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TYPE 316 STAINLESS STEEL, INCONEL, AND HAYNES ALLOY NO. 25 NATURAL-CIRCULATION BOILING-POTASSIUM CORROSION TEST LOOPS

D. H. Jansen
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OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

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METALS AND CERAMICS DIVISION

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JUNE 1965

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D. H. Jansen and E. E. Hoffman[1]

ABSTRACT

An investigation was undertaken to determine the compatibility of conventional nickel-, iron-, and cobalt-base high-temperature alloys with boiling potassium. The tests were designed to obtain quantitative information on the dissolution of the container alloys by condensing potassium and the subsequent deposition of solute in a subcooled liquid region of the test device.

Studies were conducted in natural-circulation loops, two of which were fabricated from type 316 stainless steel, two from Haynes alloy No. 25, and one from Inconel. Each of the alloys was operated at a maximum boiler-condenser temperature of 870°C for 1500 to 3000 hr, and the Haynes alloy No. 25 was also tested for 3000 hr at 980°C. The potassium condensing rates at the two temperatures were 170 to 180 and 300 g/min, respectively.

At 870°C type 316 stainless steel and Haynes alloy No. 25 exhibited comparable resistance to attack by boiling potassium, while Inconel showed greater deterioration.

Carbon was transferred from the condensing region to the subcooled liquid region in all the loop tests. Attendant with this, the elongation of the material decreased and the tensile strength increased. No evidence of preferential leaching of major metallic constituents of these alloys was detected by electron microprobe analysis of the condenser surfaces. However, limited mass transfer from the hot region to the cold region was noted.

INTRODUCTION

Future space exploration programs are in a large measure predicated on the development of light weight, efficient power plants to supply the electrical needs of space vehicles. Design studies[2] at the Oak Ridge National Laboratory have underscored the high performance achievable in

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[1]Now with Space Power and Propulsion Section, Missile and Space Division, General Electric Company, Cincinnati, Ohio.

[2]A. P. Fraas, "Boiling Potassium Reactor for Space," Nucleonics 22(1), 72 (1964).

a nuclear reactor mated to a Rankine-cycle turbine generator, using a single working fluid to cool the reactor, power a turbine, and lubricate the turbine, generator, and pump. Potassium was concluded to be the best choice for the working fluid in such a system.

One problem outlined in this study was the need for information regarding the compatibility of boiling potassium with structural alloys at system temperatures. Consequently, an evaluation of the long-term effects of boiling potassium with Inconel, type 316 stainless steel, and Haynes alloy No. 25 was started. The purpose was to compare the relative corrosion properties of high-temperature nickel-, iron-, and cobalt-base alloys with respect to potassium liquid and vapor. The studies employed natural-circulation loops, designed to supply quantitative information regarding the dissolution and deposition rates in a condenser and liquid subcooler at known condensing rates.

A total of five loop tests were conducted. The loop materials and the operating conditions for each test are given in Table 1.

Table 1. Materials and Operating Conditions of Boiling Potassium Corrosion Loop Tests

Material	Test Number	Duration (hr)	Condenser Temperature (°C)
Type 316 stainless steel – two tests	1	3000	870
(69% Fe–17% Cr–12% Ni–2% Mo)	2	3000	870
Inconel	3	1500	870
(80% Ni–14% Cr–6% Fe)			
Haynes alloy No. 25 – two tests	4	3000	870
(50% Co–20% Cr–15% W–10% Ni–3% Fe–1% Si–1% Mn)	5	3000	980

This report discusses the design, fabrication, and operating procedures of three different loop configurations that were used in the course of these investigations. The results of each test are reported in the order of the above list and the compatibility of the various loop materials with boiling potassium is compared.

POTASSIUM PREPARATION AND FILLING PROCEDURES

The potassium used in these studies was a high-purity, low-sodium grade material purchased from MSA Corporation. Prior to shipment, this grade of potassium was given a purification procedure to remove sodium and oxygen, which involves hot trapping with zirconium or zirconium-titanium for at least 24 hr at 538°C (1000°F). A typical vendor's analysis of a large, 200-lb potassium shipment is as follows:

Element	ppm
Fe	30
B	<20
Co	<10
Mn	3
Al	3
Mg	<5
Sn	2
Cu	2
Pb	<5
Cr	<5
Si	50
Ti	<10
Ni	<10
Mo	<5
V	<5
Ag	<1
Be	<5
Ca	<100
Na	100 (estimated)

Upon receipt of a shipment, further purification was performed on the material in accordance with the flow diagram illustrated in Fig. 1. The sequence of purification steps was as follows:

1. Filtration from the shipping container through a 10-μ pore size stainless steel powder compact filter to remove gross particles.

2. Gettering with titanium sponge at 650°C (1200°F) for 450 hr. The weight ratio of potassium to titanium sponge was approximately 1 1/2 to 1. This operation would reduce the oxygen content to approximately 300 ppm.

3. Two periods of cold trapping for extended times at 250°C (480°F) and 100°C (212°F) in the same container used for hot trapping. This operation would reduce the oxygen content to approximately 200 ppm.

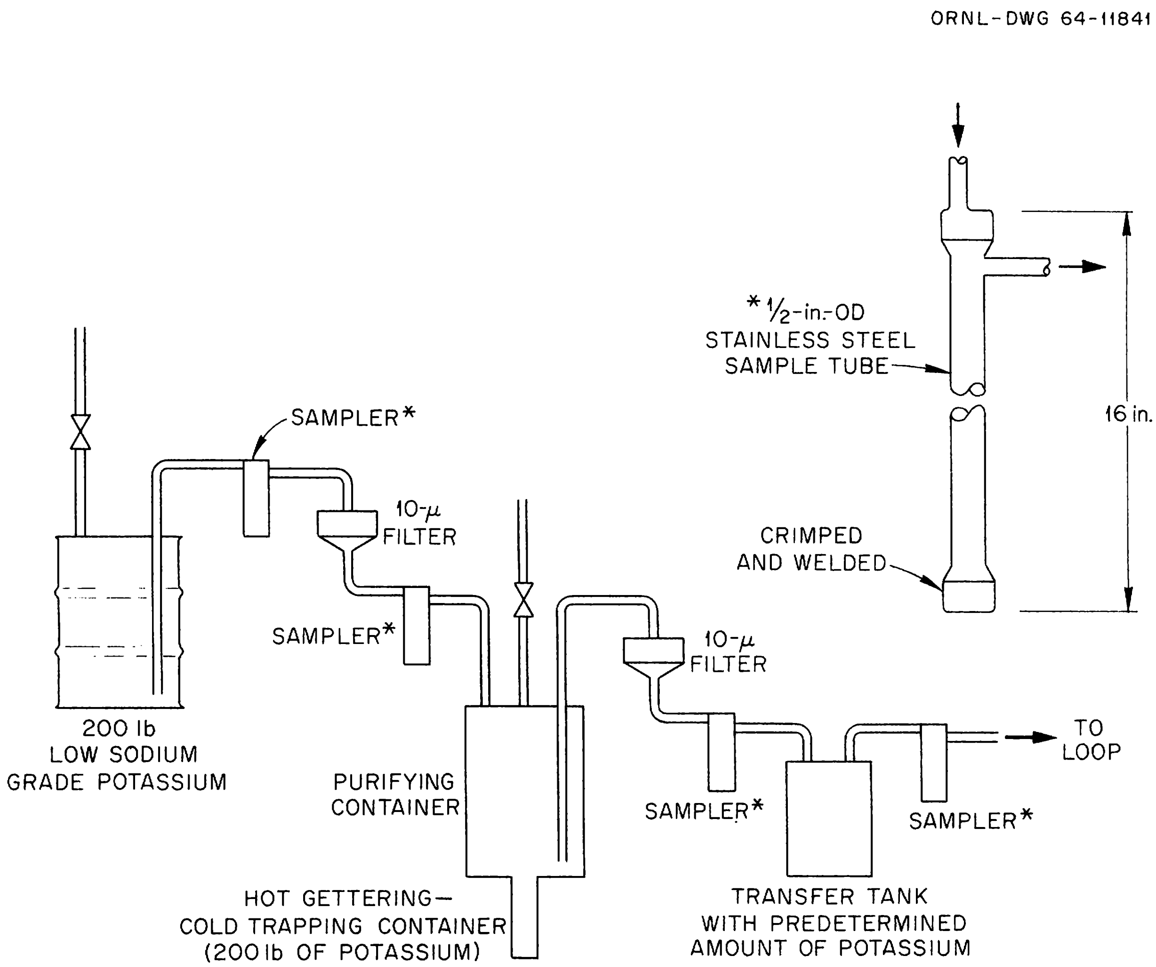

Fig. 1. Flow Diagram and Purification Sequence for Potassium Used in Loop Tests.

The vessel used for gettering, illustrated in Fig. 2, was large enough to contain the entire 200-lb shipment and was utilized for both

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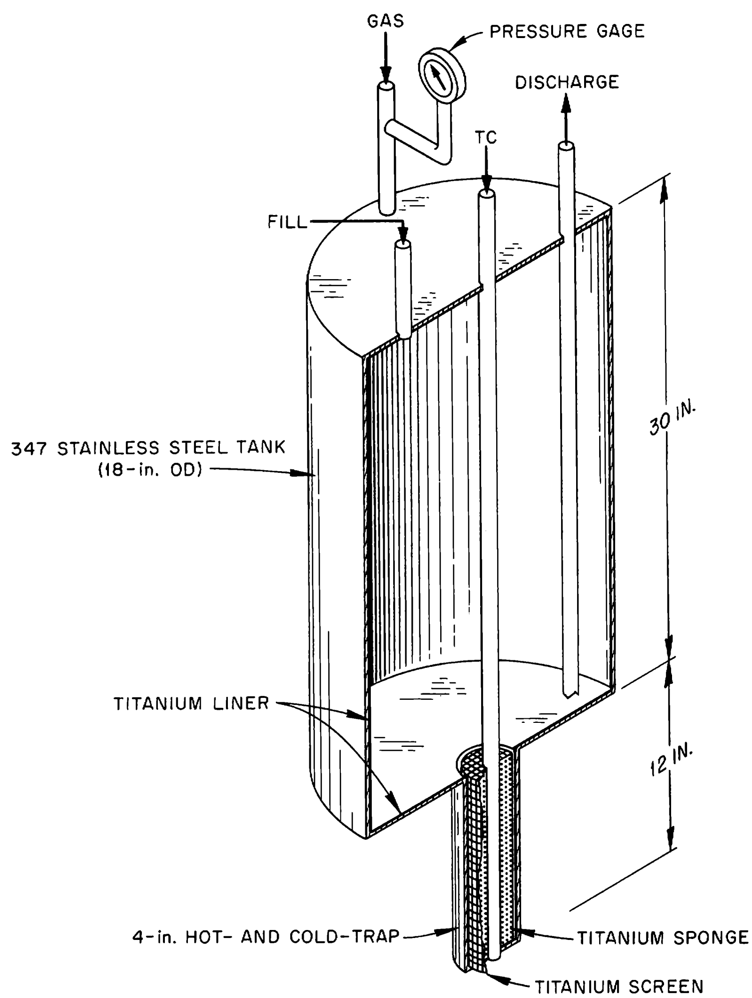

Fig. 2. Potassium Purification Tank.

the hot-gettering and cold-trapping operations. The temperatures involved in these operations and the resulting oxygen analysis[3,4] in each purification step are listed in Table 2.

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[3]J. C. White, W. J. Ross, and R. Rowan, Jr., "Determination of Oxygen in Sodium," Anal. Chem. 26(1), 210–13 (January 1954).

[4]The modified n-butyl bromide method for determining oxygen in potassium was used to analyze the potassium in all the tests discussed in this report. Subsequent experience with other analytical methods indicates that the n-butyl bromide method used generally yielded higher than actual oxygen values.

Table 2. Oxygen Contents of Potassium Shipment After Various Hot-Gettering and Cold-Trapping Steps

Process Description	Temperatures at Various Locations of Purification Tank (°C)	Time (hr)	Oxygen Content[a] (ppm) Individual Analyses	Average
As-Received			2460, 2680, 2930	2690
Hot Gettered at 650°C (using 8.4-kg Ti sponge)	Tank 650; lower leg → 650	50	260, 360, 380	330
		150	180, 500	360
		250	200, 200, 200	200
Cold Trapped[b]	Tank 250; lower leg → 80, → 45	100	330, 400, 650	460
		250	320, 380, 400	370
		400	490, 510	500
		550	200, 250, 500	320
		700	190, 270, 280, 470, 570	360
Cold Trapped[b]	Tank 100; lower leg → 35, → 30	100	180, 270, 350	270
		200	160, 420	290
		300	140, 190, 200	180
		400	180, 330	280

[a] Analytical Method: n-butyl bromide. Ref: J. C. White, W. J. Ross, and R. Rowan, Jr., "Determination of Oxygen in Sodium," Anal. Chem. 26(1), 210–13 (January 1954).

[b] Potassium Melting Point: 64°C.

Analyses, performed at ORNL, of metallic impurities in the potassium before and after all purification steps are given in Table 3. The metal analyses were determined by the spectrophotometric method. While designed specifically to remove oxygen, the purification process also produced a slight reduction in metal contaminants.

Table 3. Analyses of Metallic Impurities in Potassium Used in Natural-Circulation Loop Tests

Treatment	Element (ppm)				
	Iron	Nickel	Chromium	Titanium	Molybdenum
As-Received	<25	<25	<25	<20	<25
Hot trapped for 450 hr at 650°C	<15	<20	<10	<5	<10

Filling the loops with the potassium operating charge involved the following sequence of steps:

1. With low power applied to the heaters and with thermocouples reading approximately 500°C (930°F), the loop was outgassed for approximately 24 hr at a pressure of 5×10^{-5} torr.

2. Charging the loop with a predetermined amount of potassium was then accomplished by inert-gas pressurization of the transfer container and a potassium sample was taken at this time.

3. Outgassing of the charged loop was then performed by applying a dynamic vacuum (5×10^{-5} torr) for approximately 16 hr while the molten potassium was held at 120°C (250°F). This was considered a most important step to rid the system of all noncondensables, which would affect the potassium boiling point.

4. The evacuation line was valved off, sealed by welding, or both.

The size of each operating charge and the oxygen contents following each loop fill of same are listed in Table 4. The exceedingly high oxygen content associated with loop No. 4 is believed to stem from contamination during sampling.

Table 4. Summary of Potassium Charges Used in Natural-Circulating Loop Tests

Loop Number	Fill Number	Potassium Admitted to Loop (g)	Oxygen Analyses (ppm)	
			Before Test	After Test
1 (type 316 stainless steel)	1	1068	400[a]	217[a]
2 (type 316 stainless steel)	1	1293		Lost
	2	1370	640[a]	200
3 (Inconel)	1	1306		Lost
	2	1281		Lost
4 (Haynes alloy No. 25)	1	1212	1550[a]	63
5 (Haynes alloy No. 25)	1	1460	640[a]	Lost
	2	1366	20	55

[a]These analyses were performed according to the n-butyl bromide method. Subsequent to this, the amalgamation method was devised. Analyses not footnoted were done using this latter procedure.

DESIGN AND OPERATION

Type 316 Stainless Steel Loop (Test No. 1)

Design

The design of the first loop test to be operated under this program is shown schematically in Fig. 3. The loop consisted of a 24-in.-long boiler, a heated vapor carry-over line, a 43-in.-long condenser-subcooler, and a preheater region. The boiler was fabricated from 2-in. sched-40 pipe, and all other components from 1/2-in. sched-40 pipe. The differently sized pipes were joined with standard reducers using full penetration gas-tungsten arc welds. Thermocouple wells were used in the boiler; thermocouples spot welded to the pipe wall measured temperatures at other locations.

The condenser section which was surrounded by an annular air duct was cooled by the passage of air in a direction countercurrent to the

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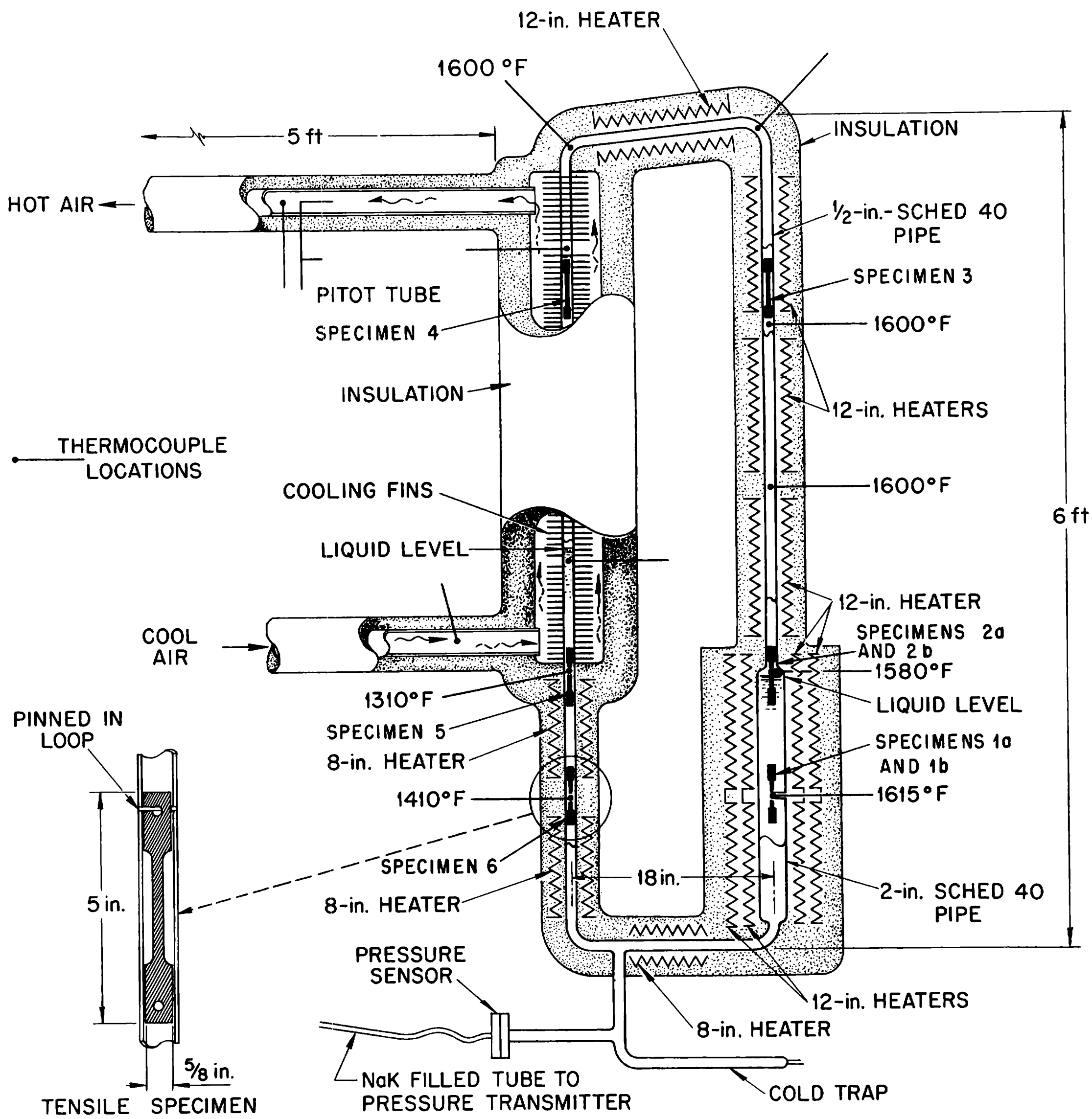

Fig. 3. Schematic of Type 316 Stainless Steel Boiling-Potassium Loop Used in First Test.

flow of potassium. Air was supplied with a 250-cfm squirrel-cage blower. To increase the cooling efficiency, nickel fins, 1/4-in. apart, were placed over the loop in this region; and the air jacket was heavily insulated to minimize extraneous heat losses. A Pitot tube, installed in the exit line of the air cooler, was used to determine the air velocity. A heat balance calculation on the air circuit was used to determine the potassium condensing rate (see Appendix A).

Two concentric sets of semicylindrical (clamshell) Nichrome heaters were used to heat the boiler. Semicylindrical heaters were placed along the vapor carry-over line to prevent condensation of vapor, as it was desired to have liquid-free, saturated vapor entering the condenser.

A diffusion cold trap on the bottom horizontal leg was intended to trap out oxide impurities during loop operation. A pressure sensor, connected just above the diffusion cold trap, monitored the potassium pressure in the loop.

Sheet tensile specimens, 0.040 in. thick, of the loop material were suspended in various regions (indicated in Fig. 1) to determine the effect of exposure to the potassium on the mechanical properties of the alloy.

Operation and Test Conditions

The loop was operated for the scheduled 3000 hr with the boiler temperature maintained at 870°C. As a precautionary measure, the test was interrupted at 1500 hr to replace the Nichrome elements in the boiler heaters.

Stable boiling prevailed during the test, as evidenced by a constant boiler temperature and only very slight pressure fluctuations in the system. These fluctuations were relatively small in magnitude (±1/4 psi) and occurred with rather high frequency, about 30 to 40/min. However, posttest examinations (to be reported more fully in a subsequent section) provided evidence of liquid carry-over into the condenser.

The average pressure in the loop during operation was 33 psia.[5] Heat balances calculated from the cooling air temperature rise and the volume of airflow through the air cooler corresponded to a potassium condensing rate of 170 g/min and a vapor velocity of 46 fps (14 m/sec) in the 1/2-in. sched-40 (0.62-in. ID) vapor carry-over line.

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[5]At the time of this test, the only vapor pressure data available gave a value of 34 psia at 870°C. More recent data from Battelle Memorial Institute indicate a value of 37 psia at 870°C. W. D. Weatherford, Jr., J. C. Tyler, and P. M. Ku, Properties of Inorganic Working Fluids and Coolants for Space Applications, WADC-TR-59-598 (AD-230065), December 1959, and A. W. Lemmon, Jr., H. W. Deem, E. A. Eldridge, E. H. Hall, J. Matolich, Jr., et al, Engineering Properties of Potassium, Final Report, NASA-CR-54017, December 31, 1963.

Type 316 Stainless Steel Loop (Test No. 2)

Design

The first type 316 stainless steel loop was followed by a second loop in which insert specimens were placed along the condenser and sub-cooler sections. Based on the operating behavior of the first loop, certain other design modifications were incorporated in this second test. These changes, shown in the schematic in Fig. 4, consisted of (1) lengthening of the boiler (from 24 to 36 in.) to minimize liquid carry-over to the condenser, and (2) the addition of a sodium jacket around the entire length of the boiler to provide more uniform heating in this region. The insert specimens in this loop consisted of 1 1/2-in.-long close-fitting sleeves (Fig. 4). The inside surfaces of alternate inserts were highly polished in order to determine if the quality of the surface finish would have any effect on the dissolution and deposition rates.

The overall loop dimensions were the same as those of the initial loop described above. The boiler pipe size (2-in. sched-40) was retained, and the sodium jacket was fabricated from a 4-in. sched-40 Inconel pipe. The double set of wrap-around heaters with Nichrome heating elements that had been used on the boiler of the first loop was replaced by a single set of heaters containing imbedded Kanthal heating elements. Heat was extracted from the condenser by the same methods as those used on the first loop. However, guard heaters were incorporated around the air jacket to eliminate heat losses in this region.

A side tube (not shown in Fig. 4) of 3/8-in. sched-40 pipe 12-in. long, equipped with a valve, was welded to the horizontal vapor carry-over line to facilitate evacuation and gas pressurization of the loop during startup and draining.

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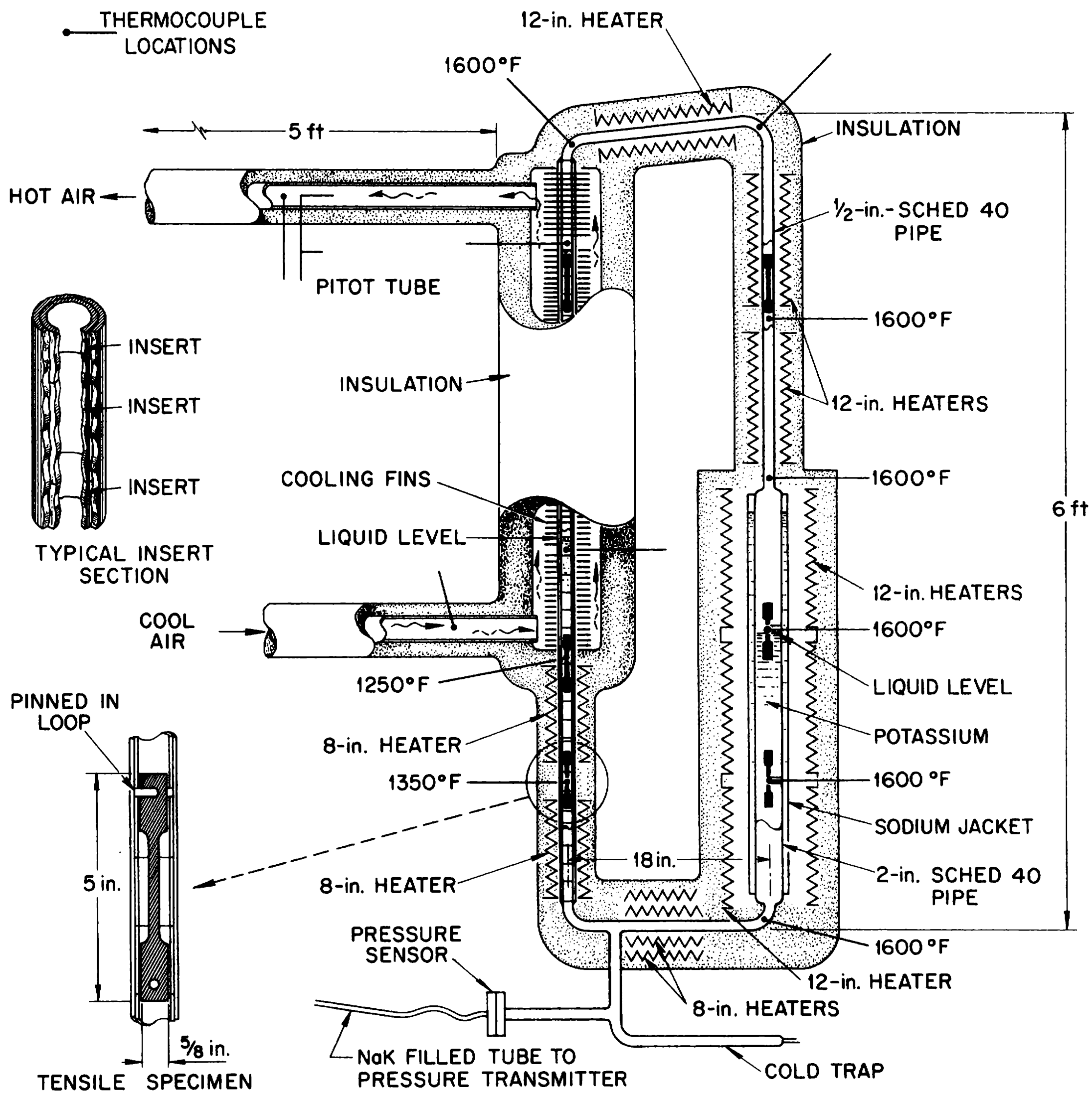

Fig. 4. Modified Boiling-Potassium Loop Containing Tubular Inserts in Condenser-Subcooler Leg and Sodium Jacket Around Boiler.

Operation and Test Conditions

From the onset of operation boiling was very unstable. The loop pressure fluctuated between 36 and 45 psia.[6] The low pressure was associated with nonboiling periods during which the boiler temperature rose abruptly. These periods were followed by sudden flashing of superheated liquid in the boiler, a concomitant temperature drop in the boiler, and a corresponding pressure surge. The fluctuations occurred on the average of every 15 sec.

After 680 hr operation of the loop, a leak[7] occurred in the vapor carry-over line in the bend above the condenser, far enough from the test section so as not to damage the inserts. The damaged section was replaced and the loop was filled with a potassium cleanup charge and operated as an all-liquid thermal-convection loop for 100 hr at 500°C. The cleanup potassium was drained and the loop was recharged with 1370 g of new potassium and restarted (see Table 4).

Heat balances calculated from the cooler circuit gave an average potassium condensing rate of 180 g/min during the test.

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[6]The design conditions were to boil the potassium and deliver the vapor to the condenser at 870°C. The observed pressure values correspond to potassium temperatures of 880 and 910°C, respectively. Due to an apparent carry-over of liquid and a subsequent cooling in the vapor line, it was necessary to drive the boiler to a higher temperature in order to deliver the material to the condenser at design conditions. The boiler temperature ranged from an average of 880°C at the bottom to 910°C in the vapor above the liquid level. The values would fluctuate approximately ±15°C due to the unstable conditions.

[7]This leak is believed to be the result of thermal fatigue cracking of the heat affected zone on the vapor line-evacuation line weld. This type of cracking, originating from boiling instabilities, will be discussed in more detail in a subsequent section.

Inconel Loop (Test No. 3)

Design

The Inconel loop design was identical to that of the second type 316 stainless steel loop described above (Fig. 4), except that none of the inside surfaces of the inserts in the condenser-subcooler region was polished.

Operation and Test Conditions

Boiling instabilities prevailed throughout the test. The instabilities were similar to those found in the second type 316 stainless steel loop although not as severe, with the pressure fluctuations ranging from 33 to 37 psia.

During operation a potassium condensing rate of 180 g/min was maintained. This value corresponded to a potassium vapor flow rate of 50 fps (15 m/sec) in the 1/2-in. sched-40 vapor carry-over line.

After 275 hr of operation at 870°C, the loop developed a leak (caused by thermal-fatigue cracking of an evacuation-line weld) near the bend at the top of the condenser. Since the failure was far enough from the test section that it did not affect the inserts, the damaged portion was cut out and replaced. The replacement pipe was butt welded to the loop and then these welds were further protected with a cover sleeve, which was fillet welded at each end. Prior to restarting, the loop was flushed by filling with potassium and operating as a thermal-convection loop for 100 hr at 500°C, emptied, and then loaded with charge for boiling operation.

After 1519 hr of operation at design conditions, the top horizontal line failed beneath a heater. The test was terminated at this time. Again, the leak was sufficiently far from the test section to permit valid weight-change measurements on the inserts. The cause of the leak is not definitely known; however, failure analyses revealed that the top horizontal pipe had sagged in the region of the failure (Fig. 5), and the amount of deflection appeared sufficient to have caused a short circuit between the pipe wall and the heating elements surrounding the pipe. However, the region adjacent to the failure point also revealed many cracks. Therefore, the possibility that the failure was due to a crack through the wall (induced by thermal fatigue caused by boiling instabilities) cannot be discounted.

