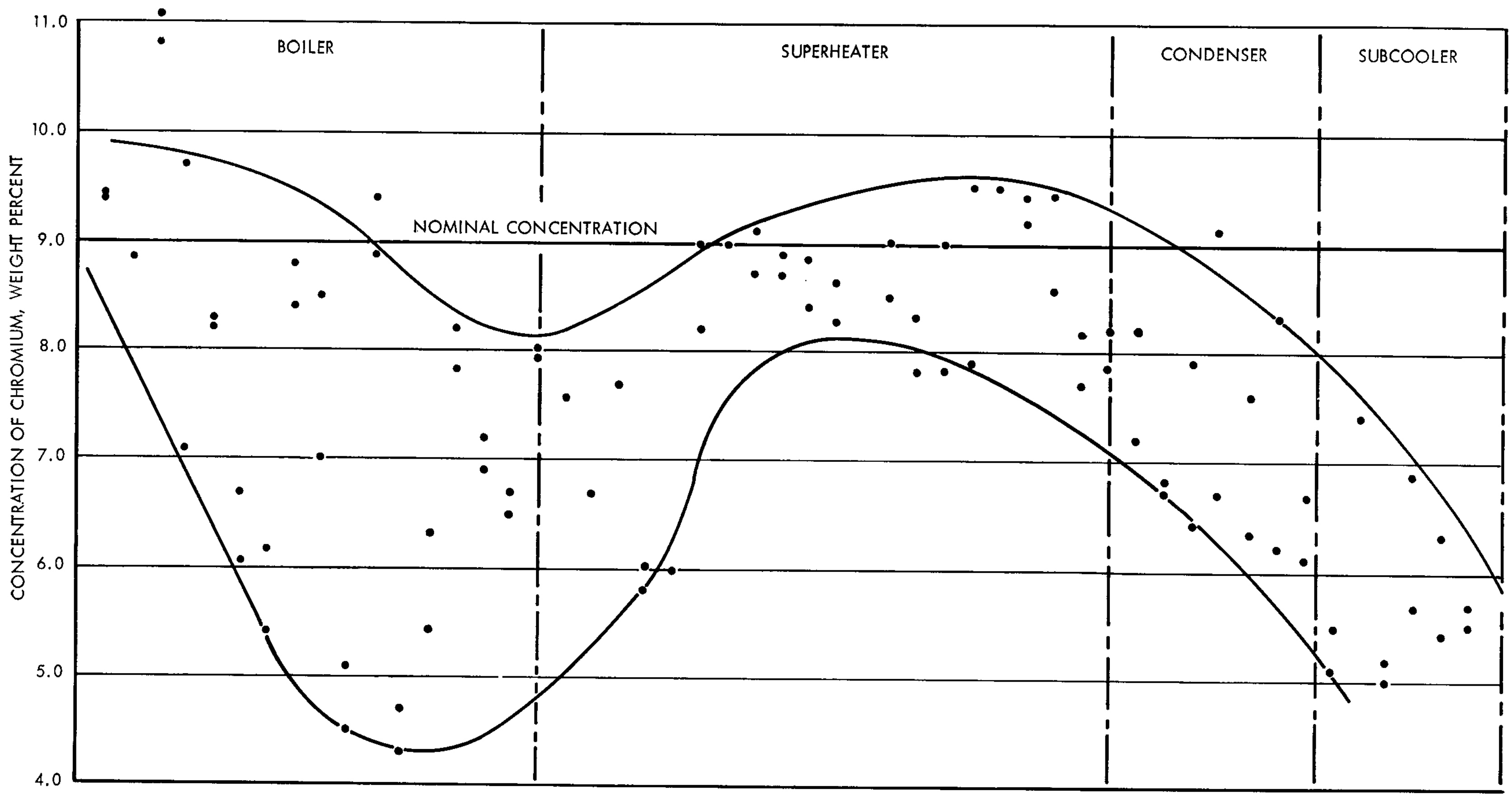


Figure 41. Variation of Chromium Content of I. D. Surface with Loop Section.

the condenser and subcooler ID surface cannot be explained by the metallographic results.

F. CROLOY 9M THROTTLING VALVE

It will be recalled that a very severe leak developed in the Croloy 9M throttling valve after 448 hours of operation and that the valve was removed and replaced with a Croloy 9M orifice. During this period of operation, the average temperature on the inlet side of the valve was 1090°F (39°F superheat), while the average outlet temperature was 758°F (9°F superheat).

Disassembly of the valve revealed that failure was brought about by "freezing" of the handle to a threaded extension welded to the bellows. This resulted in twisting of the bellows and eventual failure. Figure 42 shows the disassembled valve, and Figure 43 shows the deformed bellows.

The valve was sectioned and mounted for metallographic evaluation. Examination showed that a deposit had formed on the outlet side of the orifice in the valve. Two foreign metallic chips were also found in this area of the valve. The combination of the deposit and metallic chips caused a considerable reduction in the flow passage in the valve. This explains the control difficulties experienced during initial loop operation when fine adjustment of the valve could not be obtained. A photomicrograph of this area of the valve is shown in Figure 44.

An electron beam microanalysis was performed on the larger of the two metallic chips and on the deposit by Advanced Metals Research Corporation. A careful comparison of the iron, chromium, and molybdenum x-ray line intensities for the base alloy and the metallic chip revealed no significant differences, as seen in Figures 45 and 46. In addition, the manganese and silicon content of these two zones appeared to be identical. It is thus apparent that the metallic chip is Croloy 9M, probably a residual particle remaining in the valve after fabrication of this component.

The iron, chromium, and molybdenum distributions in the deposit are shown in Figures 47, 48, and 49, respectively. It is seen here that the deposit is iron-rich and chromium- and molybdenum-poor. The chromium shows a small gradient in the vicinity of the deposit-base metal interface but rapidly falls to less than one percent. The molybdenum content drops abruptly to approximately zero percent at the deposit-base metal interface. The iron concentration rises to roughly 90 to 95 percent. Chromium-rich phases are present in both the deposit

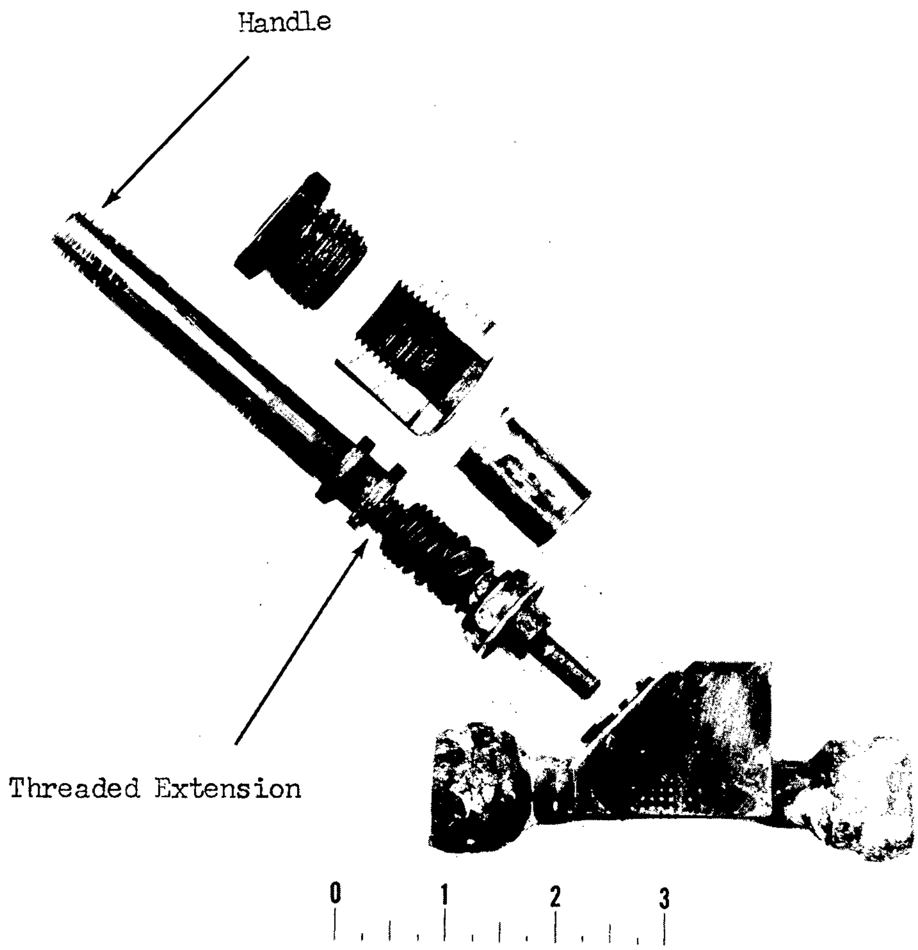


Figure 42. Disassembled Croloy 9M Throttling Valve.

