GESP-550
NASA-CR-72814

N71-19497

SNAP-8 REFRACTORY BOILER DEVELOPMENT

TOPICAL REPORT III

EVALUATION OF SNAP-8, SN-1 BOILER

Prepared by

W. H. Hendrixson
R. W. Harrison

Approved by

E. E. Hoffman
J. Zmurk

November 3, 1970

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-10610
Edward R. Furman, Project Manager
Phillip L. Stone,
Materials Structures Division

NUCLEAR SYSTEMS PROGRAMS
SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

REPRODUCIBLE COPY

NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report should be referred to:

National Aeronautics and Space Administration
Scientific and Technical Information Division
Attention: USS-A
Washington, D.C. 20546

GESP-550
NASA-CR-72814

SNAP-8 REFRACTORY BOILER DEVELOPMENT

TOPICAL REPORT III

EVALUATION OF SNAP-8, SN-1 BOILER

prepared by
W. H. Hendrixson
R. W. Harrison

approved by
E. E. Hoffman
J. Zmurk

NUCLEAR SYSTEMS PROGRAMS
SPACE DIVISION
GENERAL ELECTRIC COMPANY
Cincinnati, Ohio 45215

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

November 3, 1970

CONTRACT NAS 3-10610

NASA Lewis Research Center
Cleveland, Ohio
Edward R. Furman, Project Manager
Phillip L. Stone
Materials Structures Division

TABLE OF CONTENTS

		PAGE NO.
	ABSTRACT	vii
I.	INTRODUCTION	1
II.	BOILER EVALUATION	3
	A. PROCEDURE	3
	B. SECTIONING OF SNAP-8 SN-1 BOILER	3
	C. EVALUATION OF STAINLESS STEEL PARTS	6
	1. Outer 316 Stainless Steel Shell	6
	2. Stainless Steel Piping Separating Flowing and Static NaK	8
	D. EVALUATION OF TANTALUM PARTS	11
	E. Ta/316 SS TRANSITION JOINTS	13
	1. Ultrasonic Inspection	16
	2. Metallographic Examination	16
III.	SUMMARY	19

LIST OF ILLUSTRATIONS

Figure No.		Page No.
1	Sectioning of the SNAP-8 Boiler for Evaluation - Removal of the Outer Stainless Steel Shell at the Boiler Inlet	21
2	Appearance of the SNAP-8 Boiler Inlet with Outer Stainless Steel Shell and Bellows Removed.	22
3	Appearance of the Zirconium Getter Bundle Removed From Around the Coextruded Joint at the Boiler Inlet - Chemical Analysis Indicated High Hydrogen Concentration.	23
4	Appearance of the Boiler Exit with the Outer Stainless Steel Shell Removed	24
5	Sectioned SNAP-8 Boiler Following Testing.	25
6	ID of 321 Stainless Steel Tube Exposed to Static NaK at Distances Marked from the Header at the Boiler Inlet.	26
7	ID of Tantalum Tube Exposed to Mercury at Distances Marked from the Header at the Boiler Inlet	27
8	Dark Gray Deposit on the ID Surface of a Tantalum Boiler Tube at the Exit of the Plug Section.	28
9	Tantalum Plug and Tube Section	29
10	Microstructure of the 316 Stainless Steel Outer Tube Shell at the NaK Inlet of SNAP-8 Boiler SN-1	30
11	Hardness Across the Wall of the Outer Stainless Steel Shell.	31
12	Bend Specimens from Outer Stainless Steel Shell at NaK Inlet - Specimens Bent with ID Surface in Tension and 1T Bend Radius	32
13	OD of Stainless Steel Tube Separating the Static and Flowing NaK Circuits in Plug Area 2' Downstream of the Header at the Boiler Inlet	33
14	321 Stainless Steel Tubing Separating the Static NaK and Flowing NaK Circuits	34
15	Hardness Across the 321 Stainless Steel Tubing Separating the Flowing and Static NaK Circuits 18 Feet From the Boiler Inlet Header	35

LIST OF ILLUSTRATIONS (Continued)

Figure No.		Page No.
16	Weld Shrinkage in a Butt Weld in the 321 Stainless Steel Tubing Separating the Static and Flowing NaK Circuits 18 Feet Downstream from the Boiler Inlet. .	36
17	Posttest Microstructures of the Tantalum at the Boiler Inlet	37
18	Tantalum Tube - 0.670" ID x 0.040" Wall Used in the Construction of the SNAP-8 SN-1 Boiler in the Pre-test Condition	38
19	Tantalum Tube - 0.670" ID x 0.040" Wall Used in the Construction of the SNAP-8 SN-1 Boiler in the Pre-test Condition	39
20	Tantalum Tube - Plug Assembly at Plug Exit	40
21	The Surface Coating on the Mercury Side of the Tantalum Tubing in the Vapor Region of the Boiler	41
22	The Surface Coating on the Mercury Side of the Tantalum Tubing in the Vapor Region of the Boiler	42
23	Tantalum Tube Separating Hg from Static NaK (A) 5' Downstream of Tantalum Header at Hg Inlet (B) 21' Downstream of Tantalum Header at Hg Inlet.	43
24	The Surface Coating on the Ta-10W Turbulator Wire. .	44
25	Separation of 316 Stainless Steel Feather-Edge From Tantalum on OD of Coextruded Joint From Inlet of SN-1 Boiler.	45
26	Tantalum - 316 Stainless Steel Interface of Co-extruded Joint From Inlet of SN-1 Boiler	46
27	Hardness Traverse Across Coextruded Ta/316 Joint . .	47
28	Microstructure of the Ta/316 SS Brazed Joint From Mercury Exit of SN-1 SNAP-8 Boiler at the Location with the Most Defects as Indicated by Ultrasonic Inspection	48
29	Hardness Traverse Across Ta/316 Brazed Joint	49

LIST OF TABLES

Table No.		Page No.
I	Chemical Analysis of Zirconium Getter Foil From The Static NaK System	5
II	Dimensional Measurements of Stainless Steel - Tantalum Tube Pairs.	7
III	Chemical Analysis of Stainless Steel Boiler Tubing. .	10
IV	Chemical Analysis of Tantalum Parts From the SNAP-8 SN-1 Boiler	14
V	Chemical Analysis of Tantalum Boiler Tubing	15

ABSTRACT

The SNAP-8, SN-1 boiler which was designed and fabricated by NASA was performance tested for 13,500 hours by GE-NSP. After testing the boiler was removed from the test facility and the boiler materials were evaluated. The evaluation indicated excellent compatibility of the boiler materials with liquid metals at high temperatures and all indications point to the design life capability of 40,000 hours.

I. INTRODUCTION

The SNAP-8, SN-1 Boiler was designed and fabricated by NASA at the Lewis Research Center, Cleveland, Ohio. The boiler was delivered to GE-NSP in December 1967 for performance testing which was initiated in February 1968. The boiler successfully completed 13,500 hours of testing with only three shutdowns for repairs.[1-3] At the completion of the test program, the boiler was removed from the test facility for posttest metallurgical evaluation, the results of which are described in this report.

---

(1) Edited by R. D. Brooks, "SNAP-8 Refractory Boiler Development, Summary Report of the February 17, 1968 Boiler Failure," NASA Contract NAS 3-10610, GESP-118, April 25, 1968.

(2) R. A. Fuller, W. F. Zimmerman, "Report of the May 31, 1968 SNAP-8, SN-1 Boiler Failure," GESP-230.

(3) R. A. Fuller, W. F. Zimmerman, "Report of the August 20, 1968 SNAP-8, SN-1 Boiler Failure, GESP-231.

II. BOILER EVALUATION

A. PROCEDURE

A complete nondestructive and destructive evaluation of the SN-1 boiler was conducted. The following items of the boiler were thoroughly examined: the inlet and outlet Ta/316 SS bimetallic joints, the inlet and outlet tantalum dished heads and header plates, two stainless steel-tantalum tube pairs, the tantalum plugs, the Ta-10W turbulator wire, and the zirconium getter foil. The techniques employed to evaluate these boiler materials included: visual, dimensional, and dye penetrant inspections; chemical analysis; metallographic examination microhardness measurements; X-ray diffraction; X-ray fluorescence; and microprobe analysis.

B. SECTIONING OF SNAP-8 SN-1 BOILER

Upon completing the endurance testing of the SNAP-8, SN-1 test boiler and securing the SNAP-8 test facility, the boiler was cut from the facility and removed for the destructive evaluation. Visual examination of the boiler revealed it to be in good condition, and sectioning of the boiler was initiated.

The initial cuts for sectioning of the boiler were made at the mercury inlet as shown in Figure 1. The first cut was made through the outer stainless steel shell just forward of the header on to which the bellows was welded. The snap ring was then removed from the bearing housing and the first section was slid off the end of the 1-inch-OD stainless steel tube exposing the bellows. The bellows appeared to be in excellent condition as can be seen in Figure 1. The second cut was made at the front end of the bellows while the third cut was made through the outer stainless steel shell just forward of the stainless steel header. This second section of the outer stainless steel shell with bellows attached was slid off the end of the inner tube exposing the coextruded joint and tantalum header. Some alkali metal oxide was present but it was easily removed by wiping with a damp cloth. This

section before and after cleaning is shown in Figure 2. The zirconium getter bundle which was wrapped around the coextruded joint and exposed to the static NaK is shown in Figure 3. The zirconium foil was very brittle and analysis, shown in Table I, indicated it to be very high in hydrogen (3800 to 4800 ppm). Although the source of this high hydrogen concentration is not clear, the contamination could have occurred as a result of moisture in the air reacting with residual NaK remaining on the foil after the boiler shutdown. A pickup of approximately 200 ppm oxygen was also noted.

At the exit end of the boiler, the stainless steel shell was removed exposing the tantalum header, dished head, and zirconium getter bundle around the brazed bimetallic joint. As shown in Figure 4, no NaK oxide was present, and the parts looked very clean. The zirconium getter bundle unlike the one at the inlet was ductile. Chemical analysis results shown in Table I indicated a pickup of 1000 to 3000 ppm oxygen but no pickup of hydrogen, such as that found in the getter bundle at the boiler inlet, was noted. The oxygen pickup is understandable since the getters purpose was to remove oxygen from the NaK and the reaction rate would be higher at the higher temperature at the boiler exit (NaK inlet).

After removing the zirconium getter bundles, the coextruded joint and dished head at the boiler inlet and the brazed joint and dished head at the exit were removed by cutting through the welds between the tantalum header and the dished head. Some mercury amalgam was observed on the face of the tantalum header and the ID of the tantalum dished head at the inlet but the ID surfaces exposed to mercury at the exit were very clean. By cutting through the stainless steel and tantalum tube pairs just following the stainless steel headers, both header plates at the inlet and exit were removed from the tube bundle. A layout of all the components of the sectioned boiler is shown in Figure 5.

The outer stainless steel shell was removed by making circumferential cuts every 4-5 feet and then sliding the sections off the tube bundles. In general, the tube bundles appeared very clean although some alkali metal oxide was noted at the NaK exit. This oxide film was easily removed by wiping with a damp cloth. After cleaning in this manner, the tubes appeared very bright and shiny except in the plug region where they appeared to be oxidized.

TABLE I

CHEMICAL ANALYSIS OF ZIRCONIUM GETTER FOIL FROM THE STATIC NaK SYSTEM

LOCATION	O	N	H	C
INLET				
OUTER LAYER	960	172	4875	104
MIDDLE LAYER	1061	208	3802	90
INNER LAYER	1060	167	3917	157, 176
EXIT				
OUTER LAYER	1729	214	13	101
INNER LAYER	4284	381	12	168
PRETEST	790	25	7	109

IS-17

Two stainless steel-tantalum tube pairs were removed from the boiler for evaluation. Viewing the tubes from the mercury inlet header, the tube located at 11 o'clock position (which will be designated as Tube A) and the tube located at the 5 o'clock position (which will be designated as Tube B) were selected. These tubes are from the hottest (Tube A) and coolest (Tube B) areas of the boiler as indicated during testing by thermocouples attached to the outer shell. The selected tubes were removed by cutting into sections measuring approximately 6 feet in length and sliding each section from the tube bundle. The inner tantalum tube was then removed from the outer stainless steel tube. Dimensional measurements along the length of the tube at approximately three-foot intervals were made. These measurements, shown in Table II, indicate no distortion of the tubes during test.

After removal from the boiler, the tubes were split longitudinally. Subsequent visual examination of the tubes indicated some discoloration or stain as shown in Figures 6 and 7 but no signs of degradation. A light gray deposit was observed on the ID of the tantalum tubes throughout the boiler in the vapor region. A darker gray deposit shown in Figure 8 was observed on the ID surface of the tantalum tube at the exit of the plug section.

The tantalum tubes from each plug tube assembly were slit longitudinally and removed from the plug. Discoloration or staining of the tube and plug were noted but no apparent degradation. The sectioned assembly is shown in Figure 9. Dye penetrant inspection of the boiler revealed no defective welds. Dimensional inspection of the various parts of the boiler indicated no distortion.

C. EVALUATION OF STAINLESS STEEL PARTS

1. Outer 316 Stainless Steel Shell

The visual appearance of the outer 316 stainless steel shell after removal from the test facility was good; however, it was observed that the shell appeared more brittle than normal when cutting it with a tubing cutter during the sectioning of the boiler. Although the action of the tubing cutter normally tends to work harden the material (especially thick-walled tubing) and causes a brittle fracture before cutting all the way through, it was noticed that after cutting about half way through

TABLE II

DIMENSIONAL MEASUREMENTS OF STAINLESS STEEL - TANTALUM TUBE PAIRS

Distance from Stainless Steel Header at Boiler Inlet, Feet	Diameter Stainless Steel Tube, Inches		Diameter Tantalum Tube, Inches	
	D_1	D_2, 90° to D_1	D_1	D_2, 90° to D_1
		TUBE PAIR A[a]		
0.5	-	0.875	0.739	0.739
1.5	-	0.890	0.738	0.739
3.0	-	0.889	0.738	0.739
6.0	1.046	0.920	0.726	0.764
9.0	1.011	0.982	0.720	0.772
12.0	1.007	0.993	0.738	0.763
15.0	1.006	1.000	0.739	0.758
18.0	1.005	1.004	0.728	0.766
21.0	1.006	1.010	0.731	0.766
24.0	1.006	1.010	0.734	0.762
27.0	1.007	1.010	0.736	0.760
30.0	1.010	1.011	0.737	0.761
33.0	1.011	1.010	0.734	0.764
36.0	1.011	1.002	0.734	0.760
		TUBE PAIR B[b]		
0.5	-	0.861	0.740	0.740
1.5	-	0.877	0.739	0.740
3.0	-	0.876	0.740	0.740
6.0	1.062	0.887	0.719	0.770
9.0	1.024	0.957	0.746	0.731
12.0	1.007	0.988	0.735	0.765
15.0	1.006	0.998	0.742	0.761
18.0	1.007	1.001	0.723	0.768
21.0	1.001	1.006	0.730	0.766
24.0	1.004	1.003	0.728	0.766
27.0	1.002	1.010	0.739	0.758
30.0	1.006	1.012	0.727	0.767
33.0	1.009	1.012	0.730	0.765
36.0	1.004	1.005	0.745	0.756

(a) Tube pair from hottest position in boiler.

(b) Tube pair from coolest position in boiler.

the wall the pressure from the tubing cutter would cause the remaining portion of the wall to crack.

Metallographic examination of a specimen taken from the outer shell near the NaK inlet showed an almost continuous grain boundary precipitate, as shown in Figure 10, which could attribute to the brittle behavior. This precipitate is most likely $M_{23}C_6$ type of carbide phase. Metallographic examination of the OD and ID surfaces of the shell revealed nothing unusual as far as corrosion or oxidation were concerned. Hardness traverse across the wall showed no significant hardness gradient as shown in Figure 11.

A bend specimen cut from this same area of the boiler was bent with the ID in tension over a 1t radius and cracked after 100° bend. A specimen* from the outer shell of the BRDC-2 boiler (tested at Aerojet General) was also tested. This specimen bent under the same conditions as the previous specimen from SN-1 boiler shell, bent a full 105° without cracking. The two specimens are shown in Figure 12. The BRDC-2 boiler was operated for a total of 8700 hours while the SN-1 boiler was operated for a total of 15,000 hours. There apparently was some loss of ductility of the SN-1 316 stainless steel outer shell material, but it does not appear to be detrimental to boiler operation. However, as test time is accumulated the ductility of a given boiler shell may continue to decrease.

2. Stainless Steel Piping Separating Flowing and Static NaK

One-inch-OD x 0.035-inch-wall 321 stainless steel tubing was used to contain the static NaK around the tantalum tubes and separate it from the flowing NaK system. These tubes were welded at both ends to stainless steel header plates which in turn were welded to the outer shell. The appearance of the tubing, after removal of the outer shell, was bright and clean except in the plug region which was coated with NaK oxide. This oxide was easily removed with a damp cloth but the surface beneath was dark gray in color and rougher in comparison to the tubing in the rest of the boiler.

---

* Supplied by NASA.

An earlier report of the February 17, 1968, boiler failure,[1] described a heavy deposit of NaK oxide on the OD surface of the stainless steel tubes in the plug region. At that time the NaK oxide was mechanically removed. The roughened surface was probably the result of the mechanical cleaning and subsequent oxidation or corrosion of the stainless as a result.

