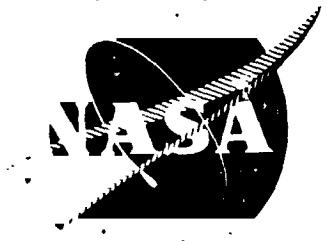


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# SNAP-8 MATERIALS REPORT FOR JULY - DECEMBER 1965

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
LEWIS RESEARCH CENTER

CONTRACT NAS 5-417



VON KARMAN CENTER  
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SEMIANNUAL MATERIALS REPORT

SNAP-8 MATERIALS REPORT FOR JULY - DECEMBER 1965

By H. Derow and B. E. Farwell

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

February 1966

CONTRACT NAS 5-417

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FOREWORD

The ultimate objective of the SNAP-8 Program is to design and develop a 35-kw electrical generating system for use in various space missions. The power source will be a nuclear reactor furnished by the Atomic Energy Commission. The SNAP-8 system uses a eutectic mixture of sodium and potassium (NaK) as the reactor coolant and operates on a Rankine cycle, with mercury as the working fluid for the turbogenerator. The SNAP-8 system will be launched from a ground base and will be capable of unattended full-power operation for a minimum of 10,000 hours. After the system is placed in orbit, activation and shutdown may be accomplished by ground command.

The Materials Program was under the management of M. F. Parkman, Head, Materials Section, Technical Support Department, SNAP-8 Division, Von Karman Center. Part of the work on this contract was performed at Aerojet-General Nucleonics under the direction of B. E. Farwell, Head, SNAP-8 Section, Metallurgy Department, Applied Science Division. The following engineers contributed to the various programs:

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The suggestions and guidance of W. Titus, Metallurgy Department, Aerojet-General Nucleonics; and P. Stone, SNAP-8 Project Office, Lewis Research Center, NASA, are gratefully acknowledged.

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Approved:



Robert Gordon, Manager  
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Distribution List

GLOSSARY

Abbreviations commonly used in the SNAP-8 Program are defined below.

Å	Angstrom units
AGN	Aerojet-General Nucleonics
ASIM	American Society for Testing Materials
BCC	Body-centered cubic microstructure
CL-4	Corrosion Loop No. 4
CTL-2	Component Test Loop No. 2
FCC	Face-centered cubic microstructure
HCP	Hexagonal close packed microstructure
Hg	Mercury
ID	Inner diameter
IR	Infrared
L/C	Lubricant/coolant
LNL	Liquid NaK loop
Mix-4P3E	Bis(mix-phenoxyphenyl)ether, a mixture of six isomers of bis(phenoxyphenyl)ether, the lubricant-coolant for the PCS
NaK	Eutectic mixture of sodium and potassium
NPS	Nuclear power system
OD	Outer diameter
ORNL	Oak Ridge National Laboratory
PCS	Power conversion system
P/N	Part number
PMA	Pump motor assembly
Rb	Rubidium
R <sub>B</sub>	Rockwell B (hardness)
R <sub>C</sub>	Rockwell C (hardness)
RPL-2	Rated Power Loop No. 2
SL-1	System Loop Test Facility No. 1
S/N	Serial number
SNAP	Systems for Nuclear Auxiliary Power



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GLOSSARY (cont.)

SS	Stainless steel
TAA	Turbine-alternator assembly
T-T	Tube-in-tube
VKC	Von Karman Center
XRD	X-ray diffraction

SUMMARY

Materials work during the second half of the 1965 calendar year provided data to guide the selection of materials for SNAP-8 system components. Metallurgical assistance was also provided in the design, development, fabrication, and testing of that system.

Test Operations Support

A study was conducted to evaluate the amount of wall thickness depletion that occurred in the 316 SS NaK containment pipe coils during operation of the two gas-fired NaK heaters used in the RPL-2 system (2300 hr for heater No. 1 and 3220 hr for heater No. 2), and to estimate the probable remaining heater life. The wall thicknesses were measured by the ultrasonic pulse-echo technique. Calculations based on estimated maximum oxidation-corrosion rates for the previous operating history, indicate that the heaters should operate for a minimum of an additional 9,100 hr before the wall thickness is reduced to the minimum allowed by the ASME Boiler Code.

Metallographic examination of a 7/8-in.-dia, 0.049-in. wall, 316 SS instrumentation tube removed from the Hg exit manifold of the -1 boiler, indicated that surface corrosion had occurred toward the hot end in the area of presumed Hg refluxing action. Assuming a constant corrosion rate, a wall depletion rate of 7.7 mils/1000 hr occurred. Presuming that this rate of wall depletion continued, it would have required slightly less than 5000 hr of total boiler operating time to reduce the tube wall thickness to the minimum allowable specified by the ASME Boiler Code for the existing vapor conditions. Instrumentation taps of 316 SS exposed to the above conditions would therefore not be satisfactory for a 10,000-hr system. It was recommended that 9Cr-1Mo or a 400 series SS be substituted for the 316 SS.

Mix-4P3E, (the L/C loop fluid) and an aliphatic hydrocarbon (probably Duoseal vacuum pump oil), as fluids and as thermally decomposed residues were found on the Hg-exposed surface in the tube-in-tube boiler and in the Hg inventory in the SL-1 system. The Hg loop and components were cleaned, however, later in the test sequence; recontamination was detected, apparently caused by an undetected pressure transducer failure that introduced silicone oil into the Hg loop.

The outer glass wrapping on the pipe insulation on NaK primary loop discolored during 1150 hr operation of SL-1. The discoloration is presumed to have resulted from slow elevated-temperature decomposition, at approximately 300°F, of the butadiene adhesive used in the outer layer of the piping thermal insulation system. It was further ascertained that the adhesive spontaneously and rapidly decomposes without a visible flame in the presence of quiescent air at some minimum temperature above 502°F but not exceeding 1065°F. The material spontaneously ignites and burns in quiescent air at some minimum

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temperature above 1065°F, but not exceeding 1200°F. The adhesive in the glass wrap of the loop pipe insulation system will add fuel to any fire in the test cell that raises the temperature of the material to the point of spontaneous ignition. Consequently, it was recommended that a review be made of the current usage of this adhesive in the test area.

A Rb injection system was installed and successfully operated during boiler tests conducted in the SL-1 test facility. The Rb addition sufficiently improved boiler performance to allow completion of boiler characterization tests. Depletion of the Rb content in the Hg occurred gradually, repeating the experience of previous loop tests on the SNAP-8 Program.

During PCS-1, Phase IV, Step 1 testing, Hg loop samples were obtained and analyzed before startup and during various loop shutdown periods. No contamination of the PCS loop during Step 1 operation was apparent. The Hg emergency dump system, however, contained a small amount of aliphatic hydrocarbon. A recommendation was made to clean this system prior to Step 2 testing to avoid possible contamination of the PCS from this source. The Hg from the main and emergency dump tank was slightly alkaline. The source of this contamination is unknown, but the quantity detected should have had no effect on system operation.

A test program was completed which indicated that Hg decontamination of components could be performed safely using a solution rather than a dewetting reaction. A 70% nitric acid solution containing 1 or 2% sodium nitrite (rust inhibitor) will adequately decontaminate components fabricated of 9Cr-1Mo, 316 SS, and low-carbon steels. No detrimental attack of the metals occurred as a result of immersion in the solution for up to 45 min.

An industrial survey indicated that the use of castings for NaK contaminant in the primary loop of the SNAP-8 system was not recommended for 10,000-hr service. Shorter time periods would be possible if periodic non-destructive evaluation of the component were performed to detect incipient leaks. Castings may be used in the other, lower temperature, NaK loops when the castings are thoroughly inspected prior to installation to ensure freedom from defects.

### Fabrication Support

Full-scale tube assembly samples representing the coiled tube-in-tube boiler were fabricated and evaluated to establish coiling limitations and design criteria. A 20.5-in. mean diameter coil sample contained no unacceptable metallurgical defects in either the 321 SS outer NaK contaminant pipe or the seven internal 9Cr-1Mo steel Hg containment tubes. It is concluded that fabrication of a SNAP-8 tube-in-tube boiler containing a mean coil diameter equal to, or greater than, 20.5 in. should not result in fabrication-induced discontinuities detrimental to 10,000-hr boiler operation life.

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A procedure utilizing a brazing alloy was established for rigidizing the interstage pressure transducers on the turbine assembly so as to protect against vibration-induced loosening during operation. Simulated joints produced with Easy Flo 3, a noneutectic silver brazing alloy applied by torch, resulted in satisfactory joints. Protection against Hg corrosion of the normally non-resistant silver alloy should be afforded by the primary seal of the Dryseal pipe thread used to mate the housing and the transducer fitting.

Stellite 6B Evaluation

Stellite 6B in the HCP condition appears to be considerably more brittle and less impact-resistant than material in the FCC condition. No decrease in tensile proportional limit or tensile yield strength was apparent, although a lower ultimate strength appears evident. The tensile properties of Stellite 6B at 1200°F do not degrade with continued exposure (up to 700 hr) at 1200°F. The static modulus of elasticity of material in the FCC or HCP condition is not significantly different. With care in design, handling, and operation, Stellite 6B in the HCP condition could prove a satisfactory alloy for SNAP-8 turbine use.

Statistically there is a 99% probability that 99% of the measured contractions attributable to the crystallographic transformation of Stellite 6B from FCC to HCP structure will fall between 0% and 0.223%. Therefore, the linear dimensions of Stellite 6B machined parts in the FCC condition will reduce when transformed to the HCP condition during turbine operation. The consequence may be an unsatisfactory change in the stack-up clearance of parts.

Transformation of Stellite 6B components from the FCC to the HCP condition can be expected over the operating period of the turbine, if the standard solution anneal and stress relief treatment is used (2250°F, air cool followed by 1650°F, air cool). The transformation may be caused by depletion of the matrix of carbon in the form of precipitation carbides. The carbon is a strong FCC stabilizer, and its removal changes the chemistry of the matrix such that the FCC structure becomes unstable and transforms to HCP with time at temperatures above 1100°F. The rate and ultimate degree of transformation remains unclear.

Complete transformation during fabrication was produced by thermal treatment of solution annealed material at 1650°F for 4 hr, followed by 1250°F treatment for 48 hr. Some material lots were more sluggish in going through metallurgical changes than others so that X-ray diffraction analysis is required to ensure completion of the reaction. By pretransforming turbine component material, potentially detrimental transformation during operation (because of volumetric contraction) can be avoided.

Based on test results produced thus far the FCC structure of Stellite 6B apparently can be stabilized against transformation to HCP by quenching

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from the solution annealing temperature rapidly during the heat treat procedure. Salt-quenched specimens exposed at 1065°F showed some slight transformation within the first 400 hr of exposure but beyond that point, up to 1083 hr accumulated thus far, no further change in either the X-ray diffraction pattern or hardness of the material has been detected. The microstructure remains essentially FCC.

### Alternate Turbine Materials Evaluation

Various alloys, considered as potential SNAP-8 turbine materials were exposed at elevated temperatures for a preliminary evaluation of their metallurgical stability. At 1065°F the alloys S-816, PH 15-7, Lapelloy, and 410 appear acceptably stable. At 1200°F all except S-816 appear to suffer a reaction which would, as a minimum, reduce their mechanical properties.

### Evaluation of -1 Boiler

After completion of a test series in RPL-2, the tube-in-shell -1 boiler was removed for material evaluation. The NaK side operated for 2350 hr and the Hg side for 1415 hr.

Dendritic-type NaK mass-transfer deposits, primarily iron with a significant amount of nickel and a lesser amount of molybdenum, were concentrated at the low-temperature NaK outlet end (1100°F). Some minor deposit was found at the NaK inlet end (1300°F). The mass transfer is presumed to have been caused by a generally prevalent uncontrolled (probably high) oxygen content of the NaK through most of the boiler operating life.

Microscopic examination of the NaK-exposed surface of the Hg containment tube revealed grain growth and concomitant decarburization (maximum approximately 0.030 in. deep), mass-transfer deposits (maximum approximately 0.002 in.), and tube cracking. The latter occurred at a 5-1/2 in. radius Hg tube bend at the Hg outlet manifold.

Macroscopic and microscopic examination of the Hg side of the tube revealed pitting (maximum depth, 0.0055 in.), mass-transfer (maximum height, 0.004 in.), unexplained surface decarburization toward the Hg outlet end, and surface cracking at various locations. The internal mild steel ribbon turbulator had collapsed during boiler operation, introducing possible flow discontinuities.

The cause of cracks near the end of the Hg inlet plug is obscure at present. The cause of cracks toward the Hg outlet end is apparently a pretest phenomenon, possibly resulting during tube manufacture. The cracks at a 5-1/2 in. radius tube bend at the Hg outlet manifold appear to have been caused by a pre-existing, excessively thin tube wall area resulting from tube ID-to-OD eccentricity produced during tube manufacture. This thin wall, combined with external surface decarburization and grain growth, 0.030 in. deep, produced circumferential expansion of the tube and enlargement of the tube diameter. Cracks in the tube wall resulted.

Corrosion Loop Program

Additions of mix-4P3E to mercury in 1/19 scale boiler loop tests caused degradation of boiler performance. The amount of mix-4P3E that would be necessary to degrade the performance of a full-scale SNAP-8 mercury boiler could not be determined from these tests. Increased liquid velocity in the preheat region of the boiler inlet plug increased the tolerance of the boiler to additions of mix-4P3E. Also, the increased liquid velocity decreased the time necessary for the boiler to regain its thermal performance or conditioned state. These tests indicate that surface contamination of boiler tubing can be a factor in producing poor thermal performance.

A multimaterial boiler inlet plug was designed and tested for 359 hr. This plug included the following materials in the preheat section:

- Carburized AISI 1020 steel
- Dynacut tool steel
- Nitrided-Nitralloy
- AISI 1020 steel
- Tantalum
- 18-4-1 tool steel
- 9Cr-1Mo steel

The AISI 1020 steel specimens, completely wetted by mercury, showed the highest corrosion rate. The other iron-based alloys were not wetted by mercury and suffered low corrosion rates. The tantalum section was not attacked by the mercury, indicating that erosion was not the predominant mechanism for loss of material in the preheat section of the boiler inlet plug. Since the entire test section in the boiler inlet plug was not uniformly wetted by mercury, the relative corrosion resistance of some of the materials tested is uncertain.

A program was completed to design and test in corrosion loop 4 (CL-4) a mix-4P3E mercury separator that would prevent mix-4P3E from entering the boiler by removing it from the Hg stream. After preliminary laboratory tests, a gravity type of separator was fabricated and installed in CL-4. With the use of a boiler inlet plug that gave a preheat liquid velocity of 6 fps, a series of mix-4P3E injections was made during loop operation to test the separator effectiveness in a dynamic loop. After each addition of mix-4P3E, no effect was noted on the boiler performance. At the end of the test, the total amount of mix-4P3E added to the loop could not be recovered from the separator. The test indicates that a gravity type of separator can reduce the amount of mix-4P3E entering the boiler and may prevent impairment of the boiler heat-transfer characteristics. Such a separator should be considered for SNAP-8 system loops.

Preliminary tests established that in situ X-ray evaluation of the CL-4 boiler Hg tube is feasible. The X-ray source is outside the boiler NaK containment pipe, and the radiographic film lies flat inside the Hg containment tube, across the tube ID.

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Tests were completed in component test loop 2 (CTL-2) to investigate the effect on boiler performance of adding rubidium to the mercury. A 25-g injection of mix-4P3E deconditioned the CTL-2 boiler, but the addition of Rb (1100 ppm) to the mercury caused boiler reconditioning. Operating time also aided the conditioning process. An effective oxidation method was used to remove Rb from the mercury. The decrease in Rb concentration during the run and the results of the mass balance on the Rb added and extracted from the mercury showed that some Rb was left in the loop.

I. TEST OPERATIONS SUPPORT

A. RATED POWER LOOP 2 (RPL-2)

This system is being operated to evaluate the performance of various SNAP-8 components, including the mercury boiler, TAA, condenser, and mercury PMA. During the report period, test support equipment was examined to establish the amount of corrosion that had occurred during system operation. An estimate was made of the amount of wall thinning which had occurred in the containment pipe of the gas-fired NaK heaters and of the amount of Hg corrosion that had occurred in a typical instrumentation line in the Hg-vapor portion of the PCS loop.

1. Gas-Fired NaK Heater Pipe Evaluation

Component and system tests at Aerojet-General require a heat source to simulate the nuclear power system (NPS) concurrently being developed by Atomics International. For this purpose, gas-fired NaK heaters are used that are capable of providing sufficient thermal power to heat the NaK to 1300<sup>o</sup>F under rated NaK primary-loop flow conditions. Two elements of operation may be detrimental to the service life of the NaK containment piping in the heaters. First, the pipe wall thickness may be reduced by removal of internal wall material by mass transfer - especially in NaK loops operated with an uncontrolled, high oxygen content. Second, oxidation of the external pipe surface may be caused by exposure to the heater flame and air. Cyclic loop operation can accentuate the oxidation rate due to spalling of the otherwise protective oxide coatings during heating and cooling.

The gas-fired NaK heaters used in the operation of RPL-2 had experienced many thermal cycling restarts during the life of the system. They has also operated for slightly less than 1000 hr without a NaK purification system which is required to control the oxygen content in the NaK (30 ppm of oxygen is the normal accepted maximum). During the operating period, a primary-loop massive NaK leak occurred resulting in significant oxygen contamination of the loop. Wall thickness measurements of the piping in two RPL-2 NaK heaters were made to ensure that oxidation and mass transfer had not unacceptably reduced the pipe wall thickness, and to estimate additional safe operating life.

a. Post-Operational Heater Pipe Wall Thickness

Ultrasonic pulse echo measurement of the NaK containment pipe wall of heaters No. 1 and 2 indicated an average thickness of



0.206 and 0.208 in., respectively. Because of data scatter only an approximation of wall loss rate and estimated remaining heater life could be made using a statistically determined post-operation heater-tube minimum wall thickness. The minimum wall thickness was statistically calculated such that with 99% confidence, 99.9% of the full NaK containment pipe length contained a wall thickness equal to or greater than 0.178 and 0.177 in. for heaters No. 1 and 2, respectively.

b. Corrosion/Oxidation Rate During Heater Operation

Determination of an exact corrosion/oxidation rate required knowledge of the pre-operation wall thickness of the heater pipes, which was not available. Therefore, an estimate was made of the maximum possible rate that could have occurred. The parts list of the heater required a 2-1/2-in. dia, schedule 40, 316 SS NaK containment pipe conforming to ASTM-A312. The permissible maximum wall thickness of this pipe is 0.220 in. Assuming that a pipe with this wall thickness was used, it is presumed (based on analytical considerations) that the wall thickness would have been reduced to 0.206 in. when the pipe was coiled to the NaK heater design configuration. This wall reduction would have occurred only in the area which experienced tensile stresses during coiling (the outside pipe wall with respect to the coil diameter), but this thickness was selected as the limiting case for analysis.

The following calculation yields the desired corrosion/oxidation rate:

$$C = \frac{(t_2 - t_1)(1000)}{T} \quad (1)$$

where

- C = maximum corrosion/oxidation rate, in./1000 hr
- t<sub>2</sub> = maximum pre-operational pipe wall thickness, in.
- t<sub>1</sub> = minimum post-operational pipe wall thickness, in.
- T = operating time of NaK heater, hr

Substituting,

$$C = \frac{(0.206 - 0.178)(1000)}{2300} = 0.012 \text{ in./1000 hr for heater No. 1}$$

$$C = \frac{(0.206 - 0.177)(1000)}{3220} = 0.009 \text{ in./1000 hr for heater No. 2}$$

c. Remaining Heater Life

Under operating conditions of the NaK heater (75 psi and 1500°F hot spot), the minimum pipe wall thickness allowed by the ASME Boiler Code is 0.068 in. Therefore, operation can continue until the pipe wall thickness is reduced an additional 0.110 or 0.109 in. for the two heaters using the estimated minimum pipe wall thickness (0.178 and 0.177 in.). Presuming that the above calculated maximum corrosion/oxidation rate (0.012 and 0.009 in./1000 hr for heaters No. 1 and 2, respectively) continues during future loop operation, the estimated additional minimum operating life is 9100 and 12,100 hours for heaters No. 1 and 2, respectively. If start-stop system cycling is minimized and the oxygen content of the NaK loop is continuously kept under approximately 35 ppm, the life of the heaters should be extended significantly beyond these estimates.

d. Conclusion

The remaining heater life is more than sufficient to fulfill the presently anticipated test operation requirements of RPL-2. However, it was recommended to test operation personnel that another wall thickness survey should be performed after an additional significant operating period to establish more reliable corrosion rates for these heaters.

2. Instrumentation Line Evaluation

A 3/8-in. dia, 0.049-in. wall, 316 SS instrumentation tube was removed from the Hg exit manifold of the -1 tube-in-shell boiler, S/N A-1, after 1415 hr of Hg operation to determine if the Hg-exposed surface had corroded. This instrumentation line was not used during boiler operation. (The cold end of this line had in fact been capped.) The hot end (connected to the boiler) is presumed to have been at the boiler Hg outlet temperature (approximately 1300°F); the cool, capped, end was a sufficient distance from the boiler to have been at 600°F or lower, as indicated by the degree of metal surface discoloration (temper color) on both the outside and inside surfaces. The tube was horizontal during boiler operation, a position which produces Hg condensing reflux action and consequential corrosion.

Metallographic examination of the tube indicated that general dissolution corrosion as well as pitting corrosion of the Hg-exposed surface had occurred toward the hot end in the area of presumed Hg refluxing action. The cold-end tube wall thickness was 0.051 in., but the pit-type corrosion toward the hot end had reduced the wall thickness to 0.040 in. Assuming uniform original tube wall thickness, and a uniform corrosion rate, a wall depletion rate of 7.7 mils/1000 hr occurred. If this rate of wall depletion continued, it would have required slightly less than 5000 hr of total boiler operating time to reduce the instrumentation tube wall thickness to the minimum allowable specified by the ASME boiler code (0.0125 in.) for the existing vapor conditions (1300°F and 265 psi). Thus, instrumentation taps

of 316 SS exposed to the above conditions would not be satisfactory for a 10,000-hr system. It was therefore recommended that 9Cr-1Mo or a 400 series stainless steel be substituted for the 316 stainless steel - in that portion of the instrumentation tube where significant Hg vapor refluxing action would be expected. A further recommendation was that the instrumentation lines should be sloped below the horizontal to minimize mercury condensing refluxing.

B. SYSTEM LOOP 1 (SL-1)

This loop is being operated to perform a 10,000-hr test on a SNAP-8 system containing all of the prototype components under development with the exception of the nuclear reactor. Gas-fired NaK heaters provide heat to the NaK primary loop inventory to simulate the reactor.

1. Hg Loop Contamination

Performance tests were conducted on the tube-in-tube (T-T) reference boiler (P/N 097444-5, S/N A-1) during September. After approximately 268 hr of operation the boiler had not achieved a fully conditioned state (Page V-5 of Reference 1). During the test period, periodic Hg analysis was performed. Indications were that contamination of the Hg loop had occurred as a result of (a) oil intrusion from the L/C loop (mix-4P3E fluid), and (b) Duoseal pump oil from the vacuum pumps. Corrosion products and particles of 316 SS base metal were also found. The 316 SS particles were attributed to uncontrolled loop assembly procedures. Infrared spectrophotometric (IR) analysis produced the following data:

<u>Date of Sample</u>	<u>Oil Content (g)</u>	
	<u>Mix-4P3E</u>	<u>Aliphatic Hydrocarbon</u>
7 Sept	1.9	1.9
14 Sept	9.5	9.5
22 Sept	4.0	16.4

The only aliphatic hydrocarbon fluid used in the system is Duoseal vacuum pump oil and it is presumed this is the contaminant detected.

