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TITLE: Evaluation of SNAP 8 Bare Refractory Double Containment Boiler No. 2 After 8700 Hours of Operation

ABSTRACT

The Hg inlet and outlet, and one tantalum tube from the SNAP-8 bare refractory double containment boiler No. 2 were metallurgically evaluated after 8700 hours of operation in PCS-1. The evaluation indicated that with the correction of some deficiencies, a five-year operational life for boilers of this type is attainable. Inherent corrosive or erosive attack of the construction materials by the NaK or the mercury was not detected. Therefore, within the specified requirements corrosion is not life limiting. The materials used in this boiler are suitable for future boiler fabrication. Minor surface defects in the tantalum, produced during fabrication, exhibited no tendency to initiate failure. Hg mass transfer products generated in non-boiler areas of the PCS-1 system, deposited in the boiler and alloyed with the surface of the Ta tubes forming intermetallic compounds but did not diffuse into the Ta. Since these deposits were wetted by Hg, they were judged not to have adversely affected boiler operation, neither did they affect the mechanical properties of the Ta. The deficiencies in the boiler, partial debonding of a Ta/316SS transition joint, failure of a Ta-10W swirl wire, and cracking of a 321SS static NaK tube can be eliminated by design, fabrication or procedural modifications. A decreased boiler pressure drop which occurred with increased operating time was attributed to organic contamination of the I.D. of the Ta tubes. Modifications of system operational procedures can eliminate or minimize this condition. The tantalum weld grain size was large, but no indications of imminent failure were detected. For optimum low cycle fatigue life, welding techniques to produce finer grained Ta weldments should be developed.

KEY WORDS: BRDC Boiler, Tantalum, Hg corrosion/mass transfer, NaK corrosion, stainless steel, (316 and 321), Ta/316 transition joint, Ta-10W

APPROVED:

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NOTE: The information in this document is subject to revision as analysis progresses and additional data are acquired.

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I. SUMMARY

Sections of the SNAP-8 bare refractory double containment boiler No. 2 were metallurgically evaluated after 8700 hours of operation in PCS-1. The Ta showed no evidence of Hg corrosion or erosion, and minor surface defects produced during fabrication and repair of the boiler exhibited no indications of propagation during operation. The Ta welds were generally excellent in quality, but the grain size was so large that the full wall thickness included only one grain in the full cross section in some locations. It is anticpated that this condition would reduce the low cycle fatigue life of the material. Hg mass transfer products, deposited in the boiler during operation, exhibited a pattern along the boiler length which correlated well with the changing Hg conditions of temperature and quality in the boiler during system operation. The mass transfer products formed intermetallic compounds with the Ta on the Hg exposed Ta tube surface but did not diffuse into the Ta and therefore had no effect on the properties of the Ta.

Several deficiencies in the boiler were also detected. Cracking of a 321SS static NaK tube was attributed to thermal cyclic-rupture. Wall thinning of the Ta tube under the crack was attributed to erosion by NaK flowing through the crack at a low volume, but a high velocity, due to periodic pressure differentials between the primary and static NaK loop. Some debonding of the Ta/316 transition joint at the Hg inlet occurred, which is a typical failure mode for this type joint. The failure of one of the Ta-10W swirl wires was attributed to hydrogen embrittlement combined with residual stresses introduced during fabrication of the boiler. The presence of organic products at the boiler inlet produced or contributed to the decreased pressure drop which occurred across the Hg side of the boiler during its operational life. All of the deficiencies are readily correctable by design, processing and/or operational modifications. With correction of the deficiencies, a five-year operational life for BRDC type boilers appears attainable.

II. CONCLUSIONS

1. A reliable five-year life for BRDC type boilers is attainable, with the incorporation of readily attainable design, processing and operational modifications.

2. The materials used in this boiler (tantalum, tantalum-10 tungsten 316SS, 321SS, zirconium) are acceptable for long duration operation in future boiler designs employing NaK and Hg as the heat exchanger fluids.

3. No detectable Hg corrosion or erosion of the Ta occurred in 8700 hours of boiler operation, thus confirming the selection of Ta as the Hg containment material.

4. General NaK corrosion of the Ta did not occur but NaK removal of oxides from the heat affected zone of two welds with resultant void formation occurred. The presence of the original oxide was the result of insufficiently pure protective inert atmosphere during the welding operation. This indicates that the static NaK concept is effective and that NaK corrosion of the Ta is not life limiting.

5. Minor defects such as fabrication tears and subsurface oxidation paths on the surface of the Ta did not propagate during boiler operation and exhibited no adverse effects on boiler performance.

6. Although minor oxygen contamination of the tantalum in the PCS-1 system may have occurred, the mechanical properties changed so little that no problem in achieving 100 system restarts is foreseen.

7. The decreased boiler pressure drop with increased operating time is at least partially attributable to organic contamination of the inside surfaces of the Ta Hg containment tubes.

8. The debonding of the hot co-extruded Ta/316SS transition joint at the Hg inlet was typical of the failure mode previously demonstrated by laboratory testing for this type of joint manufactured with the same fabrication parameters.

9. Hydrogen embrittlement, combined with residual assembly stresses caused the failure of the Ta-10W swirl wire.

10. The static NaK tube failure was caused by excessive stress, inherent in this specific boiler design resulting from thermal excursions during system startup and shutdown, and retained stresses at steady state resulting in a failure phenomenon known as cyclic-rupture.

11. Localized wall thinning of the Ta tube under the failed static NaK tube was caused by erosion due to NaK impingement from the crack in the static NaK tube.

12. The zirconium foil served its intended function of purifying the static NaK by absorbing oxygen, nitrogen, hydrogen and carbon.

13. Oxidation of the O.D. of the Ta Hg inlet dome and header occurred during a boiler repair weld operation. During subsequent boiler operation the NaK leached the oxides from the Ta thus leaving voids. This same condition was present to a minor extent at one of the original Ta tube-tube welds. These defects did not propagate and had no adverse effect on the boiler operation.

14. Mass transfer products generated in non-boiler areas of the Hg system deposited in the boiler in a pattern consistent with the condition of the Hg. At the boiler inlet the liquid Hg was initially heated causing an increase in the terminal solubility of the solute elements so that precipitation did not occur. As the quality of the Hg increased further into the boiler, less liquid remained to retain the solute elements. This resulted in saturation of the Hg, precipitation of solute, and deposition on the tube wall. The deposition reached a maximum approximately 15 feet from the inlet where the calculated quality was 98 percent. These products alloyed with the Ta to form intermetallic compounds on the I.D. of the Ta tubes but did not diffuse into the Ta or affect the mechanical properties of the Ta.

15. The presence of only one to three grains across Ta welds was typical within the BRDC #2 boiler.

16. Swaging of the tube over the plug at the Hg inlet to the boiler produced intimate contact between the tube and the plug lands. The tight fit was maintained during boiler operation, verifying the adequacy of the swaging operation for the production of inlet plug sections for boilers.

17. Scoring of the O.D. of the Ta tube was the result of its rubbing against the 321SS static NaK containment tube during thermal cycling caused by the difference in thermal expansion coefficients of the two materials. The maximum depth of affected Ta was 0.003 inches and the defects did not tend to propagate from the tube wall into the tube.

18. As operated in the PCS-1 system, the 321SS static NaK tube had more sigma phase formation than did the 316SS shell material. Both materials exhibited a 0.003 to 0.004 inch zone on the flowing NaK side which did not contain sigma phase. The absence of sigma in these zones was attributed to the leaching or deposition of interstitial elements on the tube surfaces by the flowing NaK. This effect was not present on the static NaK side of the 321SS tube or on the air exposed side of the 316SS shell material. Both materials are satisfactory for boiler use.

III. RECOMMENDATIONS

1. Design modifications should be made to reduce the stress level in the static NaK tube at the Hg inlet and in other areas where stress analysis or previous performance indicates cyclic overstress failure is probable.

2. Optimization of the fabrication techniques for the production of reliable Ta/316SS transition joints should be continued and the resultant best products tested to establish their acceptability for SNAP-8 boiler use.

3. PCS-1 operational modifications and improvements should be made to eliminate organic contamination of the Hg loop. These should include liquid nitrogen and molecular sieve traps between vacuum pumping stations and the Hg loop.

4. The chemical cleaning procedure, AGC 10319/8, should not be used for boilers containing Ta-10W turbulator wire. An alternate material, not subject to hydrogen embrittlement when so cleaned, should be considered or a modified cleaning procedure should be developed for removing heat transfer inhibiting surface films from partially deconditioned Ta boiler tubes.

