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SNAP-8 TOPICAL MATERIALS REPORT FOR 1962

VOL. III - SNAP-8 MERCURY CORROSION AND MATERIALS RESEARCH

A Report to

National Aeronautics and Space Administration

Report No. 2517 September 1963 Copy No.

Vol III

Contract NAS 5-417

GENERAL TIRE

Aerojet General Corporation Report No. 2517, Vol. III
Aerojet General Nucleonics Report No. AN-TM-192
Subcontract 274949, under NAS 5-417

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SNAP-8 MERCURY CORROSION AND MATERIALS RESEARCH

Topical Report

For the Period June, 1960 to December, 1962

By

M. F. Parkman, B. E. Farwell, D. K. Whaley, and R. V. Arabian

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AEROJET-GENERAL NUCLEONICS
A SUBSIDIARY OF AEROJET-GENERAL CORPORATION

This report is submitted in partial fulfillment of National Aeronautics and Space Administration Contract NAS 5-417. This volume is the third of four volumes that comprise the complete report.

GENERAL TIRE

FOREWORD

This work was conducted from July, 1960 to December, 1962 at Aerojet-General Nucleonics, San Ramon, California, as part of the SNAP-8 Materials Research Program, under Subcontract 274949 to Aerojet General Corporation, Azusa, California. The SNAP-8 Division of Aerojet General Corporation is developing the SNAP-8 Power Conversion System for the National Aeronautics and Space Administration under Contract NAS 5-417.

The work was carried out at different times by the following personnel:

Project Engineers, San Ramon SNAP-8 Materials Research Activities: J. R. Payne, M. F. Parkman, and R. S. Carey.

Task Engineers of work reported herein:

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ABSTRACT

Mercury corrosion of Haynes Alloy No. 25, Type 405 stainless steel, AM 350, 9Cr-1Mo and Cb-1Zr thermal convection capsules was investigated between 1000 and 10,000 hours at 1025°F and 1100°F and between 500 and 2000 hours at 1175° and 1250°F. Isothermal capsules of Haynes 25 and Type 405 stainless steel were operated for 5000 and 1000 hours. Thermal convection capsules were heated at the bottom and cooled at the top to create a thermal gradient of 85° to 150°F.

Penetration of the bottom half of the Haynes 25 thermal convection capsules increased with time and temperature up to 2½ mils maximum at 1175°F. The corrosion layer spalled off the 1250°F capsules. A mass transfer deposit that increased in amount with time and temperature formed along the top half of the capsules. Very little corrosion of the isothermal capsules occurred. Tensile specimens machined from the Haynes 25 capsules after test were pulled and indicated that the material age hardened after exposure at 1175° and 1250°F.

Small mass transfer deposits occurred along the top half of the Type 405 stainless steel capsules, increasing in amount with time and temperature, but were much smaller than in Haynes 25 capsules. The bottom halves dissolved evenly with no porous layer formed but with surface roughening and intergranular penetration up to 1 mil.

Similar results occurred with AM 350 and 9Cr-1Mo with the former about the same as Haynes 25 and the latter similar to Type 405 stainless steel.

Cb-1Zr exhibited no detectable corrosion.

author

Stress rupture properties of AM 350 in the solution annealed and equalized conditions were measured at 1300°F after aging at different temperatures.

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I. INTRODUCTION

This is a report of materials research in support of the SNAP-8 project covering the period June 1960 through December 1962. It includes work to study liquid mercury corrosion of Haynes 25,* Type 405 stainless steel, AM-350, 9Cr-1Mo, and Cb-1Zr in capsules, and to measure the mechanical properties of AM-350. Concurrent tasks to measure the solubility of elements in Hg and to study the effects of additives on mercury pool boiling heat transfer are reported separately.[1,2,3]

At the start of the SNAP-8 program, information was available[4,5] to make a preliminary selection of alloys from the standpoint of resistance to mercury corrosion. Work was to be based largely on SNAP-2 technology so Haynes 25 was the alloy of prime interest. Early work at General Electric Company[4] showed that the ferritic alloys had good resistance to mercury corrosion below 1000°, so Type 405 stainless steel was also selected for investigation as representative of those alloys and because of minimal welding problems.

In 1962, information from this program and from concurrent programs at NASA-Lewis Research Laboratory and Thompson Ramo Wooldridge indicated

* See Table 2 for nominal compositions of materials.

that Haynes 25 would not be satisfactory for long-time use in the high temperature parts of the SNAP-8 system so work was started with 9Cr-1Mo, AM-350, and Cb-1Zr. 9Cr-1Mo was of interest to replace Haynes 25 as the SNAP-8 reference material because it was the ferritic alloy with the least amount of Cr that would maintain oxidation resistance at 1300°F. AM-350 was indicated to be a promising alloy from capsule testing at Lewis Research Center. Cb-1Zr was indicated to be completely resistant to mercury corrosion at SNAP-8 temperatures and was of interest as a backup.

The objective of the capsule tests was to investigate mass transfer, stress corrosion, mode of corrosion attack, structure and composition of mass transfer products, and change in mechanical properties of the alloys during exposure to Hg between 1025° and 1250°F up to 10,000 hours.

II. TEST PLAN

A summary of the test plan is shown in Table 1. Three types of capsules were made from 5/8 in. OD tubing (see Section III-A for details). All were nearly filled with Hg, and operated with either argon or vacuum in the remaining space. Types A and B were about 11 in. long, were heated on the bottom half and cooled on the top half to induce a thermal convection circulation of the Hg. The Type C capsules were 5 in. long and were operated isothermally to distinguish the effects of circulating and static Hg.

Capsules of Type 405 stainless steel were operated only at 1025° and 1100°F because the creep strength was not adequate to withstand the vapor pressure of Hg at the higher temperatures.

Mass transfer potential was to be measured by observing the amount of corrosion at the hot (bottom) end and the amount of deposition at the cold (top) end. The amount and mode of corrosion was to be determined by micrographic examination and diametral measurements before and after the run.

Stress corrosion effects were to be measured by machining thin walls in some of the capsules (Types B and C). A wide range of stresses resulted due to the reduced wall thickness and different internal pressures at different temperatures.

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TABLE 1 SUMMARY OF CAPSULE TESTS

1025°F

Nominal Test Time, hr	Capsule Number, Type, and Material (1)	Wall Thickness Reduced Section, in.(2)	Estimated Effective Stress, psi (3)	Actual Test Time, hr	Remarks (4)
500	None	-	-	-	-
1000	HDA-16	-	1400	1000	T: Table 3 (5) } H: Table 3, Fig. 15 } P: Table 3
	HDB-22	0.027	2400	1000	
	4BA-6	-	1400	1000	} H: Fig. 32
	4BB-10	0.024	2700	1000	
2000	HDA-17	-	1400	2000	T: Table 3 H: } Table 3, Fig. 15
	HDB-23	0.026	2500	2010	(6) M: Fig. 12 } P: Table 3
	4BA-7	-	1400	2000	} H: Fig. 32
	4BB-11	0.024	2700	2000	
5000	HDA-18	-	1400	5000	(6)(7) } H: Table 3, Fig. 15
	HDB-24	0.025	2600	5000	(8) } P: Table 3
	HDBW-34	0.025	2600	5000	(9).
	HEB-7	0.027	2400	5000	(6)(10) M: Fig. 13; P: Table 3
	4DA-8	-	1400	5000	(11) M: Fig. 27 }
	4BB-12	0.023	2800	5000	} H: Fig. 32
10,000	HDA-15	-	1400	10,000	
	HDB-25	0.030	2200	10,000	
	HDBW-35	0.035	1850	10,000	M: Fig. 14
	HDC-38	0.024	2700	10,000	
	HEA-5	-	1400	10,000	
	HEB-8	0.026	1400	10,000	
	4BA-9	-	1400	10,000	
	4BB-13	0.025	2600	10,000	M: Fig. 28
	4BBW-14	0.033	2000	10,000	
	4BC-15	0.024	2700	Failed	
1250°F					
500	HDB-40	0.023	8000	500	} H: Fig. 26
	HDB-41	0.024	7700	500	(5)(22) T: Table 6
	9AA-70	-	-	500	
	9AA-71	-	-	500	With Cb strip M: Fig. 35
	AAA-50	-	-	500	Solution Annealed } M: Fig. 34
	AAA-51	-	-	500	Equalized
1000	HDA-42	-	750	1000	(6) M: Fig. 24 } H: Fig. 26
	HDB-43	0.024	7700	1000	T: Table 6
	AAA-53	-	-	1000	Equalized
	AAA-54	-	-	1000	Solution Annealed,
2000	HDA-44	-	3750	2000	(6)(23) } H: Fig. 26
	HDB-45	0.024	7700	2000	
	HDBW-46	0.024	7700	2000	(6)(24) M: Fig. 25
	9AA-72	-	-	[illegible]	
	9AA-73	-	-	[illegible]000	With Cb strip
	AAA-55	-	-	2000	Equalized
	AAA-56	-	-	2000	Solution Annealed

1100°F

Nominal Test Time, hr	Capsule Number, Type and Material	Wall Thickness Reduced Section, in.	Estimated Effective Stress, psi	Actual Test Time, hr	Remarks
500	HDA-5	-	1670	500	T: Table 4 } H: Table 4, Fig. 4 } P: None (12)
	HDB-13	0.023	3600	500	
	4BA-60	-	-	500	Cb strip;
	4BA-61	-	-	500	
1000	HDA-4	-	1670	1000	T: Table 4 } H: Table 4, Fig. 20 } P: Table 4
	HDB-10	0.023	3600	1000	
	HEA-2	-	1670	1000	T: Table 4 } H: Fig. 20 } P: Table 4
	HEA-3	-	1670	1000	
	4BB-2	0.022	3700	1525	(13) H: Fig. 33
	AAA-57	-	-	1000	Not evaluated.
2000	HDA-2	-	1670	2000	T: Table 4 (RT) } H: Table 4, Fig. 20
	HDB-26	0.020	4075	2000	(5)(14) T: Table 4 (1100°F) } P: Table 4
	4BA-5	-	1670	2000	H: Fig. 33
	AAA-58	-	-	2000	H: 302Kn (100g) P: 0.0013 in. Equalized
	AAA-59	-	-	2000	H: 326Kn (100g) P: 0.0003 in. Sol'n. Annealed
	4BA-62	-	-	2000	H: 173Kn (100g)
	4BA-63	-	-	2000	(15) H: 180; H: Fig. 21
5000	HDA-19	-	2000	5000	(6)(16) M: Fig. 16 } H: Table 4; Fig. 20
	HDB-27	0.021	4500	5000	(17) } P: Table 4
	HDC-14	0.028	2900	5000	(6)(18) M: Fig. 18
	HEB-9	0.020	2000	5000	(19) M: Fig. 20
	4BA-4	-	1670	5000	M: Fig. 29; H: Fig. 33
10,000	HDA-9	-	1670	10,000	(7) M: Fig. 17
	HDB-8	-	-	10,000	
	HDBW-3	0.022	4300	10,000	(6)
	HDC-11	0.025	3800	10,000	M: Fig. 19
	HEA-6	-	2000	10,000	
	HEB-10	0.020	4750	10,000	
	4BA-1	-	1670	10,000	H: Fig. 33 M: Fig. 30
Indef.	C-3	-	-	2000+	Weld cracked, lesion Na after 2000 hr.
1175°F					
500	HDB-28	0.036	3700	500	} H: Fig. 23
	HDB-34	0.017	7900	500	
1000	HDB-29	0.037	3600	1000	T: Table 5 H: Fig. 23
2000	HDB-31	-	-	-	Failed
	HDB-30	0.036	3700	2000	(6)(20) T: Table 5 M: Fig. 21 }
	HDBW-36	0.037	3600	2000	(21) T: Table 5 } H: Fig. 23
5000	HDA-21	-		5000	
	HDBW-37	0.025	5400	5000	(6) M: Fig. 22 } H: Fig. 23
	HEB-11	0.035	3800	5000	H: Fig. 33
Indef.	C-2	-	-	4300	See Fig. 36

(1) First character = material; H = Haynes 25; 4 = Type 405 stainless steel; 9 = 9Cr-1Mo; A = AM-350; C = Cb-1Zr.
Second character = Lot number (see Table 2)
Third character = Capsule type (see Figure 2)
Number character = order that capsule was cut from tubing lot.
(2) Types B, C, and BW capsule only
(3) Calculated from σ = internal pressure x internal radius/wall thickness
(4) T = tensile test results; h = microhardness results; P = corrosion penetration; M = photomicrograph of wall
(5) X-ray fluorescence analysis of powder in Na: Co, major constituent; decreasing relative amounts of Ni, Cr, Fe, Mn.
(6) Micros taken full length of capsule, not shown.
(7) 0.0385g corrosion product separated from Na.
(8) 0.206g corrosion products separated from Na.
(9) 0.105g corrosion products separated from Na.
(10) 0.037g corrosion products separated from Na.
(11) 0.071g corrosion products separated from Na.
(12) Mass transfer deposits at top of capsule.
(13) Thin section increased 9% in diameter during run.
(14) Crystal structure identified as α-Co by X-ray diffraction. Size analysis of powder 147 particles examined under microscope: 51.7% between 0.025 and 0.125μ; 33.3% between 0.125 and 0.63μ; 6.1% between 0.63 and 1.25μ; 4.8% between 1.25 and 12.5μ; 4.1% between 12.5 and 125μ.
(15) Contained columbium strip. Mass transport deposit formed preferentially on the Cb. No corrosion of Cb.
(16) 0.179g corrosion products separated from Na.
(17) 0.025g corrosion products separated from Na.
(18) 0.0001 to 0.0002 in. penetration over length of capsule.
(19) 0.015g corrosion products separated from Na.
(20) 0.050g corrosion products separated from Na.
(21) 0.053g corrosion products separated from Na.
(22) 0.025g corrosion products separated from Na.
(23) 0.006g corrosion products separated from Na.
(24) 0.123g corrosion products separated from Na.

Mechanical property effects were to be determined by measuring the hardness of the tubing and machining tensile specimens from the capsule walls after the runs.

Some mass transfer deposits were to be analyzed by X-ray diffraction and emission spectrography to determine structure and composition.

Haynes 25 and Type 405 stainless steel were replaced as prime SNAP-8 candidates by 9Cr-1Mo and AM-350 part way through the program. It was decided to continue the capsules that were running to their 10,000-hour scheduled shutdown but the amount of analysis was curtailed. Thus some of the objectives, particularly on the longer time capsules, were not completed on those alloys.

The 9Cr-1Mo, AM-350, and Cb-1Zr capsules were all Type A and only qualitative compatibility information was sought.

No tensile or stress rupture data were available for the solution annealed AM-350 at SNAP-8 temperatures so sheet specimens were tested to obtain these data and compared to specimens in the equalized condition.

TABLE 2

MATERIALS DATA

1. Haynes 25 Lot D--5/8 in. O.D. by 0.49 in. wall tubing.

Drawn by Superior Tube Company, heat #L-1720, welded and redrawn, annealed, pickled. Tensile strength: 136,700 psi; yield strength: 70,400 psi; 45% elongation in 2 in.; 20.13% Cr 9.74% Ni, 2.17% Fe, 15.00% W, 1.44% Mn, 0.60% Si, 0.08% C, 0.011% P, 0.010% S, bal Co. Dye penetrant inspected.

2. Haynes 25 Lot E--5/8 in. O.D. by 0.049 in. wall tubing.

Drawn by Haynes Stellite, heat #L-1627, welded and redrawn, annealed. 20.22% Cr, 9.82% Ni, 2.31% Fe, 14.89% W, 1.58% Mn, 0.74% Si, 0.09% C, 0.010% P, 0.011% S, balance Co. No tensile data. Dye penetrant and eddy current inspected.

3. Type 405 stainless steel Lot A--5/8 in. O.D. by 0.049 in. wall tubing.

Drawn by Superior Tube Company, heat #23162X, cold drawn, seamless, annealed. Tensile strength: 65,000 psi; yield strength: 35,300 psi; 39% elongation in 2 in. 12.31% Cr, 0.66% Mn, 0.39% Si, 0.21% Al, 0.04% C, 0.016% P, 0.008% S, balance Fe. Dye penetrant inspected.

4. 9Cr-1Mo--1/2 in. O.D. by 0.050 in. wall tubing

Drawn by Pacific Tube Company, heat #16376, annealed. Tensile strength: 72,150 psi; Yield strength: 42,565 psi. 35% elongation in 2 in.; 8.49% Cr, 1.04% Mo, 0.12% C, 0.49% Mn, 0.013% P, 0.009% S, 0.36% Si. Flattening, flaring and hydrostatic test satisfactory per ASTM A-199 T-9.

5. AM-350--1/2 in. O.D. by 0.050 in. wall tubing.

Drawn by Trent Tube Company, heat #99118, welded and redrawn, hydrogen annealed. Tensile strength: 142,500 psi; Yield strength: 58,100 psi; 20% elongation in 2 in.; 16.65% Cr, 2.75% Mo, 4.38% Ni, 0.082% C, 0.74% Mn, 0.019% P, 0.016% S, 0.35% Si, 0.089% N_2. 100% eddy current test, flared, 100% dye penetrant tested.

(Table 2 continued)

6. Cb-1Zr--1/2 in. O.D. by 0.49 in. wall tubing.

Supplied by Wah Chang Corporation stress, relieved at 1850°F. 98.75% Cb, 1.15% Zr, 30 ppm C, 94 ppm N, 180 ppm O.

7. Cb--0.06 in. sheet

Supplied by Wah Chang Corporation, heat #9-4432-Cb. Ingot hardness range: 55.1-63.3 BHN. Ingot chemistry (ppm):

Element	Top (ppm)	Element	Top (ppm)
Al	35	B	<1
C	<30	Cd	<5
Cr	<20	Cu	<40
Fe	2100	H_2	5
Hf	<80	Mg	<20
Mn	<20	Mo	<20
N_2	56	Ni	<20
O_2	<50	Pb	<20
Si	140	Sn	<20
Ta	<500	Ti	<150
V	<20	W	<300
Zn	<20	Zr	<500

III. APPARATUS AND EXPERIMENTAL PROCEDURES

A. CAPSULE DESIGN AND FABRICATION

The principle of the capsule design and test method is illustrated in Figure 1. A furnace (described in Section B) contained two 6 in. dia. x 6 in. long blocks, each with 19 holes and separated by insulation. The bottom block was enclosed in clam shell heaters and the top block was unheated. Capsules were mounted in the holes so that the bottom half was heated by the bottom block. The ΔT between top and bottom of capsule varied between 80° and 150°F depending on the nominal temperature, the type of material, and the length of time since start up. This situation created a natural convection flow pattern as shown in Figure 1. The liquid being heated at the bottom of the capsule ascended next to the wall and descended at the axis. The wall along the bottom half of the capsule dissolved into the rising film of liquid. When this film reached the upper half and cooled, the liquid became supersaturated and the solute precipitated to form a deposit on the wall or a powder in the liquid. By keeping all capsules under a similar Δ T, it was expected that relative differences between alloys could be measured.

This flow system had been studied by Hartnett, Welch, and Larson[6] at a length-to-diameter ratio of 21. Heat transfer had been correlated against a modified Grashof-Prantl number. The Types A and B capsules were selected with this same ratio because it was hoped that

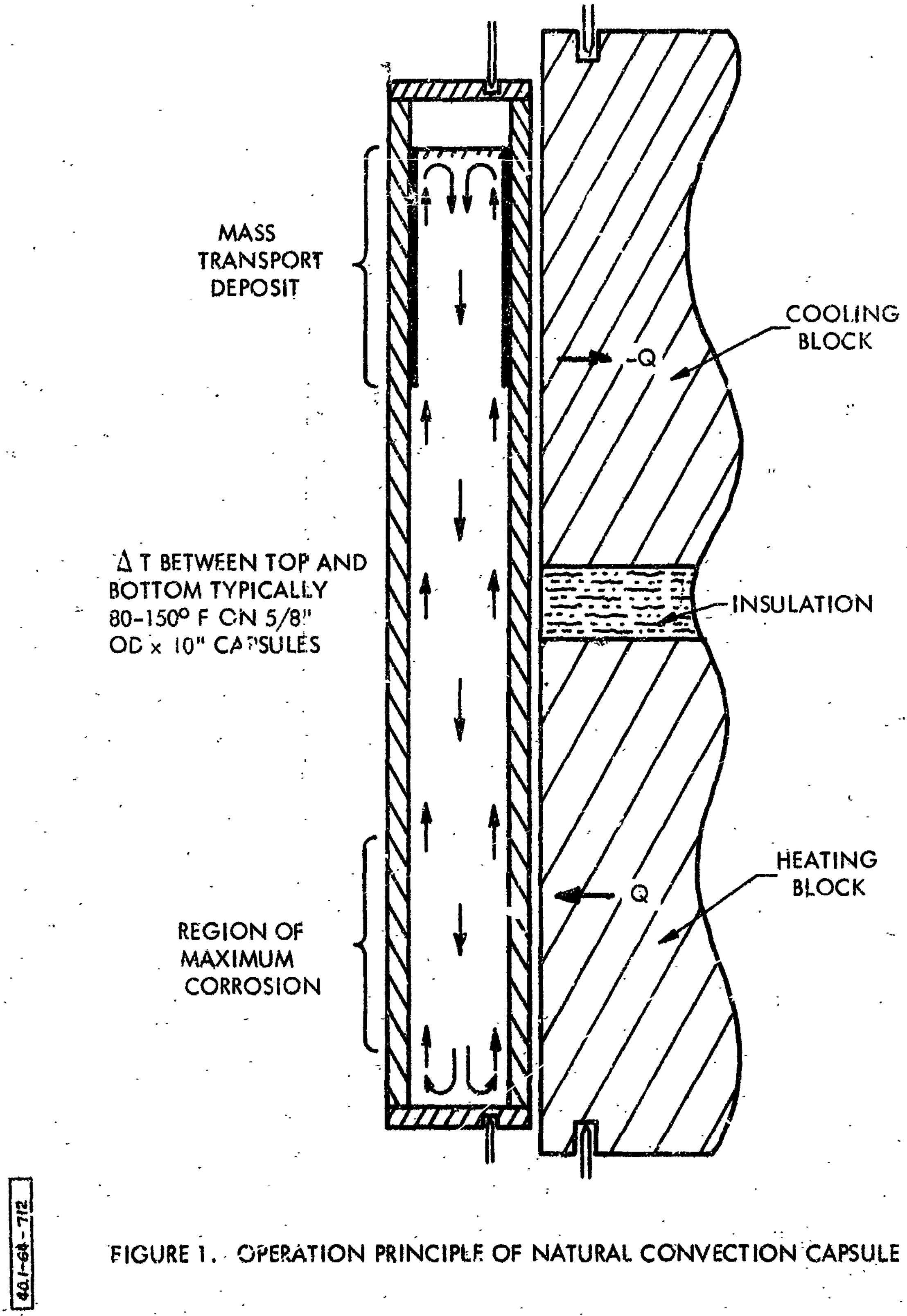


FIGURE 1. OPERATION PRINCIPLE OF NATURAL CONVECTION CAPSULE

further analysis of the system would result in an analogous correlation of mass transfer. A somewhat simpler system had been correlated in both natural and forced convection.[7] The analysis was eliminated during later changes in scope of the program so no attempt at correlation was made.

To provide further evidence that the proposed mechanism was responsible for the corrosion and mass transfer observed, short capsules were designed to operate completely within the bottom block and thus operate isothermally. It was expected that little or no corrosion would be shown in these capsules because of the lack of a driving force.