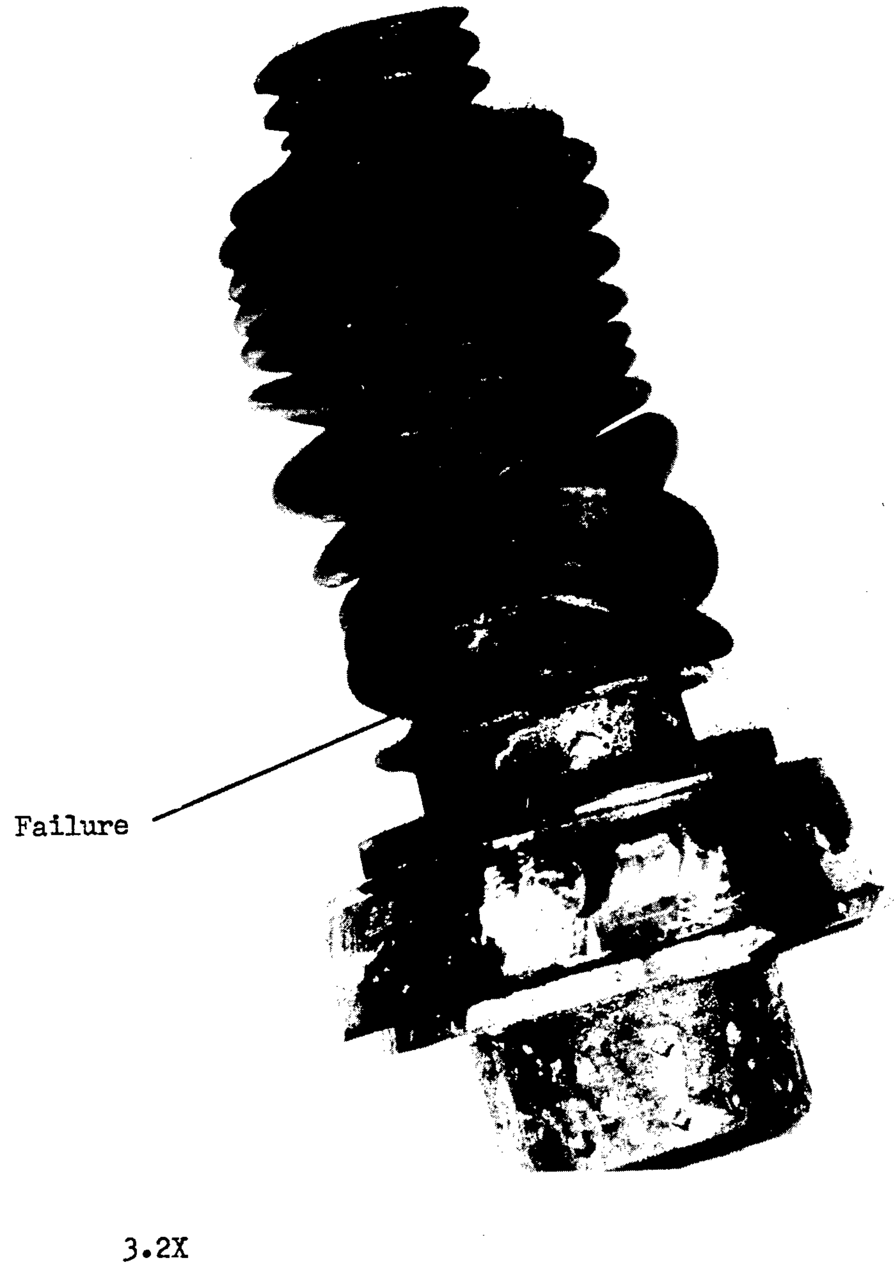
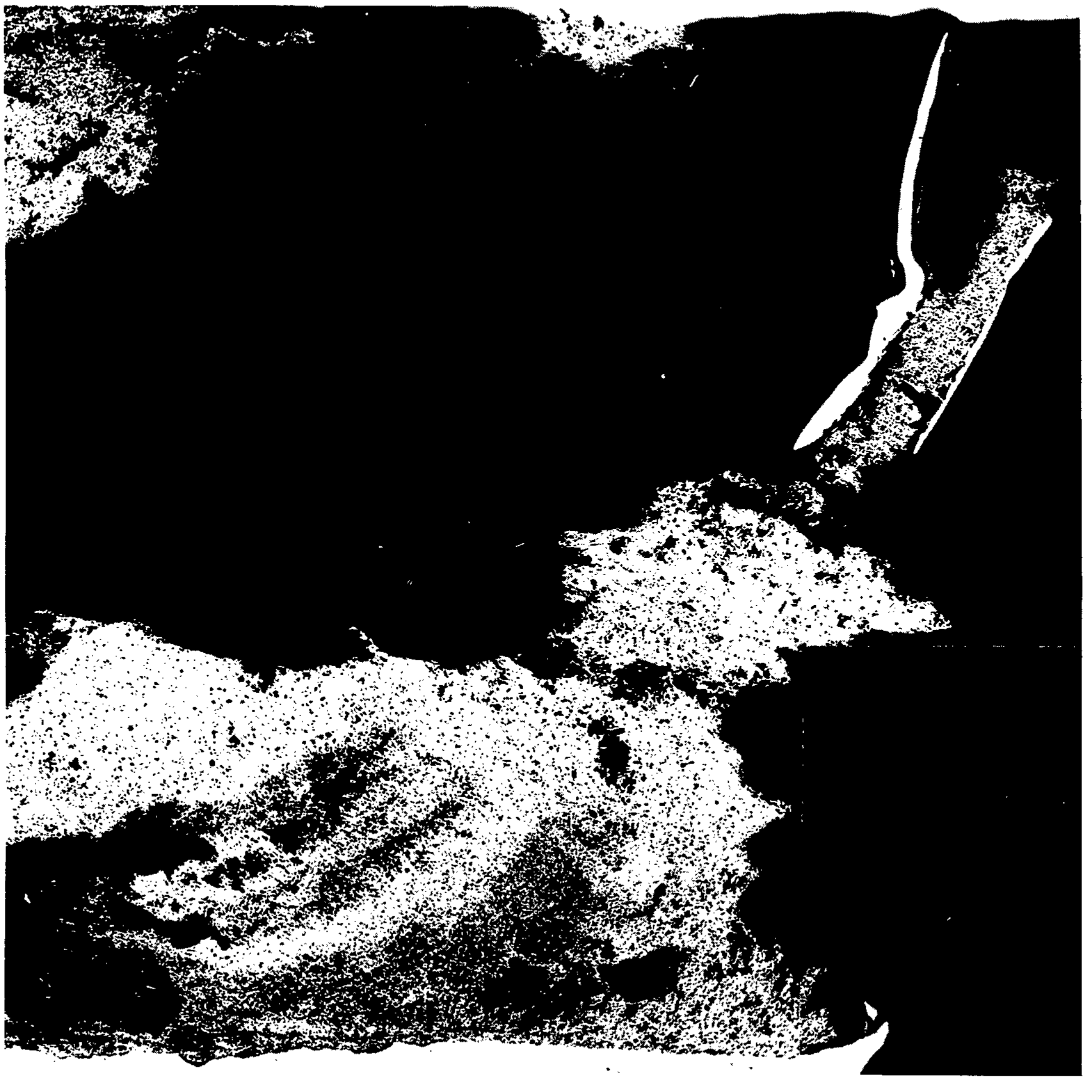


Figure 43. Type 321 SS Bellows from Croloy 9M Throttling Valve.



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100X

Figure 44. Composite Photomicrograph of the Outlet Side of the Orifice in the Croloy 9M Throttling Valve.