Metallographic examination of this area revealed some attack on the OD of the 321 stainless steel tubing to a depth of about 0.0005 inch, as shown in Figure 13. A gradient analysis for interstial elements was obtained from the inner 1/3, center 1/3, and outer 1/3 of the tubing wall. These analyses are shown in Table III along with a gradient analysis of another section of tubing further downstream. The analysis of the tubing from the plug section showed the OD of the tube to be very high in oxygen (2017 ppm) and somewhat higher in carbon (779 ppm). The high oxygen concentration at the OD is believed to be associated with the observed attack. Sections of this tubing were noted to turn white after long-term exposure to air. The white powdery coating results from oxidation of the NaK oozing from the grain boundary attack area at the surface of the tubing.

Photomicrographs shown in Figure 14 are typical of all other areas of the 321 stainless steel tubing separating the static and flowing NaK circuits. No corrosion was observed, but the areas adjacent to the surface were depleted in sigma with the effect more prevalent on the flowing NaK side. A hardness traverse across the tube wall indicated no hardness gradient as can be seen in Figure 15. Microprobe scans across the sigma depleted zone detected no change in composition. Interstitial analysis of the outer 1/3, center 1/3, and inner 1/3 of the tube wall shown in Table III, showed the oxygen and nitrogen concentrations to be somewhat higher at the surface. Apparently there has been a subtle change in composition at the tube surface which has prevented the formation of the sigma phase. The stainless steel header plates showed no sign of attack or degradation from either the static or flowing NaK sides. During the metallographic examination considerable weld shrinkage was noted in one of the center tube welds 18 feet downstream of the inlet header as shown in Figure 16. No shrinkage was noted in any of the other five welds examined.

TABLE III

CHEMICAL ANALYSIS OF STAINLESS STEEL BOILER TUBING

Sample Location	Element	Tube from Plug Area Concentration, ppm (Average)	Tube 13' from Inlet Header Concentration, ppm (Average)
Outer third of Tube Wall	O	1649, 2386 (2017)	86, 146 (116)
	N	153, 183 (168)	261, 275 (268)
	H	81, 87 (84)	16, 5 (11)
	C	762, 796 (779)	651, 677 (664)
Middle third of Tube Wall	O	19, 24 (22)	29, 39 (34)
	N	82, 80 (81)	94, 95 (95)
	H	1, 2 (2)	2, 3 (3)
	C	402, 398 (400)	640, 689 (665)
Inner third of Tube Wall	O	175, 149 (162)	164, 82 (123)
	N	105, 117 (111)	117, 128 (123)
	H	7, 7 (7)	7, 10 (9)
	C	442, 430 (436)	484, 507 (495)

D. EVALUATION OF TANTALUM PARTS

The tantalum section of the boiler separates the mercury and static NaK systems. The mercury enters the boiler through a 2-inch-OD 316 stainless steel tube. The transition from 316 stainless steel to tantalum is made at this point by means of a coextruded joint. The tantalum dished head connects the coextruded joint to the tantalum header. The seven tantalum boiler tubes are welded into the header plate along with the orifices, followed by the plugs and then the turbulator wire which extends the remaining length of the boiler tubes. As noted earlier, visual examination of the tantalum parts revealed some discoloration but no signs of degradation.

Metallographic examination of the tantalum dished head at the boiler inlet showed a minor amount of corrosion on the static NaK side. The attack shown in Figure 17 extends to a depth of 2 mils and is typical of oxygen associated alkali metal corrosion along crystallographic planes. The morphology of the corrosion suggests the possible cause as being slight oxygen contamination of the tantalum surface. The tantalum dished head at the boiler outlet showed no sign of attack although it operated at a much higher temperature (approximately 1300°F as compared to 900°F at the boiler inlet) since the tantalum dished head at the inlet was replaced during a field weld repair,[1] some contamination could have occurred at that time. Chemical analysis indicates the oxygen concentration of the tantalum at the boiler exit to be less than 10 ppm as compared to 40 ppm at the boiler inlet. The difference in oxygen concentration could also be explained by differences in the kinetics of oxygen dissolution in the alkali metal as influenced by the temperature difference.

Surface cracks were observed on the ID of the tantalum orifice, shown in Figure 17. They do not appear to be corrosion but rather a result of machining. The orifices of this boiler were drilled and not polished by honing, which is now being employed in boiler manufacture.

Metallographic examination of the pretest tantalum tubing used in the SN-1 boiler revealed severe grain boundary attack on both the OD and ID as shown in Figures 18 and 19. It is believed the defects could have resulted from surface contamination during processing and

subsequent acid pickling. Similar grain boundary voids have been observed in Cb-1Zr tubing processed similarly; however, this effect is not typical of standard quality refractory metal tubing.

The photomicrograph in Figure 20 of a representative area of the tantalum tube-plug assembly shows the grain boundary voids in the tube while the plug shows no sign of attack. Although the liquid metal exposure would tend to accentuate these grain boundary voids no corrosion was observed.

At the exit of the plug, dark gray deposits were noted on the ID of the tantalum tube as shown previously in Figure 8, and extended over a length of approximately nine inches. A section of the tube was mounted for metallographic examination, but the deposit was dissolved during the polishing process. It was discovered at this time that this deposit was water soluble. A semiquantitative spectrographic analysis of the deposit indicated the major element to be chromium (> 10%) with minor amounts (< 10%) of cobalt, iron, and nickel. All of these elements with the exception of the cobalt are major constituents of stainless steel, and cobalt is a major constituent of L605. Stainless steel and L605 are both employed in the facility piping.

Following the dark gray deposits at the exit of the plug section and for the remaining length of the boiler, a light gray film or coating was observed on the ID surface (mercury side) of the tantalum tubing. This film, shown in Figure 21, was approximately 0.0005 inch thick and considerably harder than the base metal. Microhardness measurements show the film to have a Knoop hardness of 361 as compared to an average hardness of 100 in the tantalum. It is interesting to note that the film extends down into a lap defect in the tantalum tubing shown in Figure 22 to a depth of almost 4 mils. Photomicrographs of the tantalum tubing from other representative areas of the boiler are shown in Figure 23. X-ray diffraction analysis of the film indicated it to be predominantly isomorphous with tetragonal Ni_3Ta; however, X-ray fluorescence analysis indicated the presence of iron, chromium, titanium, and some mercury in the coating as well as nickel.

Microprobe analysis showed the presence of nickel and iron in this coating with essentially no diffusion of these elements into the tantalum

base metal. The mass transfer of stainless steel constituents occurs by dissolution in the mercury from the stainless steel facility piping and concentration in the boiler vapor region by distillation. The coating was also found on the tantalum turbulator wire which extended the full length of the boiler, the tantalum header, the dished head, and the Ta/316 SS brazed transition joint at the boiler exit. Photomicrographs of the turbulator wire are shown in Figure 24.

Chemical analysis of the various tantalum parts of boiler are presented in Table IV. From these data it can be seen that the concentration of nitrogen increases from the inlet to the outlet of the boiler. Since the boiler is a counterflow boiler, the mercury exit is hotter than the mercury inlet. If the nitrogen pickup is diffusion limited then one would expect a higher concentration at the mercury outlet. It is assumed the nitrogen comes from the stainless steel and exists in the boiler as nitrogen since mercury, sodium, and potassium do not form stable nitrides.

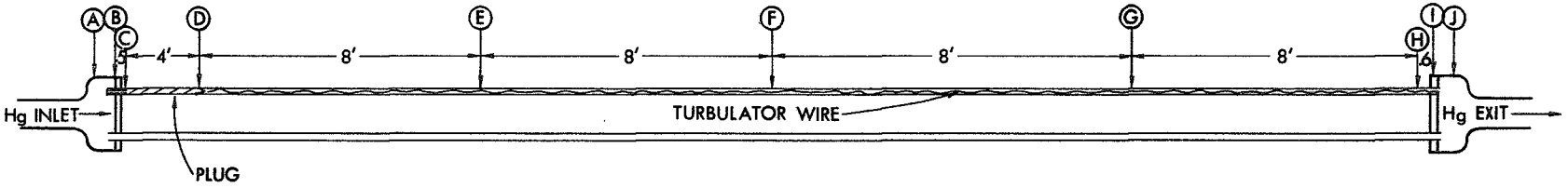
On the other hand the oxygen is highest at the boiler inlet. Since the oxygen concentration falls off very rapidly and remains fairly constant throughout the rest of the boiler, it may be that the tubing in this area was oxygen contaminated at the time of the field weld repairs. A gradient analysis of the tube from the plug inlet and from the boiler exit is shown in Table V. In both cases, analysis indicated the surface concentration of all the elements was higher than at the center of the tube, indicating surface contamination of both sides.

E. Ta/316 SS TRANSITION JOINTS

Two Ta/316 stainless steel transition joints were evaluated; the coextruded joint from the boiler inlet and the brazed joint from the boiler exit. Both joints were leak checked after removal from the boiler and found to be leak tight. After leak checking, the joints were Zyglo inspected. The brazed joint showed no indications but the coextruded joint revealed one indication, approximately 1/4-inch wide on the OD of the joint at the stainless steel featheredge. The ultrasonic inspection and metallographic examination of the joints are discussed in the following sections.

TABLE IV

CHEMICAL ANALYSIS OF TANTALUM PARTS FROM THE SNAP-8 SN-1 BOILER



	A	B	C	D	E	F	G	H	I	J		PRETEST	
	DISHED HEAD	HEADER	TUBE A						HEADER	DISHED HEAD	TUBE	HEADER	DISHED HEAD
O	40	23	110	26	24	14	20	17	9	4	20	90	72
N	9	11	9	6	10	9	32	67	17	9	21	5	16
C	25	28	32	37	33	28	24	22	35	29	12	20	28
			PLUG FROM TUBE A		TURBULATOR WIRE						PLUG	TURBULATOR WIRE	
O			184	72	146			146			100	48	
N			16	10	7			211			22	10	
C			91	26	42			71			40	20	
			TUBE B								TUBE		
O			69	32	33	15	22	28			20		
N			4	5	13	4	15	110			21		
C			60	65	55	38	43	43			12		
			PLUG FROM TUBE B		TURBULATOR WIRE						PLUG	TURBULATOR WIRE	
O			66	51	73		72				100	48	
N			10	10	27		32				22	10	
C			36	36	70		70				40	20	

TABLE V

CHEMICAL ANALYSIS OF TANTALUM BOILER TUBING

Sample Location	Element	Tube at Plug Inlet Concentration, ppm (Average)		Tube at Boiler Exit Concentration, ppm (Average)	
Outer third of Tube Wall	O	30, 50	(40)	19, 54	(36)
	N	7, 9	(8)	75, 112	(93)
	H	3, 3	(3)	3, 2	(3)
	C	78, 100	(89)	81	(81)
Middle third of Tube Wall	O	10, 10	(10)	12, 9	(11)
	N	5, 5	(5)	19, 14	(17)
	H	1, 1	(1)	< 1	(< 1)
	C	24, 30	(27)	18	(18)
Inner third of Tube Wall	O	42, 52	(47)	15, 32	(24)
	N	13, 15	(14)	81, 92	(87)
	H	2, 2	(2)	1, < 1	(1)
	C	71, 81	(76)	58	(58)

1. Ultrasonic Inspection

Modified A-scan recordings were obtained from the inspection of the Ta/316 stainless steel transition joints. This ultrasonic inspection method was developed under contract NAS 3-11846[4] for measuring the quality of tongue-in-groove braze joints.

The tongue-and-grooved standard contained reference defects of 0.005 inch, 0.010 inch, 0.020 inch, and 0.050 inch at both the ID braze interface and the OD braze interface. These defects were used to calibrate the Sperry No. 721, Ultrasonic Flaw Detector.

The two joints were ultrasonically scanned using the water immersion technique while being rotated in a small lathe.

The evaluation was performed using a No. 721 Flaw Detector, 50 W Broadband Pulser/Receiver and a 25 and 15 MH2 (megacycle) search units. The search units were "BB" styles, having 3/8-inch-diameter crystals and 1.35-inch and 2.17-inch water paths and 0.012-inch and 0.020-inch beam diameters, respectively. Recording of indications was accomplished using an Electronic Associates, flat bed, x-y recorder.

The "Modified A-Scan" recordings obtained from the inspection of the brazed joint produced several indications greater than that obtained from a 0.010-inch-diameter calibrated hole. These indications were exclusively from the inner annulus or the wide braze section of the joint.

The ultrasonic inspection of the coextruded joint verified the Zyglo indication and showed the defect to be approximately 1/4 inch wide and 100 mils long. One small internal defect was also indicated by ultrasonic inspection approximately 1/4 inch back from the 316 stainless steel featheredge.

---

[4] S. R. Thompson, J. D. Marble and R. A. Ekvall, "Development of Optimum Fabrication Techniques for Brazed Ta/Type 316 SS Tubular Transition Joints," NASA Contract, NAS 3-11846, GESP-521.

2. Metallographic Examination

Preparation of the brazed joint for the microstructural examination of longitudinal planes required special preparation because of residual stresses present in the tongue-and-groove area which can produce catastrophic failure of the braze alloy during longitudinal sectioning. To overcome this difficulty, the entire assembly was encased in thermal setting plastic before sectioning. The mounting material thus provided the required support in the braze area and prevented failure during sectioning. The same procedure was used on the coextruded joints.

When sectioning the joints, longitudinal cuts were made 1/8 inch away from the plane to be viewed. The specimens were then ground back to it before final polishing. Three longitudinal sections from each joint were selected for metallographic examination. From the brazed joint one specimen was selected from the worst area, one from the best area, and one from an intermediate area as determined from the ultrasonic inspection. From the coextruded joint, one specimen was selected from the area of the defect on the OD of the joint at the 316 stainless steel featheredge, one was selected from the area of the internal defect, and one was selected from a sound area.

The photomicrographs in Figure 25 show the separation at the featheredge of the 316 stainless steel from the tantalum on the OD of the coextruded joint. The photomicrograph shows the separation to be 0.016 inch long. Although the specimen mounted to view the internal defect indicated by the ultrasonic inspection was ground back several times in an attempt to locate the defect. It was not found. The only defect observed in the three specimens was the one on the OD of the joint at the 316 stainless steel featheredge. The photomicrograph shown in Figure 26 is typical of the Ta/316 stainless steel interface. As can be seen, there is a discontinuous phase present at the interface, but it is less than 0.0001 inch thick and apparently had no detrimental effect on the joint. The hardness traverse across the coextruded joint shown in Figure 27 indicates a favorable hardness profile.

Metallographic examination of the three specimens from the brazed joint showed the joint to be completely filled with braze alloy and

to have very little microshrinkage in it. As can be seen from the photomicrograph in Figure 28 from the worst area of the joint, one void approximately 0.003 inch in diameter is shown. Several other considerably smaller voids are present but nothing close to the 0.010-inch-diameter voids indicated by the ultrasonic inspection. The other two metallographic specimens also showed some microshrinkage voids (less < 0.002 inch) but to a lesser degree.

A microhardness traverse across the brazed joint is shown in Figure 29. It appears from this survey that some diffusion of the braze alloy into the 316 SS from the wide brazed side has occurred. Diffusion from the narrow braze side was apparently limited by the amount of braze alloy present. The hardness of the wide braze is somewhat higher than the hardness recorded for as-brazed specimens.[4] This increase of the braze hardness and increased hardness of the 316 stainless steel tongue does not appear to be detrimental to the joint.

III. SUMMARY

Posttest evaluation of the SN-1 boiler materials following testing has indicated the excellent compatibility of the selected materials with liquid metals at high temperatures. No major corrosion or degradation was observed in any of the boiler components, and all indications point to design life capability (40,000 hours). In summary, the following observations were made in regard to the various components of the boiler.

Some embrittlement of the outer stainless steel shell did occur but was not detrimental to the boiler operation. The 321 stainless steel tube, separating the static and flowing NaK systems, was in excellent condition except in the plug section which showed some surface attack (0.0005-inch) on the OD.

The tantalum dished head at the boiler inlet showed a minor amount of alkali metal corrosion on the static NaK side, which is believed to have been caused by slight oxygen contamination of the tantalum surface during the replacement of the dished head during a field weld repair.

A phase identified as predominantly isomorphous with tegragonal Ni_3Ta but also containing iron, chromium, titanium, nickel, and mercury was found on the turbulator wire and on the ID surface (mercury side) of the tantalum tubing. The phase was approximately 1/2 mil thick and a result of mass transfer of the stainless steel constituents.

Both the coextruded joint at the boiler inlet and the brazed joint at the boiler exit were in excellent condition. No deleterious effects were observed due to the exposure to mercury or NaK.