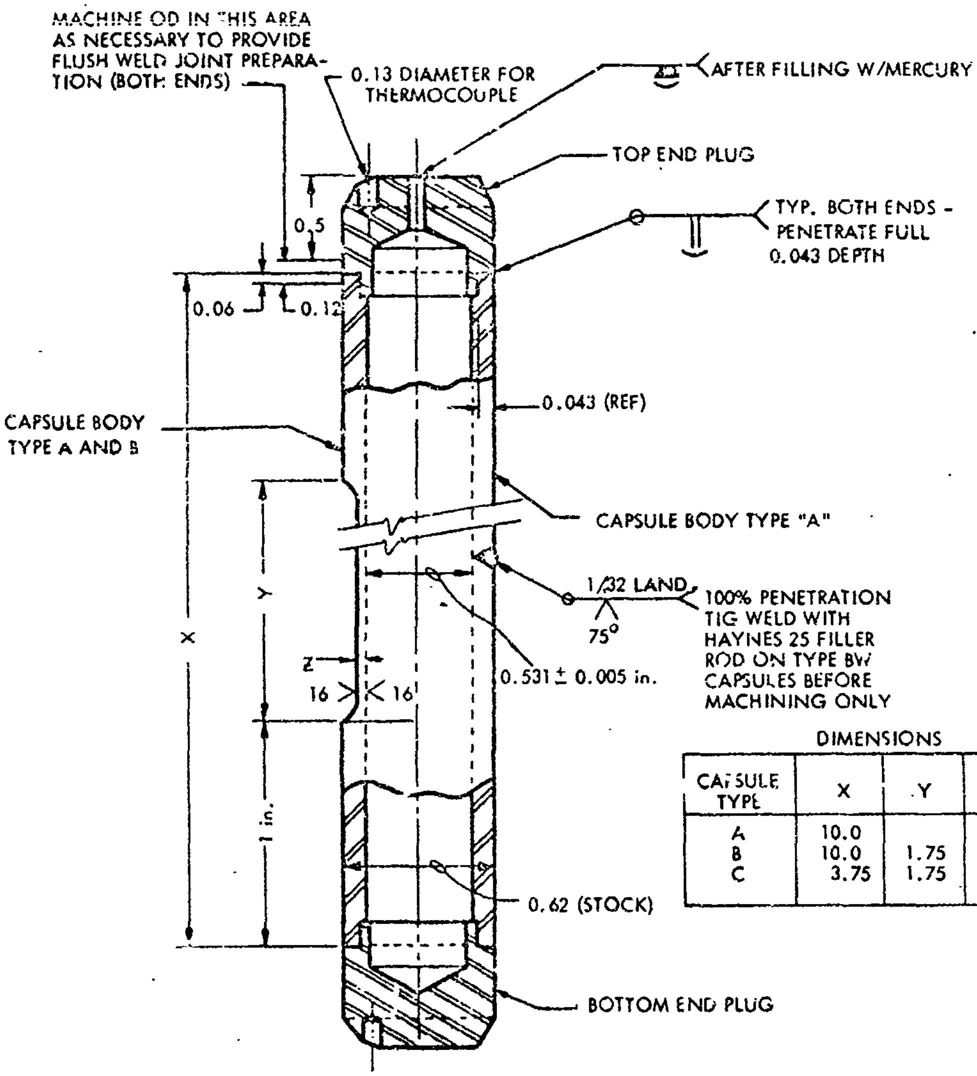
1. Haynes 25 and Type 405 Stainless Steel

Three types of capsules were used, each equipped with two types of closures depending upon whether the capsule was sealed under vacuum or in argon. Figure 2 is a composite drawing of the three capsule types with the modification for filling in argon. A set of final machined capsules with modification for filling under vacuum is shown in Figure 3. For filling under vacuum, a 1/4-in. tube is extended from the top to facilitate sealing, described below. The first 20 capsules were vacuum filled. The change to argon-filled capsules was made when it was determined that mass transfer results might be affected by a small amount of mercury refluxing in the vacuum space above the liquid.

The capsules consisted of two end plugs and a body (and the fill tube for the capsules sealed under vacuum). As-received tubing was used for Type A bodies. The inner surface of B, C, and BW type bodies was honed and a thin section was machined in the outer surface of the tube wall so that the desired stress would exist in the wall during operation. The BW capsules were the same as Type B, except that the tubing was cut and welded at the center of the thin section before the machining was done. Thus, the weld was stressed the same as the thin section.

Two lots of Haynes 25, having significantly different characteristics (Table 2), were used.

The capsules were filled with triple distilled mercury to a level calculated to maintain the liquid surface just above the top weld. Discoloration and the corrosion pattern after the runs indicated that this level was obtained.



DIMENSIONS

CAPSULE TYPE	X	Y	Z
A	10.0		SEE TABLE 1
B	10.0	1.75	
C	3.75	1.75	

FIGURE 2. CAPSULE DESIGN

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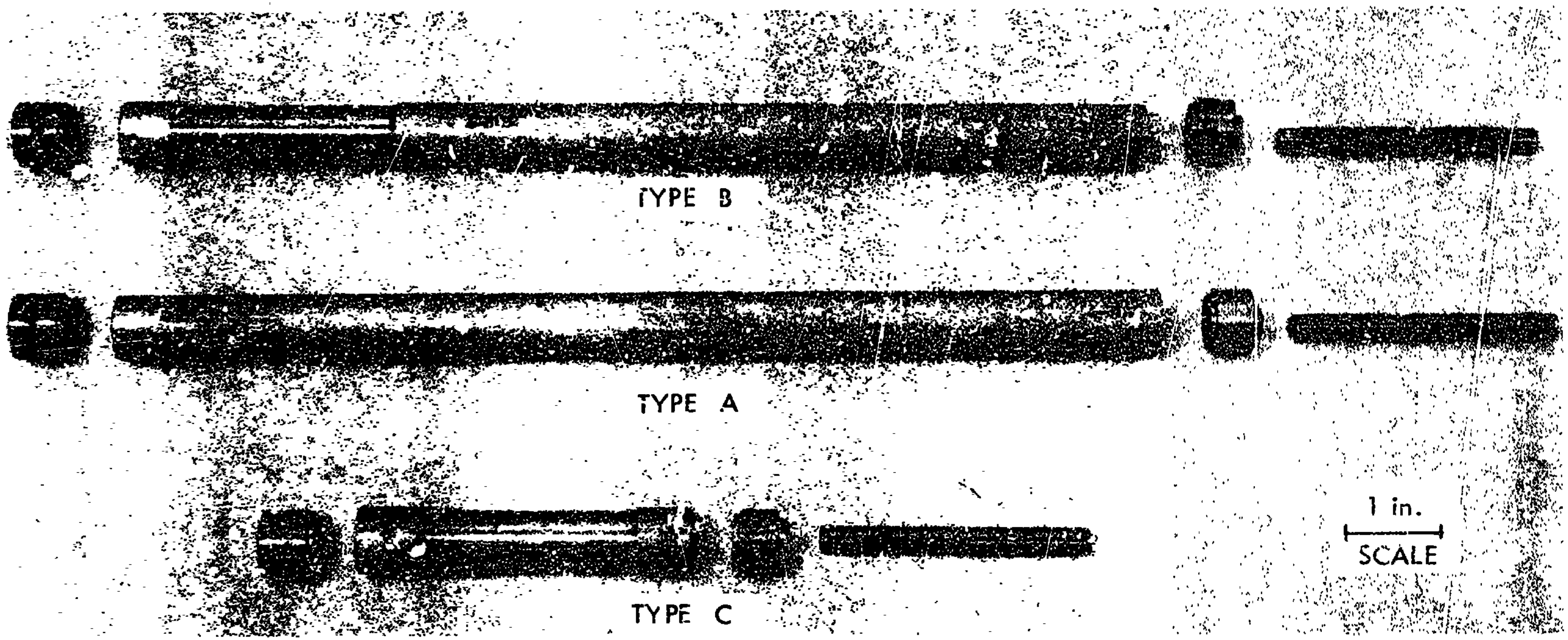


FIGURE 3. FINAL MACHINED CAPSULE PARTS

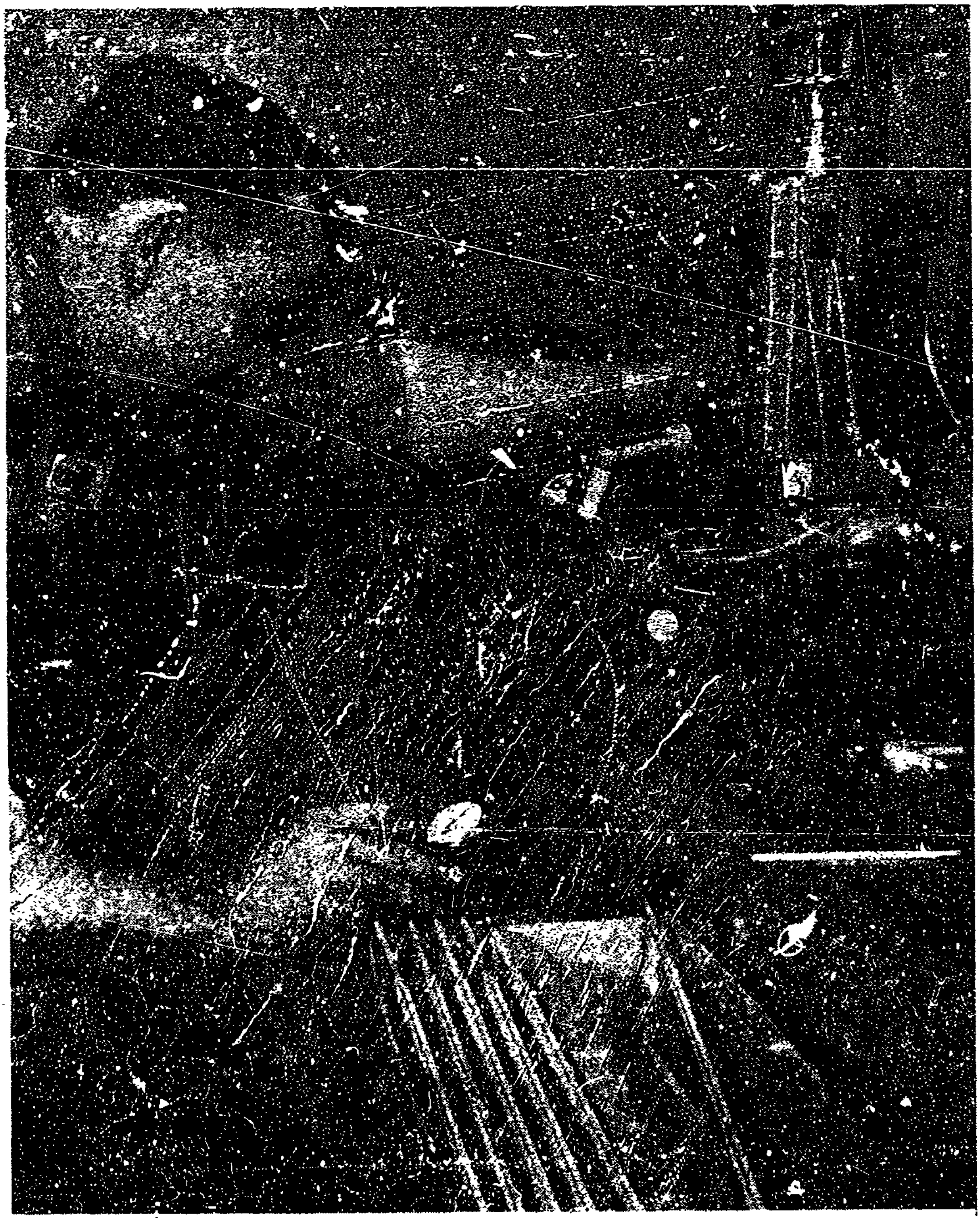


FIGURE 4. SEMIAUTOMATIC WELDING OF CAPSULE

Capsules were fabricated and prepared as follows:

1) Cut lengths of tubing from different lots, as shown in Table 1. Number the lengths at random and record to note original location from tubing. Cut short samples with each length for pre-test analysis or reference, if needed after the run.

2) Rough machine parts.

3) Final machine parts.

4) Measure diameters and wall thicknesses of bodies.

5) Degrease with trichlorethylene.

6) TIG weld with argon purge inside the capsule as shown in Figure 4.

7) X-ray welds.

8) Etch Type 405 stainless in 20% HNO_3, 4% HF for 3 minutes at room temperature. Etch Haynes 25 the same way except at 85°C.

9) Fill in air with triple distilled Hg.

Vacuum Type

10) Evacuate until pressure of non-condensible gases is below 1×10^{-4} torr (one to two days) (Figure 5a).

11) Pinch the small fill tube with a hydraulic C-clamp (Figure 5b). Preliminary tests established the force necessary to form a vacuum tight seal.

12) Remove the capsule from the evacuation facility and saw off the excess tube (Figure 5c) while holding the vacuum with the C-clamp.

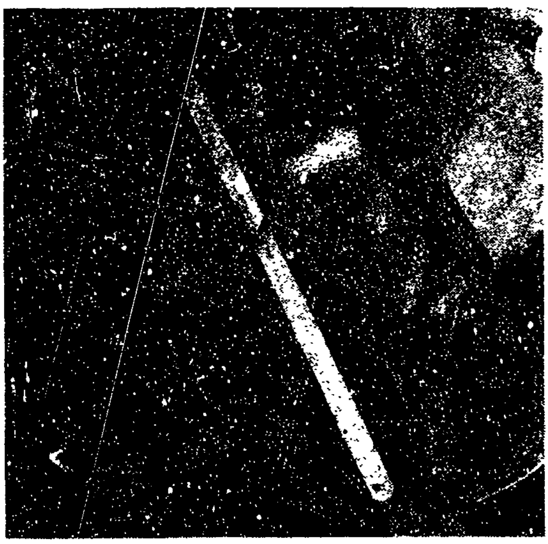
13) Weld across the end of the tube to seal the capsule. (Figure 5d) then release the C-clamp.

Argon Type

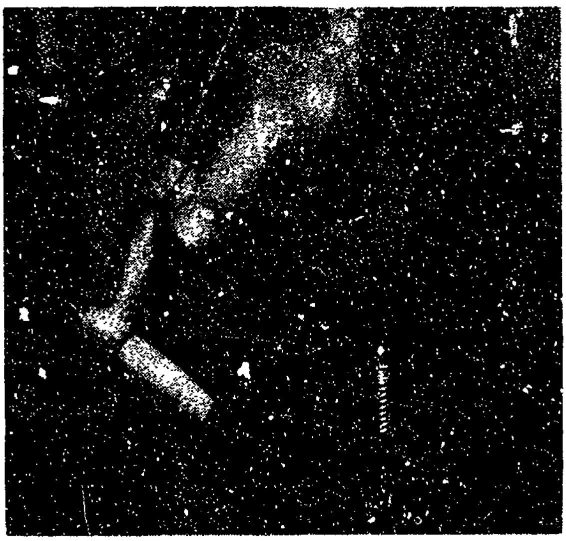
10) Place capsules in vacuum glove box and evacuate overnight.

a. EVACUATING CAPSULES

b. PINCHING FILL TUBE



c. CUTTING OFF FILL TUBE



d. WELDING FILL TUBE

FIGURE 5. EVACUATING AND SEALING VACUUM TYPE CAPSULES

11) Refill the glove box with argon and seal the capsules in the glove box with a weld bead over the small hole in the top (Figure 2).

2. Cb-1Zr

Simple A-type capsules were made from 5/8 in. OD x 1/8 in. wall tubing and closed with flat discs made from flattened tubing. They were filled and welded in the argon glove box.

3) AM-350 and 9Cr-1Mo

Simple A-type capsules were made from 5/8 in. tubing with barstock used for end plugs. Some capsules contained columbium strips as shown in Table 1. The capsules were depicted in some of the figures showing results; e.g., Figure 31.

B. FURNACE DESIGN AND OPERATION

Three furnaces, as shown in Figures 6 and 7, were used to contain the capsules. A furnace consisted of a cold wall pressure vessel containing a 6 in. long mild steel block surrounded by an Arc-O-Vac heater coiled into a helix. Another block was mounted directly above the heating block and was separated from it by 1/2 in. of insulation. The remainder of the furnace was filled with foamed silica insulation. The blocks contained 19 vertical holes in which the capsules were mounted.

The furnaces could be operated up to 295 psia pressure or under vacuum. All but short periods were conducted under vacuum less than 1 x 10^{-3} torr using a fore pump and dry ice/acetone cold trap.

The temperatures were measured with Aero Research 1/16 in. OD, MgO-insulated stainless steel sheathed Chromel-Alumel thermocouples. Conax glands were used for heater and thermocouple feed throughs. The higher temperatures also tended to result in a smaller ΔT. Thermocouples were mounted in various locations in the blocks and in the holes at both ends of 2 capsules at opposite sides of the block. Temperatures were uniform radially and the gradient between top and bottom of the capsules varied between 85 and 150°F. The tests were interrupted at 500 or 1000-hour intervals to remove capsules, take diametral measurements, or start new capsules. The wider gradient occurred at the start of each interval and narrowed after

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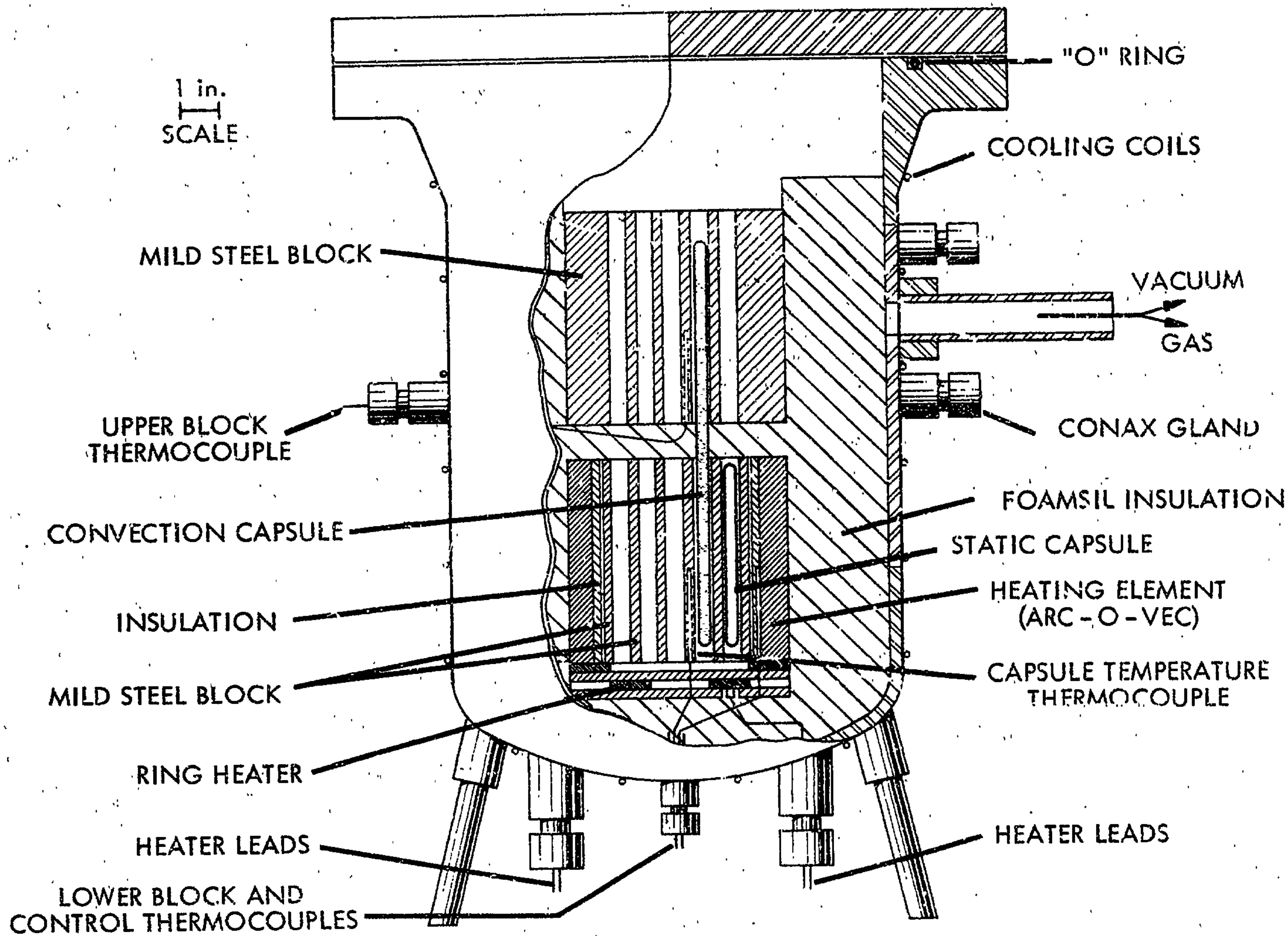


FIGURE 6. CORROSION CAPSULE FURNACE

40.1-64-717

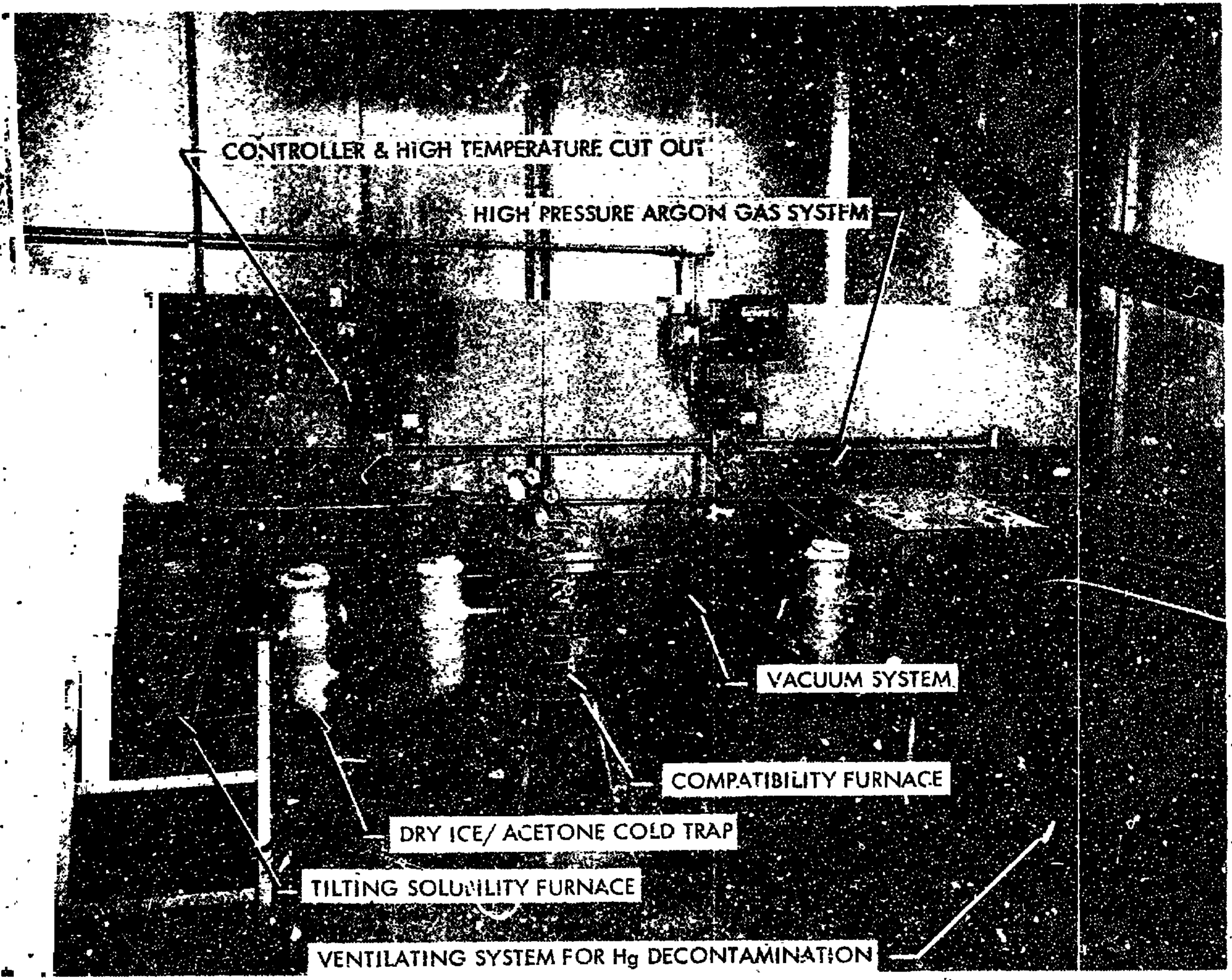


FIGURE 7. CAPSULE FURNACE FACILITY

several days operation. The bottom of the capsule remained at constant temperature and the top became hotter during the period, therefore, the nominal temperatures listed in Table 1 apply to that part of the capsule undergoing dissolution.