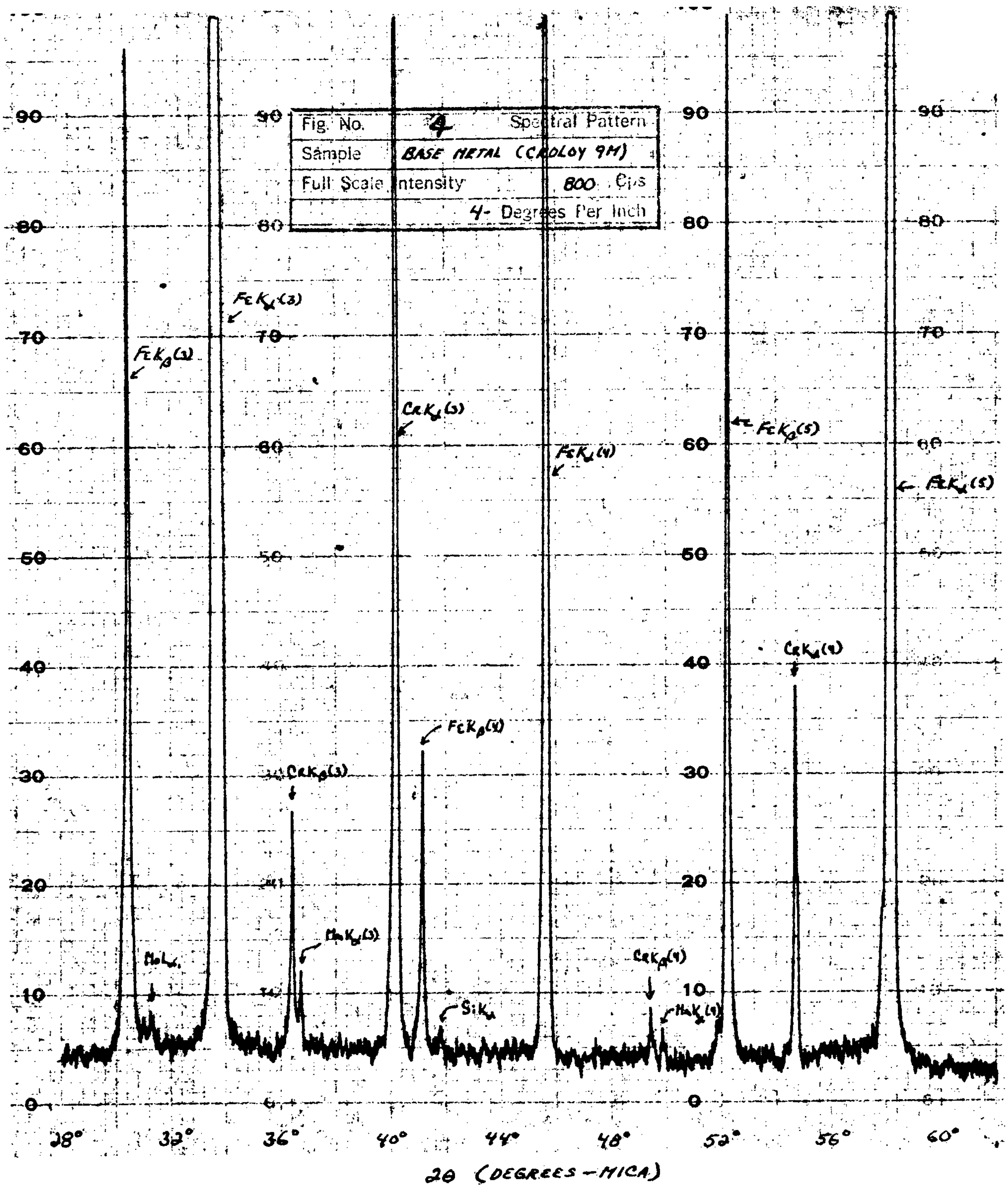


Figure 45. Spectral Pattern of Croloy 9M Base Metal.

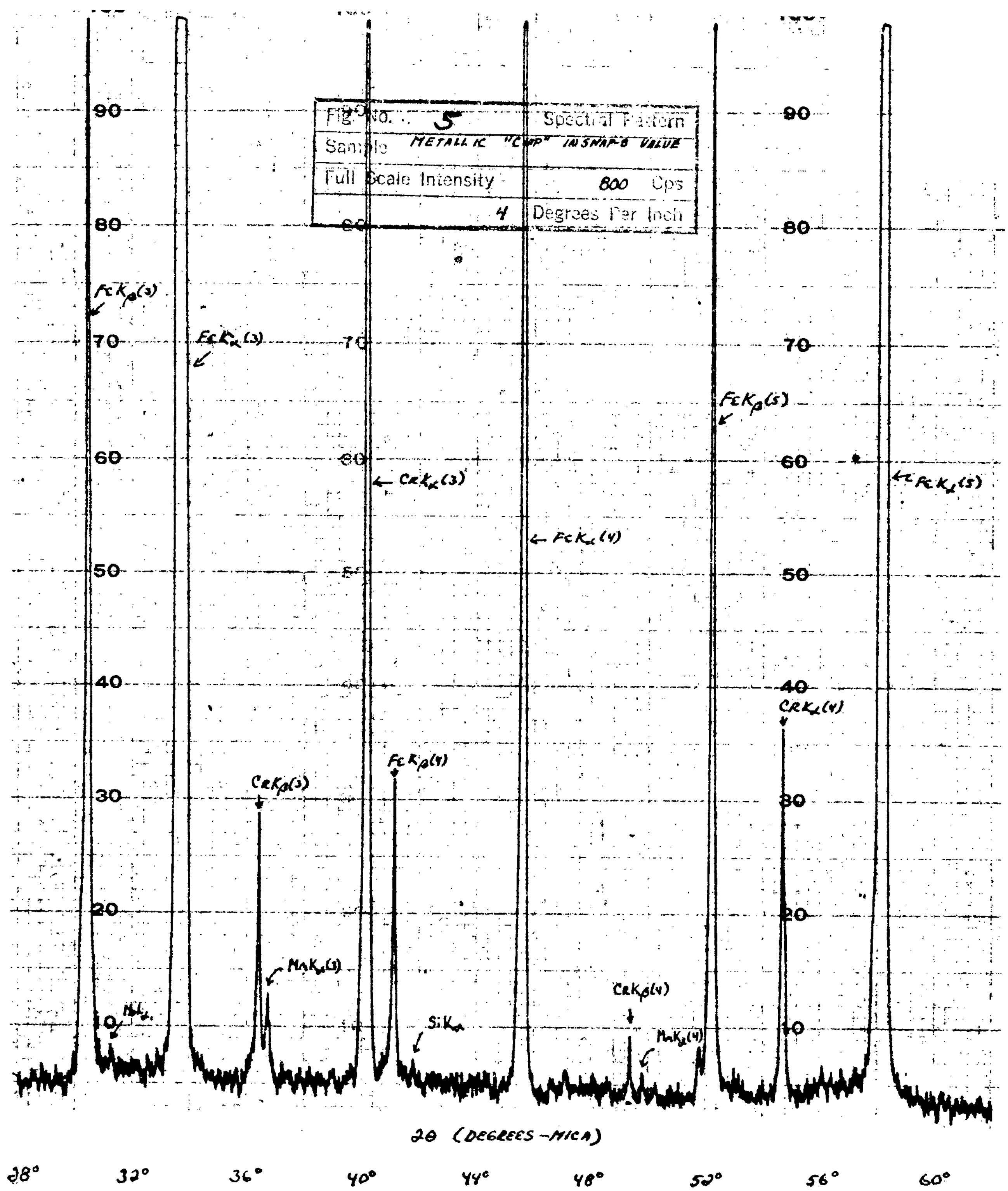


Figure 46. Spectral Pattern of the Metallic Chip Found in the Croloy 9M Throttling Valve.

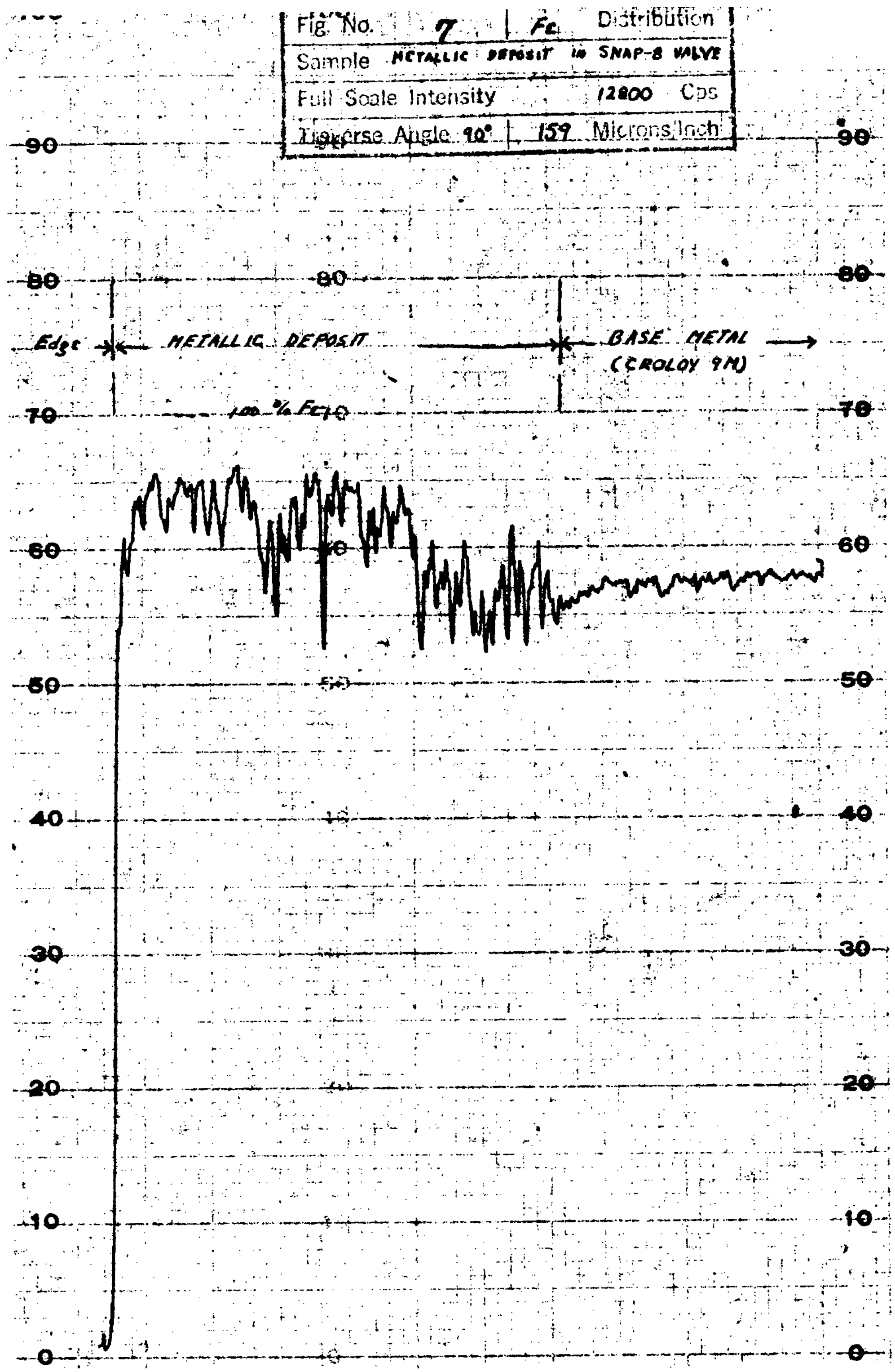


Figure 47. Distribution of Iron in the Deposit Found in the Croloy 9M Throttling Valve.