The SL-1 boiler test was terminated and the seven boiler Hg inlet plugs were removed for examination. In the tight-pitch sections, the plugs appeared clean, but in other areas, the plugs contained a thin tenacious surface coating which was identified by X-ray diffraction analysis (XRD) as  $Fe_3O_4$  and Fe. One plug was immersed in nitric acid and then in hydrochloric acid to remove a coating sample. Combustion analysis (for carbon) of all of the acid-insoluble residues indicated that the coating contained 2.15 mg of carbon for the entire plug area. The six remaining plugs were washed with carbon tetrachloride. The solvent was collected as two separate samples - one from the high-velocity areas of the plugs,

and one from the low-velocity areas. The IR analysis indicated the presence of (a) 9 mg of vacuum pump oil and 2 mg of mix-4P3E in the solvent from the low-velocity areas of the plugs, and (b) 5 mg of vacuum pump oil and 0.065 mg of mix-4P3E from the high-velocity areas.

It was concluded that the plugs had been contaminated with vacuum pump oil and mix-4P3E, and subsequently exposed to an oxidizing atmosphere at elevated temperature. Probable thermal decomposition of the oil resulted in the deposition on the plugs of a carbonaceous residue. Previous work by Rosenblum at NASA's Lewis Research Center has shown that even a very thin oil film on a metal surface will inhibit wetting by Hg. Thus it is presumed that the oil contributed to, or caused the unsatisfactory boiler performance. Consequently, all mercury components were flushed with either trichloroethylene or 1,1,1-trichloroethane to remove all oil from the loop. The effluent rinse fluids were analyzed by IR to ensure that the loop was free of oil.

Cleaning was performed by Astro Pak Co., Downey, California. Essential steps included flushing with an inhibited sodium bisulfate solution at 180°F, an alkaline permanganate solution at 200°F, an alkaline rust-removing solution at 200°F, and a 40% nitric acid solution. Intermediate wash flushes were performed with deionized water. The boiler performed satisfactorily on restart and testing was completed during October.

Toward the end of this final test period, a Hg sample was removed from the top of the Hg dump tank inventory during a temporary shutdown. Silicone oil was found in the residue by IR analysis. This oil is used in the transducers for signal transmission, and it is postulated that the oil originated from rupture of the fluid cavity wall or inadequate cleaning of the transducer prior to installation. The presence of the oil had no apparent effect on boiler performance.

## 2. NaK Primary Loop Thermal Insulation Discoloration

During the 1150-hr SL-1 primary NaK Loop operation (1100 to 1300°F NaK), the outer glass cloth wrap of the thermal insulation covering the NaK containment pipe discolored to brown from as-cured white. The insulation system consists of Kaylo block insulation in direct contact with the NaK containment pipe. The block is covered by glass cloth outer wrapping. The wrapping consists of cloth held in place by an adhesive applied in a water solution which is subsequently cured by dehydration. The discoloration appeared very shortly after startup and darkened as operation continued. In several locations - notably over fittings protruding from components insulated with cement rather than Kaylo block - the overlayer of glass cloth wrapping was charred and blackened.

An investigation was completed (Reference 2) which indicated that the discoloration probably resulted from elevated-temperature decomposition of the butadiene adhesive which was applied to the glass cloth to form the outer

wrap. Thermal exposure tests of glass cloth wrap specimens indicated that discoloration of the outer surface of the insulation system was probably caused by air exposure at approximately 300°F during loop operation. Two additional independent temperature approximations support this conclusion. First, 300°F is considered by the supplier to be the maximum use temperature of the adhesive where slow decomposition would be anticipated. At higher temperatures the decomposition becomes more rapid and would have resulted in a greater change in the wrapping appearance than occurred. Second, the calculated temperature of the outer surface of the pipe insulation during loop operation is approximately 300°F.

The adhesive in the glass cloth wrap spontaneously and rapidly decomposes without a visible flame in the presence of quiescent air at some temperature above 502°F, but not exceeding 1065°F. The adhesive spontaneously ignites and burns in quiescent air at some temperature above 1065°F, but not exceeding 1220°F.

Although the discoloration does not appear detrimental to extended loop operating life, the adhesive in the glass wrap loop pipe insulation system will add fuel to any test-cell fire that raises the temperature of the material to the point of spontaneous ignition. Consequently, a recommendation was made to the Test Operations Group to review the current usage of this adhesive in test systems.

### 3. Rubidium (Rb) Injection System

The objectives of boiler performance mapping tests in SL-1 were not being met within the allowed time schedule because immediate boiler conditioning was not achieved on start up and subsequent run-in time was becoming excessive. Previous operating experience in the RPL-2 (Reference 3) and loop tests at Aerojet-General Nucleonics (Section VII of Reference 4), showed rubidium (Rb) to be an effective Hg additive to promote immediate boiler conditioning. It was decided that Rb would be added during the current test. The Rb injection system (Figure 1), designed and constructed at AGN, was transferred to VKC for this purpose.

After checkout and operation familiarization tests (Reference 5), Hg-Rb amalgam (0.8% Rb by weight) was successfully injected into the loop during boiler characterization tests conducted from 5 November through 7 November 1965. The anticipated improvement in boiler performance was realized (Section VI,C of Reference 6). During loop operation, a decrease in Rb content of the Hg was noted, duplicating operation performance (with Rb) of the RPL-2 system and the AGN loop. It may be presumed that at least one contributor to Rb depletion is air in-leakage or oxygen, and/or water vapor contaminants in the argon cover gas system.

A history of loop operation with respect to Rb additions is shown in Figure 2.

C. PCS-1, PHASE IV

During the report period, a management decision was made that planned full-system tests would be accomplished by upgrading the RPL-2 test loop. The T-T boiler tests in SL-1 were completed and the loop was shut down. To properly identify the upgraded RPL-2, it was redesignated as PCS-1, Phase IV.

In PCS-1, Phase IV, Step 1, several sources of organic contamination of the Hg loop were possible. Previous work had shown that these organics may degrade the performance of Hg boilers. These sources include leakage of mix P3E through the Hg PMA seal, diffusion of vacuum or diffusion-pump oil into the system, rupture of diaphragms on the Hg side of the fluorinated hydrocarbon or silicone-oil-filled pressure transducers, and organics inadvertently introduced during loop construction.

Conversely, the presence of metallic alkaline metals in mercury can improve boiler performance by enhancing heat transfer from the boiler tube wall to the Hg. The most obvious source of alkaline metal is Rb residues left in the loop piping as a result of previous boiler tests with a Rb addition in the Hg. Although the Hg inventory was changed and the loop components were cleaned or changed, no attempt was made to remove Rb surface-adhering residues from any loop piping remaining from previous RPL-2 tests.

To determine the presence of organic or alkaline metal contamination in the PCS loop during Step 1 testing (22 November to 13 December 1965), Hg samples were obtained and analyzed before system operation, during various loop shutdown periods and at the conclusion of the test sequence.

Analyses of Hg samples from the main dump tank during Step 1 operations (Reference 7) indicated that no organic contamination of the Hg loop occurred. However, the mercury surface residue in the emergency dump tank contained a small amount of an aliphatic hydrocarbon. The only known fluid of this type used in the system is Duoseal vacuum pump oil. Under normal operating procedures, this contamination would not be transferred from the emergency Hg dump tank to the PCS loop. However, volatilization of this organic and subsequent diffusion into the loop, could occur during an emergency dumping operation, or in the event that emergency dump tank valve leakage should occur. Therefore, it was recommended that the emergency tank and its associated lines be cleaned prior to the initiation of Step 2 testing.

Analysis of samples from both the main and the emergency dump tanks indicated the Hg was slightly alkaline (0.01 - 0.50 ppm, calculated as potassium) after system operation. The source of the alkalinity is not apparent at present, but no effect on system operation would be expected to result from the low concentration detected.

D. Hg DECONTAMINATION STUDY

Decontamination of Hg-exposed components may be accomplished by either dewetting the metal surface so that the residual Hg "balls up" and rolls off, or dissolving the Hg through use of a proper solvent. A study was conducted to evaluate 30 to 50% strength nitric acid solutions. Nitric acid is generally used to clean acid-resistant parts such as 300 and 400 series stainless steels. It will also dissolve Hg. Preliminary tests of 46 to 70% nitric acid solutions without inhibiting additives establish that the optimum solution for Hg solution be between 62 and 70%. Therefore, in this evaluation 62 and 70% nitric acid solutions were tested, some of which also contained 1 or 2% sodium nitrite rust inhibiting additive. The following characteristics of nitric acid solutions were evaluated.

- Acid solution strength which provides optimum Hg dissolving power
- Effect of sodium nitrite (rust inhibitor) additive
- Compatibility of the SNAP-8 containment materials with candidate solutions

The containment materials were 9Cr-1Mo steel, 316 SS, and plain carbon mild steel (AISI 1010).

1. Solvent Evaluation

a. Procedure

A known weight of mercury was placed in the bottom of a special test tube\* in a  $20 \pm 1^\circ\text{C}$  bath. The tube was fitted with a stirrer\*\*, and 75 ml of a candidate solvent solution was quickly poured over the Hg. The solution was stirred for a time interval and then removed by flushing with distilled water. The unreacted mercury was vacuum-dried and weighed.

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\* 12-mm-OD by 30-mm-long tube attached to the bottom of a 30-mm-OD by 180-mm test tube. The smaller tube section held the Hg, and had a  $0.95 \text{ cm}^2$  cross-sectional area.

\*\* The stirrer blade was positioned 1.5 in. above the Hg surface.

b. Results

The rate of dissolution per unit time (minutes) not only was markedly different for the various acid concentrations but also changed rapidly during the exposure of the Hg to each solution. To more accurately correlate the reaction time with a convenient time span for Hg component decontamination, the test data were plotted (Figure 3) as the total weight of Hg dissolved as a function of elapsed time.

In most tests of the uninhibited 70% nitric acid, without additive, natural inhibition with respect to dissolution of Hg occurred. This reaction appeared to be caused by a precipitated (within the first minute of Hg acid contact) white mercurous nitrite film which coated the Hg surface, preventing further dissolution. Where precipitation did not occur, the Hg dissolution rate was very high (as illustrated by the one test point in Figure 3) the cause of the precipitate is unclear. Tests on acid solutions containing a sodium nitrite rust inhibitor showed that a solution of 1% sodium nitrite in 62% nitric acid increased the mercury dissolution rate for approximately the first 4 min, but then the rate approached that of the acid with no additive. The presence of 2% sodium nitrite did not appreciably affect the mercury dissolution rate produced by the 62% nitric acid alone.

The addition of 1 or 2% sodium nitrite to a 70% nitric acid solution significantly reduced the tendency for the inhibiting white precipitate to form. During an occasional test, the precipitate appeared with consequential marked reduction in the Hg dissolution rate. The marked scatter of the data points in Figure 3 from the presumed curves for these two solutions is indicative of the varying and unpredictable reaction.

2. Compatibility Tests

Compatibility tests were run to determine if corrosion of SNAP-8 Hg containment materials (9Cr-1Mo steel, 316 SS, and low-carbon mild steel) would result from exposure to any of the Hg decontamination solutions tested during the above solution evaluation.

a. Procedure

Specimens of 1-1/2 in.<sup>2</sup> surface area were prepared using material previously exposed to Hg in the SNAP-8 test system. They were cleaned using detergent and acetone, then weighed. The specimens were then immersed for 45 min in the various test solutions at 75°F, removed, washed in tap water, dried, reweighed, and then subjected to metallographic examination.

b. Results

None of the solutions detrimentally attacked 9Cr-1Mo or 316 SS. The weight losses were converted to depth of material removed



based on uniform surface attack. The specimens were exposed for a time considered liberal for component decontamination requirements. The depth of metal removal was only slightly more than 0.1 mil, well within acceptable limits. Low-carbon mild steel was compatible only with the nitric acid solution containing the sodium nitrite addition. The depth of metal removal was slightly less than 0.15 mil, also within acceptable limits. Metallographic evaluation showed that the solutions considered compatible with the various materials did not produce detrimental surface attack such as intergranular penetration.

### 3. Conclusions

Based on the results of both test series, it was concluded that a 70% nitric acid solution with 1% sodium nitrite additive (to provide rust inhibition) is optimum for use in Hg decontamination of components. These solutions should be used when it is necessary, or desirable, to decontaminate parts using a solvent-solute, in preference to a dewetting, reaction. However, if a white precipitate appears in the solution during decontamination, the solution should be discarded and a fresh solution prepared and used.

#### E. USE OF NaK EXPOSED CASTINGS

NaK PMA 347 SS impeller housing castings contained localized excessive porosity that resulted in NaK leaks during testing in LNL-3 (References 8, 9, and 10). An attempt to establish an adequate non-destructive acceptance test procedure was unsuccessful due to the shape of the casting, its wall thickness, and the distribution pattern of the porosity. Sealing of the suspected defective area of an as-received casting by brush-plating with iron was attempted but was not successful. Cast housings were replaced with a weldment of wrought material. Satisfactory performance resulted.

As a result of this experience with casting porosity and NaK leakage, a survey was made to establish recommendations for (1) the future use of castings for NaK service in the SNAP-8 system, and (2) disposition of valve body castings currently a part of the NaK systems in RPL-2 and SL-1.

Various organizations experienced in liquid alkali metal system operation were contacted to establish the current practice in the use of castings in such systems. A summarization of the recommendations resulting from this survey follows.

1. Valve, pipe fitting, and other NaK containment castings in the RPL-2 loop should be X-rayed to determine if incipient wall cracking or penetration has occurred. Castings with defects should be replaced. Sound components may continue in use. However, during future shutdowns, when practical, the castings should be X-rayed again to ensure continued serviceability.

2. NaK containment castings should not be used in the primary NaK loop of SL-1 for the 10,000-hr test.

3. NaK containment castings may be used in the heat rejection and radiation simulator loops of SL-1. However, such castings should be inspected by radiography and helium leak ( $10^{-7}$  std cu cm/sec allowable leak rate limit) to ensure freedom from defects. It is also recommended that these valves be inspected by radiography and helium-leak test whenever a shutdown occurs after an accumulated operating period of approximately 1000 hr after a previous inspection.

4. Non-NaK containment castings, such as NaK PMA impeller, can be used.

## II. FABRICATION SUPPORT

### A. SPECIFICATIONS

The following materials and process specifications were issued or modified during the report period:

<u>No.</u>	<u>Title</u>
AGC-10210B	Bars and Forgings, Cobalt-Base Alloy (Stellite 6B)
AGC-10214B	Tubing, Austentic Type 316 SS Seamless, Corrosion Resistant
AGC-10227C	Welding, Fusion, Liquid Metal Containment Materials
AGC-10227/2A	Welding, Fusion, 316 Stainless Steel
AGC-10227/5A	Welding, Fusion, Haynes Stellite 6B Steel
AGC-10227/6	Welding, Fusion, NaK Contaminated Piping, Procedure for
AGC-10319/6 Amend. 3	Cleaning of SNAP-8 Power-Conversion System Components Prior to Final Assembly, Procedure for
AGC-STD-1272	Cleaning, Metallic Parts
AGC-STD-1273	Tubing and Pipe, Steel, Bending Methods and Procedures

### B. TUBE-IN-TUBE BOILER

In November 1964, the SNAP-8 reference boiler design was changed from a tube-in-shell configuration to tube-in-tube. The new conceptual design

consists of a helical tube assembly coil of seven 9Cr-1Mo tubes contained within a 321 SS pipe (see Figure 4). During fabrication planning, companies with experience in coiling tube bundles comparable to the new SNAP-8 boiler could not be found. Full-scale tube assembly samples, consisting of one 360° coil, were fabricated to establish coiling limitations and boiler design criteria. They were coiled to a 20.5 in. mean diameter with various internal tube reinforcing materials. The 20.5 in. coil diameter represents a degree of conservatism in boiler design adequacy because the reference design consists of a 36 in. diameter coil. Specimens were removed from these samples and metallurgically evaluated (Reference 11).

The first assembly contained convolutions in the 321 SS at the inside compression surface of the entire coil length. The convolutions did not detrimentally affect the microstructure of the material and the tube appeared metallurgically capable of withstanding the imposed stresses of boiler operation; however, the convolutions were hydrodynamically unacceptable. The second tube-in-tube assembly evaluated was produced without convolutions and represented the developed production procedure for fabricating a boiler. The 9Cr-1Mo steel tubes were slightly work-hardened during coiling, but a flattening test (complete flattening of tube sections without incipient fracture) indicated no apparent detrimental loss in ductility.

Metallographic evaluation and microhardness measurements indicated that the 321 SS pipe was work-hardened by the coiling, but flattening tests showed that the material retained sufficient ductility for satisfactory service. Incipient fracture during the flattening test occurred in the area of the pipe subjected to tension stresses during coiling, but the amount of flattening that could be effected prior to fracture was well within the specified limits of ASTM specification requirements.

It is concluded that fabrication of a SNAP-8 tube-in-tube boiler containing a mean coil diameter 20.5 in. or greater should not result in fabrication-induced discontinuities detrimental to 10,000-hr boiler operation life.

### C. TURBINE ASSEMBLY

Analysis of data from the first TAA tests showed that interstage pressure measurements were required for complete performance evaluation. The subsequent assembly, S/N A-1, required this added instrumentation but fabrication of the 9Cr-1Mo steel turbine case had proceeded to a point where conventional welding of the interstage pressure taps was not practical. The 1350° F post-weld stress relief, required for 9Cr-1Mo steel, would probably have produced unacceptable case distortion. The selected non-welded joint consists of a mild steel fitting screwed into the 9Cr-1Mo steel TA case using a Dryseal pipe thread. The Dryseal pipe thread is presumed to provide a sufficient seal against housing leakage at the highest operating temperature, 1000° F, based on its normal application for high-pressure, high-temperature gas systems. The seal results from localized thread distortion produced by

applying a sufficient torque to the fitting during assembly. Brazing was selected to protect the assembly against loosening of the fitting which would lead to Hg vapor leakage during operation. A study was conducted to select a proper brazing alloy and establish an adequate brazing procedure.

Only silver brazing alloys exhibit a remelt temperature above the TA operating temperature and allow brazing below the transformation (hardening) temperature (1510°F) of the 9Cr-1Mo steel turbine case. It is presumed that since the Dryseal pipe joint is sufficient to maintain a proper vapor seal, Hg vapor leakage or refluxing action through the thread to the braze alloy should not occur. Any leakage which might occur through the Dryseal joint should be sufficiently small that dissolution of the braze joint is not anticipated.

Wetting and flow tests, using heliarc and acetylene-oxygen torch heating methods, were conducted on three candidate brazing alloys manufactured by Handy and Harman Brazing Products, Los Angeles.

Alloy	Constituent Elements (%)					Melting Point (%)	Flow Point (%)
	Ag	Cu	Zn	Cd	Ni		
BT	72	28	-	-	-	1435	1435
Easy Flow 45	45	15	16	24	-	1125	1145
Easy Flow 3	50	15.5	15.5	16	3	1170	1270

Easy Flow 3, applied by torch heating, was selected as the optimum combination of alloy and process. This material is a non-eutectic alloy. Although it exhibits less tendency to flow into very small crevices than the other alloys it possesses better filleting characteristics. Both of these factors are advantageous since less than optimum brazing joint clearances are available.

Simulated joints were successfully brazed using a scrap 9Cr-1Mo turbine case, Figure 5. Visual and metallographic examination showed that flow of the alloy into the threads of the joint was complete although limited to the first engaged thread in the housing because of the pinchoff produced by the intimate contact of the threads. Overheating of the joint had not occurred, as indicated by the absence of martensite in the 9Cr-1Mo case microstructure and a hardness of  $R_B 87$ . The procedure was incorporated into the TA fabrication plan.

### III. STELLITE 6B EVALUATION

Stellite 6B\* is a high-temperature cobalt-base alloy which exhibits excellent mercury erosion resistance and acceptable mercury corrosion

\* Cobalt-base-alloy containing 30% Cr, 4.5% W, 1.5% max. Mo, 3% max. Fe, 1.15% C, and 3% max. Ni.

resistance for use as the reference SNAP-8 turbine rotor and nozzle material. The turbine assembly (S/N 3-2) operated in the SNAP-8 RPL-2 system for 820 hours; then a premature failure occurred. Post-failure evaluation indicated that the precipitating cause of failure was probably unanticipated softening of Spiralox retainer springs caused by the inadvertent use of mild steel rather than a more temperature-resistant alloy. During this post-test analysis, a hitherto unreported crystallographic transformation in the Stellite 6B was detected that had apparently resulted from exposure at the turbine operating temperatures (700-1238°F). Until the metallurgical evaluation of the components of the turbine (S/N 3-2) was made, it had been thought that Stellite 6B was a stable face-centered-cubic (FCC) alloy. No information of the properties of this alloy in the transformed condition, HCP, was available from the supplier, Haynes Stellite, Kokomo, Indiana.

Based on later data, it is estimated that the turbine parts which operated at the higher temperatures were in the HCP condition for 500 or more hours, and some of the parts had relieved locally high operating stresses by cracking during operation. After these stresses had been relieved, the parts continued to function until mechanical failure occurred, caused by the retaining spring. The turbine failure was not a result of the Stellite 6B transformation; however, had the retaining spring failure not occurred, the capacity of the Stellite 6B parts to last 10,000 hr was questionable. For this reason, an investigation was started to evaluate the effects of transformation on the properties of Stellite 6B.

It was found that this transformation of crystallographic structure is accompanied by volumetric contraction and loss of ductility and impact strength, along with other changes in mechanical properties. It was concluded that Stellite 6B is a satisfactory material provided that fabrication procedures are changed to thermally induce the transformation during component fabrication, and component designs are reviewed to compensate for the estimated results of transformation from the FCC to the HCP structure.

#### A. MECHANICAL PROPERTIES

Attempts to obtain literature data on the mechanical properties of Stellite 6B in the HCP condition were unsuccessful since this transformation had not been previously reported. It was postulated that the HCP structure would be more brittle and less impact resistant based on results of the TAA S/N 3-2 failure analysis, and therefore would be less desirable than the FCC form. An initial investigation discussed below under "Transformation Studies," indicated that the transformation could be expected during turbine operation. A limited investigation was completed (Reference 12) to determine qualitatively some of the Stellite 6B, HCP, properties. Only a trend was established because of a lack of test material.

Stellite 6B in the HCP condition tends to be considerably more brittle and less impact-resistant than material in the face-centered cubic condition. From the standpoint of the use of this material in a TA, no

decrease in tensile proportional limit or tensile yield strength is anticipated although a lower ultimate strength appears evident. This is indicative of a tendency for the material to fail in a brittle fashion rather than to relieve high local stresses, as at the base at a notch, by plastic deformation. The 1200°F tensile properties of HCP Stellite 6B do not degrade due to exposure (up to 700 hours) at 1200°F prior to testing. The static modulus of elasticity of material in the FCC or HCP condition is not significantly different. With care in design, handling, and operation, Stellite 6B in the HCP condition appears to be an acceptable alloy for use in mercury-driven turbine applications.

#### B. VOLUMETRIC CONTRACTION

Crystallographic transformation of Stellite 6B is accompanied by a volumetric contraction of the material. Therefore, machined parts of Stellite 6B in the FCC condition undergo a reduction in linear dimensions when transformed to the HCP condition during turbine operation. The consequence of this may be a marked increase in the stack-up clearance of components produced during TA assembly or a decrease of this clearance which may result in overstressing of parts. A study was completed (Reference 13) to determine quantitative contraction data.

Transformation of various laboratory samples prepared from forgings, bar stock, and actual TA parts, was produced by thermally treating the specimens for 4 hr at 1650°F followed by 48 hr at 1250°F. The calculated average contraction and standard deviation were 0.092 and 0.042%, respectively. Based on these data and on the assumption that a normal distribution exists, there is a 99% probability that 99% of the measured contractions due to the crystallographic transformation of Stellite 6B from FCC to HCP structure will fall between 0% and 0.223%.