The temperature was controlled by Wheelco Model 402 controllers using a thermocouple placed between the heating block and the heater.

The furnaces originally were built with a copper heating block and an aluminum cooling block to take advantage of their high thermal conductivities. A capsule leaked Hg early in the 1100°F run, forming a low melting eutectic with the Cu and Al and destroying some of the capsules. The blocks were then replaced with the mild steel blocks.

The columbium capsules were wrapped in zirconium foil to minimize reaction with impurities in the furnace atmosphere during the run. No visible decontamination of the Cb occurred during the runs.

C. TEST METHODS

1. Capsule Decontamination and Sampling

Completed capsules were handled as follows:

1) Open by sawing at the top until a small hole is made.
2) Pour Hg into large test tube and save.
3) Cut off ends of capsule.
4) Evaporate Hg from capsule bodies by molecular distillation (~ 200°F) in the apparatus shown in Figure 8.
5) Evaporate Hg from test tube in the same apparatus (certain capsules only).
6) Recover the loose powder from Steps 4) and 5) and weigh.
7) Measure capsule diameters.
8) Cut samples for micrographic examination.
9) Cut sample for tensile test (certain capsules only).

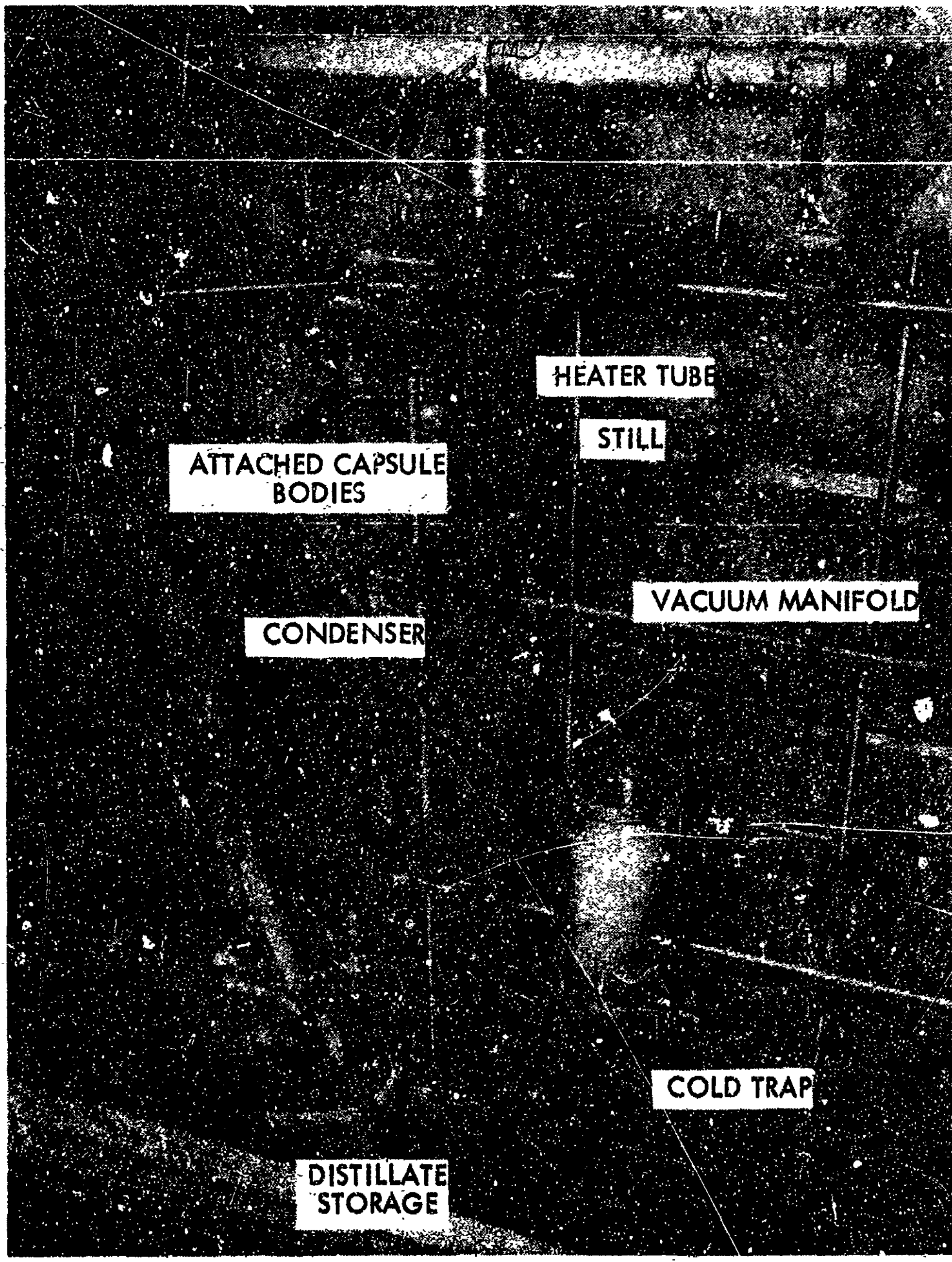


FIGURE 8. CAPSULE DECONTAMINATION STILL

40.1-64-718

2. Capsule Diameter Measurements

The inside and outside diameters and wall thicknesses of the Haynes 25 and Type 405 stainless steel capsules were measured with a micrometer along 2 diameters. Only one capsule exhibited any significant change in diameter while meaurements were made (2000-hr capsules and under) so no data will be shown. One Type 405 stainless steel capsule (No. 4BB-2) expanded about 9%, mostly during a short temperature excursion, so was removed from test.

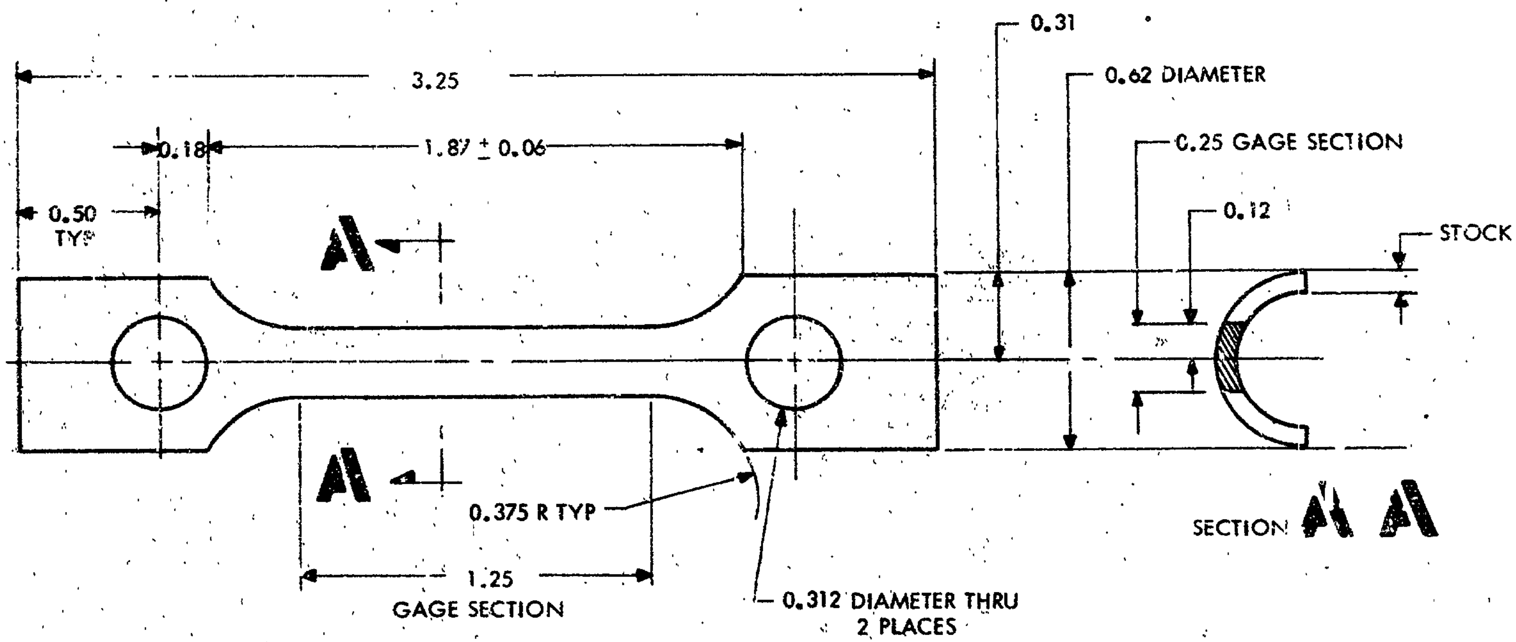
3. Hardness Measurements

It was known that Haynes 25 would age harden during exposure to temperatures in the SNAP-8 range. It was hoped that some correlation between microhardness and elongation during a tensile test could be found so that future testing could be simplified. A Kentron Microhardness Tester was used to make indentations in the micrographic samples. Readings were taken at 120° intervals around the tubing. Impressions were taken at distances from the inside and outside of the tubing as shown in figures summarizing the results. The average of the 3 impressions at similar distances was used to obtain the values plotted.

4. Tensile Testing

It was decided to measure the effects of exposure on the Haynes 25 and Type 405 stainless steel by machining tensile specimens from the walls of the tested capsules rather than use inserts. In this way the effect of stress history would be included. This required a special specimen, as shown in Figure 9. Special grips were also fabricated. Those used for room temperature tests are shown in Figure 10 and those for elevated tests in Figure 11. Tests were performed on as-received tubing to check out the grips and to obtain reference data.

401-64-719



DIMENSIONS IN INCHES

FIGURE 9. TENSILE SPECIMEN MACHINED FROM CAPSULE WALL

401-64-720

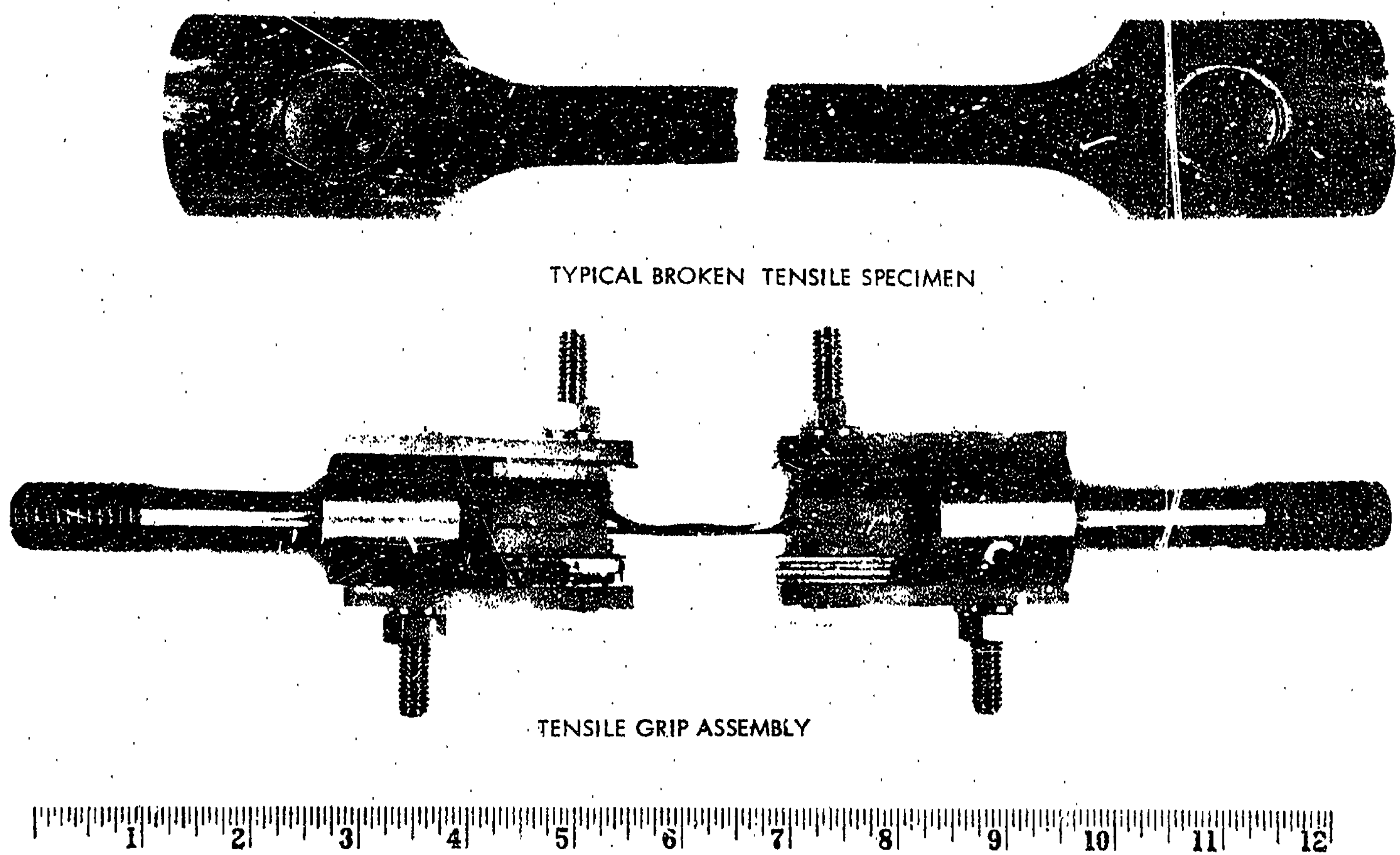


FIGURE 10. TENSILE SPECIMENS AND GRIPS USED FOR ROOM TEMPERATURE TESTING

40.1-64-721

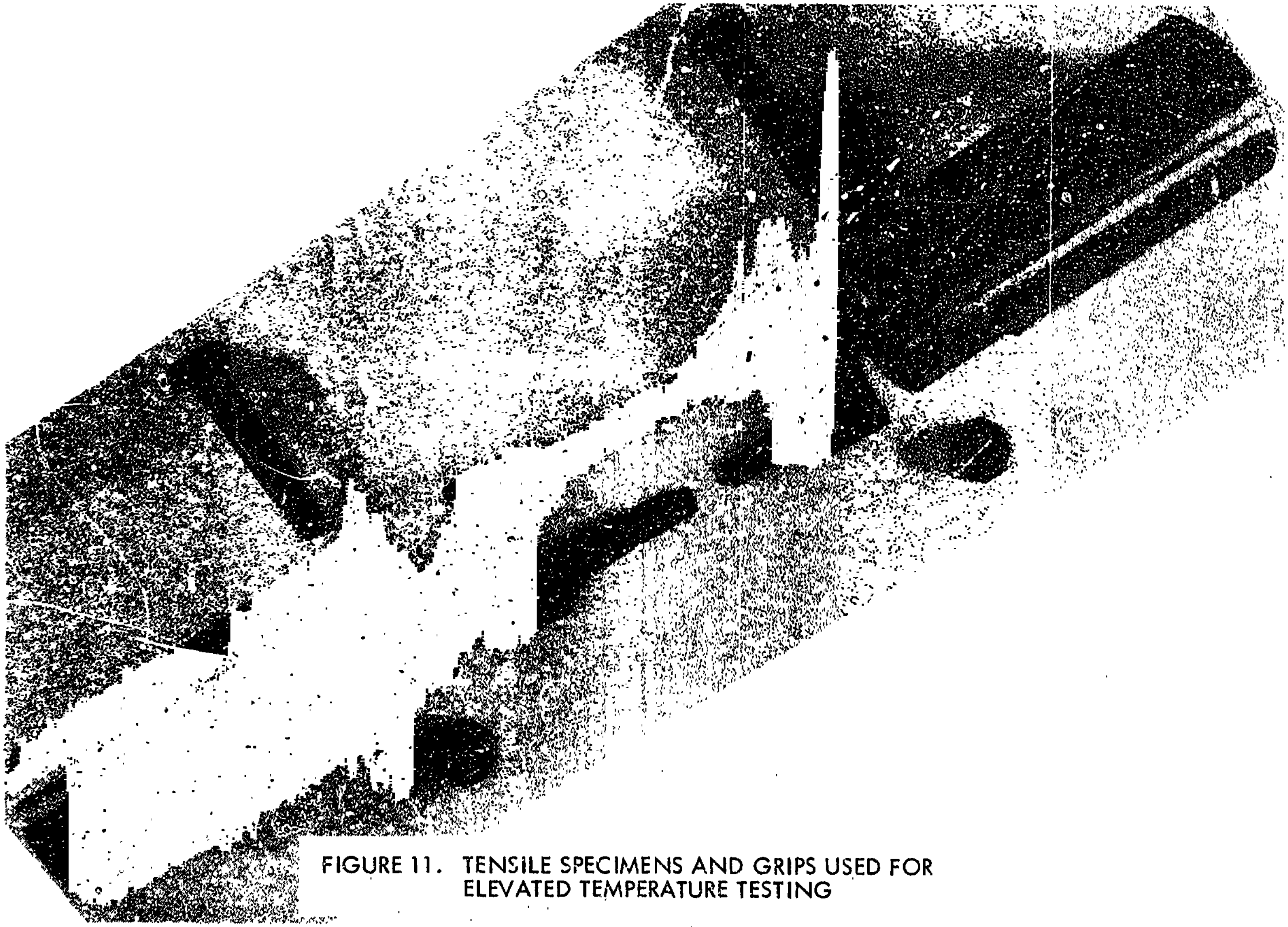


FIGURE 11. TENSILE SPECIMENS AND GRIPS USED FOR ELEVATED TEMPERATURE TESTING

IV. RESULTS

The actual lengths of time that capsules operated were listed in Table 1. Representative data will be shown in the following pages under divisions of time and temperature. In general, duplicate capsules had nearly identical results so are not shown. Data are not shown on welds since no effect different from the base metal was noted. Photomicrographs are not shown on some of the shorter time capsules; their appearance was identical to the longer time capsules except for degree.

Haynes 25, Type 405 stainless steel, 9Cr-1Mo, and AM-350 were etched with Glycergia for the photomicrographs. Cb-1Zr was etched with the following solution:

5 ml H_2O
25 ml HF
10 ml H_2SO_4
10 ml HNO_3

Immerse 20 to 30 sec, then polish. Re-immerse for shorter times and repolish as necessary.

A. HAYNES ALLOY NO. 25

1. Capsules Exposed at 1025°F

a. Capsule No. HDB-23 - 2000 Hours at 1025°F

Corrosion effects were found along the full length of the capsule (Figure 12). An evenly depleted layer was found along the bottom half of the capsule, averaging approximately 0.0006 in. deep. A mass transfer deposit was found in a layer of variable density along the top half, extending to the liquid surface. The two layers merged abruptly at the point where the bottom and top positioning blocks met in the furnace. This point corresponded to a sharp change in the temperature gradient along the capsule. The mass transfer deposit was most dense about two inches from the top weld. The largest crystals are shown to be about 0.001 in. at their longest dimension. The composition of the crystals has not been determined, but since the loose powder found in the Hg was predominantly Co (as discussed later), it is believed they are primarily solid solutions of Ni and Cr.