5. Although no indications of initiation of failure of the Ta in the BRDC #2 boiler were detected, future boilers should be constructed of recrystallized rather than cold worked Ta to provide greater ductility of the material and to reduce the possibility of failure due to low cycle fatigue and hydrogen or oxygen embrittlement.

6. Ta welding techniques to produce finer grain size weldments should be developed for the boiler application.

IV. INTRODUCTION

The SNAP-8 bare refractory double containment boiler No. 2, P/N CF751840, initiated operation in PCS-1 in March of 1968. After 1313 hours of operation, the boiler was repaired following an Hg outlet bellows failure, Reference (a). After approximately 4000 hours of operation, a PNL to static NaK leak was detected, Reference (b), but operation was continued since the leak did not affect the overall performance of the boiler. After 6138 hours of operation, a NaK to air leak occurred in the 316SS boiler shell at the NaK outlet, Reference (c). This was repaired by welding a doubler on the shell over the leaking area and operation was continued. In September of 1969, after a total of 8700 hours of Hg operation which included a total of 28 startup-shutdown cycles, the boiler was removed from PCS-1. During its operational life this boiler had produced Hg vapor for turbine operation in an acceptable manner. The performance of the boiler did degrade during its life as indicated by a decreased Hg side pressure drop. This was attributed to Hg loop contamination and not to the boiler design. After the boiler was removed from the PCS-1 system, the Hg inlet, outlet and one Ta tube were cut from the boiler for metallurgical evaluation. This memorandum reports the results of that evaluation.

V. Ta TUBE

A. VISUAL EXAMINATION

Visual examination of the O.D. of the Ta tube which had been exposed to the static NaK during boiler operation and subsequently the decontaminating solutions after removal from the PCS-1 system, revealed the presence of a black surface at the NaK outlet, Figure 1. This coloration gradually faded, and from approximately tweleve feet from the NaK outlet to the NaK inlet the tube was spottily discolored in a manner indicating probable contact with the 321SS static NaK containment tube. Except for the localized wall thinning of the Ta tube under the crack in the static NaK tube, no visual evidence of corrosion of the Ta tube was detected. However, some scoring of the Ta tube O.D. was present, Figure 2, presumably due to contact with the static NaK containment tube and relative movement between the two tubes due to differential thermal expansion

during heating and cooling of the boiler. Although the scoring was general for the full length of the Ta tube, it was most severe at the end of the straight inlet section. Five Ta tube-tube welds were present in the full boiler length. All of these visually appeared to be excellent with no apparent effect from the 8700 hours of operation.

Visual examination of the I.D. of the Ta tube revealed a dark surface discoloration at the Hg inlet. In the plug section, the tube area in contact with the plug lands was silvery appearing, indicating good contact with the plug, and the area between the lands had the dark discoloration, Figure 3. This discoloration faded and the tube had a silvery metallic appearance from about nine feet from the inlet to the outlet, Figure 1. There was no visual evidence detected which provided an indication of the Hg liquid-vapor interface. That is, there was no mass transfer pileup, wall thinning or sudden change in coloration visible. Visually, mass transfer deposits on the tube I.D. were not apparent. They were subsequently detected by metallographic examination and X-ray fluorescence analysis. The visual examination indicated that the retained Hg was physically wetting the tube wall and was an indication that the mass transfer deposits present were also wetted by the Hg. No evidence of corrosion, erosion or mechanical damage were visually detected on the I.D. of the Ta tube.

B. RESIDUE ON STATIC NAK SIDE

At the Hg inlet end, it was possible to scrape a small quantity of a black powdery residue from the O.D. of the Ta tube. From a three foot length of tube, 5.6 mg of non-magnetic material was collected. Semi-quantitative emission spectrographic, combustion-gas-chromatographic, atomic absorption and X-ray diffraction analyses were run on the residue. The results of these analyses are listed in Table I. The X-ray diffraction analysis indicated that the material was at least partly crystalline but the pattern could not be matched with existing ASTM powder diffraction patterns. All of the diffraction lines for Ta were present indicating that some of the tube material was scraped off with the residue. The large amount of Na and K present indicates the residue contained NaK compounds. The carbon may have been present in the form of NaK carbonates formed subsequent to removal of the boiler from the system when air exposure and resultant absorption

of CO_2 occurred. The presence of vanadium was surprising since alloys containing this element were not employed in the static NaK system. It is possible that the presence of this element may be explained by its being present in residual amounts in the primary NaK loop as a result of previous PCS-1 operation with boilers containing vanadium modified 9Cr-1Mo Hg containment tubes, this material could have passed from the PNL to the static NaK area through the crack in the 321SS static NaK containment tube during BRDC #2 boiler operation. The amount of residue present was very small but being concentrated at the Hg inlet end of the Ta tubes, it may have had a minor effect in decreasing the heat transfer to the Hg in this area.

The PNL NaK had contained 225 PPM Hg during boiler operation. A chemical spot test, capable of detecting 0.3×10^{-9} grams Hg per cm^2 of tube wall, was performed on the OD of the Ta tube at locations 4, 18 and 36 feet from the Hg inlet. No Hg was detected. This indicates the probability that the Hg did not contribute to the surface discoloration of the tube or the small amount of residue found at the Hg inlet end of the tube.

C. METALLOGRAPHIC EXAMINATION

Metallographic examination of the Ta tube at the end of the straight section where the most severe scoring was visually observed, indicated that the scoring extended below the original tube contour to a maximum of approximately 0.0008 inches. However, the cold flow of the Ta had caused rises at the edges of the scores up to approximately 0.0022 inches. This made the scoring appear visually to be more severe than it was. There was no indication that the scoring caused any initiation of cracking of the tube wall. There was also material galled into the Ta, Figure 2. This presumably was 321SS from the static NaK containment tube since mass transfer products were not detected metallographically on the Ta tube O.D.

No evidence of corrosion of the Ta tube O.D. remote from weld areas was detected metallographically. This indicates that the basic design concept is excellent from a NaK corrosion standpoint. Except for the wall thinning of the Ta tube which occurred under the crack in the 321SS static NaK containment tube and which was presumably caused by erosion, there was no indication that NaK corrosion of the Ta tube would be life limiting.

The metallographic examination revealed that Ta tubing in two different conditions was used in the fabrication of the boiler. The tubing in the straight inlet plug section and the last eight inches at the Hg outlet was recrystallized, but the tubing from the tube-to-tube weld at the end of the plug section to the tube-tube weld near the Hg outlet of the boiler was in the cold-worked condition, Figure 4. From the elongated grain structure produced by the cold-working, it was estimated that the Ta was cold-worked approximately fifty percent. The cold-working increased the hardness of the Ta tube as revealed by the microhardness traverses performed, Table II. The average Knoop hardness of the cold-worked material was 147 KHN (130-158 KHN range) and of the unswaged recrystallized tubing at the inlet was 108 KHN (102-113 KHN range). The only area of the parent metal tubing which exhibited a true hardness gradient was the inlet end where it had been swaged over the plug. The hardness in this area was as high as 296 KHN at the surface with an average cross sectional hardness of 151 KHN. This hardness gradient was produced by the cold-work introduced into the recrystallized Ta by the swaging operation. The cold-worked tubing comprising most of the length of the boiler tube did exhibit a variation in hardness across the wall thickness, but the pattern was not uniform and could not be interpreted as a through-wall gradient. In general, the O.D. did tend to be slightly harder than the I.D., but this was not true at all locations tested. In some areas, the center of the wall thickness was harder than either the I.D. or the O.D. If oxygen contamination of the Hg loop had occurred, a decreasing hardness gradient from the I.D. to the O.D. of the Ta tube wall would be anticipated. Therefore, it is presumed that the Ta wall hardness variation was due primarily to the previous cold-work history of the Ta tube. Cold-worked Ta exhibits lower ductility than recrystallized Ta; the ductility decreasing with increasing amounts of cold reduction. Although mechanical tests were not performed to determine actual values, the cold-worked Ta in this boiler would exhibit lower ductility than if it had been recrystallized. Therefore, an undetermined but shorter low cycle fatigue life would be anticipated. No indications of cyclic stress failure were detected in the boiler tube, but for optimum resistance to the low cycle fatigue failure, future boilers should be fabricated from recrystallized Ta. No evidence of Hg

corrosion of the Ta was detected by metallographic examination of various sections from the Hg inlet to the outlet end of the boiler. In the plug section, the tube I.D. was expanded in the areas where it had been swaged over the lands of the plug, Figure 5. Intimate contact between the tube and the plug lands had been achieved by the swaging operation and maintained through the 8700 hours of operation of this boiler verifying the suitability of the inlet plug section fabrication procedure.