#### C. CRYSTALLOGRAPHIC TRANSFORMATION STUDIES

Stellite 6B parts in the TA (S/N 3-2) were subjected to a heat treatment, during fabrication, consisting of a solution anneal at 2250°F before machining and either a stress relief at 1650°F (rotors diaphragms and nozzles) or at 1100°F (rotor stacking bolt) afterward. Stellite 6B specimens taken from the same lot from which parts were made, and treated in the same manner, showed the structure after either heat treatment to be FCC with an approximate lattice parameter of 3.57 angstroms (A). X-ray diffraction (XRD) analysis (employing a diffractometer) of components removed from the TA indicated that transformation to an HCP structure had occurred with approximate lattice parameters of  $a = 2.55 \text{ A}$  and  $c = 3.95 \text{ A}$ . The degree of transformation varied, as indicated in the tabulation below.

<u>TA Component</u>	<u>4-Hr Post-Fab* Stress Relief Temp (°F)</u>	<u>Est Oper Temp (°F)</u>	<u>Post Operation Hardness R<sub>C</sub>**</u>	<u>Relative Transformation to HCP (by XRD)</u>
1st-Stage Nozzles	1650	1238	48-51	Complete
1st-Stage Rotor	1650	1150	48-51	Complete
1st-Stage Shroud	1650	1052	48-51	Complete
2nd-Stage Diaphragm	1650	1030	48-51	Complete
2nd-Stage Rotor	1650	937	48-51	Complete
3rd-Stage Rotor	1650	801	41-42	Almost Complete
3rd-Stage Diaphragm	1650	827	ND	Almost Complete
4th-Stage Rotor	1650	704	41-42	Almost Complete
4th-Stage Diaphragm	1650	762	ND	Almost Complete
Rotor Stacking Bolt Head	1100	1160	42	Partial
Rotor Stacking Bolt Threads	1150	457	40	Minor

ND = Not Determined

It appeared that the crystallographic changes were due either to in-fabrication heat treat procedure or to the exposure temperature of the component. Studies were conducted to characterize the Stellite 6B transformation reaction as a function of these two parameters.

#### 1. Transformation as Affected by Stress Relief

To confirm the apparent differing effect of stress relief temperature, several specimens were solution annealed at 2250°F, and then either stress relieved at 1650°F or given no stress relief. They were subsequently exposed at 1200 or 1400°F to promote transformation. Those specimens which had been given no stress relief prior to exposure were, in effect, being stress relieved during the exposure period. Results of XRD examination are shown below.

\* All parts were hot forged from bar stock, then solution annealed at 2250°F and rapid air cooled before machining.

\*\* Initial hardness after stress relief was R<sub>C</sub>40.

<u>Specimen Source</u>	<u>Stress Relief* Temp (°F)</u>	<u>Exposure Temp (°F)</u>	<u>Exposure Time (hr)</u>	<u>Transformation FCC to HCP</u>
Forging**	1650	1400	24	Complete
Forging**	1650	1200	90.5	Complete
***	None	1200	278	Minor to Partial

It was concluded based on detected transformations in all samples that any Stellite 6B components in the FCC condition would transform to the HCP condition during TA operation. The rate and ultimate degree of transformation remained an unknown.

## 2. Preassembly Crystallographic Transformation

The potentially detrimental volumetric contraction resulting from transformation during TA operation could be avoided if complete transformation was produced by heat treatment prior to assembly. Therefore, based on the stress relief tests above, and additional developed data, the following two-cycle heat treatment was established for complete transformation of solution-annealed (2250°F) material from the FCC to HCP structure during Stellite 6B component fabrication:

- a. 1650°F soak for 4 hours
- b. 1250°F soak for 48 hours.

Subsequently, 36 Stellite 6B turbine assembly production parts including rotating wheels, diaphragms, shrouds, and stacking bolts were heat treated in this manner to produce crystallographic transformation. X-ray diffraction analysis and dimensional measurements indicated that complete transformation and anticipated dimensional contraction had occurred in all parts.

An anomaly occurred in that the hardness of three rotating wheels, of the 36 parts so treated, was below the anticipated hardness,  $R_C$  48-51. Two wheels contained areas measuring  $R_C$  46 and the third exhibited a uniform hardness of  $R_C$  44. The cause of this difference remains obscure. The parts were considered to have been satisfactorily transformed for the purpose of avoiding transformation contraction during turbine operation.

\* All material had been solution annealed at 2250°F and rapid air cooled.

\*\* Material from same lot used to fabricate turbine assembly S/N 3-2 parts.

\*\*\* Three specimens representing three different material sources:

- (1) First stage shroud from turbine assembly S/N 3-2.
- (2) Forging from same lot used to fabricate above.
- (3) Bar stock from different material lot.



### 3. Nonresponse to Transformation Heat Treatment

As more experience with preassembly transformation was accumulated additional mill heats of Stellite 6B were processed which exhibited sufficiently sluggish transformation behavior that the developed treatment of 1650°F -4 hr + 1250°F -48 hr would not produce the desired HCP structure. Subsequent exposure at 1400°F for 112 hr produced primarily HCP structures, but a measurable difference between parts in the amount of retained FCC and in the relative hardness still existed. This difference not only occurred from heat to heat but also from part to part within the same piece of metal. After an additional 546 hr of exposure at 1200°F, the hardness of all parts was  $R_C$  48/49. Trace amounts of FCC structure were still detected at this time, the measured amount differing between parts.

These results indicate that unexplained reaction sluggishness in some Stellite 6B may exist, but ultimate transformation occurs to an essentially all-HCP structure and higher hardness under sufficient conditions of time or temperature, or both. Therefore during TA operation given sufficient time, Stellite 6B components should transform to HCP. Since attainment of metallurgical equilibrium is a time-temperature function it would be anticipated that the first stage, in a turbine containing "sluggish" Stellite 6B components operating at the highest temperature would most likely transform sooner than the other Stellite 6B components. The last stage, which operates at the lowest temperature, would be the least prone to transform and may take as long as 10,000 hours or more. This study reinforces the desirability of preassembly transformation of all Stellite 6B turbine components. This procedure will avoid during TA operation, the thermal contraction reaction which accompanies transformation.

### 4. Stabilization of the FCC Structure

Analysis of the XRD data accumulated during all the transformation studies indicated that the FCC-to-HCP transformation temperature for solution-annealed Stellite 6B lies between 1650°F and 1400°F. It is hypothesized that transformation occurs because the matrix is depleted of carbon (a strong FCC stabilizer) by precipitation of carbides during the stress relief treatment. The reaction is triggered by subsequent exposure to the TA operating temperature. Theoretically, carbon can be retained in the matrix if Stellite 6B is not heated to, or held within, the rapid-carbide-precipitation temperature range (1400°F to 2100°F). Carbon depletion can also be avoided if the cooling rate through this range is sufficiently rapid. That this carbon retention may be possible is evidenced by the relatively slow transformation of specimens and the TA rotor stacking bolt head which were exposed to 1160°F with no prior 1650°F stress relief. However, elimination of a 1650°F stress relief probably is not sufficient as evidenced by some transformation even in this material. Therefore cooling-rate control after the 2250°F solution annealing treatment appears to offer more promise than stress relief temperature changes.

A measurable difference in the amount of transformation was noted (on the XRD trace) in the three specimens, described in Section III,C,1 above, which had been exposed to 1200°F with no post-solution anneal stress relief. This difference was qualitatively correlated with the different cooling rates experienced after the 2250°F soak. The variation in rate apparently resulted from the difference in specimen size. The slower cooling rate occurred in the more massive cross-section and correspondingly the most massive specimen was found to contain the greatest amount of HCP. Exposure of these three specimens was therefore continued at 1200°F in an effort to determine more fully the time-transformation relationship as affected by cooling rate after solution annealing. To further evaluate the theory of the effect of cooling rate on transformation, two additional specimens, sections of the fourth-stage diaphragm of TA S/N 3-2, were added to the study. One was water-quenched from the solution-annealing temperature (2250°F) and the other was salt-bath-quenched to 1000°F, then still-air cooled. These specimens represent cooling rates significantly higher than either of the three previous specimens that were rapid-air cooled. Semi-quantitative XRD data and hardness measurements during exposure times up to more than 1200 hr at temperatures between 800 and 1200°F appear in Figures 6 and 7. After approximately 400 hr of exposure, both fourth stage diaphragm sections exhibited an increase in hardness to  $R_C$  40 from the annealed hardness of  $R_C$  38. Both sections also exhibited slightly increasing amounts of the HCP with increasing exposure time. After 1083 hr at 1065°F and 1025 hr at 800°F, neither the hardness nor the crystal structure of either specimen was altered from that which existed after 400 hr of exposure. These results indicate that stabilization of the more ductile FCC structure may be possible at least at the lower turbine operating temperatures. Exposure was continued of only the 1065°F specimen.

#### IV. ALTERNATE TURBINE MATERIALS EVALUATION

A study was conducted to evaluate the metallurgical stability of various alloys considered as candidates for replacing Stellite 6B as the SNAP-8 reference turbine material should the need of a substitution arise. The materials evaluated are described below.

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Alloy	Nominal Chemical Composition (%)							Pre-Exposure Heat Treatment
	C	Cr	Ni	Fe	Co	Mo	Others	
S-816	0.38	20.0	20.0	4.0	Bal	4.0	Cb and W 4.0 each	2200°F for 1 hr; water quench 1400°F for 24 hr; air cool
PH 15-7	0.09	15.0	7.0	-	-	2.5	Al 1.0	1950°F for 1 hr; air cool; 1400°F for 1/2 hr; rapid cool (within 1 hr) to 55°F; hold 1/2 hr; 1050°F for 1-1/2 hr; air cool
Lapelloy	0.30	12.0	0.30	Bal	-	2.75	V 0.25	2000°F for 1 hr; oil quench; 1200°F for 2 hr; air cool
410	0.15	12.0	-	Bal	-	-	-	1550°F for 1 hr; furnace cool to 1100°F; air cool

Specimens were exposed in an air atmosphere oven at 1065 and 1200°F to determine the long-term effects on metallurgical stability. Periodic room-temperature hardness measurements are reported in Figures 8 and 9. Quantitative crystal structure determinations by XRD were also made.

The precipitation-hardening steel (PH 15-7) contains a predominantly martensitic structure in the heat-treated condition tested (TH 1050). The composition of the alloy is such that the microstructure exhibits a mixture of two crystallographic phases, gamma and alpha structures (FCC and BCC, respectively), in the equilibrium state. Therefore, under sufficient conditions of time or temperature, or both, the primarily martensitic structure should change. The XRD evaluation detected such a change at both exposure temperatures (1065 and 1200°F) during the first 730 hours. From that time to the end of the exposure period, no further change was detected by XRD.

That metallurgical equilibrium was essentially reached is also confirmed by a hardness decrease measured after 730 hr at 1065°F. The room temperature hardness changed from  $R_C$  35 after 730 hr to  $R_C$  33 after 2846 hr. At 1200°F the room temperature hardness of  $R_C$  23 after 730 hr remained unchanged to the end of the exposure period (1884 hr). The difference in hardness produced by the two exposure temperatures is an anticipated reaction. The material, having been tempered during the heat treat process at 1050°F, is further tempered and softened to a degree which is proportional to the increase in the subsequent exposure temperature. Since the time required to complete this change is also a function

of the exposure temperature, it would be anticipated that 1200°F exposure would result in faster attainment of equilibrium (cessation of hardness change) than 1065°F.

The S-816 specimens appeared to be the most stable of the several alloys evaluated. No metallurgical change from the original FCC microstructure occurred, as indicated by periodic XRD examination. Room-temperature hardness measurements through 2426 hr exposure at 1065°F and 2696 hr at 1200°F indicated no deviation from the unexposed hardness of Rc 30.

Lapelloy is a heat-treatable alloy which is hardened by quenching from the austenitizing temperature to produce a martensitic structure. This structure is then tempered at 1200°F (made more ductile and impact-resistant although of lower strength). Subsequent exposure at 1200°F would result in further tempering of the martensite. That this occurred is evidenced by a significant change in hardness (Rc 34 to Rc 23) during exposure. No such change occurred at the 1065°F exposure temperature. This exposure temperature is sufficiently below the 1200°F tempering temperature used as part of the initial heat treatment so that metallurgically stable material would be anticipated.

The 410 SS was exposed only at 1200°F. Like Lapelloy, it would not be expected to transform from the BCC structure because of its composition. Also like Lapelloy softening would be expected to result from exposure at this temperature. This reaction is evidenced by the reduction of hardness from the pre-exposed, annealed condition (R<sub>B</sub> 95) to R<sub>B</sub> 84 at the end of the exposure period.

The exposure of these materials allowed a preliminary evaluation of their metallurgical stability. At 1065°F, those alloys evaluated appear acceptably stable; at 1200°F, all except S-816 appear to suffer a reaction which would reduce their mechanical strength. To fully evaluate the applicability of any of these materials for the higher-operating-temperature turbine components, more complete evaluation of the exposure effects would be required.

## V. EVALUATION OF -1 BOILER (P/N 092020-1F, S/N A-1)

### A. INTRODUCTION

The interface component between the NaK primary loop and the Hg loop in the SNAP-8 system is the boiler. This component transfers heat from the NaK to the Hg, producing Hg vapor to drive the turbine-alternator. The -1 boiler (Figure 10) is a combination cross-counter flow, tube-in-shell heat exchanger. The mercury flows in four 60-ft-long tubes which are coiled on two double-lead helices. A plug to restrict flow is placed in the inlet to each of the four parallel flow passages, giving a liquid velocity of 0.8 ft/sec. The plug in this restricted flow section is a solid rod spaced

from the inside of the tube by a wire spring forming a spiral flow path for the mercury. This insert continues through the boiler for 10 ft. Downstream of the plug, the spiral flow is maintained by a twisted ribbon insert which continues for the remainder of the boiler length. The swirl flow serves to separate the high-density liquid from the vapor, making the boiler operation insensitive to gravity and increasing heat transfer rates. The mercury coils are surrounded by two concentric cylindrical shells which form an annular flow passage for the reactor coolant, NaK-78 (the eutectic mixture of sodium and potassium). The Hg tubes are 0.902-in. ID by 0.125-in. wall 9Cr-1Mo steel, and the shells are 316 SS. The plug consists of a 0.600-in. OD low-carbon steel rod and 0.135-in. dia low-carbon steel wire. The ribbon is also of low-carbon steel, 0.016 in. thick.

Loop testing of the boiler (S/N A-1) was initiated in July 1964 using the RPL-2 facility. After successful completion of a performance test program in July 1965, the boiler was removed from the loop and a metallurgical evaluation was initiated. During the report period, collection of metallurgical data was completed. Analysis of the data was started. The NaK- and Hg-exposed surfaces were examined for corrosion effects, and analysis was performed to determine the cause of surface cracking in one of the Hg-containment tubes.

## B. OPERATING HISTORY

### 1. NaK Side

The NaK side of the boiler operated for 2350 hr. The nominal rated design conditions are listed in Table 2. At the time of installation of the -1 boiler in the RPL-2, the NaK primary loop was operating without a NaK purification system because of design deficiencies and operating difficulties. Purification to some unknown level was achieved toward the beginning of the boiler test history, primarily through multiple hot dumps at the start of each operating period. During this period a massive leak in the NaK primary loop was produced by an electric NaK heater failure. Although the loop was made operative, the resultant oxygen contamination was so great that during the subsequent operation period significant mass transfer was observed in many of the components. Toward the middle of the test period the cold trap of the purification system was made operative. Although plugging meter runs were not made, it is presumed, based on periodic examination of components (the absence of NaK mass transfer deposits), that an oxygen content of 25-40 ppm was maintained during the last 1140 hr of a total NaK side operating time of 2350 hr.

### 2. Hg Side

The rated design parameters of the Hg side of the boiler, and typical performance characteristics during the test period, are shown in Table 2. The boiler started the test series with a less than satisfactory heat transfer capability (unconditioned state), and saturated vapor was not produced (Reference 14). From November 1964 through March 1965, the boiler

was operated intermittently for a total of approximately 300 hr with a Rb additive in the Hg to promote full conditioning and attainment of rated boiler outlet conditions. Testing continued thereafter until a total 1415 hr of operating time was logged. During the last half of the total test period (approximately 700 hr), boiler characterization tests were conducted under varying operating conditions.

C. NaK SIDE EVALUATION

1. Visual Mass Transfer Deposits

Dendritic-type NaK mass-transfer deposits found during boiler dissection (Figure 11) consisted primarily of iron with a significant amount of nickel and a lesser amount of molybdenum. The heaviest mass transfer deposit occurred in a NaK drain fitting which protruded approximately 3 in. from the NaK outlet end (1100°F) of the boiler shell. This stub apparently acted as a cold finger so that the contained static NaK was below the NaK mainstream temperature (1100°F). The stagnant fluid and thermal gradient associated with the cold finger probably accounted for the heavy deposit concentration in this area.

A very small deposit was located at the NaK inlet end (1300°F) in the vicinity of the Hg tubes (Figure 11). This deposit is presumed to have occurred because of one, or a combination, of two conditions. First, an unanticipated NaK thermal gradient may have existed between the main NaK stream and the localized mass adjacent to the Hg tubes (where they penetrated the NaK flow baffle); these tubes carry approximately 1265°F Hg vapor into the Hg outlet manifold. Second, the Hg tubes may have produced a flow restriction resulting in a relatively low NaK flow area. The deposits were similar in appearance to those found in SNAP-8 simulated NaK test loops operated by Oak Ridge National Laboratories (ORNL) with intentionally induced high (approximately 80 ppm) oxygen contents (Reference 15). The deposit composition differed, however, in that no chromium was found in the RPL-2 boiler deposit while that element was found in the ORNL loop. The reason for this is obscure at present. Based on ORNL work it is presumed these deposits were caused by RPL-2 operation with an uncontrolled high oxygen content caused by lack of NaK purification and gross oxygen contamination of the loop resulting from the failure of the electric NaK heater in the primary loop during the early part of the boiler test history.

2. Microscopic Evaluation of One Hg Containment Tube Coil

Complete metallographic evaluation of the NaK exposed surface of one full 9Cr-1Mo steel Hg containment tube coil (identified as No. 0-4 in Figure 11) was performed. Concomitant grain growth and surface decarburization, intergranular corrosion, mass-transfer deposits, and surface cracks were found (Figure 12).

a. Grain Growth and Decarburization

Grain growth and concomitant decarburization were found at the NaK exposed surface over the full length of the tube coil. The maximum depth of the affected zone (0.030 in.) was measured at the NaK inlet end of the Hg containment tube. The depth of the affected zone generally decreased along the length of the tube from the NaK inlet end toward the outlet end. However, for a presently unexplained reason an irregularity in this pattern of decreasing depth appeared in the 30 ft and 42-1/2 ft (from the NaK inlet end) specimens of Figure 12. Although these also show a grain growth zone, the grain size and the depth of the affected area is less than that present in adjacent areas downstream (with respect to NaK flow direction). The results of ORNL confirm our findings of 9Cr-1Mo tube surface decarburization and grain growth with the maximum effect occurring at the maximum NaK temperature to which the 9Cr-1Mo steel is exposed. It was also found by ORNL that these effects are independent of the oxygen content of the NaK. Thus, significant time of RPL-2 operation under oxygen-contaminated conditions cannot be presumed to have affected the depth of this zone found in the -1 boiler tubing after the total 2350 hr of NaK loop operation.

Aerojet-General Nucleonics (AGN) also conducted a corrosion loop test simulating the SNAP-8 NaK primary loop, Reference 4. The pattern of decarburization and grain growth was not the same as that found in the -1 boiler. The single tube-in-tube boiler operated at AGN did not experience decarburization and grain growth in the last half of the NaK flow passage (i.e., from the midpoint of the 60 ft boiler tube length to the NaK outlet). The thermodynamics of the system as well as the design was different, and a more complete comparative analysis is required to account for the different results.

b. Mass-Transfer Corrosion Effects

Metal mass-transfer deposits similar to those found in the boiler were also found in loops operated at AGN and ORNL. The ORNL tests indicated that mass transfer is markedly increased by high oxygen content of the NaK. It is therefore presumed that the mass transfer in the present boiler was a result of the generally high oxygen content of the RPL-2 system throughout the 2350-hr exposure of the NaK side of the boiler. The mass transfer (see area C in various micrographs in Figure 12) is presumed to be a mixture of metallic particles and oxide deposits based on analytical work completed by ORNL.

Intergranular attack in the tube surface was found by ORNL at the NaK high-temperature end of the coil; such attack also was noted in the SNAP-8 -1 boiler (see 0 and 13-1/2 ft photos in Figure 12). It is theorized by ORNL that this attack is due to initial preferential intergranular chromium depletion of the material by the oxygen-rich NaK.

c. External Surface Tube Cracks

Cracks were found at the NaK inlet end in a section of the No. C-4 Hg tube which was bent to a 5-1/2 in. radius for transition from the coil into the Hg outlet manifold (Figure 11). The cracks are presumed to have been

caused by excessive creep resulting primarily from a markedly reduced tube wall thickness. However, a contributing cause was also decarburization and grain growth in this area. The thin wall was apparently due to an eccentricity between the ID and OD in the original tube. The wall thickness was further reduced when the 5-1/2 in. radius bend was produced. A more detailed analysis appears below in the section on "surface cracking."

#### D. Hg SIDE EVALUATION

One of the four tubes contained mass transfer deposit directly at the Hg outlet manifold (Figure 13). There apparently was a sufficient difference in the performance of this one tube compared to the others which produced untypical Hg vapor conditions during the test period, not representative of the previously described boiler outlet conditions. Such a deposit would be anticipated at the manifold only if the tube operated with no superheat length for a major portion of the test period. It is not clear at present why one of four tubes should exhibit mass-transfer deposits at the Hg outlet.

The No. 0-4 tube coil, one of the 2 coils in the outer of the two-layer tube bundle, see Figure 10, was completely dissected, using a band saw. The coil was cut transversely into sections comprising 180° of a single turn. Each section was then cut longitudinally. The surfaces of the tube, twisted tape, and the Hg inlet plug and associated wire were examined for surface effects. Significant findings are summarized below and in Figures 13 and 14.

##### 1. Macrographic Examination of Tube Coil No. 0-4

Surface effects found at the Hg inlet region (including the tube, plug, and wire) up to the 23-ft point are presumed to be liquid Hg corrosion effects. There appeared to be no orientation of the attack with respect to gravity. The surface deposits resulted from the precipitation of soluble corrosion products from the liquid Hg as it vaporized.

##### 2. Microscopic Examination of Tube Coil No. 0-4

###### a. Pitting

Pitting was found in the first 23 ft of the No. 0-4 tube (measured from the Hg inlet). Figure 14 shows a micrograph of a section of the tube 11 ft from the inlet. The pits were approximately circular, with a diameter-to-depth ratio between 1 and 10 and a maximum depth of 5.5 mils. Figure 15 describes the maximum pit depth distribution along the tube length. There was no apparent orientation of pitting or maximum pit depth with respect to either gravity or centrifugal forces on the flowing Hg. An analysis of the data with respect to temperature, or vapor quality, or both, will be performed in an effort to correlate pitting with localized Hg conditions.