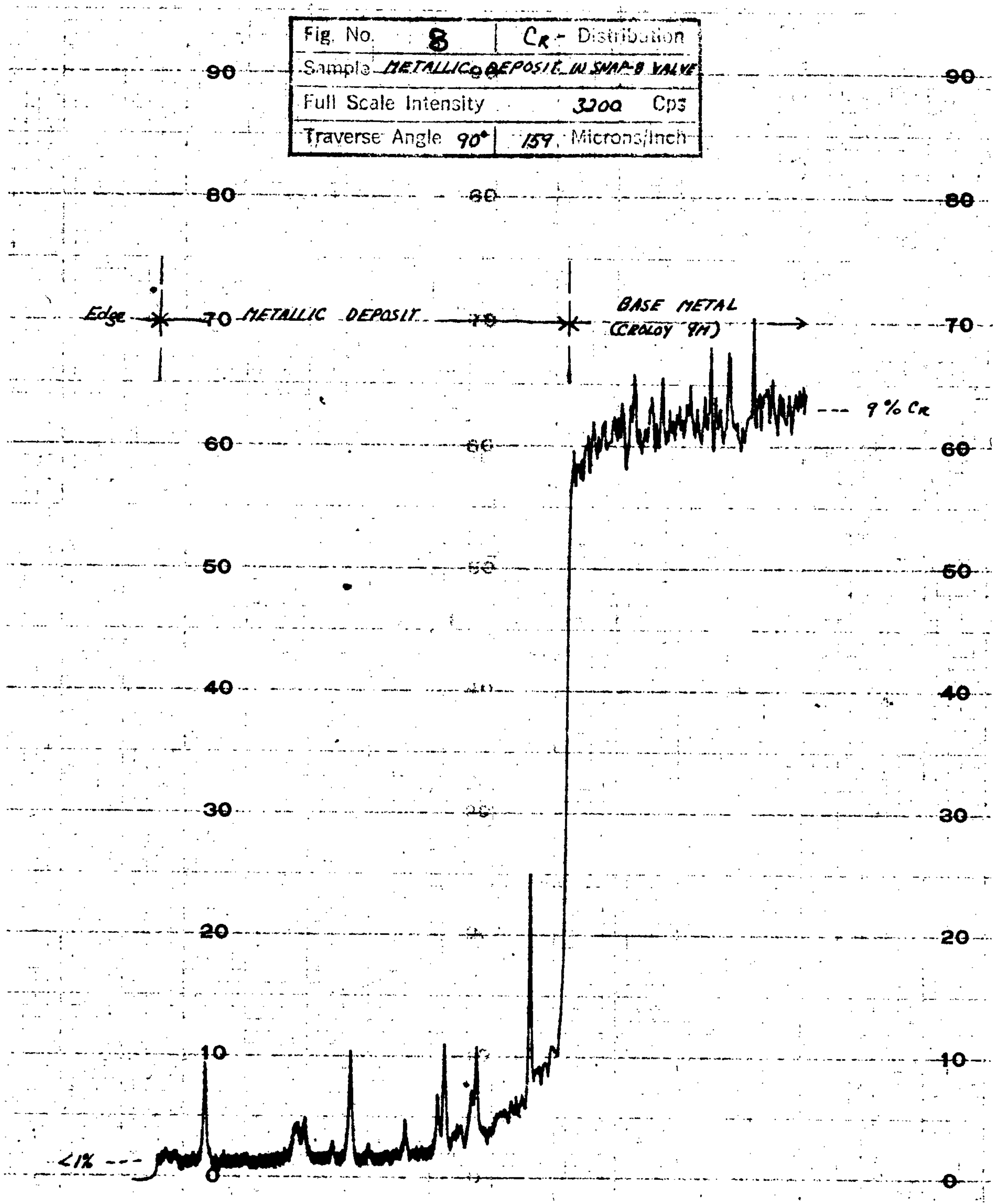


Figure 48. Distribution of Chromium in the Deposit Found in the Croloy 9M Throttling Valve.

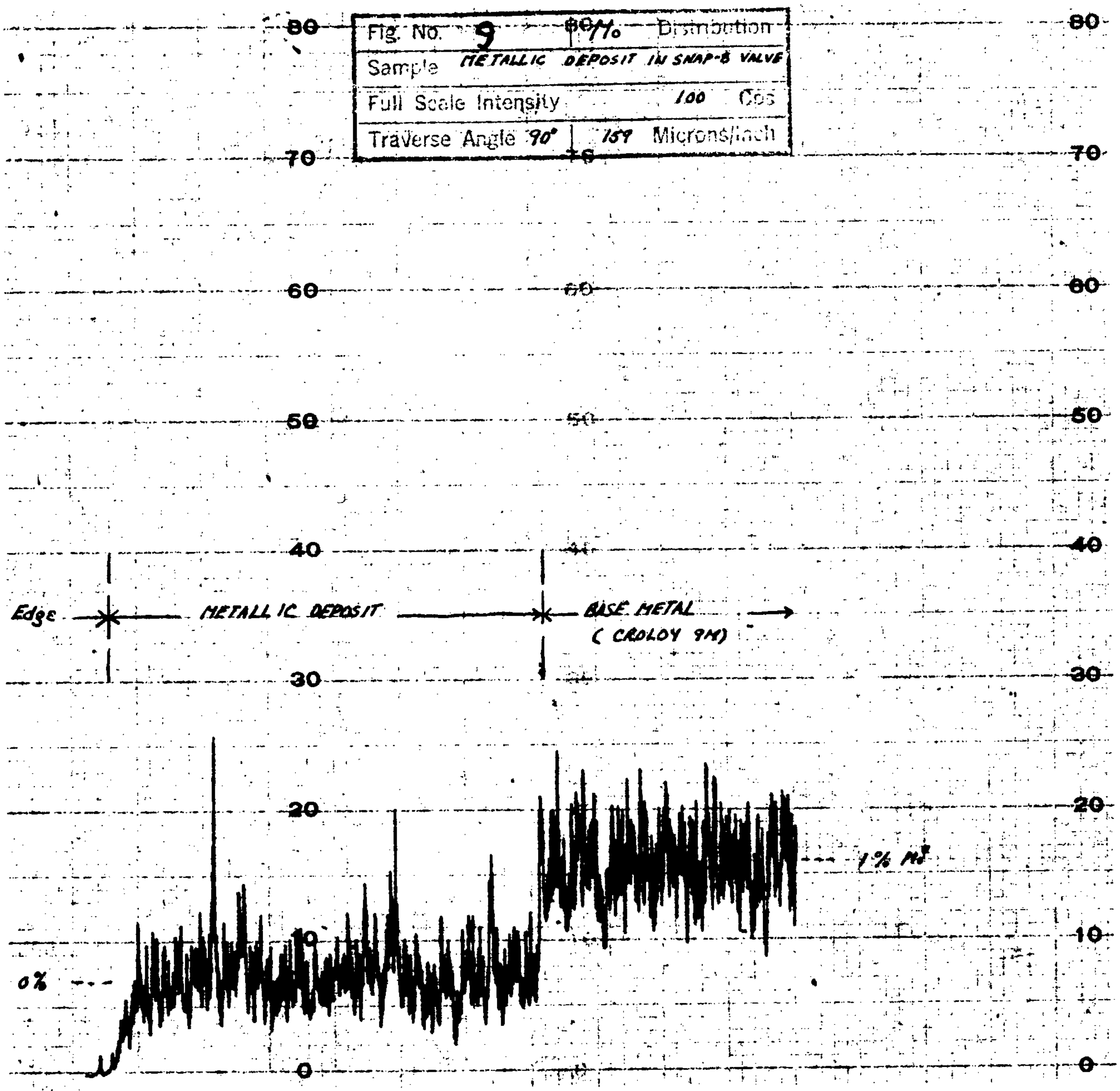


Figure 49. Distribution of Molybdenum in the Deposit Found in the Croloy 9M Throttling Valve.

and the base metal as evidenced by the sharp intensity spikes. These phases are very likely carbides. The balance of 5 to 10 percent material not seen in the analysis (90 to 95 percent iron, approximately one percent chromium and molybdenum) is probably a result of the porosity in the deposit rather than the presence of an additional element. The sharp decrease in both the chromium and molybdenum at the deposit-base metal interface suggests that iron has been removed from another part of the system and has been transported to the throttling valve. This is in agreement with the chemical and x-ray fluorescent evaluations of the remainder of the system.