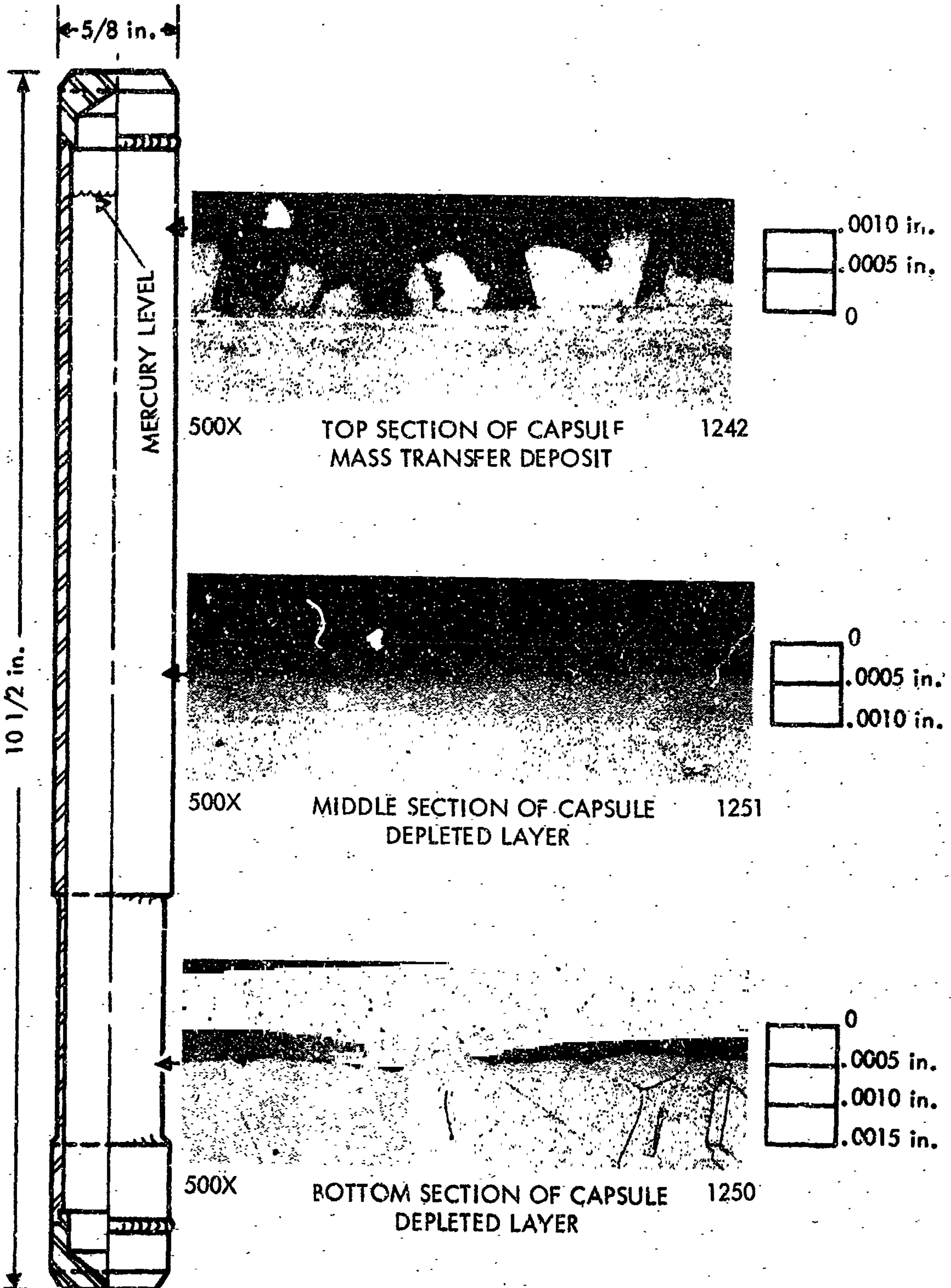
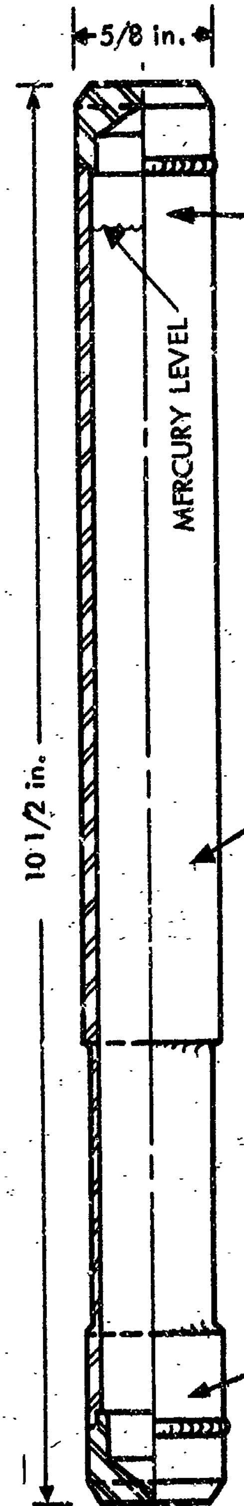


FIGURE 12. HAYNES 25 CAPSULE NO. HDB-23 TESTED AT 1025°F FOR 2000 HOURS

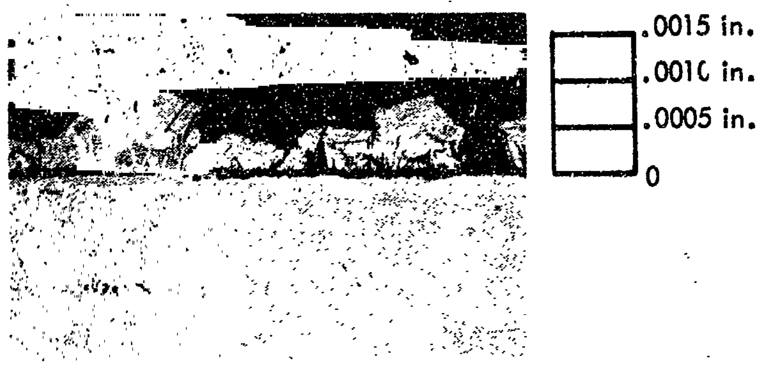
4A1-64-722

b. Capsule No. HEB-7 - 5000 Hours at 1025°F

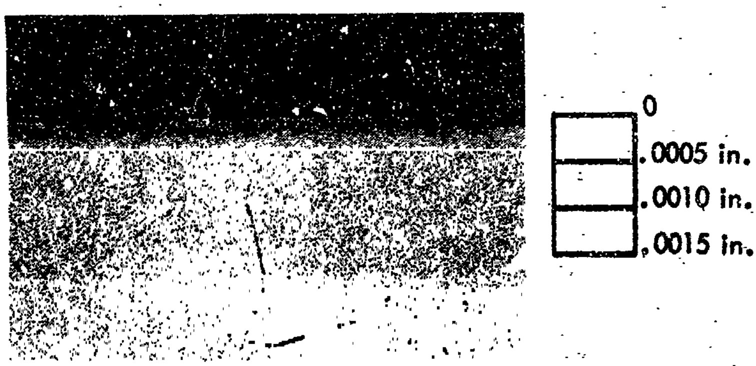
Corrosion effects were found along the entire length of the capsule (Figure 13). An even, depleted layer approximately 0.0015 in. deep, extended along the bottom part of the capsule. The depleted layer tapered off until it disappeared about 1 in. below the start of the mass transfer deposit The mass transfer deposit extended to within 2 in. of the top weld in the capsule. No evidence of spalling of the depleted layer could be found.



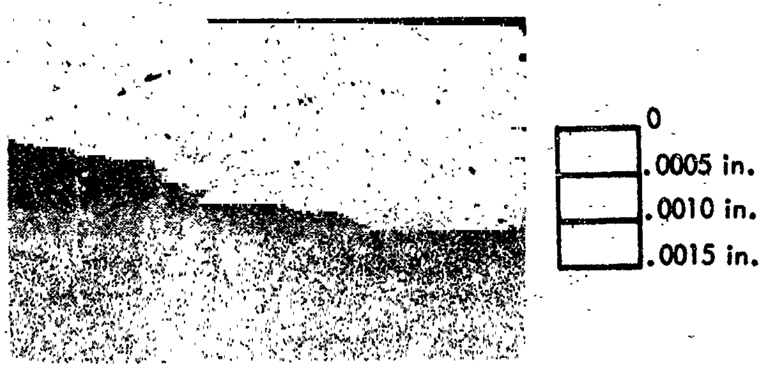
COMPATIBILITY CAPSULE



500X TOP SECTION OF CAPSULE 1836
MASS TRANSFER DEPOSIT



500X MIDDLE SECTION OF CAPSULE 1839
DEPLETED LAYER



500X BOTTOM SECTION OF CAPSULE 1841
DEPLETED LAYER

FIGURE 13. HAYNES 25 CAPSULE NO. HEB-7 TESTED AT 1025°F FOR 5000 HOURS

401-84-783

c. Capsule No. HDBW-35- 10,000 Hours at 1025°F

Examination of the microstructure along the entire length of the capsule showed a typical depleted surface layer of the tube wall at the bottom of the capsule. A mass transfer deposit was found at the top of the capsule. Typical microstructures are shown in Figure 14. The depleted layer was approximately 0.002 in. deep. No spalling of the depleted layer was observed.

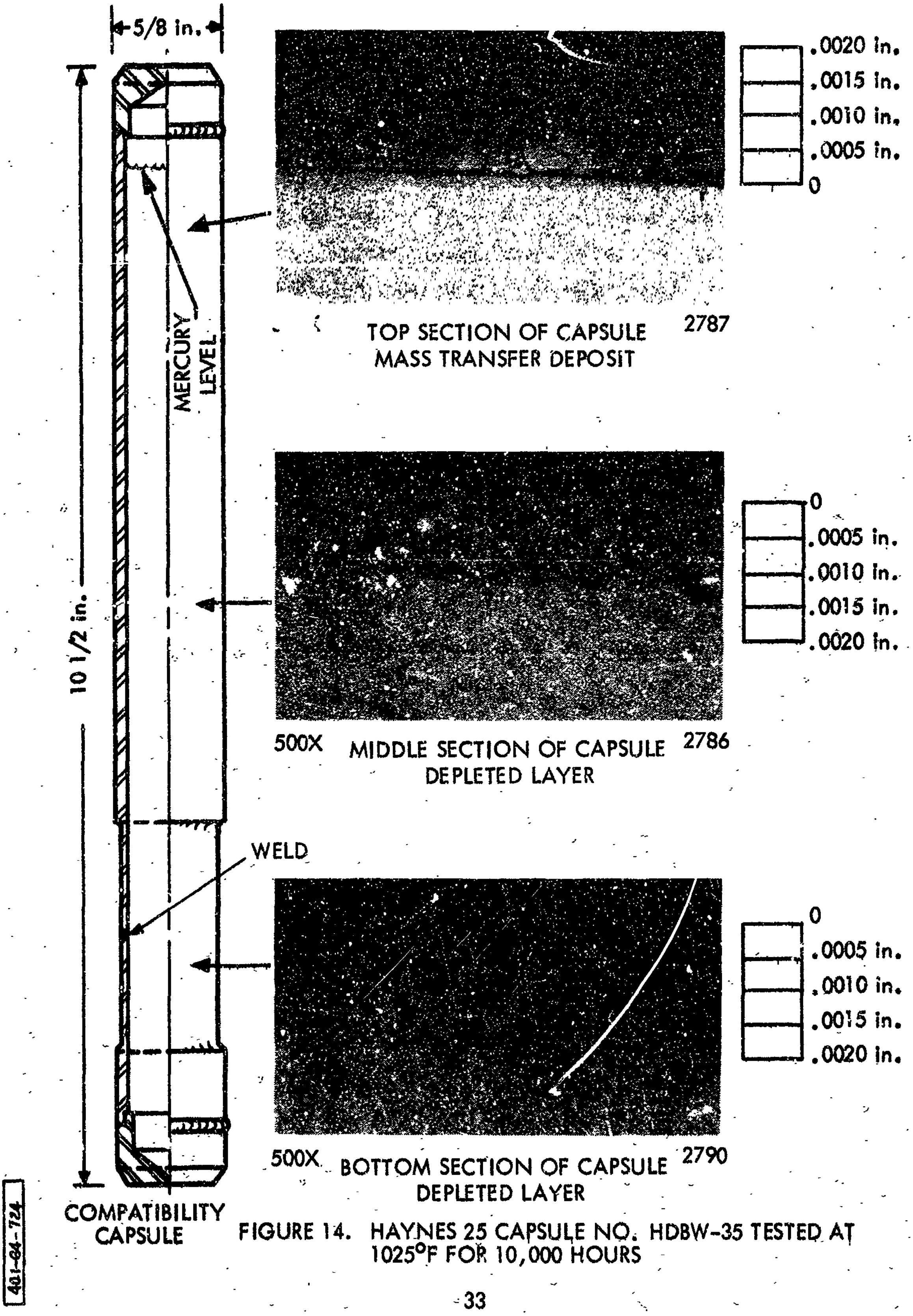


FIGURE 14. HAYNES 25 CAPSULE NO. HDBW-35 TESTED AT 1025°F FOR 10,000 HOURS

d. Summary of Results of Haynes 25 Tested at 1025°F

The microhardness of the capsule material decreased to 339 Knoop (100 gram load) from the original hardness (408 Knoop) of the Haynes 25 tubing used to make the capsule bodies. A microhardness survey of the Haynes 25 capsules exposed at 1025°F is shown in Figure 15. Table 3 summarizes the results from the 1025°F capsules.

TABLE 3 - SUMMARY OF TEST RESULTS FROM HAYNES 25 COMPATIBILITY CAPSULES EXPOSED AT 1025°F

	Room Temperature Tensile Test				
Time, hr	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 in., %	Room Temperature Knoop Hardness with 100 gm load	Maximum Penetration of Depleted Layer, in.
0	138.6	70.3	38 (1)	408	none
1000	149	78.2	44 (2)	354	0.0003
2000	150	81.0	61 (2)	385	0.0006
5000	143.4	79.7	45 (1)	380	0.0015
10,000	---	---	---	339	0.002

(1) Average of three tests

(2) One test

No elevated temperature tensile tests were made of 1025°F compatibility capsules.

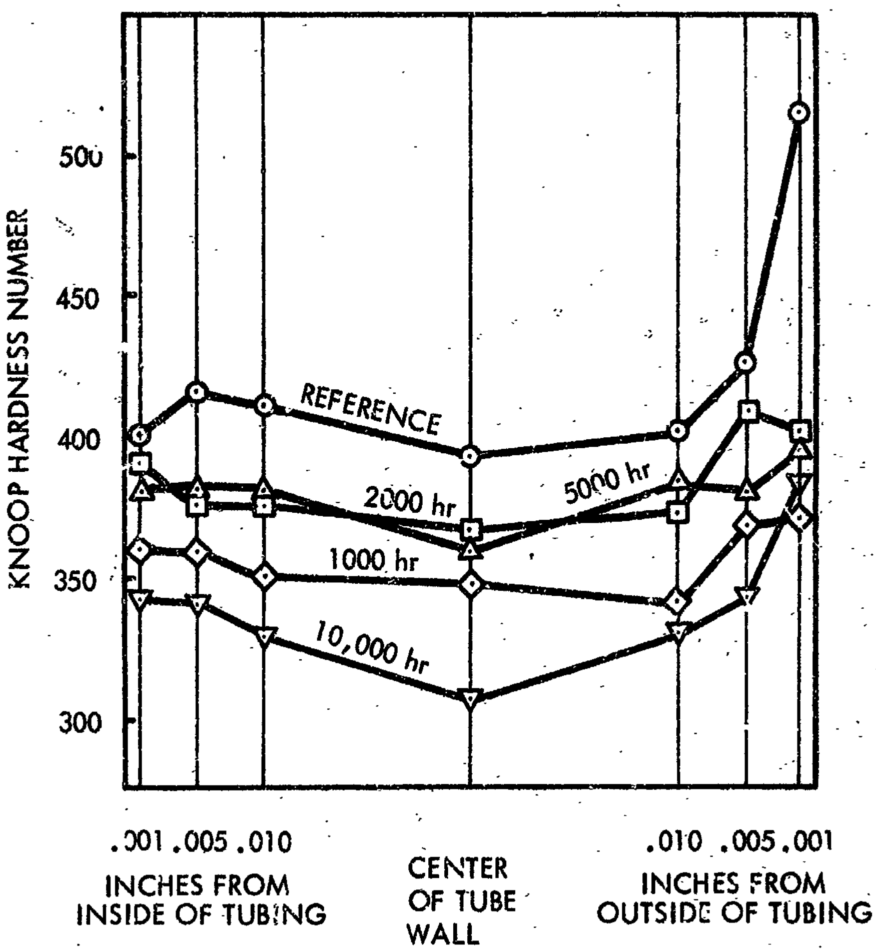


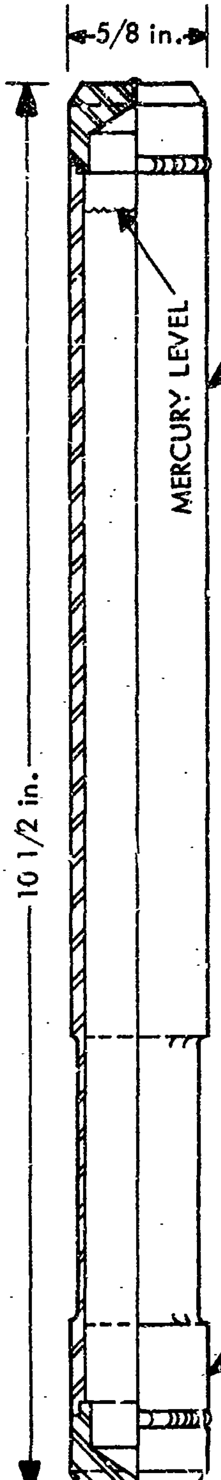
FIGURE 15. MICROHARDNESS SURVEY, HAYNES 25 LOT D EXPOSED TO MERCURY AT 1025°F

401-64-725

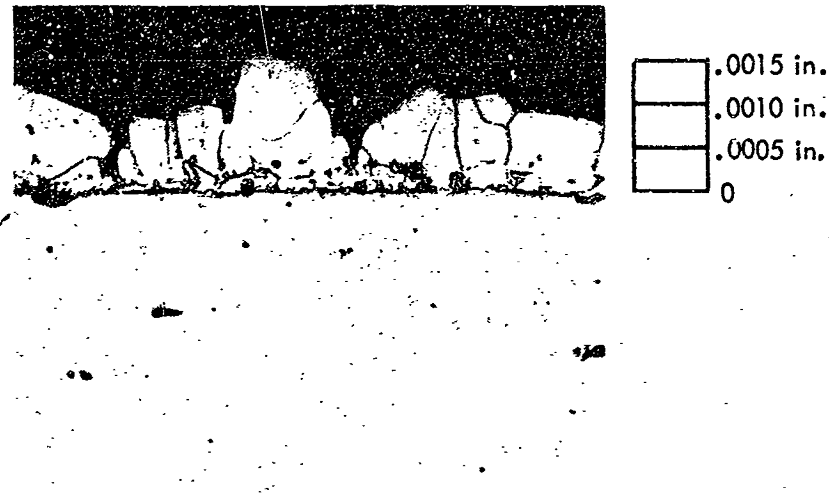
2. Capsules Exposed at 1100°F

a. Capsule No. HDA-19 - 5000 Hours at 1100°F

This capsule showed the same corrosion pattern found in the 5000 hour, 1025°F capsule. The depleted layer was approximately 0.0013 in. deep and extended to the middle of the capsule. No evidence of spalling of the depleted layer was found. The mass transfer deposit formed near the top of the capsule built up to 0.0015 in. maximum. Typical sections of the top and bottom portions of the capsule are shown in Figure 16.

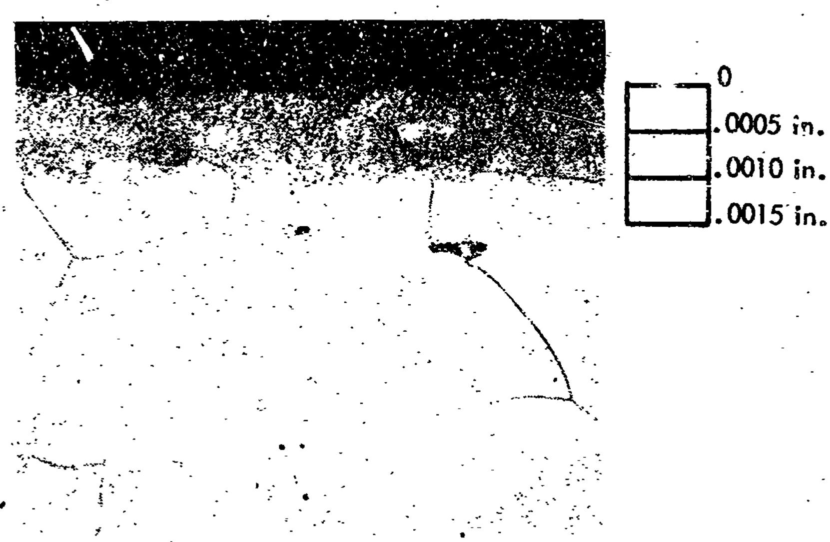


COMPATIBILITY CAPSULE



500X TOP SECTION OF CAPSULE 1943

MASS TRANSFER DEPOSIT



500X BOTTOM SECTION OF CAPSULE 1946

DEPLETED LAYER

FIGURE 16. HAYNES 25 CAPSULE NO. HDA-19 TESTED AT 1100°F FOR 5000 HOURS

40.1-64-726

b. Capsule No. HDA-9 -10,000 Hours at 1100°F

Corrosion effects were found along the entire length of the capsule. A depleted layer was found along the bottom section of the capsule approximately 0.002 in. deep, maximum. The depleted layer tapered off to a very thin layer, but it extended under the mass transfer deposit found at the top of the capsule. No evidence of spalling of the depleted layer is evident. Typical sections are shown in Figure 17.

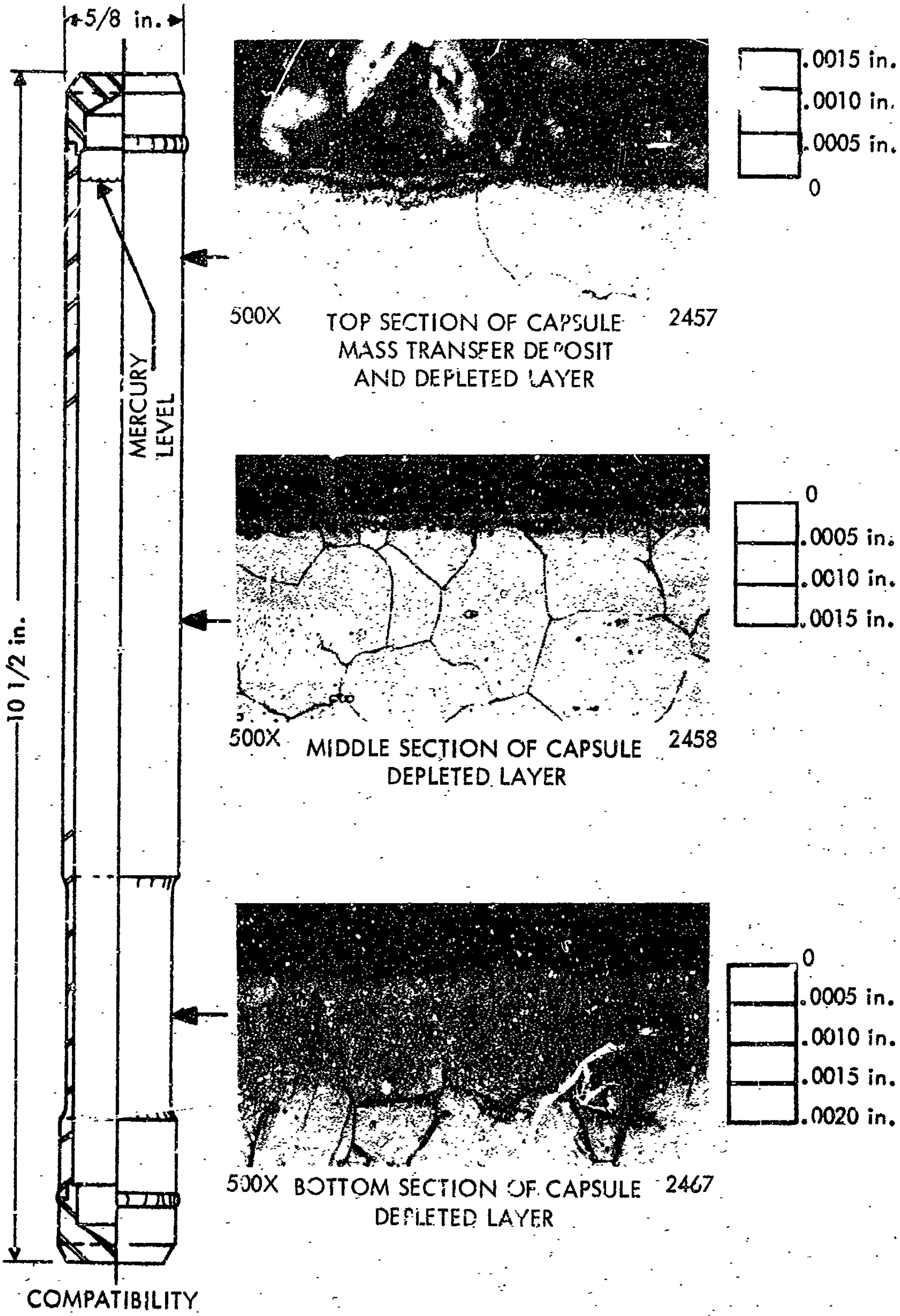


FIGURE 17. HAYNES 25 CAPSULE NO. HDA-9 TESTED AT 1100°F FOR 10,000 HOURS

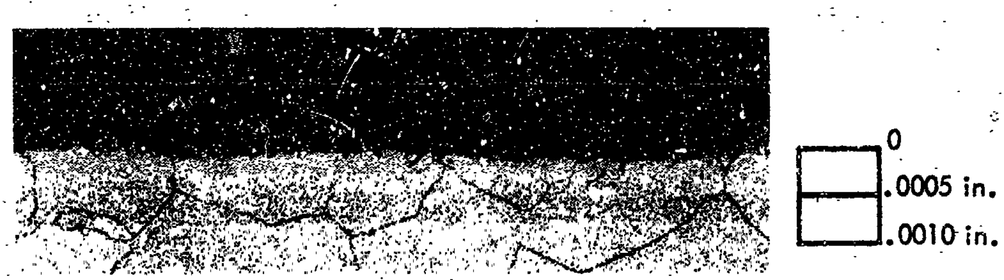
441-64-727

c. Capsule No. HDC-14 - 5000 Hours at 1100°F

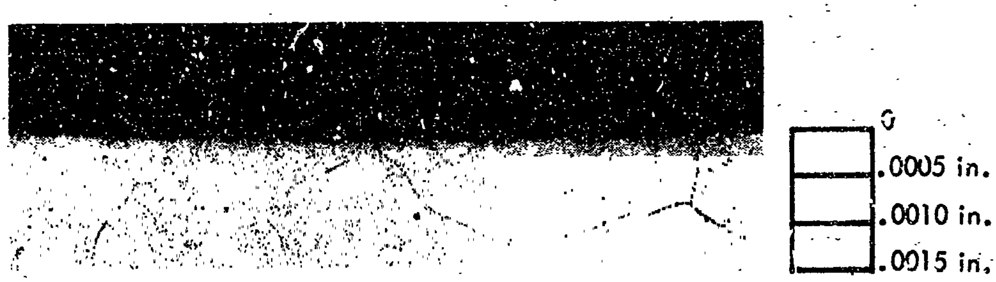
This capsule and a similar 10,000 hour capsule operated under nearly isothermal conditions.