###### b. Mass Transfer

Microscopic mass-transfer surface deposits were found in the length 13 to 57 ft from the Hg inlet end. The maximum depth at the 47-ft point, was



4 mils (see Figure 14). It is postulated, confirmed by the operating history, Table 2, that the boiler operated for significant time periods with a very short superheat length. This would explain the presence of deposits only 3 to 4 ft from the Hg outlet in this tube coil.

c. Decarburization

Toward the Hg outlet end, the surface contained a decarburized layer approximately 1 mil thick, shown in the 55-ft located micrograph of Figure 14. Pre-existing decarburization resulting from tube manufacture does not appear probable since all tubes were part of one fabrication lot and other tube sections in this one coil do not exhibit the same phenomena. In order to determine whether Hg corrosion effects could have produced surface decarburization, this same area in two other tube coils removed from the boiler is being examined.

d. Surface Cracking

Tube cracks on the Hg-exposed surface were found in three areas (see Figure 13): at, and immediately beyond, the end of the Hg inlet flow restrictor plug, 55 to 58 ft from the Hg inlet, and at the 5-1/2 in. radius bend at the Hg outlet manifold. The latter area contained cracks on both the inside and outside surfaces and appeared associated with tube wall thinning.

The cracks at the end of the flow restrictor plug, in the approximate length between 7 and 15 ft from the Hg inlet. All cracks lie perpendicular to the longitudinal axis of the tube. They appear in a series of discrete groups separated by approximately 3/4 to 1-1/2 in. of sound metal (see 10-1/2 ft photo in Figure 13) and they lie in the portion of the tube wall which is subjected to a tensile stress during the coiling operation (on the outside tube section with respect to the coil diameter). The cause of these cracks is somewhat obscure at present, primarily because of the distribution and uniform pattern. It could be presumed that they were caused by thermal stresses produced by the high heat flux which probably was present in this area. Another possibility exists that these cracks were a consequence of induced coiling stresses in combination with operating conditions. Future analysis of the findings with respect to boiler operating conditions will confirm location of the high heat flux area. This in combination with evaluation of additional tube coils may aid in determining the probable causes of this cracking.

The cracks toward the Hg outlet end of the tube, approximately 55 to 58 ft from inlet, appear very straight and form a criss-cross pattern, Figure 13, suggesting that they were cut into the surface by a tool rather than produced by any phenomena related to boiler performance. It is postulated that these cracks were produced during tube manufacture and were undetected in final inspection. Support of this theory of pre-existence stems from the appearance of the micrograph of one of these

cracks (the 55-ft section shown in Figure 14). A surface decarburization layer follows the contour of the crack and appears to be of uniform thickness throughout. Since the decarburized layer thickness in the crack is not different from that on the tube surface, the decarburizing phenomenon must have been reacting on the crack as long as the surface proper and this would require existence of the crack prior to startup. If the crack had appeared during boiler operation it would be expected that the decarburized layer in the crack would be thinner than on the actual tube surface; a time delay would be required for the Hg and/or internal pressure to cause propagation of the crack from any pre-existing microscopic surface discontinuity. This time delay would have resulted in a difference in decarburization depth.

e. Distortion of Ribbon Turbulator

The 0.016 in. thick AISI 1010 steel twisted ribbon insert, downstream of the inlet plug, was not a single length. The 60 ft long Hg containment tube was fabricated using short lengths because 60 ft tubes were not available. Prior to butt welding of the adjacent tube lengths the twisted ribbon was inserted in each. The tubes were joined but the ribbon was only abutted against the adjacent ribbon.

X-rays taken after tube coiling, during fabrication, (Figure 16a) show that the adjacent ribbon ends were properly abutted and the twist was uniform and consistent. After boiler operation, the ends of adjacent sections had separated up to 11 in. and the ribbon had collapsed (Figure 16b) from its original twist form. The distortion is presumed to have been caused by elevated-temperature relieving of locked-in stresses induced in the thin ribbon during twisting and subsequent tube coiling. The relief occurred during exposure to the boiler operating temperature. The collapse of the ribbon must be presumed to have caused local Hg flow discontinuities which, based on boiler test data, did not detrimentally affect boiler operation. A material having higher strength (at 1100-1300°F) than carbon steel, and/or possibly increased thickness, should prevent collapse based on previous operation and evaluation of Haynes 25 boilers (containing Haynes 25 turbulator ribbons).

f. Rubidium Analysis

The RPL-2 system operated for approximately 500 hr after Rb was no longer used as an addition in the Hg to promote boiler conditioning. However, there remained a Rb residue, probably as an oxide, in the two tube coils evaluated. Using distilled water as a solvent flush and performing subsequent titration using phenolphthalein solution, one tube coil was found to contain 0.34 g, and the other 0.11 g, of Rb. The presence of Rb indicates that once used, a formed surface residue is not removed by the Hg vapor flow or by changing the contaminated Hg inventory; more thorough cleaning such as by a loop water flush may be required if complete Rb removal is desired.

It may be that satisfactory boiler performance toward the end of the operating period, when Rb was no longer present as a Hg additive, was caused by an adherent Rb residue on the boiler tube wall. Such a residue could continue to promote efficient heat transfer. There is insufficient test data at present to allow a substantiated analysis of the validity of this theory.

E. CRACKED 5-1/2 INCH RADIUS TUBE BEND AT Hg OUTLET MANIFOLD

Two of the four boiler tube coils (identified as I-1 and O-4 in Figure 11) contained surface cracks at the 5-1/2 in. radius tube bend which is the transition from the tube coil shape to the Hg outlet manifold. Separate consideration is given to this cracked area because the indications appear on both the inside and outside tube wall surface (see Figures 11 and 13). Analysis indicated that they probably were caused by the same phenomena.

1. Metallurgical Evaluation

The maximum depth of the internal surface defects in Coil O-4 was 0.012 in. (Figure 17). Some cracks were nearly as wide as they were deep. External intergranular cracks extended to a depth of 0.045 in. (Figure 17). A layer of marked grain growth extended to a depth of approximately 0.030 in. This is consistent with the structure found further down the tube length toward the Hg inlet.

2. Dimensional Evaluation

Dimensional measurements of the dissected sections showed an ID to OD eccentricity in Coils I-1 and O-4 which resulted in an area of significant wall thinning below that normally expected of the as-received tube. Therefore, a detailed dimensional analysis of tube dimensions in this area was made on all four coils (Table 3). This evaluation included comparison between the bend area and a straight section of the tube coil immediately adjacent to the bend on the downstream side. This straight section, which is about 4 in. long, is the terminus of the coil at the Hg outlet manifold; it should have experienced the same boiler operating conditions of temperature, pressure, flow, etc., as the bend area. The as-received tube dimensions in these two adjacent areas should have been extremely close, though perhaps not exact, because the normal anticipated variation between two points, within approximately 6 in. along the length of an as-fabricated tube, is very small.

It appears that all tubes contained some ID to OD eccentricity when purchased, as evidenced by the differences in wall thicknesses found in the straight tube section. The minimum wall dimension in the bend of all tubes was found at or near the tension side relative to the bend radius (top of Plane 1 in Section A-A of Table 3). One would expect that some decrease in the as-received minimum wall thickness would occur during the bending operation, and correspondingly that some increase would occur in the maximum wall thickness

which was located at or near the compression side of the tube relative to the bend radius. This generally occurred, but there was an anomalous condition in the minimum wall thickness in Tubes I-2 and O-3 in that the straight section was thinner than the corresponding area in the bend. This anomaly is unexplained at present. The wall thinning is markedly apparent in the case of Coil O-4, which contained the most severe cracked condition. It is not known why the wall of Tube O-4 shows slightly more reduction than that of Tube I-1. It may be the result of a difference in the results of bending the two tubes to the 5-1/2 in. radius or due to the operating conditions which may have caused more creep in the O-4 tube wall.

Comparison of the outside diameters of three of the tubes indicates that prior to boiler fabrication they were reasonably round and very close to the nominal dimension. This conclusion is drawn specifically from the measurements taken on the Hg outlet straight sections as well as subsequent measurements also made on sections at the boiler Hg inlet where the exposure temperature (1100°F) was considerably lower than the outlet temperature. For example, the tube outside diameter at the Hg inlet end of Coil I-1 was 1.144/1.151 and that of Coil O-3 was 1.144/1.152. The respective Hg outlet end straight section outside diameters (Table 3) are 1.148/1.149 and 1.145/1.154.

### 3. Tube Wall Stress

Previous analyses have shown that the limiting stress in the boiler occurs at the mercury exit/NaK inlet end of the boiler tube where there is no thermal stress but where the temperature is maximum. Considering the tube in this region to be a cylinder of inside radius  $a$ , outside radius  $b$ , acted upon by an internal pressure  $P_i$  and external pressure  $P_o$ , the expressions for the principal tangential and axis stresses at any radial position,  $r$ , are given by:

$$\sigma_t = \text{Tangential Stress} = \frac{a^2 P_i - b^2 P_o}{b^2 - a^2} + \frac{(P_i - P_o) a^2 b^2}{r^2 (b^2 - a^2)} \quad (1)$$

$$\sigma_a = \text{Axial Stress} = 0 \text{ for an unrestrained cylinder.}$$

For the boiler Hg tube Coil No. O-4 in the region of reduced wall, the following conditions apply:

$$\begin{aligned} a &= 0.488 \text{ in.} & P_i &= 275 \text{ psia} \\ b &= 0.582 \text{ in.} & P_o &= 30 \text{ psia} \end{aligned}$$

Solving for the stresses at the inner radius where they are maximum:

$$\sigma_t = 1371 \text{ psi}$$

$$\sigma_a = 0$$

For plastic flow (creep) the limiting stress is the constant flow stress,  $\sigma_o$ , defined as:

$$\frac{2 \sigma_o}{\sqrt{3}} = \sigma_t - \sigma_a \quad (2)$$

or

$$\sigma_o = 1190 \text{ psi}$$

This latter tube wall stress in Coil O-4 is approximately 28% higher than the comparable stress in tube Coil O-3 (931 psi) which does not contain the severely reduced wall thickness.

#### 4. Diametral Creep

Diametral measurements indicated creep had occurred in the tubes in the area of the cracks. The effective creep rate, assuming uniform secondary creep, of the two tube coils containing cracks was more than 1 order of magnitude higher than anticipated (12.1 and 12.9 x 10<sup>-4</sup> %/hr as opposed to the design rate of 1 x 10<sup>-4</sup> %/hr). The uncracked tube was only slightly higher than anticipated: 1.62. It is therefore presumed that the cracks are indicative of incipient stress rupture failure. The intergranular orientation of the cracks and the presence of the significant decaurized and enlarged grain surface area, which has been shown to result in decreased creep and stress rupture strength, support this conclusion.

In addition to the fact that the cracks in the tube wall are intergranular, rather than transgranular, there is no evidence of localized tube wall reduction. These are typical criteria for stress-rupture tube wall failure.

It is presumed that an imposed stress which would result in this amount of creep leads to an estimated total tube life, before rupture, of approximately 2500 hr (or about 1100 hr in addition to the number accumulated).

5. Discussion

Although the No. 0-4 (cracked) tube coil with its thin wall was subjected to a significantly higher wall stress than the No. 0-3 (uncracked) coil, the increase does not appear sufficient to justify the significantly higher creep rate. The stress for 1% creep in 10,000 hr for 9Cr-1Mo steel is 1100 psi. Increasing this stress to 1190 psi increases the total creep in 10,000 hr to only 1.6%.

Previous work (References 4 and 15) indicates that grain growth and concomitant decarburization will materially decrease the creep and stress rupture strength of 9Cr-1Mo steel.

One, or a combination of these factors could have contributed to an overstressed condition of the two sections in tubes No. 0-4 and I-1. These factors would tend to reduce the load-carrying capacity of the tube and could have resulted in the observed cracking and diametral growth.

F. DISCUSSION

The corrosion pattern in the -1 boiler was similar to that found in the boiler operated in CL-3 at AGN for 4400 hr, but differed markedly in degree. Pits in the -1 boiler appeared to be much shallower and also were more numerous. The deposit layer was more uniform and was thinner. The difference in most respects was probably due to the different operating times (4400 hr vs 1415 hr for the CL-3 and -1 boilers, respectively); however, the deep concentrated pits in the CL-3 boiler (illustrated in Figure 21 on Reference 4) were not found in the -1 boiler. Unless it is presumed that attack was accelerated with time, there is an inconsistency in that the ratio of operating times (CL-3/RPL-2) is 3:1 but the ratio of maximum pit depth is 7:1. Further analysis of the operating conditions of the RPL-2 boiler may provide additional data for comparison, however, at this time, it is postulated that this apparent accentuated pitting of the CL-3 boiler is due in part to the flow conditions introduced by the 0.062-in.-dia wire turbulator of the CL-3 boiler as opposed to those introduced by the ribbon turbulator of the RPL-2 -1 boiler. This is explained using Figure 17a. The Hg vapor flow through the CL-3 boiler tube containing the helical wire turbulator is essentially in the tube's axial direction with the wire acting only on the liquid Hg drops forced against the tube wall. Thus the push of the vapor on any Hg drop upstream of the wire applies a component force parallel to the wire, resulting in movement of the drop, and also in a force component perpendicular to the wire which produces no movement of the drop along the longitudinal direction of the tube. The ribbon in the RPL-2 boiler, because it covers the full tube ID, produces a helical flow to the entire stream. All force on a Hg drop acts to push it through the tube with no force component against a resisting surface (frictional forces excluded). Therefore, there should be less tendency for Hg drops to dwell in any area in the latter boiler. It is postulated that the dwell time of the Hg drop at any point has a marked effect on the depth of pitting corrosion produced. If this is indeed the case, it would appear that a boiler with a wire turbulator should exhibit the greater effect. Subsequent full-scale SNAP-8 boilers (tube-in-tube design) were fabricated with wire turbulators. Post-test examination should provide more data for comparative analysis.

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The cracks found in the area of the plug exit cannot be explained at this time. The cracks near the Hg outlet, and at the 5-1/2 in. radius tube bend at the Hg outlet manifold, appear to stem from a cause other than boiler operation. Greater quality control applied to tube manufacture, and acceptance and boiler design changes could reduce the amount of damage. Again, subsequent tube-in-tube SNAP-8 boilers were designed and fabricated with this in mind. Ultrasonic inspection of tubing and elimination of any small radius bends are two such items. To ascertain whether a cracking problem exists, perhaps due to some deficiency in the 9Cr-1Mo tube material or its incapacity for withstanding anticipated boiler operating conditions, several actions were called for:

- o Additional tube coils from this tube-in-shell boiler will be evaluated for cracking
- o Tubing of subsequent SNAP-8 tube-in-tube boilers were fabricated and inspected using stringent nondestructive inspection procedures
- o The tubing for these boilers was centerless-ground on the outside surface, and vapor-honed on the inside surface to remove any microscopic discontinuities which might otherwise propagate into cracks because of high heat flux or temperature during boiler operation.

Evaluation of these boilers after a significant accumulated operating period will provide additional information with which to estimate the inherent suitability of 9Cr-1Mo steel as the Hg containment tube alloy for the SNAP-8 system.

Decarburization and concomitant grain growth produced by exposure of 9Cr-1Mo steel in the SNAP-8 primary NaK loop reduce the creep and stress-rupture strength. This was shown by independent tests on this alloy conducted at Aerojet-General Nucleonics and at Oak Ridge National Laboratory (References 4 and 15). A sufficient number of tests have not been run, however, to statistically determine the reduction to be anticipated. One obvious method of overcoming this reduction is to increase the tube wall thickness. However, this may accentuate the problem of thermally induced cracking since the imposed stress is a function of wall thickness. An alternate method - one which has met with apparent success - is to increase the initial long-time-elevated-temperature strength of the material through modification of the chemistry. The Steel and Tube Division of Timkin Roller Bearing Company, Canton, Ohio, added vanadium and columbium, along with other minor elements, to the standard 9Cr-1Mo chemistry. This produced an alloy which, in preliminary tests (Reference 16), exhibits 10,000-hr stress rupture strengths of approximately twice that of 9Cr-1Mo of standard chemistry. Additional testing is required to statistically establish acceptable design data and to determine how much reduction, if any, results from NaK exposure under SNAP-8 conditions on these properties.

VI. CORROSION LOOP PROGRAM

A. INTRODUCTION

The objectives of this program are to determine corrosion and mass-transfer patterns in the mercury and NaK loops of the SNAP-8 system, and to evaluate the corrosion resistance of the SNAP-8 reference materials with regard to the 10,000-hr life requirement. In addition, the corrosion loops have been used to investigate mercury boiler heat-transfer characteristics.

Corrosion Loops 1 and 2 (CL-1 and CL-2) were constructed of Haynes 25 alloy. Operation of the first loop was completed in 1962. The second loop was converted into Component Test Loop 2 that was operated to check the performance of certain components to be used in subsequent loops, and to run mercury-boiler performance tests.

The mercury-containment material for CL-3 and -4 was 9Cr-1Mo steel. The NaK primary loop was constructed of 316 SS with a section of Hastelloy C in the low-temperature area, and sections of chromalized Hastelloy N and 347 SS in the high-temperature area. Type 316 SS was used for the NaK condensing loop.

Corrosion Loop 3 completed its operating time in December 1964. The results of the evaluation of this loop were reported previously (Reference 4). Corrosion Loop 4 operation was started in January 1965; all of its operating time during this report period was used to conduct mercury boiler performance tests.

B. CORROSION LOOP 4

1. Mix-4P3E Additions

a. Introduction

The SNAP-8 system employs two space seals that separate the lubricant and cool fluid (mix-4P3E) from the mercury. One seal is used in the TAA, and the second seal is used in the mercury pump. There is some mix-4P3E diffusion into the mercury system from these seals during startup and continuous operation. The space seals are dynamic and do not function properly until rated speed is achieved. During startup, liftoff seals are employed to prevent leakage until the dynamic seals are operating. If the liftoff seals do not function properly or become worn during operation, they may provide the source of mix-4P3E contamination on restart.

It has been demonstrated (Reference 17) in loop tests at Aerojet-General Nucleonics that organic contaminants in the mercury "decondition" or degrade the performance of a SNAP-8 type forced-convection



once-through boiler. The purpose of this series of tests in CL-4 was to (1) determine the capability of a SNAP-8 type mercury boiler to digest mix-4P3E without degradation of thermal performance, and (2) determine the capability of the boiler to "condition" or recover original performance if such thermal performance were lost.

The tests performed in CL-4 are summarized. A complete description of the tests is given in Reference 18.

b. Test Run 11 with 0.9 ft/sec Liquid Velocity  
Boiler Inlet Plug

The object of this run was to determine the effect on the boiler performance when mix-4P3E is added to the mercury and to observe the reconditioning ability of the boiler using a low velocity plug.

Prior to the run, the boiler was considered conditioned. After loop operation had stabilized, mix-4P3E was admitted to the boiler in amounts to total 1, 3, 6, and 12 g.

There was no effect on the boiler performance when additions were made while the loop was operating; however, when the mercury boiling was restarted after the last addition, the boiler performance (as indicated by the NaK temperature profile) was found to be degraded. From this series of tests, it was observed a restart of mercury boiling was more detrimental to boiler performance than continuous operation, when mix-4P3E is present in the loop.

The boiler was operated for approximately 72 hr after 12 g of mix-4P3E had been added to the mercury stream and the boiling had been stopped and restarted. No conditioning of the boiler was noted during this time.

c. Test Runs with 4.5 ft/sec Liquid Velocity  
Boiler Inlet Plug

(1) Run 12

The object of this run was to determine the effectiveness of an intermediate liquid velocity plug in reconditioning a mix-4P3E contaminated boiler.

No attempt was made to clean the boiler after removal of the boiler inlet plug from the previous run. The loop was operated for 150 hr during the run. There was a continuous improvement in boiler performance noted both from the NaK shell temperature profiles and the calculated average heat flux for the boiler. At the end of the run the boiler was considered to be conditioned.

(2) Run 13

The object of this run was to determine the effect on boiler performance when mix-4P3E was added to the mercury and the ability of the boiler to recondition when the 4.5 ft/sec boiler inlet plug was installed.

Additions of mix-4P3E were made to the mercury in two steps so that a total of 1 and 3 g were injected into the mercury. The mercury boiling was interrupted and restarted after each mix-4P3E addition since Run 11 indicated the boiler performance decreased more rapidly when this was done.

Boiler performance was degraded after a total of 3 g of mix-4P3E was injected and the boiling was stopped and restarted. An operating time of 100 hr was necessary to recondition the boiler.

(3) Run 14

The object of this run was to determine the effect of a mix-4P3E addition to the mercury when it was injected into the boiler at startup.

When the mercury boiling was started, 3 g of mix-4P3E was added with the mercury. This amount of mix-4P3E caused a decrease in boiler performance, and the mercury boiling was continued to recondition the boiler. The loop was operated for approximately 70 hr when a plant-wide power failure caused a loop shutdown for 1 hr. Loop operation was continued for another 70 hr (140 hr total). At the end of this time, the boiler was considered conditioned.

(4) Run 15

The object of this run was to determine the effect of removing the noncondensable gas generated by the breakdown of mix-4P3E in the rate of boiler conditioning. In previous runs the noncondensable gases generated by the breakdown of the mix-4P3E in the boiler were not removed prior to reconditioning the boiler.

The loop was started and 3 g of mix-4P3E was injected into the mercury causing the boiler performance to degrade. Mercury boiling was stopped and the mercury system was evacuated to remove the noncondensable gases. Mercury boiling was resumed and there was no improvement in performance or conditioning rate when compared to Runs 13 and 14.

d. Test Run 16 with 6 ft/sec Liquid Velocity  
Boiler Inlet Plug

The object of this run was to determine the effect of mix-4P3E addition to the mercury on the performance of the boiler when a high-liquid-velocity inlet plug was installed.

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The loop was started and it was necessary to add 12 g of mix-4P3E to the mercury before the boiler performance was degraded. The boiler was conditioned after operating for 24 hr.

e. Summary

This series of tests showed that additions of mix-4P3E to the mercury causes degradation of the performance of SNAP-8-type mercury boilers. The amount of mix-4P3E that would be necessary to degrade the performance of a full-scale SNAP-8 mercury boiler could not be determined from these tests.

Increased liquid velocity in the preheat region of the boiler inlet plug increased the tolerance of the boiler to additions of mix-4P3E. Also, the increased liquid velocity decreased the time necessary for the boiler to regain its thermal performance or condition.

Surface contamination of the boiler tubing appears to be one factor responsible for poor thermal performance of SNAP-8 type mercury boilers.

2. Multimaterial Boiler Inlet Plug Test

a. Introduction

During the test program to develop mercury boiler inlet plugs (Reference 19) in Corrosion Loop 4, corrosion was seen on the inlet plugs where the mercury preheat liquid velocity was increased by a tightly spiraled wire wrapped around the plug. The corrosion was observed after operating times of 100 to 150 hr, and the corrosion rate was estimated to be great enough to destroy the geometric pattern of the plug within 10,000 hr.

A boiler inlet plug was designed (Figure 18) and tested that included several diverse materials in the preheat section including the reference SNAP-8 material for mercury containment (9Cr-1Mo). The objective of the test was (1) to determine the difference in corrosion between several materials at the conditions in the boiler inlet plug region, and (2) to determine whether or not erosion is a predominant factor in loss of material from the inlet plug.

A summary of the multi-material inlet plug test is given below, and a complete description of the test can be found in Reference 20.

b. Summary

The test of the multi-material plug included the following materials in the preheat section of the boiler:

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- Carburized AISI 1020 steel
- Dynacut Tool Steel
- Nitralloy-Nitrided Case
- AISI 1020 Steel
- Tantalum
- 18-4-1 Tool Steel
- 9Cr-1Mo Steel

After fabrication was completed, the plug was installed in the CL-4 boiler. The loop was started and 110 hr of operating time was necessary to condition the boiler so that the reference NaK profile was obtained. Operation was continued for an additional 249 hr, making a total operating time of 359 hr. A shutdown occurred after 213 hr of total operating time so that the corrosion of the inlet plug could be evaluated.

The evaluation of the materials that made up the plug included visual inspection, weight loss measurements, microhardness measurements, and metallography. Both AISI 1020 steel specimens were completely wetted by mercury. They also showed the highest corrosion rate. The other iron-base alloys were not wetted by mercury and suffered low corrosion rates. The tantalum section was not attacked by the mercury, indicating that erosion was not the predominant mechanism for loss of material in the preheat section of the boiler inlet plug.