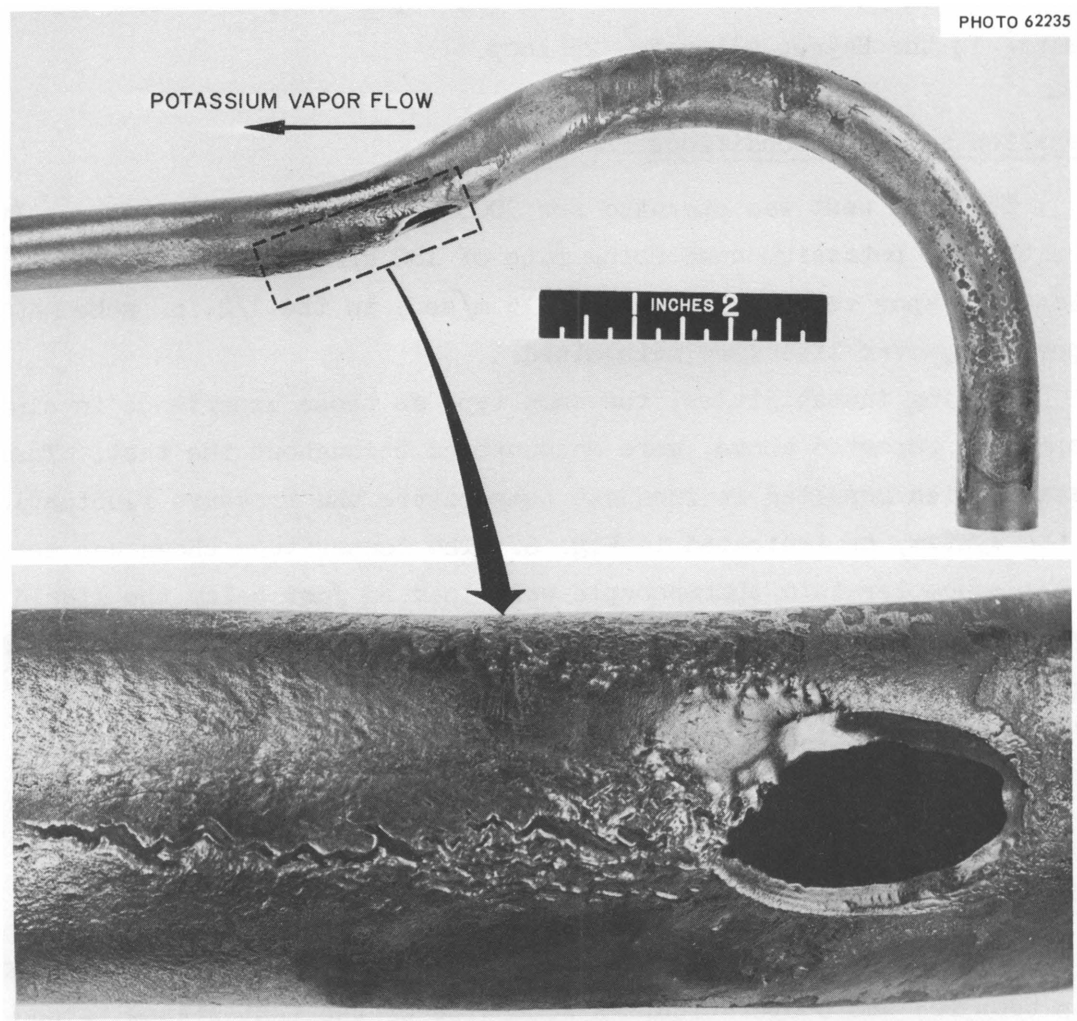

Fig. 5. Point of Failure on Top Vapor Carry-Over Line of Inconel Boiling-Potassium Loop, Operated for 1500 hr at 870°C.

Haynes Alloy No. 25 Loop (Test No. 4)

Design

The configuration of the 870°C Haynes alloy No. 25 loop test was identical to that of the Inconel and the second stainless steel loop described above. Although seamless pipe was used for the vapor carry-over line in the other loops, limited availability of material dictated that seam-welded material be used for the vapor carry-over line and inserts in the Haynes alloy No. 25 loop.

Operation and Test Conditions

The loop test was operated for 3000 hr at design conditions. During operation, a potassium condensing rate of 180 g/min (24 lb/hr) and a potassium vapor velocity of 50 fps (15 m/sec) in the 1/2-in. sched-40 vapor carry-over line were maintained.

Boiling instabilities, the same type as those experience in similar loop tests reported above, were encountered throughout the test. These instabilities resulted in frequent temperature and pressure fluctuations in the system, as indicated in Fig. 6. The temperature shown was measured in the subcooler in a thermocouple well located just below the liquid level. As can be seen, pressure surges due to flashing of superheated liquid in the boiler were followed by temperature rises as large as 70°C. These surges occurred on an average of every 90 sec.

Haynes Alloy No. 25 Loop (Test No. 5)

Design

Several modifications were made in fabricating a Haynes alloy No. 25 loop test for 980°C operation. A schematic of the test system is shown in Fig. 7, and Fig. 8 shows the fabricated loop before installation of the heaters and insulation.

In place of the short tubular inserts used in the other designs, the condenser-subcooler pipe itself consisted of twelve very accurately machined and weighed cylinders, each approximately 9 in. long, which were butt welded together. With this arrangement, a weight per unit

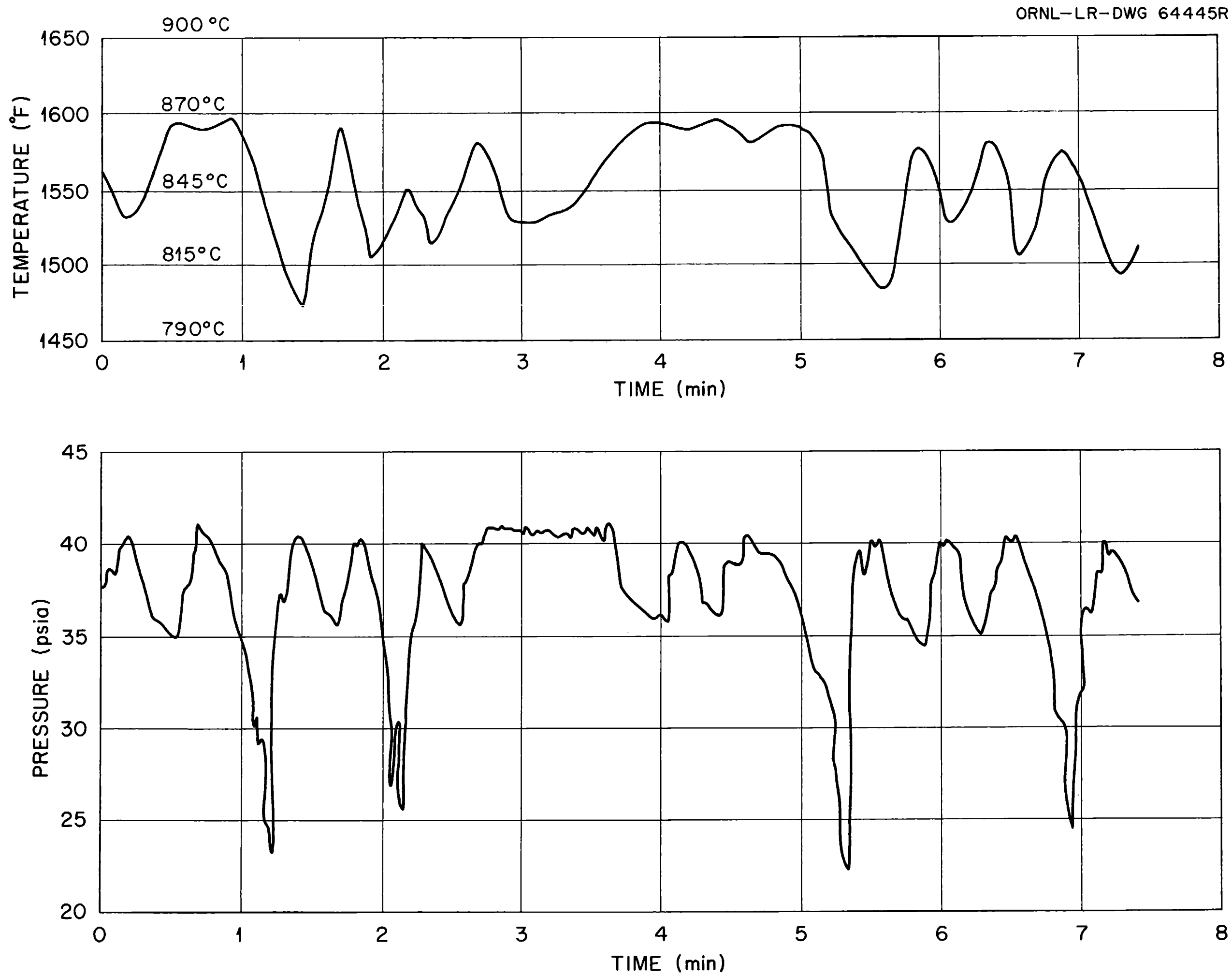

Fig. 6. Temperature and Pressure Profiles for Haynes Alloy No. 25 Boiling-Potassium Loop Test No. 4.

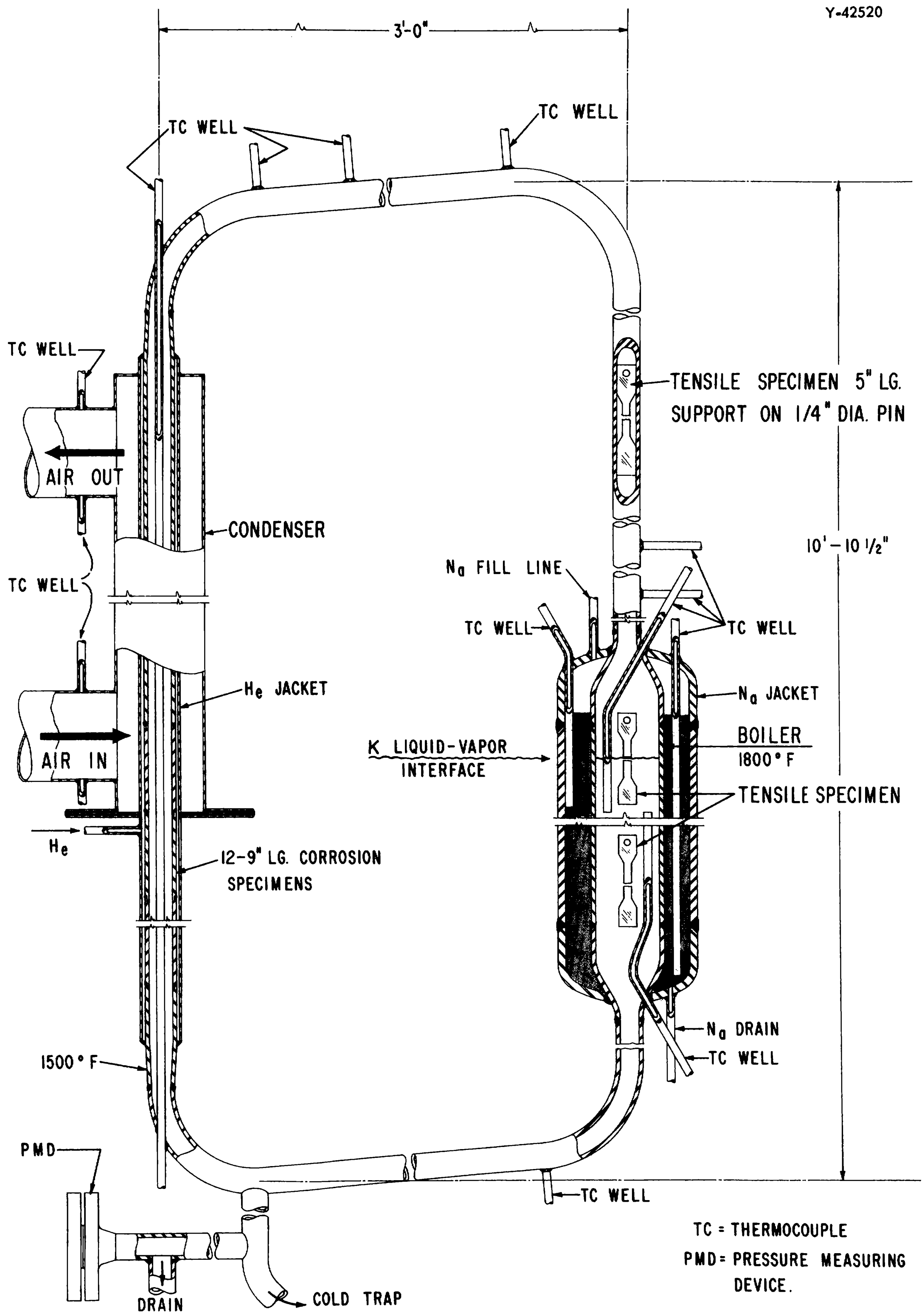

Fig. 7. Haynes Alloy No. 25 Natural-Circulation Boiling-Potassium Corrosion Loop (Test No. 5) for 980°C Operation.

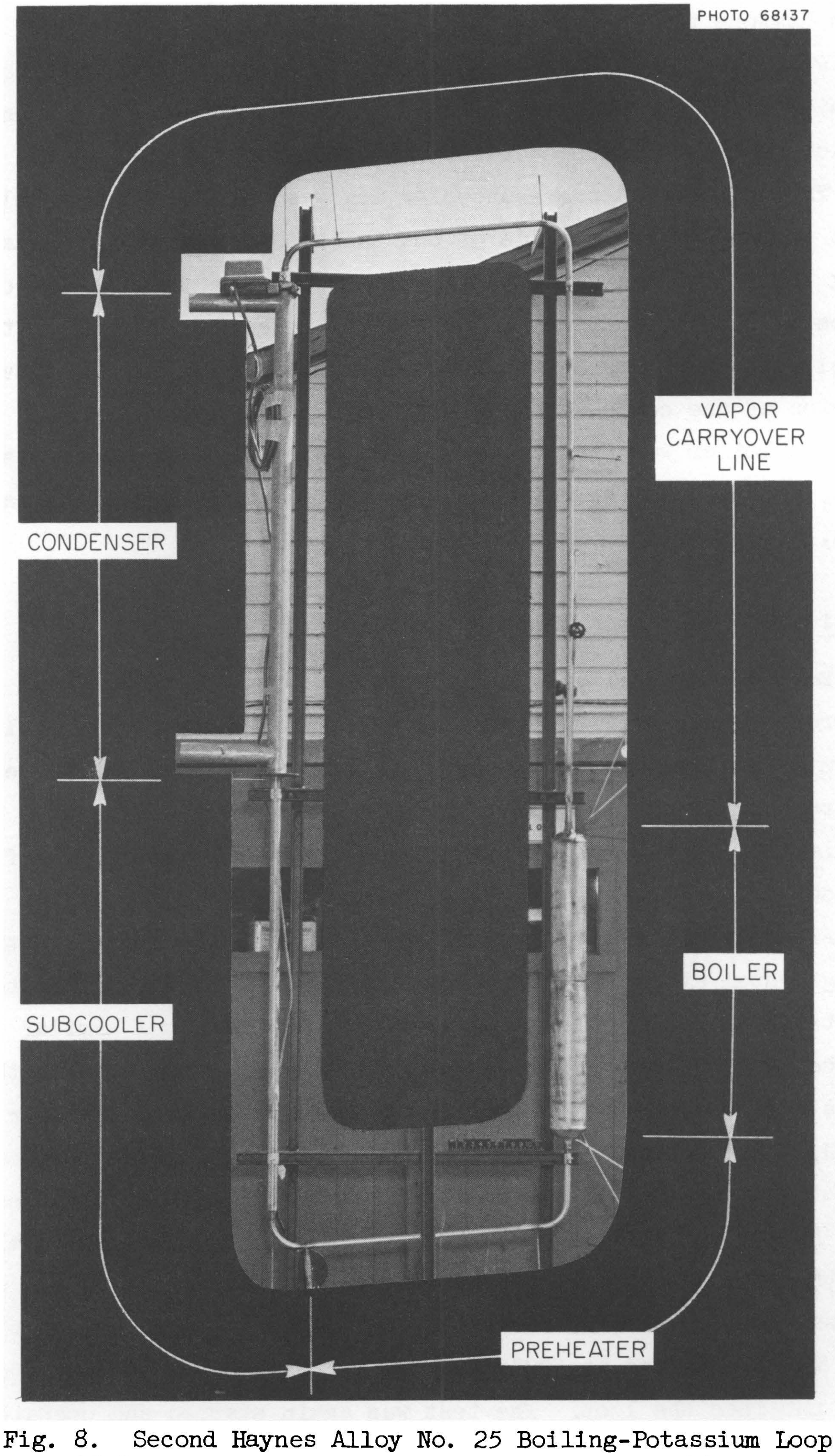

Fig. 8. Second Haynes Alloy No. 25 Boiling-Potassium Loop (Test No. 5) for 980°C Operation; Boiler is on the Lower Right.

length "before test" was obtained for each insert. After test, the butt welds were machined away and the resulting tubular "specimens" accurately dimensioned and weighed. From these data, weight changes per unit area were determined for the condenser and subcooler regions.

The entire condenser-subcooler leg was shielded with a helium jacket to protect the outside of the machined inserts from oxidation during test. Cooling air impinged on the outer surface of the condenser portion of this helium jacket. A specially designed diaphragm-type Inconel bellows was used at the bottom of the air cooler to provide for expansion of the condenser-subcooler leg.

The boiler was fabricated from a 2-in. sched-40 pipe. A sodium jacket, used around the boiler to provide a uniform heat flux in this region, was fabricated from 1/4-in.-thick plate.

Operation and Test Conditions

Boiling instabilities again plagued loop operation. With the test at operating temperature (980°C) the pressure in the system cycled a maximum of 35 psi, between 45 and 80 psia. (The potassium temperature corresponding to 80 psia is 980°C.) Boiler temperature changes of approximately 10°C accompanied the large pressure cycles. The frequency of these large cycles varied from 20 sec to 1 hr.

After approximately 100 hr operation, a leak occurred in the helium cooling system at the Inconel bellows. This was repaired, the test was restarted, and the system was operated under conditions similar to those described above until 760 hr operation had been accumulated by the test. At this time, a leak occurred in the horizontal vapor carry-over line (Fig. 9). Failure analyses indicated the rupture was due to cracking in the fusion zone of the seam weld extending longitudinally along the pipe. Due to the brittle nature of the 1/2-in. sched-40 seam-welded pipe, the entire vapor carry-over line was cut out and replaced with seamless 3/4-in. sched-40 material. With this accomplished, a cleaning charge was circulated at 500°C for 100 hr, dumped, and a second operating charge put into the loop. The test was again started and operated under similar conditions, as indicated above, until a total of 3000 hr had been accumulated.

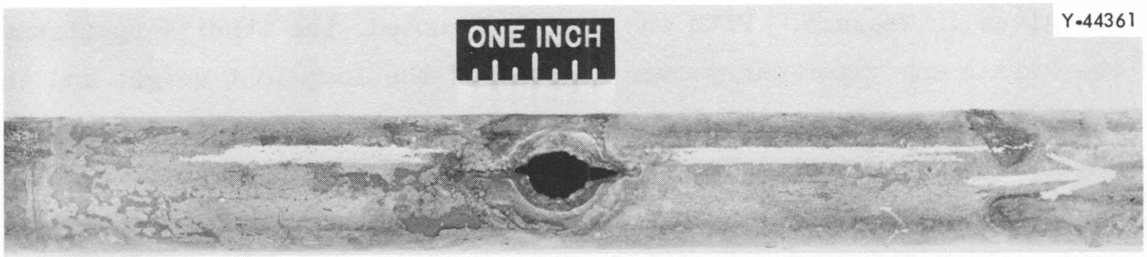

Fig. 9. Failure in the Horizontal Vapor Carry-Over Line of Haynes Alloy No. 25 Boiling-Potassium Loop Test After 760 hr of Operation. Failure occurred in the weld fusion zone of the pipe.

During operation at design temperature, a condensing rate of 300 g/min was maintained. The equivalent vapor velocity in the 1/2-in. sched-40 pipe was 60 fps (18 m/sec) while the vapor in the 3/4-in. sched-40 replacement pipe was 27.5 fps (8.4 m/sec).

TEST TERMINATION

All loop tests were terminated in essentially the same manner. At the scheduled time, the potassium was drained from the loop through a 10-μ pore size filter into a drain tank. A sample of the potassium bath was taken at this time.

The residual potassium after draining was removed by distilling at approximately 400°C for 24 hr.

After distillation, the inserts and tensile specimens were carefully removed for weight-change measurements. The loops were sectioned into their several components and usually split longitudinally for visual examination. Samples from the various regions were selected for metallographic examinations and chemical analyses.

RESULTS

Type 316 Stainless Steel Loop (Test No. 1)

Weight Changes

Weight-change determinations in loop No. 1 were made by means of tensile specimens suspended at six different positions around the loop. Results are given in Table 5. With one exception noted, the tensile specimens from the boiler and vapor carry-over regions of the loop lost weight and the specimens in contact with liquid potassium in the subcooler and preheater regions gained weight. The weight losses are attributed to dissolution of material in the hotter regions of the loop and the weight gains to the subsequent deposition of material in the cooler regions.

The maximum weight loss recorded (−11 mg/in.^{2}) is equivalent to a uniform reduction in wall thickness of 0.08 mil.

The weight gain on specimen No. 3 is attributed to a pickup of material by physical contact with the anchor pin (Fig. 10).

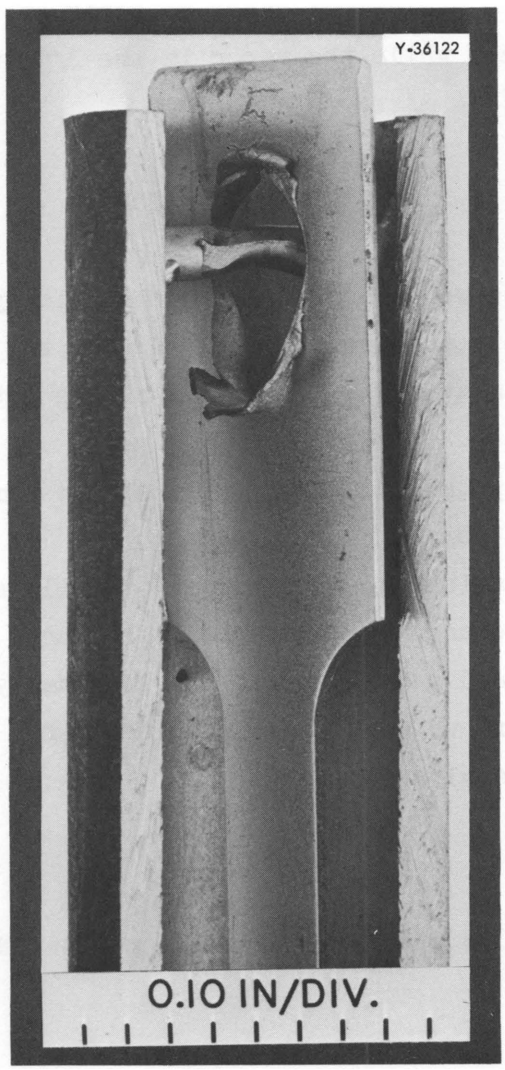

Fig. 10. Anchor Point of Tensile Specimen No. 3 From the First Type 316 Stainless Steel Boiling-Potassium Loop. The specimen, located in the vapor carry-over line above the boiler, shows damaged hole presumably from impacts by slugs of liquid potassium from the boiler. Reduced 14%.

Table 5. Weight-Change and Room-Temperature Mechanical Property Data on Type 316 Stainless Steel Sheet Tensile Specimens Following Exposure in First Boiling-Potassium Loop for 3000 hr

Gage Length Dimensions: 2 × 0.25 × 0.040 in.

Specimen Number[a]	Test Environment	Exposure Temperature (°C)	Weight Change (mg/in.^{2})	Mechanical Property Data		
				Yield Strength (psi)	Tensile Strength (psi)	Elongation in 2-in. Gage (%)
				× 10^3	× 10^3	
1-A	Boiler, liquid	870	−11.0	23.9	78.6	56
1-B	Boiler, liquid	870	−10.6	23.8	78.0	58
2-A	Boiler, liquid-vapor interface	870	−8.9	23.7	79.3	57
2-B	Boiler, liquid-vapor interface	870	−8.7	22.0	80.7	56
3	Vapor, hot leg	870[b]	+8.2[c]	21.8	79.5	61
4	Vapor, condenser	805[b]	−4.1	22.4	80.5	56
5	Cold Leg, subcooler	710[b]	+29.6	32.3	86.2	43
6	Cold Leg, subcooler	765[b]	+18.1	29.1	86.5	44
Control-1	Vacuum	870		26.7	80.1	55
Control-2	Vacuum	805		30.7	90.9	48
Control-3	Vacuum	765		30.6	90.8	48
Control-4	Vacuum	710		30.5	88.4	52

[a]Specimen locations and numbers are identified in Fig. 3.

[b]Exterior wall temperatures; other temperatures were determined by means of thermocouples projecting into the center of the pipe in wells.

[c]Specimen mechanically damaged during test by "bumping"; the damage affected its weight.

Visual and Metallographic Results

The maximum attack observed in the loop occurred on the inside boiler wall in the vicinity of the liquid level. A number of specimens from this locality were examined, and the attack appeared to be limited to one particular quadrant of the pipe circumference. Subsurface voids were found to a depth of 2 mils extending for a distance of 70 mils longitudinally along the boiler wall. In Fig. 11 the maximum attack in this vapor-interface region is compared to a region of the boiler below the interface. The depletion of precipitate seen at the surface of both specimens is attributed to the removal of carbon and chromium. This is discussed in more detail under "Chemical Results."

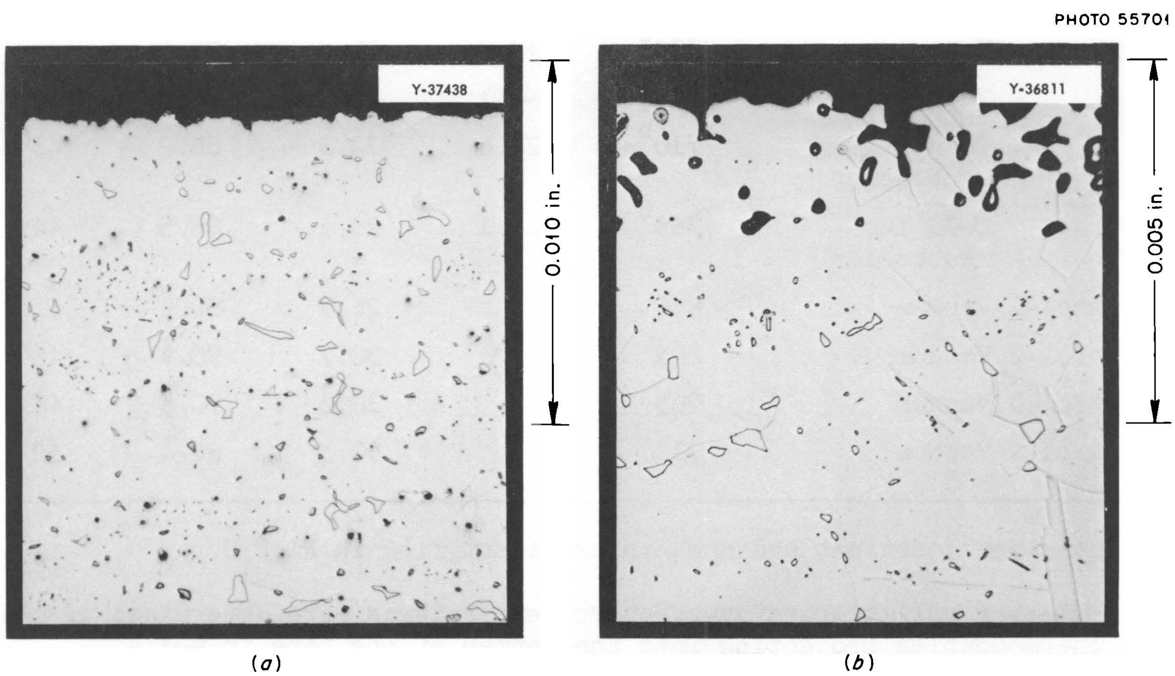

Fig. 11. Sections of the Surface of the Boiler Wall From (a) A Region Below the Liquid Level and (b) at the Liquid-Vapor Interface of Test No. 1. Etchant: aqua regia.

Metallographic examination of the inner walls of the condenser and subcooler regions disclosed that carbon was being removed from the condenser and was subsequently being deposited on the wall of the subcooled liquid region. This is illustrated in Fig. 12. The discontinuous globules seen in the photomicrograph of the condenser (Fig. 12a) were identified by preferential etching (see Appendix C) as either chi or sigma phase. X-ray diffraction patterns on material extracted from the matrix clearly identified this phase as sigma (FeCr).

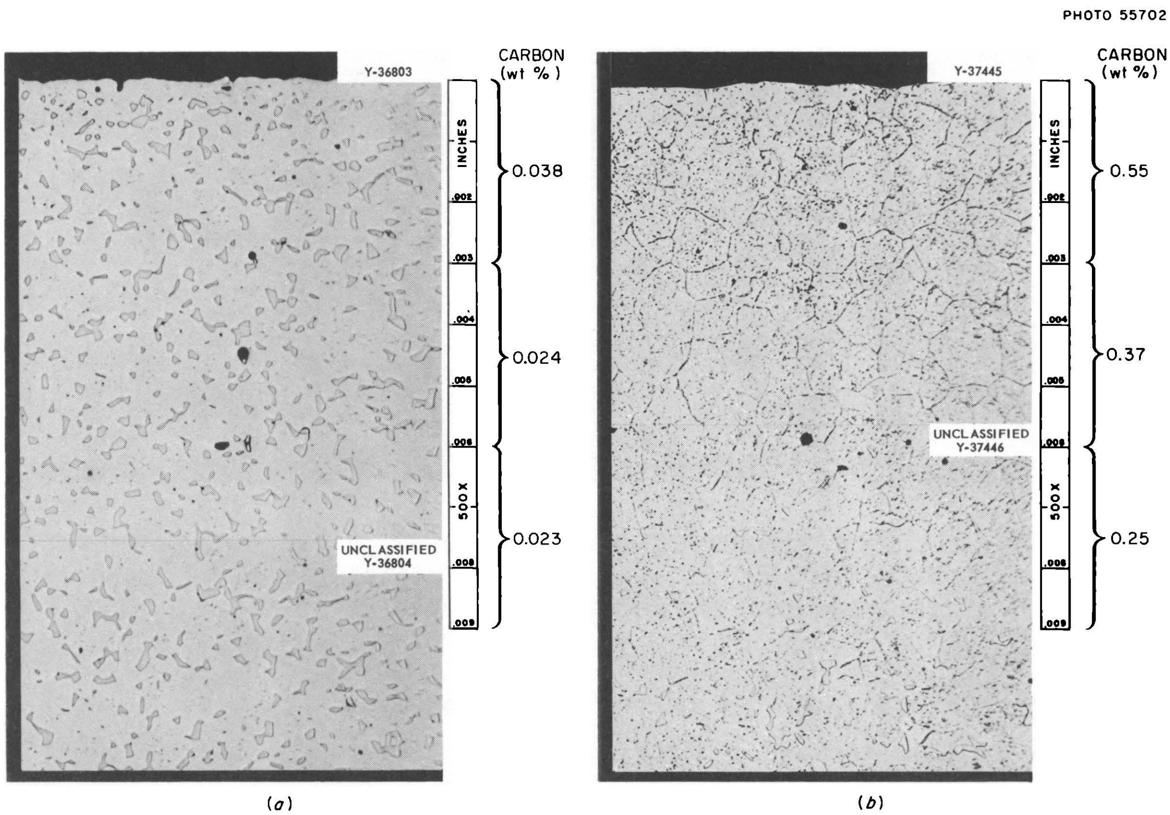

Fig. 12. Pipe Wall From (a) Top of Condenser and (b) From Subcooler. The carbon gradient shown indicates removal from the condensing region and subsequent deposition in the cooler liquid region (b). Etchant: aqua regia. Reduced 37%.