A depleted layer varying from 0.00013 to 0.0003 in. thick was found along the length of the 5000 hour isothermal capsule. No correlation between depth and position along the capsule was noted. The elements leached from the depleted layer apparently precipitated in the Hg and no mass transfer deposit was formed. Photomicrographs of the top, middle, and bottom sections of the capsule are shown in Figure 18.

500X TOP SECTION OF CAPSULE 1452
DEPLETED LAYER



500X MIDDLE SECTION OF CAPSULE 1453
DEPLETED LAYER



500X BOTTOM SECTION OF CAPSULE 1454
DEPLETED LAYER

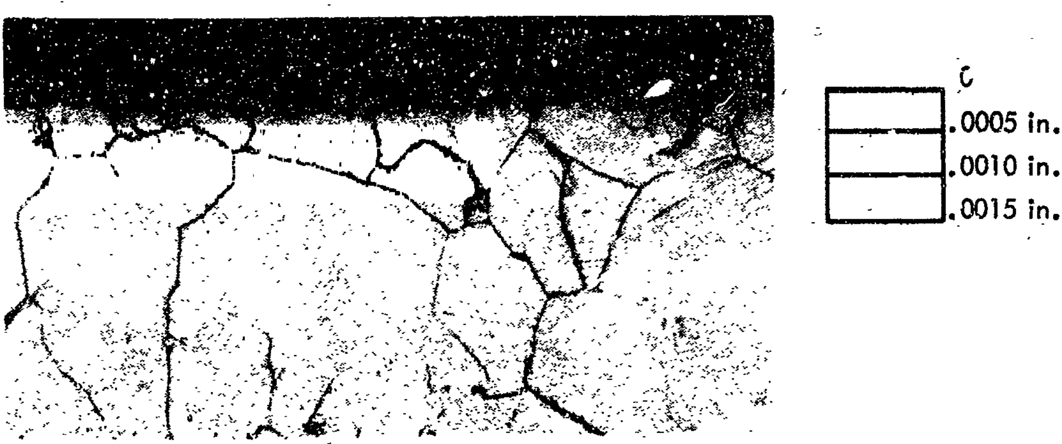
FIGURE 18. HAYNES 25 CAPSULE NO. HDC-14 TESTED AT 1100°F FOR 5000 HOURS

401-64-728

d. Capsule No. HDC-11 - 10,000 Hours at 1100°F

A very thin depleted layer was found along the entire length of the capsule and a mass transfer deposit was evident near the top. Typical sections of the top and bottom of the capsule are shown in Figure 19. This capsule was a vacuum type capsule and it is believed that the mass transfer deposit resulted from refluxing in the space above the liquid.

500X TOP SECTION OF CAPSULE 2455
MASS TRANSFER DEPOSIT
AND DEPLETED LAYER



500X BOTTOM SECTION OF CAPSULE 2456
THIN DEPLETED LAYER

FIGURE 19. HAYNES 25 CAPSULE NO. HDC-11 TESTED AT 1100°F FOR 10,000 HOURS

441-64-729

e. Summary of Results of Haynes 25 Tested at 1100°F

A microhardness survey of the Haynes 25 capsules exposed to Hg at 1100°F is shown in Figure 20, and results from these capsules are summarized in Table 4. (The lot designations in Figure 20 indicate capsules made from different ingots of Haynes 25, see Table 2).

TABLE 4

SUMMARY OF TEST RESULTS FROM HAYNES-25 COMPATIBILITY CAPSULES EXPOSED AT 1100°F

	Room Temperature Tensile Test			Tensile Test at 1100°F				
Time, hr	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %	Room Temperature Knoop Hardness with 100 gm Load	Maximum Penetration of Depleted Layer, in.
0	138.6	70.3	38(1)		Not Tested		408	None
500	154	75	48(2)		Not Tested		374	None
1000	149	76	57(3)		Not Tested		378	0.0005
2000	131	83	20(2)	83.3	63.3	24(2)	400	0.0008
5000	136.2	86	21		Test Not Planned		393	0.0013
10,000		Test Not Completed			Test Not Planned		409	0.002

(1) Average of three tests

(2) One test

(3) Average of two tests

40.1-64-730

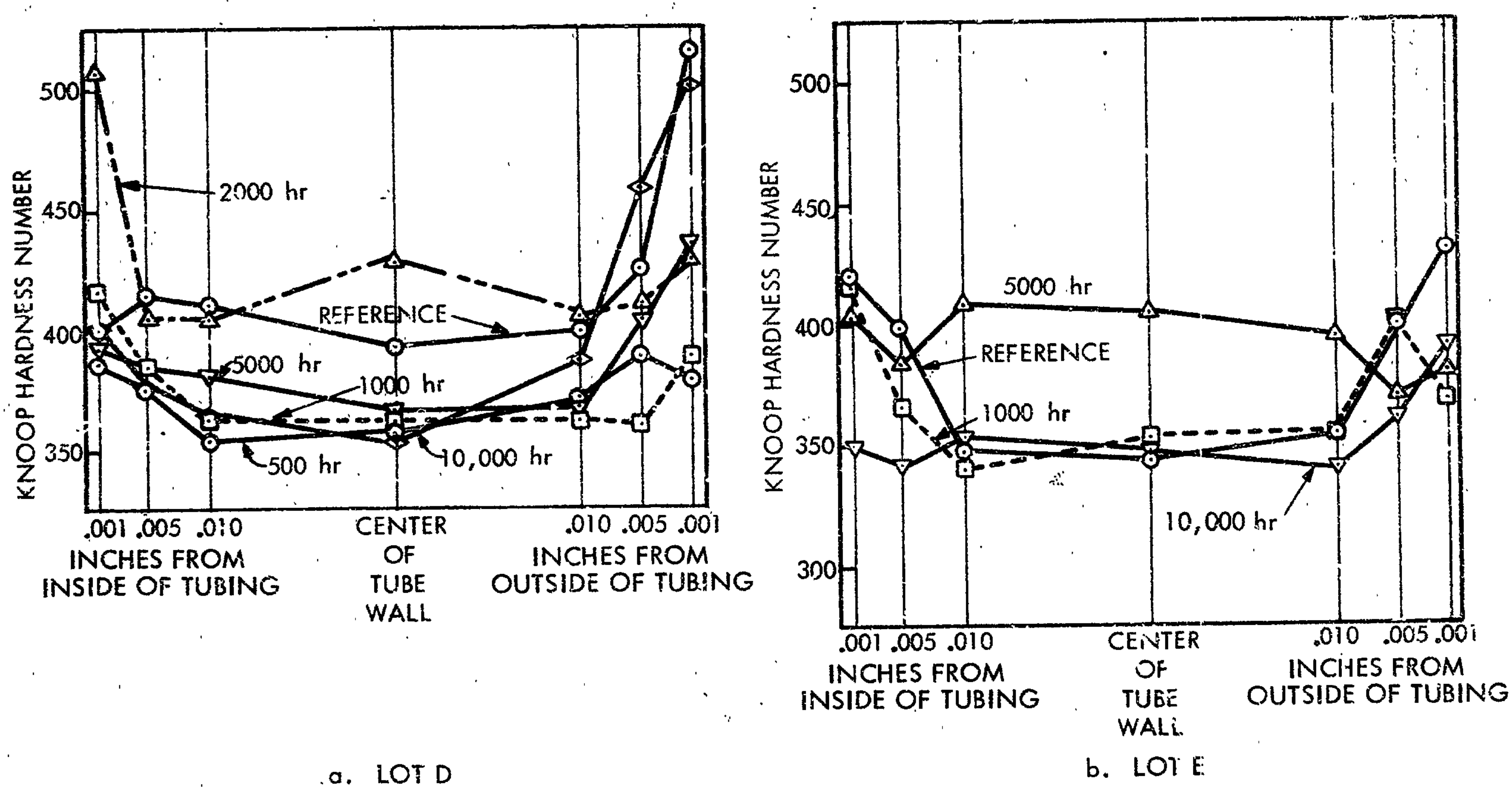
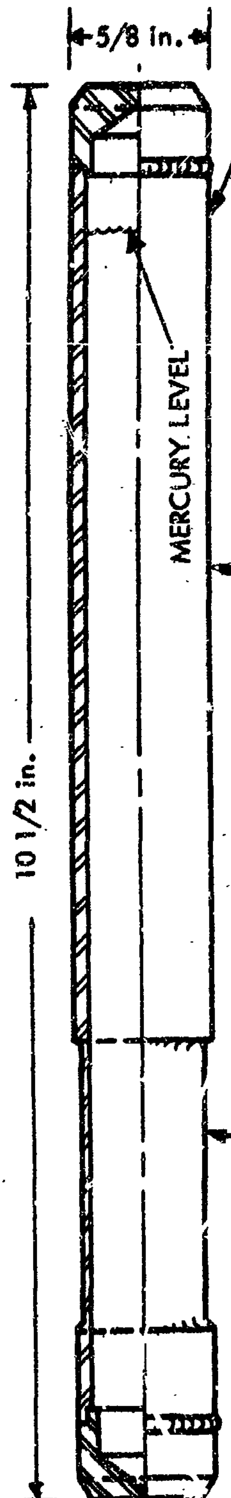


FIGURE 20. MICROHARDNESS SURVEY, HAYNES 25 EXPOSED TO MERCURY AT 1100°F

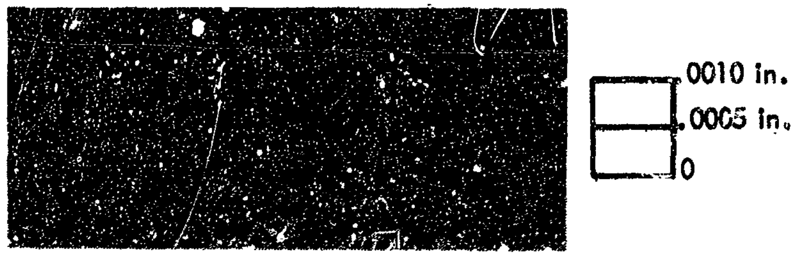
3. Capsules Exposed at 1175°F

a. Capsule No. HDB-30 - 2000 Hours at 1175°F

This capsule showed the same pattern found in the 2000 hour 1025°F capsule, except that the depleted layer apparently was spalled in some places. Also, the depleted layer did not graduate into the mass transfer deposit but continued along under the deposit for a distance of about three inches. The largest crystals were approximately 0.0015 in. in maximum dimension. Typical photomicrographs are shown in Figure 21.

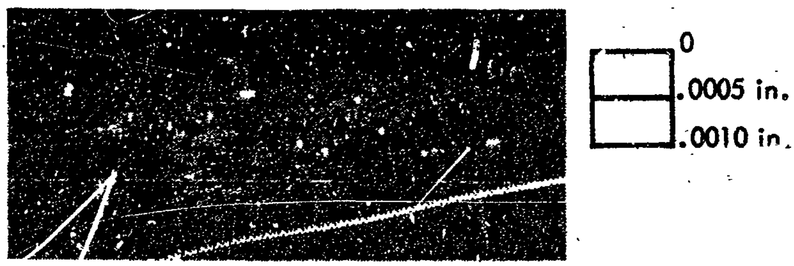


COMPATIBILITY
CAPSULE



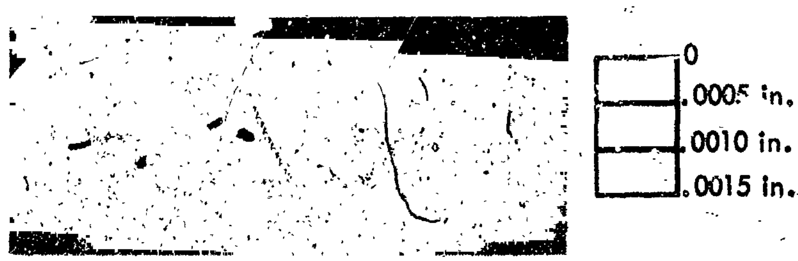
500X 1286

TOP SECTION OF CAPSULE
MASS TRANSFER DEPOSIT



500X 1278

MIDDLE SECTION OF CAPSULE
DEPLETED LAYER



500X 1277

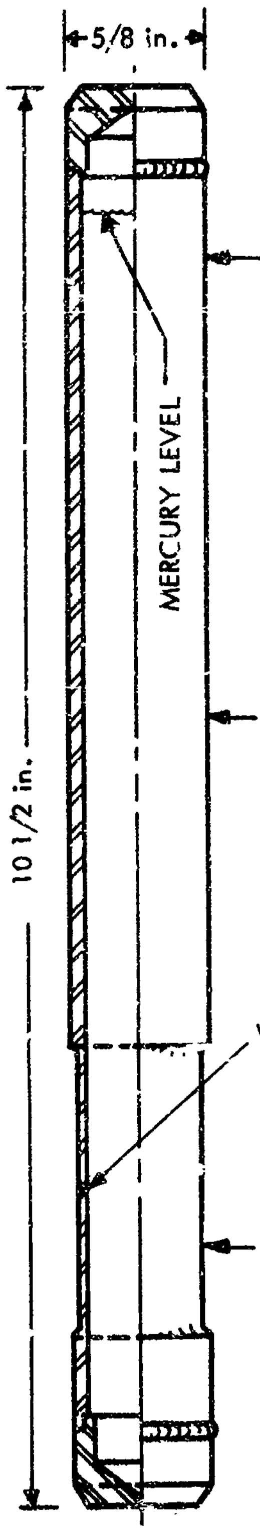
BOTTOM SECTION OF CAPSULE
DEPLETED LAYER

081-64-731

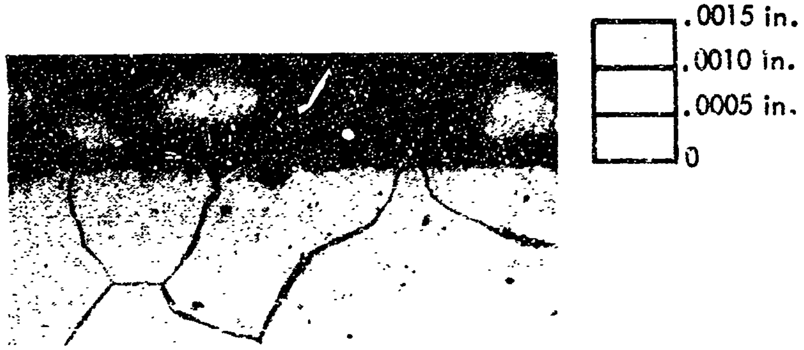
FIGURE 21. HAYNES 25 CAPSULE NO. HDB-30 TESTED AT 1175°F FOR 2000 HOURS

b. Capsule No HDBW-37 5000 Hours at 1175°F

The depleted layer found near the bottom of the capsule had spalled in several areas (See Figure 22). The depleted layer extended down in small V-shaped areas in what appeared to be intergranular attack. The intergranular attack did not appear to advance rapidly, however, but stayed just ahead of the depleted layer. A depleted layer was found along the entire length of the capsule, extending under the mass transfer deposit at the top. Spalling of the depleted layer took place only in the bottom of the capsule where the temperature was highest.

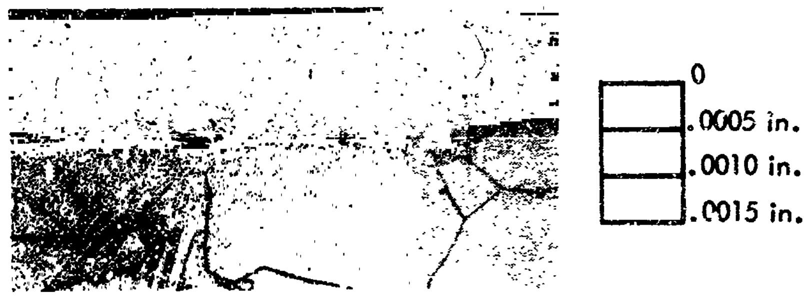


COMPATIBILITY
CAPSULE



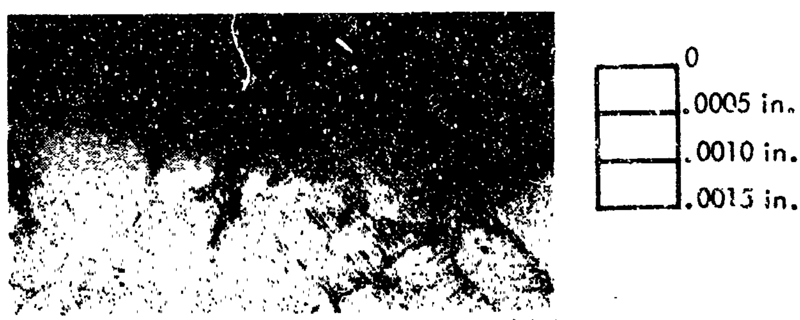
500 X 2454

TOP SECTION OF CAPSULE
MASS TRANSFER DEPOSIT



500X 2453

MIDDLE SECTION OF CAPSULE
DEPLETED LAYER



500X 2452

BOTTOM SECTION OF CAPSULE
AREA WHERE DEPLETED LAYER HAS SPALLED

FIGURE 22. HAYNES 25 CAPSULE NO. HDBW-37 TESTED AT 1175°F FOR 5000 HOURS

c. Summary of Results of Haynes 25 Tested at 1175°F.

The results of microhardness surveys are shown in Figure 23; test results are summarized in Table 5.

TABLE 5

SUMMARY OF TEST RESULTS FROM HAYNES-25 COMPATIBILITY CAPSULES EXPOSED AT 1175°F

	Room Temperature Tensile Test			Tensile Test at 1175°F			Room Temperature Knoop Hardness with 100 gm Load	Maximum Penetration of Depleted Layer, in.(6)
Time, hr	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %		
0	138.6	70.3	38(1)		Not Tested		408	None
500		Not Tested			Not Tested		418	0.0001
1000	125	92	15(2)	86.3	57	7.0(2,4)	409	0.0002
2000	122	109	4(2,3)	87.6	55.1	15(2,5)	445	0.0011
5000		Test Not Completed			Test Not Planned		498	0.0015

(1) Average of three tests

(2) One test

(3) The tensile specimen used in this test had a butt weld across the center of the reduced section. The butt weld was made in the capsule body and machined on both sides when the capsule was fabricated.

(4) The tensile test failed in the grips. Values of units and elongation are low

(5) No weld in the center of the reduced section

(6) Depleted layer apparently did not form, or spalled off.

40.1-64-733

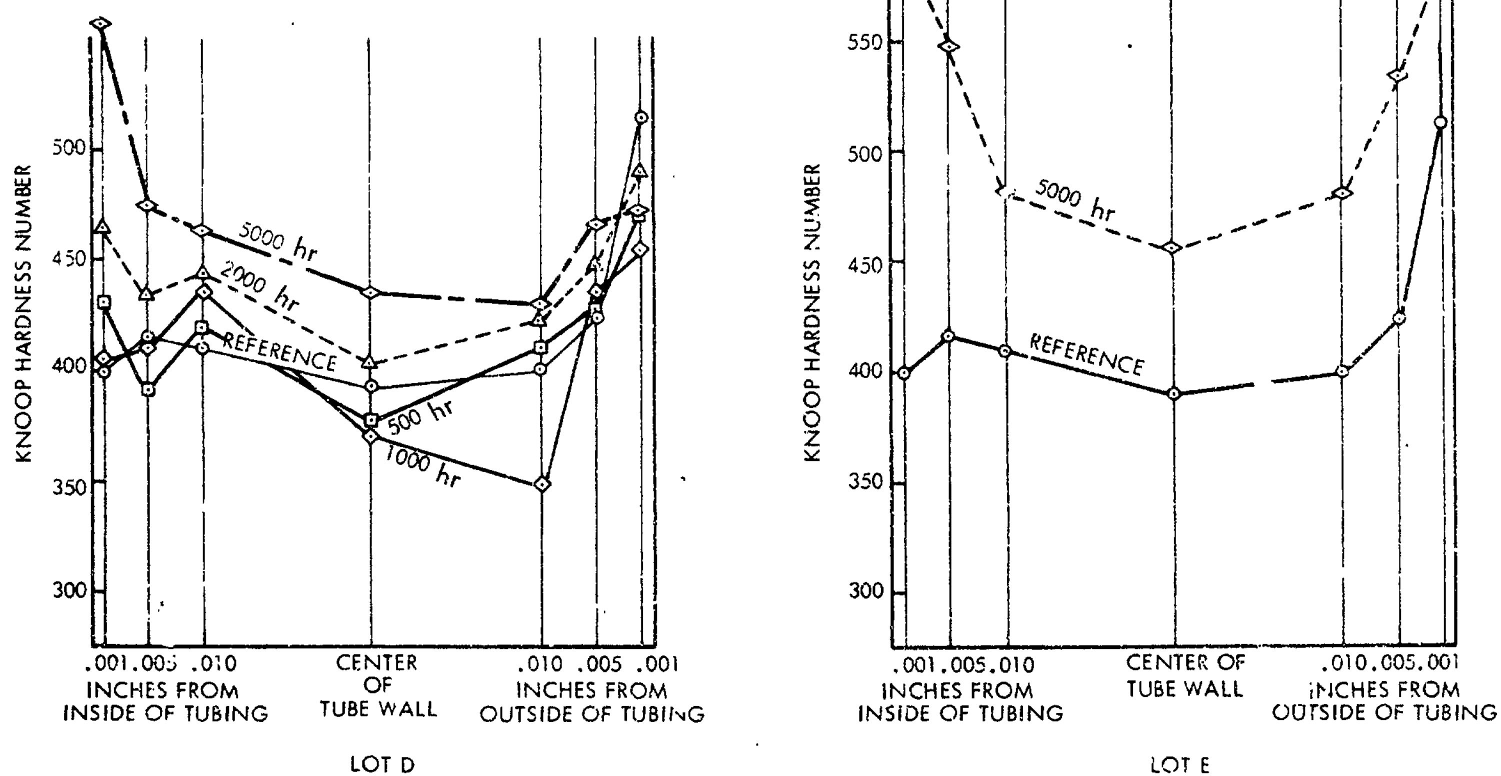


FIGURE 23. MICROHARDNESS SURVEY, HAYNES 25 EXPOSED TO MERCURY AT 1175°F

4. Capsules Exposed at 1250°F

a. Capsule No. HDA-42 - 1000 Hours at 1250°F

The same pattern as in the 1025°F, 2000 hour capsule was again found, however, almost complete spalling of the depleted layer appeared to have occurred. A mass transfer deposit was evident over 2½ square inches near the top of the capsule. The mass transfer deposit appeared to be more dense than the deposits observed in the 1025° and 1175°F capsules. The greatest thickness of the mass transfer layer was 0.0006 in. Typical photomicrographs are shown in Figure 24.