D. Ta TUBE-TUBE WELDS

Five Ta tube-tube welds were present in the tantalum boiler tube evaluated. Metallographic examination of each of these welds was performed. The weld metal grain sizes were so large that the full wall thickness consisted of two to three grains. This is an undesirable condition from the standpoint of potential low cycle fatigue failure, Reference (d), and from the standpoint of inadvertent but possible oxide contamination during fabrication or elevated temperature operation. Oxygen introduced at the surface of Ta, such as could occur if an air leak into the hot system developed or if the welding atmosphere deteriorated after welding but before the material cooled, tends to penetrate more deeply into the material along grain boundaries than through the grains. With a few large grains, the oxide penetration at the grain boundaries would be deeper into the wall thickness than for many small grains which provide a more devious and longer path through the wall. Although no apparent difficulty occurred with this boiler due to the large grain size weld metal, it is desirable to develop a welding technique which will produce finer grained weldments to improve the reliability of the boiler.

The welds were metallographically defect free, but the O.D. of the tube-tube weld heat affected zone located 12.5 feet from the boiler inlet showed evidence that the welding atmosphere had not been sufficiently pure to prevent oxygen pickup as it had with the other welds. The heat affected zone of this weld showed the effect of NaK leaching of oxygen from preferential planes and grain boundaries to a depth of 0.0008 inches into the tube wall from the O.D., Figure 6. There was no metallographic evidence that these areas tended to cause cracking of the Ta tube.

Microhardnesses of the weld metal in each tube-tube weld were determined, Table III. In four of the welds the O.D. and I.D. were harder than the core of the wall thickness. In the fifth weld very little hardness gradient existed and the surfaces were slightly softer than the core. Although no chemical anayses were performed on the welds, it is presumed that the variations in hardness are due to interstitial contamination, possibly nitrogen, during the welding operation because hardness gradients were not generally detected in the parent metal tube wall remote from the welds. This indicates that the hardness gradients are not an operationally produced effect.

E. INTERSTITIAL ELEMENTS ANALYSES

Vacuum fusion and conductometric carbon analyses were performed on full cross sections of the Ta tube wall from three locations, the Hg inlet and outlet and the center of the tube length, Table IV. It was not possible to correlate the amount of interstitial elements present with the operating history since the original analysis of the tubing was not available. The small amount of hydrogen present may be the result of the aqueous decontamination of the static NaK side of the boiler after removal from the system. Its concentration is so low that no noticeable effect on the mechanical properties would be anticipated, but if the boiler had been restarted it is anticipated that hydrogen would have been outgassed at elevated temperatures. The presence of higher oxygen contents at the inlet and outlet than at the center of the tube could be the result of Hg side contamination from the vacuum atmosphere employed during the system startup sequence prior to Hg injection. Any air inleakage into the Hg system would have occurred outside the boiler and the oxygen would then diffuse through the system, entering the boiler through the Hg inlet and outlet. As the oxygen passed into the boiler its partial pressure would decrease due to the gettering action of the Ta it previously passed. Thus, it would be anticipated that the inlet and outlet Ta would contain higher concentrations of oxygen than the center of the tube. Although the presence of the oxygen would increase the hardness and strength and reduce the ductility slightly, the amount present was well below the 500 ppm maximum established on the basis of low cycle fatigue failure in 100 thermal cycles, Reference (e). If it is assumed that the original concentration of oxygen in the Ta at the outlet was 40 ppm and that the increase in oxygen level

resulted from contamination during exposure to the vacuum atmosphere during system start-ups and shutdowns, a total of 100 such cycles could be performed before reaching the 500 ppm limit of oxygen level. It does not appear therefore that interstitial element contamination of the Ta as the boiler was operated in PCS-1, would limit the life of the boiler to less than the required 100 thermal cycles.

F. DEPOSITS ON I.D. OF Ta TUBING

No deposits were detected metallographically on the I.D. of the Ta tube in the straight Hg inlet section. However, infrared spectrographic analysis of solvent washings from this area revealed the presence of aliphatic hydrocarbons, and analysis of scrapings from the tube revealed the presence of small amounts of Fe, Ni and C. It is presumed that the carbon is the result of thermal decomposition of organics which entered the Hg system; mix 4P3E, octoil, and duoseal oil have frequently been detected in small quantities in various locations in the Hg system. The presence of organic material at the Hg inlet indicates that the decreasing pressure drop across the boiler was contributed to significantly by organic contamination.

From the end of the Hg inlet plug straight section to the Hg outlet, however, metallic deposits were observed, Figure 7. The circumferential pattern of deposition was not well defined by the examination. In general, there was a thin layer of deposits directly on the I.D. of the Ta tube, Figures 7 and 8. This appeared to be a diffusion zone. There also were random deposits which bore a similarity to, but were not truly characteristic of, dendritic growth patterns. These deposits were up to 0.002 inches thick in some locations but undetectable in others on the same transverse tube section. Electron microprobe analyses revealed that these areas were primarily Ta with localized areas containing Ni, Fe, Cr and Co. The composition pattern of these particles and the tube surface are illustrated in the radiation displays, Figures 9 and 10. These displays are from two areas of the same transverse tube section fifteen feet from the Hg inlet. Also, the relative concentration of Ni is illustrated in Figure 11 which represents four line traces from the specimen metallurgical mounting material on the I.D. into the Ta tube wall at different circumferential locations on the same transverse cross section. These deposits could have been burrs produced during metallographic sectioning; however, this is not likely since they were not present on the

recrystallized inlet samples of tubing or at the welds. Repeated grinding down and repolishing of metallographic samples did not eliminate the deposits. It is presumed, therefore, that this I.D. surface condition was produced during the tube drawing operation. Since these particles were attached to the tube wall only in localized areas, they were closer to the temperature of the Hg stream than was the hotter main tube wall during boiler operation. This could account for the absence of the uniform surface deposit layer on the protrusions where due to the lower temperature a significant degree of Hg boiling heat transfer did not occur. At the higher temperature tube wall, surface boiling and resultant depostion did occur. The presence of these particles and deposits does not appear to have adversely affected the Ta boiler tubing.

The uniform diffusion zone deposits on the tube wall were friable as indicated by the metallographically observed cracks and by attempts to determine their hardness. The deposits tended to crack and break apart when penetrated by the diamond during microhardness testing. The best determination that could be made indicated the deposit had a hardness of 363 KHN, considerably harder than the Ta tube and indicative of lower ductility. There appeared to be no diffusion of the elements of the mass transfer deposits into the Ta beyond the visible deposit layer. This was indicated by the absence of a hardness gradient in the Ta below the deposits as well as by the absence of the deposit elements in the tantalum below the deposit/Ta interface as indicated by microprobe analysis.

A pattern of mass transfer product deposition was present along the length of the Ta tube from the Hg inlet to the outlet. This was observed by a difference in the X-ray fluorescence analyses of flattened tube I.D. surfaces at various locations along the length of the tube, Figure 12. The fluorescence analyses indicated not only a difference in thickness of the deposits but also a change in composition at the various locations. These analyses correlate well with the condition of the Hg during boiler operation. In the plug region of the boiler where the cold incoming Hg was being heated and boiling initiated, no deposits were detected metallographically. By scraping the I.D. surface of the straight Hg inlet tube, and thus providing a concentrated sample the presence of small quantities of Fe, Ni, and C was established. As the quality of the Hg increased farther into the boiler, the amount of deposits present increased until

a maximum was reached at approximately fifteen feet from the inlet where the calculated quality reached 98 percent. From that point to approximately twenty-four feet from the Hg inlet, the amount of deposits present decreased. The amount of deposits then increased relatively rapidly within about one foot and then increased more gradually toward the boiler outlet. The X-ray fluorescence analysis indicated the presence of Fe, Cr, Ni, Co and Ta in all of the deposits. Ag was detected at only one location, 15 feet from the inlet, where the calculated quality was 98 percent.