Deposition of material on the inside surface of the subcooler manifested itself with a general darkening of the loop walls. The extremity of these mass-transfer deposits can be seen in Fig. 13.

As indicated previously, 0.040-in.-thick tensile specimens were suspended in various locations around the loop. Surface etching was

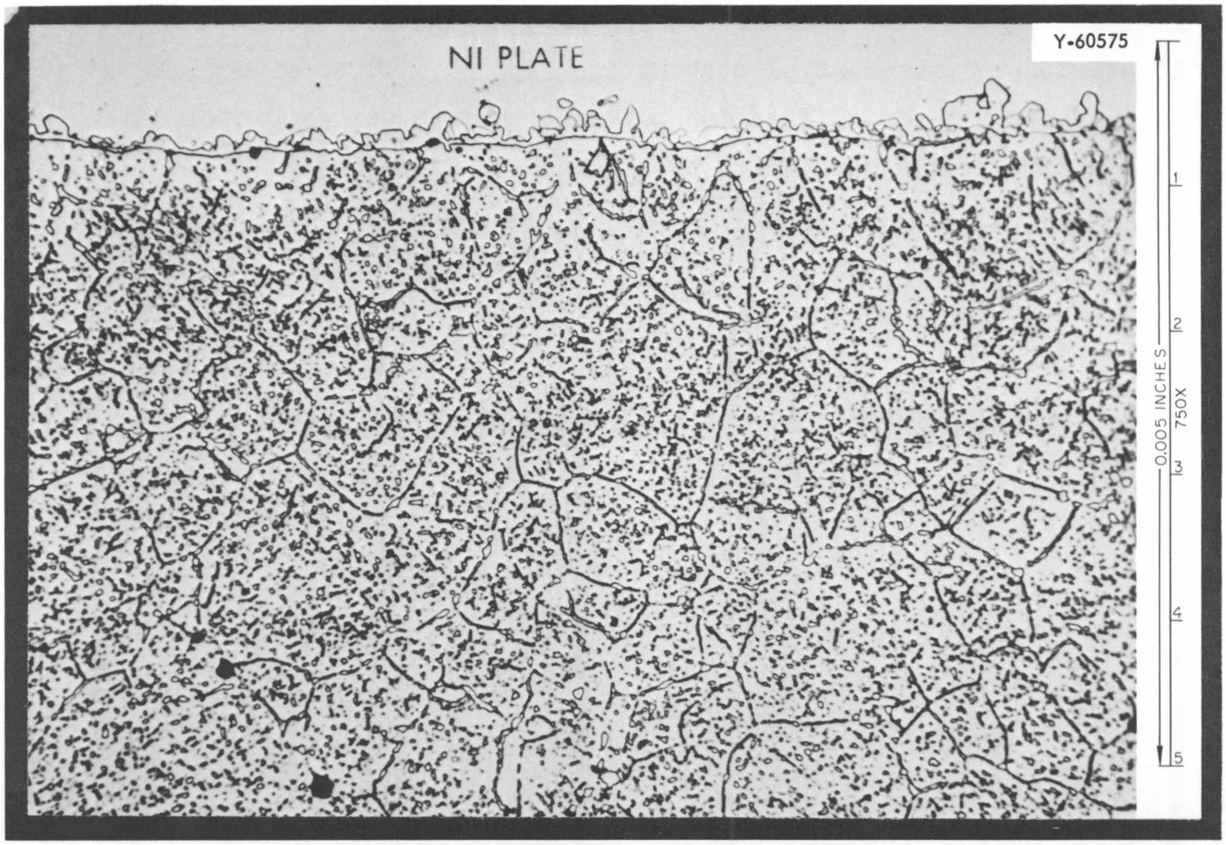

Fig. 13. Mass-Transfer Deposits Observed on the Surface of the Subcooler in Loop Test No. 1. Specimen was nickel plated to preserve edge during metallographic preparation. Etchant: lactic acid, HNO_3, HCl.

visually apparent (Fig. 14) on the tensile specimens that had been subjected either to liquid potassium in the boiler or potassium vapor in the vapor carry-over line. The surfaces of the two specimens located in the subcooled liquid regions were slightly darkened (specimens 5 and 6 in Fig. 14). Note that specimen 3 in Fig. 14 sustained heavy damage at the point where it was anchored to the loop, indicating that a substantial force was acting to tear it from its mounting. The specimen prior to its removal from the loop is pictured in Fig. 10. The tensile specimen was obviously damaged by a repetitive vertical bumping action. In view of the small cross-sectional area presented by the specimen to the flowing potassium and the small pressure drop associated with it, the dry vapor flow fluctuations seem unlikely to have moved the specimen with the force implied by the extent of damage. It is therefore concluded that periodic entrainment of liquid slugs in the potassium vapor resulting from boiling instabilities caused the damage.

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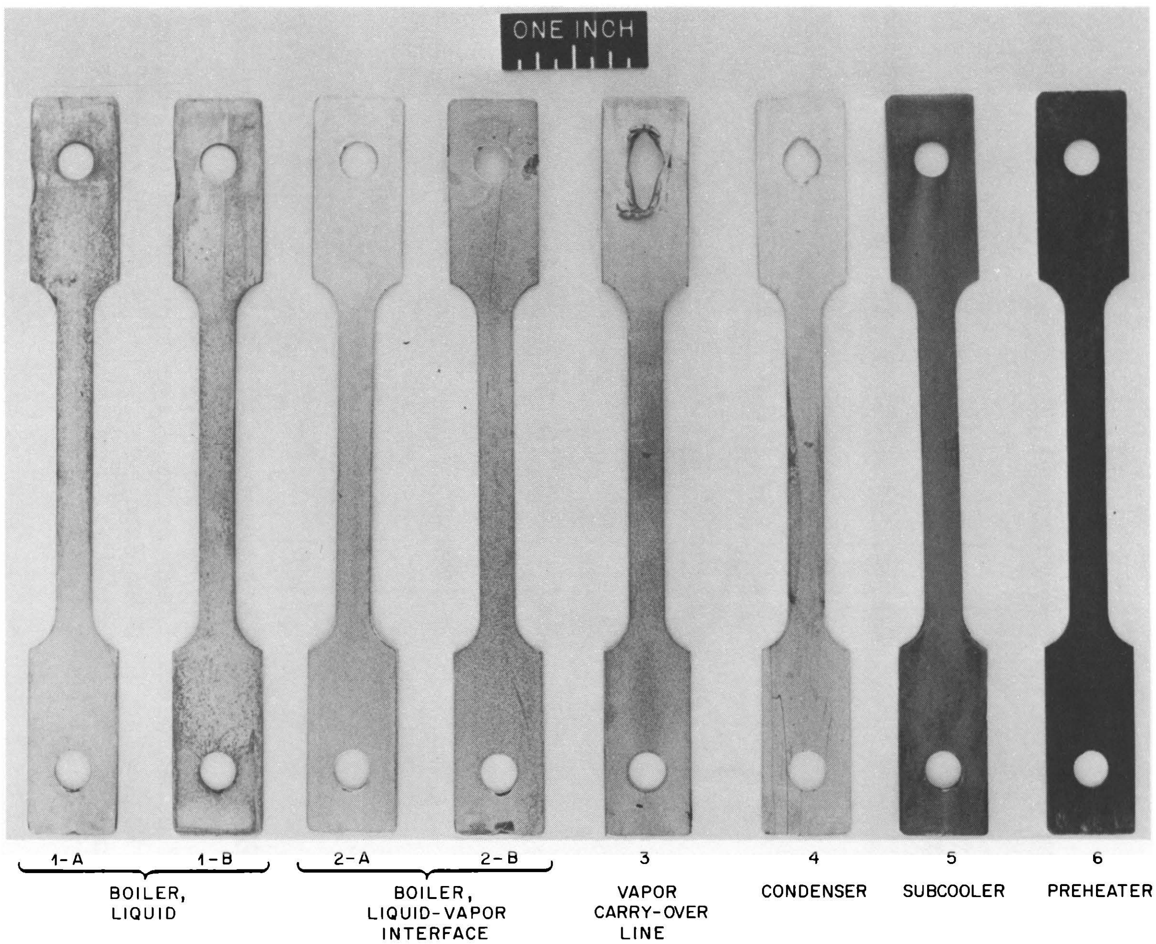

Fig. 14. Tensile Test Specimens Taken From Various Sections of the First Type 316 Stainless Steel Boiling-Potassium Loop, Which Operated for 3000 hr at 870°C. The location of each specimen during the test is indicated. Reduced 38%.

The surfaces of tensile specimens from the boiler and subcooler regions of the loop are shown in Fig. 15. A slight surface dissolution of the boiler specimen at grain boundaries is evident, while the specimen from the subcooler was covered with a porous mass-transfer deposit to a depth of approximately 3 mils. Photographs of the shoulders on these tensile specimens at 9× are shown in Fig. 16.

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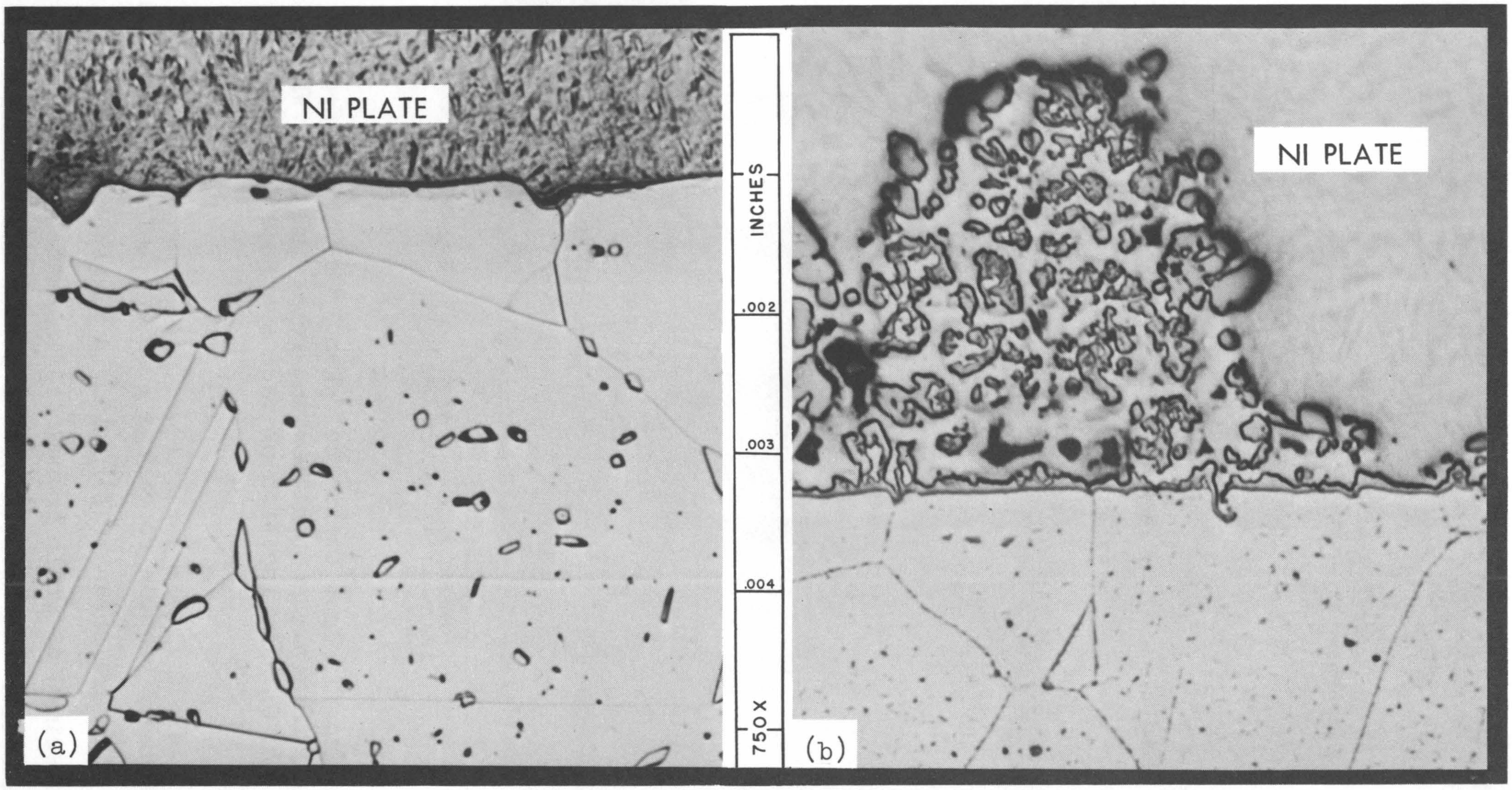

Fig. 15. Surfaces of Type 316 Stainless Steel Tensile Specimens From (a) Boiler (Liquid, 870°C) and (b) Subcooler (Liquid, 710°C) Following 3000 hr Exposure in Boiling-Potassium Loop Test No. 1. The deposited crystals in (b) were found to contain $Cr_{23}C_6$. Specimens were nickel-plated to preserve edge during metallographic polishing. Etchant: glyceria regia.

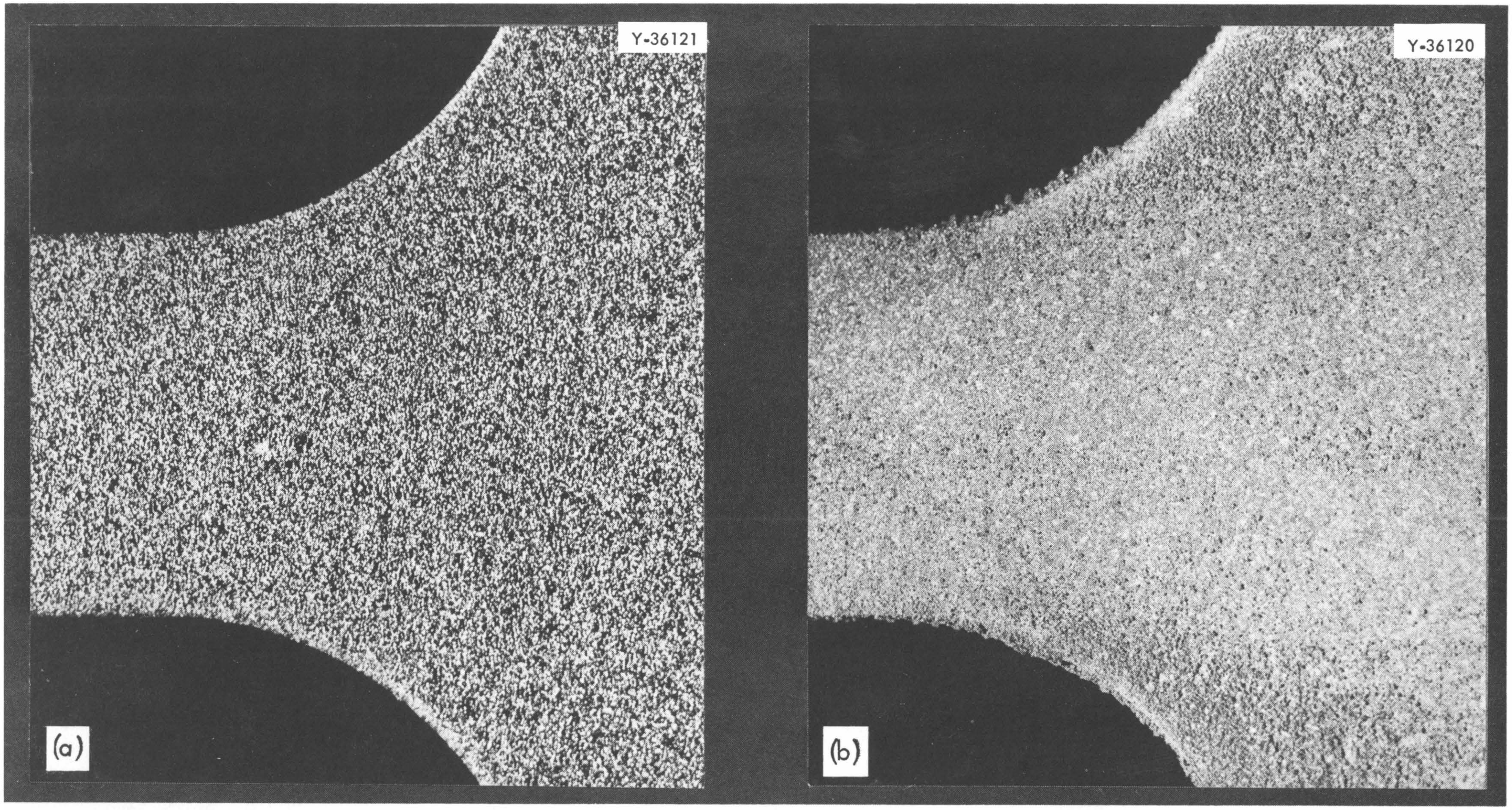

Fig. 16. Photomacrographs of Sheet Tensile Specimens From (a) Vapor Zone (870°C) and (b) Subcooler Region (710°C) of Loop Test No. 1. The shiny, etched appearance on (a) and the buildup of mass-transfer deposit on (b) can be seen.

Figure 17 shows a highly magnified cross section of a tensile specimen located at the liquid level in the boiler. A feathery, ferrite-bearing structure can be seen at the surface. X-ray analyses on this surface indicated the presence of alpha-iron. This is attributed to the preferential leaching of austenite-stabilizing elements and is discussed in more detail later. This type structure was not detected on the boiler wall adjacent to the same tensile specimen.

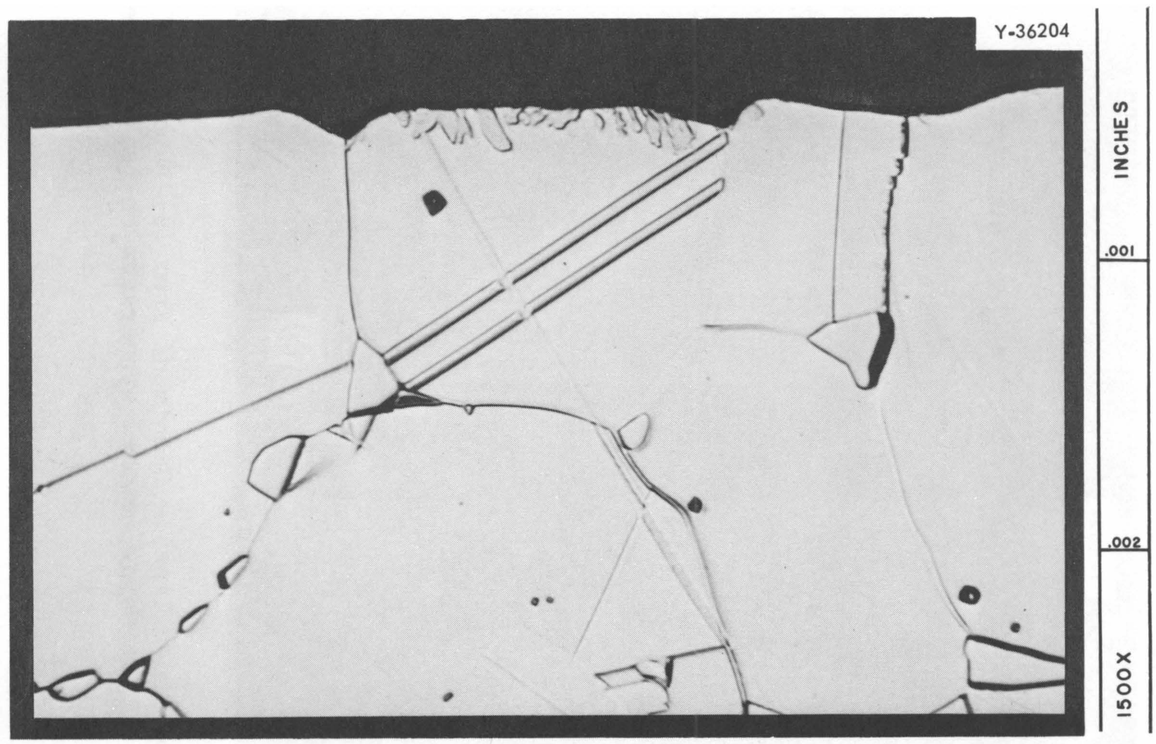

Fig. 17. Surface of Tensile Specimen Located at the Liquid-Vapor Interface in the Boiler of Loop Test No. 1. A feathery, ferritic phase can be seen at the surface. Etchant: glyceria regia.

When this loop test was started (March 1960), problems associated with boiling instability in potassium systems were still to be fully recognized. As already discussed, relatively stable boiling prevailed during this first test. However, subsequent natural-circulation loops were plagued with severe instabilities. More recent studies[8] have shown

[8]E. E. Hoffman, "Boiling-Potassium Stability Studies," Metals and Ceramics Division Ann. Prog. Rept. May 31, 1963, ORNL-3470, pp. 114–118.

that stable boiling is produced by the activation of vapor-bubble nucleation sites. The nucleation site in this first type 316 stainless steel test appears to have been a "built-in" crack produced during welding of the thermocouple well into the boiler wall. Figure 18 shows the location and configuration of this nucleation site. At high magnification, the effect of the boiling potassium on the fissure can be seen as a series of subsurface voids and a "wormhole" type attack.

A series of four weld samples from the butt weld located at the bottom of the boiler (exposed to liquid potassium at 815°C) and four weld samples from the butt weld at the top of the boiler (exposed to potassium vapor at 870°C) were examined metallographically. No evidence of attack could be found in these welds.

Chemical Results

As indicated above, selective removal of material from the exposed surface in the boiler was suggested in metallographic observations (Fig. 11). To investigate the nature of this preferential leaching, successive 0.003-in.-thick layers from the inside wall of the liquid region of the boiler were machined, analyzed, and compared to the "before-test" analyses. The results of these analyses are shown in Table 6 and indicate preferential removal of chromium, carbon, nitrogen, oxygen, and possibly nickel.

Perhaps the most significant chemical analyses were those obtained from the surfaces of the condenser and subcooler regions. Turnings machined in 3-mil increments to a depth of 9 mils were taken from each region in order to determine whether concentration gradients existed in the wall as a result of the exposure to potassium. The turnings were analyzed for iron, nickel, chromium, molybdenum, and carbon, and the results are listed in Table 7. The most significant changes in composition were depletion of carbon and, to a slight extent, chromium from the inside surface layer of the condenser and enrichment of these elements in the subcooler region. The carbon depletion from the condenser wall is in accord with the carbide-free-surface zone, apparent in metallographic examinations (Fig. 12). The results in Table 7 suggest that the

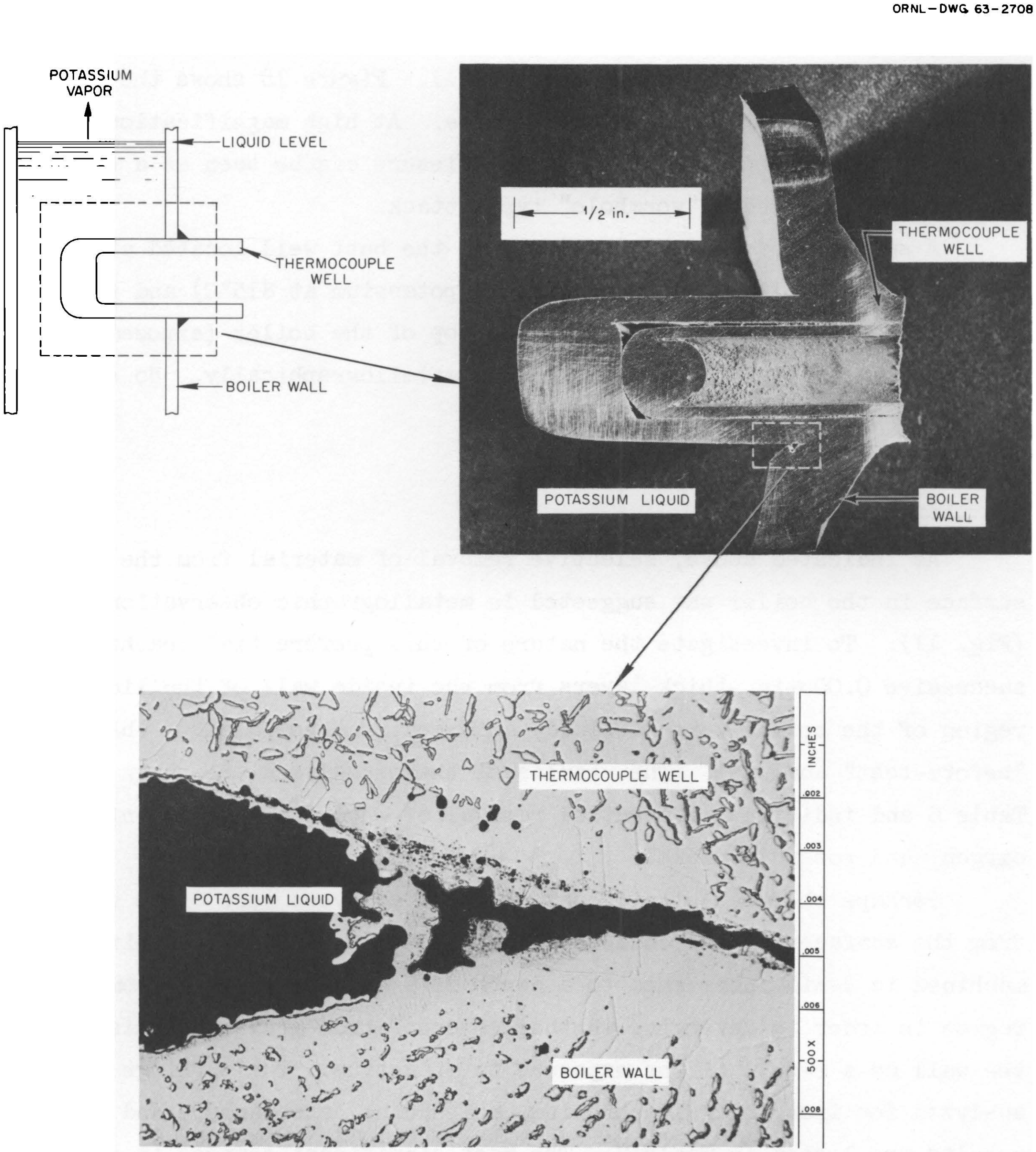

Fig. 18. Configuration of Favorable Vapor-Bubble Nucleation Site Located at the Boiler Wall-Thermocouple Well Interface of the First Type 316 Stainless Steel Boiling-Potassium Natural-Circulation Loop, Operated for 3000 hr at 870°C. Reduced 38%.

Table 6. Analyses of Type 316 Stainless Steel Incremental Layers From Liquid Region of Boiler Wall Before Test and After 3000-hr Exposure to Potassium at 870°C (Loop Test No. 1)

Sample	Element (wt %)						
	Fe	Ni	Cr	Mo	C	O	N
Before Test	65.14	13.92	15.90	2.03	0.043	0.051	0.046
After Test[a]							
0[b]–3	67.14	13.39	15.27	2.06	0.026	0.020	0.008
3–6	66.46	13.58	15.58		0.020	<0.001	0.006
6–9	65.47	13.55	16.05	2.02	0.020	0.001	0.007

[a]After-test analyses made on successive 3-mil-thick layers machined from the inside surface.

[b]Inside surface.

Table 7. Chemical Analyses of Machine Turnings From Surface of Condenser and Subcooler of Type 316 Stainless Steel Boiling-Potassium Loop Test No. 1, Operated for 3000 hr at 870°C

Location of Sample	Depth of Turnings	Materials Analyzed				
		Fe	Ni	Cr	Mo	C
Condenser, 870°C	0[a]–3	67.85	10.71	17.08	2.49	0.038
	3–6	68.07	10.57	17.05	2.38	0.024
	6–9	68.67	10.67	16.58	2.35	0.023
Subcooler, 710°C	0[a]–3	65.44	11.36	18.61	2.43	0.550
	3–6	68.36	10.69	17.21	2.43	0.370
	6–9	67.85	10.83	17.22	2.44	0.250
As-Received Material		67.63	10.35	17.28	2.40	0.080

[a]Inside surface of specimen.

dissolution of metallic constituents at the condenser wall was almost completely nonselective in character. The lack of any selective leaching was further substantiated by results of an electron-beam microprobe analysis made on a sample from the condenser. This was conducted by running a traverse from the inside surface of the condenser wall into the interior of the specimen for a distance of 20 mils. Results of scans to determine the compositional gradients of nickel, chromium, molybdenum, and iron indicated that the condensing potassium had no detectable selective leaching effect on these elements in the alloy.

Following the completion of tensile tests,[9] sections of tensile specimens near the point of fracture were analyzed for carbon. These analyses, listed in Table 8, indicate that carbon was lowered in all the specimens that were exposed to the hottest potassium, the final carbon concentrations ranging from 0.019 to 0.024 wt %. Further, the carbon content in specimens located in the subcooler increased to a level of 0.17 to 0.19 wt %. The "before-test" carbon content was 0.06 wt %. This carbon migration pattern is the same as was found for the adjoining wall specimens (Tables 6 and 7).

It has already been mentioned that deposits of crystals were observed on both the tensile specimens and the loop wall in the subcooler region of the loop after test (see Fig. 16b). The chemical analyses of these crystals, given in Table 9, indicate that the material is preferentially enriched in chromium, nickel, and manganese relative to the container alloy. The presence of chromium is further substantiated by x-ray analyses, which yielded diffraction patterns matching $Cr_{23}C_6$. These deposits must owe their origin to solute-bearing liquid arriving at the subcooler through one or more of the following sources: (1) liquid condensate from the condenser, (2) liquid carry-over expelled from the boiler through the condenser, or (3) back diffusion of saturated liquid from the boiler to the subcooler. As discussed later, evidence that condensate did transport some solute into the subcooler was attested by the loss in weight which occurred for tensile specimens hung in the vapor and condenser

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[9]Results of the tensile tests are presented in a later section of this report.

Table 8. Carbon Analyses of 0.040-in.-Thick Sheet Tensile Specimens[a] Taken From Type 316 Stainless Steel Boiling-Potassium Loop Test No. 1, Operated for 3000 hr at 870°C

Position in Loop	Specimen Number	Temperature (°C)	Carbon Content (wt %)
Boiler, liquid	1	870	0.019
Boiler, liquid-vapor interface	2	870	0.019
Hot Leg, vapor	3	870	0.024
Condenser, vapor	4	815	0.020
Subcooler, liquid	5	705	0.190
Subcooler, liquid	6	765	0.170

[a]As-received analysis: 0.06 wt % C.