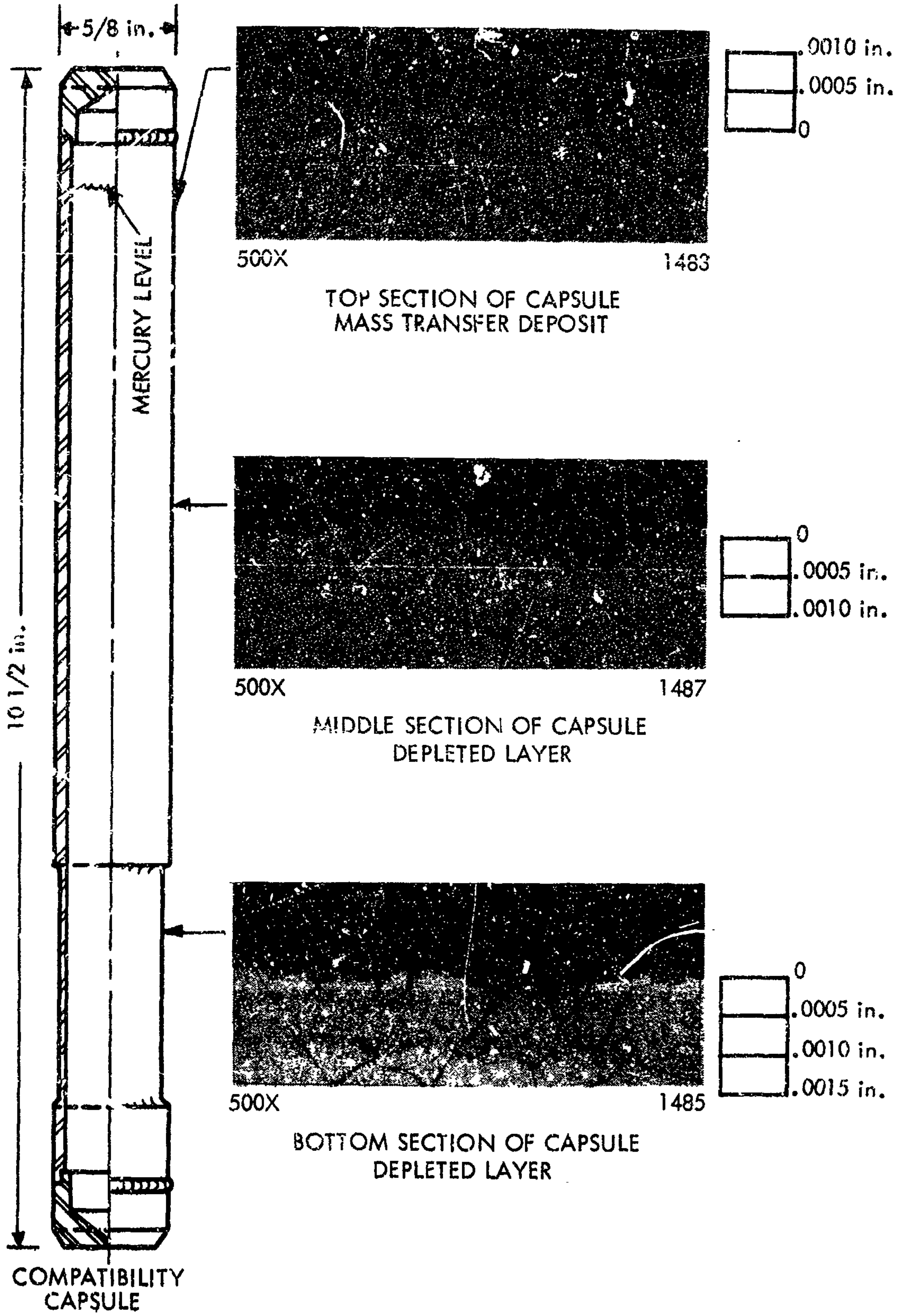


FIGURE 24. HAYNES 25 CAPSULE NO. HDA-42 TESTED AT 1250°F FOR 1000 HOURS

AGI-64-724

b. Capsule No.HDBW-46 .- 2000 Hours at 1250°F

The depleted layer found along the length of the capsule was thinner than in the capsules exposed at lower temperatures, again indicating almost complete spalling. The depleted layer extends down in small V-shaped areas before the overall surface is attacked. Figure 25 shows a typical section of this corrosion pattern. The attack did not appear to be entirely intergranular. The mass transfer deposit was similar to that found in the capsules exposed at lower temperatures except that it was more dense and the depleted layer continued under the deposit.

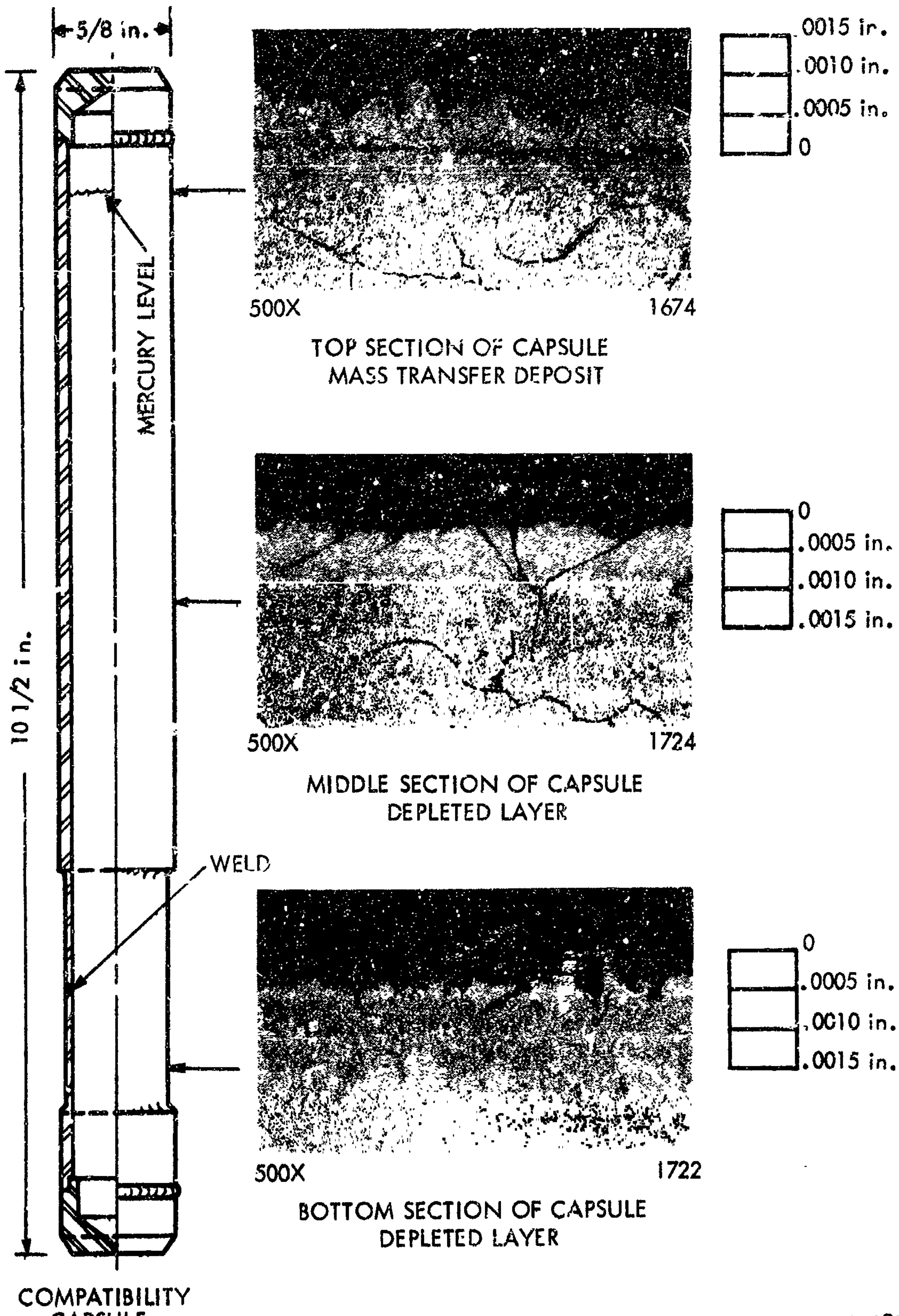


FIGURE 25. HAYNES 25 CAPSULE NO. HDBW-46 TESTED AT 1250°F FOR 2000 HOURS

c. Summary of Results of Haynes 25 Tested at 1250°F

Microhardness of the 1250°F capsules is presented in Figure 26 and results are summarized in Table 6.

TABLE 6

SUMMARY OF TEST RESULTS FROM HAYNES-25 COMPATIBILITY CAPSULES EXPOSED AT 1250°F

	Room Temperature Tensile Test			Tensile Test at 1250°F				
Time, hr	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %	Ultimate Tensile Strength, ksi	Yield Strength (0.2% Offset) ksi	Elongation in 1 inch, %	Room Temperature Knoop Hardness with 100 gm Load	Maximum Penetration of Depleted Layer, in.(6)
0	138.6	70.3	38(1)		Not Tested		408	None
500		Not Tested		91.1	62.1	12(3)	441	0.0002
1000	118.6	109.3	--(2,3)	91.4	71.9	6(3)	505	0.00035
2000(A)	139	109.6	4.0(3)				540	0.00075
(B)	155	137.3	1.0(3,4)	86.4		(2,4,5)		

(1) Average of three tests

(2) The tensile test failed in the grips. No elongation could be measured and the ultimate tensile strength value is low.

(3) One test

(4) This tensile specimen had a butt weld across the center of the reduced section. The butt weld was made in the capsule body and machined on both sides when the capsule was fabricated. The tensile specimen broke in the weld.

(5) The tensile specimen broke immediately after the load was applied. The yield strength and elongation could not be measured.

(6) Depleted layer apparently partially spalled.

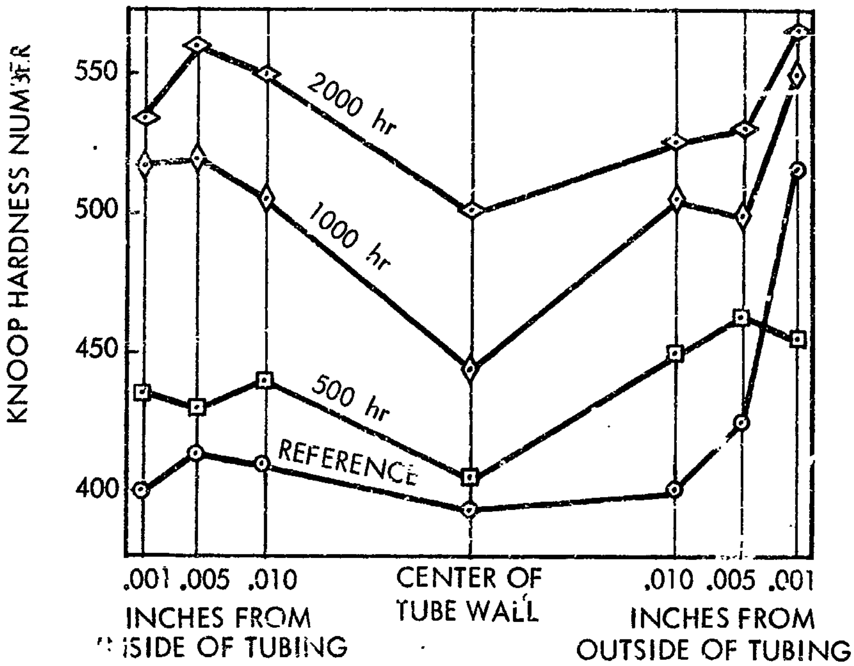


FIGURE 26. MICROHARDNESS SURVEY, HAYNES 25 LOI D
EXPOSED TO MERCURY AT 1250°F

401-64-736

5. Corrosion Product Analysis

The corrosion products which were generated in the 1025°F 2000 hr, 1100°F 5000 hr, 1175°F 2000 hr, 1250°F 1000 hr, and 1250°F 2000 hr capsules and were not attached to the walls were recovered from the mercury. This was done by distilling the mercury at a very slow rate and low temperature (about 200°F) to prevent carryover as very fine particles. Analysis of the powder by X-ray diffraction indicated that the main corrosion product from all of these capsules was cobalt, but nickel, chromium, and iron were present in lesser amounts. No indication of tungsten was found in any of the corrosion products from the capsules tested. X-ray diffraction showed only α-Co. It appears that cobalt nucleates and grows in the liquid phase, but only to very small particles, while nickel and chromium nucleate on a surface and grow into large crystals.

6. Mechanical Properties Tests

Room temperature tensile tests made on tensile specimens (Figure 9) cut from Haynes 25 compatibility capsules (Tables 4 through 6) showed loss of room temperature tensile ductility in Haynes 25 after exposure to Hg at 1100°F, 1175°F, and 1250°F for times up to 2000 hours. It was therefore decided to run elevated temperature tensile tests on specimens cut from the exposed Haynes 25 compatibility capsules. An improvement in ductility would be expected at the higher temperatures, as compared to the room temperature ductility.

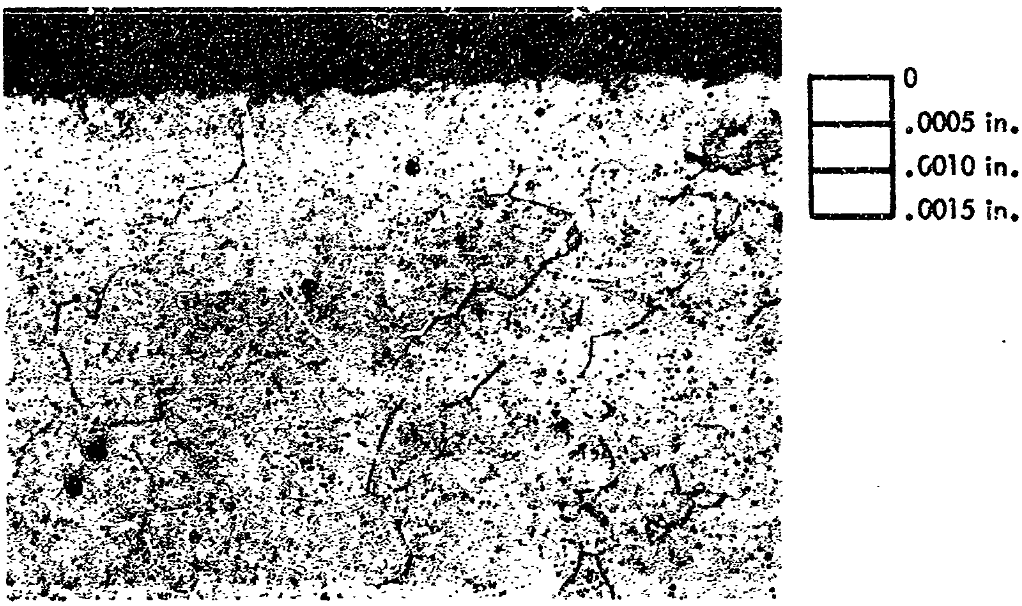
Elevated temperature tensile tests were completed on Haynes 25 specimens as listed in Tables 4 through 6 . The test temperature for each capsule was the same as the exposure temperature during the compatibility test, to avoid further age hardening of the Haynes 25. No elevated temperature tests were made on the Haynes 25 compatibility capsules exposed at 1025°F because the room temperature tests indicated no loss of ductility when compared to the unexposed Haynes 25.

B. TYPE 405 STAINLESS STEEL

1. Capsules Exposed at 1025°F

a. Capsule No. 4BA-8 - 5000 Hours at 1025°F

No corrosion layer of any type was found on the Type 405 stainless steel capsule exposed for 5000 hours at 1025°F. A mass transfer deposit was formed at the top of the capsule, indicating that an even solution attack must have taken place. Figure 27 shows a typical microstructure of the inside of the capsule.



500X 2031

INSIDE SURFACE APPROXIMATELY 1/2 IN.
FROM THE BOTTOM WELD

FIGURE 27. TYPE 405 STAINLESS STEEL CAPSULE 48A-8 TESTED AT 1025°F FOR 5000 HOURS

481-G4-737

b. Capsule No. 4BB-13 - 10,000 Hours at 1025°F

The microstructures of typical sections of the inside surface of this capsule are shown in Figure 28. A slight mass transfer deposit was formed at the top, or coolest, part of the capsule, but it was much less than that formed in the 10,000 hour, 1025°F Haynes 25 capsule. No depleted layer was formed onthe bottom section of the capsule, indicating that solution attack of the entire wall had taken place rather than selective leaching of individual constituents.

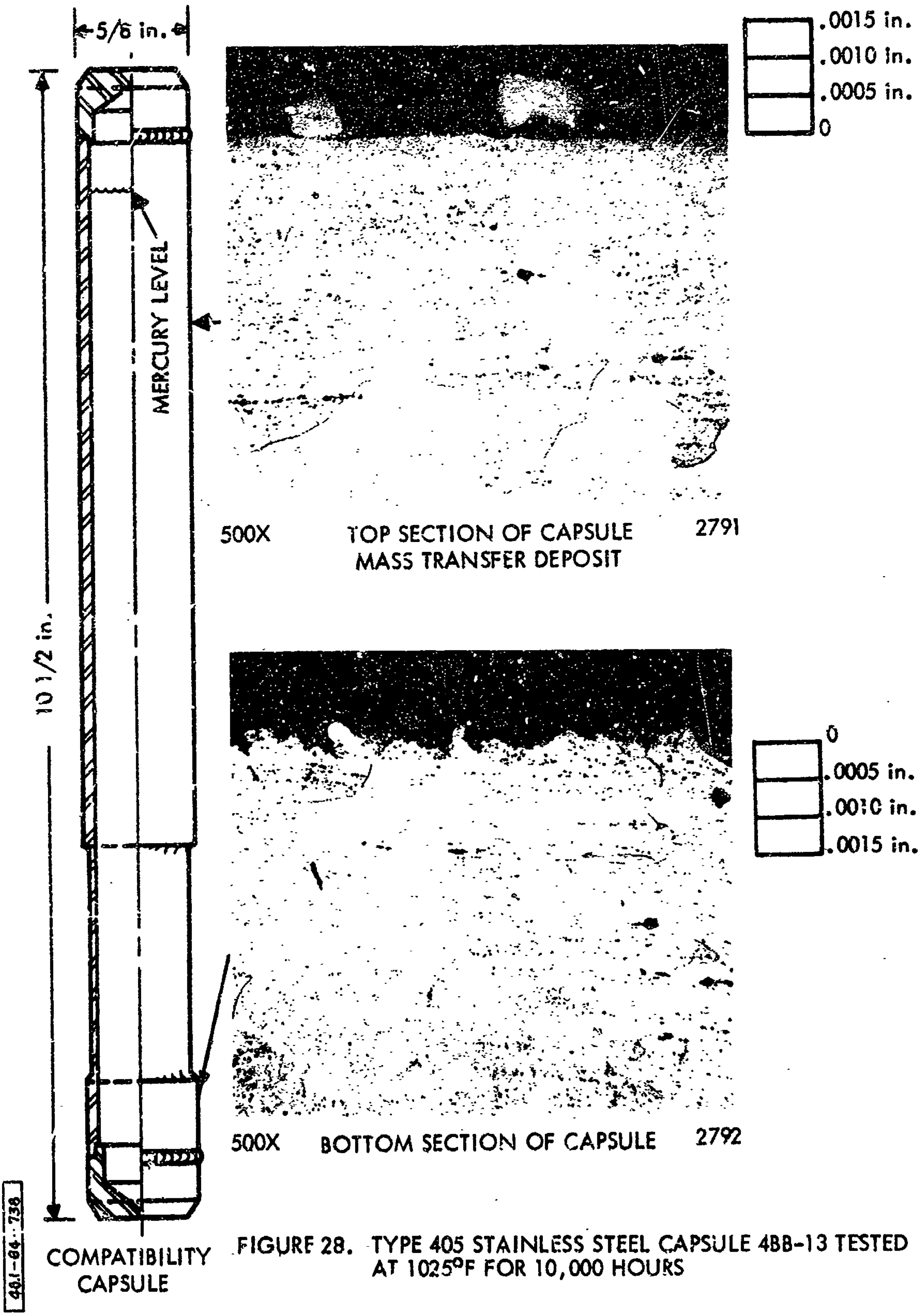
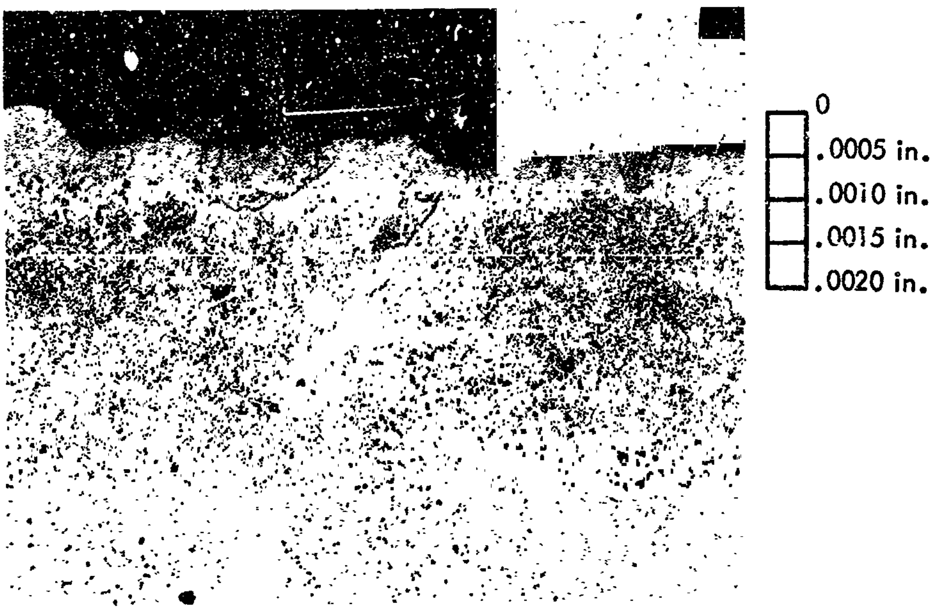


FIGURE 28. TYPE 405 STAINLESS STEEL CAPSULE 4BB-13 TESTED AT 1025°F FOR 10,000 HOURS

2. <u>Capsules Exposed at 1100°F</u>

a. Type 405 Stainless Steel Capsule No. 4BA-4
5000 Hours at 1100°F

No corrosion layer was found. A slight roughening of the surface occurred but no intergranular penetration occurred. Figure 29 shows a typical microstructure from the capsule exposed for 5000 hours at 1100°F.