This axial pattern of deposition can be explained on a basis of elemental solubility in Hg and the change in condition of the Hg as it passes through the boiler. The Hg entering the boiler inlet at approximately 500°F had passed from the condenser through the liquid lines, Hg PMA, isolation valve, flow control valve, and TSE valves. During this flow period, elements in the Hg in excess of the solubility limit at 500°F had precipitated and deposited in the liquid region, probably primarily in the valves where the most turbulence occurs. As the Hg was heated as it entered the boiler, the solubility of the elements in it increased and no precipitation occurred. However, as boiling started the amount of liquid Hg present decreased and the elements present in solution reached saturation and precipitation commenced. When the quality approached 100 percent, only the Hg liquid carry-over was available for the elements to remain dissolved in and essentially all of the precipitation was completed. Some of the solid precipitates did not attach themselves to the tube wall but were carried on by the vapor stream. Some of these particles deposited downstream in the boiler and a few were probably carried out of the boiler and into the vapor line. The relatively small but rapid increase in the amount of deposits between 24 and 25 feet from the inlet is attributed to the flattening of the temperature profile from that point to the Hg outlet. Up to approximately twenty-four feet into the boiler either the Hg temperature is increasing and/or the quality is increasing. Beyond twenty-four feet the temperature increases only slightly so that the elemental solubility in the liquid carryover increases only slightly. However, the size of the carryover droplets decreases due to Hg evaporation so that less material is soluble in the droplets and precipitation from them recommences. Therefore, it is assumed that relatively sudden dip

in the amount of deposits present at 24 feet from the Hg inlet is the result of the relatively sudden decrease in temperature rise rate at about that point and the reinitiation of precipitation due to a decrease in the amount of liquid Hg present.

A goniometer X-ray diffraction trace was made on the I.D. of the flattened Ta tube fifteen feet from the Hg inlet where the most deposit was indicated by the X-ray fluorescence analysis. Many diffraction peaks were obtained and the results could be intepreted as indicating the presence of Cr_2Ta, $TaNi_3$ and Fe_7Ta_3. The diffraction peaks for Cr, Ni, Fe and Co were not present. This indicates that alloying of the mass transfer products with the Ta occurred and resulted in a layer of inermetallic compounds on the surface of the Ta. The formation of this deposit does not appear to be boiler life limiting since it does not appear to have affected the mechanical properties of the Ta tube material. It may have an effect on heat transfer into the Hg in the areas where it is present but this would probably be minimal since the thickness is small and the deposit does appear to have been wetted by the Hg. It is also possible that since the deposits are friable they could break loose from the tube and be carried along with the Hg vapor stream. Based on the absence of Ta in residues obtained from the Hg liquid portion of the PCS-1 system, however, this does not appear to have occurred to any significant extent in the 8700 hours of boiler operation.

G. Ta TUBE AT Hg INLET HEADER

Where the Ta tube exited from the inlet header on the upstream side, smooth slight depressions were present to a depth of 0.0004 to 0.0015 inches into the tube. This indicates that tube bending stresses at the header during boiler operation did not deleteriously affect the Ta tubes. There was no indication of incipient failure of the tube at this location.

H. Ta TUBE WALL THINNING UNDER STATIC NaK TUBE CRACK

Although the failed section of the 321SS static NaK tube could have been replaced, the Ta Hg containment tube immediately under the crack had suffered localized wall thinning. Metallographic examination revealed that as much as 0.0185 inch of the 0.040 inch thick wall had been removed on the O.D. of the Ta

tube immediately under the crack in the static NaK containment tube. The reason for this localized wall thinning is not clear. Metallographically the thinning was smooth rather than rough, no differences in hardness or structure were visible in the thinned area as opposed to unthinned areas. No evidence of subsurface oxidation was present in any location on the Ta tubes. It is thought possible that short term, low temperature surface oxidation of the Ta may have occurred due to oxygen entering the static NaK system through the cracked 321SS tube during the time between the failure and repair of the boiler shell. The oxygen would have to be supplied from the PNL where the NaK/air leak occurred. The possibility of this occurring was minimized by keeping both the PNL and static NaK system under argon pressure after the failure occurred until both systems were evacuated to be refilled with NaK. The argon used did contain some oxygen. Localized formation of sodium tantalate on the Ta tube under the crack and subsequent solution or erosion by the NaK flowing through the crack of this material or of the Ta itself may have produced the wall thinning. Erosion is considered the probable cause of the Ta tube wall thinning. The PNL NaK flow rate at the O.D. of the static NaK tube was four ft/sec. It was estimated that during periods when a pressure differential existed between the PNL and static NaK systems, the NaK flow rate through the crack reached 17 ft/sec. This would also be the approximate impingement velocity on the Ta tube O.D.

VI. Ta DOME AT Hg INLET

Metallographic examination of the Ta dome section joining the Ta/316 transition joint to the Hg tube header was performed. A Ta weld had been made in this area in repairing the boiler after the outlet bellows failure after 1313 hours of operation, Reference (a). Although the weld was excellent, the parent metal on both sides of the weld on the O.D. showed indications of having been oxidized. This manifested itself as intergranular and preferential crystallogrphic plane penetration of the Ta on the O.D. of the dome, Figure 13. The oxidation occurred during the repair welding as a result of an insufficiently pure inert atmosphere. During subsequent system operation, the NaK leached the oxides from the part leaving voids. These defects showed no indications of having propagated during boiler operation, and no other indications of NaK or Hg corrosion or mass transfer deposition were detected, Figure 14.

The O.D. of the Ta dome had surface tears, extending 0.003 inches into the wall, which probably occurred during the fabrication of the part, Figure 15. These defects did not appear to have propagated during system operation and although they are undesirable, do not appear to have caused any adverse effects on boiler operation.

The repair weld metal, heat affected zone and parent metal in the weld area all exhibited a hardness gradient, Table V. Each was harder on the I.D. than on the O.D. with the weld being the hardest. This presumably was due to the solution of oxygen into the material during the repair weld and possible from some contamination from the Hg side by the system atmosphere during startups and shutdowns. Since the weld metal is considerably harder than the parent metal immediately adjacent to it, it is thought that the welding atmosphere probably produced a greater effect than the system atmosphere.

VII. Ta DOME AT Hg OUTLET

The Ta weld joining the header to the outlet dome was examined metallographically. Although a few minor inclusions were observed in the weld metal, the joint appeared to be excellent, Figure 16. The hardness in the heat affected zone and the parent metal was somewhat erratic but showed no gradient across the wall thickness from O.D. to I.D., Tables V and VI. This is the same condition which existed at the inlet dome except the hardness level of the outlet is lower.

The Ta dome at the Hg outlet had the same type of O.D. fabrication tears exhibited on the inlet. These did not appear to have caused any adverse effects; no indications of propagation or of corrosion were detected. On the header to which the dome was welded the original machining grooves were still present indicating that corrosion had not occurred and that this material had been machined without producing surface tears.

No evidence of corrosion of the Ta on the Hg side of the dome was detected. However, sporadic mass transfer deposits up to 0.0005 inches thick were present.

Cross-sections through both the inlet and outlet dome sections exhibited flow patterns parallel to the contour of the part. There was no evidence that variations in working had produced adverse effects.

VIII. Ta TUBE-HEADER-ORIFICE WELD AT Hg INLET

A section of the Ta tube-header-orifice weld at the Hg inlet was examined metallographically, Figure 17. No defects were detected; however, the wall thickness of the tube in the weld metal consisted of only one grain. The hardness of this weld averaged 130 KHN with the Hg exposed surface being 129 KHN and the core being as high as 140 KHN. This is the same condition that existed at the outlet tube to header weld except at the inlet header the hardnesses were somewhat higher. No indications of corrosion, erosion or mass transfer product deposition were detected metallographically.

IX. Ta TUBE TO HEADER WELD AT Hg OUTLET

The Ta tube to header weld at the Hg outlet was also of excellent quality; no defects were detected metallographically, Figure 18. However, in the weld metal one grain comprised the entire wall thickness in some locations. The average hardness of the weld metal was 116 KHN with the weld softer on the surface (99 KHN) than below the surface (up to 129 KHN). This gradient is presumed to be due to the welding technique and not due to operational effects. The crown of this weld is exposed to the Hg side of the boiler. If system atmosphere contamination had occurred, the Hg exposed surface would have been harder than the core of the weld because Hg will not remove oxygen from the Ta as NaK does.

The area below the weld where the tube had been rolled into the header appeared satisfactory from a metallurgical standpoint, Figure 19. No evidence of incipient failure or other deleterious effects were observed. Although the rolled area appeared to provide some support of the tube by the header, there was not intimate contact along the entire rolled area between the tube and the header.

X. Ta ORIFICE

A longitudinal section through one of the Hg inlet orifices was examined metallographically. No indications of corrosion, erosion, or mass transfer product deposition were detected. The Ta walls of the orifice were smooth with no indications of machining tears indicating that the final reaming to size

had been a good operation. Within the part tolerance, no increase in orifice size was detected by mechanical measurements. Microhardness determinations revealed that the hardness outward from the Hg exposed surface was somewhat erratic, 118-145 KHN, but it did not exhibit a definable gradient. This indicates that oxygen contamination during system operation was minimal.