Table 9. Chemical Analyses of Mass-Transfer Crystals Found on a Sheet Tensile Specimen Taken From Subcooler (705°C) of Boiling-Potassium Loop Test No. 1 After 3000 hr Exposure

Material	Analyses (total μg)	Analyses Adjusted to 100%	Type 316 Stainless Steel Composition (wt %)
Iron	26	13	68
Nickel	68	33	11
Chromium	87	43	16
Manganese	20	10	2 (max, nominal)
Molybdenum	2	1	2–3

regions. However, dissolution appears to have been quite nonselective with respect to metallic constituents comprising the condenser (see Table 7), and chromium should have amounted to no more than 17% of the total metallic species carried in by condensate. This amount of chromium appears small in comparison with the mass of chromium-rich material found in the subcooler. Therefore, it is surmised that the subcooler deposits originated either from the liquid carry-over or was formed by back diffusion in the low-velocity liquid (i.e., from the boiler), through the preheater, and up to the subcooler region. This thesis is also borne out by the selective removal of carbon and chromium (Table 6) from the boiler wall and the formation of alpha-iron on the surface of specimens located in this region (Fig. 17).

Tensile Tests

Room-temperature tensile tests with the sheet specimens mentioned earlier are reported in Table 5. The control specimens were heat treated in vacuum for 3000 hr to provide a reference for evaluating the combined effects of heat treatment and exposure to potassium. No large differences in the mechanical properties of these specimens were found. However, the yield strengths of the specimens from the hotter regions were slightly lower than those of specimens from the cooler regions of the loop and the control specimens.

Hardness Measurements

Hardness surveys were conducted on metallographic specimens cut from the condenser and subcooler region (Fig. 12). Indentations were taken every 3 mils across the total wall thickness of the condenser-subcooler. The range was 173 to 194 DPH, with an average of 184 DPH, for the condenser wall specimen (Fig. 12a), and 194 to 219 DPH, with an average of 206 DPH, across the subcooler wall.

Oxidation Resistance of the Loop Material

Investigations have shown that accelerated oxidation occurs on commercial heat-resistant alloys if no atmospheric circulation occurs and if volatile metal oxides such as molybdenum oxide (MoO_3) are present (type 316 stainless steel contains 2 to 3% Mo).[10] Consequently, the possibility of any catastrophic oxidation of the loop material at the insulated boiler and hot vapor areas was of considerable interest. Metallographic examination of the outside surface of the boiler indicated a maximum oxide layer thickness of only 3 mils and a penetration in the form of stringers approximately 1-mil deep. No evidence of accelerated attack was found.

Deposit in Cold Trap

Approximately 0.5 g of a black, flaky deposit was found in the diffusion cold trap and in a 10-μ filter through which the potassium was filtered upon termination of the loop. The analyses of this material are shown in Table 10. The analyses of the iron, nickel, and chromium, adjusted to 100%, approximate the nominal composition of type 316 stainless steel with the exception of the carbon content. X-ray analyses on this flaky deposit indicated a lattice parameter of 3.65 A, while that of type 316 stainless steel is in the 3.58 to 3.60 A range.

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[10]A. de S. Brasunas and N. J. Grant, "Accelerated Oxidation of Metals at High Temperatures," Trans. Am. Soc. Metals **44**, 1144 (1952).

Table 10. Analyses of Deposit Taken From Cold Trap of First Boiling-Potassium Type 316 Stainless Steel Loop Test (870°C – 3000 hr)

Material	Weight (%)	Adjusted to 100% (Fe, Ni, Cr)	Type 316 Stainless Steel (nominal)
Iron	26.86	60	70
Nickel	8.37	19	12
Chromium	9.60	21	17
Potassium	2.66		
Carbon	19.00		0.1

The high carbon concentration of this material may have resulted from precipitation of carbon or a carbon-rich phase associated with the depletion of carbon in the hotter sections of the loop.

Potassium Pickup by Type 316 Stainless Steel

Evidence that potassium had been picked up by the type 316 stainless steel was indicated by the analyses of a 20-mil-diam wire of this material used as a hanger for the sheet tensile specimens in the boiler. The "before-test" potassium content was 20 ppm while the "posttest" sample analyzed 35 ppm K. The amount of potassium picked up by the stainless steel also appeared to depend on the ambient temperature. Tensile specimens from the 870 and 765°C regions of the loop analyzed 100 and 65 ppm K, respectively. These increases are from a 35-ppm K content before test. This additional potassium content was not detected metallographically. The amount present in the specimens does not appear to have any effect on the mechanical properties of the alloy.

Type 316 Stainless Steel Loop (Test No. 2)

Weight Changes

As noted earlier, the second type 316 stainless steel loop incorporated forty-four 1 1/2-in.-long tubular inserts in the condenser-subcooler and vertical portions of the preheater. The inserts were intended to yield a continuous weight-change profile for these areas to obtain quantitative information on dissolution and subsequent deposition of material at a known potassium condensing rate. The inside surface of of alternate inserts was mechanically polished (~ 4-μin. finish) to afford an indication of any surface finish effects on weight-change results. In addition to these insert specimens, sheet-type tensile specimens were suspended at several points around the loop and provided additional weight-change data.

The weight-change results for the insert specimens are plotted as a function of loop position in Fig. 19. The results (shown as dark triangles) indicate a uniform removal of 8 $mg/in.^2$ (1.2 mg/cm^2) in the condenser and a maximum deposition of 90 mg/in.^{2} (14 mg/cm^2) in the subcooler at the coldest region (the cooling air intake). The weight loss of 8 mg/in.^{2} is equivalent to a uniform wall thickness reduction of 0.06 mil. Considering the similarity of weight changes between alternate inserts, surface quality (comparing as-machined with mechanically polished finishes) had no apparent effect on the magnitude of dissolution or deposition rates.

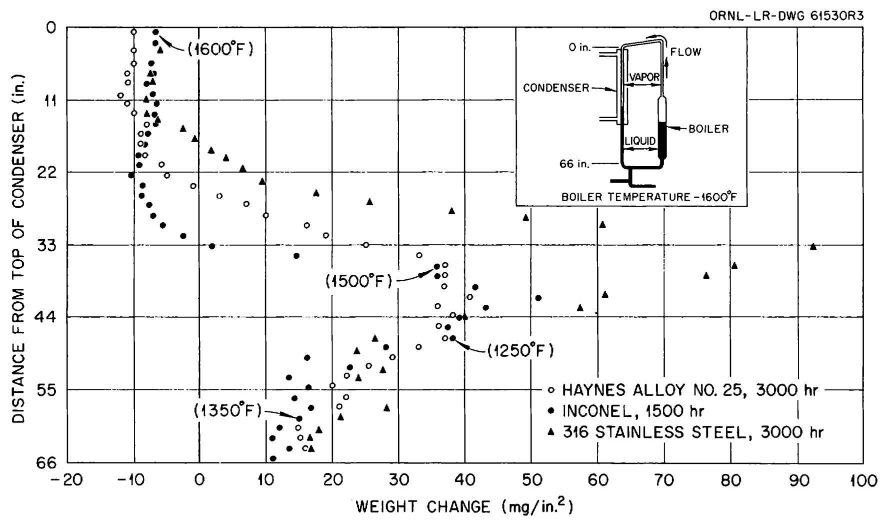

Fig. 19. Profile of Weight Changes of Inserts From Condenser Legs of Boiling-Potassium Loop Tests Operated with a Condenser Temperature of 870°C (10 mg/in.^{2} is Approximately 1.5 mg/cm^2).

The weight changes recorded for tensile specimens at various loop positions are summarized in Table 11. A specimen which was suspended in the condenser showed a weight loss identical in magnitude to that exhibited by surrounding insert specimens. However, the losses in this region appear to be consierably less than those which occurred in the boiler and vapor carry-over line. Weight gains are evident for specimens in both the subcooler and preheater regions.

Visual Findings

Like the first stainless steel loop, crystalline deposits were visible in the subcooled liquid region of loop No. 2, commencing at the liquid

Table 11. Weight-Change, Carbon Content, and Room-Temperature Mechanical Property Data on Type 316 Stainless Steel Sheet Tensile Specimens Following Exposure in Second Boiling-Potassium Loop for 3000 hr

Gage Length Dimensions: 2 × 0.25 × 0.040-in.

Specimen[a] Location and Environment	Temperature (°C)	Weight Change (mg/in.^{2})[b]	Mechanical Property Results			Carbon Content (wt %)
			Yield Strength[c]	Tensile Strength	Elongation in 2 in. Gage (%)	
			× 10^3	× 10^3		
Test Specimens						
Boiler, liquid	910	−22.4	20.6	74.4	62.0	0.01
Boiler, interface	910	−20.7	23.0	76.7	57.0	0.01
Carry-Over Line, vapor	870	−12.9	22.0	78.4	58.0	0.02
Condenser, vapor	870	−7.3	23.5	78.2	57.5	0.03
Subcooler, liquid	675	+6.3	30.8	85.0	47.0	0.16, 0.18
Preheater, liquid	700	+24.7	31.2	75.9	15.5	0.21, 0.27
Control Specimens						
Vacuum	705		55.1	88.4	51.5	
Vacuum	760		47.5	90.8	47.5	
Vacuum	870		55.5	80.1	55.5	

[a]All specimens were 0.040-in.-thick sheet annealed at 1230°C for 30 min and water quenched prior to exposure.

[b]10 mg/in.^{2} is approximately 1.5 mg/cm^2.

[c]At 0.2% offset.

interface below the condenser. The mass of these deposits can be discerned from the weight gains of the insert specimens in this region, shown in Fig. 19.

The deposit on an insert located at the approximate liquid level in the condenser is shown in Fig. 20. Metallic deposits were also

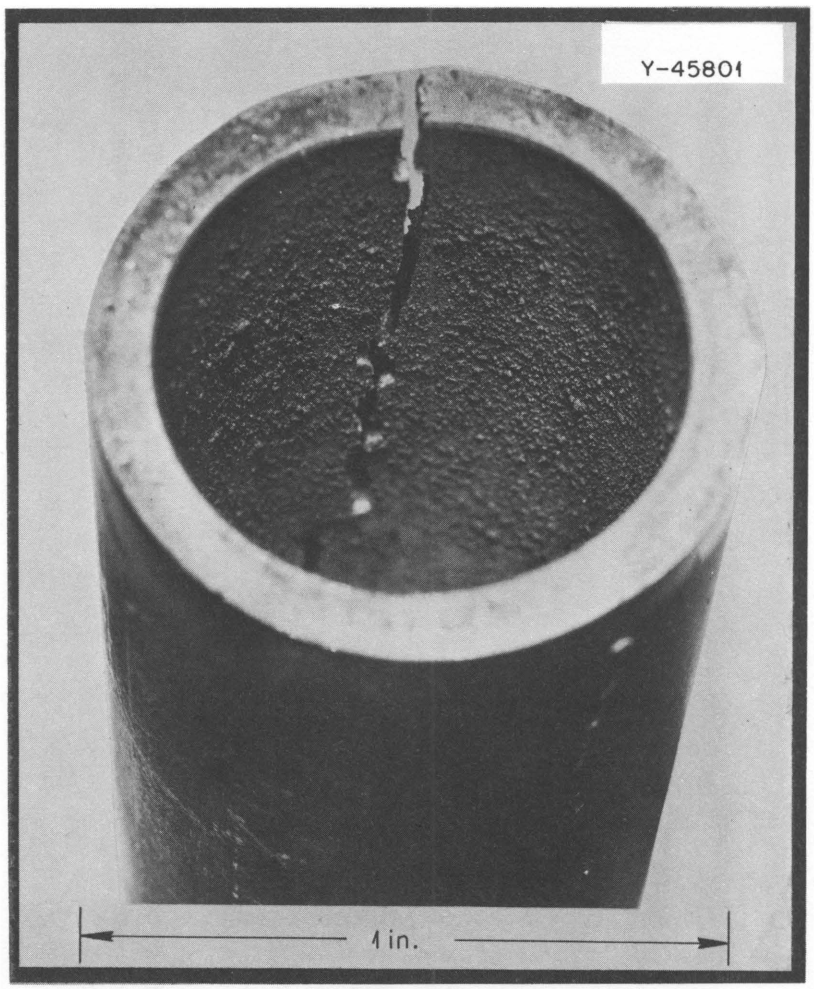

Fig. 20. Insert Located at the Approximate Liquid Level in the Condenser Showing Mass-Transfer Deposits and Thermal Fatigue Cracks in Second Type 316 Stainless Steel Boiling-Potassium Loop (3000 hr – 870°C).

observed in the horizontal portion of the preheater line as shown in Figs. 21 and 22. When the pipe was split, it was noticed that the crystals were loosely adherent and had accumulated on the bottom half of

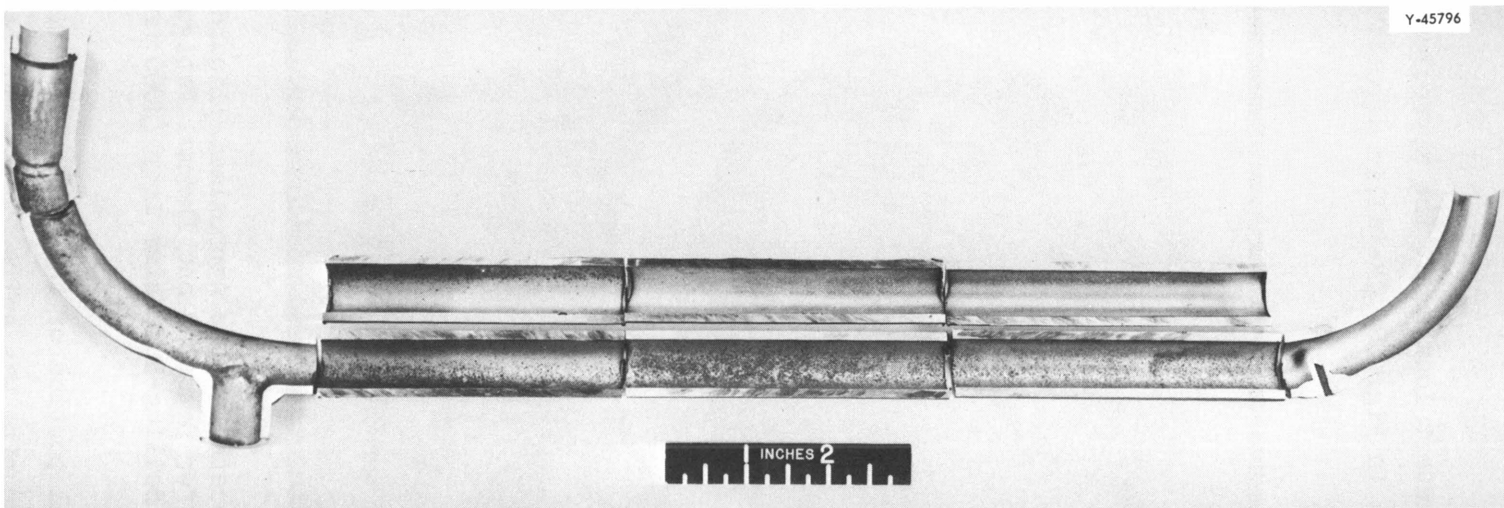

Fig. 21. Horizontal Portion of the Preheater From the Second Type 316 Stainless Steel Boiling-Potassium Loop Test. A mass-transfer deposit adhering to the bottom half of the tube can be seen.

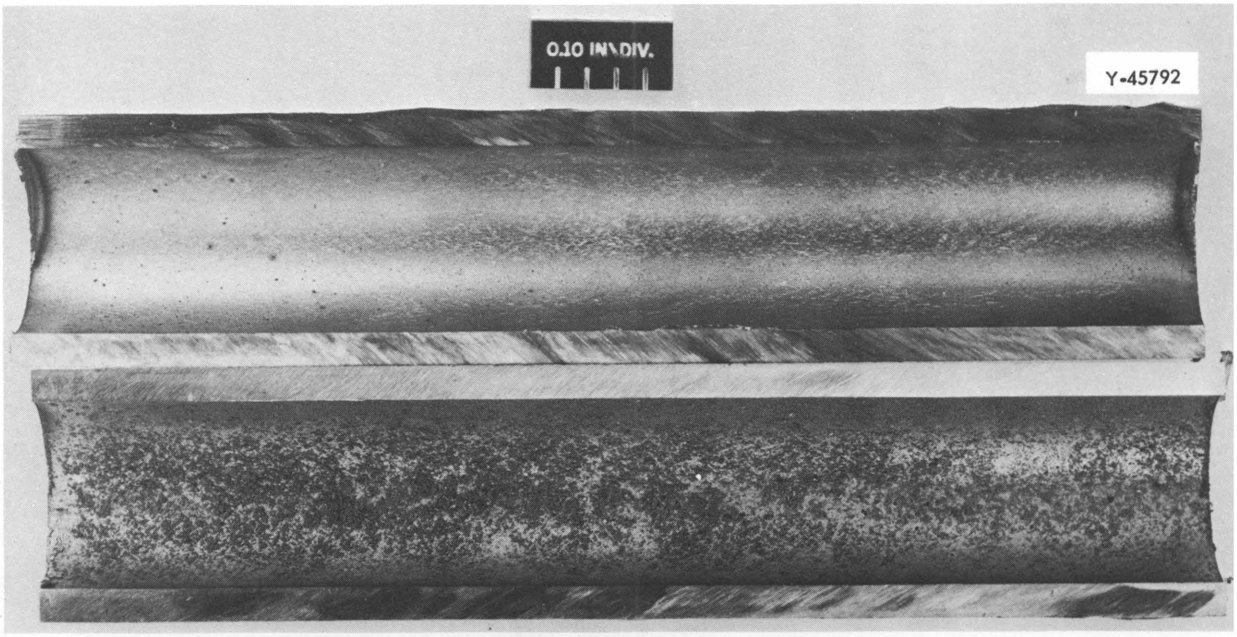

Fig. 22. Enlargement of a Portion of Center Piece Shown in Fig. 21.

the pipe during loop operation. A metallic deposit was also found in the bottom portion of the boiler.

Extensive cracking was observed in three of the insert specimens located at or nearest the approximate liquid level in the condenser (Figs. 20 and 23). These cracks extended through the total wall thickness (0.109 in.) for the entire length of these three inserts and are attributed to plastic strain fatigue caused by rapid thermal fluctuations in this region. The thermal fluctuations, in turn, were the result of an oscillating liquid level cuased by the boiling instabilities.

Metallographic Results

The results of metallographic studies of loop No. 2 are summarized in Table 12.

The metallographic appearance of inserts from the subcooled liquid region is shown in Fig. 24. Specimens from this region revealed both a mass-transfer deposit overlaying the surface and a precipitate within the base metal. This precipitate is believed to be a carbide, resulting from diffusion of carbon into the metal, since a large amount of carbon

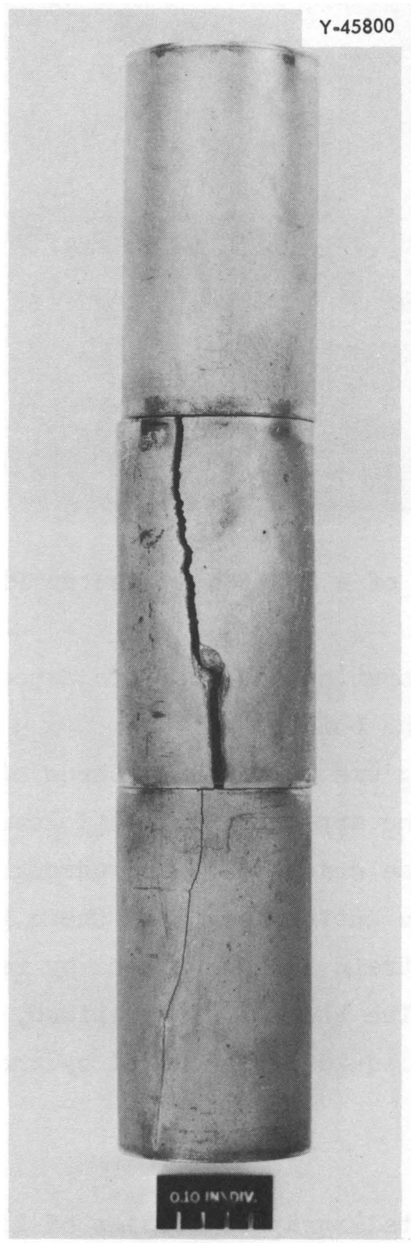

Fig. 23. Inserts From the Approximate Liquid Level in the Condenser of Second Type 316 Stainless Steel Boiling-Potassium Loop Test, Which Operated for 3000 hr at 870°C.

Table 12. Results of Metallographic Examination of Specimens From Various Regions of the Second Type 316 Stainless Steel Boiling-Potassium Loop Test Operated for 3000 hr

Location of Specimen	Temperature During Test (°C)	Results
Boiler		
Liquid zone	910	Intergranular attack to a depth of 2 mils (max); decarburization to a depth of 5 mils
Liquid-vapor interface	910	No cracks observed; surface roughening to a depth of 2 mils
Condenser		
Vapor zone	870	Intergranular attack varying from a minimum of 2 mils to a maximum of 6 mils
Liquid-vapor interface	730–840	Fatigue cracks extending completely through insert specimen (100 mils); metal crystal deposits 10 mils thick
Subcooled Liquid	700	No cracks; metal crystal deposits 10 mils thick

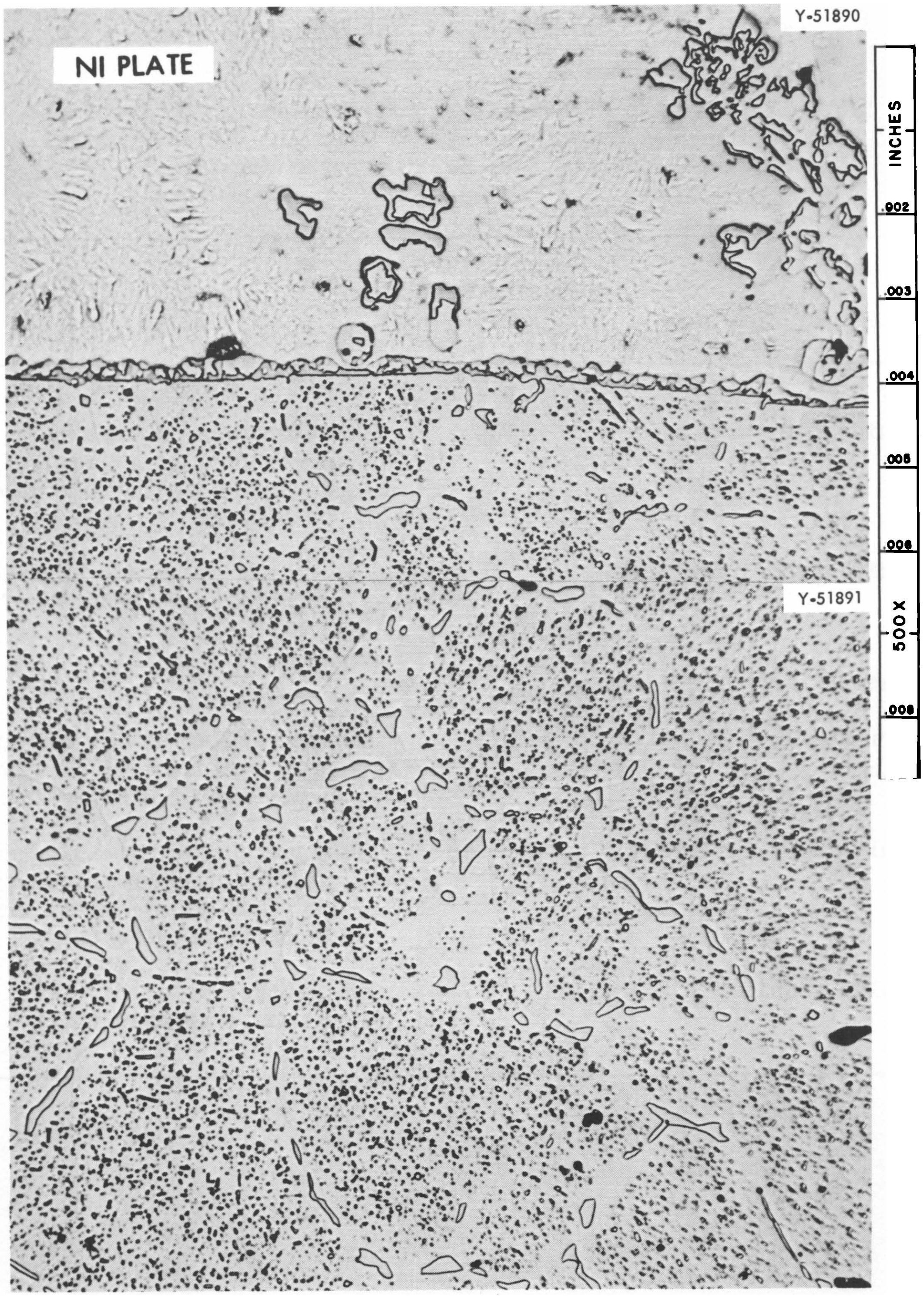

Fig. 24. Surface of Insert Located in the Subcooler of the Second Type 316 Stainless Steel Boiling-Potassium Loop for 3000 hr. Specimen was nickel-plated following test to preserve deposits and specimen edge during metallographic preparation. Etchant: aqua regia.

was detected in machinings from this region (reported later in "Chemical Analyses" section). The presence of $Cr_{23}C_6$ on the tensile specimen taken from the subcooler of the first type 316 stainless steel loop also supports this belief.

Metallographic examinations of the boiler wall in contact with liquid potassium revealed the exposed surface to be almost depleted of carbide precipitate (Fig. 25). A similar observation had been made for

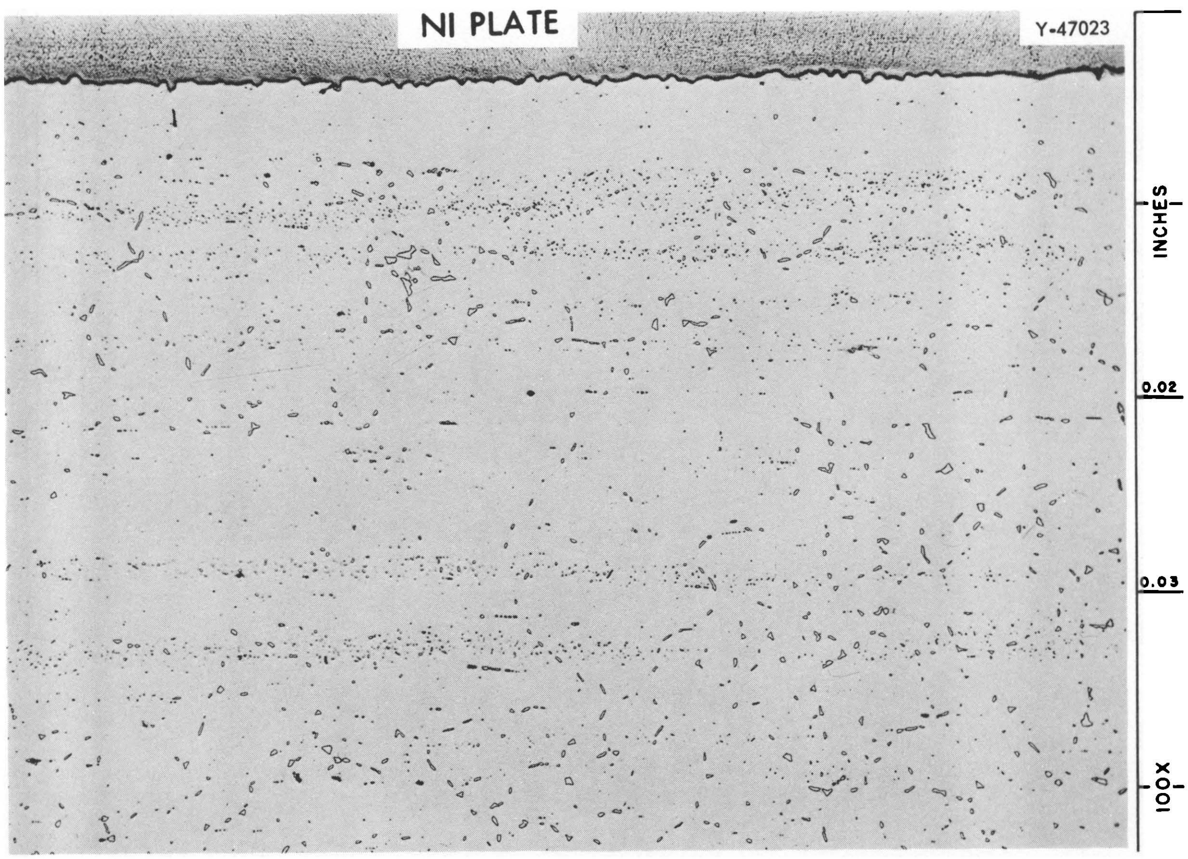

Fig. 25. Section of the Vapor Region of the Boiler Wall From the Second Type 316 Stainless Steel Potassium Loop. The temperature at this location was approximately 910°C. Etchant: aqua regia.

the first type 316 stainless steel loop test (Fig. 11). The metallographic examination of inserts located in the condenser showed subsurface voids to a depth of about 6 mils, and like the boiler, a selective removal of a fine precipitate near the surface (Fig. 26). The

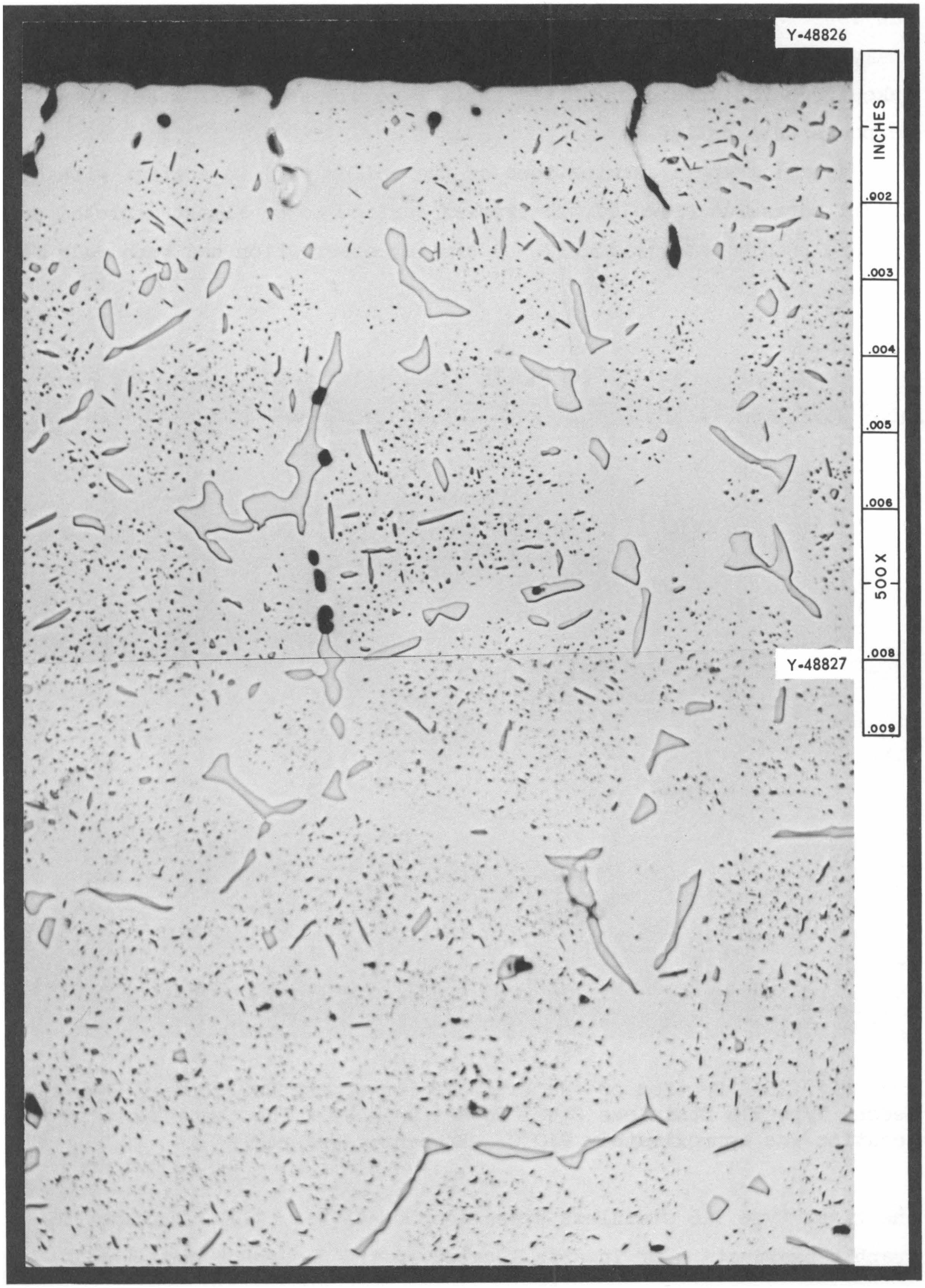

Fig. 26. Surface of Condenser From Second Type 316 Stainless Steel Boiling-Potassium Loop, Operated for 3000 hr at 870°C. Etchant: aqua regia.

large, globular particles in Fig. 26 are similar in appearance to the iron-chromium sigma phase observed in the condenser region of the first stainless steel loop. Some of the deeper voids may be due to the cracking of these sigma particles during thermal fluctuations in this region and the subsequent pulling out of the same during metallographic preparation of the specimen.