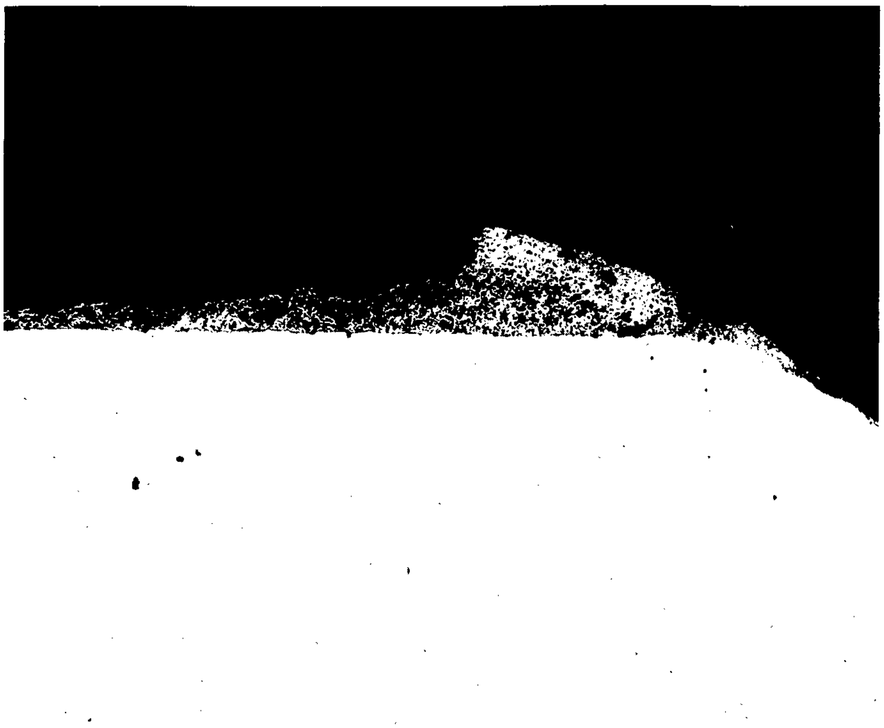
The stem (Croloy 9M) and the bellows (Type 321 SS) from the valve were also examined metallographically. As seen in Figure 50, some slight deposition was observed on the valve stem. The stem also suffered some corrosion and/or erosion damage as shown in Figure 51. The maximum depth of attack was approximately four mils. Some slight attack ($<$ one mil) was also observed on the Type 321 SS bellows, as shown in Figure 52.

G. CARBON DIFFUSION OF CROLOY 9M/TYPE 316 SS TUBING

A measure of the metallurgical stability of Croloy 9M was made by microhardness tests upon metallographic sections taken from various portions of the loop. The hardness values were obtained on a Kentron hardness tester using a 100 gm load. The hardness values obtained are presented in Table 7 as Knoop and also the equivalent Rockwell hardness.

Two hardness traverses were made of the loop tube wall: (1) from the boiler section (wall temperature - 1128°F) and (2) the superheater outlet (immersion thermocouple - 1264°F). The results are listed in Tables 8 and 9.

The carbon diffusion of the Croloy 9M/Type 316 SS tubing as indicated by the initial metallographic observations of loop specimens prompted a series of carbon analyses to be run on various loop sections. Samples of the tube were taken at 0.005" layers starting from the ID surface. Three sections of the loop wall, boiler, superheater, and condenser, were sampled in this manner and their carbon contents were determined. A total of nine samples (total depth 0.045 inches) were taken from each section except the condenser for which only six samples were obtained (total depth 0.030 inches). The results of these analyses are presented in Table 10 and plotted in Figure 53.



Unetched 100X

Figure 50. Deposition on the Stem of the Croloy 9M Throttling Valve.

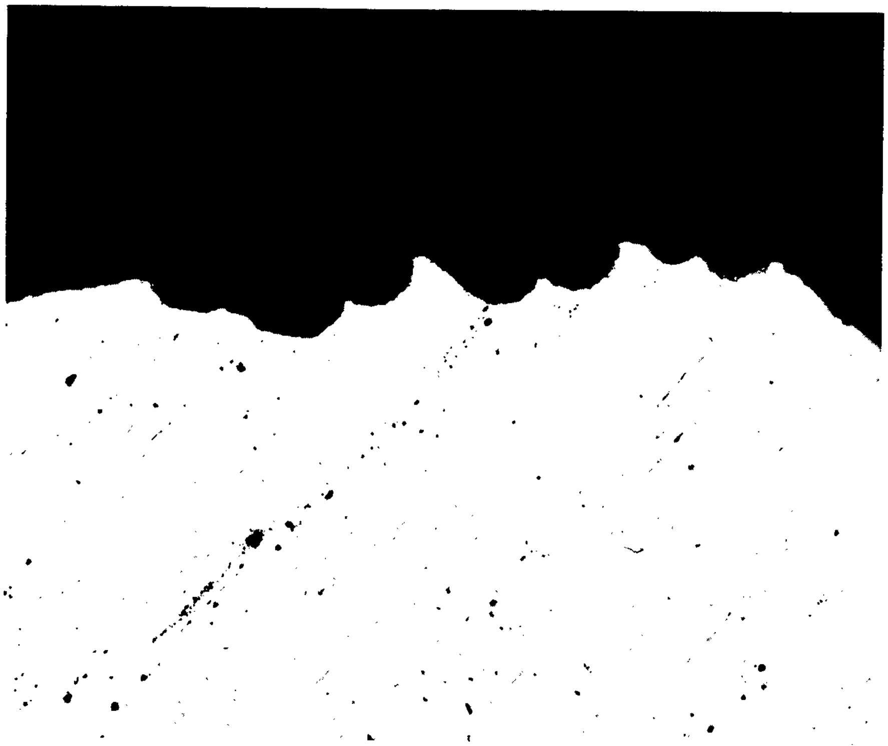


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Figure 51. Attack on the Stem of the Croloy 9M Throttling Valve.

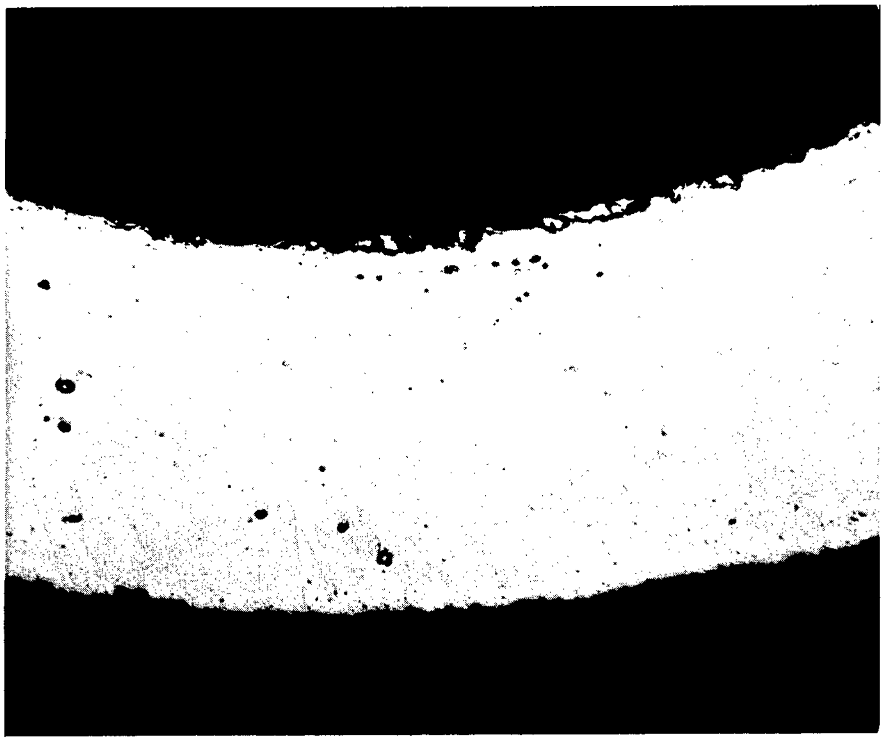

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Figure 52. Section of the Type 321 SS Bellows from the Croloy 9M Throttling Valve.

TABLE 7

HARDNESS VALUES OF LOOP MATERIAL FROM VARIOUS SECTIONS AFTER 2900 HOURS OF OPERATION

Specimen Number	Location	Average Operating Temperature* °F		Hardness Knoop	Hardness Rockwell Equivalent
56	Boiler Inlet	1059	Croloy	185	$R_B 90$
64	Boiler Mid Section	1128	Croloy 316 SS	222 249	$R_B 96$ $R_B 98$
67	Superheater Inlet	1074 Immersion	Croloy 316 SS	165 189	$R_B 85$ $R_B 90.5$
84	Superheater	1375	Croloy 316 SS	163 260	$R_B 84$ $R_B 99$
87	Superheater	1400	Croloy 316 SS	182 289	$R_B 89$ $R_C 24$
91	Superheater	1264 Immersion	Croloy 316 SS	187 325	$R_B 90$ $R_C 29$
105	Condenser Outlet	550	Croloy	180	$R_B 89$
107	Subcooler	346	Croloy	175	$R_B 87.5$
Original Material	1" diameter	-	Croloy 316	162 215	$R_B 85$ $R_B 96$

*Wall temperature unless otherwise indicated.