XI. Ta INLET PLUG

Sections of the multi-passage Ta plug from the inlet end and from the Hg outlet end were examined metallographically. The sections were identical metallographically. The Ta was in the recrystallized condition, but the swaging operation had upset the lands producing some mushrooming effect, Figure 5. The land machining marks were still visible on the sides of the lands and at the base of the grooves there was some roughening presumably due to the machining operation. Microhardness traverses indicated the swaging had produced some work hardening of the lands. The hardness of the flutes decreased from the top of the lands toward the center of the plug. The hardness showed a variation between lands indicating that the cold work during swaging was not completely uniform. At sporadic and isolated locations some particles up to 0.00017 inches thick were present in the grooves. This material may have been mass transfer products but there was insufficient material present for analysis. It did not appear that Hg corrosion had occurred and no indications of potential plug failure were detected.

XII. Ta-10W SWIRL WIRE

A two-inch long section of wire was found in the turbine Hg inlet filter after it was removed from PCS-1. Chemical analysis revealed the wire was Ta-10w, the material used for the swirl wire in the boiler. Boroscope examination revealed that a section of this wire was missing from the center Hg tube of the boiler at the outlet end. Metallographic examination of the wire removed from the filter revealed it contained numerous cracks, (Figure 20); however, the wire could be free bent 180° without further cracking. Sections of Ta-10w wire which had been used for coiling experiments and new wire procured for the fabrication of another boiler could also be bent 180° without fracturing. Metallographic examination did not reveal the presence of cracks in these two

wires. All three wires were in the cold worked condition and each had the same hardness, Rockwell C 38. Cleaning per AGC 10319/8 of the two sections of wire which had not been in the boiler resulted in their becoming embrittled. In bending these sections of wire, fracture occurred with essentially no permanent set. It was presumed that hydrogen embrittlement of the wires occurred during cleaning. Separate sections of wire were electrically coupled to 316SS and to platinum during exposure to 2-1/4 pounds KOH per gallons water (that solution in the cleaning operation most likely to cause embrittlement) at 210-220°F for 1-1/2 hours to determine if these materials would provide anodic protection and prevent the embrittlement. Both wires were embrittled after this treatment indicating that anodic protection was not effective. Sections of the embrittled wire were exposed at elevated temperature in vacuum to outgas the hydrogen. Ductility was completely restored, as indicated by bend testing, after exposure at 1000°F for 16 hours or at 1500°F for 2-1/2 hours, confirming that hydrogen had produced the embrittlement. During the life of the boiler, the swirl wire had been exposed to hydrogen several times. During the initial hot outgassing of the boiler, hydrogen was evolved presumably from the tantalum in the boiler. After the bellows failure, the Hg side of the boiler was chemically cleaned, as described above, which would cause absorption of hydrogen not only by the wire but by the Ta tubes. This hydrogen was subsequently removed by hot outgassing prior to Hg injection and during the subsequent system operation. After the failure of a PNL line at the gas fired heater outlet and the concomitant replacement of a Hg PMA, hydrogen from an undetermined source was evolved in the Hg loop. Also, organics have been detected in the Hg system; decomposition of these materials produces hydrogen.

It was concluded that hydrogen embrittlement of the Ta-10w swirl wire in the boiler had occurred at least one time during the operation of the boiler in PCS-1. This embrittlement in conjunction with residual stresses in the wires from coiling and from inserting them in the boiler tubes had caused the failure of the wire. Subsequent system heatup and operation had outgassed the hydrogen from the wires restoring their ductility. In addition to the piece of wire which had completely broken loose from the boiler, other wire cracking may have

occurred which could have resulted in additional loss of wire from the boiler during continued operation. This potential condition in conjunction with other deleterious conditions in the boiler made it undesirable to continue operating with this component.

XIII. Ta/316SS TRANSITION JOINT AT Hg INLET

The hot co-extruded Ta/316SS transition joint at the boiler Hg inlet was partially debonded. Hand held transducer, ultrasonic inspection revealed that the 316SS had debonded on the O.D. for a longitudinal distance of approximately 1/8 inch around the entire circumference of the joint. In addition, one area indicated debonding approximately 1-1/2 inches around the circumference for a longitudinal distance of approximately 1/2 inch. Since previous laboratory exposures of this type of joint had indicated that debonding would continue to failure of the joint and subsequent leakage (Reference (f)), the joint was destructively sectioned for metallographic examination. In the area where ultrasonic inspection had indicated debonding to a depth of 1/2 inch, the metallographic examination revealed debonding for a distance of 0.323 inches on the O.D., Figure 21. In addition, on the I.D., the tantalum had debonded from the 316SS for a distance of 0.012 inch in the plane examined. Both the 316SS and the Ta were in the cold worked condition and at their interface a third phase was present. These conditions are typical of those found in the samples, produced by the same technique, which were previously investigated. A program is presently in progress under the control of NASA LeRC to improve and optimize the fabrication techniques and reliability of hot co-extruded Ta/316SS joints. No evidence of Hg corrosion or mass transfer product deposition were noted on either the 316SS or the Ta of the transition joint.

IX. 321SS STATIC NaK TUBE FAILURE

Another undesirable condition was cracking of the lowest 321SS static NaK containment tube which failed in the transition from round to oval cross section at the Hg inlet. This cracking was a cause of leakage between the static NaK and the PNL, but sectioning and post operational testing of the boiler was not continued to confirm that this was the only leakage path.

Stress analysis indicated that this tube had been subjected to a stress of 44 KSI and that failure could be attributed to cyclic thermal stressing which occurred during the heating and cooling of the boiler for system startups and shutdowns, (Reference (g)). A factor which caused additional stress in the failed tube but which was not included as part of the stress analysis was the longitudinal rubbing and galling of the bottom of the tantalum tube with the 321SS tube surrounding it. This was detected both visually and metallographically.

Visual examination of the failed tube revealed that although the cracking had undoubtedly occurred under tensile loading, stress reversal had forced the fracture surfaces together under compressive loading so that the edges of the crack were cold worked and extruded both outward and inward slightly. Metallographic examination revealed that the 321SS had a normal structure except at the fracture surfaces where compressive yielding had occurred (Figure 22). The general structure has annealed, equiaxed grains. Approximately one percent sigma phase was present. This structure was identical with the structure of the top 321SS tube which had not failed. Each tube in the boiler at this location is subject to different stress levels because of the thermal gradients in the area. At the compressively yielded fracture surfaces of the failed tube a different metallographic structure existed, although X-ray diffraction indicated the presence of only austenite. The cold worked area was slightly lighter in color in the unetched condition and appeared as "salt and pepper" in the etched condition. Annealing the fractured material at 1900^{o}F did not change this structure. A step scan microprobe analysis for Fe, Cr and Ni across the crack surface structure into the unaffected parent metal revealed that at the crack surface the analysis was Fe - 79%, Cr - 8.7%, and that the analysis changed approximately linearly across the abnormal structure to Fe - 69%, Cr - 18%, in the unaffected material. The Ni concentration was constant across this area. The zone of abnormal structure was approximately 0.00157 inches thick in the area analyzed. The cause of the chemical gradient across the cold worked zone is not completely understood. Since the tubing was exposed only to NaK at elevated temperature, it is presumed that this eutectic alloy with low oxygen content caused the preferential leaching of chromium from the fracture surface. Possibly the higher energy state due to the cold working of the fracture edges contributed

to leaching at the fracture edges than was apparent in the remainder of the tubing. In addition, with changes in PNL or static NaK pressure, a flow of NaK through the crack up to an estimated velocity of 17 ft/sec occurred. The velocity of the PNL NaK over the O.D. of this tube is 4-1/2 ft/sec. The higher flow rate through the crack may also have contributed to preferential leaching in this area..

XX. ZIRCONIUM FOIL IN STATIC NaK SYSTEM

The corrugated Zr foil getters which were wrapped around the inlet and outlet Ta/316SS transition joints in the static NaK system, were removed after the boiler had been decontaminated of NaK. The foil from the NaK outlet was discolored dark grey and was friable at all locations. The foil from the NaK inlet was discolored grey and was friable in the outer layers but was clean silvery metallic colored on the I.D. side of the inner wraps away from the edges. These clean areas could be bent without breakage. Carbonates were present on the foil from both areas. Presumably these formed due to absorption of carbon dioxide from the air by the residual NaK after the boiler had been cut from the system. The variability in color and friability of the foil from the NaK inlet area indicates a variable degree of contamination of the Zr by C, H, N, and/or O, within the wraps of the foil and produced presumably by the shielding of the inner wraps by the outer wraps. Chemical analysis of the foils for C, H, N, and O tended to confirm this variability, Table IV. From the analysis results obtained, only qualitative conclusions can be drawn. The carbon, oxygen, and hydrogen content of both foils was higher than in the original foil. The hydrogen increases may have, in part at least, occurred during decontamination. The foil at the NaK inlet end of the boiler showed an increase in nitrogen content, but the foil at the outlet did not and may actually have decreased in nitrogen content. The increase in nitrogen in the inlet foil presumably is due to absorption from the NaK of nitrogen which was dissolved in the NaK at the argon/NaK interface in the expansion tank. The closer proximity of the inlet foil to the expansion tank and the static condition of the NaK would make the transfer of the nitrogen impurity in the argon essentially a diffusion process. As such, it would be expected to be gettered preferentially by the inlet foil. It appears that the Zr foil served its function of purifying the static NaK during boiler operation.