Since the entire test section in the boiler inlet plug was not uniformly wetted by mercury, the relative corrosion resistance of some of the materials tested is uncertain.

### 3. Mix-4P3E Mercury Separator Tests

#### a. Introduction

Previous tests (Reference 1) in CL-4 indicated that small amounts of the SNAP-8 lubricant-coolant fluid (mix-4P3E) are detrimental to the heat transfer characteristics of the mercury boiler. From examination of the boiler inlet plug, it was apparent a large part of the mix-4P3E was thermally decomposed where the mercury mix-4P3E mixture first contacted the plug region heated by the NaK. As a result of these observations, a task was started to design and test in CL-4 a mix-4P3E mercury separator that would prevent mix-4P3E from entering the boiler. The objective of the separator design effort was to obtain information that could be used to design a test-support-equipment separator for the systems loops at SNAP-8 Division, Von Karman Center.

Two design concepts were analyzed (1) a gravity-type separator, and (2) a thermal-decomposition type separator. Design of the thermal-decomposition type separator and the gravity separator proceeded concurrently. It was originally intended to proceed the thermal-decomposition separator with the gravity-type separator so that ideally a small steady flow of mix-4P3E into the thermal-decomposition separator would occur. This would reduce the rate of gas generation in the thermal-decomposition separator. Testing of a glass model of the gravity-type separator showed no detectable mix-4P3E in the underflow. Effort on the thermal-decomposition separator was stopped in favor of the gravity type separator since the measurement and analysis problems with the thermal-decomposition type separator were judged to be beyond the scope of the program.

b. Summary

A glass model of a gravity-type separator was fabricated and tested. A series of test runs was made over a range of mercury flow rates (500-1000 lb/hr) and the results showed a high separator efficiency.

Since the results of the glass gravity-type separator were favorable, a separator with similar design parameters was fabricated and installed in CL-4. With the use of a boiler inlet plug that gave a preheat liquid velocity of 6 ft/sec, a series of mix-4P3E injections was made during loop operation to test the separator effectiveness in a dynamic loop.

After each addition of mix-4P3E, no effect was noted on the boiler performance; however, the total amount of mix-4P3E added to the loop could not be recovered from the separator at the end of the test.

The test performed (Reference 21) indicates that a gravity-type separator could reduce the amount of mix-4P3E entering the boiler sufficiently to prevent any impairment of the boiler heat-transfer characteristics, and should be considered for the design of a test-support-equipment separator for the system loops.

4. In Situ Hg Boiler Tube Evaluation

A task was started to evaluate the feasibility of using an X-ray technique for in-situ examination of the internal Hg containment tube of tube-in-tube CL-4 boiler. Such examinations would be made during periodic loop shutdowns, and would be conducted to spot incipient cracks. Preliminary tests thus far completed established that evaluation of the tube wall is possible when X-ray film is exposed while positioned inside the tube, with an X-ray source external to the tube. Figure 19 describes the test exposures. The tube-in-tube boiler design militates against placement of the film against the outside surface of the Hg containment tube. This area is not conveniently accessible; otherwise, unnecessary and undesirable boiler cutting would be required. In addition, the passage is blocked against deep insertion of the film along the tube length by intermittently spaced Hg support brackets.

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Although results with the film in either internal position produced satisfactory radiographs, the single wall technique utilizing a flat film inside the tube appears to be the optimum position. The film can be easily inserted into the tube, and is self-locating. The disadvantage of the alternate single wall technique (i.e., the use of a curved film) is the requirement of somehow holding the film against the inside tube wall. This becomes especially difficult as the depth of insertion increases.

C. RUBIDIUM ADDITIONS TO COMPONENT TEST LOOP 2

A task was initiated during the last report period to investigate the effect on the CTL-2 boiler performance created by adding rubidium to the mercury. Its objectives were to determine (1) the effect of the SNAP-8 L/C fluid (mix-4P3E) on boiler performance, (2) the amount of Rb necessary to improve the performance of a deconditioned boiler, and (3) the amount of Rb lost during an extended loop run.

The tests with CTL-2 were completed during this report period, and the results are reported in Reference 29.

The conclusions reached from the experiments made during this task are as follows:

- A 25-g injection of mix-4P3E deconditioned the CTL-2 boiler
- The addition of Rb (~ 1100 ppm) to the mercury caused the CTL-2 boiler to condition. Operating time also aided in the conditioning process
- The oxidation method used to remove Rb from the mercury in CTL-2 was effective as measured by the analysis of mercury for Rb.

The decrease in Rb concentration during the run, and the results of the mass balance on the Rb added and extracted from the mercury, showed that some Rb was left in the loop.

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TABLE 1CORROSION OF SNAP-8 CONSTRUCTION MATERIALS BY CANDIDATE  
Hg DECONTAMINATION FLUIDS

Solution	Calculated Corrosion Rate <sup>(1)</sup> (microin./hr) for Materials		
	9Cr-1Mo	1010	316 SS
62% Nitric Acid			
Uninhibited	30	Dissolved in 3 min	85
1 wt% sodium nitrite	10 <sup>(2)</sup>	139	11
1 wt% each sodium nitrite and sodium dichromate	115	Dissolved in 3 min <sup>(3)</sup>	6
70% Nitric Acid			
1 wt% sodium nitrite	115	140	113
2 wt% sodium nitrite	117	84	117

- (1) Calculation based on measured specimen weight loss. Average of two specimens unless otherwise indicated.
- (2) One specimen only. Second specimen exhibited an extremely high corrosion rate presumed to be due to dislodgement of mass transfer surface deposit.
- (3) One specimen only. Second specimen exhibited corrosion rate of 148 microin./hr.

Table 1

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TABLE 2

SNAP-8 -1 BOLLER<sup>(1)</sup> PERFORMANCE HISTORY IN RPL-2

Operating Period	July 1964 to June 1965
NaK Side	
Total operation	2350 hr <sup>(2)</sup>
Nominal boiler design conditions	
Flow (lb/hr)	32,000
Temperature (°F)	
Inlet	1300
Outlet	1100
Hg Side	
Total operation	1415 hr
With rubidium	320 hr (Nov. 1964 to Mar. 1965)
Nominal boiler design conditions	
Flow (lb/hr)	11,400
Temperature (°F)	
Inlet	513
Outlet	1265
Pressure (psia)	
Inlet	340
Outlet	270
Vapor outlet conditions (typical of various test periods)	
90% quality	470 hr <sup>(3)</sup>
Saturated vapor	240 hr
9 ft superheat length	125 hr
27 ft superheat length	460 hr
42 ft superheat length	120 hr

(1) (P/N 092020-1F S/N A-1).

(2) Approximately the last 300 hr included a NaK purification system.

(3) Flow was 50% of nominal to enhance conditioning.

**TABLE 3**  
**DIMENSIONAL EVALUATION OF -1 BOILER TUBES (1)**

Tube Coil No.	Cracks at Hg Outlet Bend	Wall Thickness (inches)				Outside Diameter (inches)				Effective Creep Rate (2) at Bend x 10 <sup>-4</sup> %/hr
		At Bend		Straight Section		At Bend		Straight Section		
		Min	Max	Min	Max	Plane 1	Plane 2	Plane 1	Plane 2	
I-1	Yes	0.103	0.136	0.106	0.132	1.171	1.165	1.148	1.149	12.1
I-2	No	0.116	0.135	0.114	0.131	--	--	--	--	--
O-3	No	0.119	0.136	0.117	0.126	1.142	1.164	1.145	1.154	1.62
O-4	Yes	0.094	0.144	0.107	0.137	1.169	1.170	1.147	1.153	12.9

(1) Nominal tube dimensions 1.152 in. OD by 0.125 in. wall

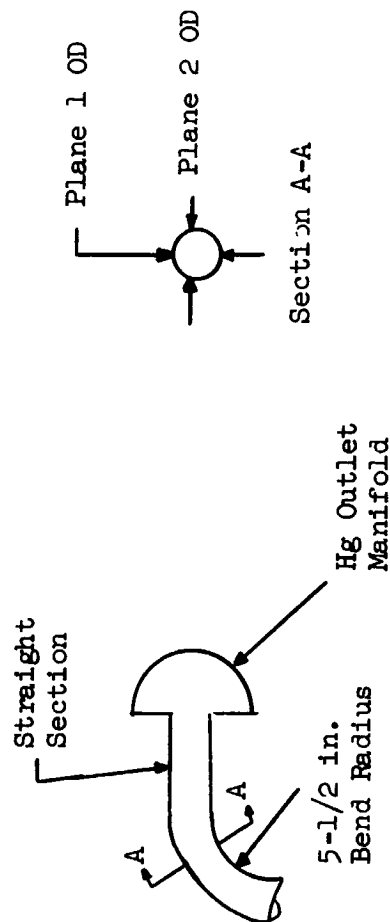
(2)  $\bar{\epsilon} = 1.15 \ln \frac{D_2}{D_1}$

where

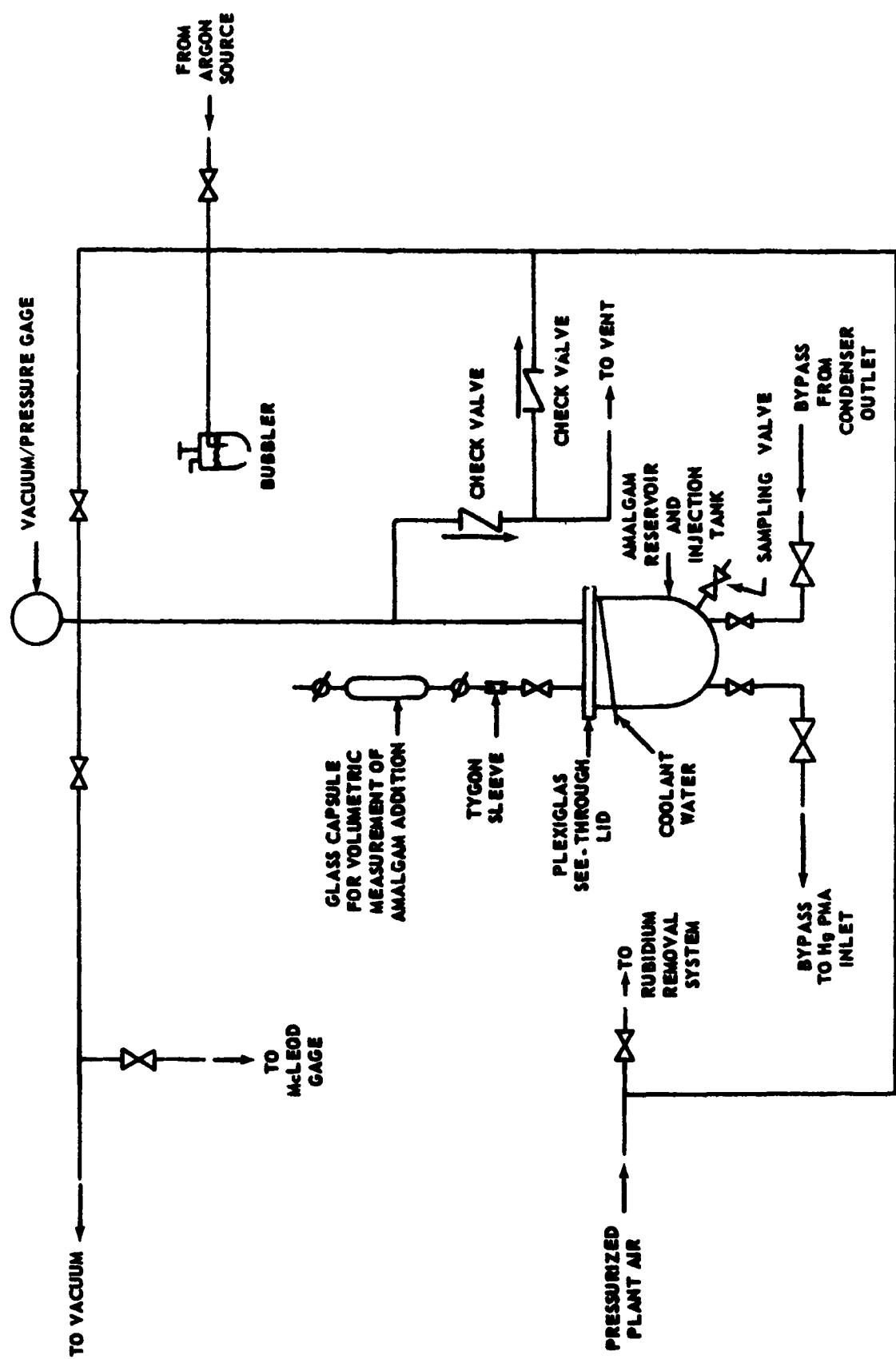
$\bar{\epsilon}$  = effective creep rate, %/hr

$D_2$  = final tube diameter, in.

$D_1$  = initial tube diameter, in.



1265-NF-1096B

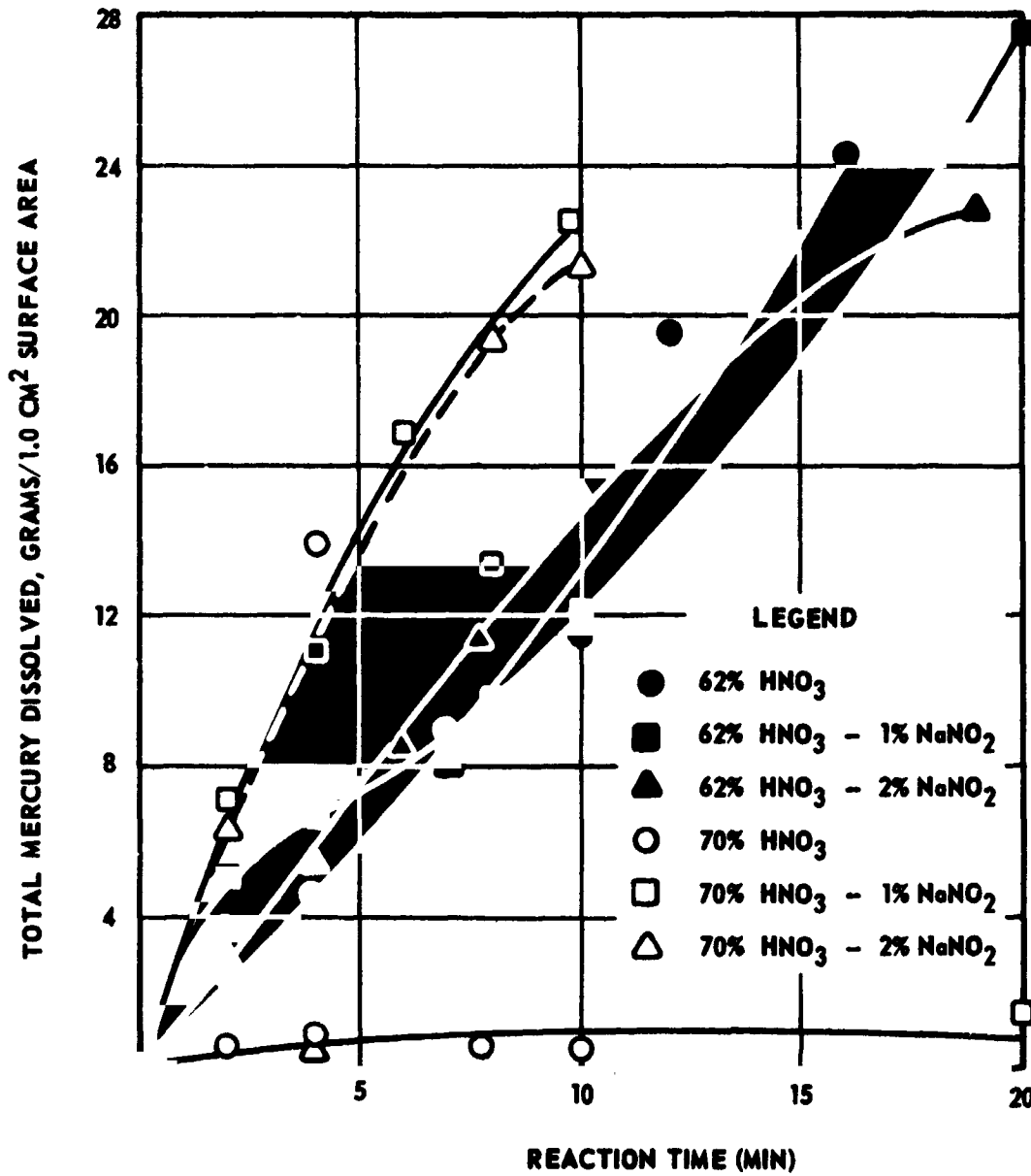


Schematic of Rb-Hg Amalgam Injection System Used in FCS-1/SL-1 During November 1965

Figure 1

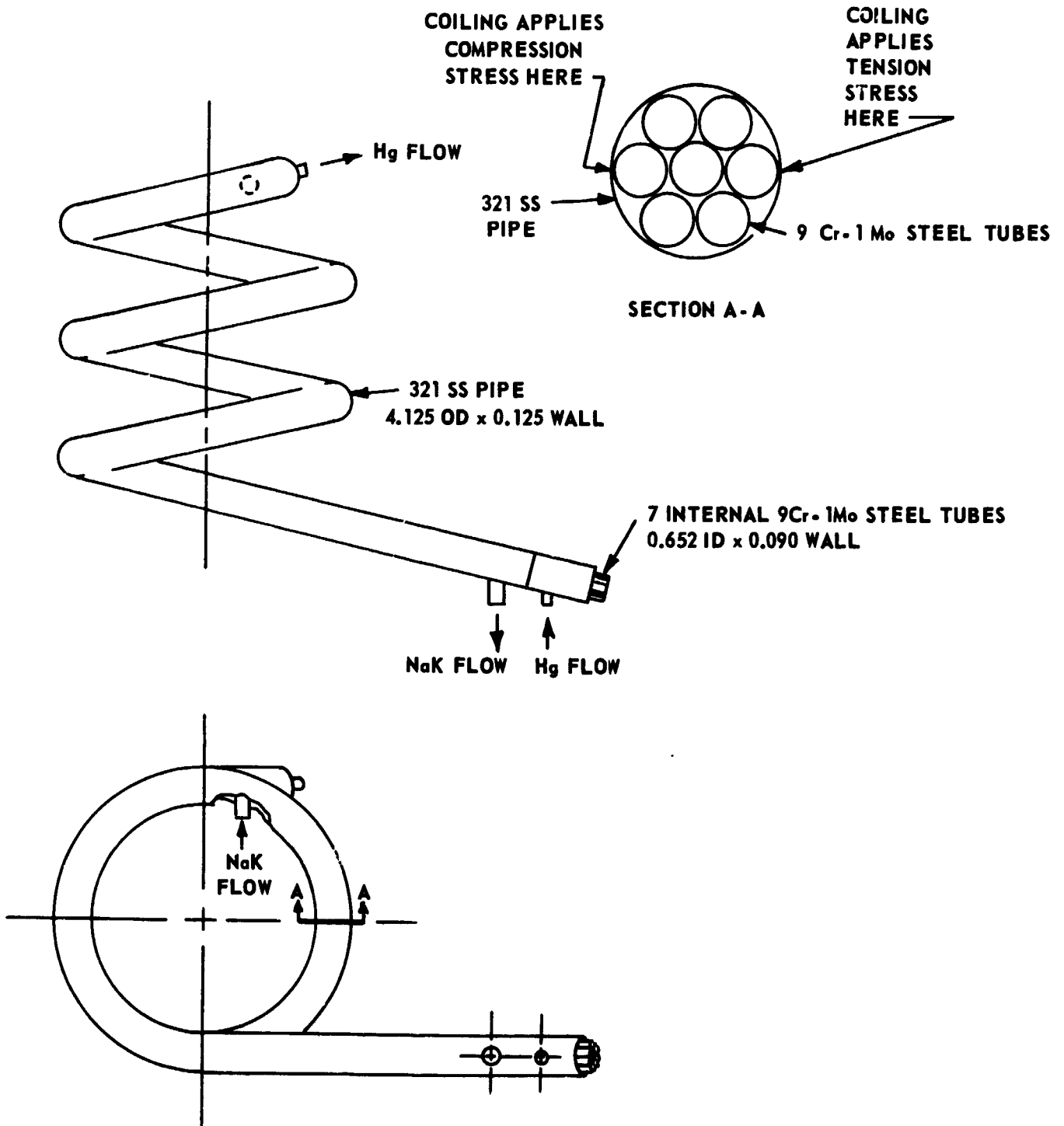


1265-NF-1097A



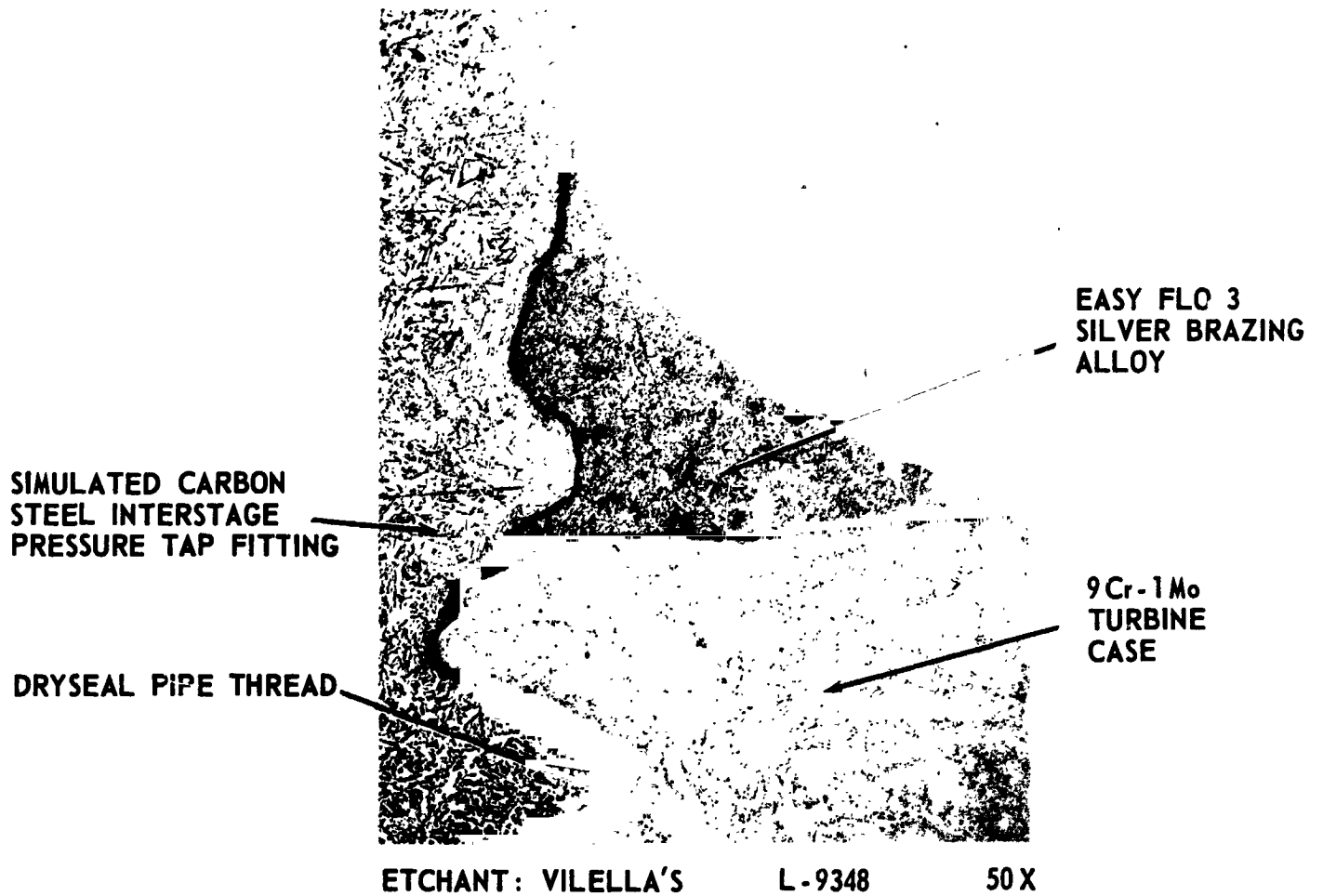
Dissolution Rate of Hg by Various Nitric Acid Solutions at Room Temperature