Chemical Results

Results of chemical analyses (normalized to 100%) of the mass-transfer deposits from various regions of the loop are listed in Table 13. The crystalline deposits were found to be preferentially

Table 13. Chemical Analyses of Mass-Transfer Deposits Found in Various Regions of Second Type 316 Stainless Steel Boiling-Potassium Loop Test

Location of Deposit	Prevailing Temperature (°C)	Analyses[a] (wt %)		
		Iron	Nickel	Chromium
Liquid level, condenser	720	51	15	34
Bottom of boiler	815	55	19	26
Tensile specimen, subcooler	675	43	26	31
Type 316 stainless steel (before test)		70	12	18

[a]Normalized to 100%.

enriched in chromium and nickel relative to the original stainless steel pipe. This preferential enrichment was also noted in analyses of the crystals collected from the first stainless steel loop test (Table 9).

Incremental layers, 3 mils thick, were machined from the inside wall of several inserts from various regions of the condenser leg. Analyses of the turnings (Table 14) indicate that carbon was transferred from the condensing region and subsequently deposited and diffused into the subcooler wall. This carbon transfer is in agreement with the metallographic appearance of specimens from these two regions (Figs. 24 and 26) and also

Table 14. Carbon Analyses of Turnings Machined From Type 316 Stainless Steel Inserts Following 3000 hr Exposure to Potassium (Test Loop No. 2)

Location of Sample	Temperature During Test (°C)	Location of Turnings (mils from inside wall)	Carbon Content[a] (wt %)
Vapor region of condenser	870	0–3	0.036
		3–6	0.030
		6–9	0.037
Approximately 10 in. below liquid-vapor interface of condenser	705	0–3	0.50
		3–6	0.30
		6–9	0.20
Preheated portion of liquid return line 30 in. below liquid-vapor interface	745	0–3	0.52
		3–6	0.42
		6–9	0.31

[a]Original carbon content: 0.08 wt %.

with the analyses obtained from the condenser and subcooler of the first type 316 stainless steel boiling-potassium loop test (Fig. 6). The analyses of incremental layers machined in a similar manner from the boiler wall (below the liquid-vapor interface) show no significant change from the before-test analyses. This is in contrast to the results of the first stainless steel test loop which showed a preferential loss of chromium in this region (Table 6).

Tensile Tests

The results of tensile tests on 0.040-in.-sheet specimens suspended in various locations around the loop are listed in Table 11. Results are also included for control specimens that were held in evacuated capsules for 3000 hr at the indicated temperatures.

The ultimate tensile strength of type 316 stainless steel was only slightly decreased as a result of exposure to potassium in the 675 to 870°C temperature range. The yield strength of specimens exposed to potassium liquid and vapor at 870°C was significantly reduced. Specimens exposed to the potassium in the 675 to 730°C range also sustained reductions in yield strength, but not as severely as those exposed at the higher temperatures.

With the exception of the tensile specimen exposed to liquid potassium at 700°C in the vertical preheater leg, no significant change in elongation values was observed. Each tensile specimen was analyzed for carbon at the point of fracture in an attempt to determine the significance of the one relatively low elongation value (15.5%). The results are listed in Table 11. The carbon content of the low ductility specimen was 0.24%. Since tensile tests on specimens exposed to potassium under similar conditions showed more than 40% elongation,[11] the brittle nature of this one specimen was thought at first to be anomalous. However, metallographic examination of the fractured region of this specimen indicated a different type of fracture from the other tensile specimens. Figure 27 shows the appearance of the fracture of the subcooler specimen with 0.17% C, and Fig. 28 shows the fracture of the low ductility specimen with 0.24% C.

The more ductile specimen (47% elongation) in Fig. 27 has failed transgranularly, while a definite intergranular fracture is apparent in the less ductile specimen (15.5% elongation). It would appear that the combination of sufficient carbon content and the appropriate thermal history creating a nearly continuous grain boundary carbide phase would explain the rapid degradation in tensile elongation. It appears that the combination of slightly higher carbon content (0.24% vs 0.17%) and different thermal history (700°C vs 675°C) has resulted in a continuous grain boundary carbide phase (Fig. 25) which in turn has produced a sharp decrease in the tensile elongation (15.5% vs 47%). In other investigations, the room-temperature ductility of type 304 stainless steel was reduced from 83 to 11% when it was carburized to 0.23% C at

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[11]Data from first type 316 stainless steel loop test, Table 5 this report.

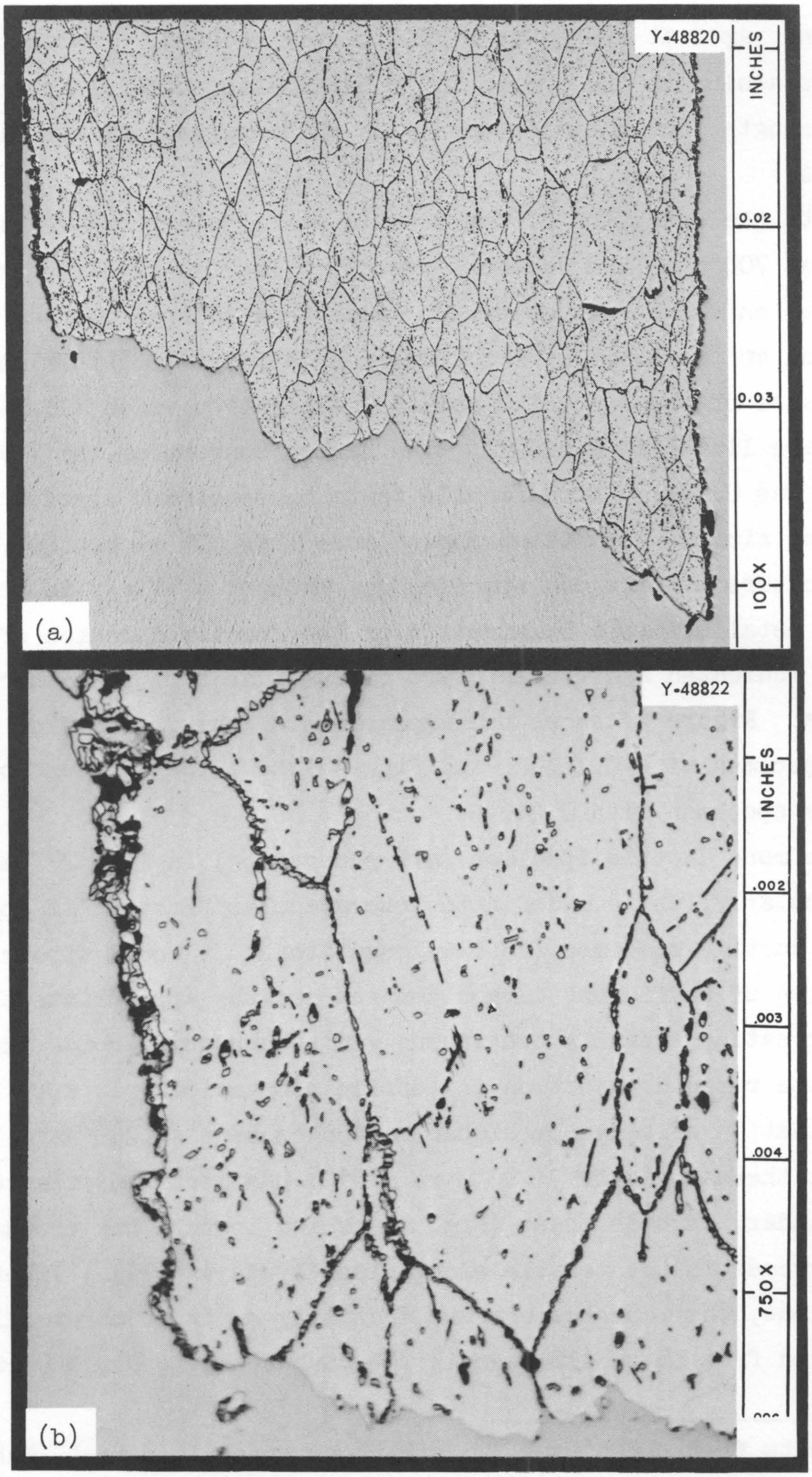

Fig. 27. Fracture of Sheet Tensile Specimen From Subcooler (675°C) of Second Type 316 Stainless Steel Boiling-Potassium Loop. The specimen contained 0.17% C and had 47% elongation in 2 in. The entire cross section at the point of fracture is shown in (a), while (b) shows a higher magnification of the same field. Note the transgranular fracture and the elongated grains. Etchant: modified aqua regia. Reduced 2.5%.

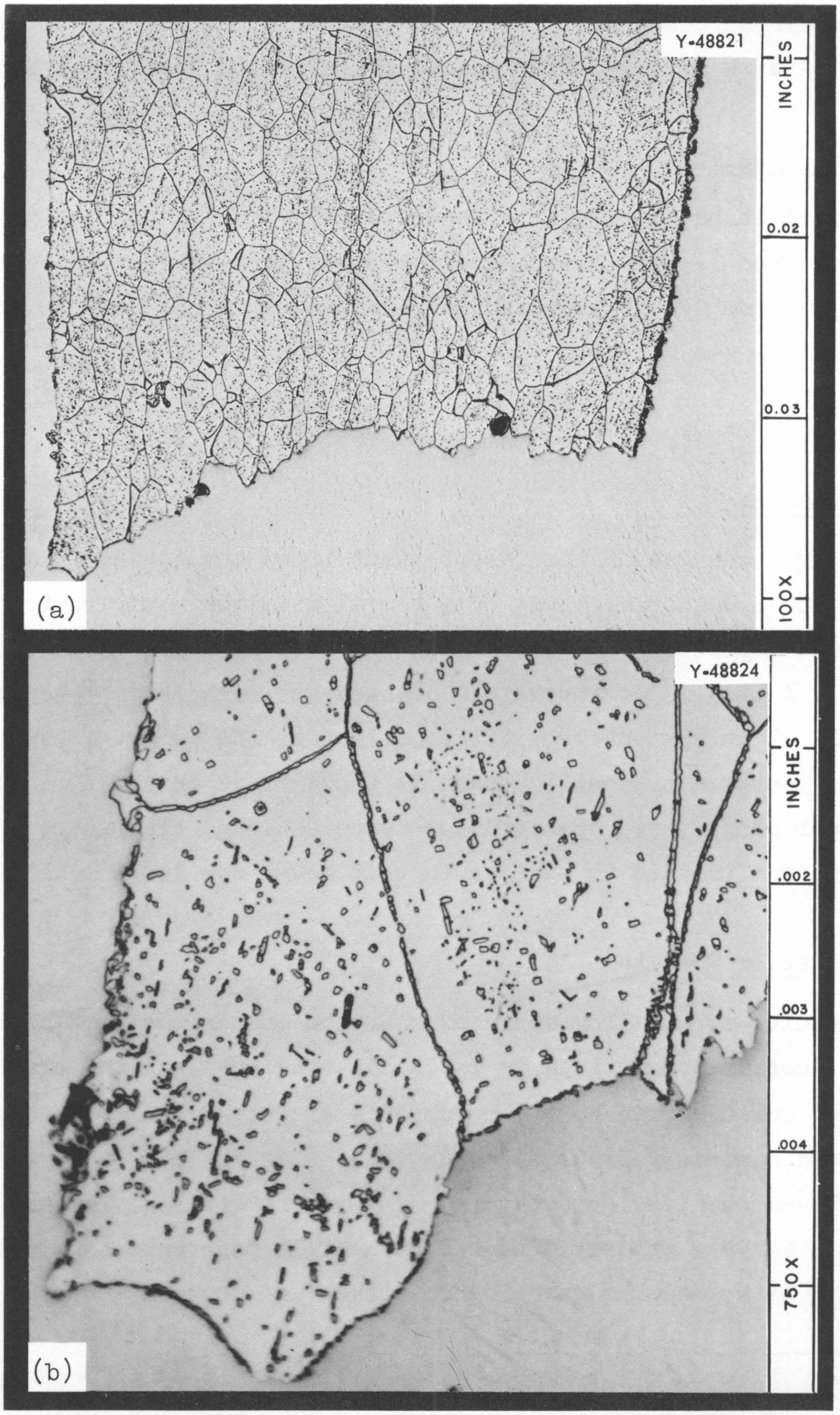

Fig. 28. Fracture of Sheet Tensile Specimen From Preheater (700°C) of Second Type 316 Stainless Steel Boiling-Potassium Loop. The entire cross section of the point of fracture is shown in (a), while (b) shows a higher magnification of the same field. The specimen contained 0.24% C and had 15.5% elongation in 2 in. Note the intergranular fracture. Etchant: modified aqua regia.

815°C in carbon monoxide.[12] In this case also the low ductility was associated with intergranular cracking similar to that shown in Fig. 28.

Microprobe Analyses

An electron microprobe traverse similar to the one performed on the first type 316 stainless steel loop was performed on the condenser section of loop 2. No preferential leaching of iron, nickel, chromium, or molybdenum was detected.

Inconel Loop (Test No. 3)

Weight Changes

Weight changes of the inserts that lined the condenser and subcooler showed uniform removal of material from the vapor region and subsequent deposition of material in the subcooled and preheated liquid regions. A profile of the weight changes for each insert is shown in Fig. 19. It is obvious from the profile that the weight losses on the Inconel inserts are about the same as those found on the type 316 stainless steel loop, which operated for twice as long. The shape of the weight-change profile is similar to that found for loops 1 and 2.

Metallographic Results

Results of metallographic examination of various components of the Inconel loop are summarized in Table 15. The most serious effect of loop operation was the wide-spread occurrence of intergranular cracks in the boiler and condenser sections. The reducer at the bottom of the boiler, which showed the heaviest cracking, is pictured in Fig. 29a and b. Cracks in this area were readily detected by a Zyglo-penetrant examination. Less extensive intergranular cracking or attack was apparent between the lower

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[12] H. E. McCoy, Jr. and D. A. Douglas, Jr., Effect of Environment on the Creep Properties of Type 304 Stainless Steel at Elevated Temperatures, ORNL-2972, (Sept. 1962).

Table 15. Results of Metallographic Examinations on Components of Inconel Boiling-Potassium Loop (Test No. 3) Operated for 1500 hr

Location of Specimen	Temperature of Specimen During Test (°C)	Metallographic Notes[a]
Boiler		
Liquid zone	880	Intergranular cracking or attack to a depth of 20 mils
Liquid-vapor interface	870	Intergranular attack or cracks or both to a depth of 15 mils
Condenser	870	Intergranular attack or cracks or both to a depth of 15 mils; decarburization and grain growth to a depth of 30 mils
Subcooler	675	Mass-transfer deposit 5 mils thick; 10 mils of carburization; no attack or cracks

[a]In all cases, the maximum attack or cracking is the value reported.

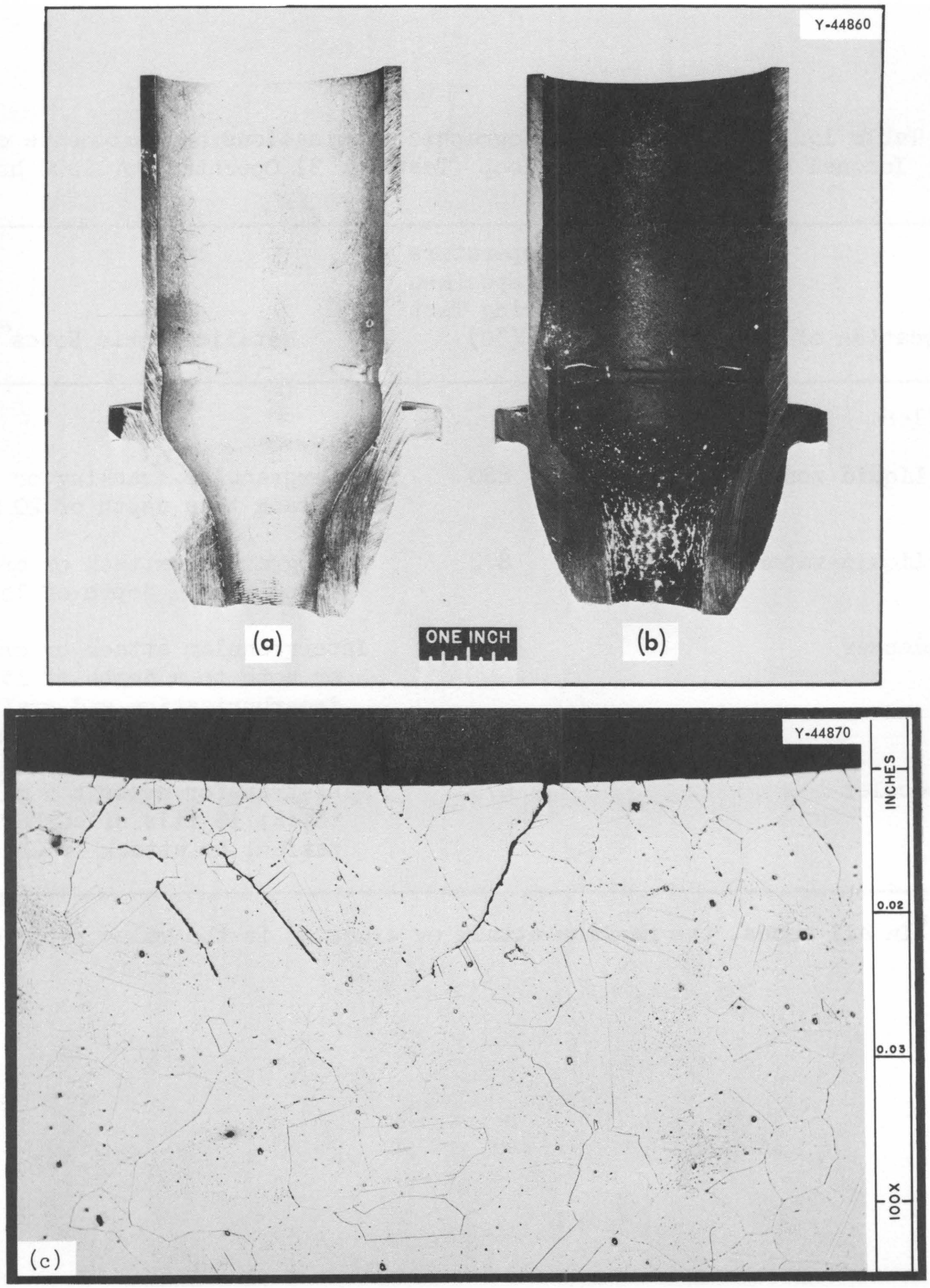

Fig. 29. Region From Bottom of Boiler From Inconel Boiling-Potassium Loop (Test No. 3), 1500 hr – 870°C. Photograph (a) was taken with white light, (b) with ultraviolet light after the specimen was treated with a fluorescent penetrant. Picture (c) is a cross section of (b) where the penetrant indicated the severest cracks. Etchant: modified aqua regia. Reduced 17%.

liquid region of the 2-in. sched-40 boiler wall, as shown in Fig. 30,

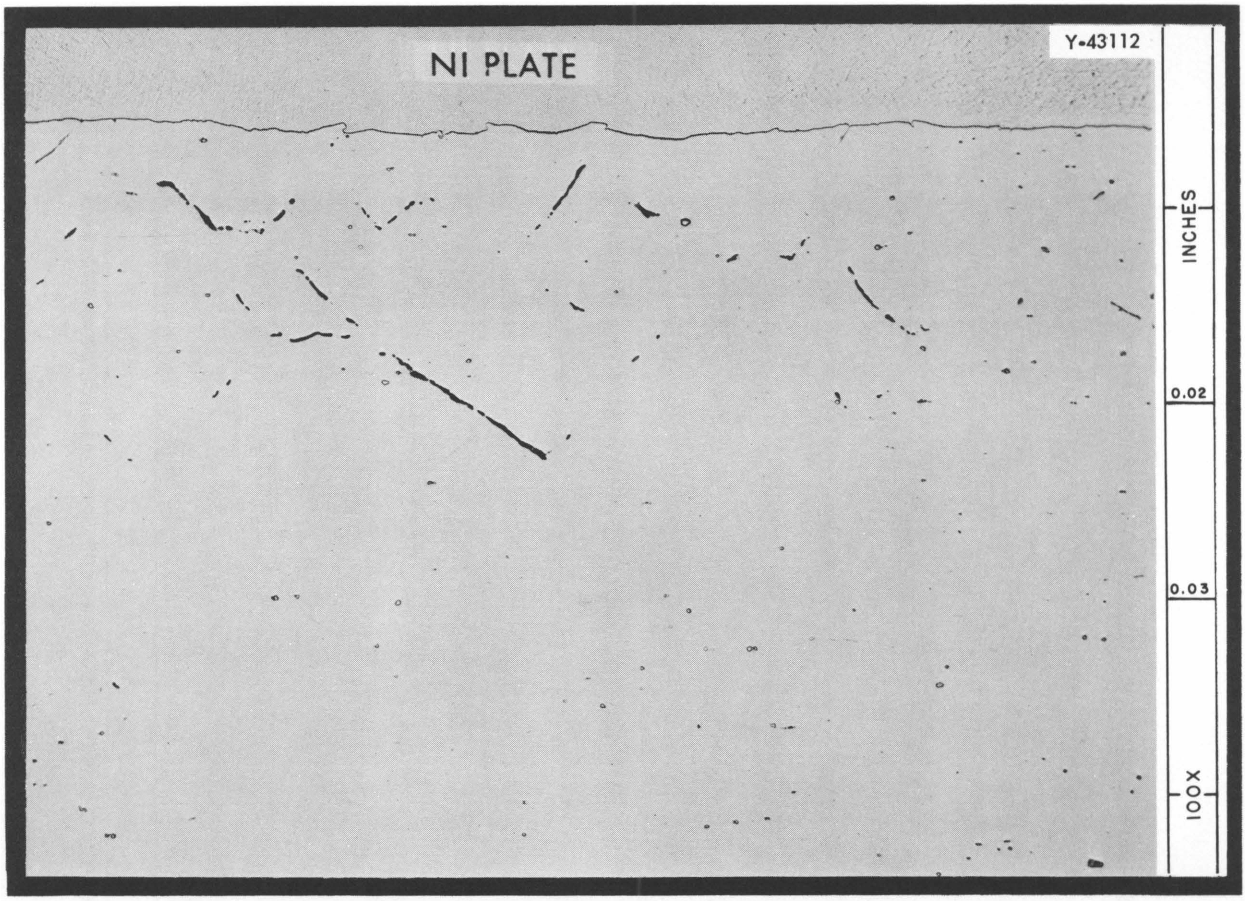

Fig. 30. Inside Surface of Liquid-Exposed Portion of Boiler Wall Taken From Inconel Boiling-Potassium Loop (Test No. 3), 1500 hr – 870°C. Note the subsurface intergranular attack. The nickel plate is to protect the specimen edge during metallographic preparation. As-polished.

and the liquid-vapor interface, as shown in Fig. 31. Similar cracking or attack was apparent in metallographic examinations of the condenser inserts. The surfaces of weight-change specimens from the top and bottom of the condenser are compared in Fig. 32, and Fig. 33. In addition to intergranular cracks, these specimens also exhibited some grain growth and decarburization, as seen in Fig. 33a.

It is not certain whether the grain boundary damage (15 mils max depth) in these specimens is due primarily to solution attack, thermal

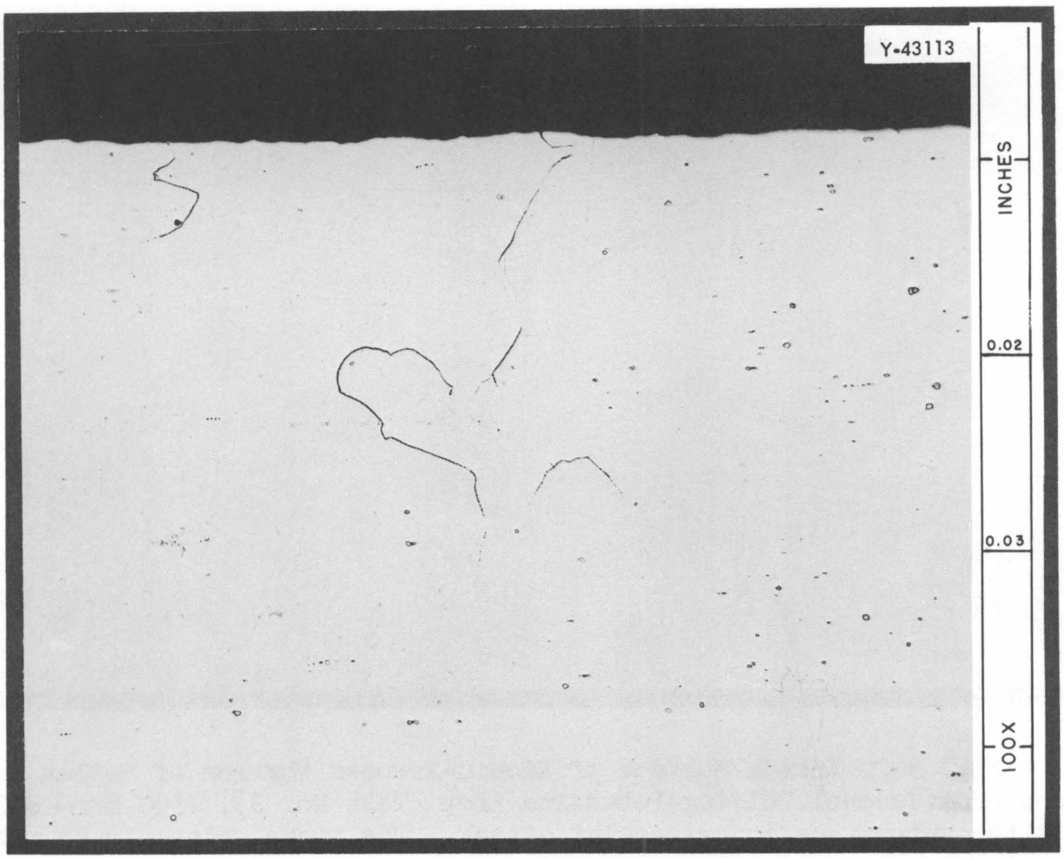

Fig. 31. Surface of Boiler Wall in the Vicinity of Liquid-Vapor Interface of Inconel Boiling-Potassium Loop (Test No. 3), 1500 hr – 870°C. Note cracks to a depth of 15 to 20 mils. As-polished.

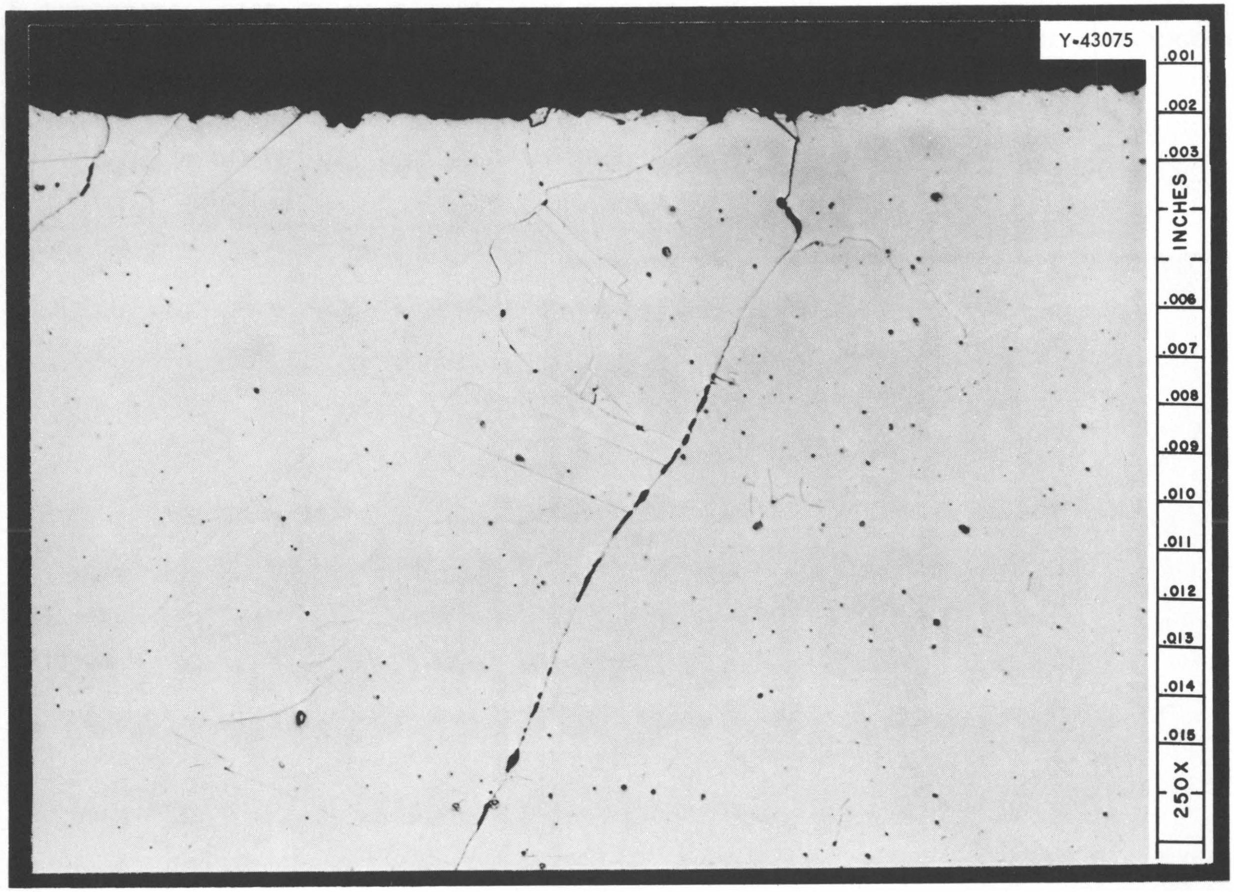

Fig. 32. Surface of Insert Taken From the Condenser of Inconel Boiling-Potassium Loop (Test No. 3), 1500 hr – 870°C. Note the deep intergranular attack. Etchant: modified aqua regia.