XXI. 316SS SHELL

Sections of the 316SS boiler shell from the NaK inlet end and from the NaK outlet end were examined metallographically. The outlet sample which operated at 1300°F contained sigma phase except for approximately 0.004 inches on the I.D. of the wall thickness. Using a lineal counting method (Reference (h)) it was estimated that 1.5 volume percent sigma phase was present except in the I.D. area. The distribution of this phase at the grain boundaries was relatively uniform and it did not form a continuous network. Therefore, it would not cause deleterious embrittlement of the steel, although it would tend to reduce the ductility and the stress rupture strength. Free bend testing of samples of this material did not cause cracking in 180° bends with the shell I.D. on the inside or on the outside of the free bend. The absence of sigma phase in the I.D. layer is attributed to a NaK effect altering the chemistry by either leaching out sigma formation enhancing elements or depositing sigma depressant elements. Step scan microprobe analysis revealed that the Fe, Cr and Ni content in the non-sigma containing area were identical with that in the sigma containing area. It is presumed that a change in the interstitial element content caused the suppression of sigma formation.

The 316SS from the NaK outlet did not contain sigma phase as revealed by metallographic examination. This area operated at 1150°F as opposed to the 1300°F operating temperature at the NaK inlet end indicates the temperature dependence of sigma phase formation.

NaK corrosion was not detected on the I.D. of the shell material at either the inlet or outlet ends. However, mass transfer products up to 0.003" thick were present at the NaK outlet end, Figure 23. These products are the result of corrosion in the high temperature portion of the PCS-1 system. Presumably this corrosion occurred primarily in the gas-fired heater tubing used to simulate the reactor in the PCS-1 testing. Ultrasonic wall thickness determinations indicate the 316SS heater tubing wall has thinned an average of 0.0008 inches per 1000 hours of operation. Due to the non-isothermal nature of the system, the corrosion occurs at the highest temperature portion of the system and deposition occurs at the coldest areas of the system when due to the lower NaK temperature, the solubility of elements in solution decreases and precipitation occurs.

XXII. 321SS STATIC NaK TUBE

Metallographic examinations of samples of the 321SS static NaK tube from the NaK inlet and from the NaK outlet end of the boiler were performed. Using the lineal counting method, Reference (g)), it was estimated that the Nak inlet material which operated at 1300°F contained 3.8 volume percent sigma phase and the outlet end material, which operated at 1150°F, contained 1.2 percent sigma, Figure 24. This again demonstrated the temperature dependence on sigma phase formation. In both locations the sigma was uniformly distributed without forming a continuous network structure. As with the 316SS shell material, the static NaK tube from the outlet exhibited a zone approximately 0.003 inches thick on the O.D. which had been exposed to the flowing NaK where sigma phase had not formed, Figure 24. This zone was not present on the I.D. which had been exposed to the non-flowing static NaK or at the NaK outlet end of the tube on either the I.D. or the O.D. Mass transfer products were present on the O.D. of the tube at the NaK outlet end, Figure 25. These deposits were similar in appearance to those on the I.D. of the shell material at the NaK outlet.

XXIII. COMPARISON OF SIGMA PHASE IN 316SS AND 321SS

The metallographic examinations performed indicated that for the particular lots of material used for BRDC #2 boiler, the 316SS was less prone to form sigma phase than the 321SS. From the 1300°F operating temperature area, the 321SS contained 3.8 percent and the 316SS 1.5 percent sigma. From the 1150°F operating temperature area, the 321SS contained 1.2 percent sigma phase and the 316SS did not contain sigma in amounts detectable by optical metallography. This condition is anticipated when decarburization of the 316SS does not occur during system operation. Since the carbon is combined with Ti as a stable compound in the 321SS, decarburization by NaK is not anticipated as it could be with the less stable chromium carbides formed in 316SS. However, since the carbon is combined with Ti in the 321SS, it is not present in solution in the austenite as a stabilizer or available to combine with the Cr to effectively reduce the matrix Cr. In the 316SS the carbon combines with Cr to effectively reduce the Cr content of the austenite matrix and thus reduces the sigma forming tendency. In addition, the 316SS has a higher Ni content than 321SS. Nickel also is an austenite stabilizer and tends to suppress sigma formation.

Although confirming chemical analyses were not performed, it appears that the 316SS shell material in the BRDC #2 boiler did not undergo decarburization as a result of contact with the flowing NaK. Therefore, as anticipated, it formed less sigma phase than the 321SS at the same temperatures. In both materials the sigma phase had not formed a continuous network in the grain structure and while some reduction in ductility of the materials occurred, no drastic embrittlement resulted after 8700 hours at temperatures up to 1300°F.

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b) SNAP-8 Failure Report No. 1190, 3/4/69

c) SNAP-8 Failure Report No. 1195, 5/27/69

d) S. S. Manson, "Thermal Stress and Low-Cycle Fatigue", McGraw-Hill Book Co., 1966

e) H. Bleil, "Tantalum Reaction Potential with Low Pressure Air at 1350°F in SNAP-8 Boiler During Pre-Injection Hot Outgassing", TM 4923:68-518, 3/11/68

f) H. Bleil "Thermal Exposure Evaluation of Tantalum/316SS Transition Joints", TM 4923:69-579, 12/2/69

g) S. Krikopulo, "Crack on the Static NaK Oval Tube", Analysis No. SA-8-240, 12/2/69

h) R. T. Howard and M. Cohen, "Quantitative Metallography by Point-Counting and Lineal Analysis", Transactions AIME, Iron and Steel Division, Vol. 172, pp 413-476, 1947