TABLE 8

HARDNESS TRAVERSE OF BOILER WALL SECTION AFTER 2900 HOURS OF LOOP OPERATION

Average Wall Temperature 1128°F. Specimen Location TC11.

Depth from I.D. Surface Inches	Hardness Knoop	Hardness Rockwell Equivalent*	Remarks
0.003	230	96	Croloy
0.0055	255	97	Croloy
0.0085	255	97	Croloy
0.0115	220	95.5	Croloy
0.0145	230	96.5	Croloy
0.019	212	94	Croloy
0.022	222	96	Croloy
0.023	222	96	Croloy
0.0245	217	95	Croloy
0.0265	173	87	Large Grain
0.0285	335	R_C31	Croloy 9M/316 SS Interface
0.031	246	98	Type 316 SS
0.0345	253	98.5	Type 316 SS
0.0365	253	98.5	Type 316 SS
0.048	225	96	Type 316 SS
0.0565	246	98	Type 316 SS
0.070	249	98	Type 316 SS

*R_B unless otherwise noted.

TABLE 9

HARDNESS TRAVERSE OF SUPERHEATER WALL SECTION AFTER 2900 HOURS OF LOOP OPERATION

Average Immersion Thermocouple Temperature 1264°F. Specimen Location TC33

Depth from I.D. Surface Inches	Hardness Knoop	Hardness Rockwell Equivalent	Remarks
Edge	186	R_B90	Croloy
0.005	196	R_B92	Croloy
0.010	186	R_B90	Croloy
0.016	186	R_B90	Croloy
0.021	186	R_B90	Croloy
0.029	275	R_B100	Croloy 9M/316 SS Interface
0.035	294	R_C24	Type 316 SS
0.040	289	R_C22	Type 316 SS
0.045	286	R_C22	Type 316 SS
0.051	279	R_C20	Type 316 SS
0.056	286	R_C22	Type 316 SS
0.062	294	R_C24	Type 316 SS
0.067	279	R_C20	Type 316 SS
0.073	279	R_C20	Type 316 SS
0.078	279	R_C20	Type 316 SS
0.084	282	R_C21	Type 316 SS

TABLE 10

CARBON CONTENT OF LOOP WALL AT VARIOUS DISTANCES FROM I.D. SURFACE

Distance from I.D. Surface	Carbon - Percent		
	Boiler	Superheater	Condenser
1^{st} 0.005"	0.131	0.052	0.304, 0.428
2^{nd} 0.005"	0.140	0.024	0.121
3^{rd} 0.005"	0.131	0.033	0.133
4^{th} 0.005"	0.126	0.005	0.122
5^{th} 0.005"	0.110	0.037	0.125
6^{th} 0.005"	0.108	0.067	0.150
7^{th} 0.005"	0.100	0.123	-
8^{th} 0.005"	0.084	0.150	-
9^{th} 0.005"	0.080	0.132	-

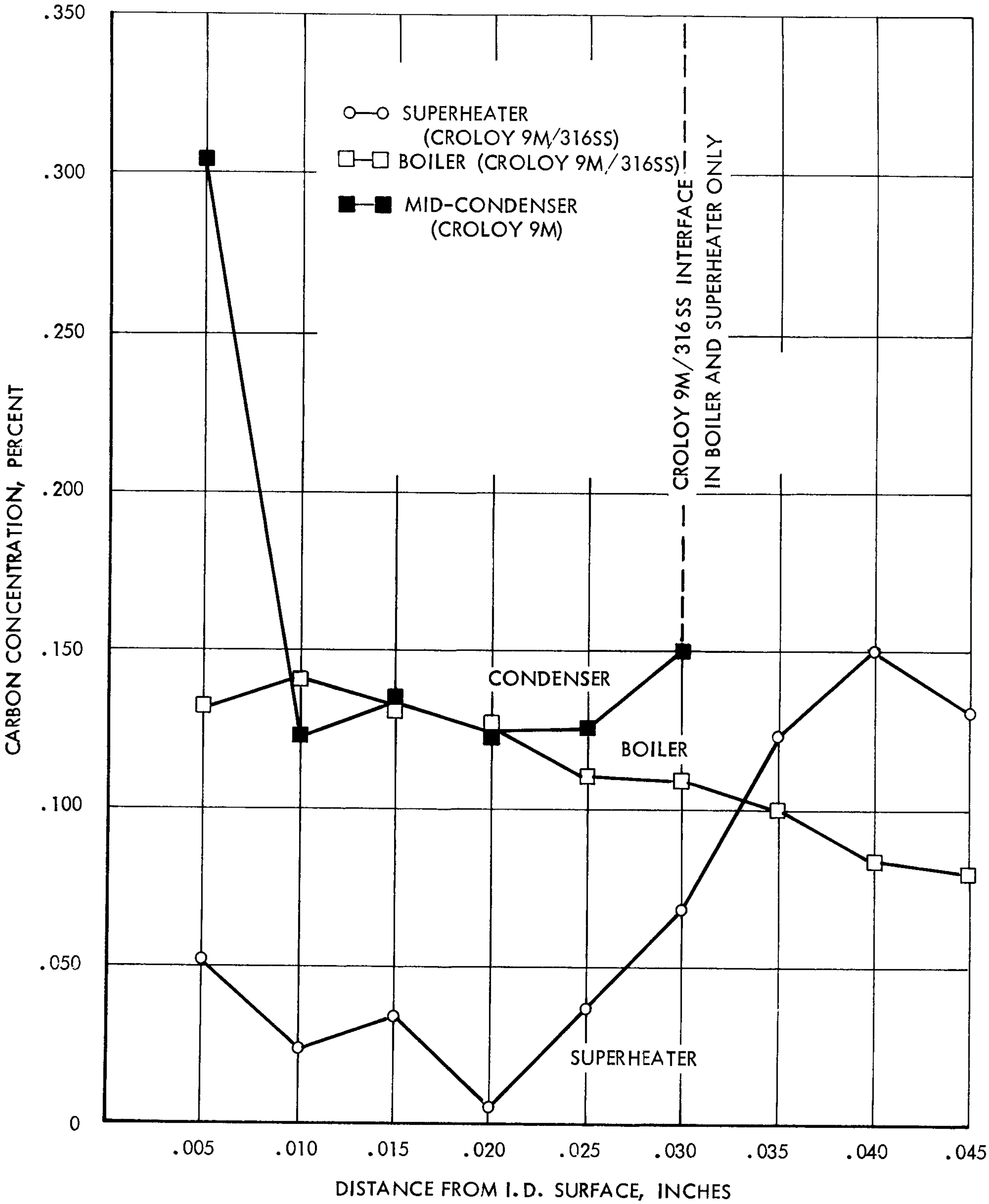

Figure 53. Carbon Content of Loop Wall at Various Distances from the I. D. Surface.

It is apparent from these carbon analyses that the Type 316 SS—Croloy 9M composite tubing is subject to metallurgical changes because of carbon diffusion at temperatures above 1200°F. Furthermore, it is indicated from these results that carbon is lost by the Croloy 9M by diffusion to the Type 316 SS cladding.

H. SUPERHEATER CREEP FAILURE

As stated in a previous section of this report, operation of the loop was suspended 30 hours before scheduled shutdown. The cause of this premature shutdown was the bulging of the superheater outlet tubing by creep resulting in breakage of the heater ribbon.

The microstructural change of the Croloy 9M in the superheater outlet section of this loop is clearly displayed in the photomicrograph of Figure 37. The normal grain structure of this ferritic steel has been replaced by an extremely low carbon, coarse grain ferrite. Apparently, long time elevated temperature exposure with intimate contact to Type 316 SS promoted the migration of carbon from the Croloy 9M to the austenitic stainless steel, accompanied by grain coarsening of the ferritic steel. The loss of carbon by the Croloy 9M did not change its original hardness (R_B85—96) significantly. The acquisition of carbon by the Type 316 SS cladding material was manifested by a profusion of carbides (Figure 37) and an appreciable change in hardness, R_B96 to R_C29. The electron beam microanalysis of this section of tubing revealed extensive molybdenum and chromium-rich precipitates in the Type 316 SS, which are probably carbides. The electron beam microanalysis also revealed a diffusion zone at the Type 316 SS—Croloy 9M interface of 1.7 to 2.2 mils in thickness (see Figures 54 and 55). This diffusion zone is also displayed in the photomicrograph of Figure 37. This extensive diffusion zone in the superheater outlet section (operated at 1405 ± 10°F) is compared with that observed in the boiler mid-section (operated at 1142°F and displayed in the photomicrograph of Figure 32). The diffusion zone in this lower-temperature region of the loop was found to be less than 0.3 mil in thickness, as revealed by the electron beam microanalysis (see Figures 56 and 57). The observed gross carbon diffusion and metallurgical structural changes of the Type 316 SS—Croloy 9M tubing in the 1405 ± 10°F temperature region is, of course, attributed to the excessive temperature.