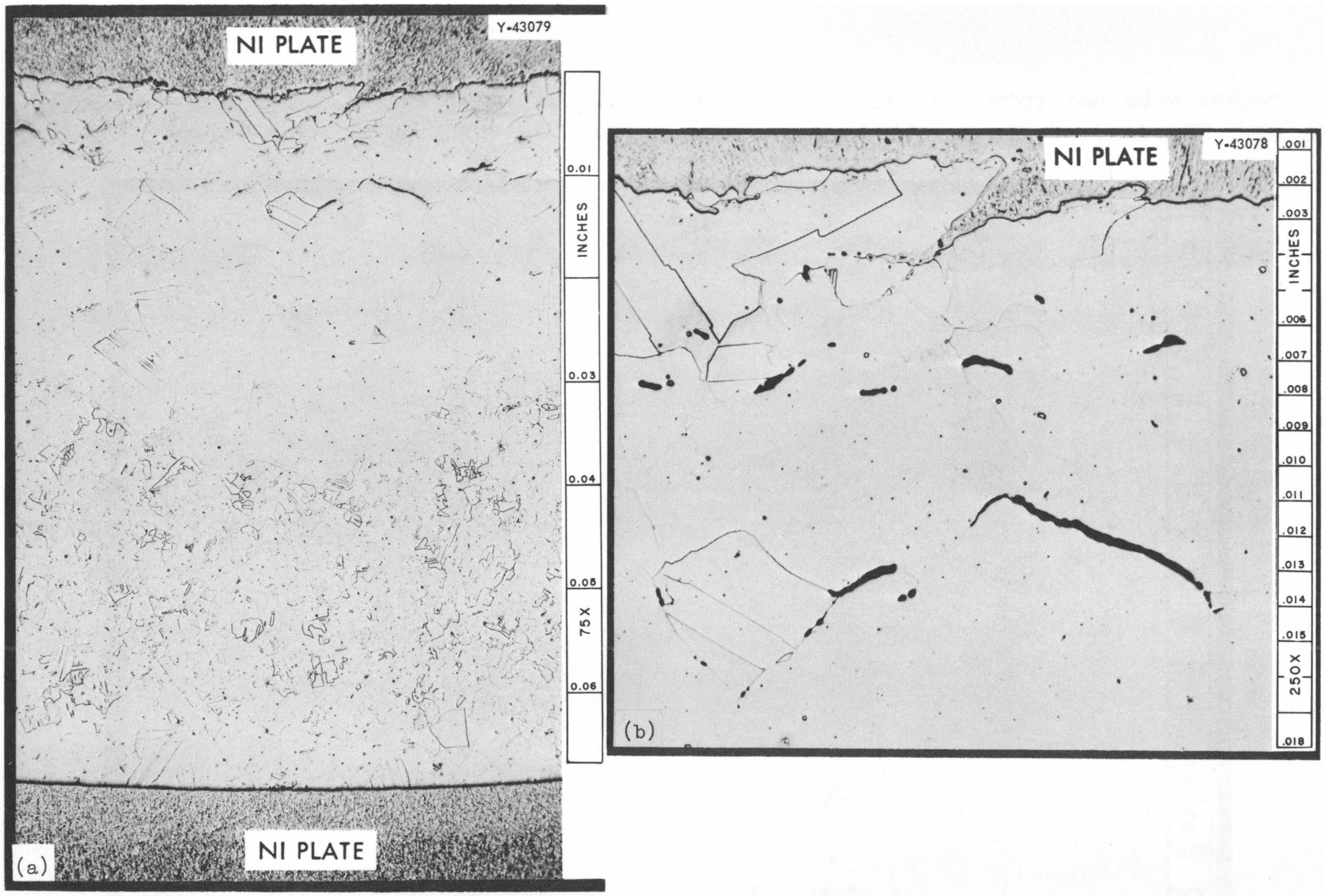

Fig. 33. Insert Taken From Region of the Liquid Level in the Condenser of the Inconel Boiling-Potassium Loop Tested for 1500 hr at 870°C. The entire wall thickness is shown in (a), while an enlarged portion of the Inconel-potassium surface is shown in (b). Nickel-plate is for edge retention during polishing. Etchant: modified aqua regia. Reduced 22%.

fatigue cracking, or to the synergistic effects of both. The low amplitude of the temperature fluctuations in the condenser region suggests that the damage in this area must stem principally from solution attack along the grain boundaries. However, as noted in a subsequent section, examinations of these grain boundary areas with an electron-beam microprobe showed no compositional differences in these regions from the grain matrices.

The metallographic appearance of surfaces of inserts from the subcooler is shown in Fig. 34b. A buildup of precipitates is evident below the exposed specimen surface and complements the finding of second-phase depletion at the condenser surfaces (Fig. 34a). Chemical analyses of the surface layers, which are shown in Fig. 34 and discussed in more detail below, indicate that this leaching and subsequent deposition and diffusion are associated with a carbon transfer.

Chemical Results

Mass-transfer deposits were found on the surface of inserts located in the subcooler. Analyses of these crystals (adjusted to 100 wt %) are listed below along with the anlyses of the "before-test" condenser material:

Condenser Material	"Before Test"	Deposit
Iron	8.6	20.8
Nickel	76.2	75.0
Chromium	15.3	3.7
Carbon	0.05	0.6

The crystal deposit was enriched in iron and carbon and depleted in chromium relative to the base material. This differs from the nickel-rich crystals that have been consistently found in Inconel systems containing all-liquid sodium.[13]

[13] E. E. Hoffman, ANP Quart. Prog. Rept. May 10, 1954, ORNL-2061, pp. 125–27.

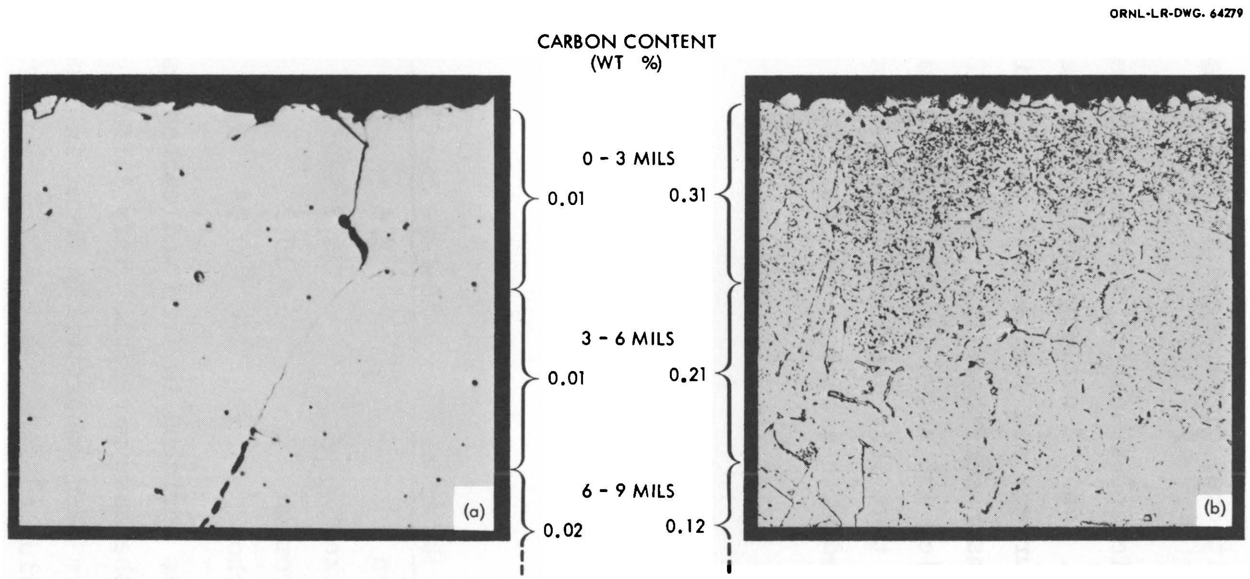

Fig. 34. Inside Surface of Inserts From (a) Condensing Region and (b) Subcooled-Liquid Region of Inconel Boiling-Potassium Loop Test, Operated for 1500 hr at 870°C. Note the carbon gradient in the two regions. Original carbon content: 0.05 wt %. Etchant: modified aqua regia. 500x. Reduced 31%.

Compositional changes occurring at the boiler surface were evaluated by chemically analyzing 3-mil-thick incremental layers from the boiler wall. The results are listed in Table 16 and indicate a preferential

Table 16. Analyses[a] of Inconel Boiler Wall Before and After 1500 hr Exposure to 870°C Potassium

Specimen Description	Element			
	Iron	Nickel	Chromium	Carbon
Before Test	7.3	77	16	0.03
After Test[b]				
0[c]–3 mils below surface	7.4	76.4	16.2	0.01
3–6 mils below surface	7.0	76.9	16.1	0.01
6–9 mils below surface	6.9	76.9	16.2	0.04

[a]Based on 100%.

[b]After-test analyses made on successive 3-mil-thick layers machined from the inside surface.

[c]Inside surface.

removal of carbon with no significant changes in the major metallic components of the alloy. This preferential removal of carbon from the surface is in accordance with observations made on loops 1 and 2. Similar analyses of incremental layers from the inside surfaces of various insert specimens indicated that carbon transferred from the condenser to the subcooler. The carbon contents of condenser and subcooler specimens are compared in Fig. 34 along with the microstructures of these two regions. No transfer of the major constituents (Fe, Ni, Cr) from the condenser to the subcooler regions was detected in these incremental layers. This pattern of carbon transfer and lack of preferential leaching of the major constituents of the alloy are similar to the results from the stainless steel loop tests.

Microprobe Traverse

The extent of preferential leaching of material from the condenser was further examined by electron-beam microprobe analysis. The results of this analysis, shown in Fig. 35, indicate no detectable leaching of

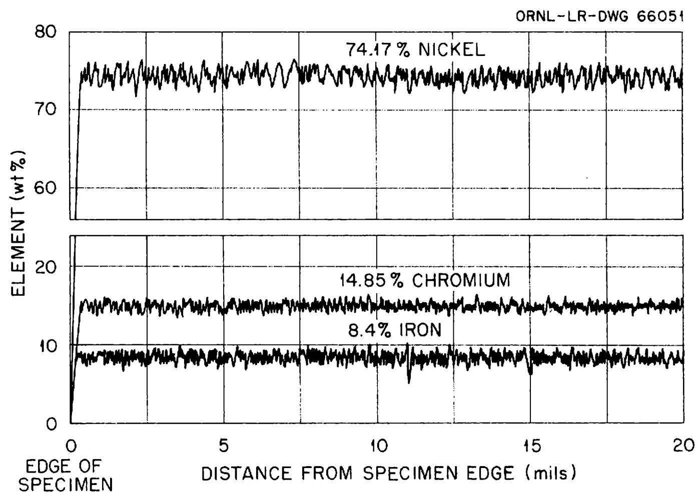

Fig. 35. Concentration Profiles of Fe, Ni, and Cr as Determined by Electron-Beam Microanalysis in an Inconel Specimen Exposed to Potassium at 870°C for 1500 hr (Loop Test No. 3).

iron, nickel, or chromium from the condensing surface. The microprobe traverse extended for a distance of 20 mils into the wall of the condenser.

Haynes Alloy No. 25 Loop (Test No. 4)

Weight Changes

Approximately 10 $mg/in.^2$ (1.6 mg/cm^2) was removed uniformly from the inserts located in the condenser. The dissolution that occurred is equivalent to less than 0.0001 in. of uniform surface removal. These weight changes and the relative positions of the inserts are plotted in Fig. 19. The general shape of the weight-change profile is similar to that in the other loop tests, with losses in the condenser almost identical to those found for the type 316 stainless steel.

Visual and Metallographic Results

The most serious damage to the loop material was cracking that occurred in the longitudinal weld zone of the condenser inserts (seam-welded pipe was used for inserts). As in previous loops, this cracking is attributed to thermal fatigue induced by alternating surges of potassium vapor and liquid. These cracks are best discerned with a fluorescent penetrant. Figure 36 shows nine of the 44 inserts tested in the condenser and subcooler which were treated with Zyglo penetrant and then photographed with ultraviolet light. The brightly fluorescing lines running parallel to the tubing axis designate cracks in inserts 1, 6, 11, 15, 21, and 27. No cracks are evident in inserts 32, 37, and 43, which were located in the liquid region (subcooler). An enlargement of the weld bead of insert 15 in Fig. 36 is shown in Fig. 37 and indicates a general network of fine cracks in addition to the large cracks lining the weld zone.

The depth of cracks on inserts located in the condenser ranged from several mils to the entire pipe wall cross section. Shown in Fig. 38 is insert 23 from the region of the liquid level in the condenser, which completely cracked through the weld fusion zone. The higher magnification microstructure in Fig. 38b shows that the cracks are intergranular in nature. Figure 39 shows the inside surface of an insert from the top of the condenser and illustrates the intergranular cracking in regions away from the weld areas.

Cracks also were observed in the boiler in the vicinity of the liquid-vapor interface. These cracks, illustrated in Fig. 40 are also believed to have resulted from thermal fatigue. Cracks were not observed in the regions of the boiler in contact with vapor only, as shown in Fig. 40a. Oscillations in the liquid level of the boiler produced heavy cracking near the liquid-vapor interface, as illustrated in Fig. 41. Figure 41a shows this region under normal illumination and Fig. 41b after treatment with Zyglo fluorescent penetrant.

The most severe corrosion in the loop was in the form of 2-mil-deep subsurface voids found at the liquid level in the boiler. Attack in the vapor carry-over line (870°C) was limited to surface roughening and

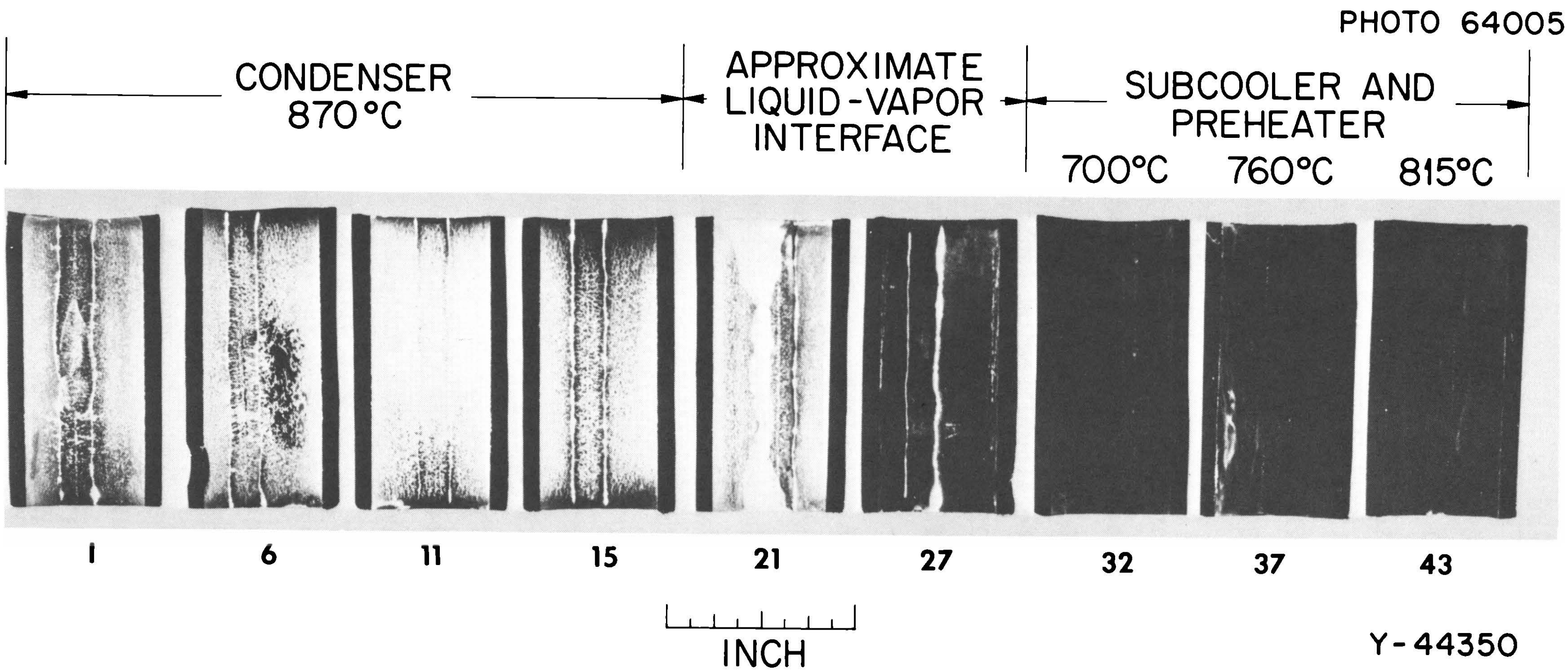

Fig. 36. Inserts From Condenser, Subcooler, and Preheater of Haynes Alloy No. 25 Boiling-Potassium Loop Operated at 870°C for 3000 hr (Loop Test No. 4). Pictures were made with ultraviolet light after Zyglo-penetrant treatment. Inserts were numbered in sequence from top of condenser to bottom of vertical portion of preheater.

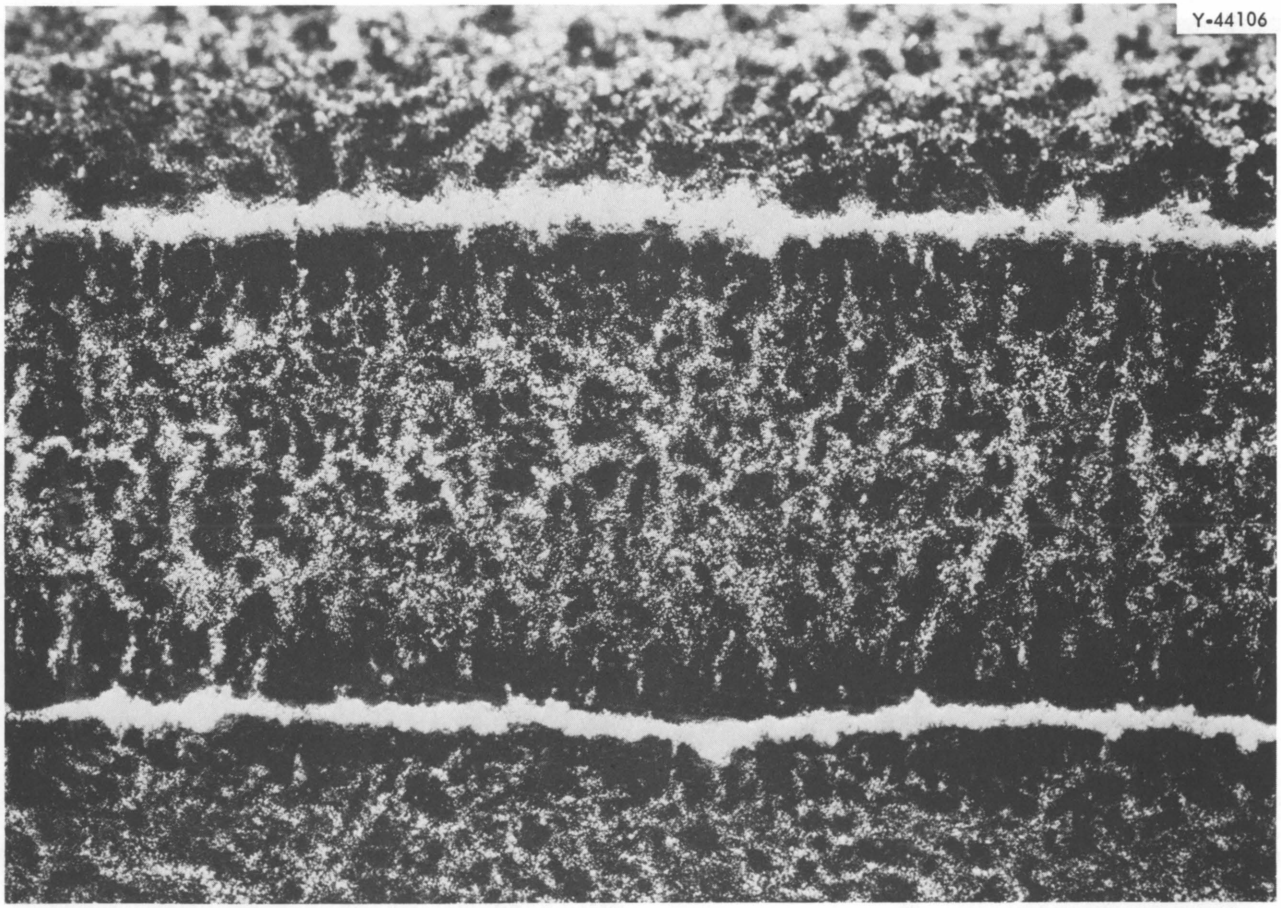

Fig. 37. Closeup of Weld Bead in Insert 15 Shown in Fig. 36. The specimen was treated with Zyglo fluorescent penetrant and photographed with ultraviolet light. 15×.

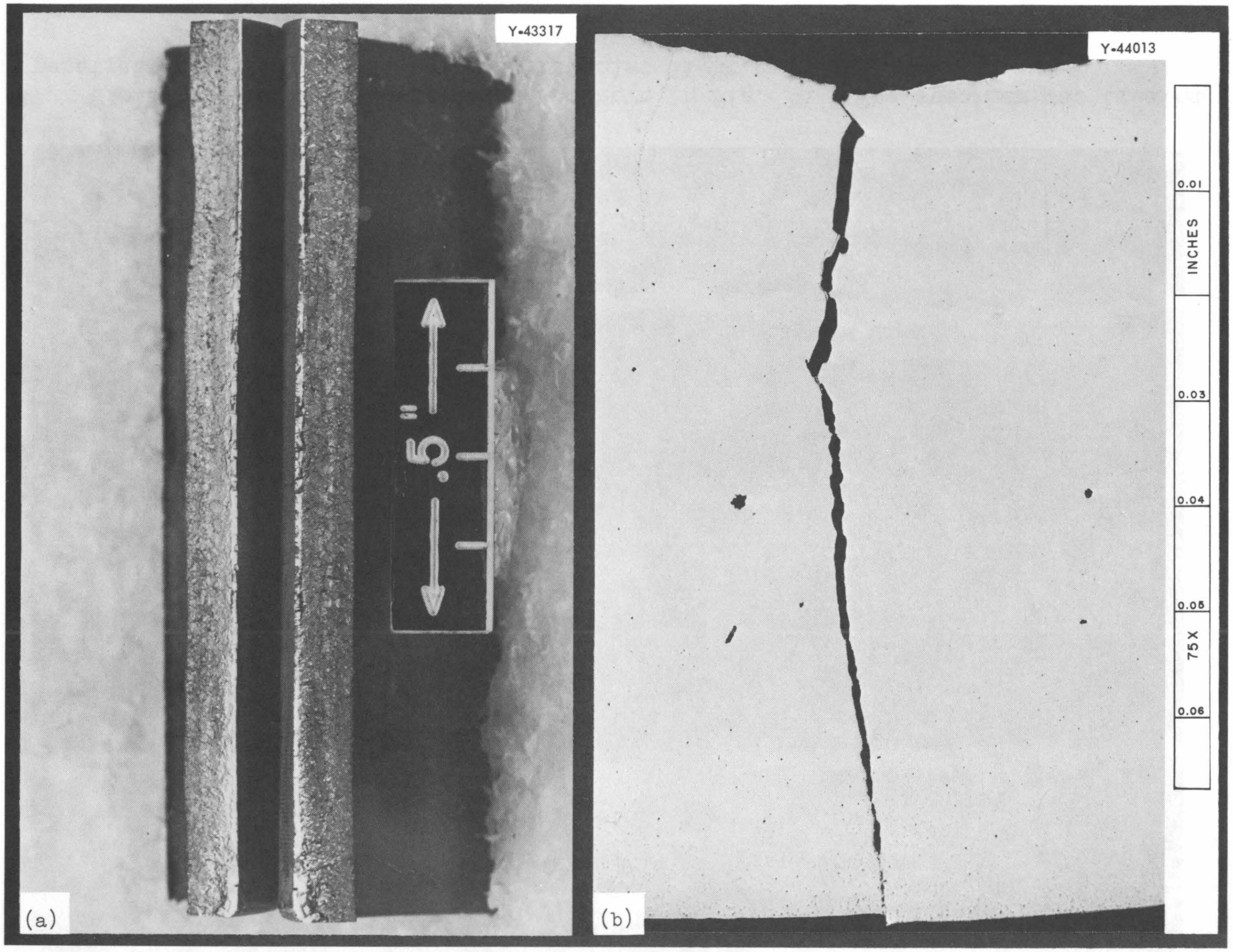

Fig. 38. Insert 23, Located in the Region of the Liquid Level in the Condenser of Loop Test No. 4. The crack in (a), which propagated through the wall, was located in a weld fusion zone. A higher magnification shows the intergranular cracking in (b). Reduced 19%.

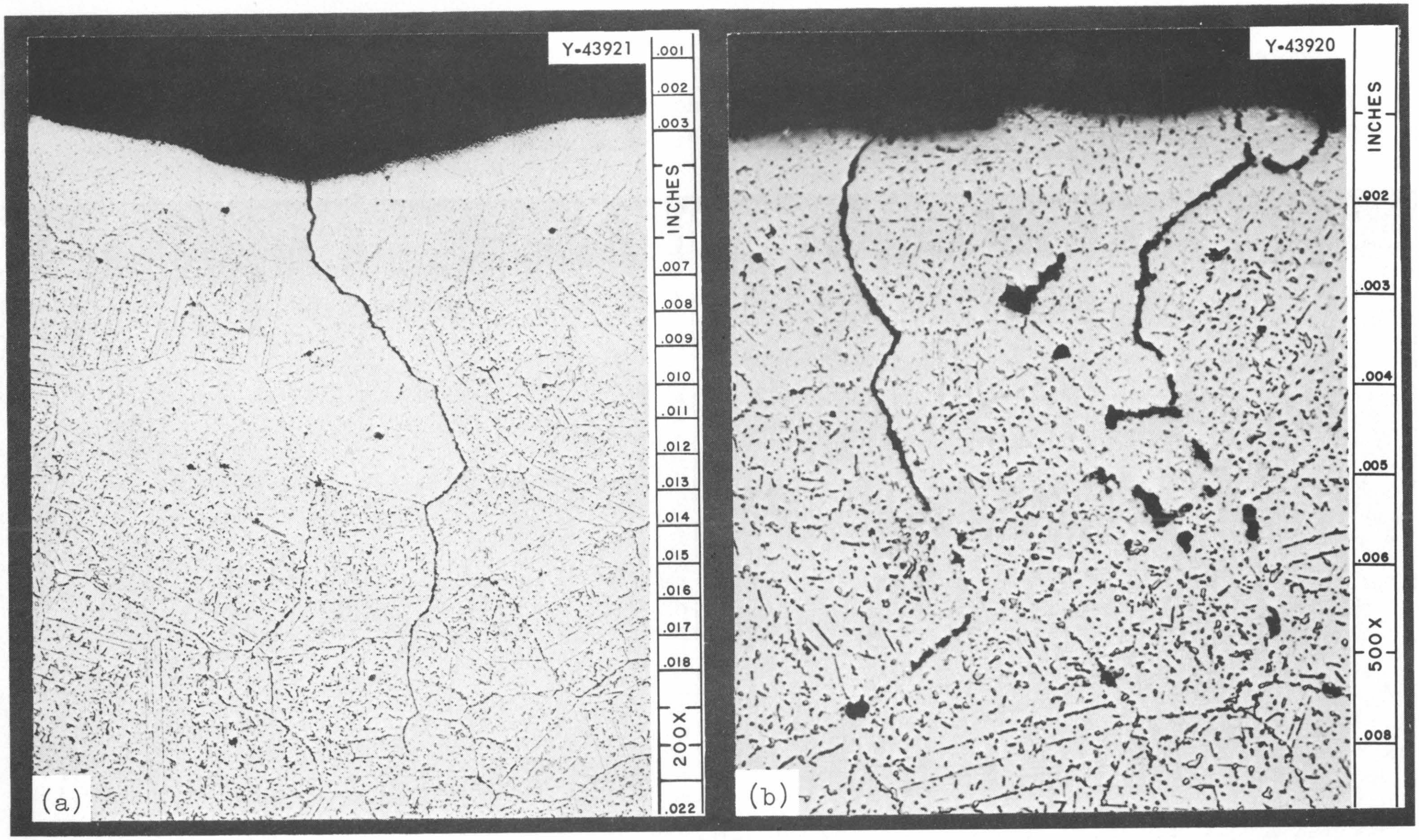

Fig. 39. Sections of Condensing Surface on the Insert From the Top of the Condenser of Loop Test No. 4 Showing Cracks in Regions Other Than the Weld Zone. In (a) is shown the weld bead while (b) is a nonwelded area. Etchant: modified aqua regia. (a) 200×; (b) 500×.