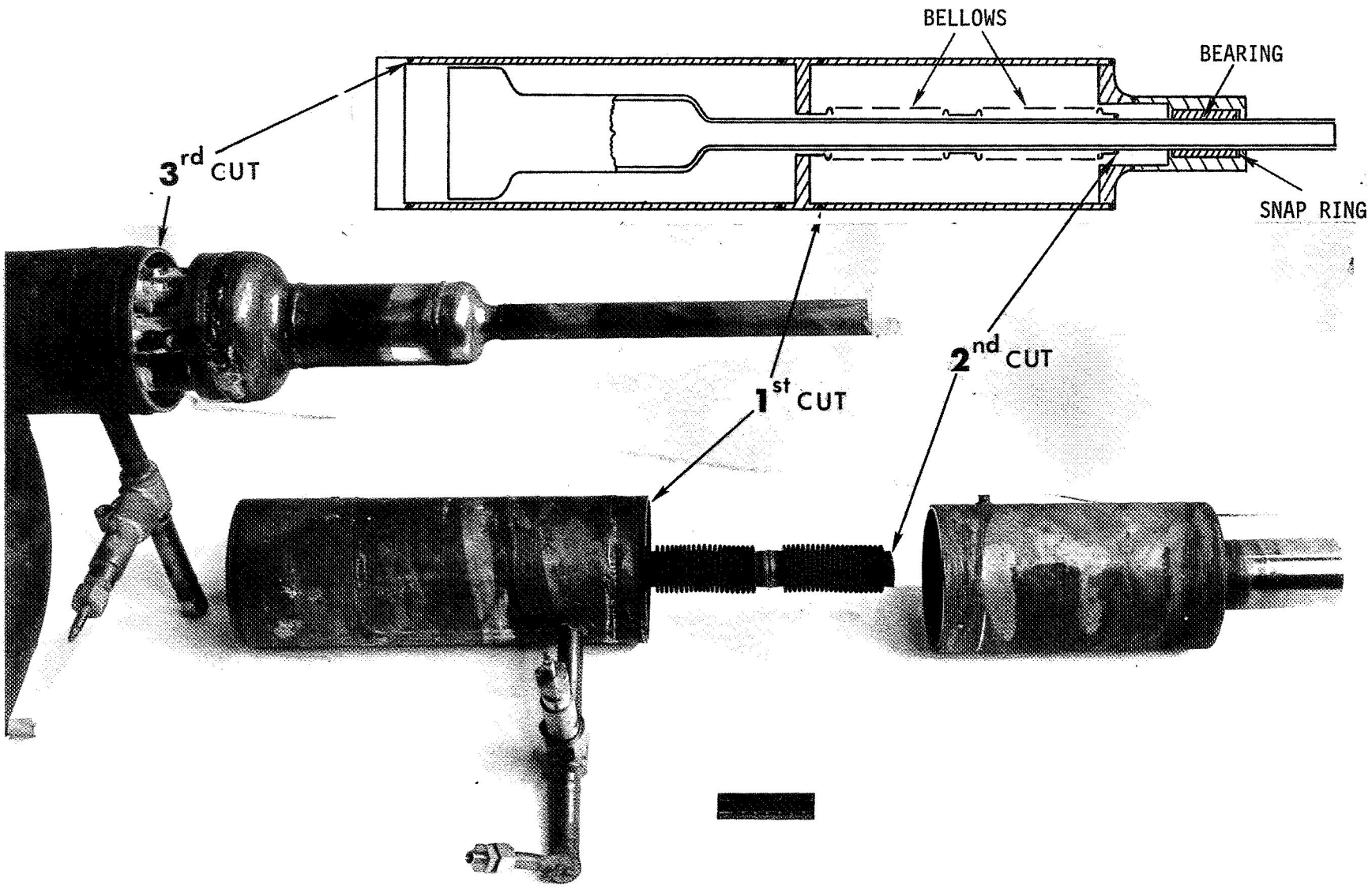
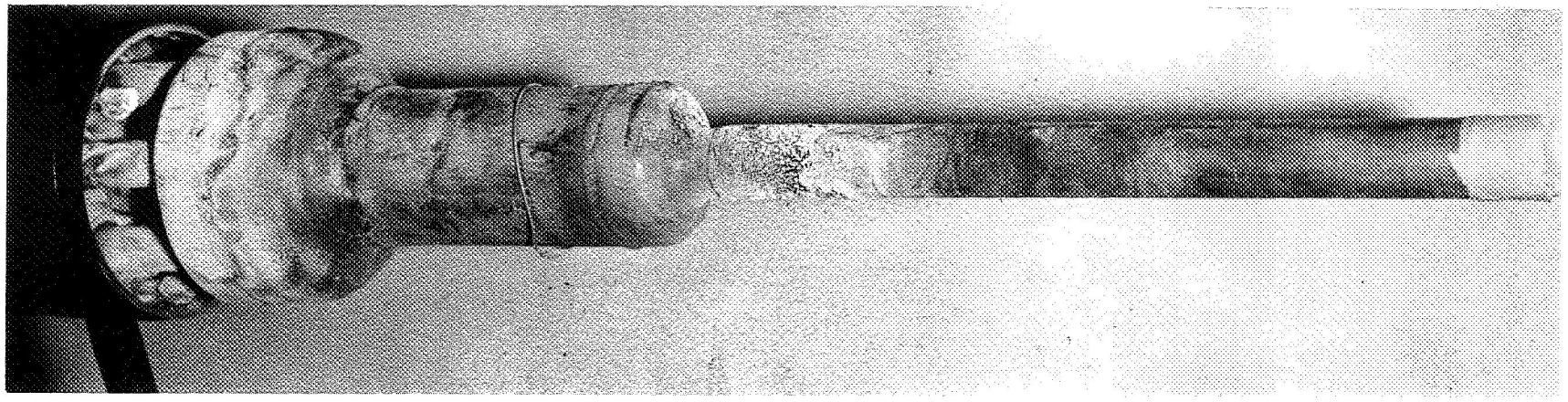
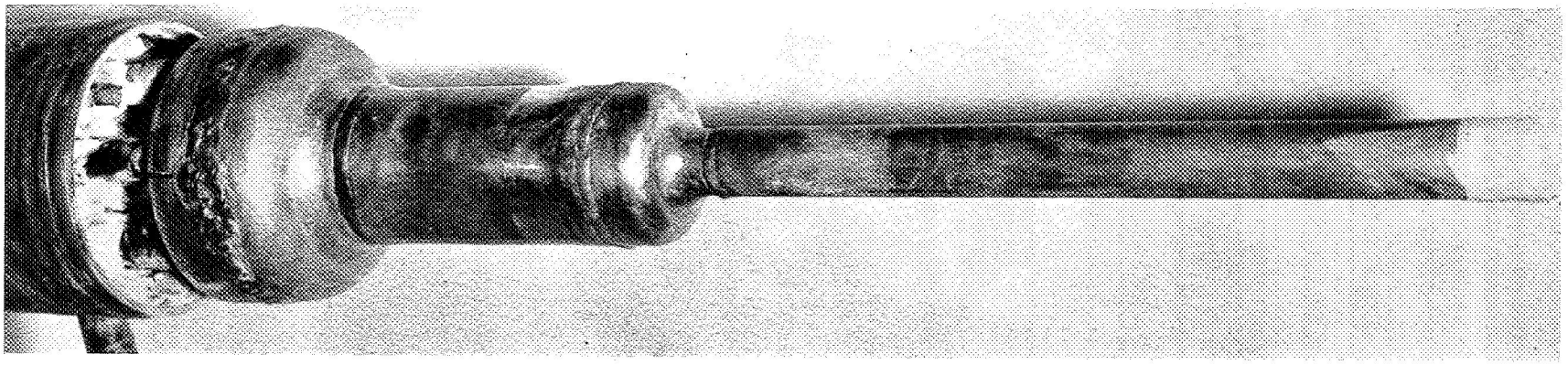


Figure 1. Sectioning of the SNAP-8 Boiler for Evaluation - Removal of the Outer Stainless Steel Sheel at the Boiler Inlet. (P70-1-12C)

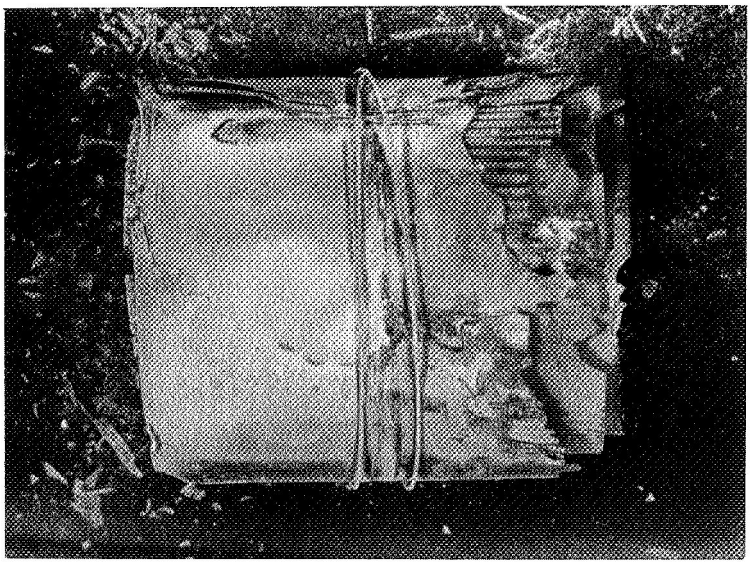


THE ASSEMBLY AS IT APPEARED WHEN OUTER SHELL WAS REMOVED



AFTER ASSEMBLY WAS CLEANED WITH DAMP CLOTH

Figure 2. Appearance of the SNAP-8 Boiler Inlet with Outer Stainless Steel Shell and Bellows Removed. (P70-1-12A & B)



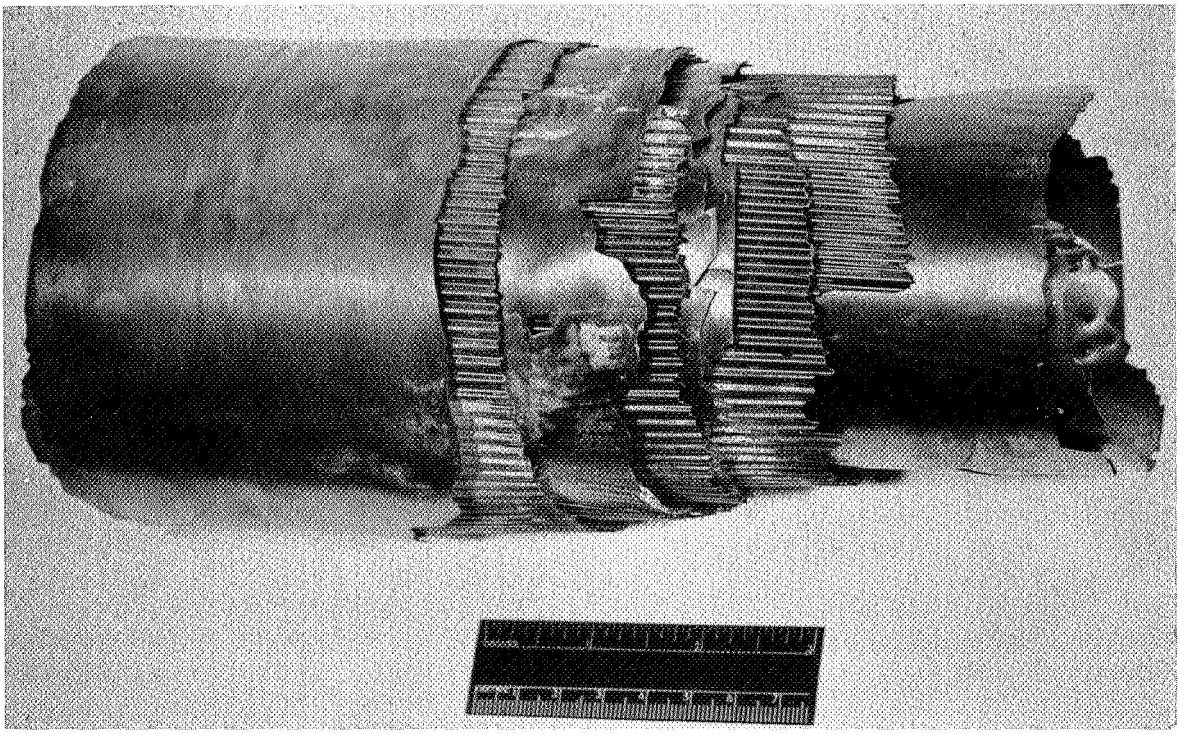


Figure 3. Appearance of the Zirconium Getter Bundle Removed From Around the Coextruded Joint at the Boiler Inlet - Chemical Analysis Indicated High Hydrogen Concentration. (P70-1-12F & H)

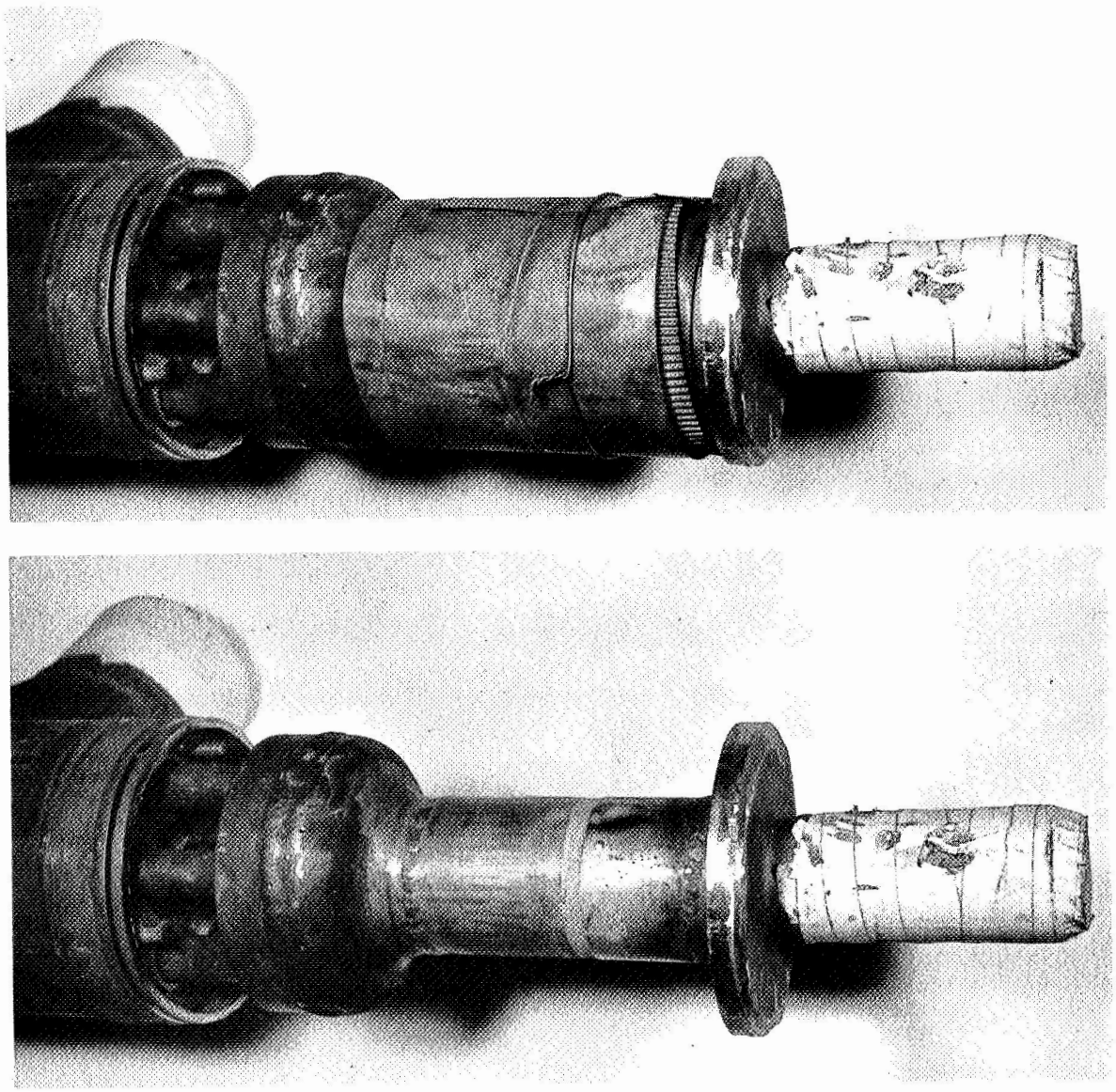


Figure 4. Appearance of the Boiler Exit with the Outer Stainless Steel Shell Removed. (Upper P70-1-12D, Lower P70-1-12E)