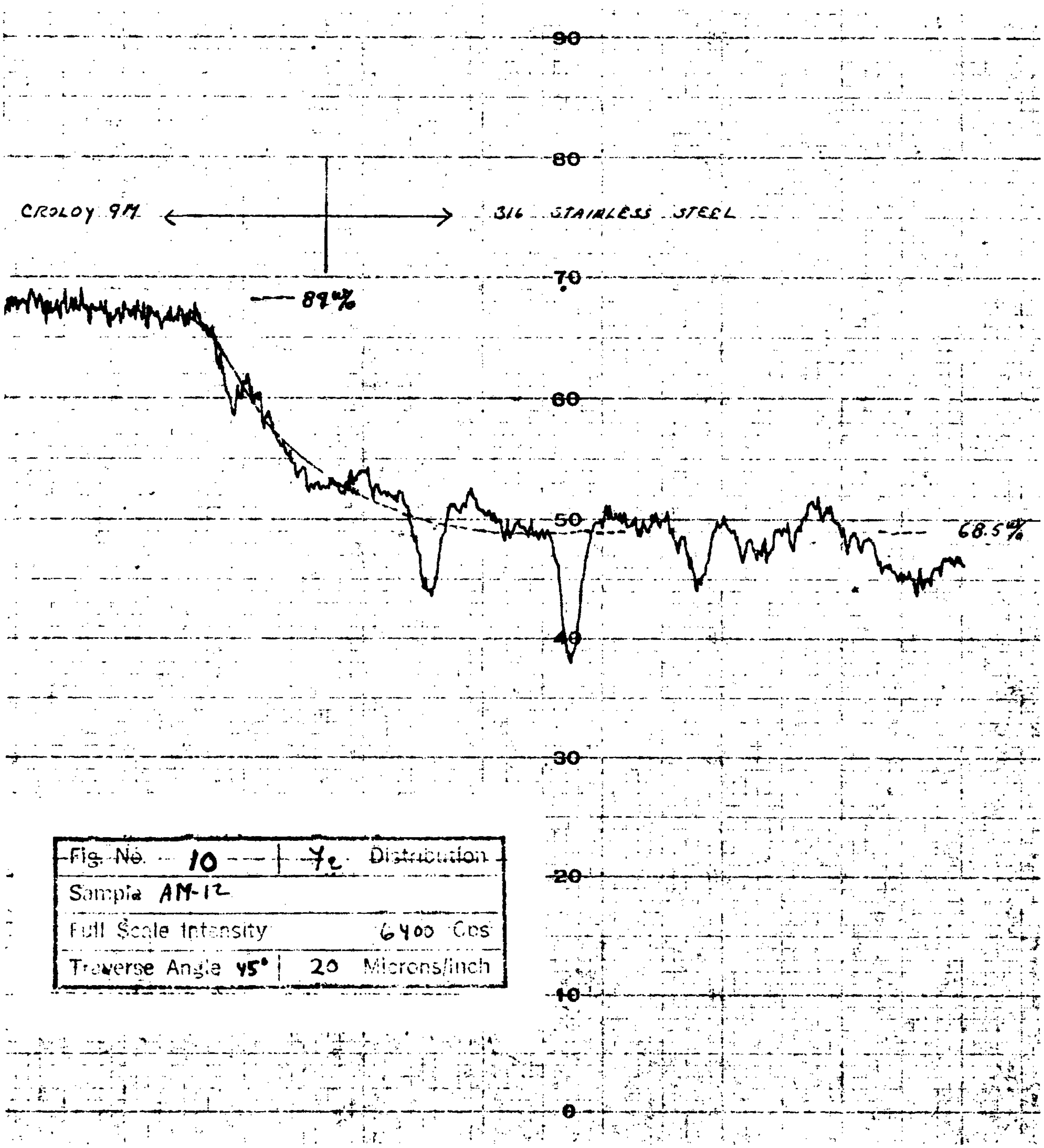

Figure 54. Distribution of Iron at the Type 316 SS-Croloy 9M Interface in the Superheater Outlet.

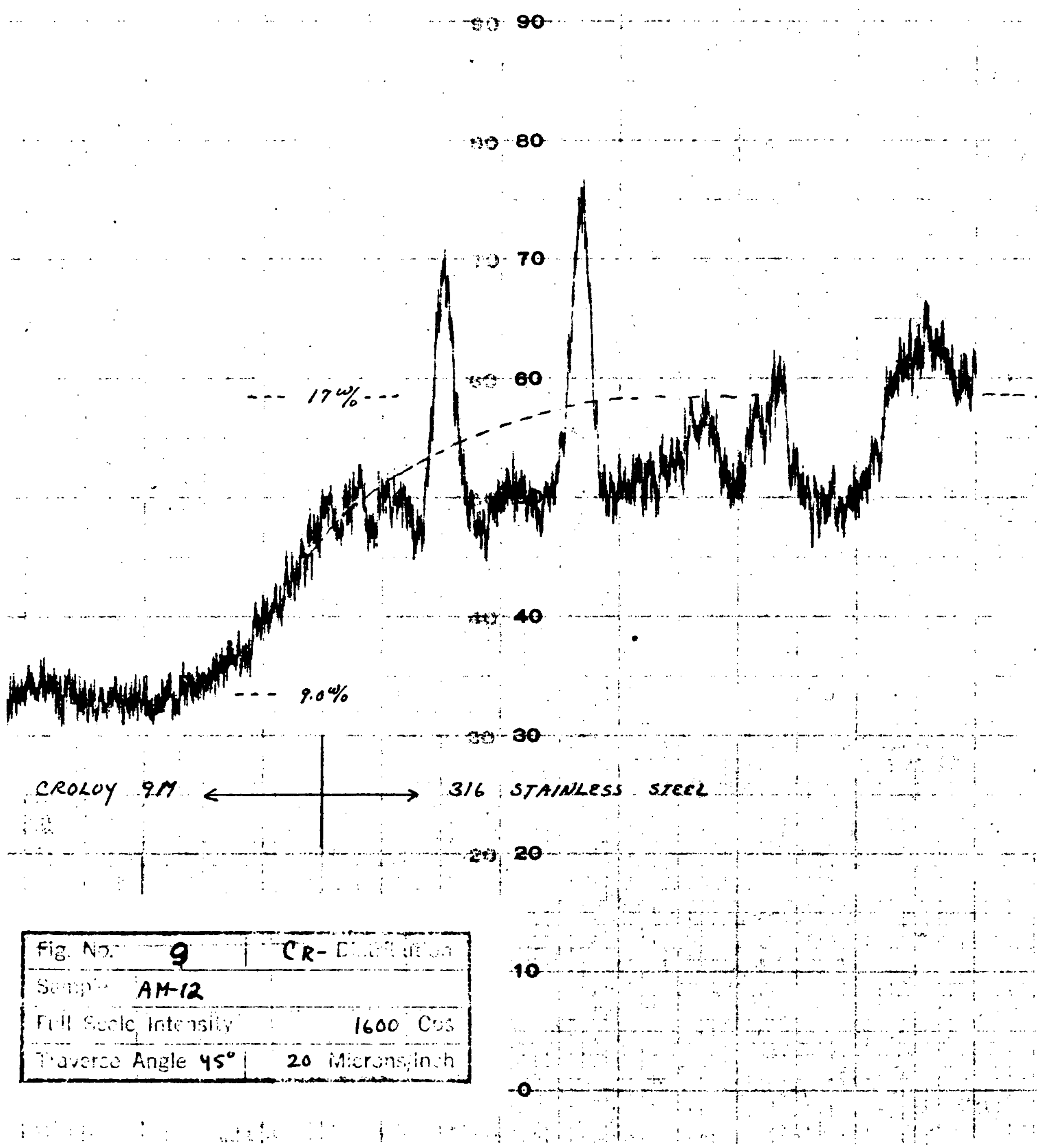

Figure 55. Distribution of Chromium at the Type 316 SS-Croloy 9M Interface in the Superheater Outlet.