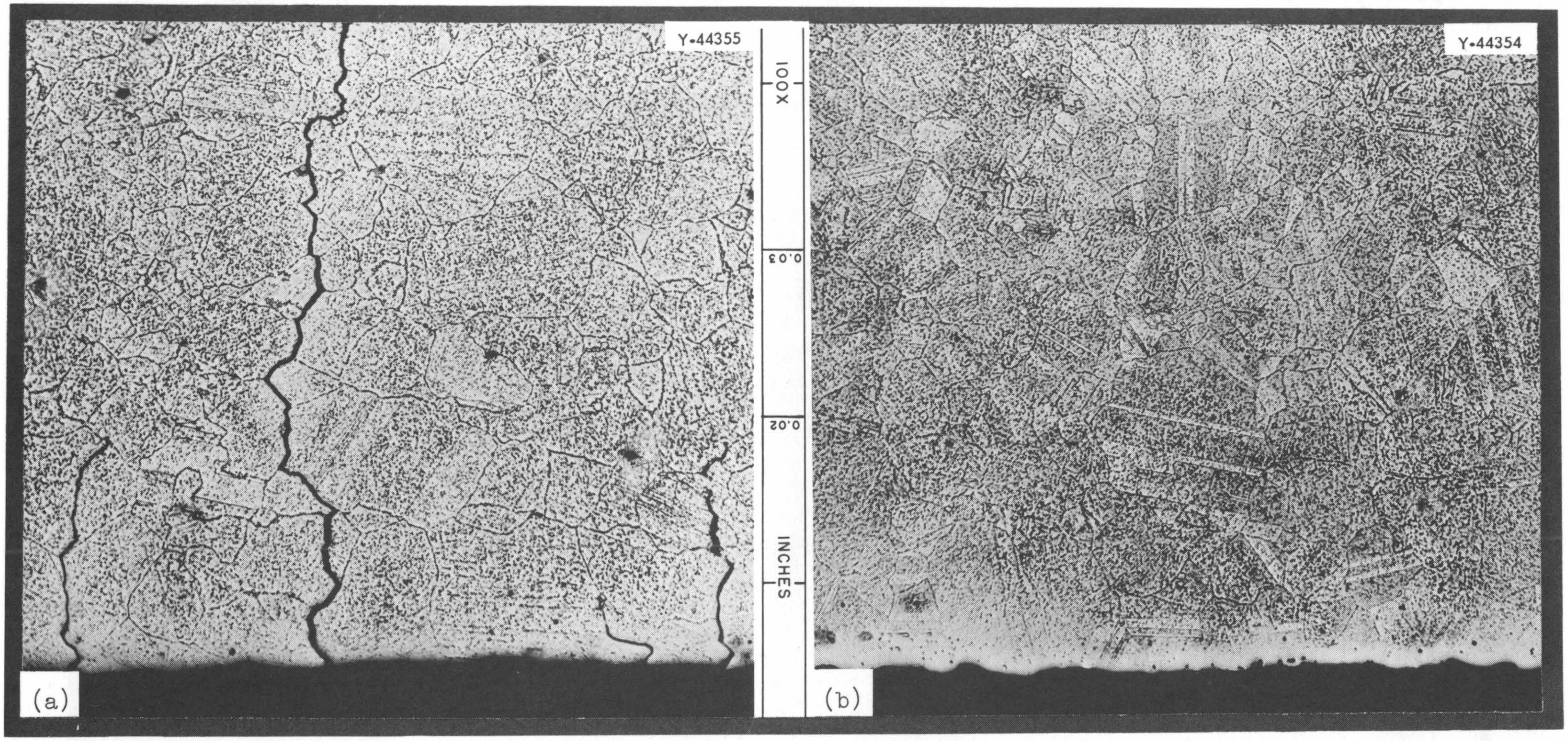

Fig. 40. Sections Cut From Wall of Boiler Section of Haynes Alloy No. 25 Loop Test No. 4. (a) Section in contact with vapor. (b) Section taken where liquid level fluctuations occurred. Etchant: modified aqua regia. Reduced 9.5%.

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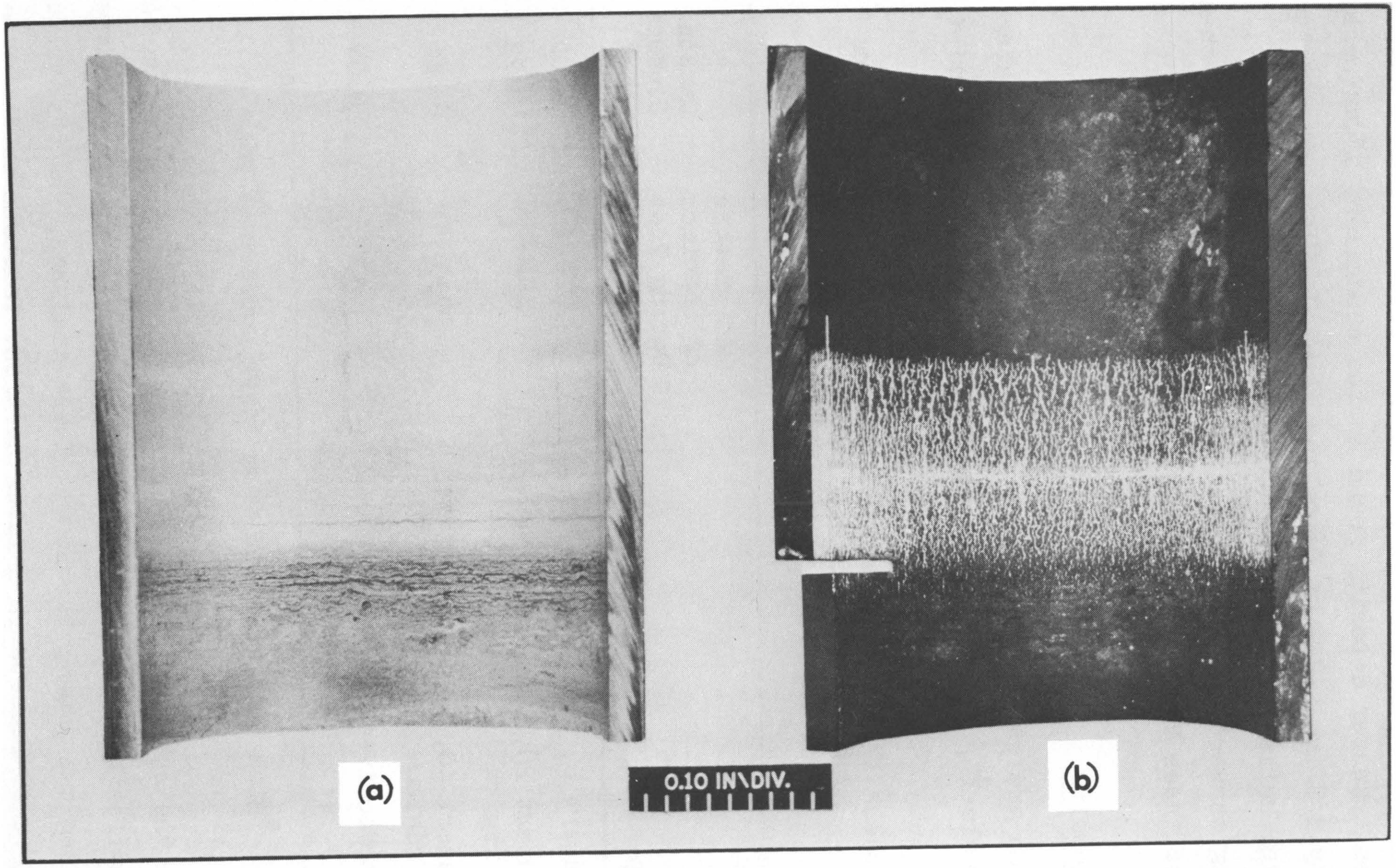

Fig. 41. Surface of the Boiler at the Liquid-Vapor Interface of the Haynes Alloy No. 25 Boiling-Potassium Loop Test No. 4. (a) Photograph taken with white light. (b) Photograph taken with ultra-violet light after treatment with Zyglo penetrant.

scattered subsurface voids to a maximum depth of 1 mil (Fig. 42). Inserts located in the subcooler (lowest temperature approximately 700°C) exhibited a mass-transfer deposit approximately 1/2 mil thick (Fig. 43). In addition to the mass-transfer deposits found in the subcooler, a narrow band (approximately 1/8 in. wide) of deposited metal 0.040 in. in thickness was found in the boiler at the region of the liquid level (Fig. 44). (Deposits in this region were not encountered in previous tests.) The chemical analyses of these deposits are discussed later.

Chemical Results

Carbon analyses of the condenser, subcooler, and preheater inserts, shown in Table 17, indicate a carbon loss in the condenser and a carbon

Table 17. Carbon Analyses on Turnings Machined[a] From Haynes Alloy No. 25, Inserts Following 3000 hr of Exposure to Potassium, Loop Test No. 4

Specimen Number and Location	Temperature of Specimen During Test (C°)	Carbon Content (wt %)
Insert 8, condenser region	870	0.05
Insert 28, subcooled liquid region	675	0.22
Insert 40, preheated liquid region	730	0.16
As-received material		0.09

[a]Nine mils machined from inner surface of pipe wall.

increase in the subcooler and preheater sections. A similar carbon movement was manifested by the analyses of the tensile specimens suspended in various regions around the loop. These analyses, which are listed with tensile test results in a later section, indicate an increase in carbon content from 0.09 to 0.18 wt % in the tensile specimen located in the subcooler. The specimen suspended in the boiler (liquid) analyzed only 0.056 wt % C.

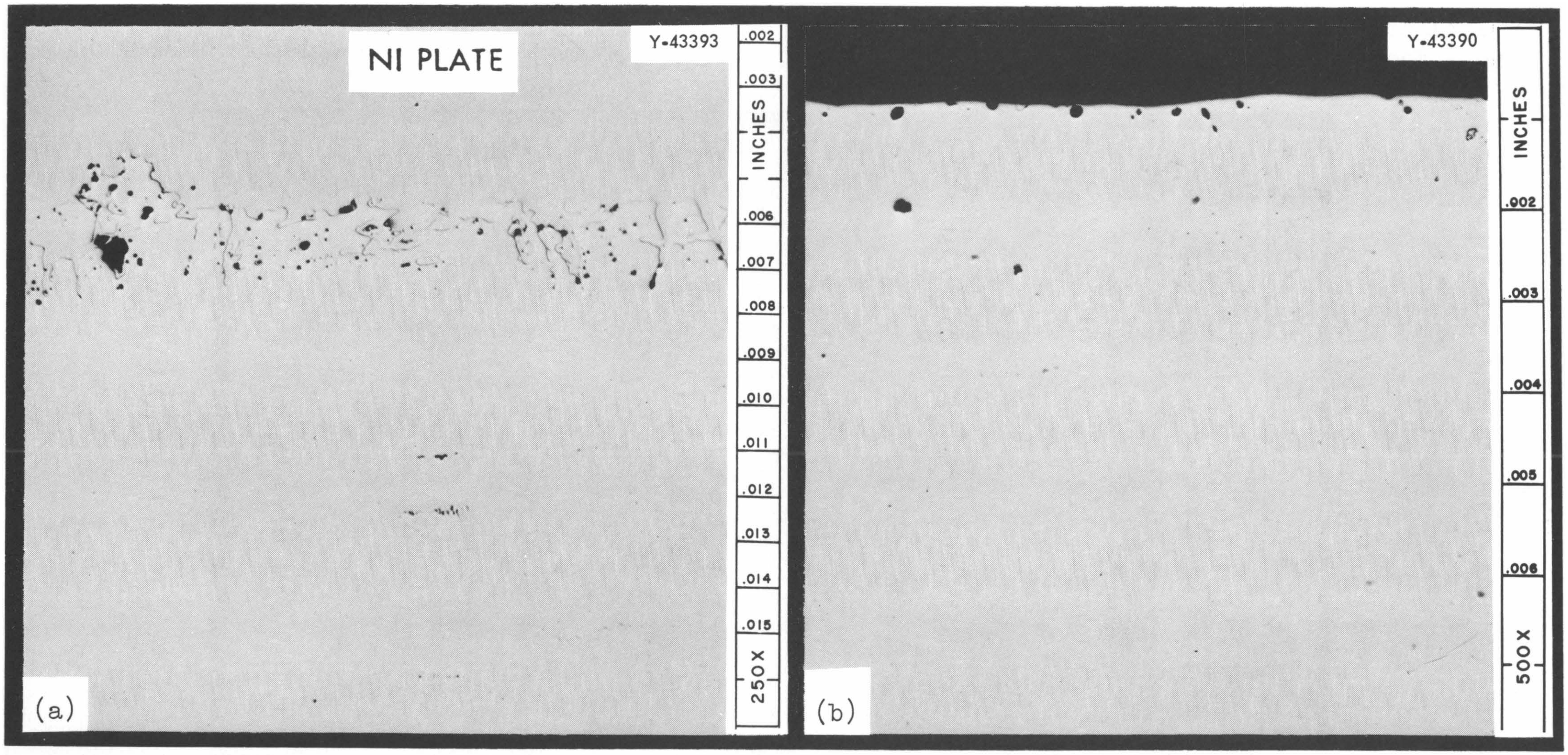

Fig. 42. (a) Inner Surface of Boiler Wall at Liquid Level From Loop Test No. 4 Showing 2 mils of Subsurface Voids, and (b) Surface of Vapor Carry-Over Line Above Boiler. Specimen (a) was nickel plated to preserve edge during polishing. Specimens are in as-polished condition.

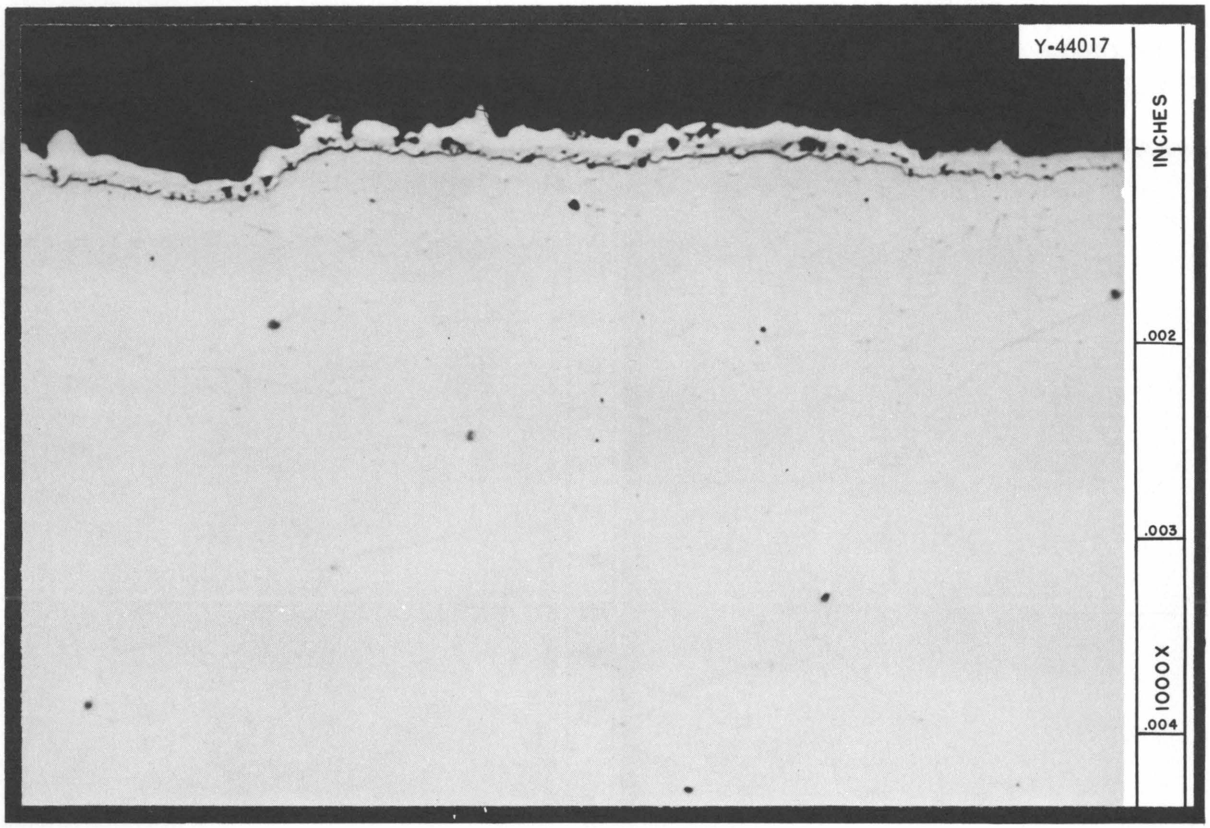

Fig. 43. Surface of Insert From Subcooler of Haynes Alloy No. 25 Boiling-Potassium Loop Test No. 4, Operated for 3000 hr at 870°C Condenser Temperature. Note mass-transfer deposit on surface of specimen. Specimen is in as-polished condition.

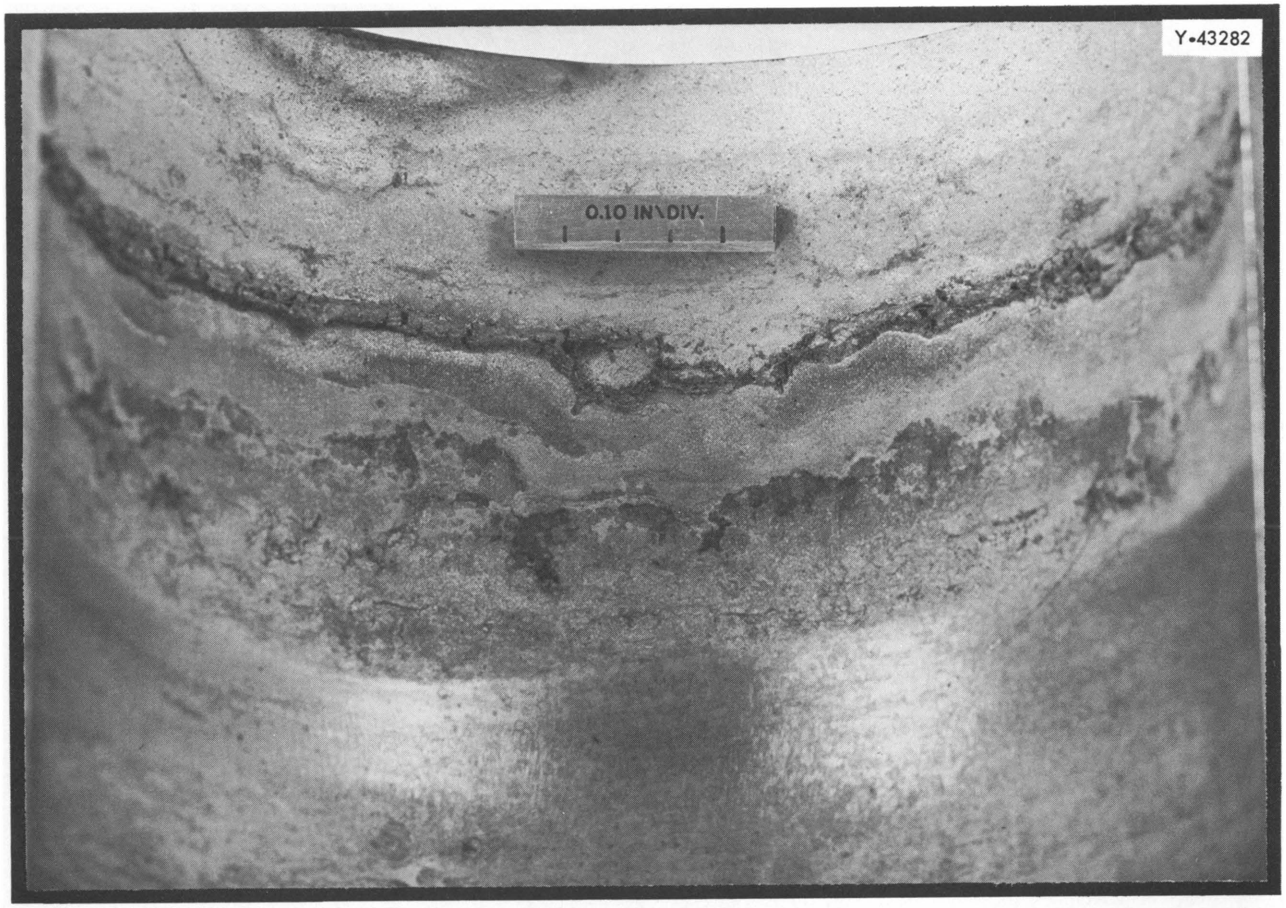

Fig. 44. Closeup of Ring of Metallic Crystals Found at the Liquid Level in the Boiler of Loop Test No. 4. These crystals were preferentially enriched in cobalt.

In line with the carbon increases found in the subooler specimens, x-ray analysis of the 1/2-mil deposit on these specimens (Fig. 43) showed the presence of $Cr_{23}C_6$ and Cr_7C_3. Chemical analyses of the ring of mass-transfer crystals located at the liquid level in the boiler showed the following constituents:

	Crystal Deposit	Nominal Haynes alloy No. 25
Cobalt	65	50
Chromium	7	20
Nickel	14	10
Tungsten	9	15
Iron	2	3

Tensile Tests

The results of tensile tests on the sheet specimens located in various regions of the loop are given in Table 18. The room-temperature ductility of specimens from the hottest region of the loop was slightly greater than that of control specimens heated in argon for the same period at the same temperatures. Conversely, specimens from the sub-cooled liquid regions of the loop were less ductile than control specimens heated in argon at similar temperatures. These effects are undoubtedly attributable to alterations in the carbon concentration of the specimens. The carbon contents of turnings from the insert (see Table 17) and of the tensile specimens from the loop (Table 18) indicate that the carbon had transferred to the components in the loop where low ductility was encountered. Specimens from the boiler, which had ductilities higher than values generally reported for solution-annealed material at 870°C, were depleted in carbon.

Haynes Alloy No. 25 Loop (Test No. 5, 980°C)

Weight Changes

The condenser inserts lost from 10 to 18 mg/in.^{2} (1.6 to 2.8 mg/cm^2), while the subcooler inserts gained 40 to 70 mg/in.^{2} (6.4 to 10.8 mg/cm^2). The overall weight-change profile is shown in Fig. 45. The condenser

Table 18. Results of Room-Temperature Mechanical Property Tests of Haynes Alloy No. 25 Control and Test Specimens[a] From Boiling-Potassium Loop Test No. 4 Operated for 3000 hr at 870°C

Temperature of Tension Test in Air	Specimen Exposure Environment	Exposure Temperature (°C)	Carbon Content[b] (wt %)	Mechanical Property Results: Elongation in. 2 in. (%)	Tensile Strength (psi)	Yield Strength[c] (psi)
					× 10^3	× 10^3
Test Specimens						
20°C	Boiler zone, liquid	870	0.056	12.0	150.6	72.6
	Boiler zone, liquid-vapor interface	870		9.5	153.9	77.0
	Vapor carry-over line	870	0.070	10.0	151.2	75.5
	Condenser, vapor	870	0.083	11.5	159.2	77.5
	Subcooler, liquid	730		5.5	162.2	90.0
	Preheater, liquid	750	0.180	2.5	151.6	89.2
870°C	Boiler zone, liquid	870		55.0	33.5	27.2
	Boiler zone, liquid-vapor interface	870		60.0	33.7	30.8
Control Specimens						
20°C	Argon (3000 hr)	870	0.087	9.0	153.4	78.3
	Argon (3000 hr)	815	0.087	7.5	165.8	88.6
	Argon (3000 hr)	760	0.087	4.0	156.0	91.3
	Argon (3000 hr)	705	0.087	10.5	154.0	114.0
	Argon (3000 hr)	650	0.087	13.0	169.0	137.0
	As-annealed	20	0.087	36.0	142.8	69.0

[a]All specimens were 0.040-in.-thick sheet, annealed at 1230°C for 30 min, and water quenched prior to exposure.

[b]Carbon concentration prior to potassium exposure: 0.087%.

[c]At 0.2% offset.

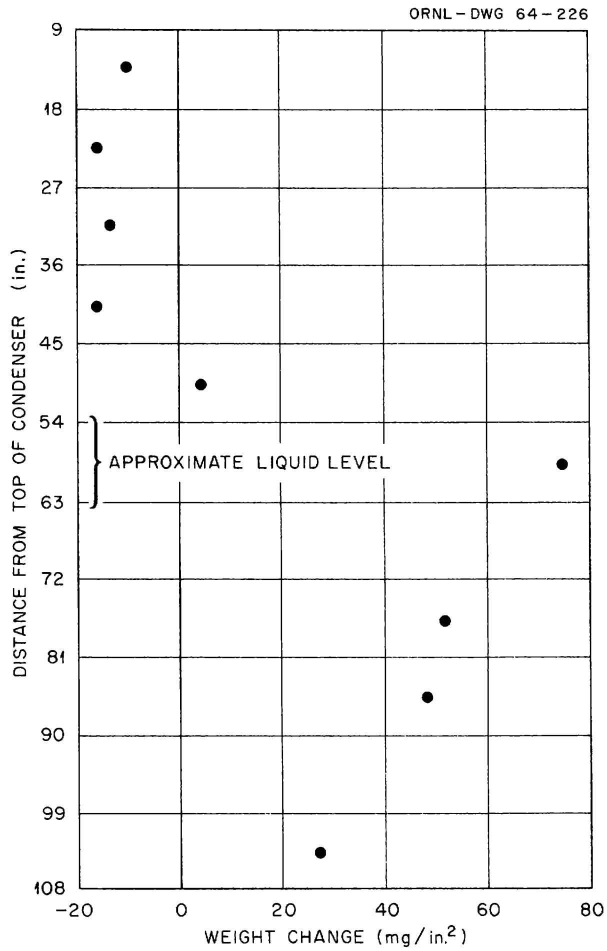

Fig. 45. Profile of Weight Changes of Welded Inserts From Condenser-Subcooler of Haynes Alloy No. 25 Boiling-Potassium Loop Test No. 5, 3000 hr at 980°C.

weight loss is slightly greater than that found in the first Haynes alloy No. 25 loop, which operated at a somewhat lower temperature (870 vs 980°C).

Visual and Metallographic Results

Inserts contained in the condenser section showed negligible corrosion beyond a slight surface roughening (Fig. 46a). Inserts located near the liquid level exhibited cracks as deep as 28 mils and a mass-transfer deposit approximately 7 mils thick (Fig. 46b). The mass-transfer deposits became heavier in the subcooler (Fig. 47), and reached a maximum thickness of 12 mils in the bend between the bottom of the subcooler and the

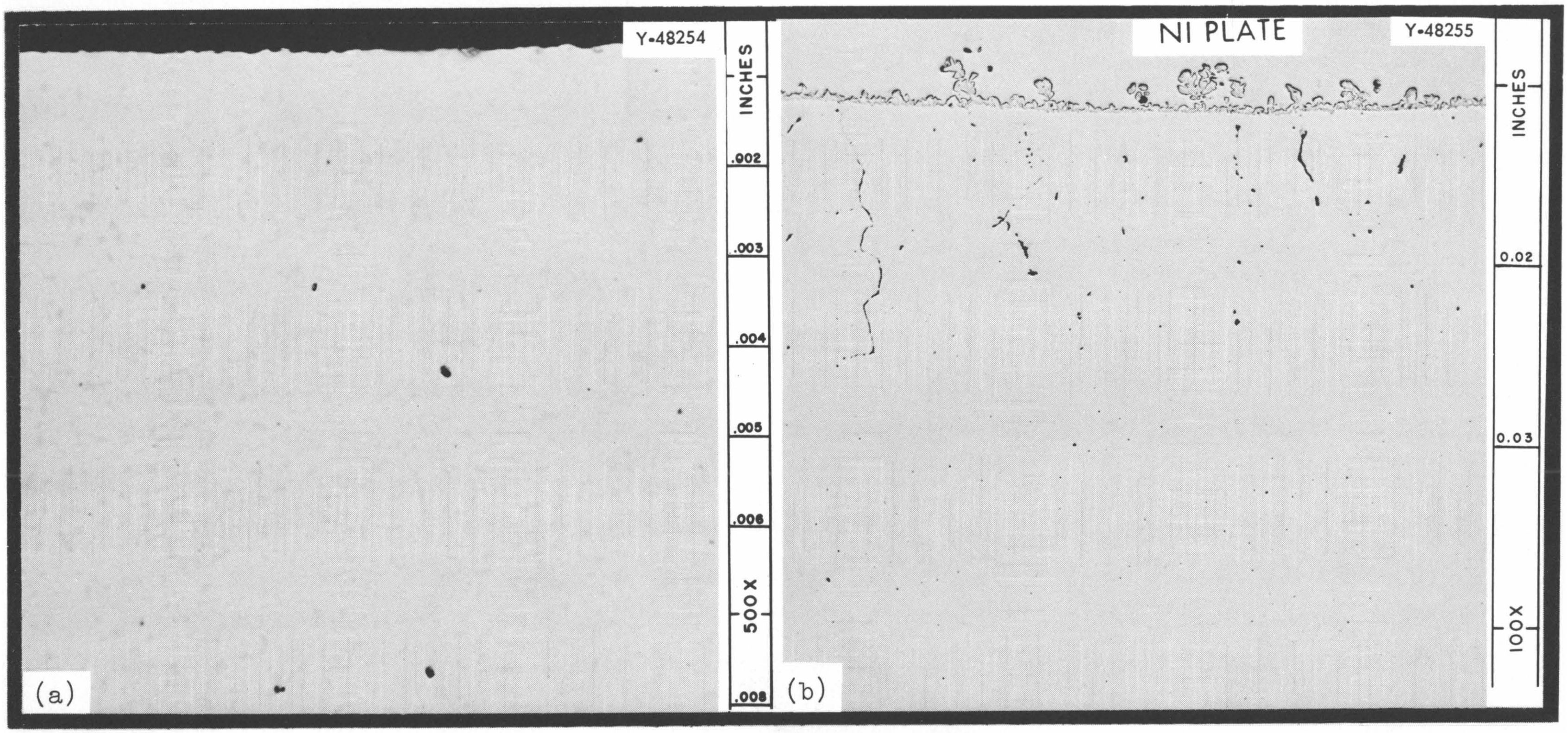

Fig. 46. Insert Specimens From Haynes Alloy No. 25 Boiling-Potassium Loop (Test No. 5) After Operation for 3000 hr at a Boiler Temperature of 990°C. (a) Condenser region. (b) Liquid-vapor interface region in condenser. Note mass-transfer deposits and cracks in (b). Specimen (b) was nickel plated for edge preservation during metallographic polishing. As-polished.