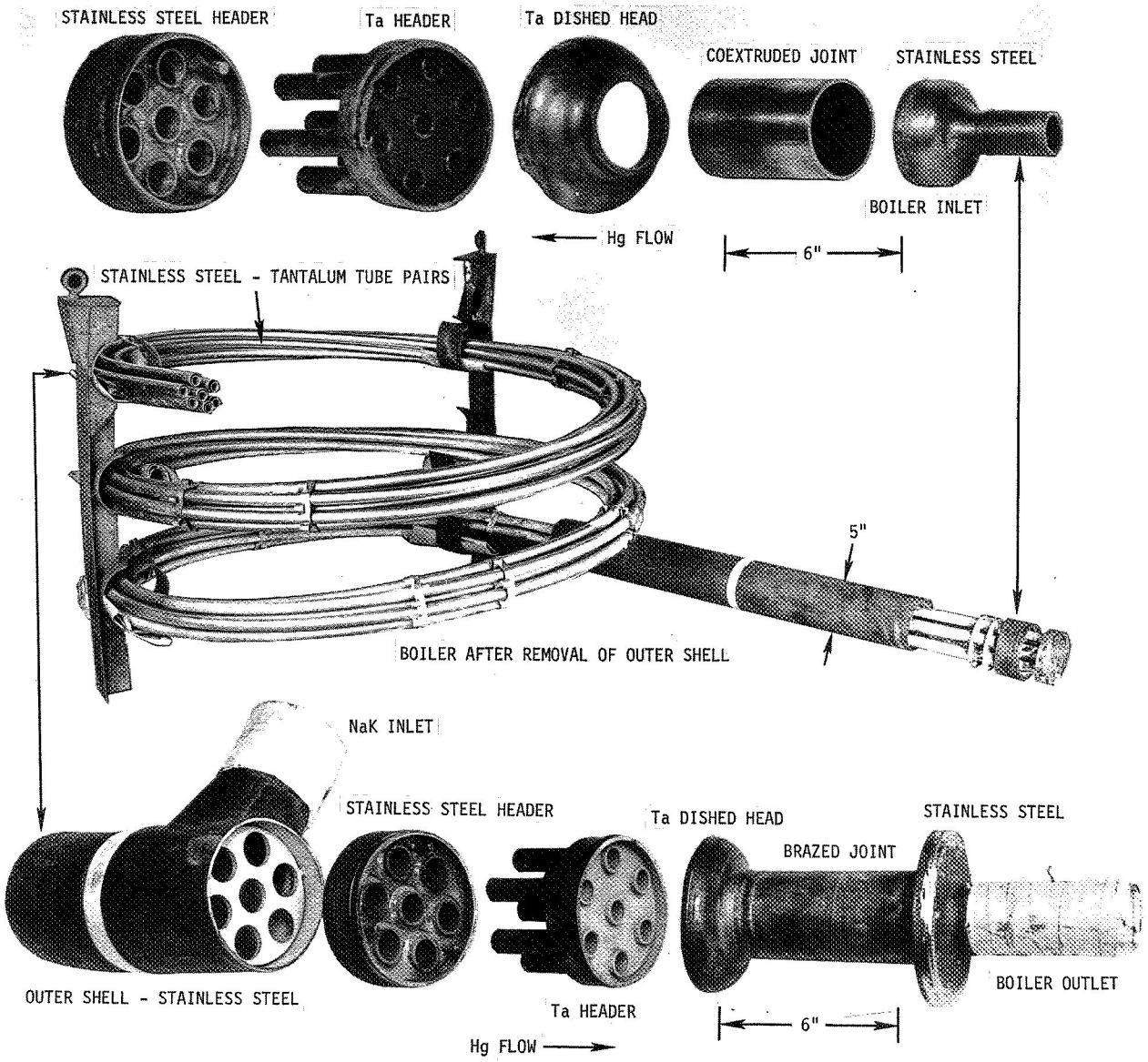


Figure 5. Sectioned SNAP-8 Boiler Following Testing. (P70-1-12S, Q, J)

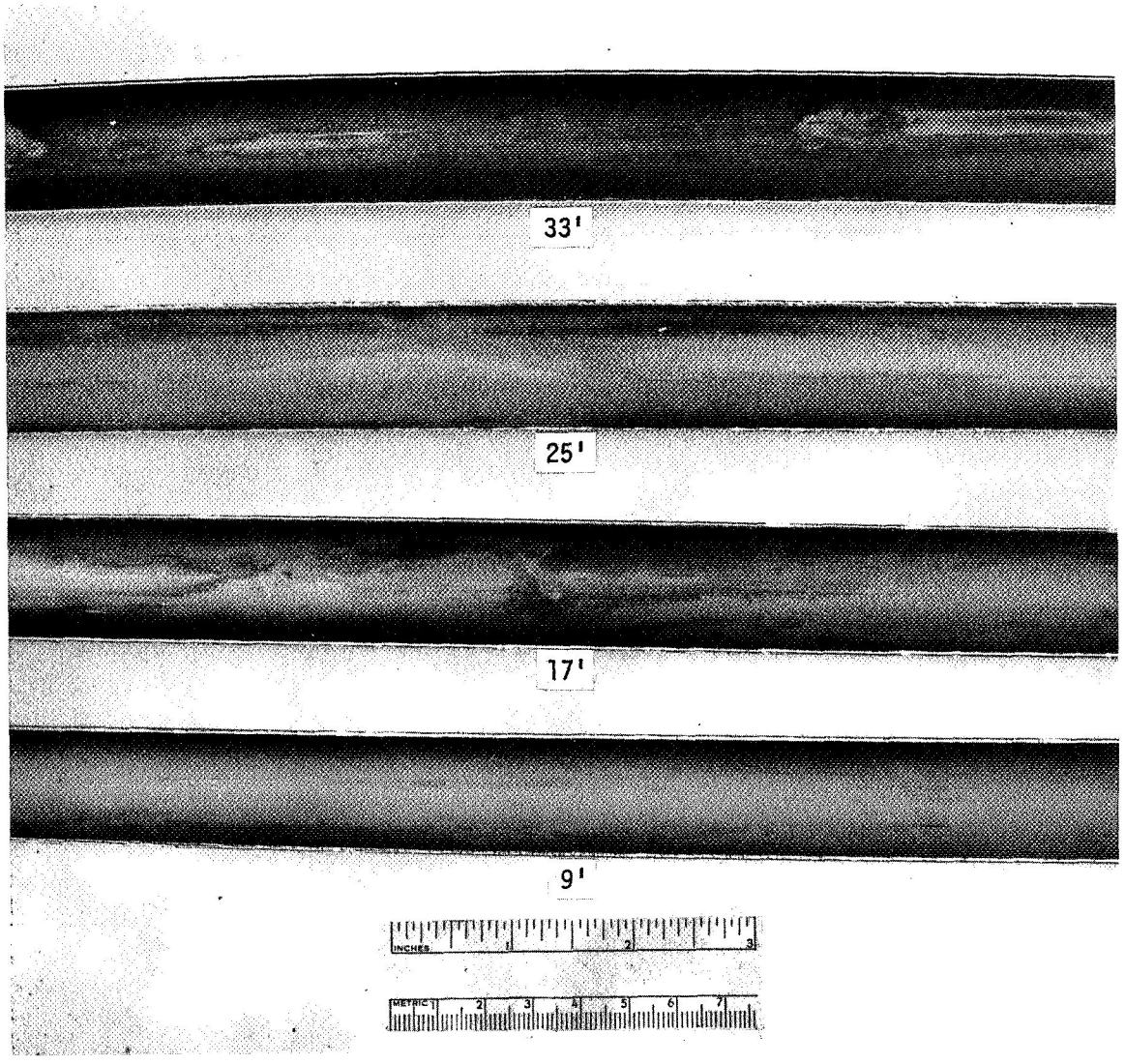


Figure 6. ID of 321 Stainless Steel Tube Exposed to Static NaK at Distances Marked From the Header at the Boiler Inlet. (P70-3-14C)

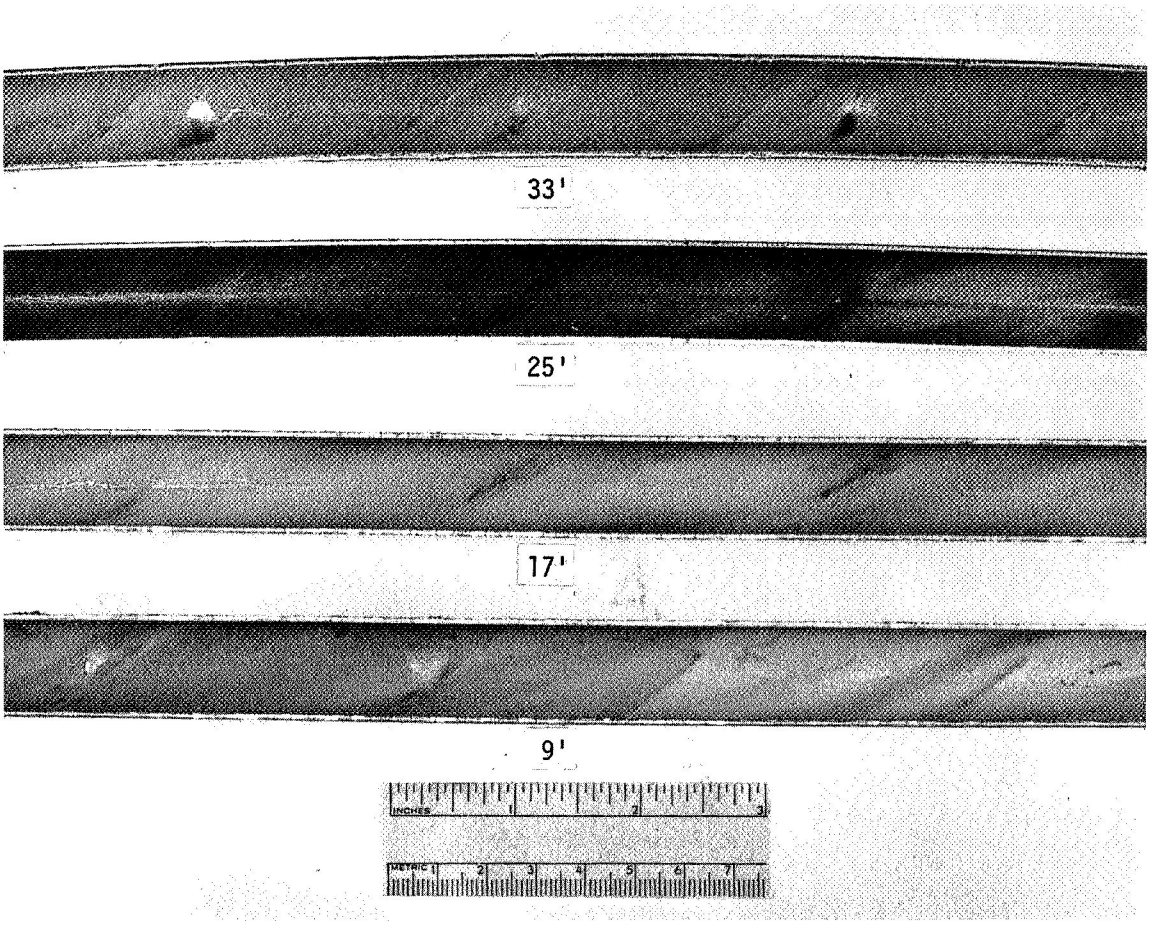


Figure 7. ID of Tantalum Tube Exposed to Mercury at Distances Marked From the Header at the Boiler Inlet. (P70-3-14D)

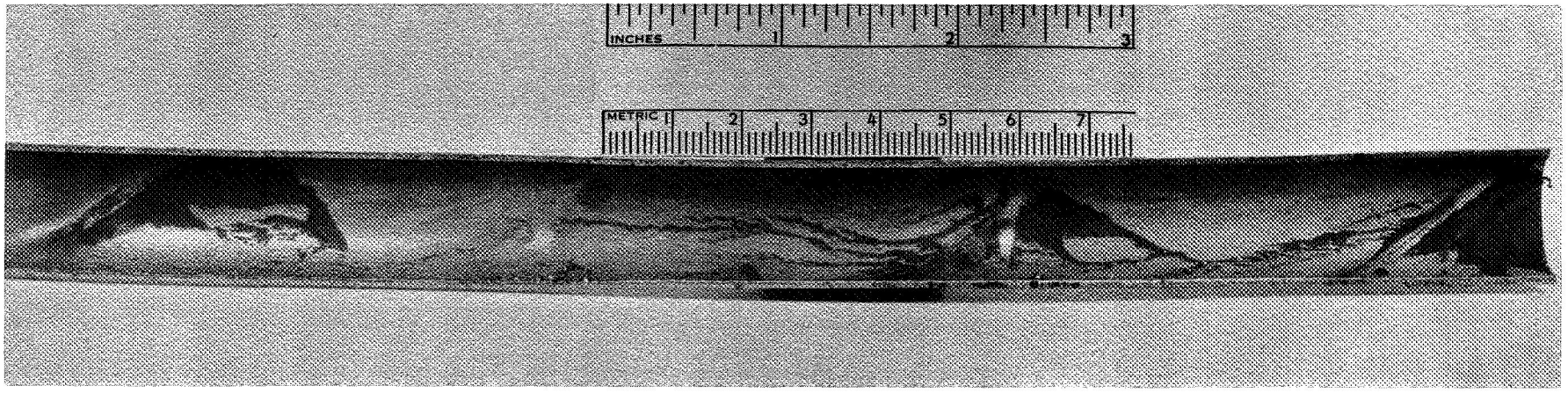


Figure 8. Dark Gray Deposit on the ID Surface of a Tantalum Boiler Tube at the Exit of the Plug Section. (P70-3-14E)

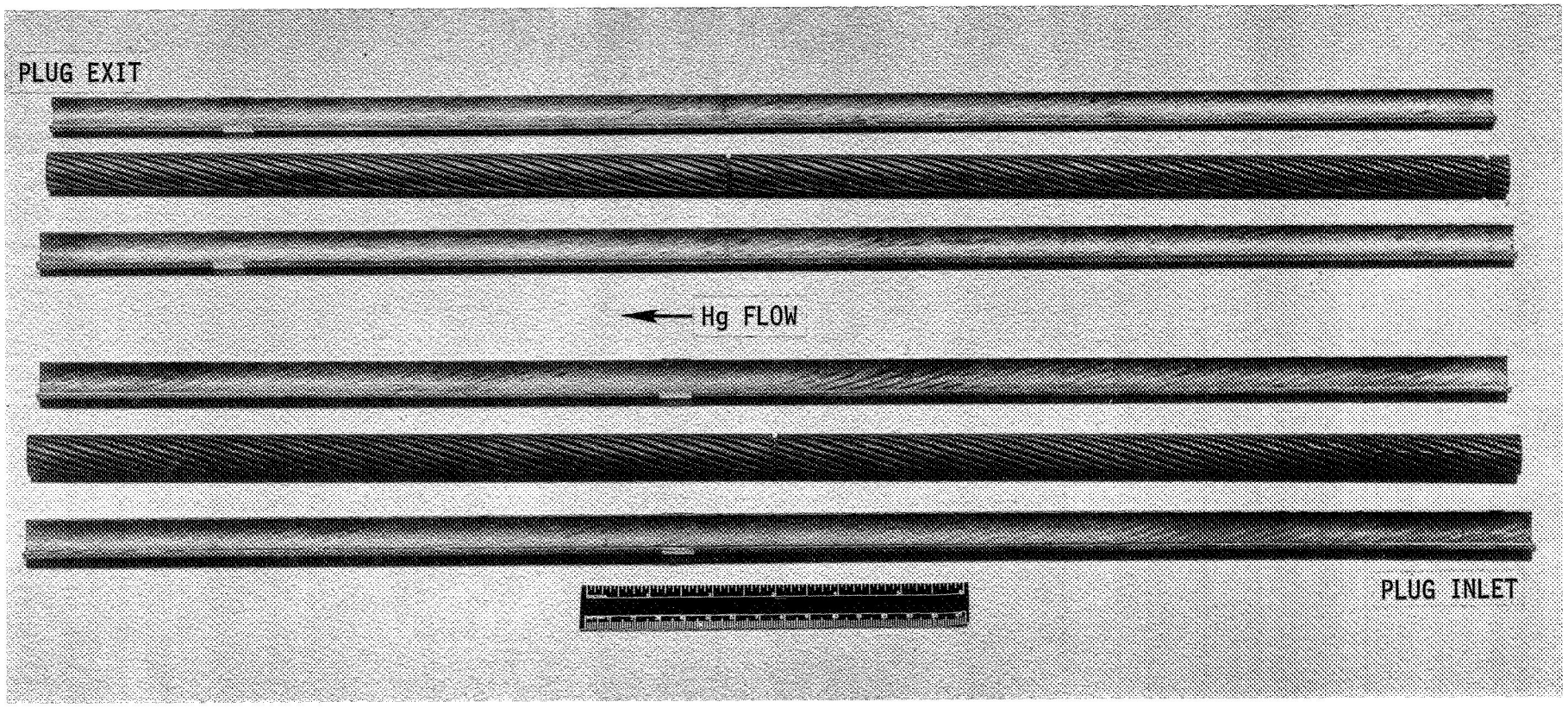


Figure 9. Tantalum Plug and Tube Section. (P70-3-28A)

H24041C Etchant: 10% Oxalic

Figure 10. Microstructure of the 316 Stainless Steel Outer Tube Shell at the NaK Inlet of SNAP-8 SN-1 Boiler.