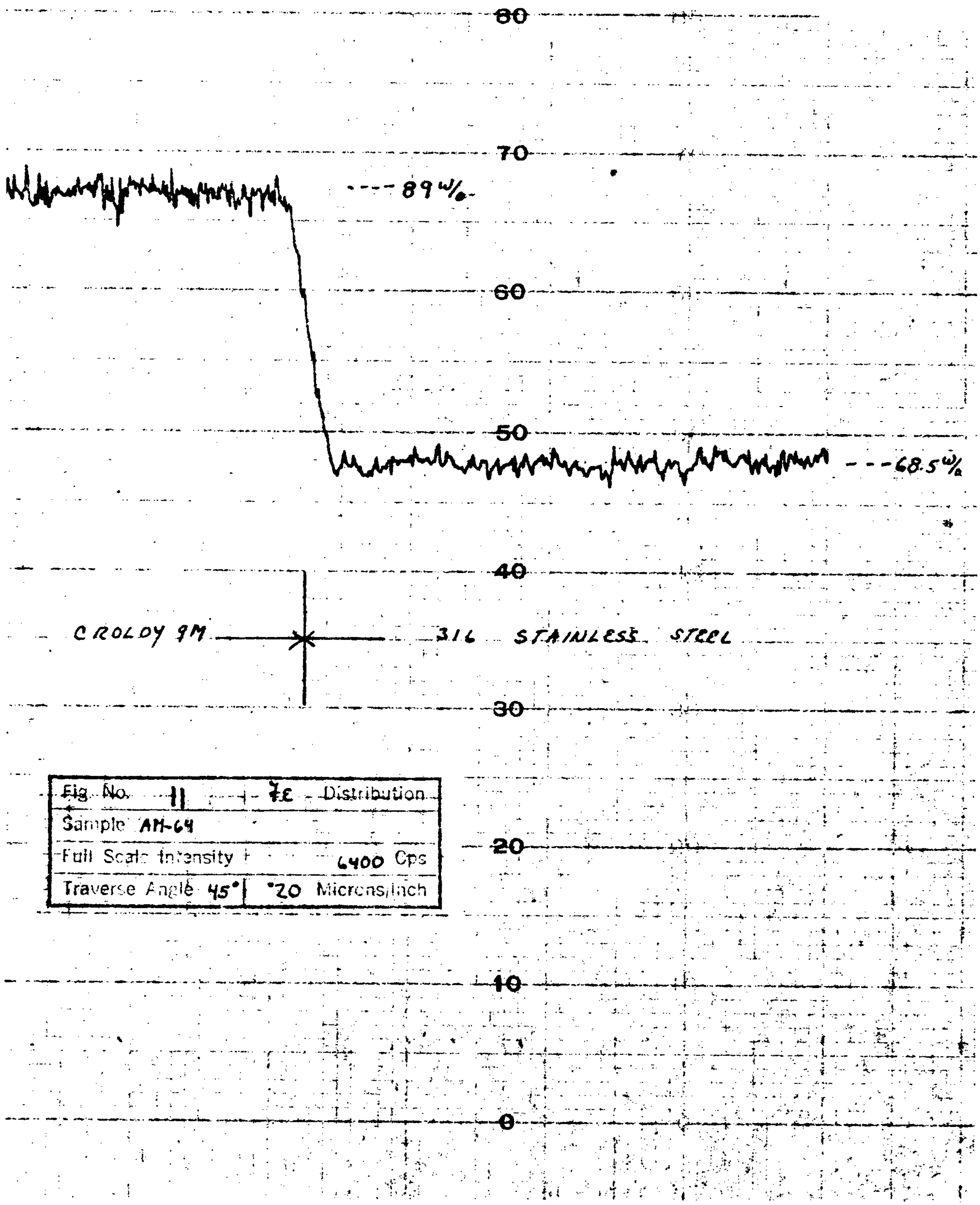

Figure 56. Distribution of Iron at the Type 316 SS-Croloy 9M Interface in the Boiler Mid-Section.

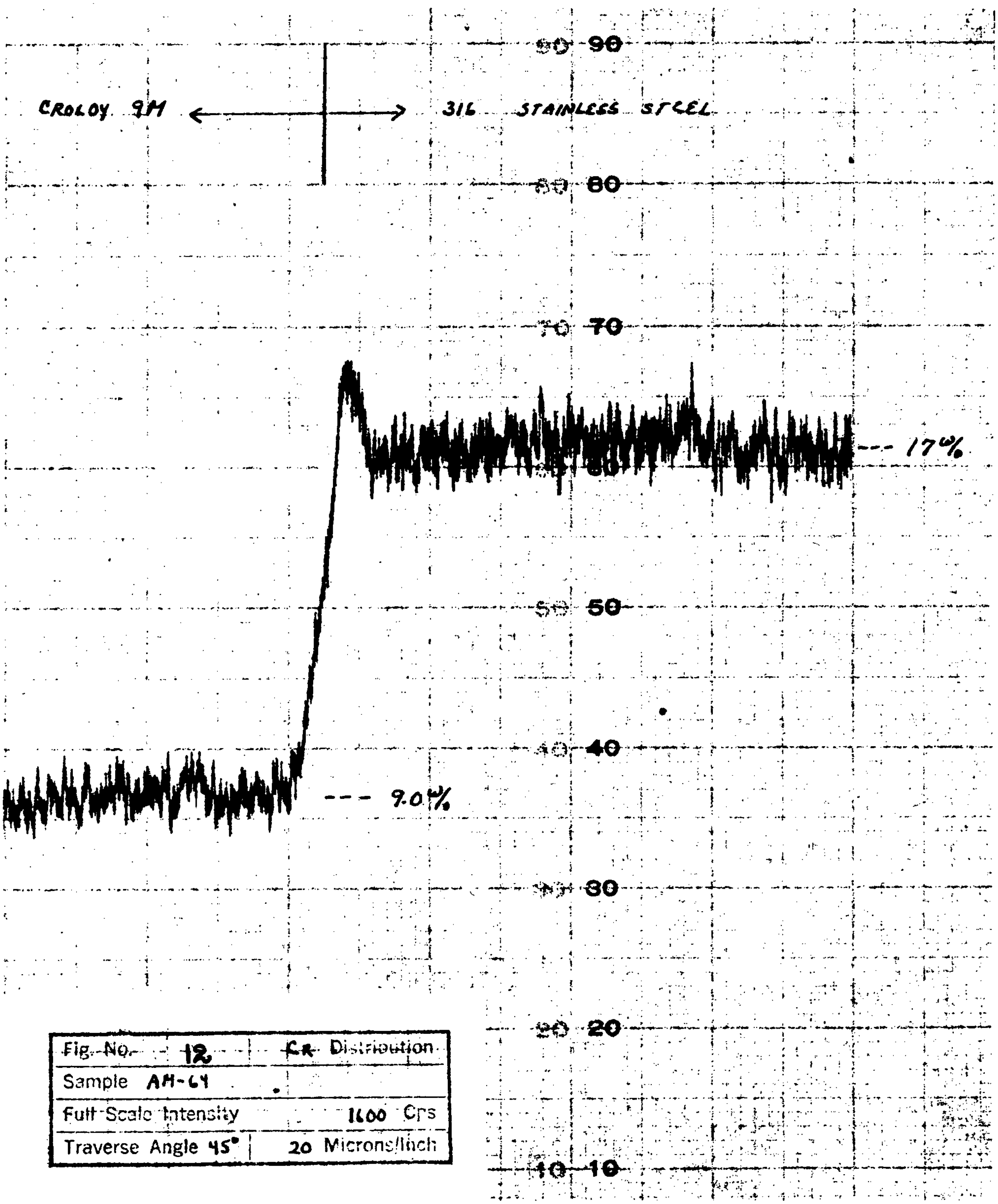

Figure 57. Distribution of Chromium at the Type 316 SS-Croloy 9M Interface in the Boiler Mid-Section.

The wall temperature of this failed portion of the superheater, by excessive creep, was 1405 ± 10°F as indicated and recorded by several thermocouples. At this elevated temperature, the creep strength of Croloy 9M is practically nil; stress to cause 0.001% per hour creep is less than 1500 psi. Since the available strength of the Croloy 9M was thus practically zero, the hoop stress for the superheater outlet section was calculated based only on the Type 316 SS clad portion of the tube and mercury vapor pressure of 269 psia. A value of 1965 psi hoop stress was obtained which is substantially lower than the reported 0.0001% per hour creep strength of 4000-5000 psi.[6, 7] Since the hoop stress was not excessive for this austenitic steel, the reported temperatures were reviewed for possible error. Since three thermocouples reported 1405 ± 10°F and since the Type 316 SS did not have oxide scale, there was no reason to doubt the reported temperature. However, the observed metallurgical changes in the Type 316 SS mentioned above may be the cause of the accelerated creep and thus could be the primary cause of failure of the tubing. Obviously, the cause of the creep failure of the duplex tubing in the superheater outlet section cannot be determined without further mechanical testing.

CONCLUSIONS

On the basis of the specific test conditions reported, the following conclusions are made:

1. Corrosion data for this system agree favorably with data of other investigators.

2. The greatest penetrations occurred in the low vapor quality regions of the boiling section of the loop and in the superheater outlet where condensation is believed to have occurred.

3. The dry portions of the superheater suffered negligible attack.

4. The degree of corrosion which existed in the condenser and subcooler could not be detected by metallography because of the extremely rough surface of the as-received material.

5. The vapor trap and vapor corrosion product separators removed 54.3 percent of the corroded metals found in the system, while the liquid corrosion product separator removed 24.7 percent of the corroded metals. The total efficiency of 79 percent agrees favorably with data from other systems employing corrosion product separators.

6. The feasibility of corrosion product separation in a forced circulation, two phase, mercury system has been demonstrated.

7. The observed attack in the loop appeared to be of the general dissolution type.

8. Type 316 SS-Croloy 9M duplex tubing exhibited carbon diffusion from the Croloy 9M to the Type 316 SS at temperatures above 1200°F.

9. The Type 316 SS-Croloy 9M duplex tubing exhibited excessive creep after 2918 hours at 1405 ± 10°F, apparently a result of deteriorative metallurgical changes in the Type 316 SS.

10. Deposition and corrosive and/or erosive attack were observed in the Croloy 9M throttling valve after 448 hours of loop operation.

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