500X 1380

BOTTOM SECTION OF CAPSULE
APPROXIMATELY 1/2 IN. FROM THE WELD

FIGURE 29. TYPE 405 STAINLESS STEEL CAPSULE NO. 4BA-4 TESTED AT 1100°F FOR 5000 HOURS

b. Caspule No. 4BA-1 - 10,000 Hours at 1100°F

No depleted layer was found on the inside surface. Solution attack appeared to have taken place along the entire length of the capsule. No mass transfer deposit, such as that found in the 405 stainless steel capsule exposed at 1025°F, was found in the top section of the 1100°F capsule. A microstructure from the bottom of the capsule is shown in Figure 30.

Corrosion products were found in the Hg. Apparently these corrosion products precipitated in the Hg instead of nucleating on the wall and growing as crystals. The difference in the behavior of the corrosion products formed in the 1025° and 1100°F capsules may be due to the temperatures in the top portions of the capsules. The top sections of the 1025°F capsules operated at approximately 850°F, while those in the 1100°F capsules operated at about 960°F.

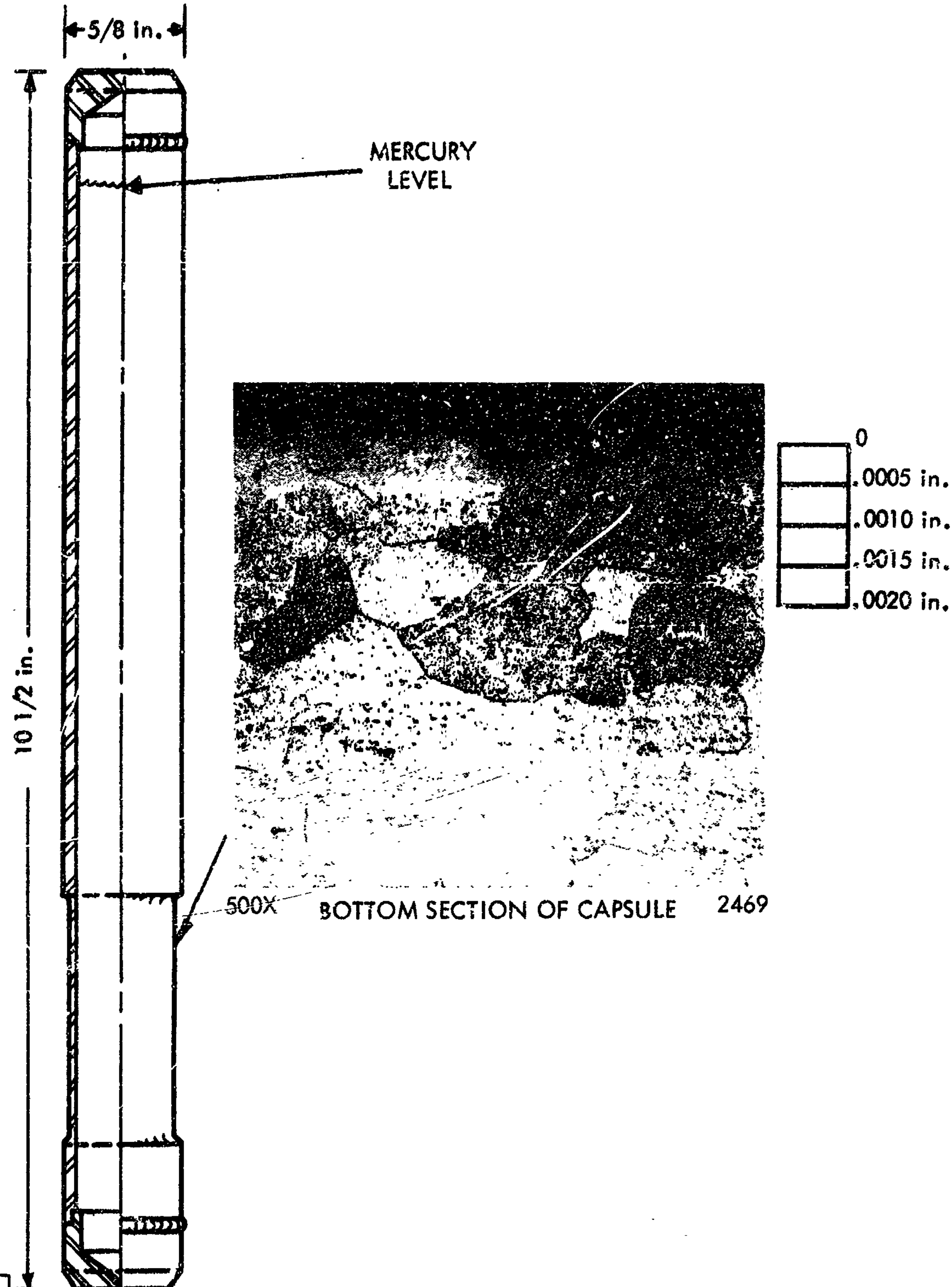


FIGURE 30. TYPE 405 STAINLESS STEEL CAPSULE NO. 4BA-1 TESTED AT 1100°F FOR 10,000 HOURS

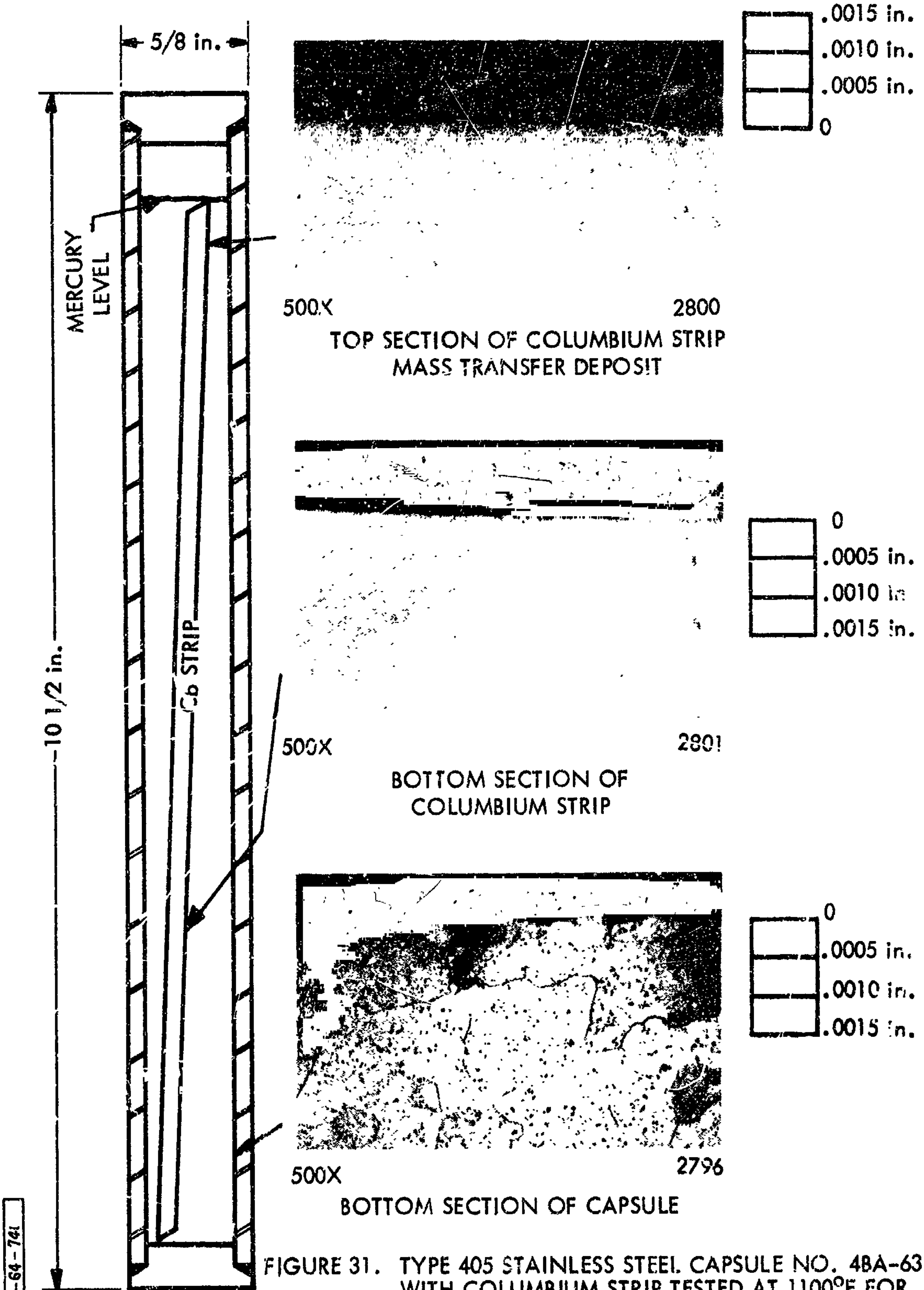
c. Capsule No. 4BA-63 - 2000 Hours at 1100°F with Columbium Strip

This capsule contained a columbium strip inside the capsule to test for bimetallic effects when Cb and Type 405 stainless steel are exposed to Hg. Figure 31 shows the position of the columbium strip in the capsule and typical microstructures of the strip and the capsule wall.

Mass transfer deposits were found on the top sections of the columbium strip and the capsule. The mass transfer deposits were similar to those found in the other 1100°F type 405 stainless steel capsules without a columbium strip.

The samples of the columbium strip were metallographically color stained to reveal any reaction with the mass transfer deposits or corrosion at the bottom section of the columbium strip. No indications of corrosion or reaction of the Cb with the mass transfer deposits were observed.

Microhardness measurements on the top and bottom of the columbium strip after exposure indicated that there was no change in hardness.



COMPATIBILITY CAPSULE

FIGURE 31. TYPE 405 STAINLESS STEEL CAPSULE NO. 4BA-63 WITH COLUMBIUM STRIP TESTED AT 1100°F FOR 2000 HOURS

d. Summary of Microhardness Tests

A summary of microhardnesses is shown in Figures 32 and 33. The capsules with Cb strips are not included.

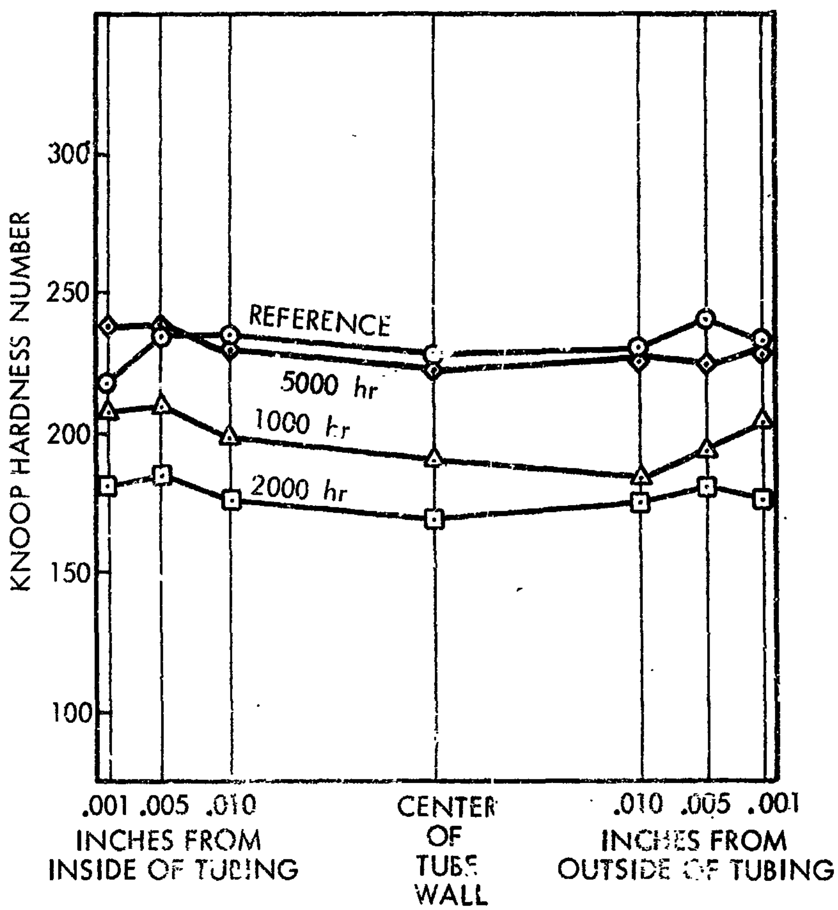


FIGURE 32. MICROHARDNESS SURVEY, TYPE 405 STAINLESS STEEL EXPOSED TO MERCURY AT 1025°F

461-64-742

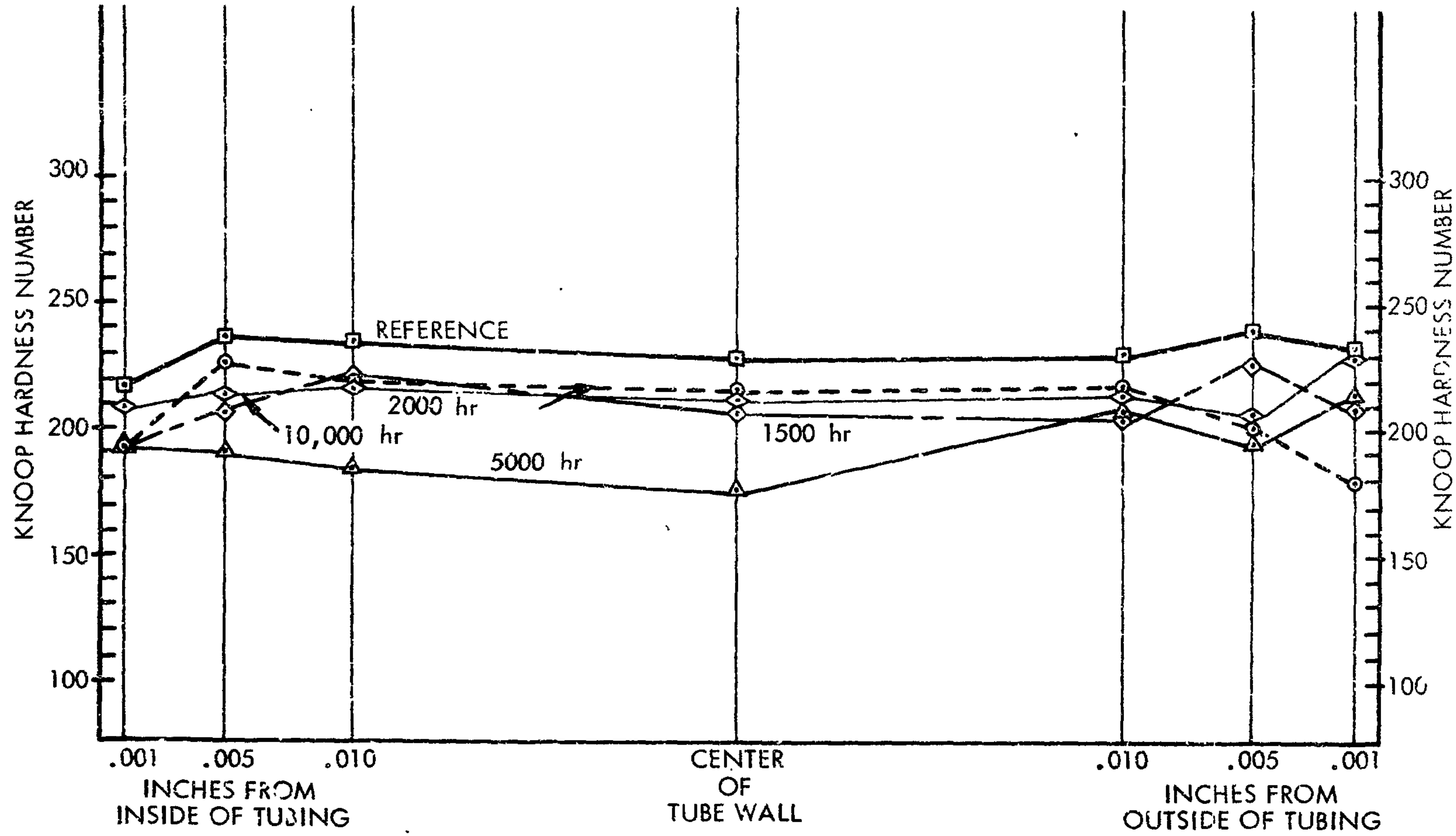
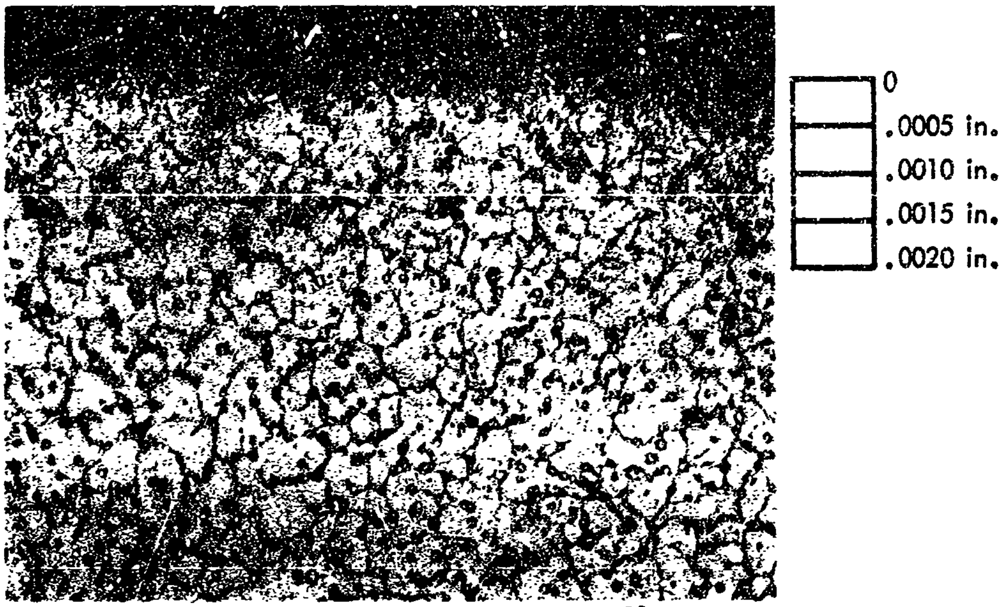


FIGURE 33. MICROHARDNESS SURVEY, TYPE 405 STAINLESS STEEL EXPOSED TO MERCURY AT 1100°F

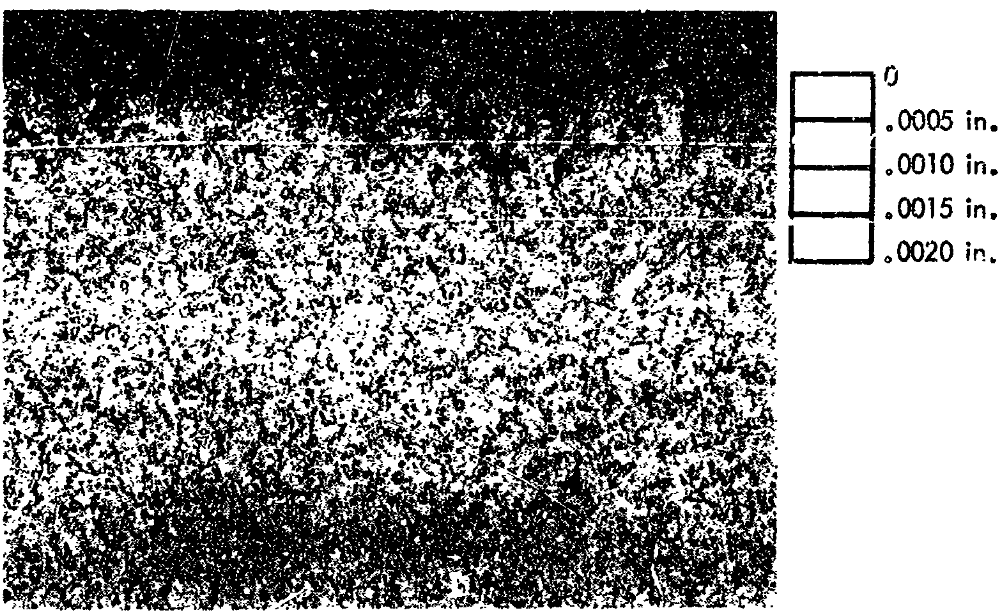
C. AM-350 (CAPSULES NO. AAA-50 AND AAA-51 - 500 HOURS AT 1250°F)

One capsule of AM-350 was tested in the solution annealed condition (1875°F followed by a water quench) and a second capsule was tested in the equalized condition (solution anneal plus 1425°F for 3 hours followed by air cooling and 1025°F for 3 hours followed by air cooling). A very small amount of mass transfer deposit formed at the coolest (top) end of each capsule. As illustrated in Figure 34, the bottom portion of the equalized AM-350 capsule was attacked to a greater degree than was the solution annealed AM-350 capsule.

Equalized AM-350 stainless steel therefore appeared to be less resistant to mercury corrosion than the solution annealed AM-350 after exposure for 500 hours at 1250°F. Analysis of the 1000-hour and 2000-hour capsules of equalized and solution annealed AM-350 must be completed before any firm conclusion can be reached concerning the suitability of this alloy. Other investigators have reported that equalized AM-350 was more resistant to Hg than solution annealed AM-350 under refluxing conditions.



500X SOLUTION ANNEALED
INSIDE SURFACE AT BOTTOM OF CAPSULE



500X EQUALIZED 2635
INSIDE SURFACE AT BOTTOM OF CAPSULE

FIGURE 34. AM-350 CAPSULES TESTED AT 1250°F FOR 500 HOURS

D. 9% CHROMIUM - 1% MOLYBDENUM ALLOY (CAPSULE NO. 9AA-71 WITH COLUMBIUM STRIP - 500 HOURS at 1250°F)

Figure 35 shows the position of the columbium strip in the capsules and the microstructures of the capsule wall and the columbium strip. Mass transfer deposits were found in the top portion of the capsule both on the columbium strip and the wall of the capsule. No depleted layer was found at the bottom of the capsule. The lack of a depletion layer plus the presence of the mass transfer deposits indicate that an even solution type attack must have taken place. This is typical of the type of attack found in the Type 405 stainless steel compatibility capsules.

There was no reaction between the mass transfer deposits (assumed to be Cr) and the Cb, judging from the microstructure of the latter. This was after an exposure of 500 hours at between 1100 and 1150°F.*

The microhardness of the columbium strip increased after exposure. The microhardness of the top section of the strip was 145 Knoop (100 gram load) and that of the bottom section was 139 Knoop. The original hardness of the columbium strip was 130 Knoop.

*Top of this 1250°F capsule operated at a temperature 100 to 150°F lower than that of the bottom.