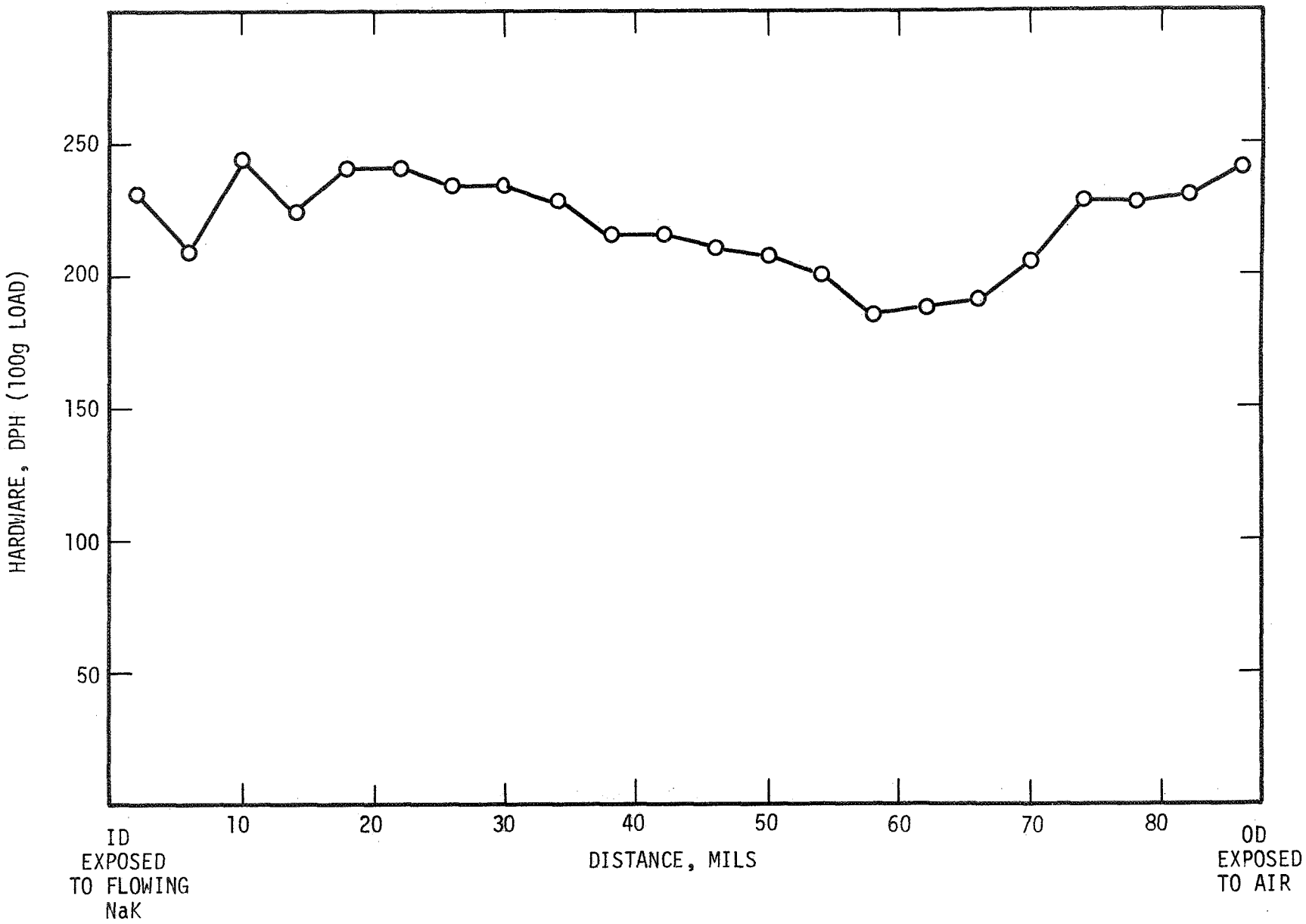
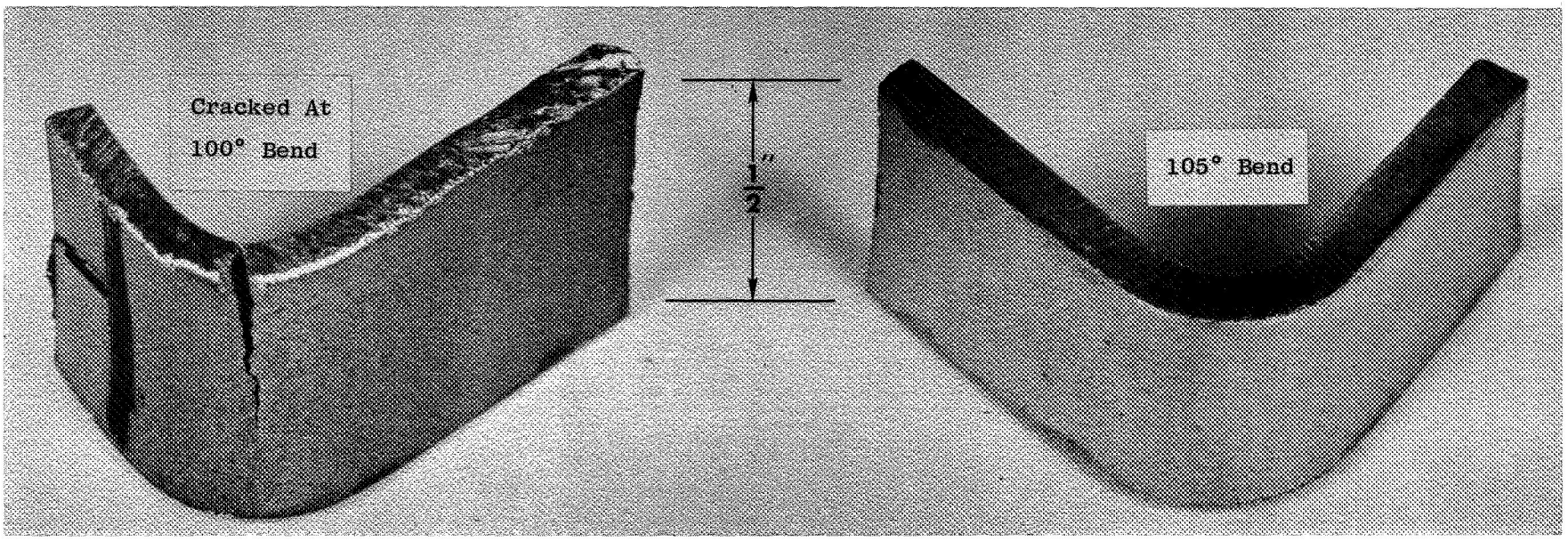


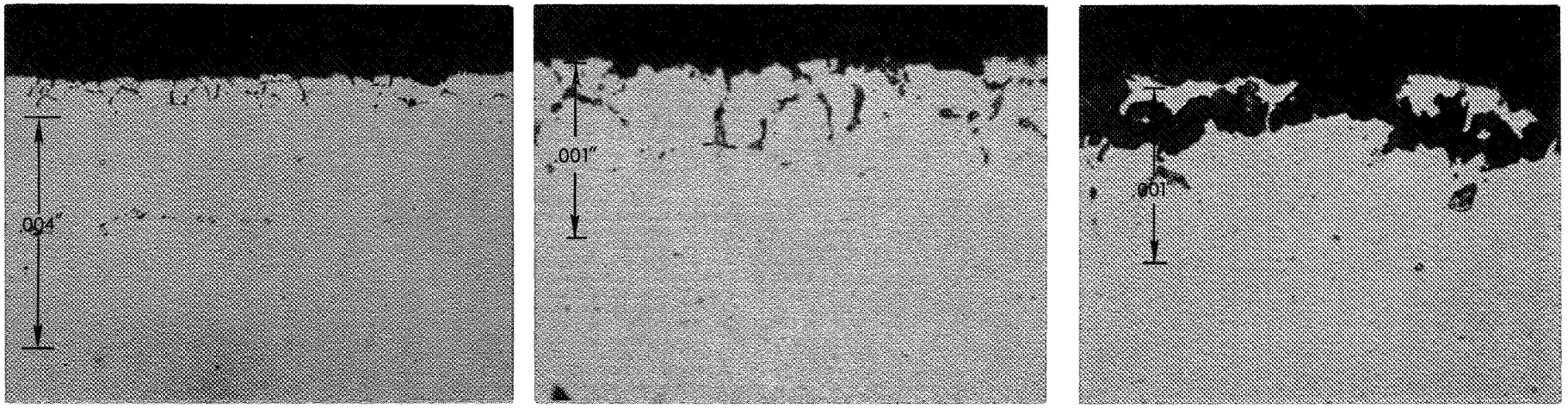
Figure 11. Hardness Across the Wall of the Outer Stainless Steel Shell.



SN-1
Boiler

BRDC-2
Boiler

Figure 12. Bend Specimens From Outer Stainless Steel Shell at NaK Inlet - Specimens Bent With ID Surface in Tension and 1T Bend Radius. (P70-8-12)



H23021A AS POLISHED H23021C ETCHANT: 10% OXALIC H23021B ETCHANT: 10% OXALIC

Figure 13. OD of Stainless Steel Tube Separating the Static and Flowing NaK Circuits in Plug Area 2' Downstream of the Header at the Boiler Inlet.

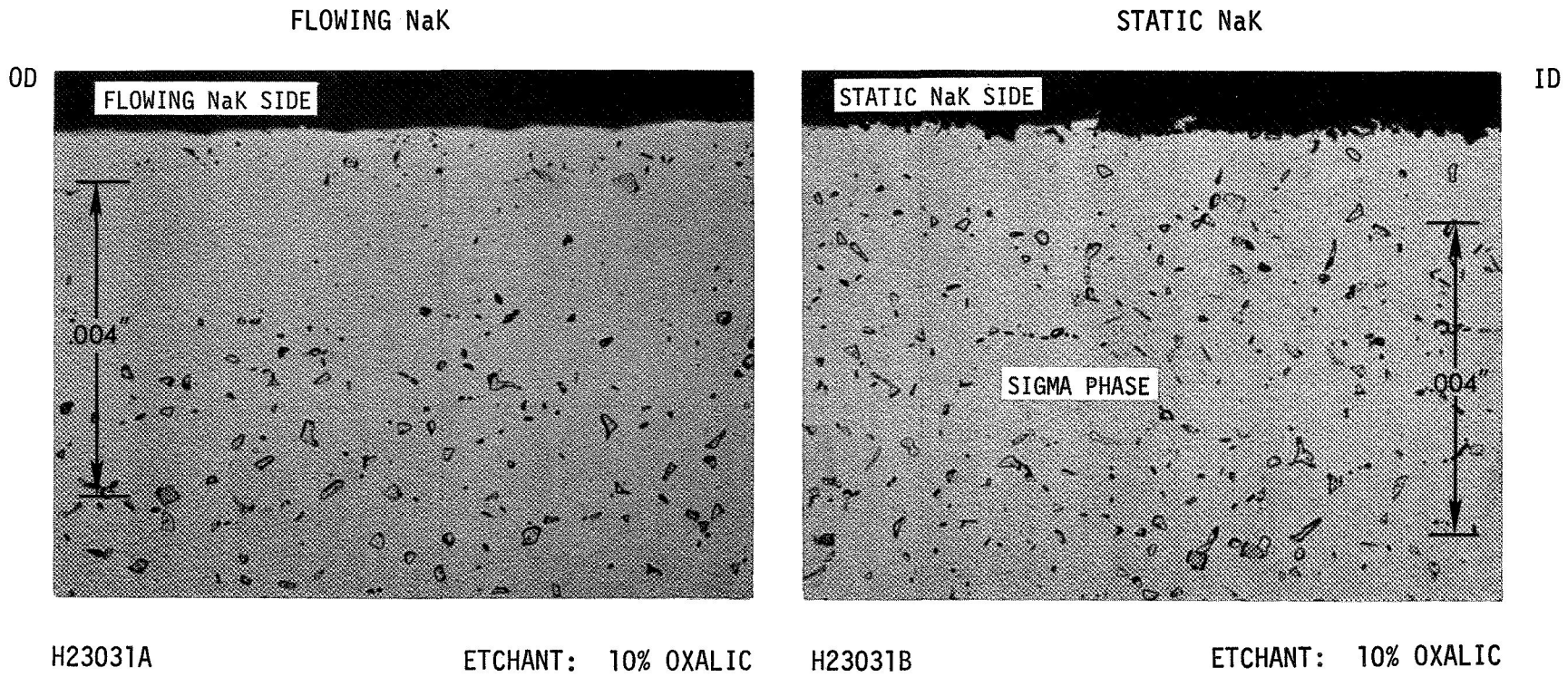


Figure 14. 321 Stainless Steel Tubing Separating the Static NaK and Flowing NaK Circuits.

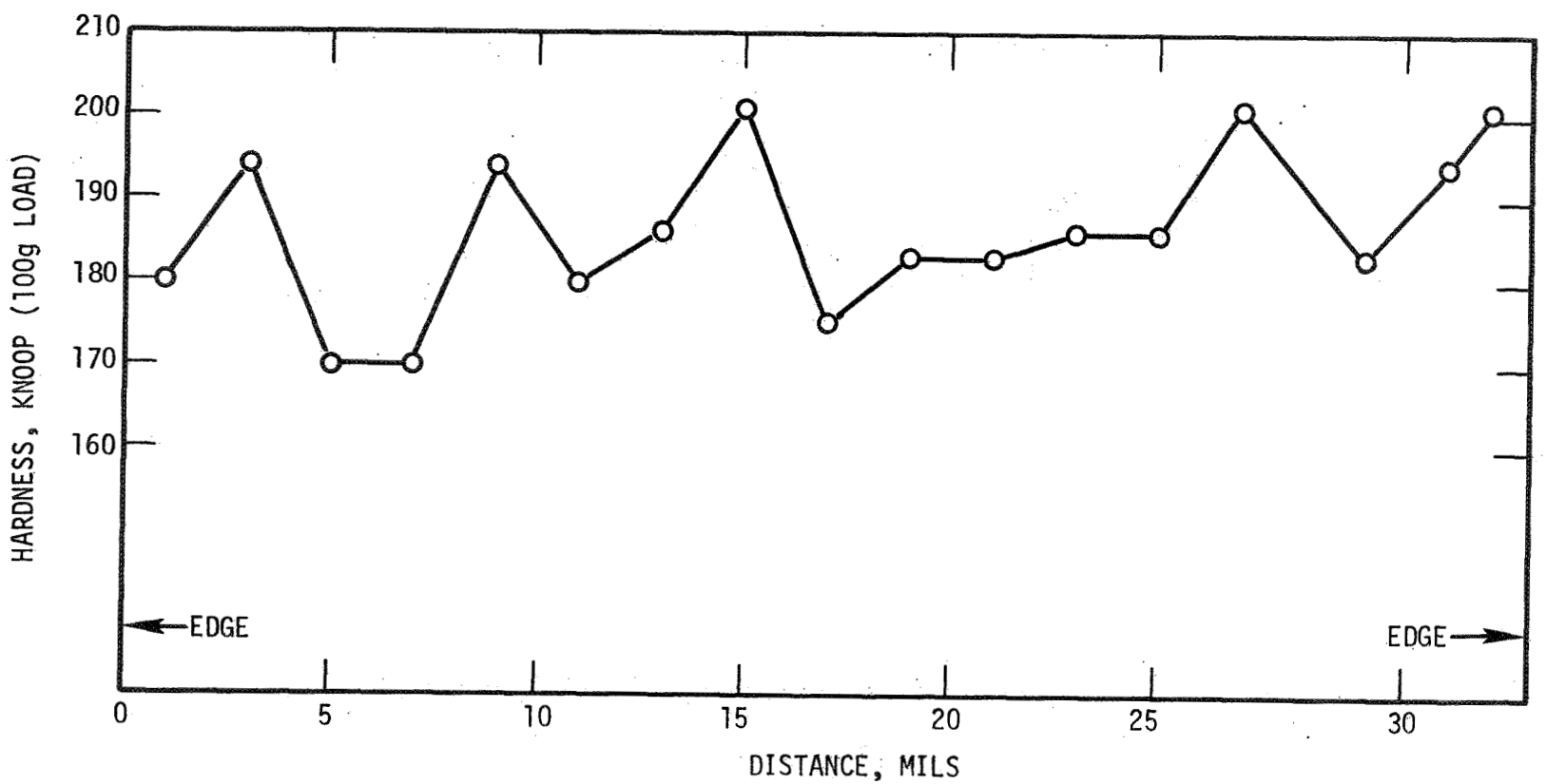
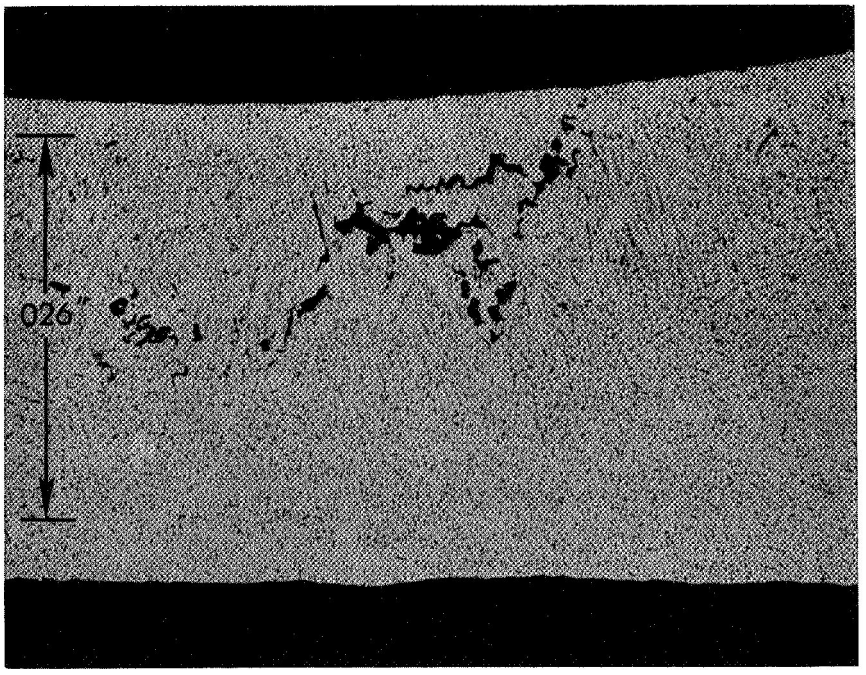


Figure 15. Hardness Across the 321 Stainless Steel Tubing Separating the Flowing and Static NaK Circuits 18 Feet From the Boiler Inlet Header.