Figure 3



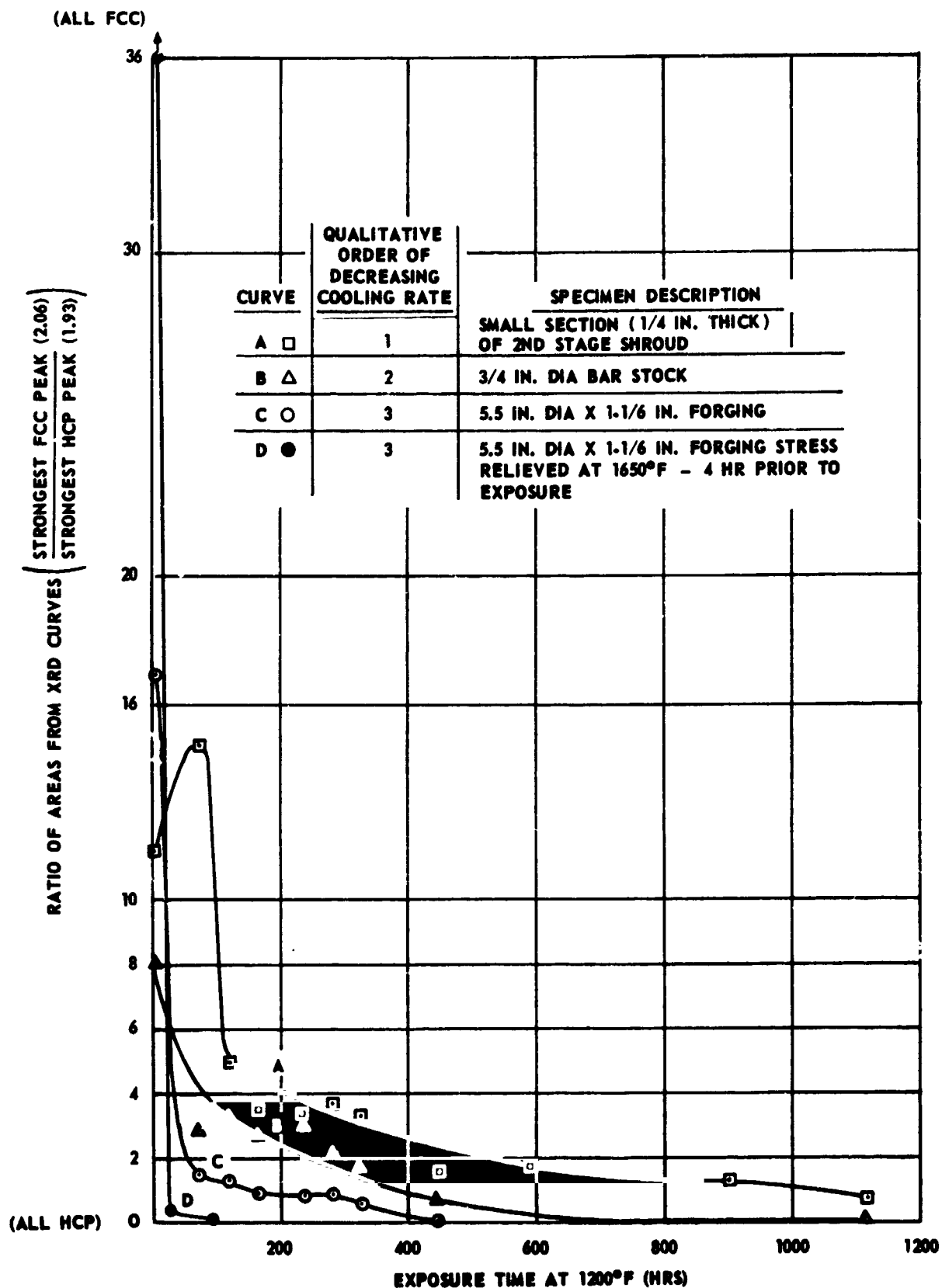
Tube-in-Tube Boiler Conceptual Design

Figure 4



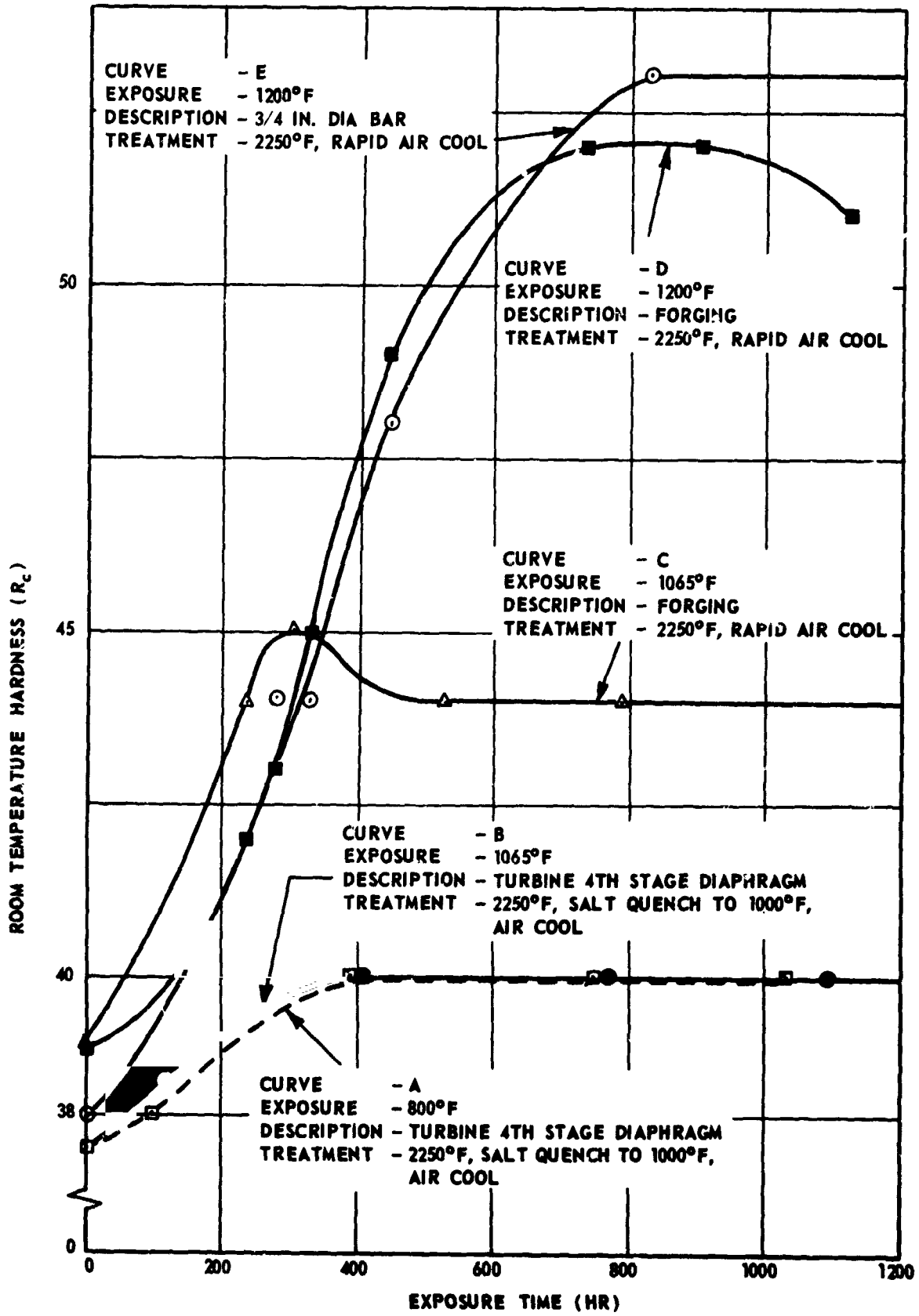
Simulated Turbine Assembly (S/N A-1) Brazed Joint Between Carbon Steel Interstage Pressure-Tap Fitting and 9Cr-1Mo Steel Turbine Case. Joint Produced by Torch Brazing Using Easy Flo 3 Braze Alloy and Flux.





Effect of Air Cooling Rate from the Solution-Annealing Temperature (2250°F) on the Rate of Stellite 6B transformation from FCC to HCP at 1200°F

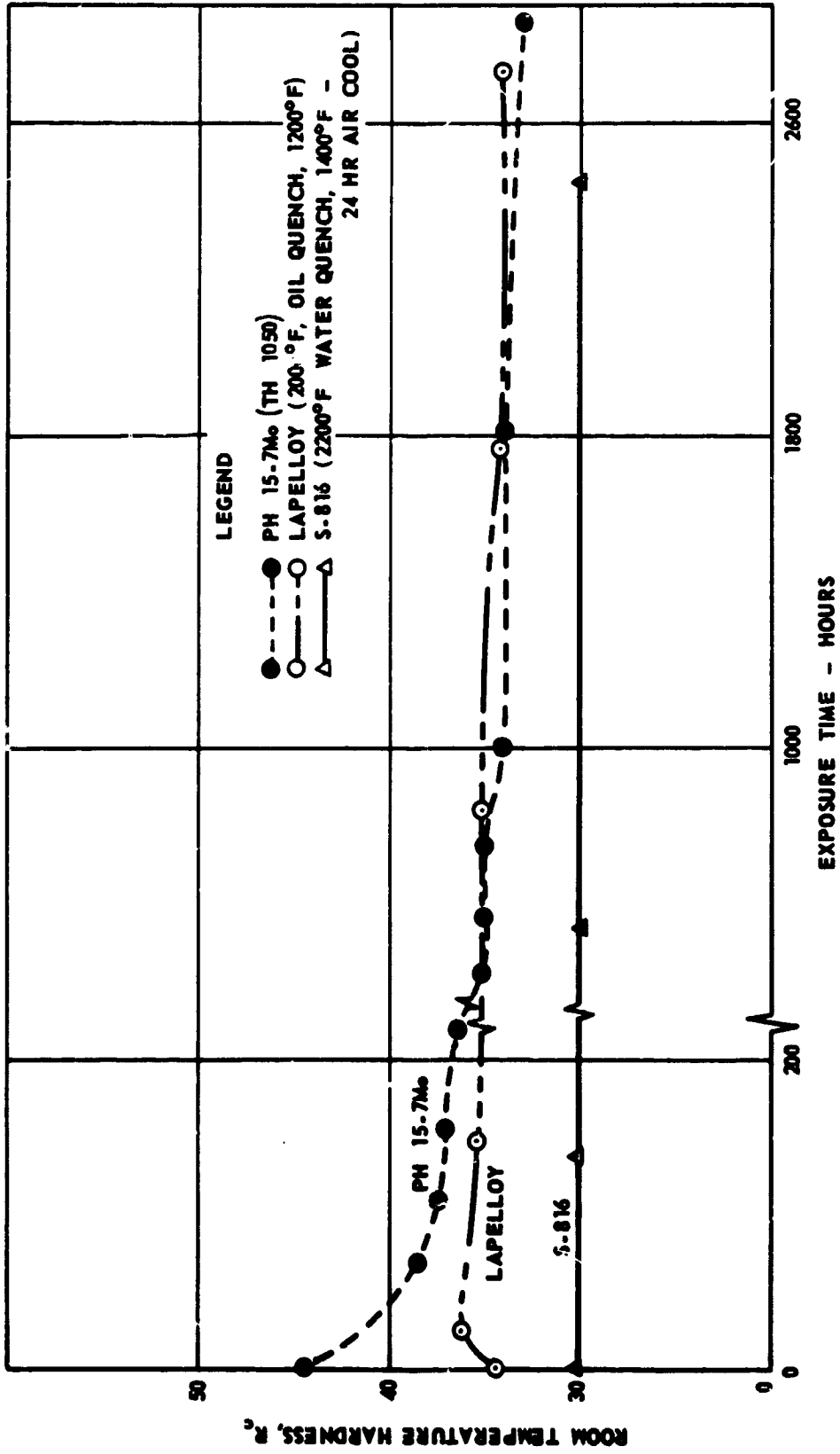
Figure 6



Effect of Cooling Rate from the Solution-Annealing Temperature (2250°F) on the Hardness of Stellite 6B with Subsequent Exposure at 800, 1065 and 1200°F.

Figure 7

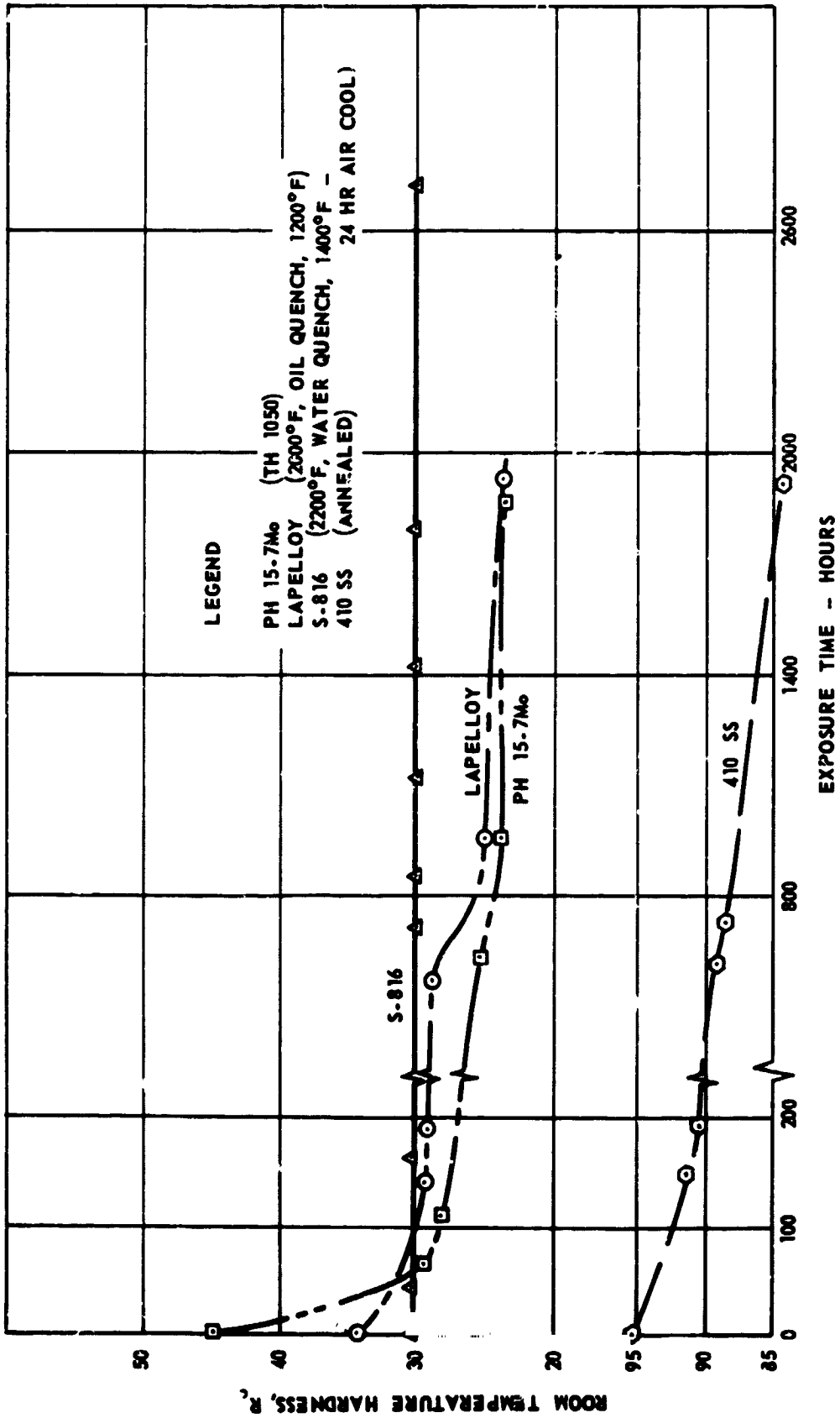
A266-NF-1146A



Effect of Exposure Time at 1065°F on the Room-Temperature Hardness of Various Turbine Alloys

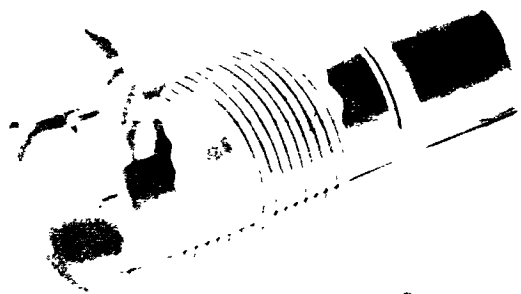
Figure 8

A266-NF-1147A



Effect of Exposure Time at 1200°F on the Room-Temperature Hardness of Various Turbine Alloys

Figure 9



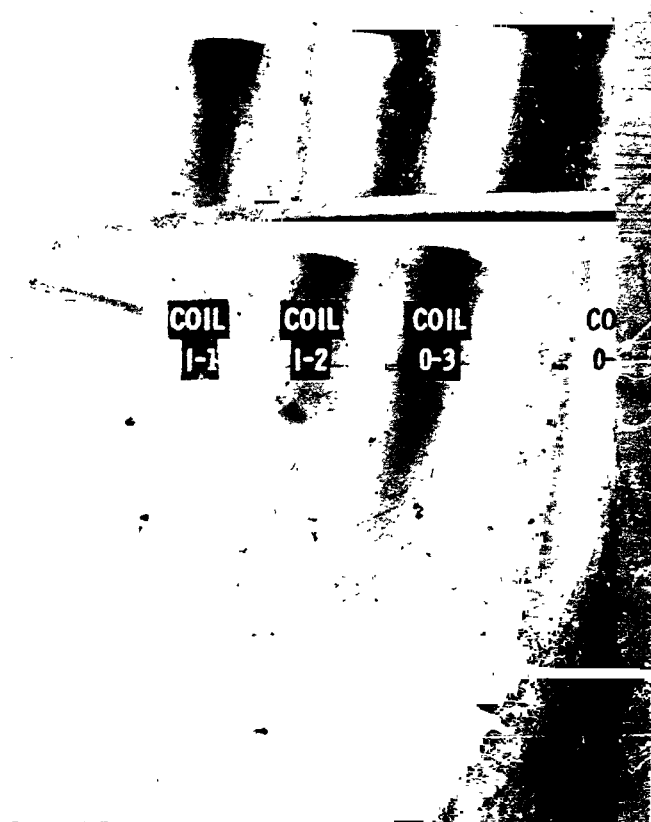
**Cutaway Drawing of Tube-in-Shell Boiler**



**SNAP-8 Tube-in-Shell Boiler**

**Figure 10**

B266-NF-1145/A



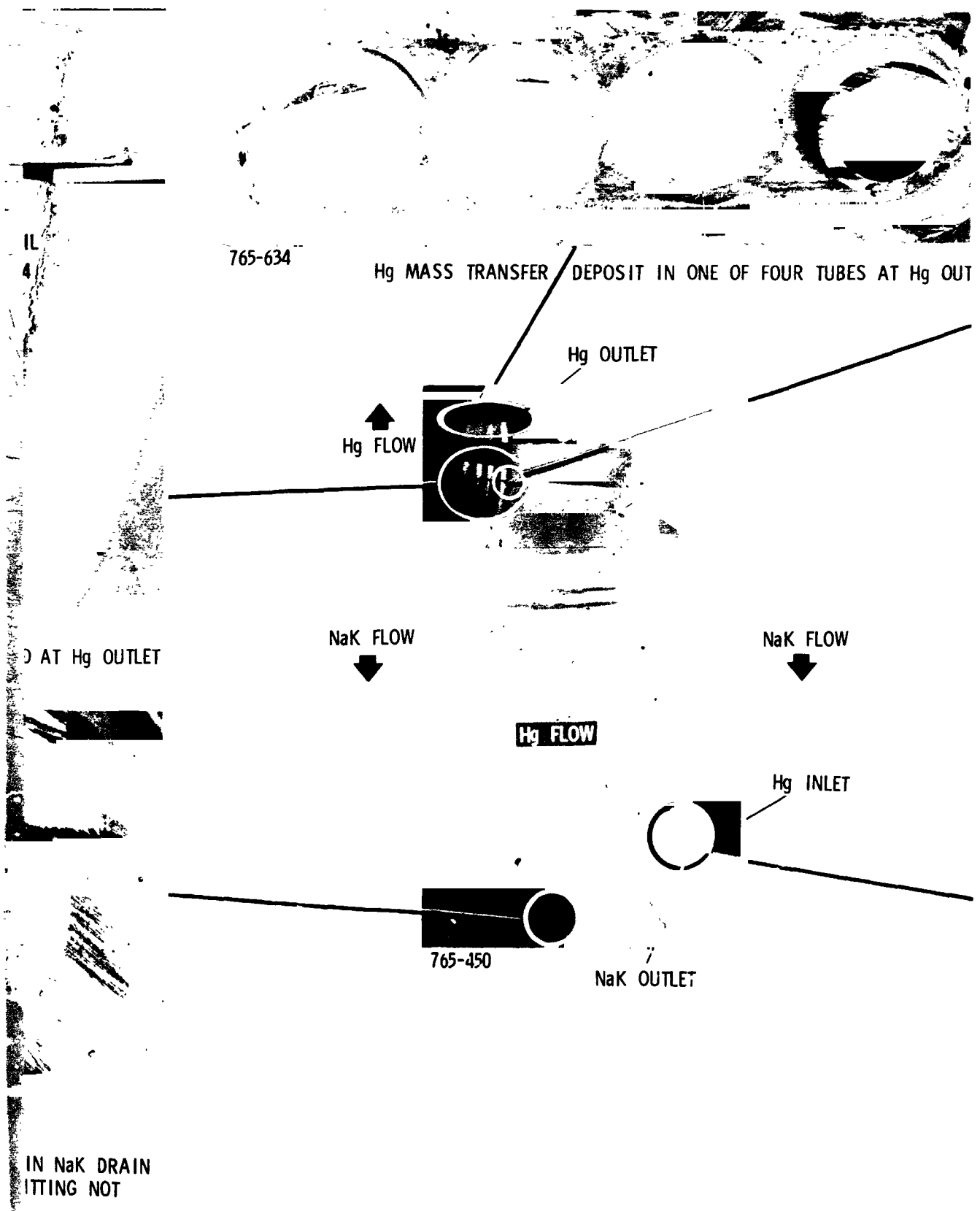
765-451

Hg TUBE SURFACE CRACKS AT 5 1/2 IN. R. BEN



765-633

NaK MASS TRANSFER DEPOSIT  
FITTING AT NaK OUTLET END (F  
SHOWN ON BOILER)



Visual Appearance of NaK Side of -1 Boiler (P/N 092020-15 S/N A-1) After Removal of Outer Shell. Boiler Removed from RPL-2 After 2350 hr of NaK-Side Operation.