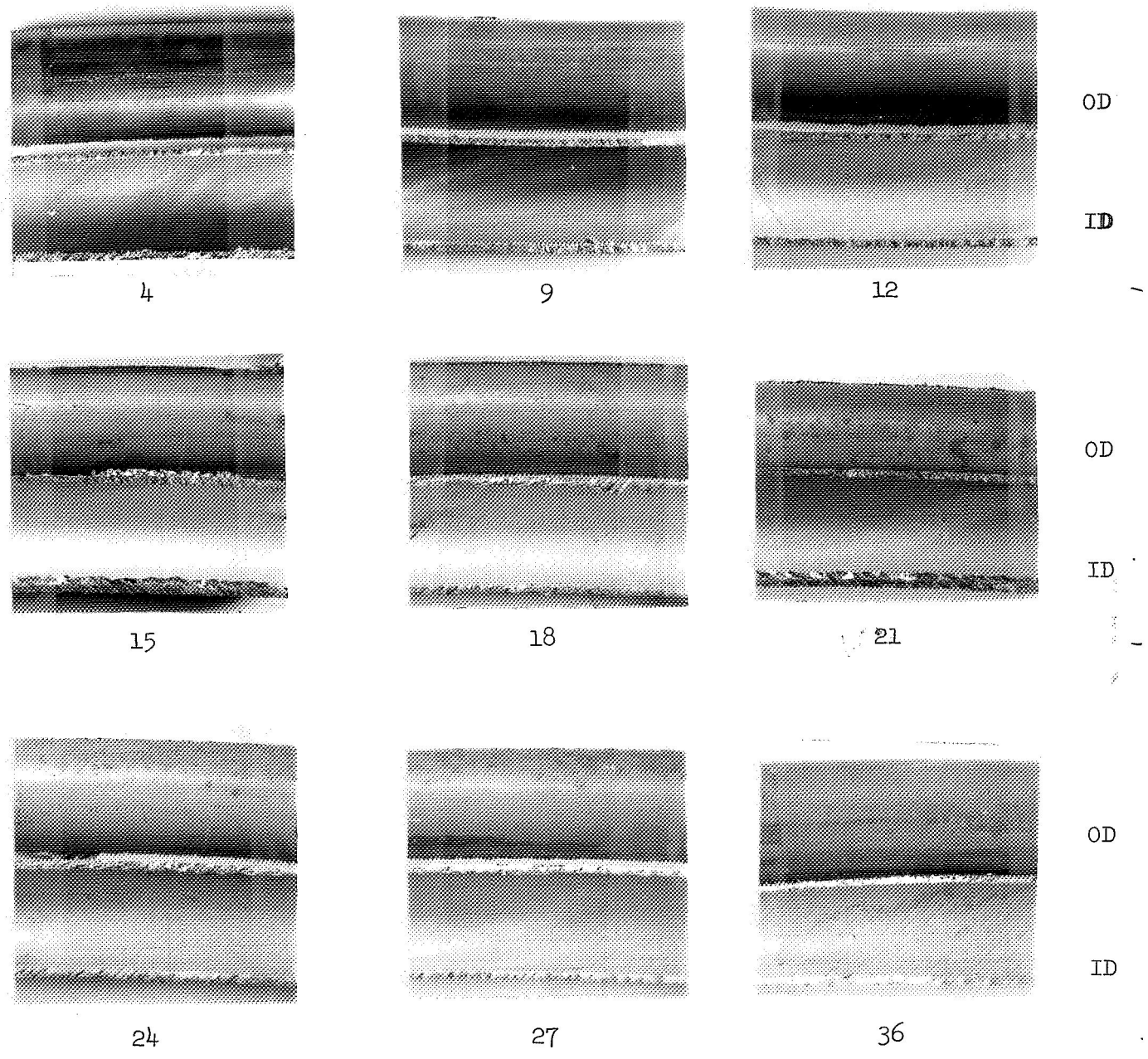
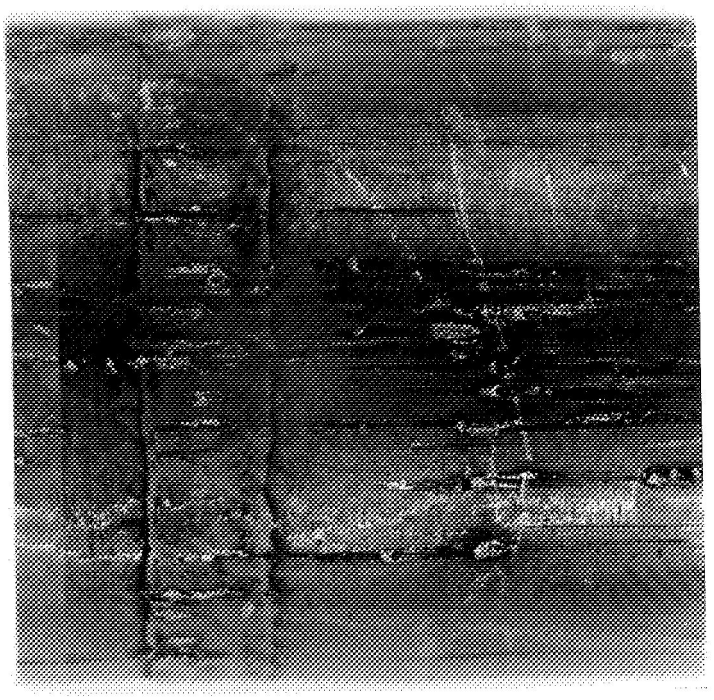


Figure 1 - Appearance of I.D. and O.D. of Ta tube at various distances in feet from the Hg inlet. Approximately full size.

1051



5X 0410 250X 0420

Figure 2 - Scoring and galling of O.D. of Ta tube at end of straight Hg inlet section. Left - general appearance, right - transverse cross-section through tube showing material galled into O.D.

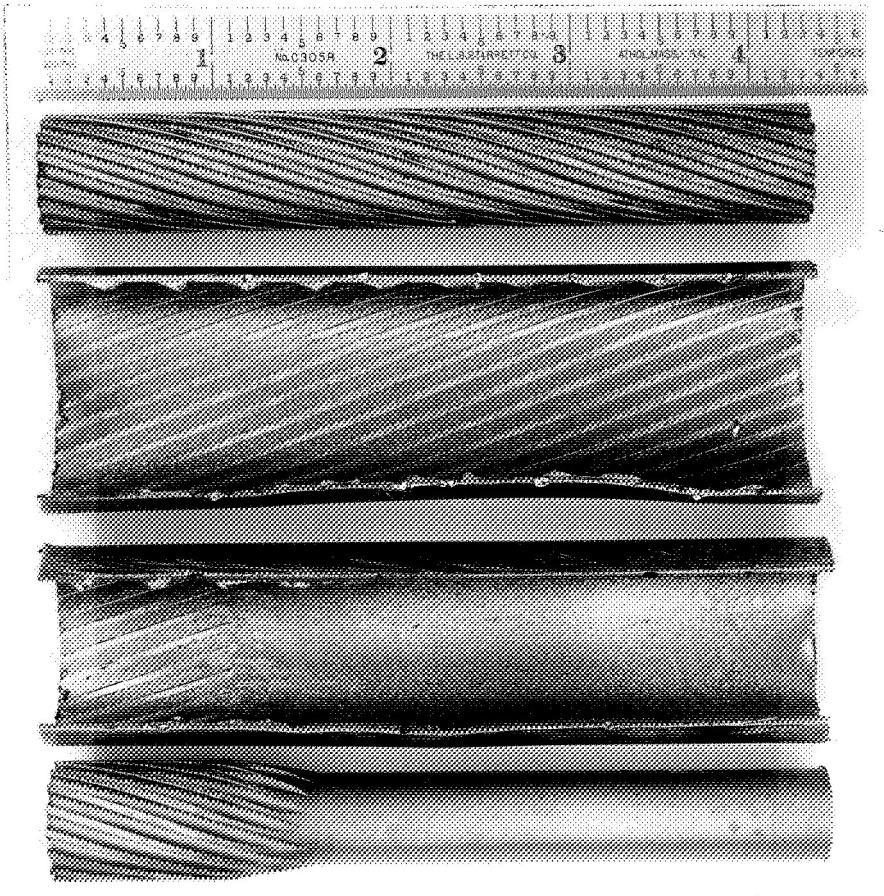


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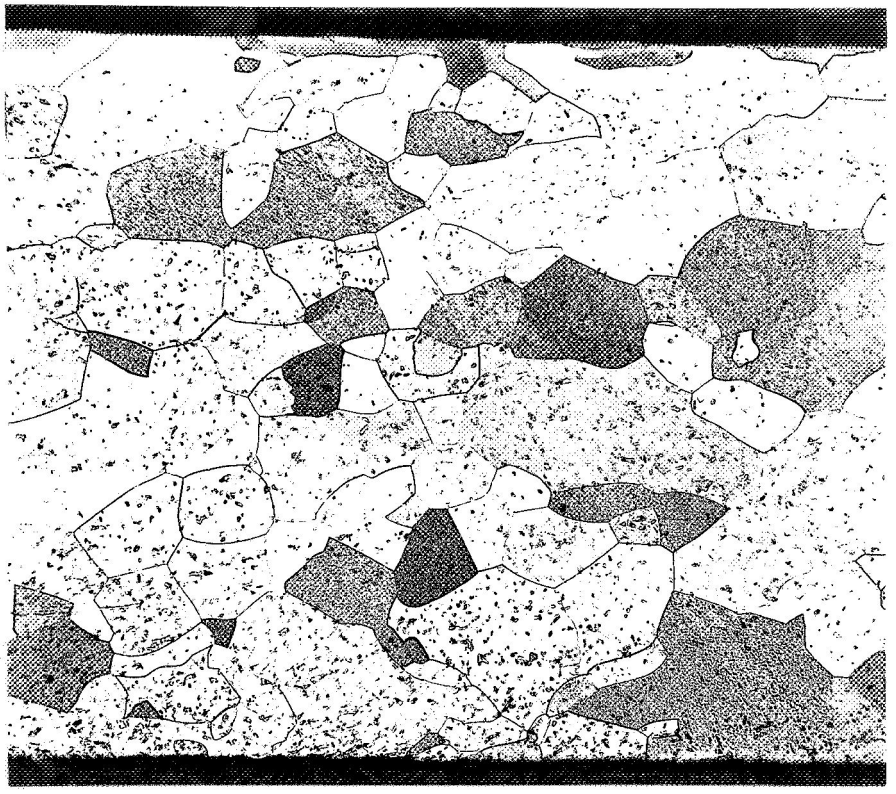
Figure 3 - Multipassage Ta plug and I.D. of Ta tube from Hg inlet of boiler.