H23041C Etchant: $30NH_4F$, $50HNO_3$, $20H_2O$

Figure 16. Weld Shrinkage in a Butt Weld in the 321 Stainless Steel Tubing Separating the Static and Flowing NaK Circuits 18 Feet Downstream From the Boiler Inlet

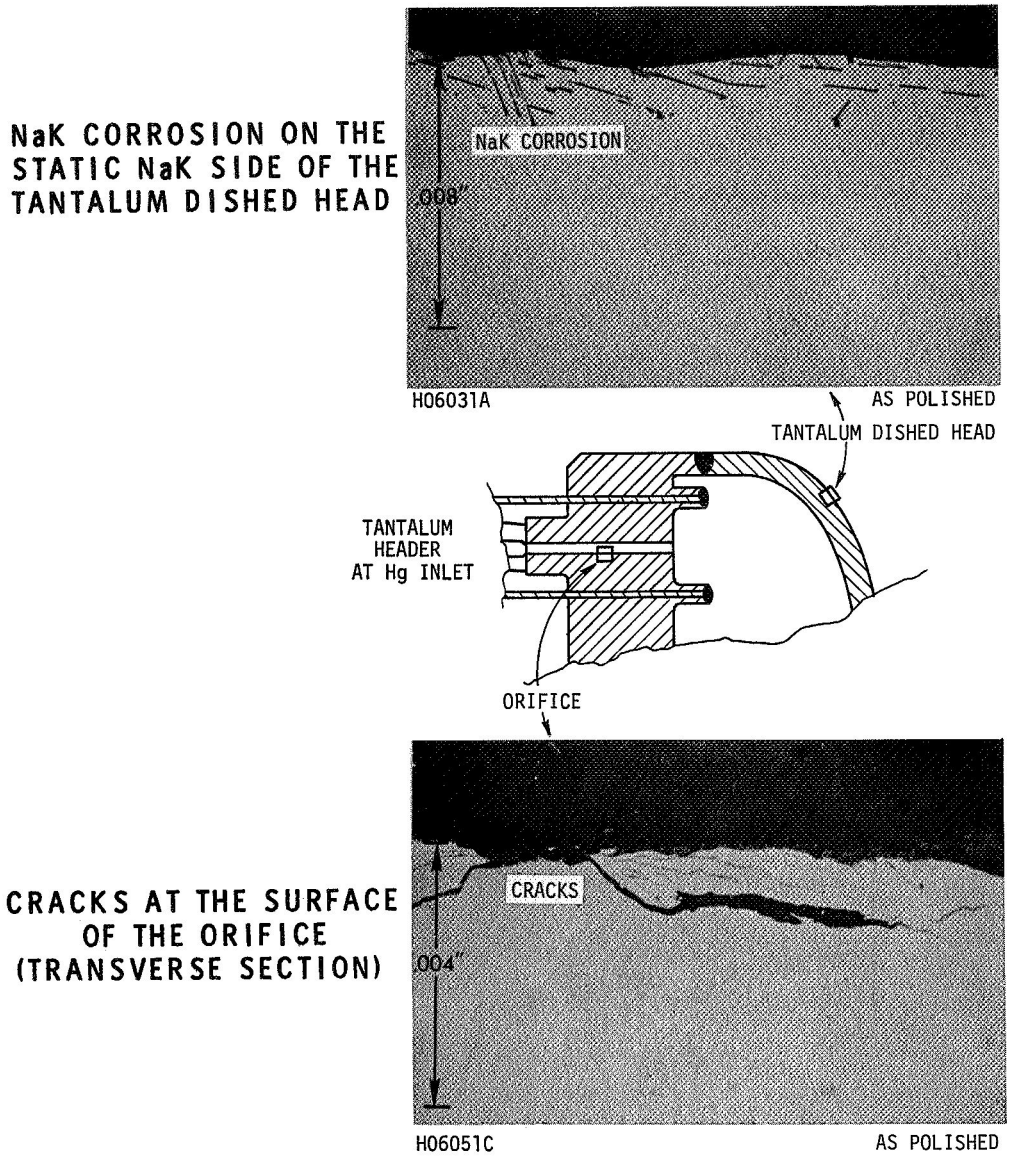
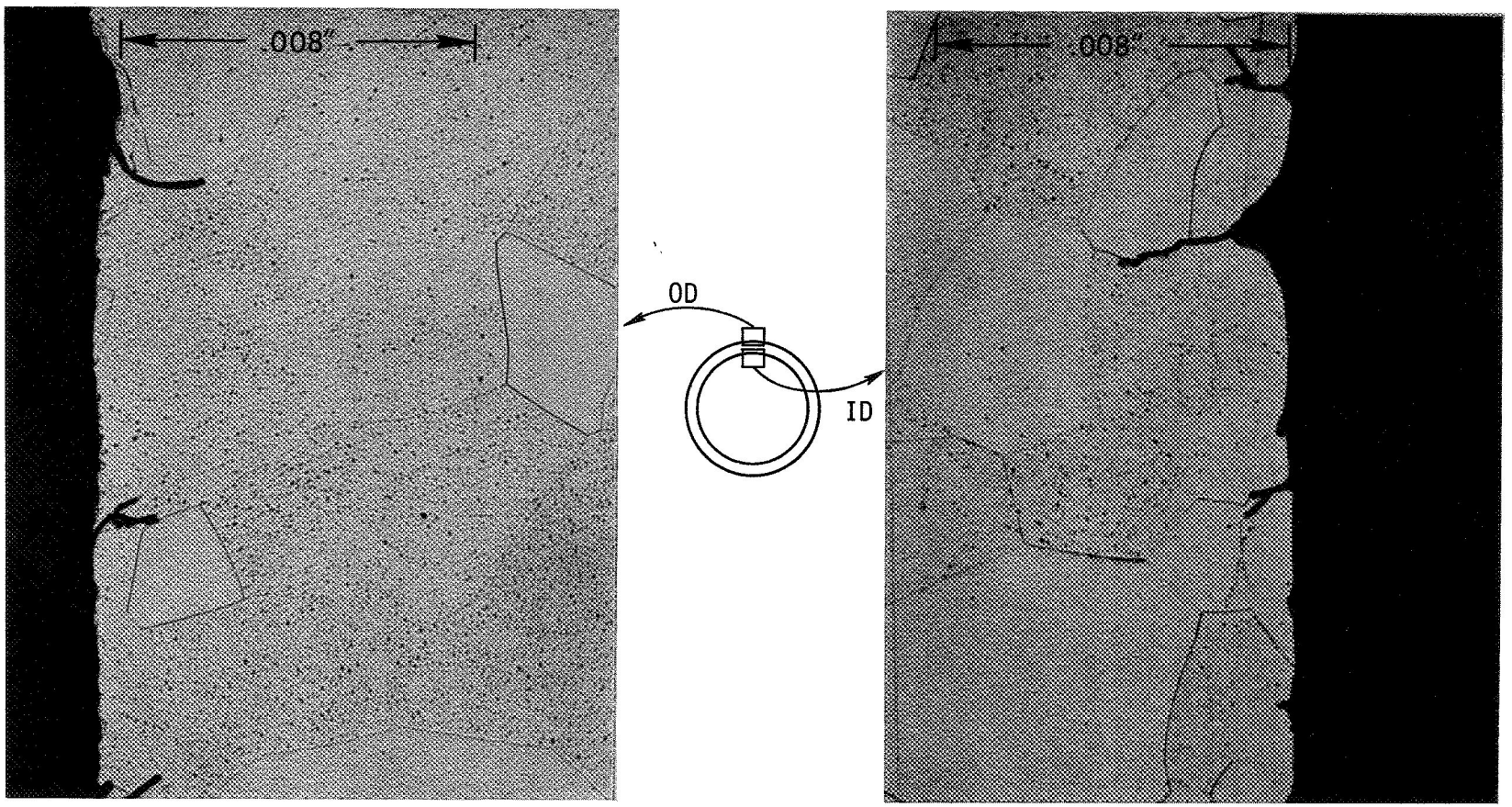


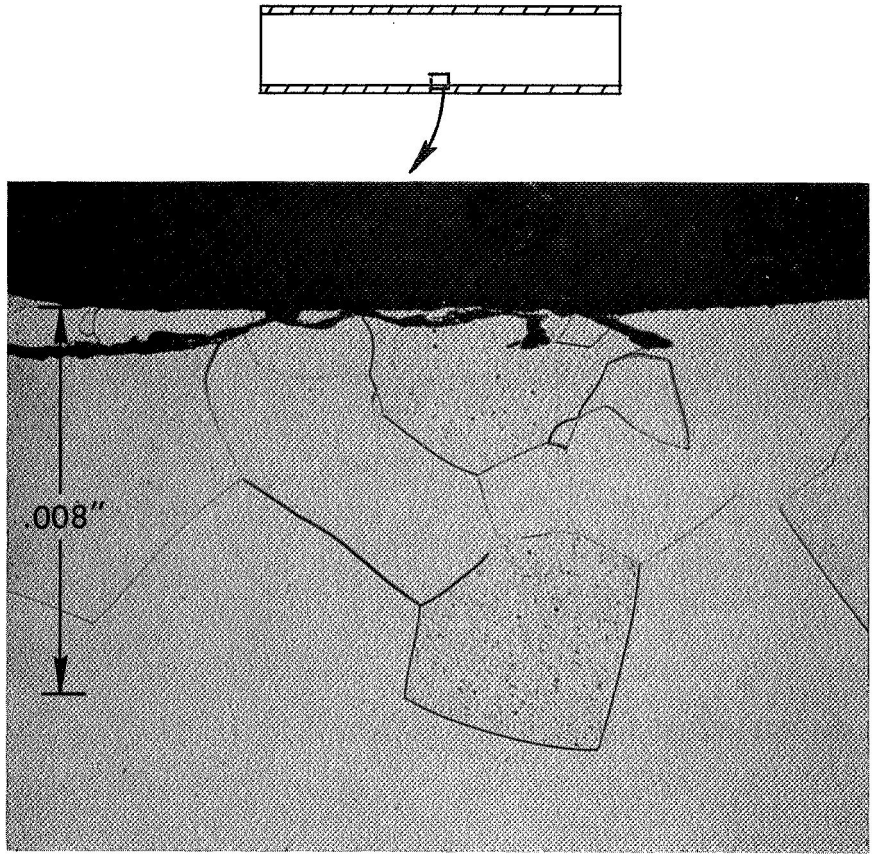
Figure 17. Posttest Microstructures of the Tantalum at the Boiler Inlet.



H24021B ETCHANT: $30NH_4F$, $50HNO_3$, $20H_2O$

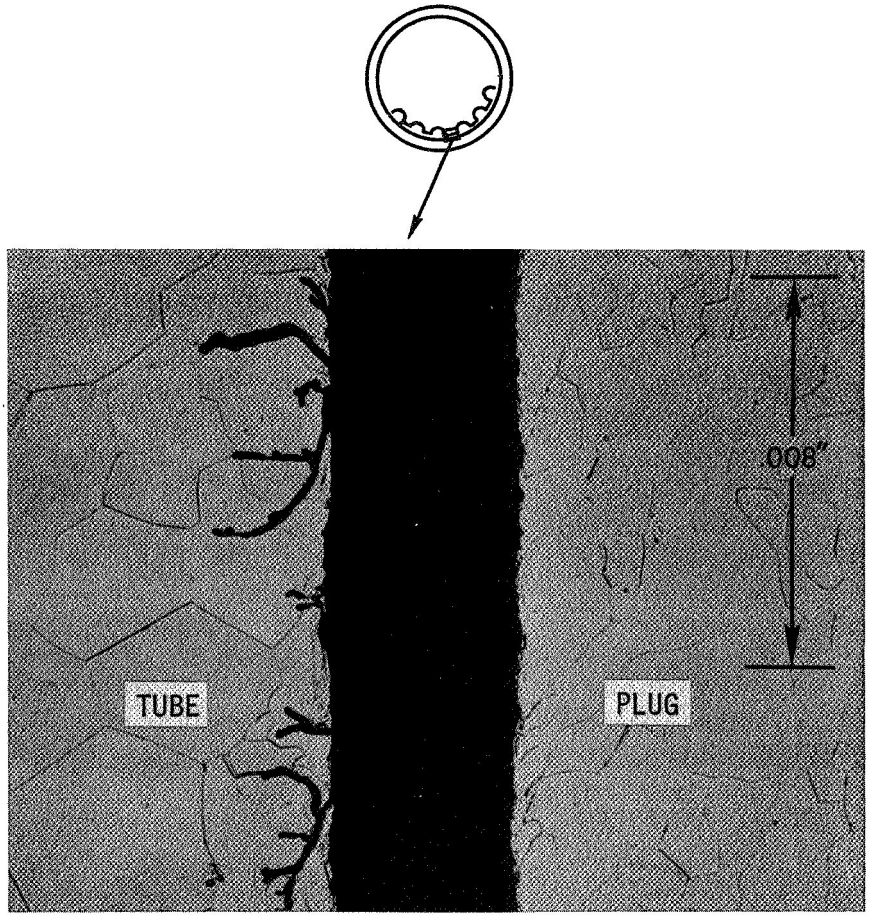
H24021C ETCHANT: $30NH_4F$, $50HNO_3$, $20H_2O$

Figure 18. Tantalum Tube - 0.670" ID x 0.040" Wall Used in the Construction of the SNAP-8 SN-1 Boiler in the Pretest Condition.



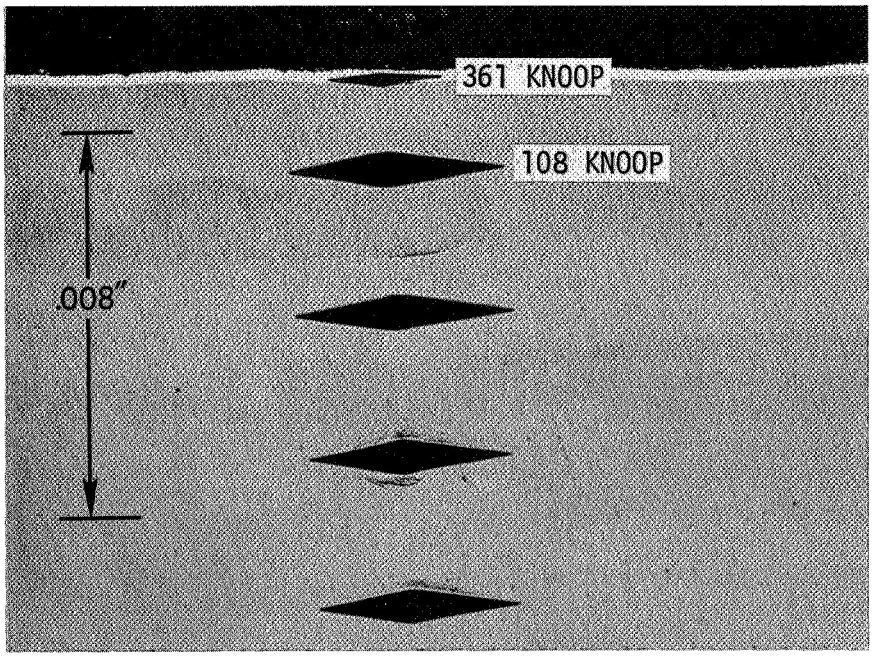
H24021A ETCHANT: $30NH_4F$, $50HNO_3$, $20H_2O$

Figure 19. Tantalum Tube - 0.670" ID x 0.040" Wall Used in the Construction of the SNAP-8 SN-1 Boiler in the Pretest Condition.



H19031A ETCHANT: 30%NH_4F, 50%HNO_3, 20%H_2O

Figure 20. Tantalum Tube - Plug Assembly at Plug Exit.



H06041A AS POLISHED

Figure 21. The Surface Coating on the Mercury Side of the Tantalum Tubing in the Vapor Region of the Boiler.