LET



765-452

NaK MASS TRANSFER DEPOSIT AT  
NaK INLET END OF BOILER

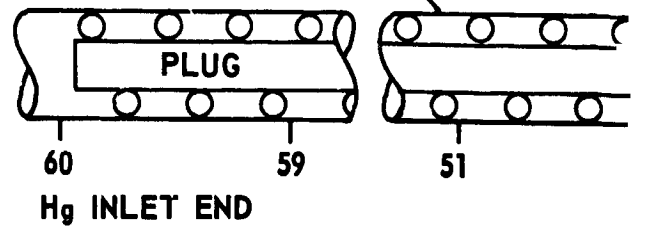
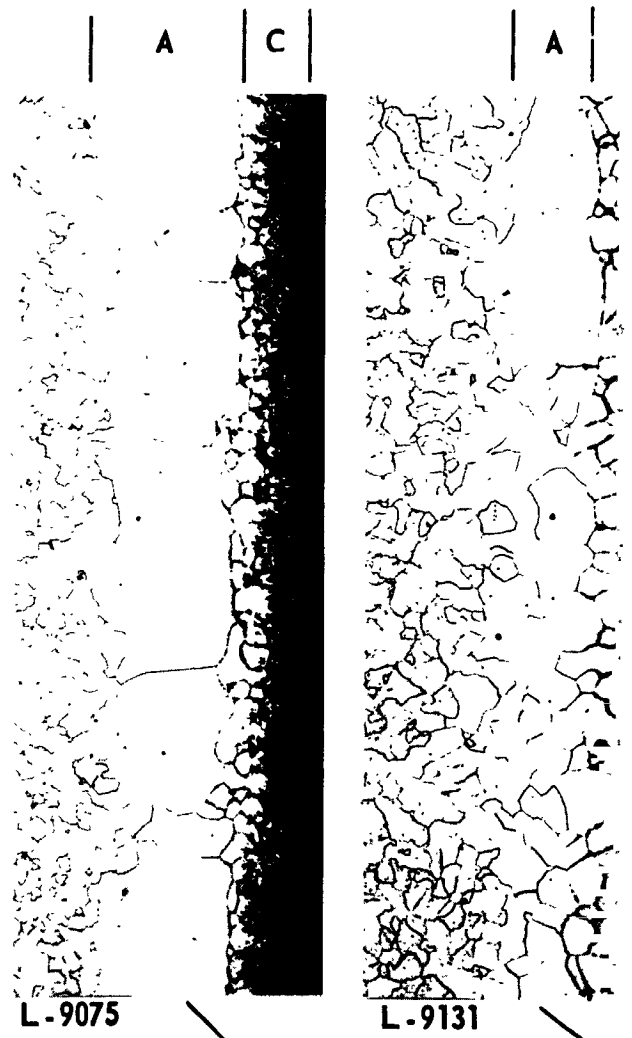


765-632

NaK MASS TRANSFER DEPOSIT AT NaK OUTLET

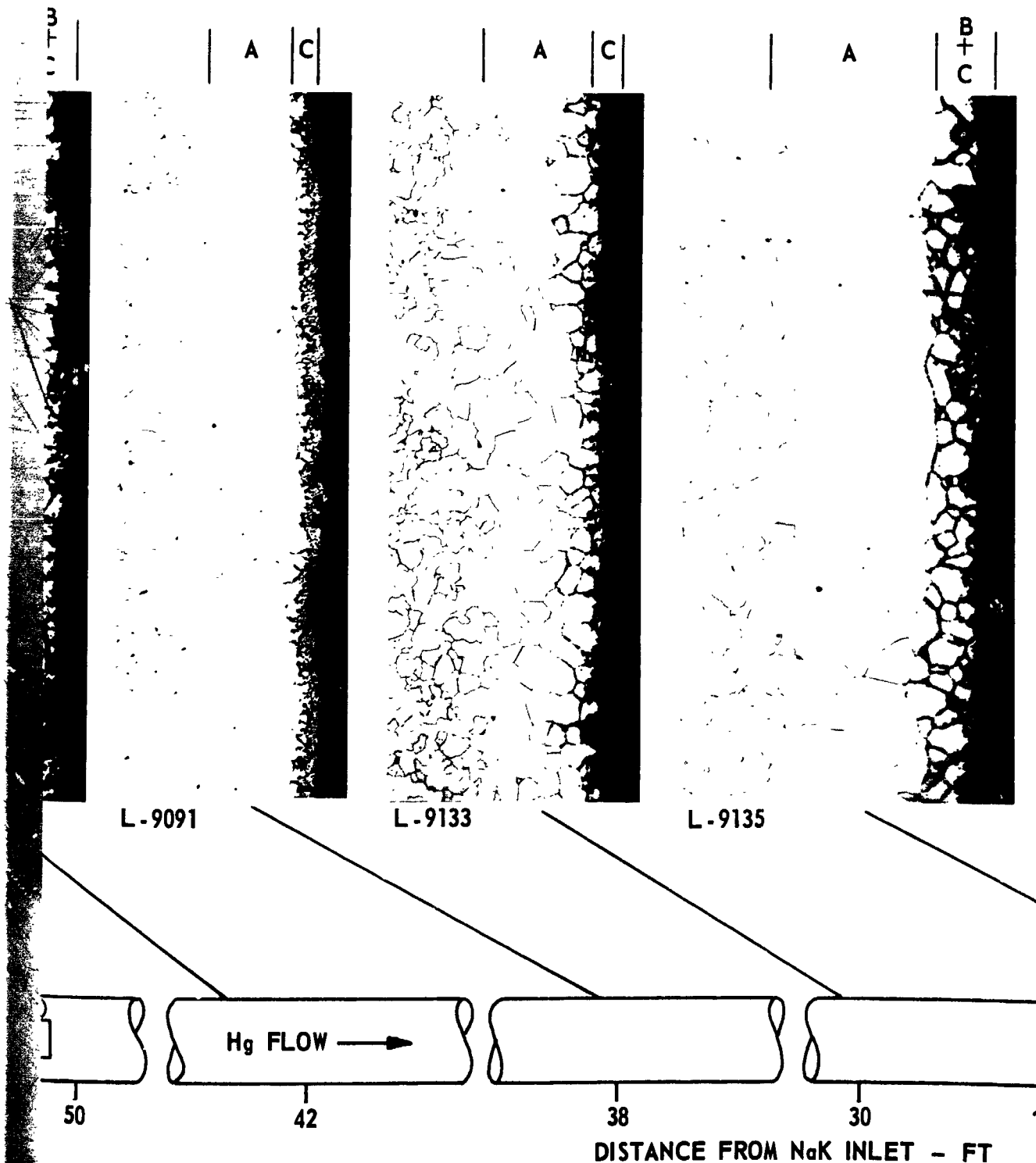


Bl66-NF-1161/A



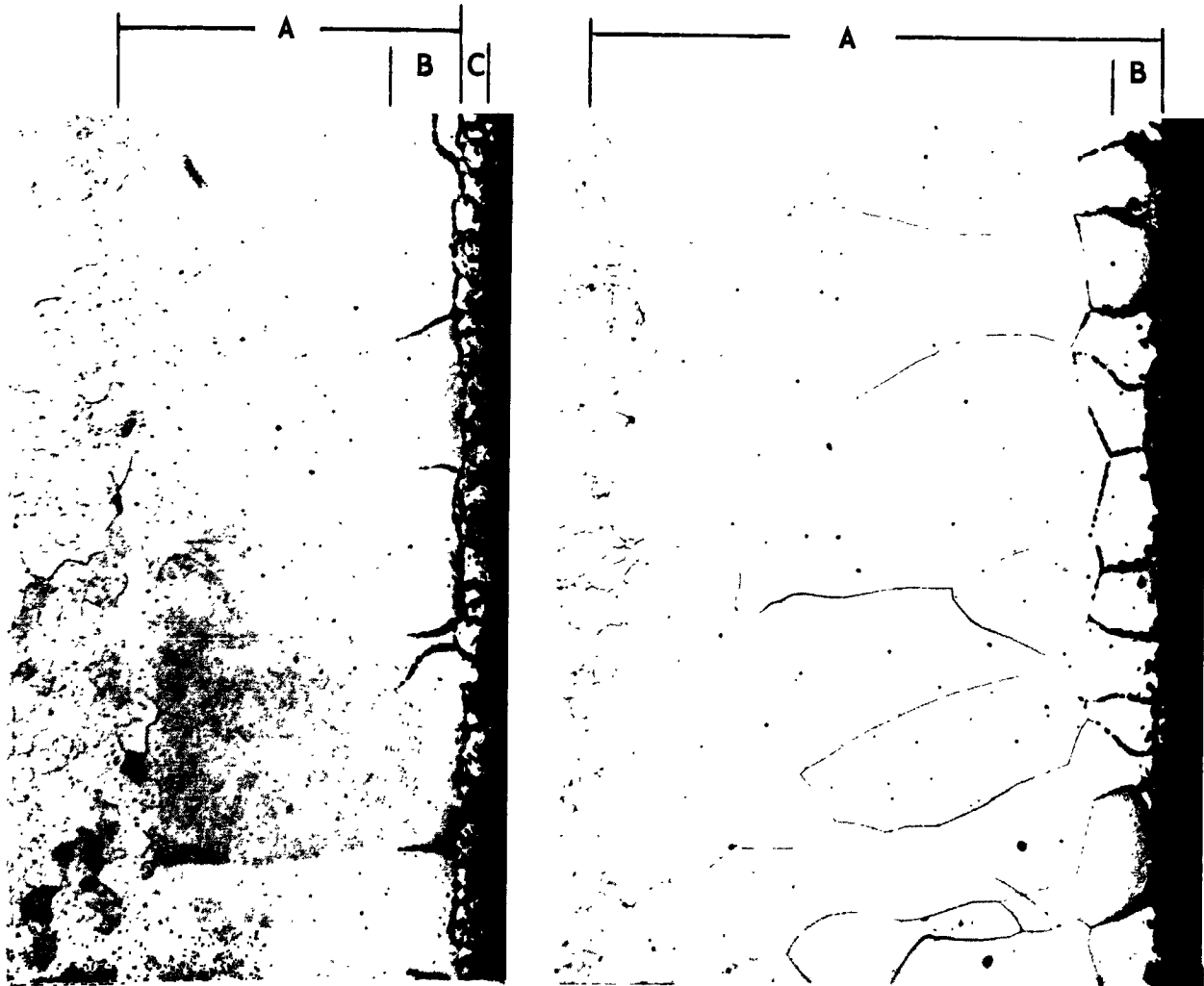
0 INCH .005 .010 .015 .020

SCALE (150 X)



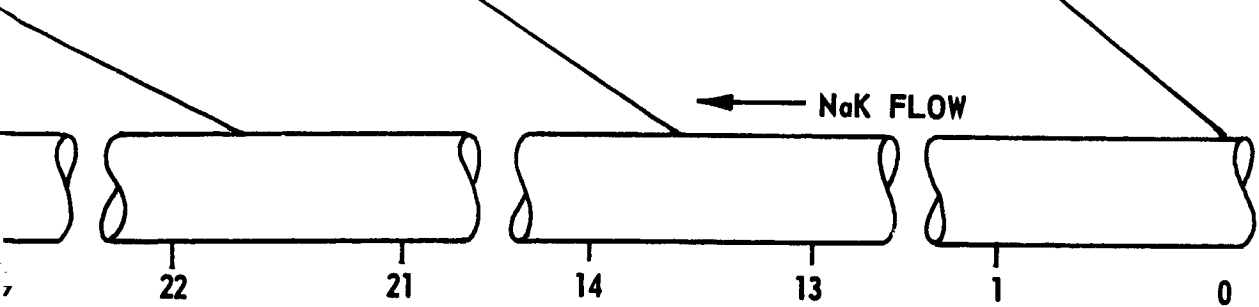
NaK-Exposed Surfaces of Hg Containment Coil No. O-4  
 After 2350 hr of NaK-Side Operation in RPL-2 (Vile:  
 All Areas Show Concomitant Decarburization and Grai.  
 The Former is Delineated at Higher Magnification.  
 Corrosion (Area B) and Mass-Transfer Deposits (Area  
 Present.

.025



L-9152

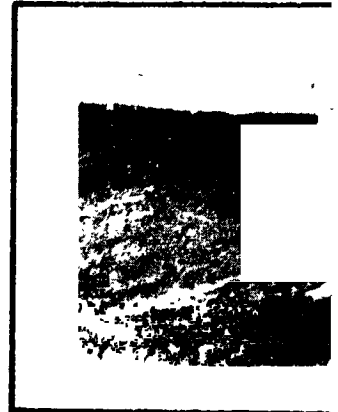
L-9159



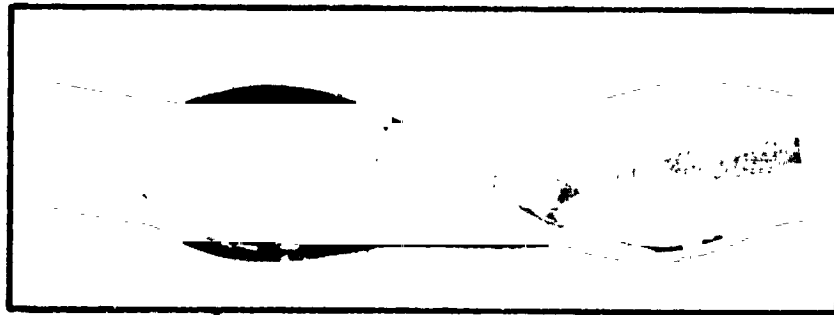
THIS END CONNECTS TO 5-1/2 IN. RADIUS BEND AT Hg OUTLET MANIFOLD

of -1 Boiler  
lla's Etch).  
n Growth (Area A).  
Intergranular  
C) Are Also

Figure 12-3



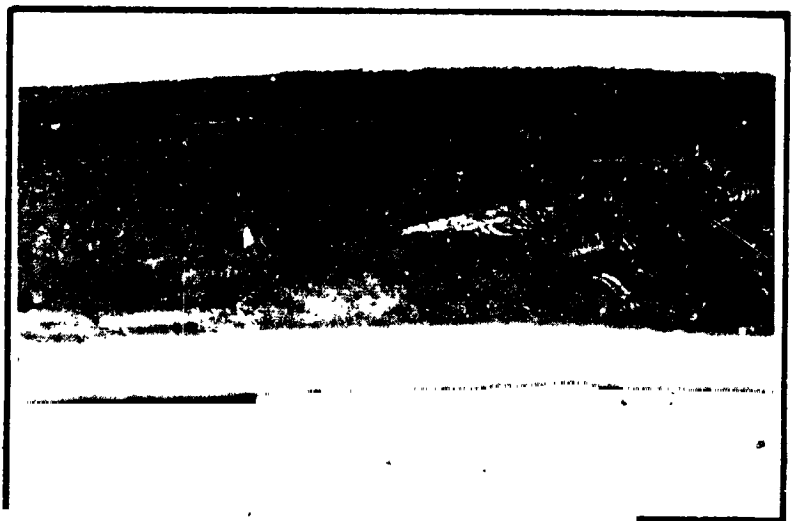
L-9057



P865-163

SURFACE ATTACK ON PLUG

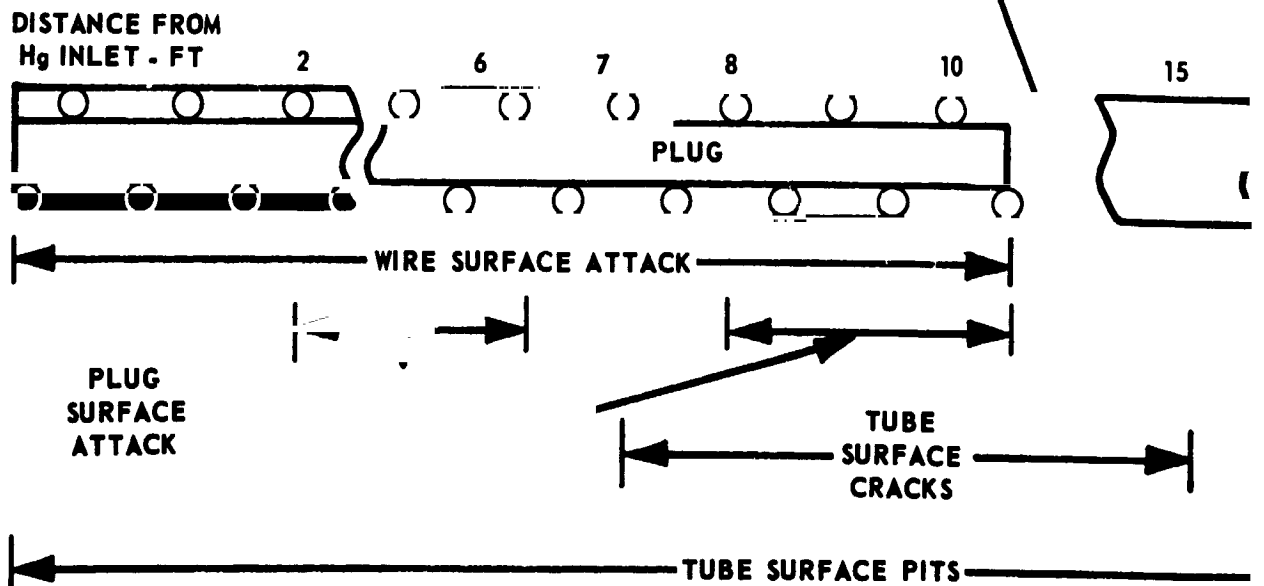
1X

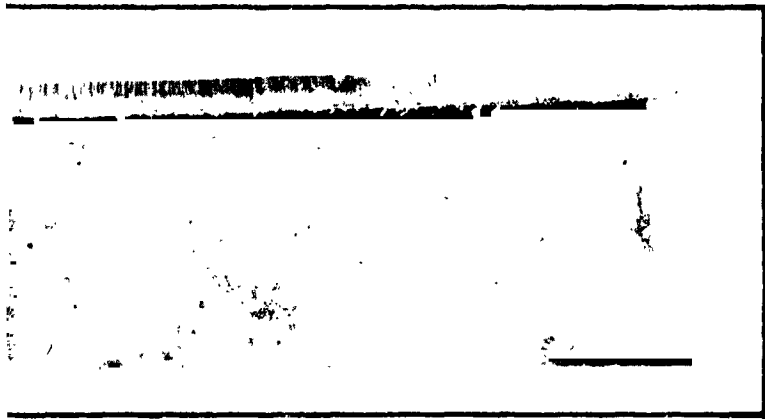


L-9058

TUBE CRACKS

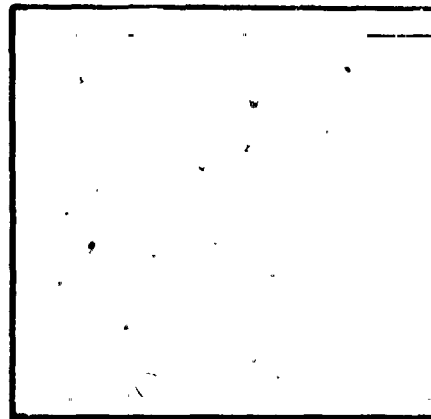
2-3/4X





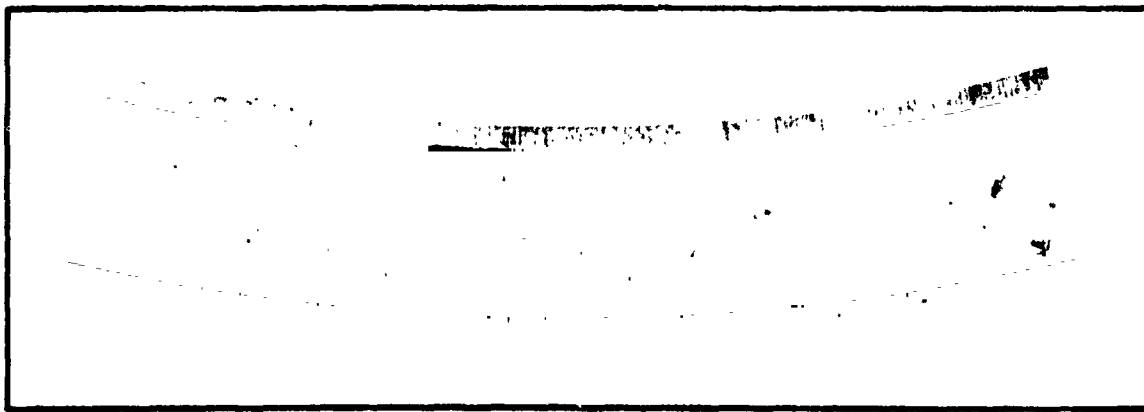
TUBE PITTING

2-1/4X



L-9456

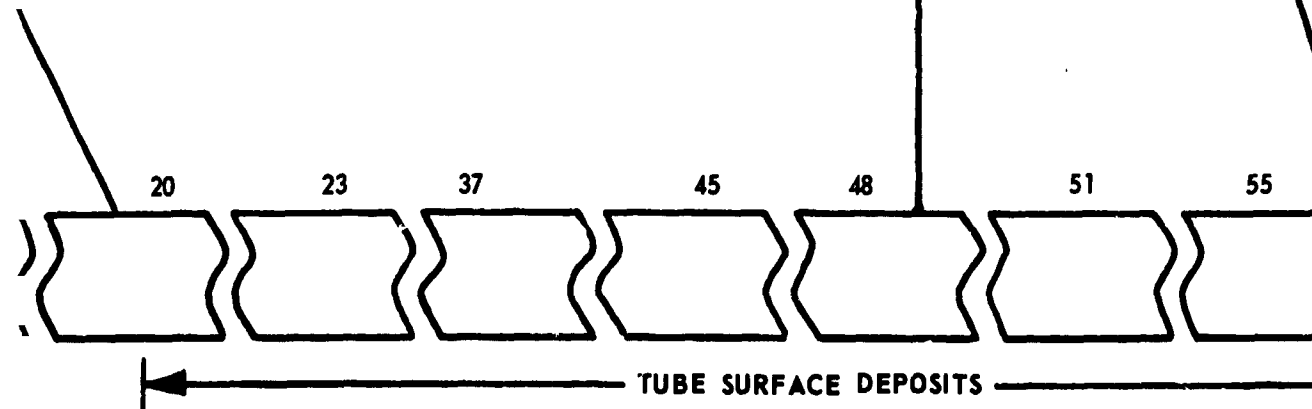
T



P865-166

UNIFORM MASS TRANSFER  
DEPOSIT

1X

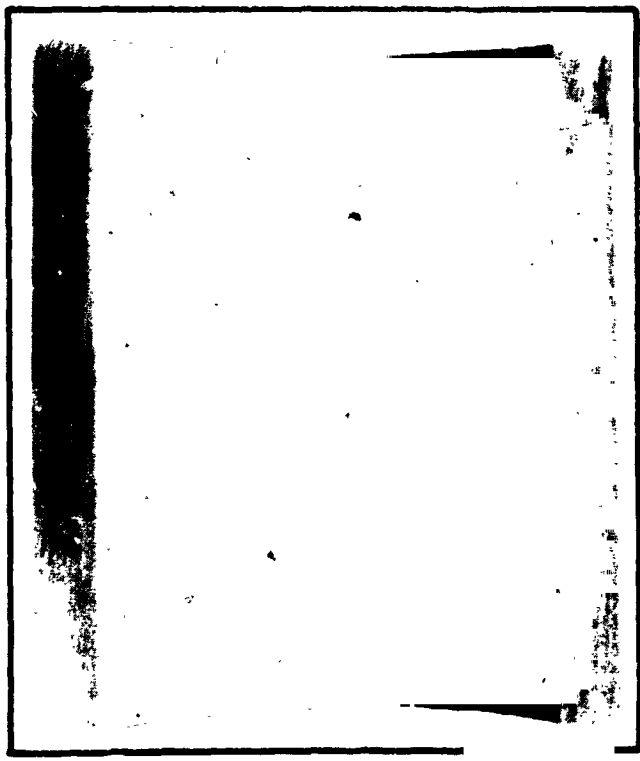


Mercury Side of Tube Coil No. 0-4 of -1 Boiler Removed From RPL-2  
After 1415 hr of Mercury Operation