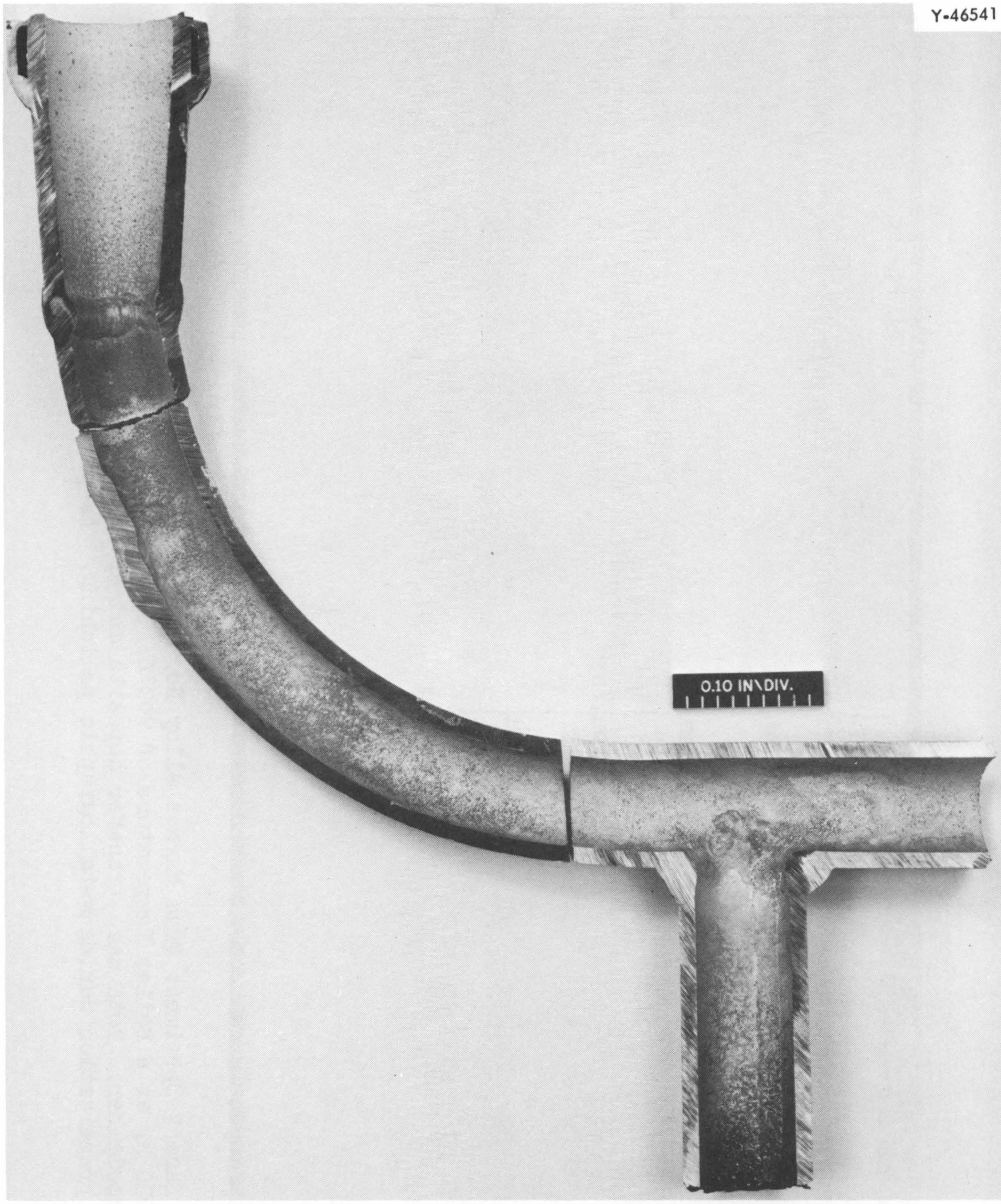

Fig. 47. Portion of Loop Between Subcooler and Preheater of Test Loop No. 5 Showing Extent of Mass-Transfer Deposit. The temperature in this region was 800°C.

preheater section, which had been maintained at 800°C during the test (Fig. 48). The amount of mass transfer deposits in this loop test were conspicuously greater than those found in the lower temperature Haynes alloy No. 25 loop (Test No. 4). This fact also manifests itself by comparing the insert weight changes (Figs. 19 and 45).

Metallographic examination of the boiler wall revealed a small amount of surface roughening, as shown in Fig. 49. However, the surface appearance was almost identical to that of a control sample heat treated in vacuum for 3000 hr at 980°C. Zyglo-penetrant inspection of the liquid and liquid-vapor interface regions of the boiler showed no cracks. This is in contrast to the first Haynes alloy No. 25 loop, which operated with an 870°C boiler temperature and showed severe cracking (Fig. 41 of this report).

Chemical Results

Chemical analyses of the mass-transfer crystals taken from the walls of the subcooler (see Fig. 48) are listed in Table 19. The analyses indicated that the crystals were enriched in iron, nickel, chromium, and manganese relative to the base material.

Table 19. Analyses of Crystals Collected From the Subcooler (800°C) of Haynes Alloy No. 25 Boiling-Potassium Loop (Test No. 5) Operated for 3000 hr at a Boiler Temperature of 980°C

Material	Composition of Crystals[a] (wt %)					
	Iron	Nickel	Chromium	Cobalt	Manganese	Tungsten
Crystal deposit	8.0	13.6	54.6	21.9	2.8	0[b]
Nominal alloy composition	3.0	10.0	20.0	50.0	1.4	15

[a]Adjusted to 100 wt %; elements listed totaled 82% of sample weight.

[b]Lower limit of detection was 0.5 wt %.

Chemical analyses (Table 20) of turnings taken from the surfaces of condenser and subcooler inserts indicate that carbon transferred from the

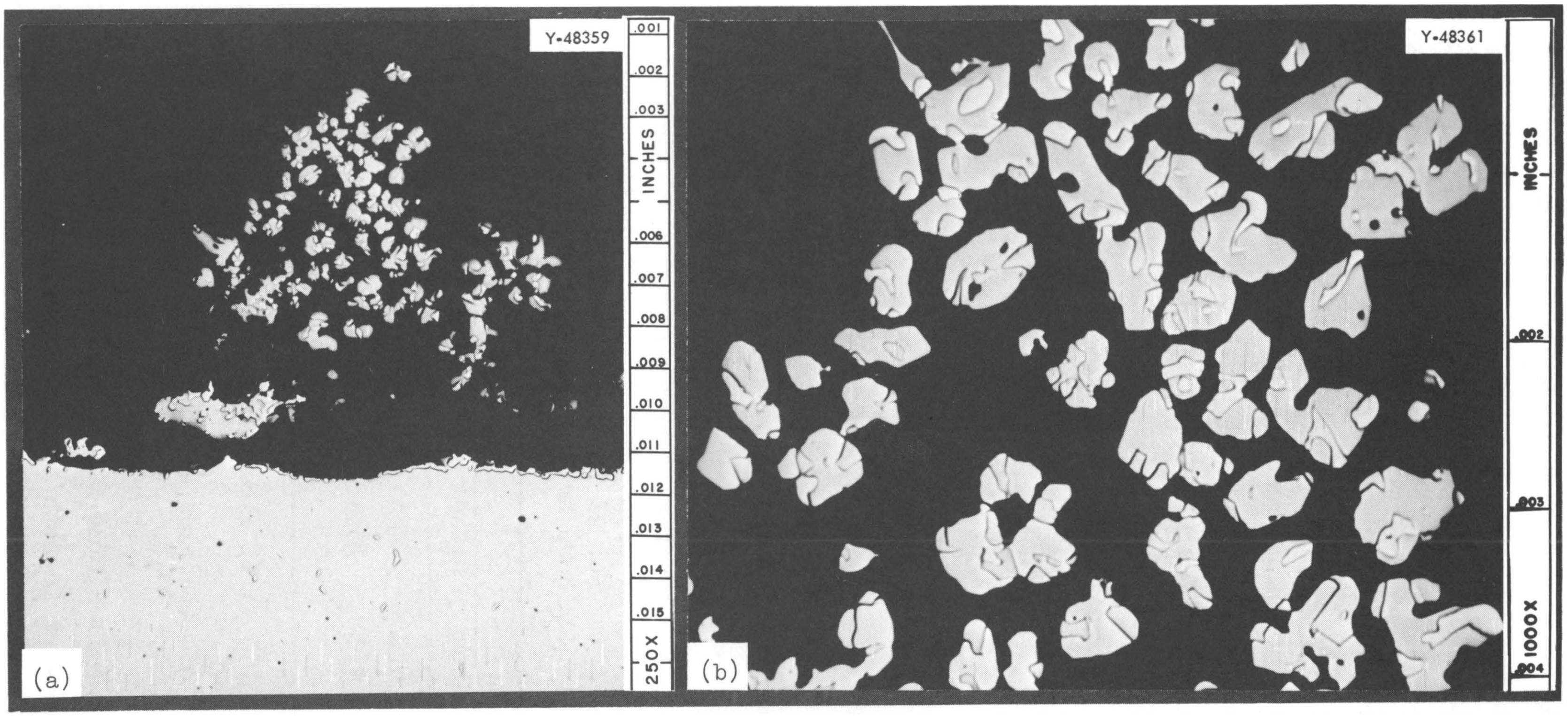

Fig. 48. Insert From Subcooler of Haynes Alloy No. 25 Loop (Test No. 5). (a) Mass-transfer deposit. (b) Deposit at higher magnification. Note that two phases are present in the deposit. As-polished. Reduced 8.5%.

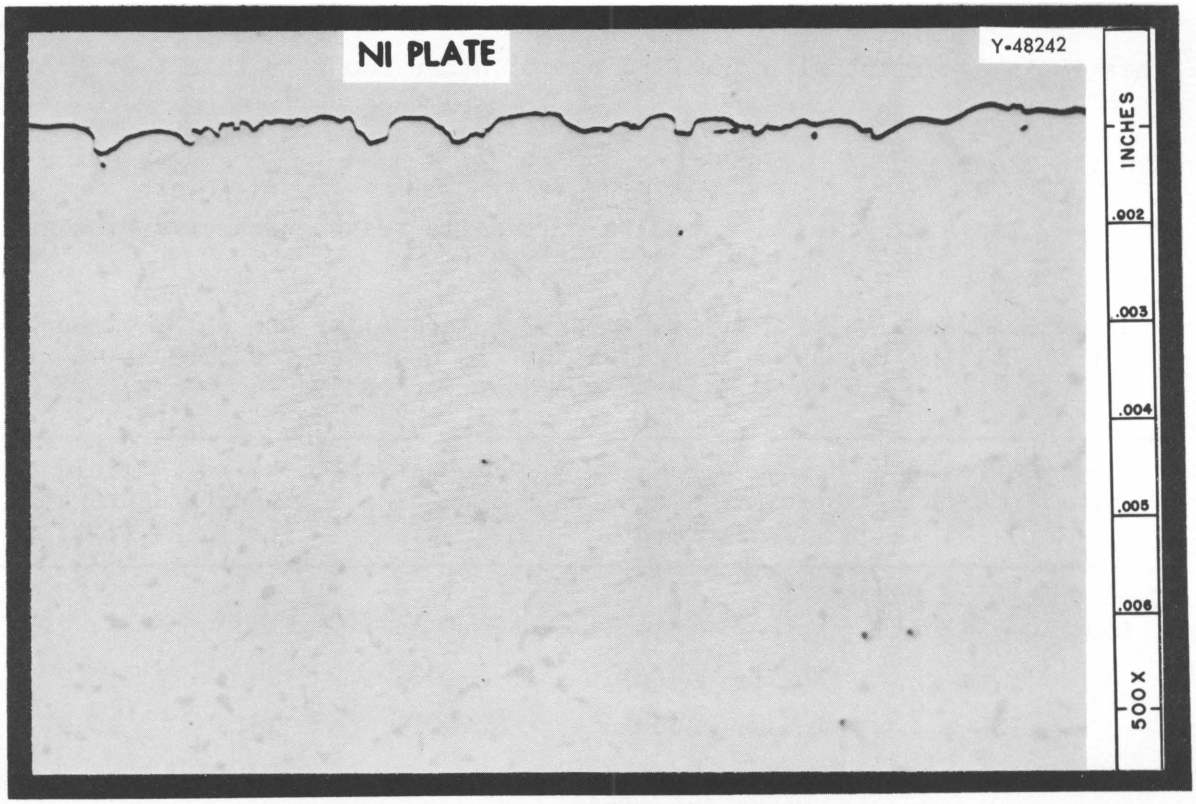

Fig. 49. Surface of Boiler at the Region of the Liquid-Vapor Interface From Haynes Alloy No. 25 Boiling-Potassium Loop Operated for 3000 hr at 980°C (Test No. 5). Specimen was nickel-plated to preserve edge during metallographic preparation. As-polished.

Table 20. Carbon Contents of Turnings[a] Taken From Various Regions of Haynes Alloy No. 25 Boiling-Potassium Loop (Test No. 5) Operated for 3000 hr at a Boiler Temperature of 980°C

Location of Turnings	Temperature (°C)	Carbon Content (wt %)
Condenser	980	0.04[b]
Subcooler	810	0.38[b]
As-received		0.09

[a]Total depth of turnings: 9 mils.

[b]Values are an average of at least three chemical analyses.

condenser and subsequently deposited and diffused in the subcooler regions. This is in agreement with the findings of other loops in this test program.

Tensile Tests

Table 21 gives the results of tensile tests on sheet specimens

Table 21. Results of Tensile Tests of Haynes Alloy No. 25 Specimens From Boiling-Potassium Loop (Test No. 5) Operated for 3000 hr with a Boiler Temperature of 980°C

Temperature of Tensile Test (°C)	Specimen[a] Exposure Environment	Elongation in 2 in. (%)	Tensile Strength (psi)	Yield Strength[b] (psi)
Test Specimens from Loop				
Room	Boiler, liquid	19.5	130.3	56.2
980	Boiler, liquid	50.0	18.5	15.9
Room	Boiler, liquid-vapor interface	18.5	137.7	60.0
980	Boiler, liquid-vapor interface	57.5	20.5	16.2
Control Specimens				
Room	Heat treated in vacuum	23.5	153.0	66.9
980	Heat treated in vacuum	48.5	22.3	18.1

[a]All specimens were 0.040 in. thick, annealed at 1180°C for 30 min, and water quenched prior to exposure; tests conducted in air.

[b]At 0.2% offset.

that were suspended in various locations around the loop and control specimens that were held in evacuated capsules at 980°C for 3000 hr. The mechanical properties of the specimens exposed in the liquid region of the boiler showed no significant differences from those exposed at the liquid-vapor interface at the same temperature. The room-temperature tensile and yield strengths of these specimens were significantly lower than those of the control specimens exposed to vacuum. However, when measured at 980°C (in air), the tensile and yield strengths of the specimens exposed to potassium were similar to the control specimens.

CONCLUSIONS

A rigorous corrosion analysis of the present loop systems is unavoidably confounded by the occurrence of boiling instabilities in all but one of the systems investigated (ironically, the first). These instabilities were responsible for the imposition of thermal fatigue cracks along many of the surfaces undergoing corrosion evaluation and also for undefined vapor conditions during a significant fraction of test operation. It is indeed unfortunate that at the time these tests were initiated (1960 to 1962) solutions to the boiling instability problem in potassium were unknown; in fact, it was through this series of tests that the magnitude and consequences of this problem were first recognized. In any case, due circumspection must be exercised in extrapolating the present results to systems where stable boiling is the normal mode of operation.

Metallographic examinations of condenser inserts as well as certain areas of the boiler wall revealed intergranular surface cracking in all but one test. The exception was the first type 316 stainless steel loop which operated under apparently stable boiling conditions. Cracks were most pronounced in the regions subjected to an oscillating liquid level but also occurred in other locations such as the vapor carry-over line where temperatures fluctuated rapidly. The Haynes alloy No. 25 loop which operated at 870°C was more susceptible to cracking, especially in the weld zones, than the Inconel and type 316 stainless steel loops.

Results of these loop tests indicate that type 316 stainless steel and Haynes alloy No. 25 have better resistance to corrosive attack by boiling potassium at 870°C than does Inconel. The weight losses per unit area in the condenser region (compared at an equivalent condensing rate) were of the same magnitude (8 to 10 $mg/in.^2$) for the stainless steel and the Haynes alloy No. 25 loops; however, the latter showed less overall corrosive attack in the liquid portion of the boiler region.

The relative condenser weight losses of Haynes alloy No. 25 loops operated at 870 and 900°C indicate that dissolutive corrosion of this alloy does not increase inordinately with increasing temperature. Furthermore at the higher test temperature, problems associated with aging embrittlement and fatigue cracking were less severe. These observations suggest that the excellent high-temperature strength of this alloy may give it potential for operating to approximately 1000°C as a container for boiling potassium.

Mass transfer deposits were found in the subcooler sections of all loops. In one loop, deposits extended into the preheater leg and the lower portion of the boiler; however, it is not certain whether their presence in these regions was caused by supersaturation and deposition or by washing from the subcooler during periods of boiling instability. The location of deposits strongly suggests that nucleation of mass transfer crystals takes place within the liquid rather than at loop surfaces, since the heaviest concentrations were found on surfaces which faced upward and on which settling apparently had occurred. Once settled, the deposits tended to be quite adherent to the wall. Assuming that a significant fraction of the mass transfer crystals were at one time suspended in the liquid, the use of filters or periodic flushing of the boiler could be quite beneficial in removing precipitated residues.

Analyses of mass transfer deposits taken from the cooler regions of the type 316 stainless steel loops indicated an enrichment in nickel and chromium. These deposits also contained relatively large amounts of carbon, and the presence of $Cr_{23}C_6$ type carbide was confirmed in x-ray patterns of the deposits. Deposits in the Inconel test systems were found to be enriched in iron but depleted in chromium. This latter finding is in contrast to the nickel-rich deposits collected in all-liquid Inconel-sodium systems operated in the past. Despite the compositional variations of deposits compared to the base metal, analyses of turnings taken from the condenser surface of each test system indicated no significant preferential leaching of major metallic constituents. This was verified by electron-beam microprobe analyses.

An additional mass-transfer effect manifested in each of the loops involved the migration of carbon. In all cases, surfaces exposed to dry or condensing vapor together with surfaces in the hottest portions of the boiler were depleted of carbon. Conversely, surfaces in the subcooler and preheater regions exhibited increases in carbon concentration. The transfer of carbon from the vapor region to the subcooler in each test had a minor but measurable effect on the short-time mechanical properties of the alloys.

It is not expected that carbon losses or gains of the magnitude encountered here will have a major effect on the creep properties of type 316 stainless steel. However, based on previous experience with type 304 stainless steel, some reduction in creep rate is expected as the carbon content of the material increases; attendant decreases in rupture ductility may also be anticipated.[14]

Obviously, the alloys examined in this study cannot be regarded as completely inert or noble container materials in boiling potassium. However, the potential of the alloys for potassium containment becomes greater when it is recognized that the condenser temperature in the present 870°C natural-circulation loops exceeds that of a typical Rankine cycle system operating at a comparable boiler temperature. This necessarily follows from the large temperature drop which is experienced across the vapor turbine of the latter system.

The major corrosion problem associated with a conventional iron- or cobalt-base alloy potassium system does not appear to stem from dissolutive attack _per se_ but rather from the formation of mass transfer crystals. Such crystals, while too few to pose a serious plugging problem, could lead to difficulties in such strategic locations as small clearance bearings. The nature and location of deposits, however, suggests that filters placed ahead of critical components may be effective in blocking the entry of deposits.

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[14] H. E. McCoy, Jr. and D. A. Douglas, Jr., _Effect of Environment on the Creep Properties of Type 304 Stainless Steel at Elevated Temperatures_, ORNL-2972, (Sept. 1962).

ACKNOWLEDGMENTS

Special thanks are extended to L. R. Trotter and J. W. Hendricks of the Metals and Ceramics Division who supervised the fabrication of all the test loops and the operation of four of them. Additional thanks are owed to J. C. Amos[15] and L. C. Fuller of the Reactor Division for their efforts in designing and operating the Haynes alloy No. 25 test loop that operated at 980°C boiler temperature.

The metallographic work associated with this investigation was performed by L. R. Shrader and E. D. Bolling of the Metals and Ceramics Division.

Thanks are also extended to the following groups and individuals whose efforts made this report possible: The Spectrochemistry and Special Analyses Groups of the Analytical Chemistry Division, the Graphic Arts Department, and the Reports Office of the Metals and Ceramics Division; W. C. Thurber and J. H. DeVan of the Metals and Ceramics Division for their review and criticism of this report and to S. Peterson for his editorial assistance.

[15]Now with the General Electric Company of Evendale, Ohio.

APPENDIX A

APPENDIX A – SUMMARY OF LOOP TEST RESULTS

Loop Material	Test Time (hr)	Temperature, °C (°F) Boiler (max)	Condenser	Subcooler (min)	Condensing Rate (g/min)	Insert Weight Change Condenser (mg/in.^{2})	Subcooler (mg/in.^{2})	General Metallographic Results	General Chemical Results
Type 316 Stainless Steel	3000	870 (1600)	870 (1600)	675[a] (1250)	180			b	c
Type 316 Stainless Steel	3000	910 (1670)	870 (1600)	675[a] (1250)	180	–8 (uniform)	+90 (maximum)	d	e
Inconel	1500	870 (1600)	870 (1600)	675[a] (1250)	180	–8 (uniform)	+50 (maximum)	f	c
Haynes alloy No. 25	3000	870 (1600)	870 (1600)	675[a] (1250)	180	–10 (uniform)	+40 (maximum)	g	e
Haynes alloy No. 25	3000	980 (1800)	950 (1740)	800[h] (1470)	300	–14 (nonuniform)	70 (maximum)	i	e

[a]Approximate temperature differential in test section: 675°C(1250°F).

[b]Subsurface voids at liquid level in boiler 2 mils deep. Surface roughening in condenser approx 0.5 mil deep.

[c]Transfer of carbon from condenser to subcooler. No preferential removal of Fe, Ni, Cr from condenser.

[d]Surface roughening to 0.002 in. in boiler. Subsurface voids in condenser 10–12 mils deep. Severe cracking in condenser at liquid level. Mass-transfer deposit in subcooler 8 mils deep.

[e]Transfer of carbon from condenser to subcooler.

[f]Intergranular attack to 20 mils in boiler; 15 mils in condenser. Mass-transfer deposit in subcooler 5 mils deep.

[g]Surface roughening in boiler to approximately 0.5 mil. Numerous cracks in boiler at liquid level. Severe cracking in condenser especially in weld zones. Scattered mass transfer in subcooler 0.5 mil thick.

[h]Approximate temperature differential in test section: 800°C(1470°F).

[i]Surface roughness to < 0.5 mil in depth in boiler and condenser. Mass-transfer deposit 7 mils deep in subcooler. Cracks to 28 mils in depth at liquid level in condenser.

APPENDIX B

APPENDIX B

Sample Calculations to Determine the Potassium Condensing Rate

From readings of the prevailing barometric pressure, the relative humidity, and the exit temperature of the cooling air, the density of this air was found to be 0.0381 lb/ft^3. With this value and the current manometer reading, an air velocity of 1745 ft/min was obtained. Then,

$$\begin{aligned} \text{Volume flow rate} &= \text{velocity (ft/min)} \times \text{area of exit pipe } (ft^2) \\ &= 1745 \text{ ft/min} \times 0.043 \text{ } ft^2 \\ &= 75 \text{ } ft^3/\text{min.} \end{aligned}$$

$$\text{Heat removed} = q = \text{vol flow rate } (ft^3/\text{min}) \times \text{density } (lb/ft^3) \times \Delta T(°F) \times \text{specific heat (Btu/lb-°F)} \times \text{conversion factor}$$

$$= 88{,}100 \text{ cal/min.}$$

$$88{,}100 \text{ cal/min} = x(\text{g/min}) \times \Delta H_{vapor} + x(\text{g/min}) \times C_p(\text{liquid}) \times \Delta T_{liquid}$$

where x is the condensing rate, ΔH_{vapor} is the heat of vaporization, C_p(liquid) is the specific heat of the liquid, and ΔT_{liquid} is the temperature drop in the liquid.

Solving, x = 180 g/min.

APPENDIX C

APPENDIX C

Identification of Phase at Condenser Surface From First Type 316 Stainless Steel Loop Test

The preferential etching techniques[16] used as an aid in identifying the prominent phase observed during metallographic examination of the condenser of the first type 316 stainless steel loop (see pp. 24–25) are outlined below. In Fig. 50, (a) shows the structure in the as-polished condition and (b) etched with a HCl-picral etchant to show most of the phases in relief. The same field is shown in (c) but etched with alkaline potassium ferrocyanide to outline carbides and ferrite, and upon long exposure faintly reveal sigma phase. The last photomicrograph (d) is the same area etched with electrolytic chromic acid, which attacks sigma or chi phases and carbides and outlines ferrite. The specimen was repolished between each step to remove the preceding etch.

Since no reagent is available to differentially stain sigma and chi, and due to the similarity of the chemical compositions of these phases, an x-ray diffraction pattern was obtained for the phase after its isolation. The patterns clearly identified the material as sigma phase.

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[16]P. K. Koh, "Occurrence of Chi Phase in Molybdenum-Bearing Stainless Steels," Trans. AIME (Feb. 1953).

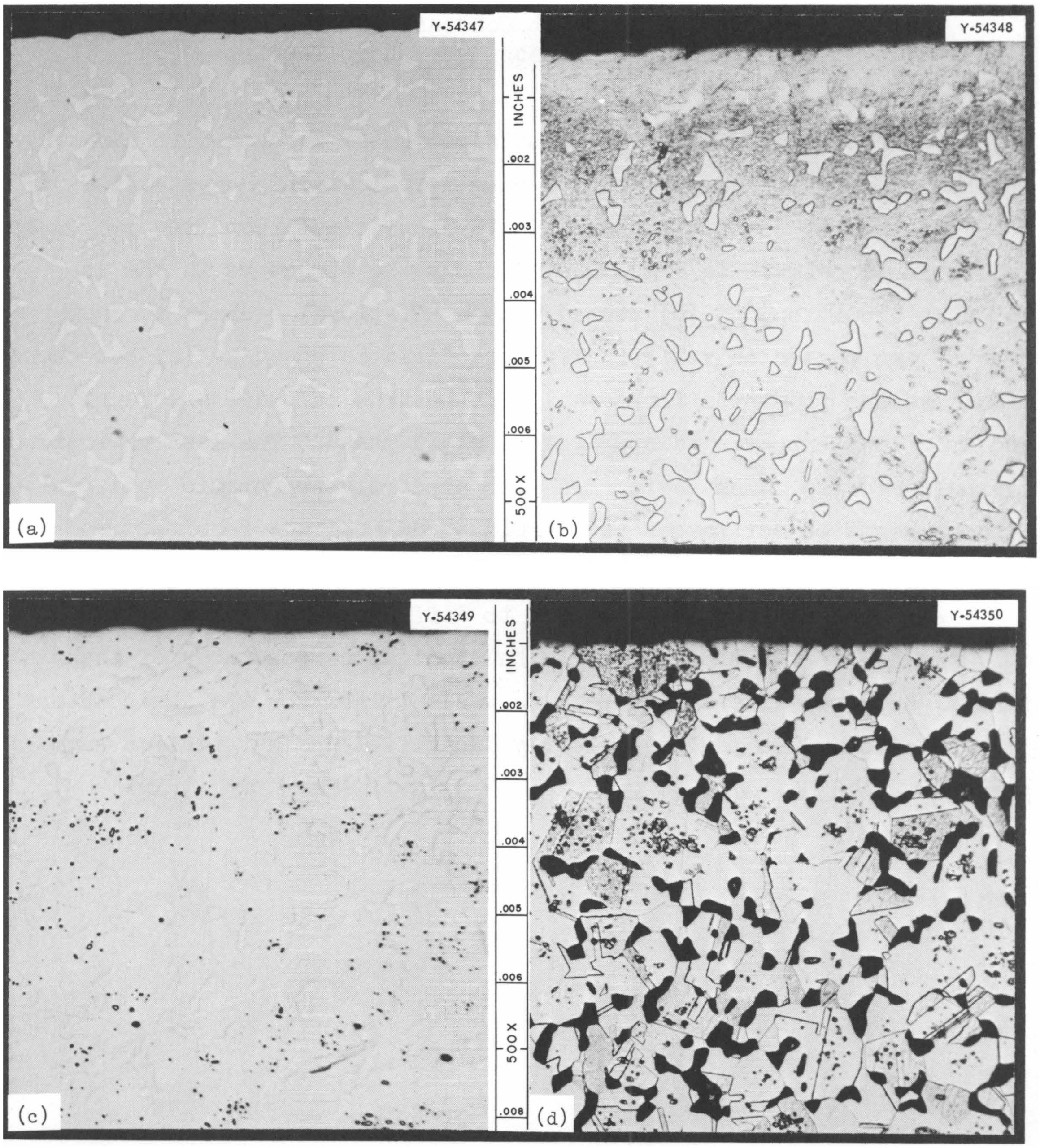

Fig. 50. Condenser Surface of First Type 316 Stainless Steel Loop Test, Showing Effects of Various Etchants on Chi Phase. See text for details. Reduced 22%.

APPENDIX D

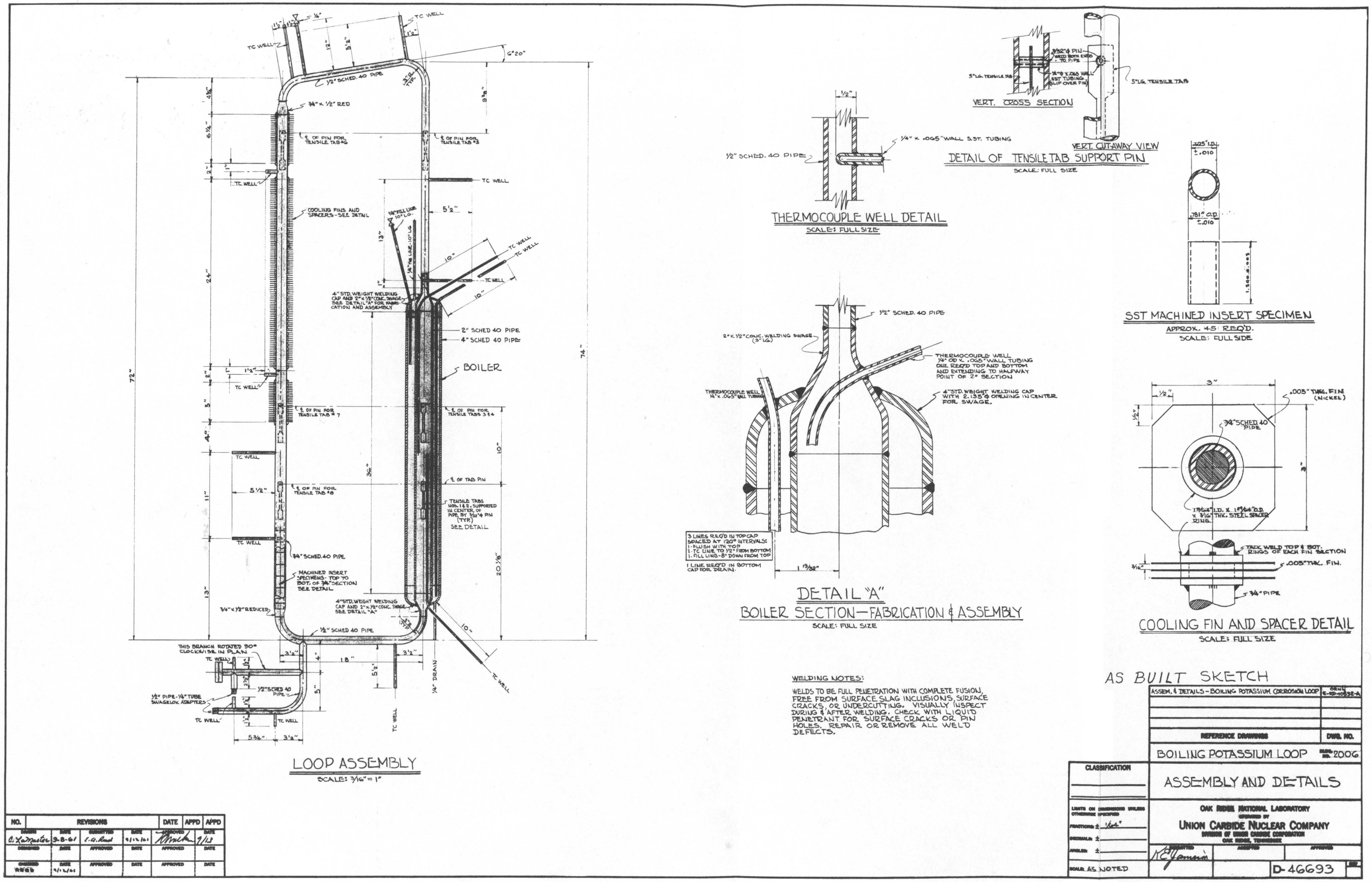

Fig. 51. As-Built Sketch Assembly and Details – Boiling-Potassium Corrosion Loop.

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