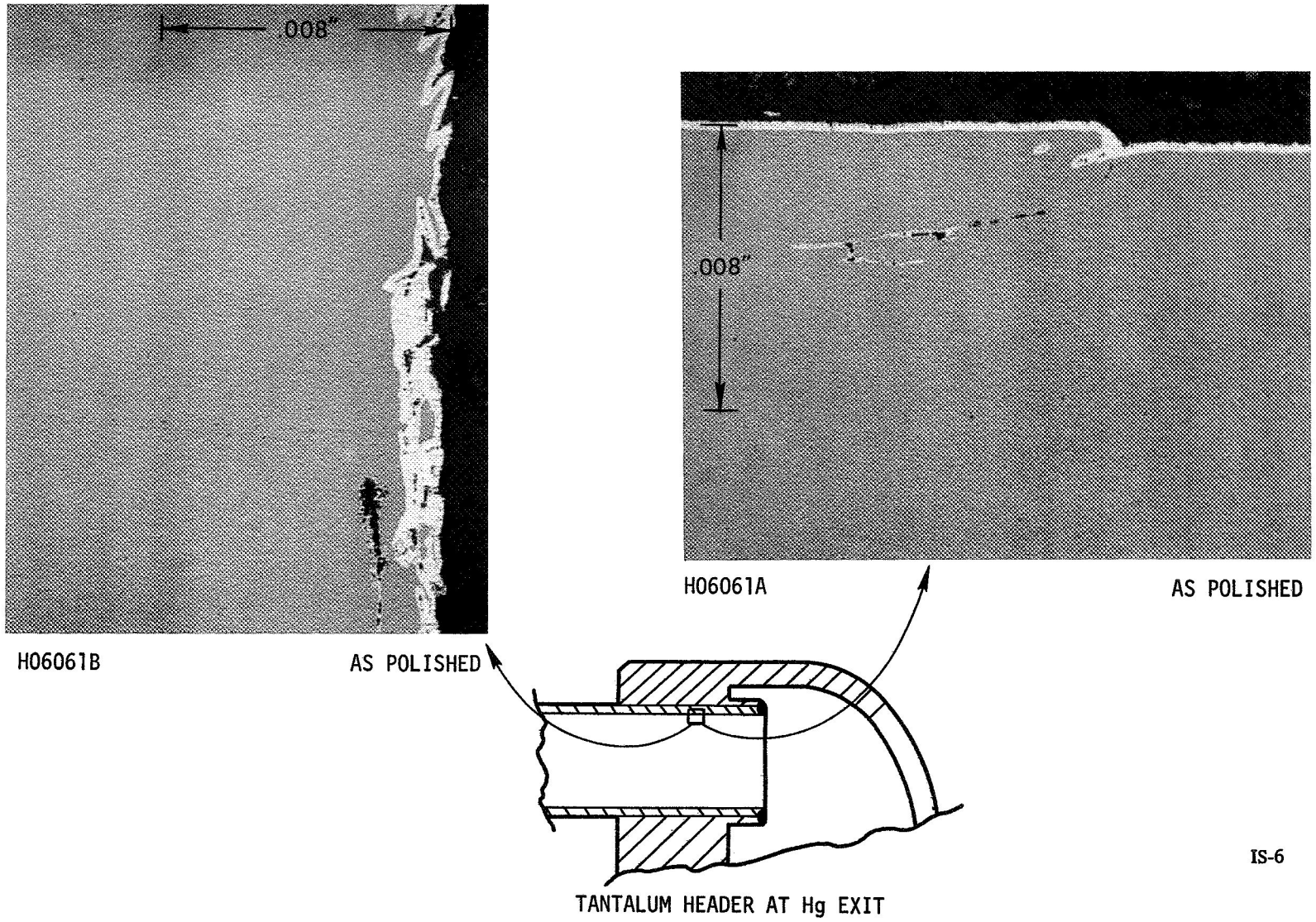


Figure 22. The Surface Coating on the Mercury Side of the Tantalum Tubing in the Vapor Region of the Boiler.

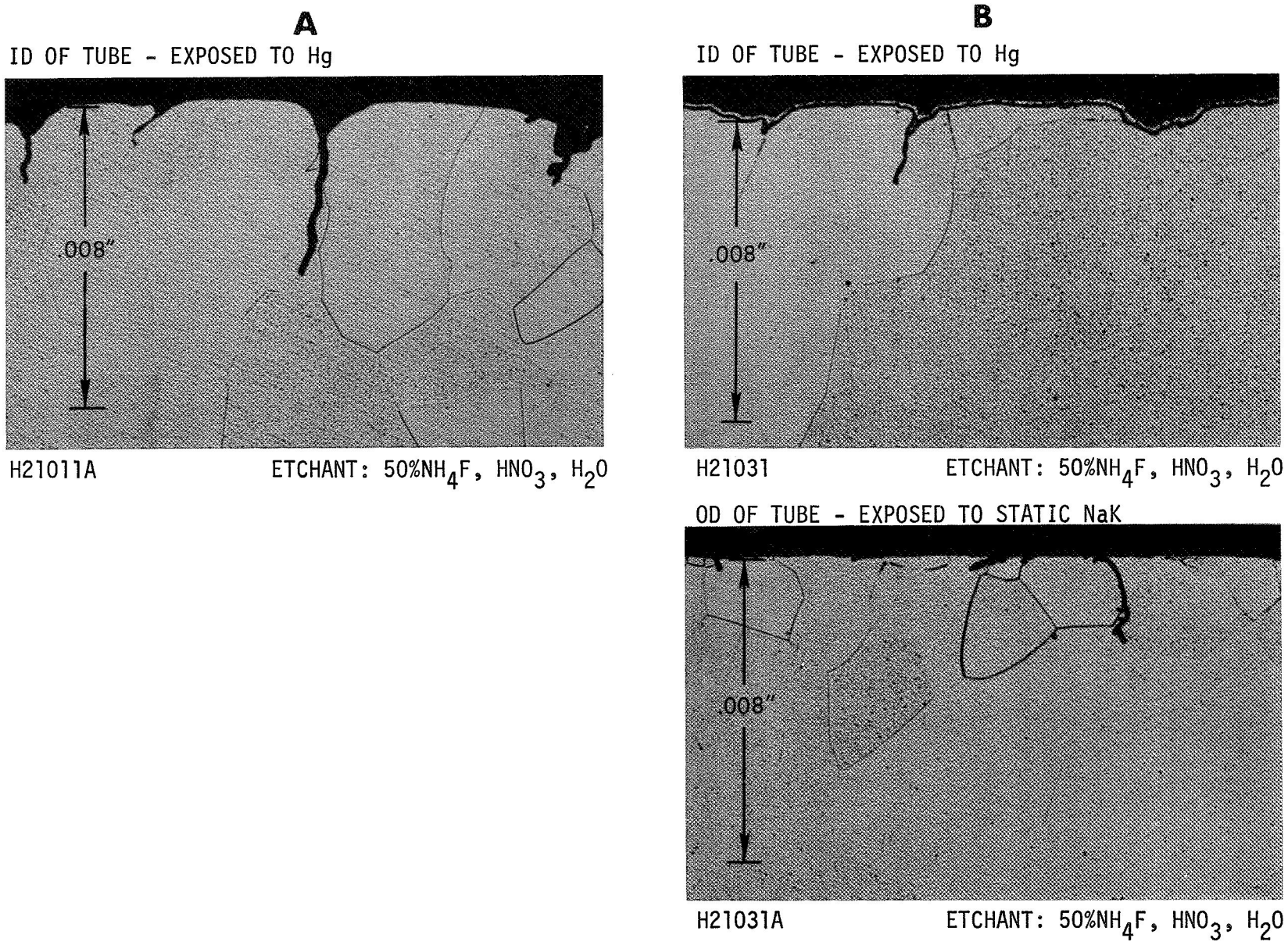
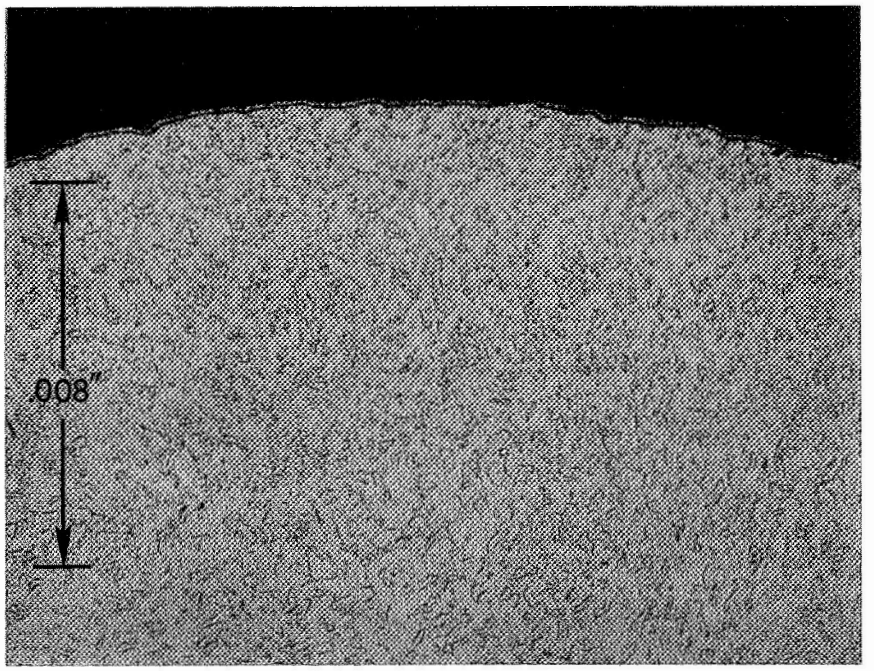
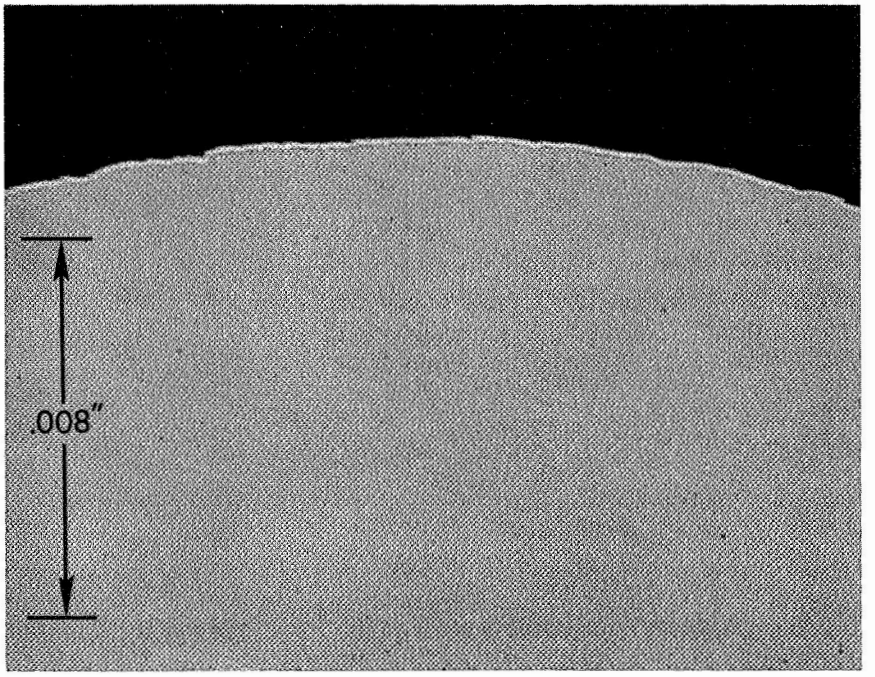


Figure 23. Tantalum Tube Separating Hg from Static NaK (A) 5' Downstream of Tantalum Header at Hg Inlet (B) 21' Downstream of Tantalum Header at Hg Inlet.

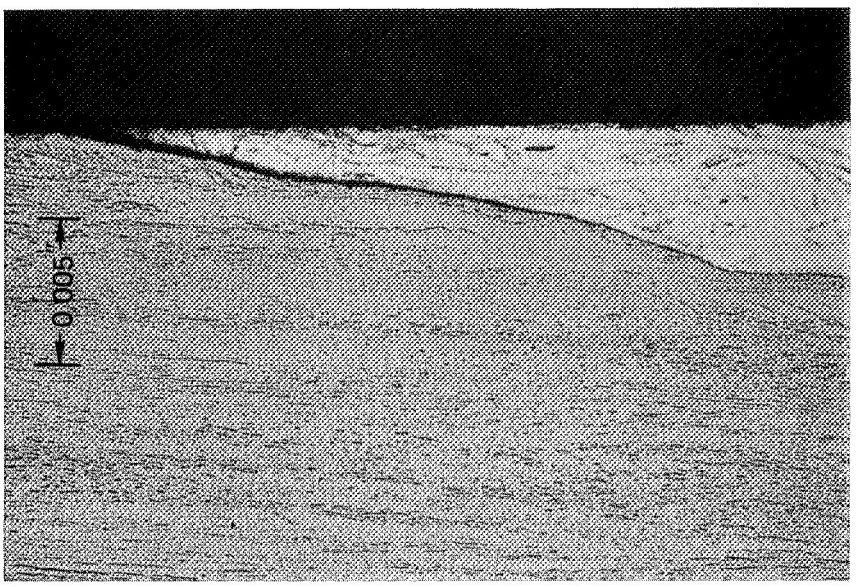


H24011B ETCHANT: $30NH_4F$, $50HNO_3$, $20H_2O$



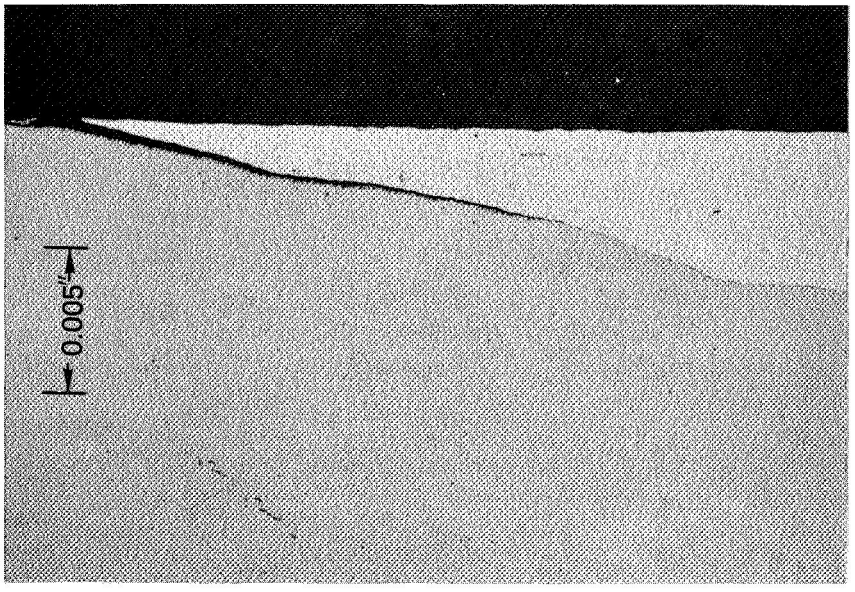
H24011A AS POLISHED

Figure 24. The Surface Coating on the Ta-10W Turbulator Wire.



H92021B

ETCHANT: NH_4F
HNO_3
H_2O



H92021A

AS POLISHED

Figure 25. Separation of 316 Stainless Steel Feather-Edge From Tantalum on OD of Coextruded Joint From Inlet of SN-1 Boiler.

H92041B

ETCHANT: NH_4F
HNO_3
H_2O

Figure 26. Tantalum - 316 Stainless Steel Interface of Coextruded Joint From Inlet at SN-1 Boiler.

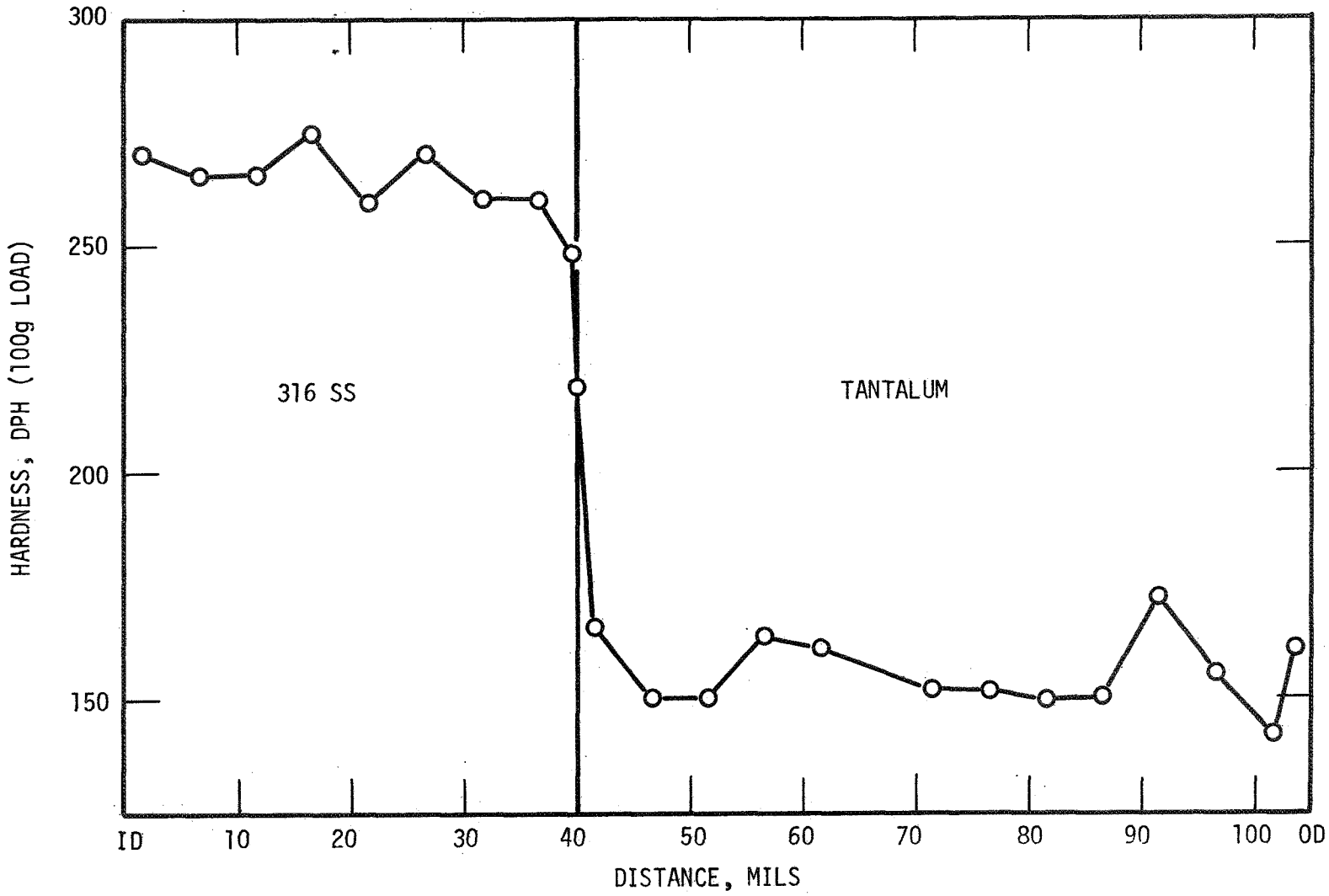


Figure 27. Hardness Traverse Across Coextruded Ta/316 Joint.