**TUBE CRACKS IN CRISS-CROSS  
PATTERN**

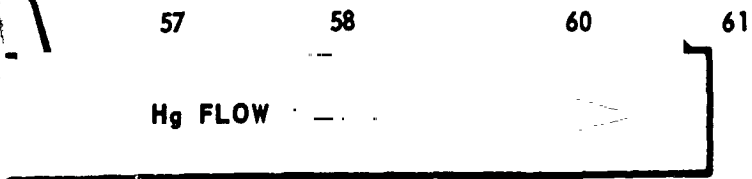
3X



L-8955

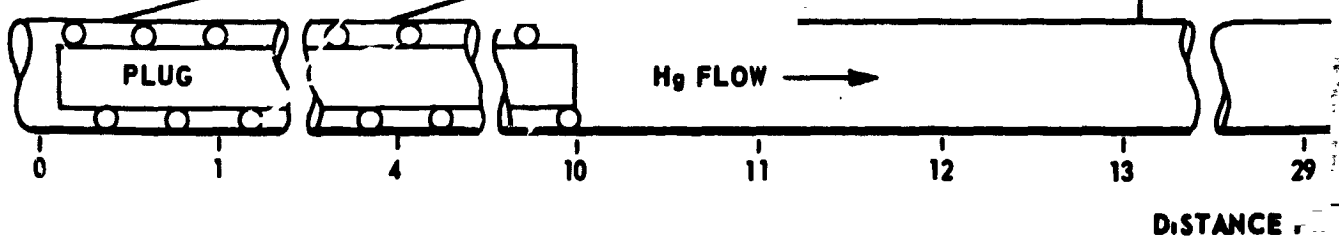
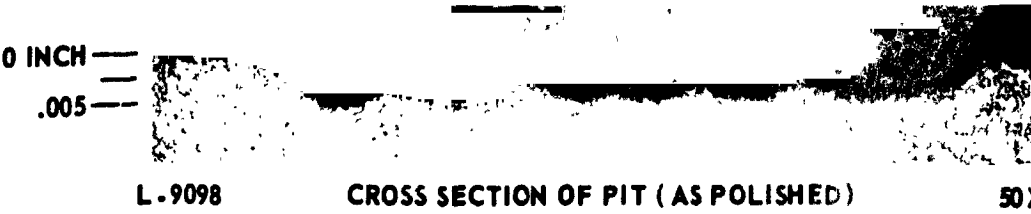
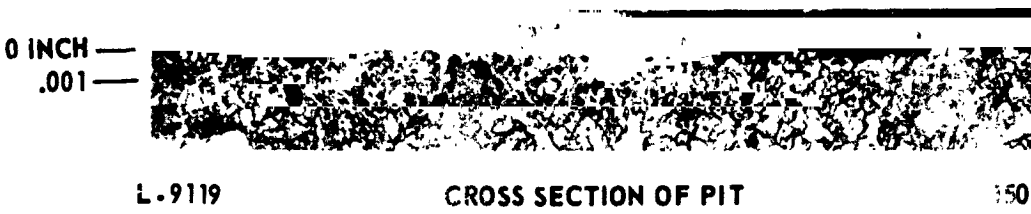
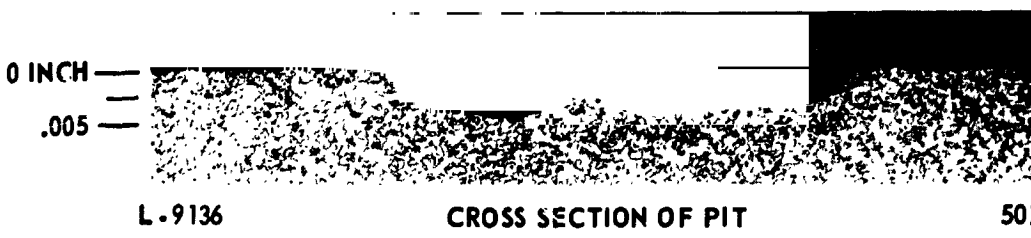
2-1.2X

**CRACKS AT 5-1/2 IN. RADIUS  
TUBE BEND**

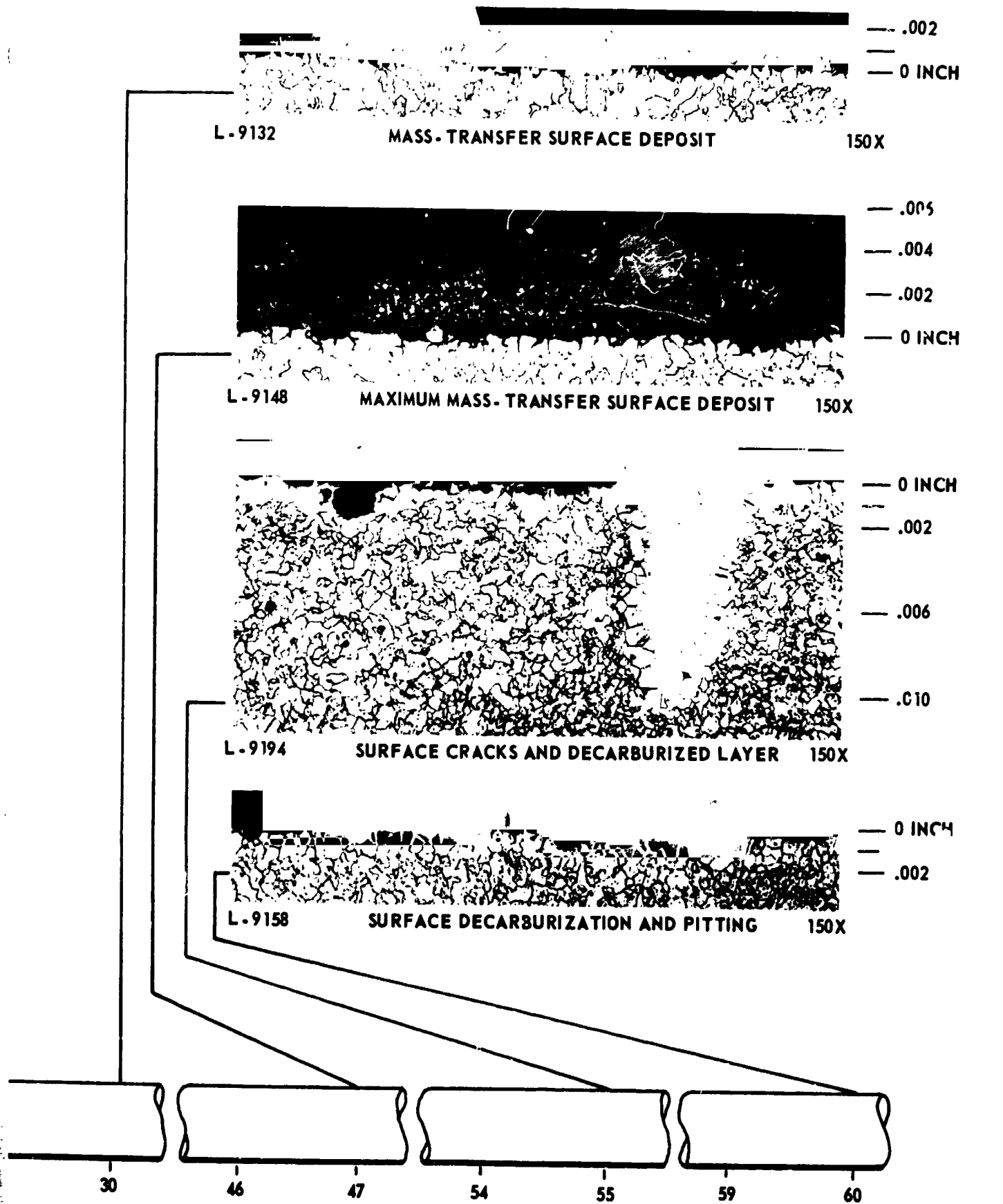


**TUBE  
SURFACE  
CRACKS**

Figure 13.3



Mercury Exposed Surfaces of Tub  
Boiler After 1415 hr of Operat



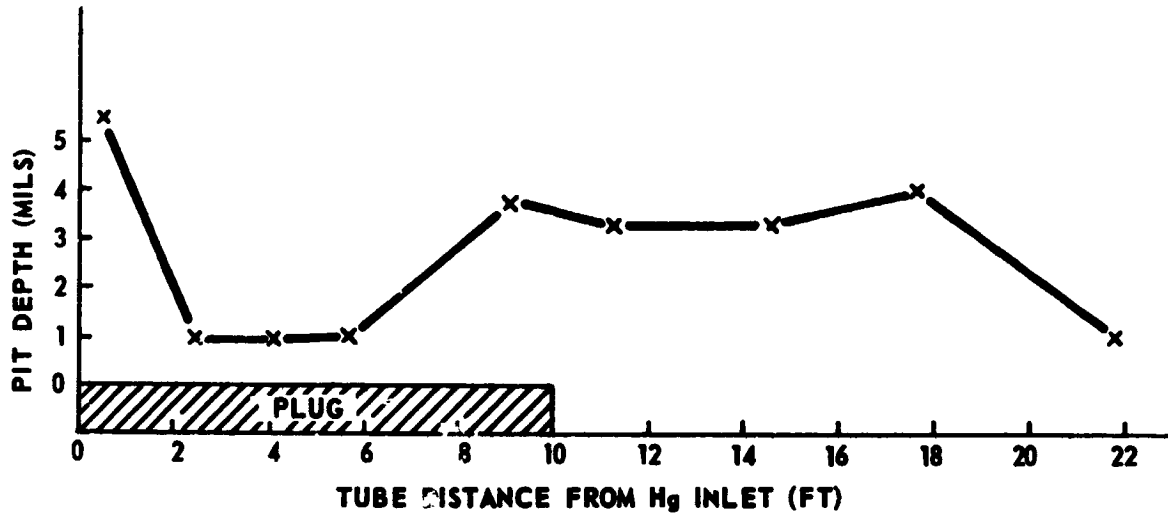
DM H<sub>g</sub> INLET (FT)

Coil No. O-4 Removed from -1  
ion in RPL-2 (Vilella's Etch)

Figure 14 - 2

THIS END CONNECTS  
TO 5- 1/2 INCH RADIUS  
BEND AT H<sub>g</sub> OUTLET  
MANIFOLD





Distribution of Pit Depth of Mercury-Exposed Surface of Tube Coil  
No. 0-4 Removed from the -1 Boiler After 1415 hr of Operation in RPL-2

Figure 15

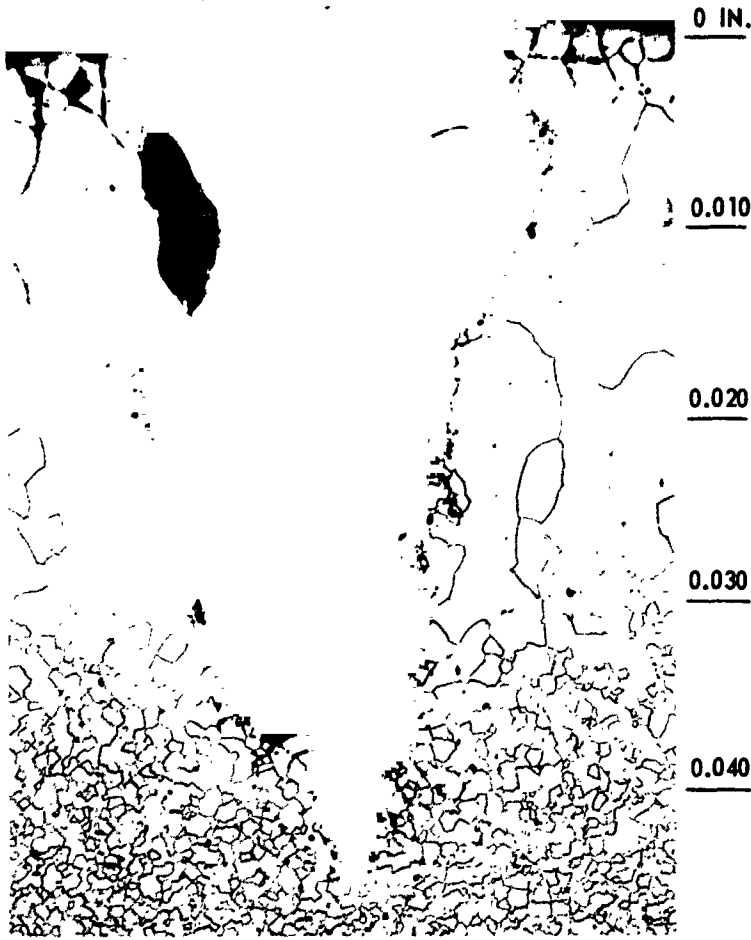


a. DURING BOILER FABRICATION - X-RAY DIRECTION IN PLANE OF COIL



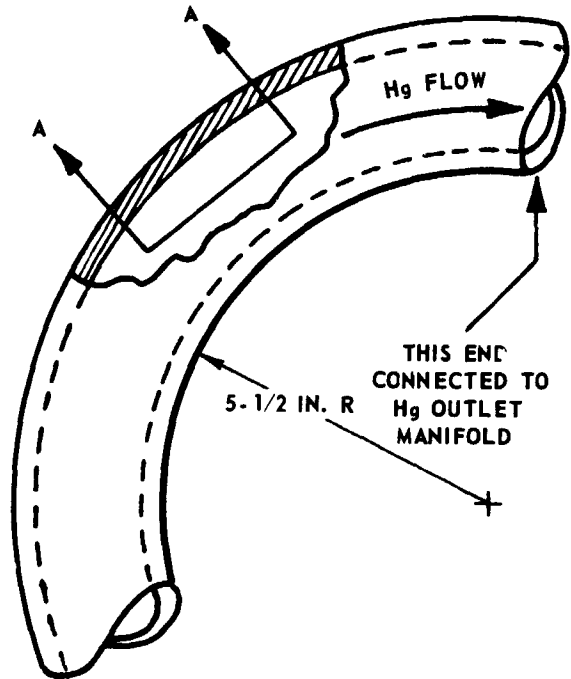
b. AFTER BOILER OPERATION  
X-RAY DIRECTION TRANSVERSE  
TO PLANE OF COIL

X-ray of Twisted Ribbon in Mercury Containment Tube of -1 Boiler  
(P/N 092020-1F, S/N A-1) Boiler Removed from RPL-2 After  
1415 Hr of Mercury-Side Operation

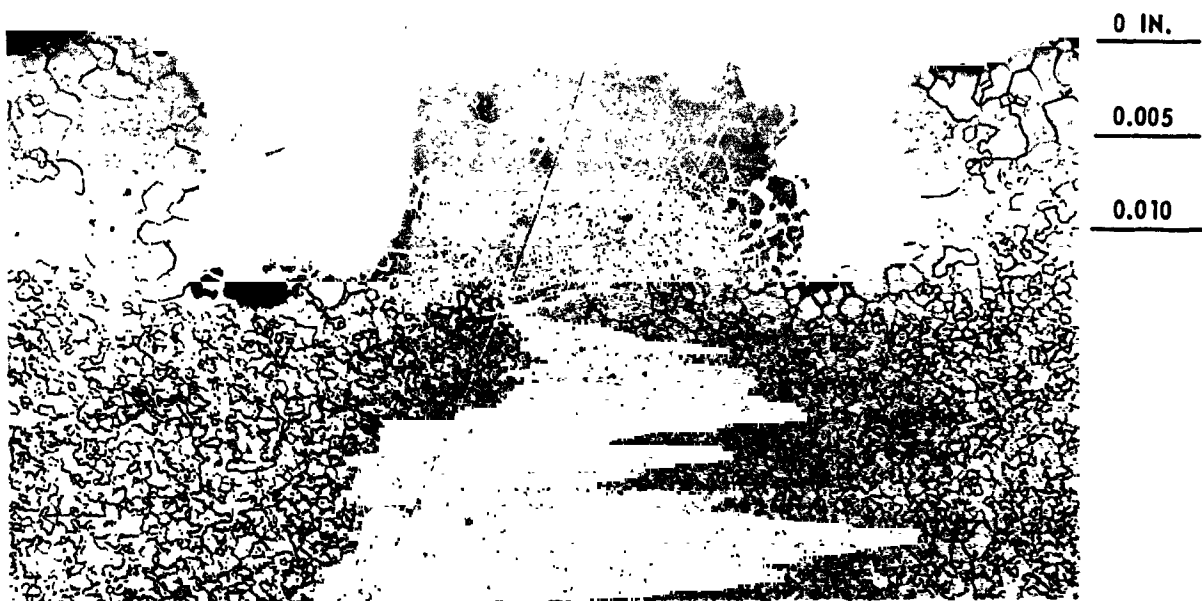


ETCHANT: VILLELA'S L-8952 100 X

C. MAXIMUM EXTERNAL SURFACE (NaK SIDE) CRACK DEPTH AT SECTION A-A



A. SCHEMATIC DEFINING EVALUATED AREA



ETCHANT: VILLELA'S L-8954 100 X

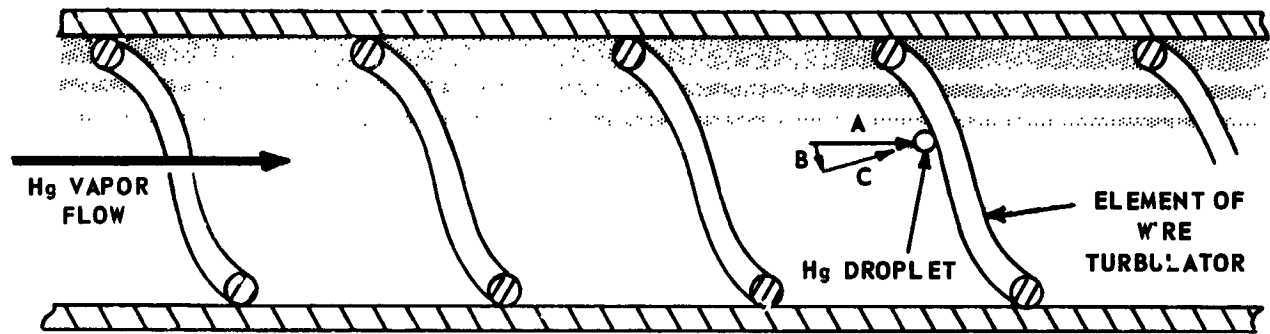
B. INTERNAL SURFACE (Hg SIDE) CRACKS AT SECTION A-A

Surface Cracks at the 5-1/2-in. Radius Bend at the Hg Outlet Manifold in -1 Boiler Removed from RPL-2. Hg Side Operated 1415 hr; NaK Side Operated 2350 hr.

Figure 17

A266-NF-1150A

966-NF-1205

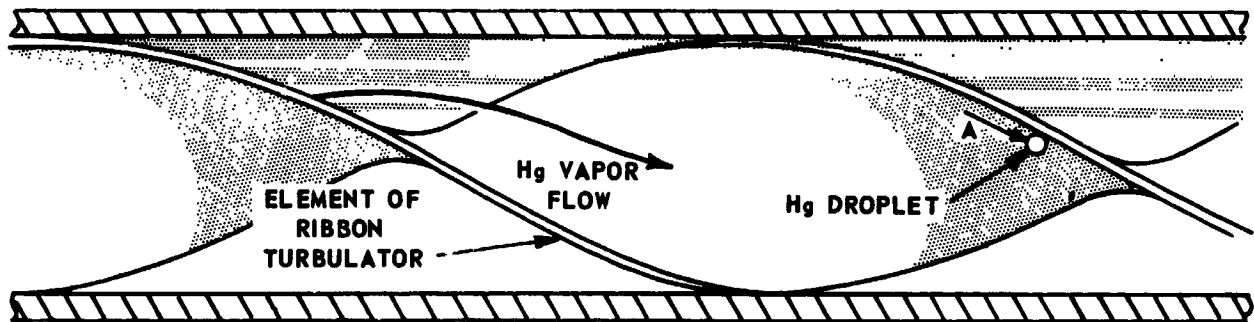


A. WIRE TURBULATOR

VECTOR A IS FORCE EXERTED ON Hg DROPLET BY FLOWING VAPOR

VECTOR B IS FORCE COMPONENT APPLYING HELICAL MOVEMENT OF Hg DROPLET ALONG WIRE

VECTOR C IS FORCE COMPONENT PUSHING Hg DROPLET AGAINST WIRE AND RESULTING IN NO AXIAL MOVEMENT OF DROP ALONG TUBE UNLESS IT IS LARGE ENOUGH FOR SOME PORTION TO ROLL OVER TOP OF WIRE



B. RIBBON TURBULATOR

VECTOR A IS FORCE EXERTED ON Hg DROPLET BY FLOWING VAPOR

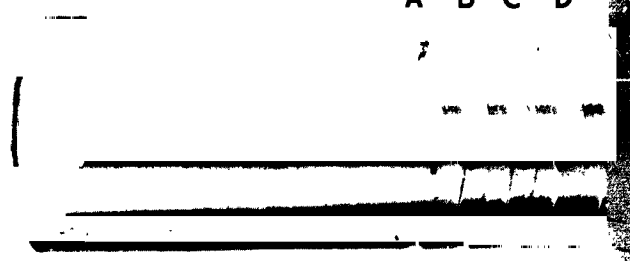
ALL OF FORCE COMPONENT APPLIES HELICAL MOVEMENT TO Hg DROPLET ALONG RIBBON. THEREFORE THERE IS LESS TENDENCY OF DROPLET TO "HANG" UP WHEN COMPARED TO CASE ABOVE.

"Plan Cutaway View of Hg Boiler Tube Showing Force Applied by Hg Vapor Flow in Moving Liquid Hg Droplet Axially Along Tube. Two Cases Considered: Tubes With Wire and Ribbon Turbulators"

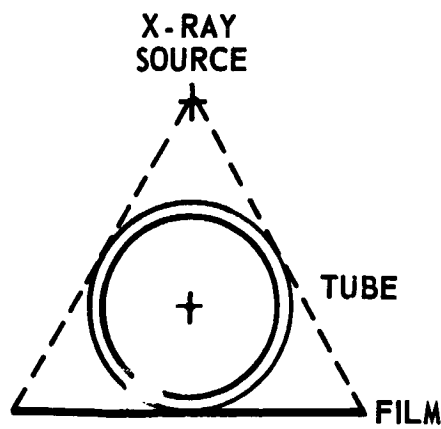
Figure 17a



MACHINED NOTCH  
A B C D



TUBE SPECIMEN (FULL)  
9Cr-1Mo STEEL (.816 OD x .0



CURV  
AGAIN

DOUBLE TUBE WALL POSITION

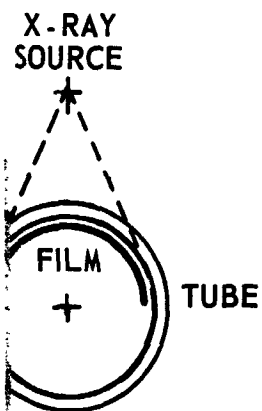
In Situ X-ray Evaluation of Tub  
Hg-Containment-Tube Cracking -

ES

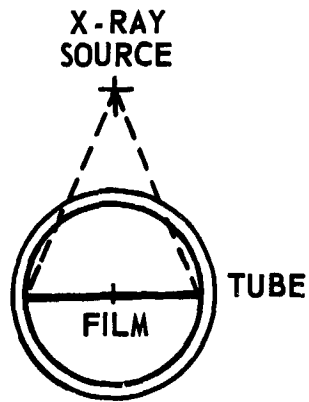
NOTCH DEPTH

A	.010
B	.008
C	.006
D	.004

(SIZE)  
(.79 WALL)



ED FILM HELD  
T INSIDE WALL



FLAT FILM  
THROUGH TUBE CENTER

SINGLE TUBE WALL POSITION

Re-in-Tube Boiler for Incipient  
X-ray Film Position Evaluation