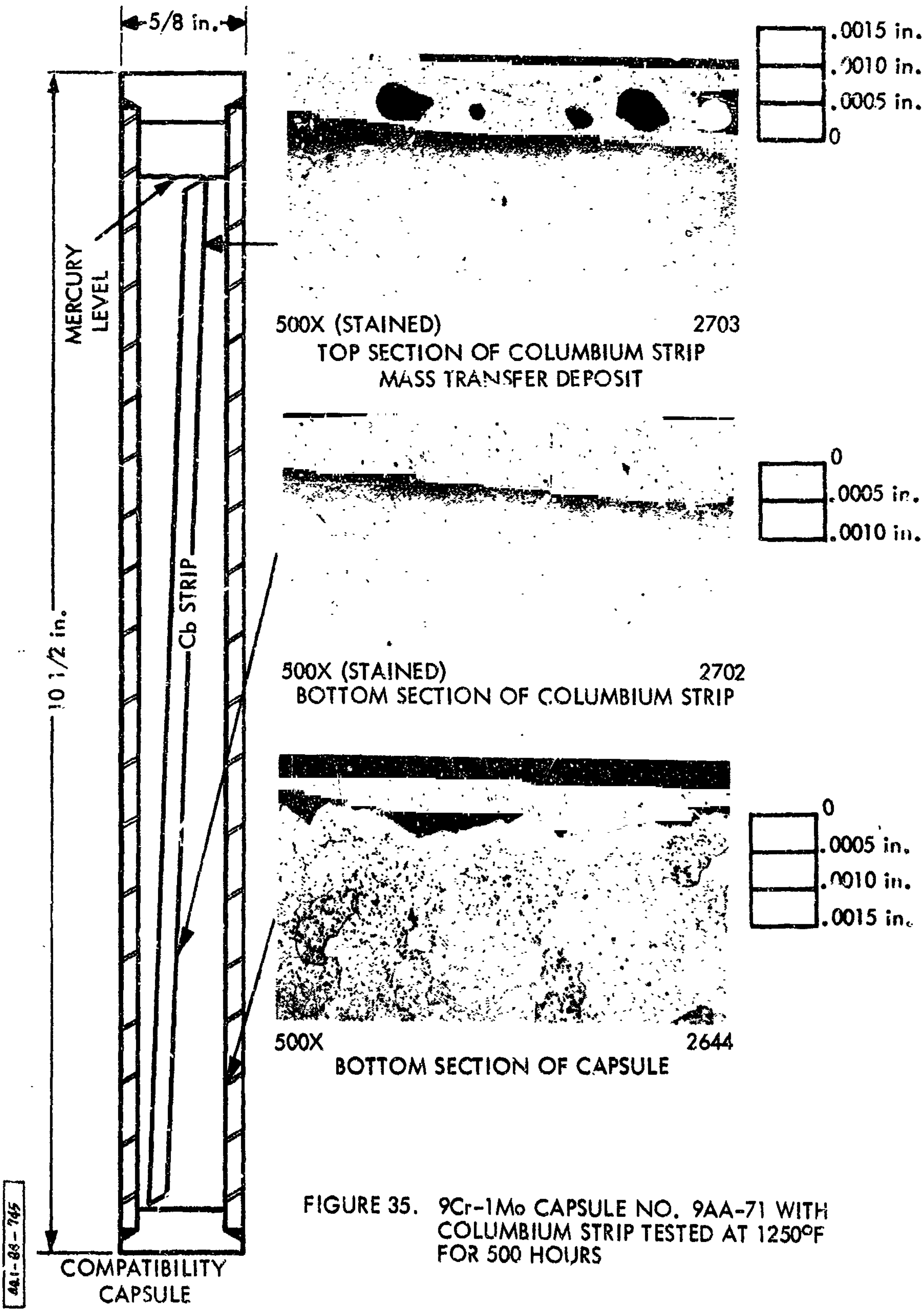


FIGURE 35. 9Cr-1Mo CAPSULE NO. 9AA-71 WITH COLUMBIUM STRIP TESTED AT 1250°F FOR 500 HOURS

E. COLUMBIUM - 1% ZIRCONIUM ALLOY

Two Cb-1Zr capsules were tested. One capsule was exposed for 4300 hours at 1100°F and the other capsule was exposed for 2500 hours at 1175°F. The capsule in the 1175°F furnace developed a leak in a closure weld between the 2000 and 2500 hour period, driving all the Hg from the capsule.

Both columbium capsules were sectioned for metallographic and micro-examination of the inside surface of the tubing. The metallographic samples indicated that the Hg had not attacked the inside surface of either capsule. Micro-examination of the inside surfaces of the capsules revealed no mass transfer deposit at the top of the capsules or any form of attack by the Hg. The mercury removed from the 4300 hour, 1100°F capsule was bright and clean with no evidence of the precipitates or powders found in the Haynes 25 and Type 405 stainless steel compatibility capsules exposed at similar temperatures (Figure 36).

Microhardness readings (Knoop, 100 gram load) were made on a ring cut from the bottom of each Cb-1Zr capsule and an unexposed section of the same lot of tubing. The average hardness of the 1175°F capsule (182 Knoop) and the 1100°F capsule (189 Knoop) was less than that of the unexposed tubing (228 Knoop). Stress relief of the tubing probably accounts for the reduction in hardness of the Cb-1Zr material.[8]

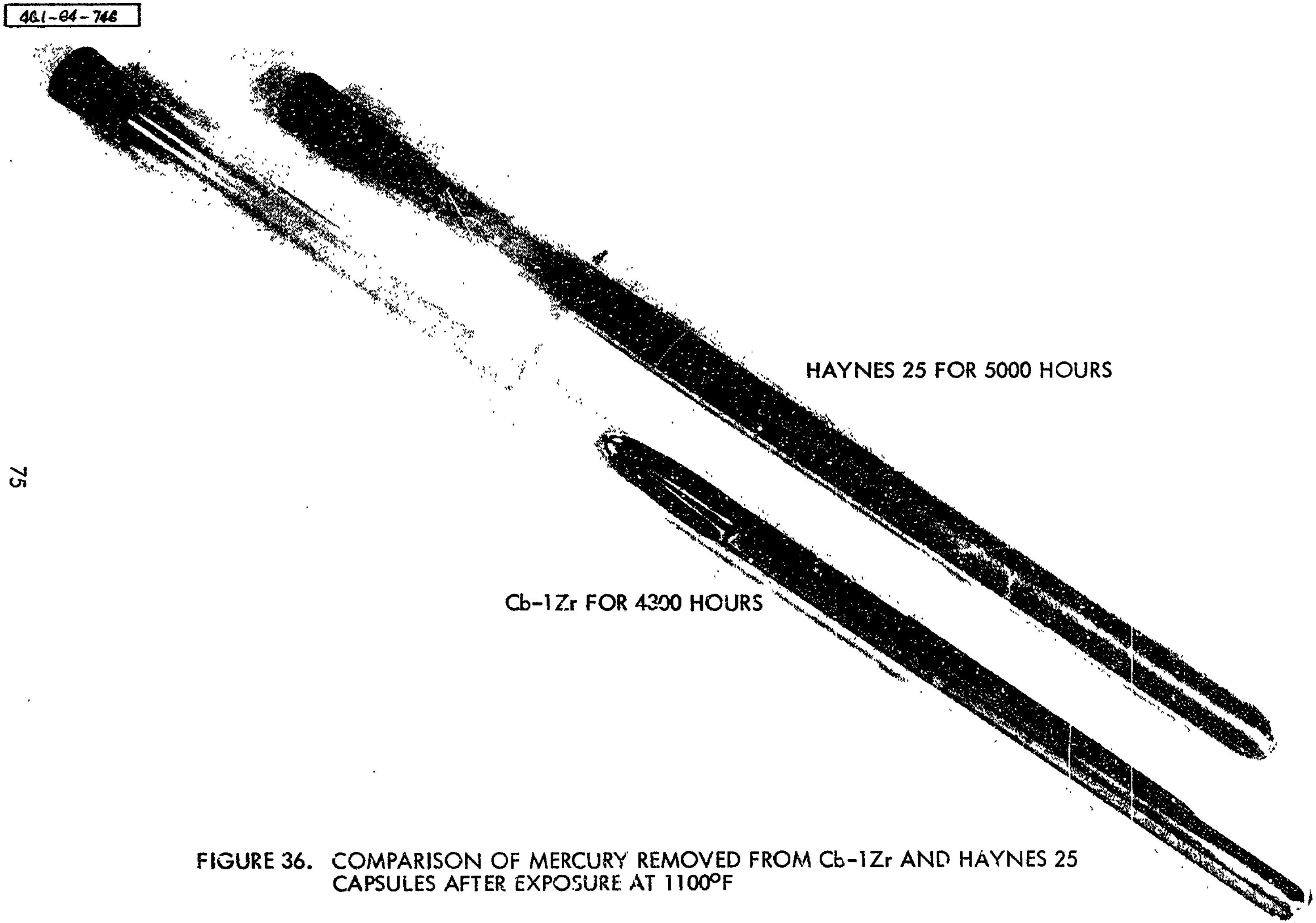


FIGURE 36. COMPARISON OF MERCURY REMOVED FROM Cb-1Zr AND HAYNES 25 CAPSULES AFTER EXPOSURE AT 1100°F

V. DISCUSSION OF HAYNES 25 CORROSION TESTS

A. CORROSION

The results of the capsule tests to determine the corrosion effects of Hg on Haynes 25 are summarized below. Some solution of the capsule wall in the liquid phase hot zone, subsequent mass transfer and deposition of mass transfer products in the cold zone was noted in all Haynes 25 capsules at all test temperatures, except in the isothermal capsules, where little corrosion was evident. Analysis of results showed that the constituents involved in the mass transfer effects are Ni, Cr, and Co, although not necessarily in that quantitative order. The thickness of depletion layers varied from 0.0005 in. in the 1025°F, 2000-hour capsule test to roughly 0.0020 in. in the 1100°F, 10,000-hour capsule and the 1025°F, 10,000-hour capsule. Mass transfer deposits ranged from 0.0005 to 0.0015 in. in thickness.

From the patterns found after sectioning some capsules along their lengths, it is evident that measurement of the depth of the depleted layer near the bottom of the capsule can be misleading when judging the amount of attack, especially in those capsules exposed at the higher temperatures. The corrosion pattern found in the capsules evaluated thus far indicates that the depleted layer is removed or spalls at the higher exposure temperatures (1175° and 1250°F). The thin depleted layers found on the 1250°F compatibility capsules and the evidence of spalling found on the 1175°F, 2000 hour capsule lead to this conclusion. Therefore, the penetration figures reported for the higher-temperature capsules are believed to be low, since they do not include the spalled material.

No evidence of stress corrosion by Hg was found in any of the Haynes 25 capsules with a reduced wall thickness in the area near the bottom of the capsule. The stress in the reduced wall varied up to 8000 psi.

The isothermal capsules (1100°F, 5000 and 10,000 hours) illustrate the relatively small amount of corrosion and mass transfer that takes place when there is little or no ΔT in the system. When the Hg becomes saturated with solute, no mechanism remains for further corrosion. Actually, because of a small ΔT (< 10°F) from the bottom to the top of the heating block a slight circulation and mass transfer potential probably existed. In the regular compatibility capsules, the mercury circulates slowly due to a ΔT of about 150°F along the capsule length, creating a much larger mass transfer potential.

B. MECHANICAL PROPERTIES

The Haynes 25 capsules exposed to test temperatures in the range from 1175° to 1250°F exhibited considerable age hardening, as shown in Figure 37. This degree of age hardening could be detrimental if Haynes 25 were used as the containment material for Hg in the SNAP-8 system. The effect of age hardening on other mechanical properties of Haynes 25, such as impact strength and notch sensitivity, was not determined.

The values obtained from the tensile tests indicate that the mechanical properties of Haynes 25 are affected by age hardening. However, the tensile values are not the same as typical or minimum values obtained for Haynes 25 aged in air at the test temperature. It should be remembered that the tensile specimens machined from compatibility capsules are not standard test specimens and that they have been exposed to Hg; therefore, the values can only be compared with results from tensile tests conducted with the same type of specimens exposed to similar environments.

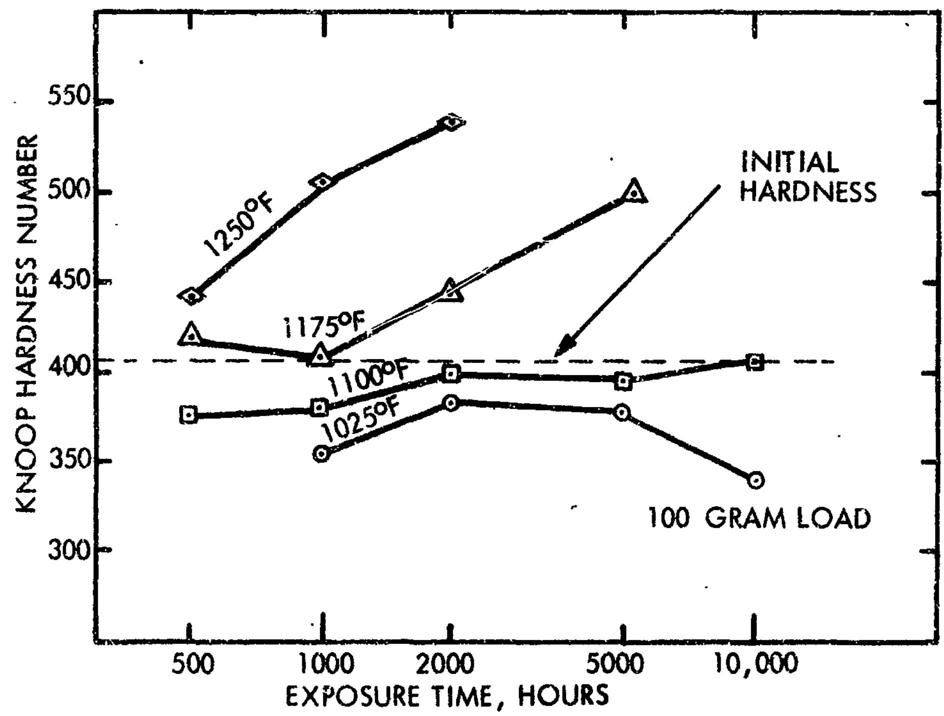


FIGURE 37. KNOOP HARDNESS VERSUS EXPOSURE TIME FOR HAYNES 25

40.1-64-2326

As would be expected, the ductility of Haynes 25 decreases as the hardness increases. Figure 38 is a plot of tensile elongation versus hardness for the compatibility specimens tested. There appears to be poor correlation between tensile elongation and hardness around 400 Knoop; however, the increasing hardness of the Haynes 25 exposed to temperatures of 1175°F and higher is a good indication of loss of room temperature ductility. Age hardening of this alloy, as evidenced in hardness increases and decreases in ductility, does not occur below 1100°F.

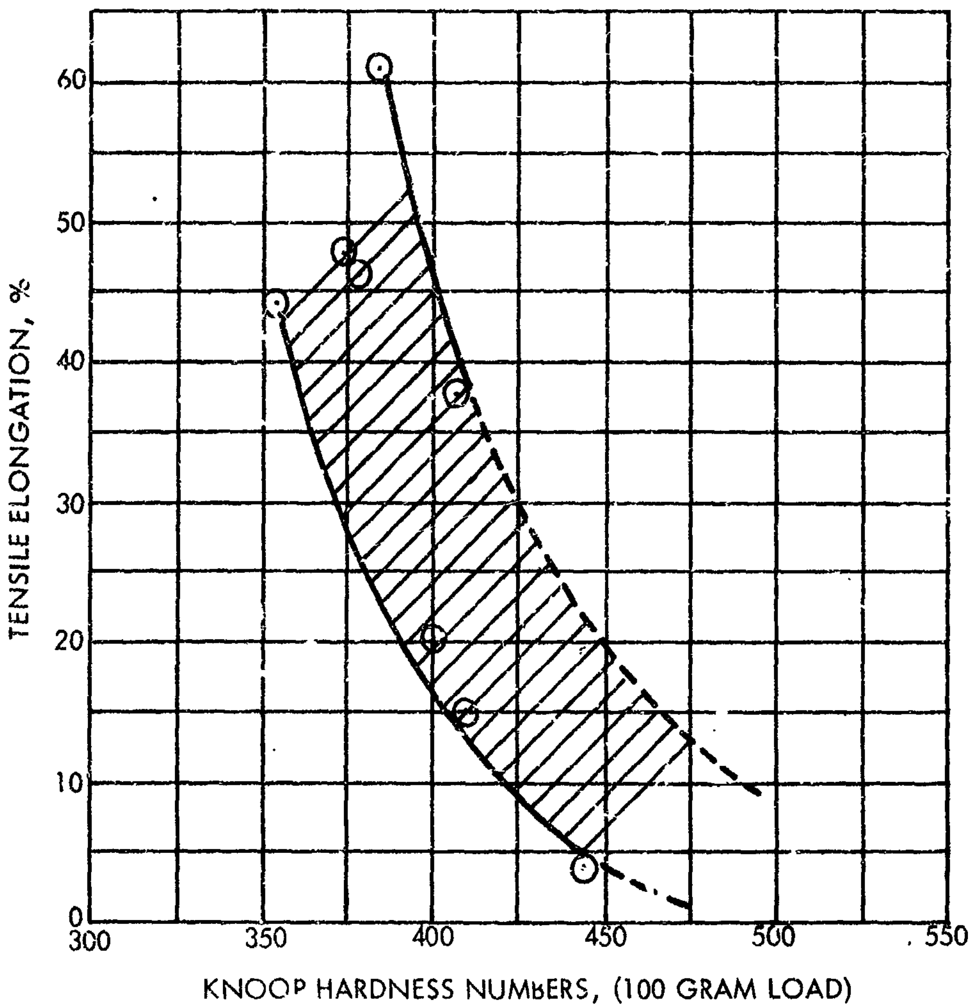


FIGURE 38. TENSILE ELONGATION VERSUS KNOOP HARDNESS FOR HAYNES 25 TENSILE TEST SPECIMENS

VI. MECHANICAL PROPERTIES OF AM-350

AM-350 alloy in the solution annealed condition was selected as a candidate material for containing Hg in the SNAP-8 system. Tensile and stress rupture data were not available for AM-350 in the solution annealed condition at the maximum temperature of the SNAP-8 system (1300°F). Solution annealed AM-350 sheet tensile specimens were placed in the compatibility furnaces and exposed to vacuum for periods up to 2000 hours at 1025°, 1100°, 1175°, and 1250°F.

The tensile test specimens originally were to be tested at room temperature after exposure in the compatibility furnaces to determine the effect of long-time exposure on the tensile properties of solution annealed AM-350. After the first set of tensile specimens exposed for 1000 hours at 1250°F were tensile tested, it was decided to perform stress rupture tests on the balance of the exposed specimens. The stress rupture tests would give more useful information on the effects which long-time exposure would have on the mechanical properties of AM-350.

Some tests are being run with equalized AM-350. The equalized condition (1425°F for 3 hours followed by air cool: 1025°F for 3 hours followed by air cool) is an overaging heat treatment used to improve the machinability of AM-350. The Hg corrosion resistance of AM-350 is improved when the material is in the equalized condition.* The stability of the material at 1300°F may also be improved after it is subjected to the overaging heat treatment.

*Concluded from other programs. See Section IV.C.

The results of stress rupture tests (Table 7) completed during the reporting period are plotted in Figure 39. Extrapolation of these test results indicate that solution annealed AM-350 should have a stress rupture strength of 7000 psi for 1000 hours and 4500 psi for 10,000 hours at 1300°F.

TABLE 7 - SOLUTION ANNEALED AM-350 RUPTURE TESTS AT 1300°F

Pre-Test Exposure				
Time, hr	Temperature, °F	Stress, psi	Time to Rupture, hr	Elongation in 1 in. after rupture, %
Unexposed		15,000	60	42
"		10,000	543	36
"		10,000	580	35
"		8,000	1359	38
"		8,000	1447	11(1)
"		8,000	1464	11(1)
2000	1100	20,000	3.4	36
2000	1100	15,000	14.3	36
2000	1100	10,000	126.0	20
2000	1025	10,000	101	27
2000	1025	8,000	485	27
2000	1025	8,000	540	24
2960	1175	8,000	216	46
2960	1175	6,000	892	21
EQUALIZED AM-350(2)		15,000	182	56
		15,000	137	55
		10,000	2076	45
		10,000	(Test not completed at end of reporting period)	
		8,000	(Test not completed at end of reporting period)	

(1) The electrical power failed after 1400 hours of the test. The specimen cooled to room temperature before the test was restarted.

(2) 1425°F for 3 hours, air cool, then 1025°F for 3 hours, air cool.

401-64-749

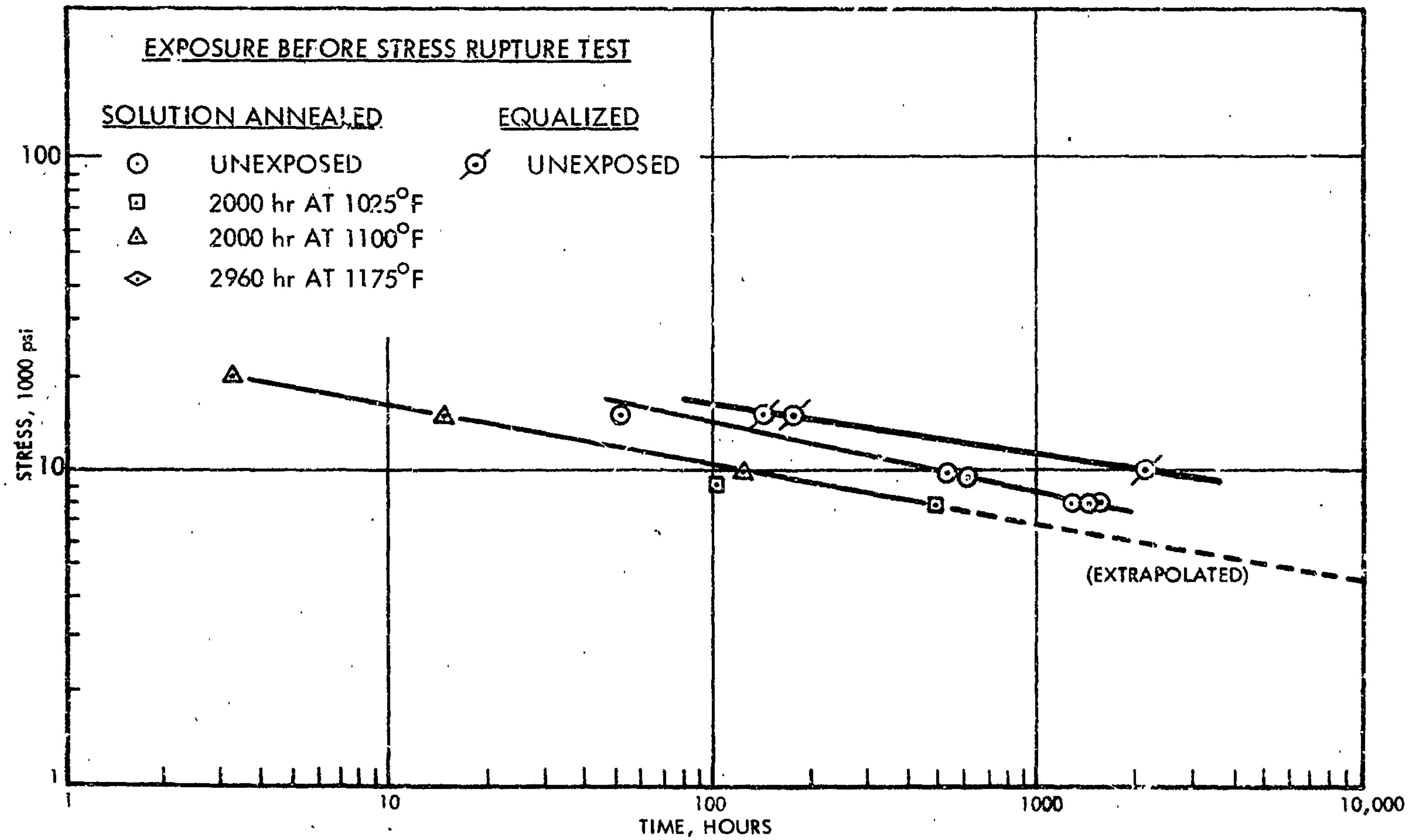


FIGURE 39. STRESS RUPTURE OF AM-350 AT 1300°F

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