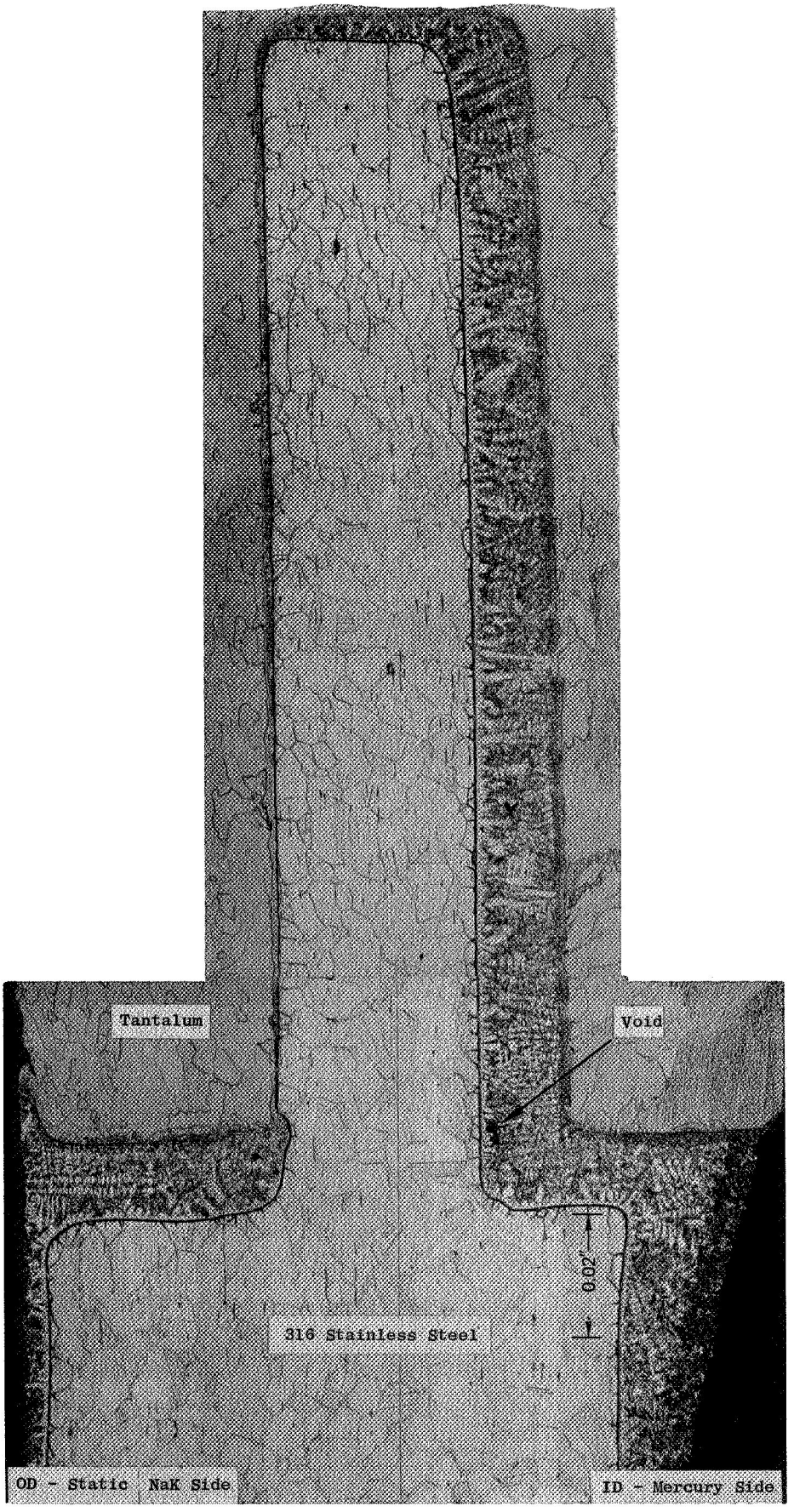


Figure 28. Microstructure of the Ta/316 SS Brazed Joint From Mercury Exit of SN-1 SNAP-8 Boiler at the Location With the Most Defects as Indicated by Ultrasonic Inspection. (H91031A, B, C, D)

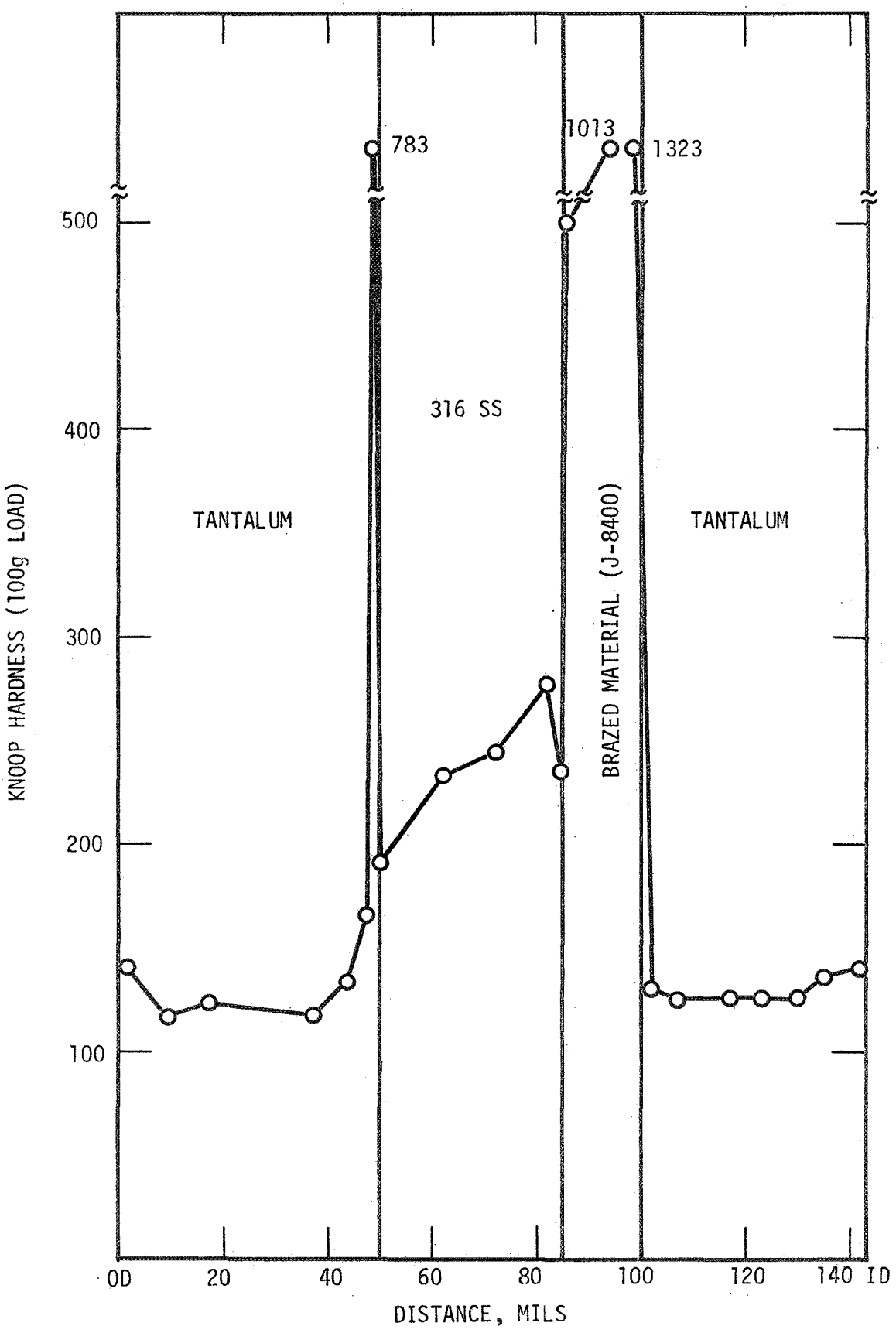


Figure 29. Hardness Traverse Across Ta/316 Brazed Joint.

DISTRIBUTION LIST
TOPICAL REPORT
CONTRACT NAS 3-10610

NASA
Washington, D.C. 20546
Attn: P. R. Miller (RNP)

NASA
Washington, D.C. 20546
Attn: James J. Lynch (RNP)

NASA
Washington, D.C. 20546
Attn: George C. Deutsch (RR)

NASA
Washington, D.C. 20546
Attn: Dr. Fred Schulman (RNP)

NASA
Washington, D.C. 20546
Attn: H. Rochen (RNP)

NASA
Scientific and Tech. Info. Facility
P.O. Box 33
College Park, Maryland 20740
Attn: Acquisitions Branch
(SQT - 34054) 2 Copies

NASA
Ames Research Center
Moffett Field, California 94035
Attn: Librarian

NASA
Goddard Space Flight Center
Greenbelt, Maryland 20771
Attn: Librarian

NASA
Langley Research Center
Hampton, Virginia 23365
Attn: Librarian

NASA
Manned Spacecraft Center
Houston, Texas 77001
Attn: Librarian

NASA
George C. Marshall Space Flight Ctr.
Huntsville, Alabama 35812
Attn: Librarian

NASA
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, California 91103
Attn: Librarian

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Librarian

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: H. O. Slone, MS 500-201

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: G. M. Ault, MS 105-1

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: P. L. Stone, MS 106-1

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: G. M. Thur, MS 500-201

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: John E. Dilley, MS 500-309

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Technology Utilization, MS 3-19

Report Distribution List - NAS 3-10610 - Topical (Continued)

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: Report Control Office, MS 5-5

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: R. L. Davies, MS 106-1

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: V. F. Hlavin, MS 3-14
(Final Only)

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: E. R. Furman, MS 500-202
3 Copies

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: M. J. Saari, MS 500-202

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: R. English, MS 500-201

NASA
Lewis Research Center
21000 Brookpark Road
Cleveland, Ohio 44135
Attn: M. Gutstein, MS 500-201

National Bureau of Standards
Washington, D.C. 20546
Attn: Librarian

AFSC
Aeronautical Systems Division
Wright-Patterson AFB, Ohio 45433
Attn: Charles Armbruster (ASRPP-10)

AFSC
Aeronautical Systems Division
Wright-Patterson AFB, Ohio 45433
Attn: T. Cooper

AFSC
Aeronautical Systems Division
Wright-Patterson AFB, Ohio 45433
Attn: Librarian

Army Ordnance Frankford Arsenal
Bridesburg Station
Philadelphia, Pennsylvania 19137
Attn: Librarian

U.S. Atomic Energy Commission
Tech. Info. Service Extension
P.O. Box 62
Oak Ridge, Tennessee 37831

U.S. Atomic Energy Commission
Washington, D.C. 20545
Attn: M. J. Whitman

U.S. Atomic Energy Commission
Washington, D.C. 20545
Attn: J. M. Simmons

Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60440
Attn: Librarian

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201
Attn: R. T. Nichoff, DMIC

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201
Attn: Librarian

Brookhaven National Laboratory
Upton, Long Island, New York, 11973
Attn: Librarian

Brookhaven National Laboratory
Upton, Long Island, New York 11973
Attn: Dr. D. H. Gurinsky

Brookhaven National Laboratory
Upton, Long Island, New York 11973
Attn: Dr. J. R. Weeks

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
Attn: J. Devan

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
Attn: R. MacPherson

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
Attn: Librarian

Office of Naval Research
Power Division
Washington, D.C. 20360
Attn: Librarian

Bureau of Weapons
Research and Engineering
Materials Division
Washington, D.C. 20546
Attn: Librarian

U.S. Naval Research Laboratory
Washington, D.C. 20390
Attn: Librarian

Aerojet-General Corporation
Electronics Division
Azusa, California 91703
Attn: Librarian

AiResearch Manufacturing Company
Division of the Garrett Corporation
Sky Harbor Airport
402 South 36th Street
Phoenix, Arizona 85034
Attn: Librarian

AiResearch Manufacturing Company
Division of the Garrett Corporation
9851-9951 Sepulveda Boulevard
Los Angeles, California 90009
Attn: Librarian

IIT Research Institute
10 West 35th Street
Chicago, Illinois 60616
Attn: Librarian

Babcock & Wilcox Company
Research Center
Alliance, Ohio 44601
Attn: Librarian

North American Rockwell Corporation
Atomics International Division
8900 DeSoto Avenue
Canoga Park, California 91304
Attn: Librarian

North American Rockwell Corporation
Atomics International Division
8900 DeSoto Avenue
Canoga Park, California 91304
Attn: P. B. Ferry

North American Rockwell Corporation
Atomics International Division
8900 DeSoto Avenue
Canoga Park, California 91304
Attn: T. A. Moss

AVCO
Research & Advanced Development Dept.
201 Lowell Street
Wilmington, Massachusetts 01887
Attn: Librarian

Electro-Optical Systems, Inc.
Advanced Power Systems Division
Pasadena, California 91107
Attn: Librarian

Fansteel Metallurgical Corporation
North Chicago, Illinois 18201
Attn: Librarian

General Dynamics Corporation
General Atomic Division
John Jay Hopkins Lab.
P.O. Box 608
San Diego, California 92112
Attn: Librarian

General Electric Company
Space Division
3198 Chestnut Street
Philadelphia, Pennsylvania 19104
Attn: Librarian

General Electric Company
Vallecitos Atomic Laboratory
Pleasanton, California 94566
Attn: Librarian

General Dynamics/Fort Worth
P.O. Box 748
Fort Worth, Texas 76101
Attn: Librarian

General Motors Corporation
Allison Division
Indianapolis, Indiana 46206
Attn: Librarian

Hamilton Standard
Division of United Aircraft Corp.
Windsor Locks, Connecticut 06096
Attn: Librarian

Hughes Aircraft Company
Engineering Division
Culver City, California 90230
Attn: Librarian

Lawrence Radiation Lab.
Livermore, California 94550
Attn: Librarian

Lockheed Missiles and Space Div.
Lockheed Aircraft Corporation
Sunnyvale, California 90221
Attn: Librarian

The Martin Company
Nuclear Division
P.O. Box 5042
Baltimore, Maryland 21203
Attn: Librarian

Martin Marietta Corporation
Metals Technology Laboratory
Wheeling, Illinois 60090

Materials Research Corporation
Orangeburg, New York 10962
Attn: Librarian

McDonnel Aircraft
St. Louis, Missouri 63166
Attn: Librarian

MSA Research Corporation
Callery, Pennsylvania 16024
Attn: Librarian

National Research Corporation
70 Memorial Drive
Cambridge, Massachusetts 02142
Attn: Librarian

North American Rockwell
Los Angeles Division
Los Angeles, California 90009
Attn: Librarian

United Aircraft Corporation
Pratt & Whitney Aircraft Division
400 Main Street
East Hartford, Connecticut 06108
Attn: Librarian

Republic Aviation Corporation
Farmingdale, Long Island, New York 11735
Attn: Librarian

Sandia Corporation
P.O. Box 5800
Albuquerque, New Mexico 87116
Attn: Librarian

Sandia Corporation
P.O. Box 5800
Albuquerque, New Mexico 87116
Attn: Don Johnson

Solar
2200 Pacific Highway
San Diego, California 92112
Attn: Librarian

Southwest Research Institute
8500 Culebra Road
San Antonio, Texas 78220
Attn: Librarian

Superior Tube Company
Norristown, Pennsylvania 19404
Attn: L. Shaheen

Philco Corporation
Aeronutronics
Newport Beach, California 92663
Attn: Librarian

General Electric Company
Nuclear Systems Program
Space Division
Cincinnati, Ohio 45215

General Electric Company
Evendale, Ohio 45215
Technical Information Center
Building 700, Mail Drop N-32

TRW Inc.
23555 Euclid Avenue
Cleveland, Ohio 44117
Attn: Librarian

Union Carbide Corporation
Materials Systems Division
1020 W. Park
Kokomo, Indiana 46901
Attn: Librarian

Union Carbide Corporation
Materials Systems Division
1020 W. Park
Kokomo, Indiana 46901
Attn: Technology Department

Wah Chang Corporation
Albany, Oregon 97321
Attn: Librarian

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh, Pennsylvania 15236
Attn: R. W. Luckman

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh, Pennsylvania 15236
Attn: G. G. Lessman

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh, Pennsylvania 15236
Attn: D. R. Stoner

Westinghouse Electric Corporation
Astronuclear Laboratory
P.O. Box 10864
Pittsburgh, Pennsylvania 15236
Attn: Librarian

Whittaker Corporation
Nuclear Metals Division
West Concord, Massachusetts 01781
Attn: P. Lowenstein

Geoscience, Ltd.
410 S. Cedros Avenue
Solana Beach, California 92075
Attn: H. Poppendiek