1051



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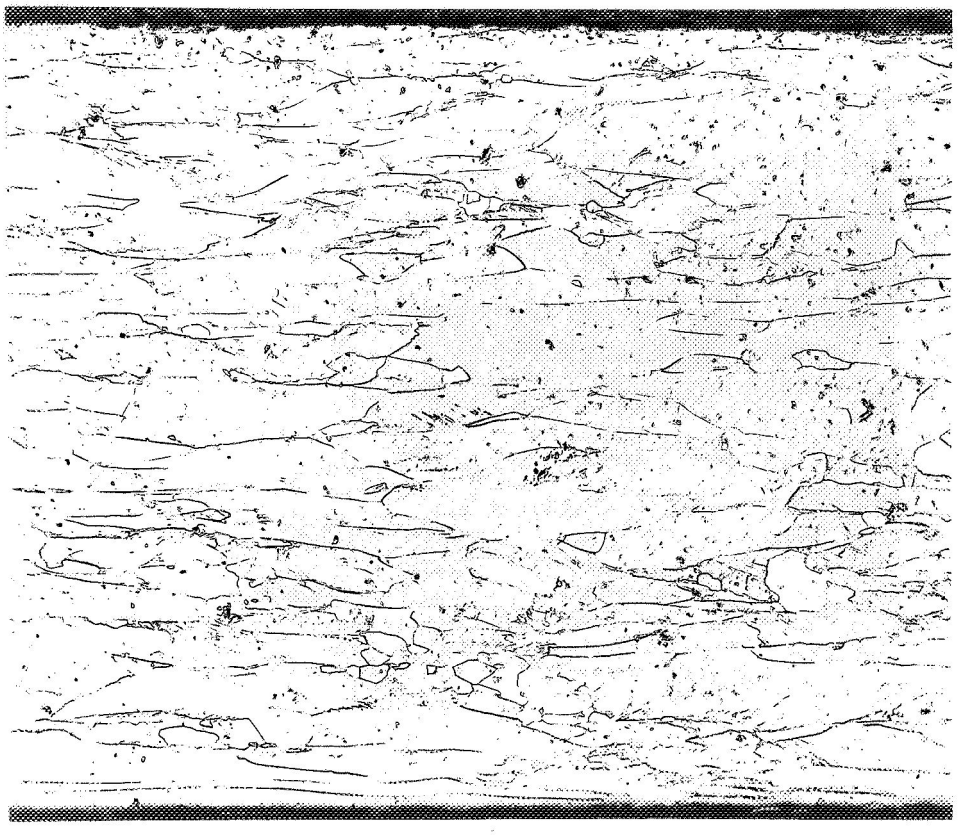
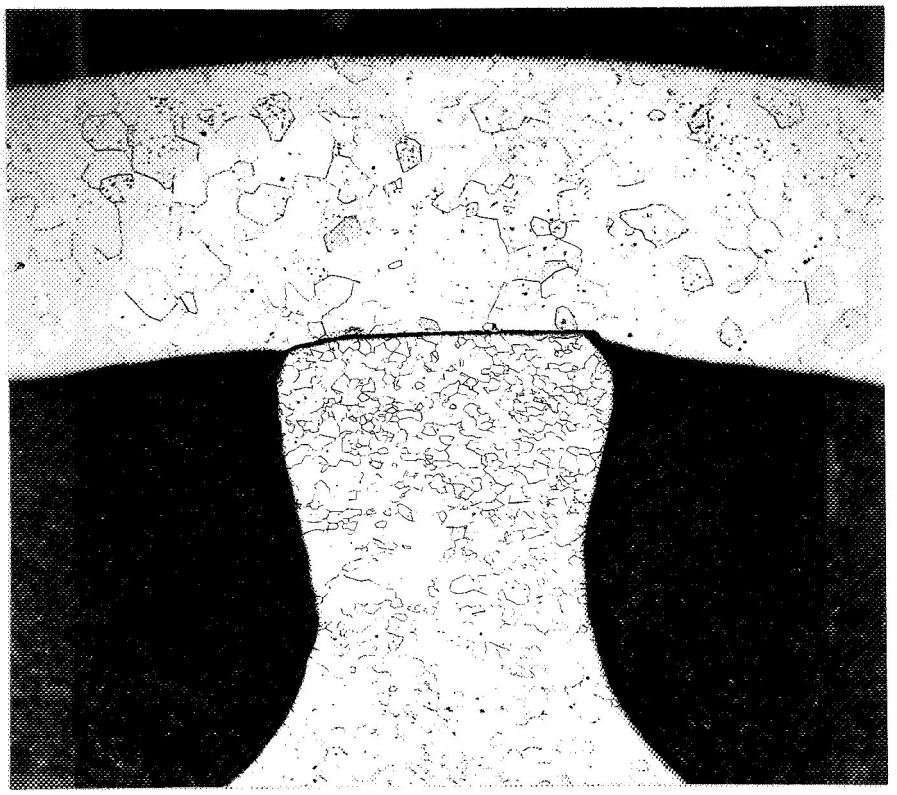


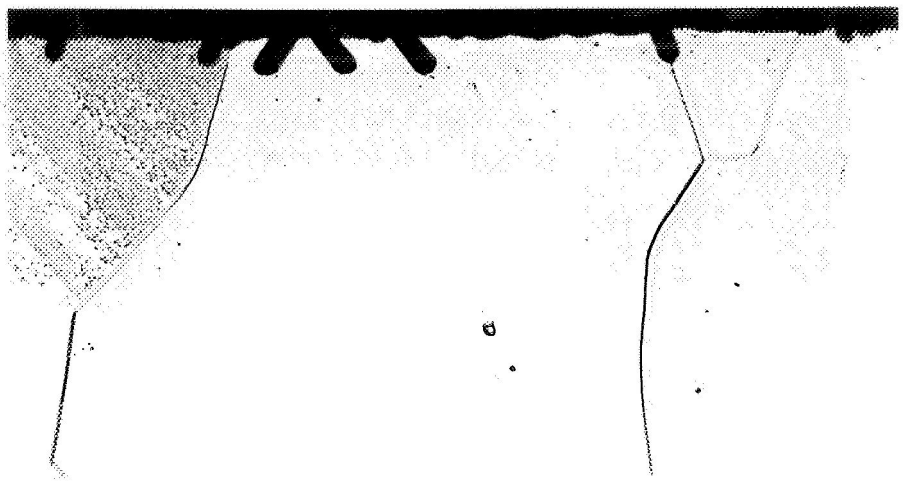
0445

Figure 4 - Longitudinal cross-sections through Ta tube. Recrystallized structure from Hg inlet straight section, top; and cold-worked structure 18 feet from Hg inlet 100X, Glyc + HF + HCl + HNO_3 etch.



40X Glyc + HF + HCl + HNO_3 etch. 3700

Figure 5 - Transverse section through Ta tube and plug at Hg inlet.



250X 0458

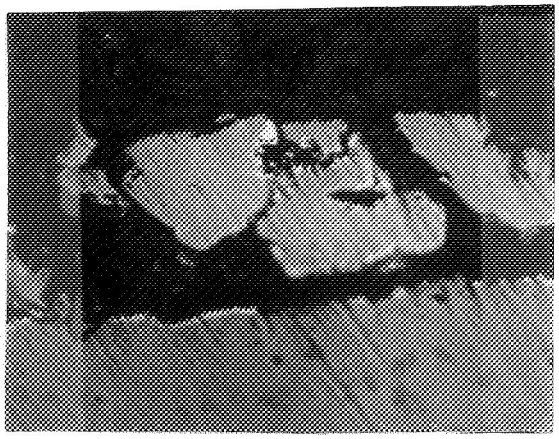
Figure 6 - Longitudinal cross-section through heat affected zone of Ta tube weld $36\frac{1}{2}$ feet from Hg inlet. Crevices in O.D. surface were produced by NaK corrosion along grain boundaries and preferential crystallographic planes and was caused by oxygen contamination during welding.

500X 0443 500X 0447

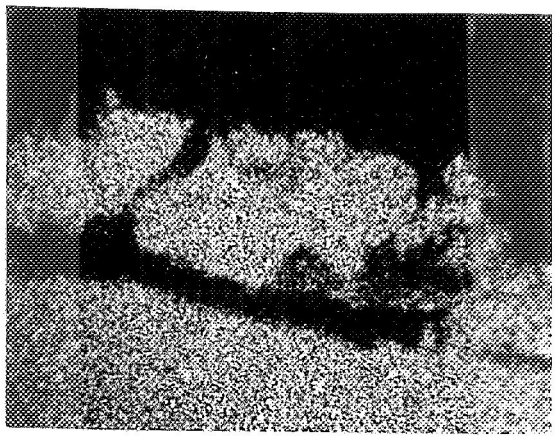
Figure 7 - Transverse sections through Ta tube showing I.D., Hg exposed surface, 14 inches from Hg inlet, left; and 36 feet from Hg inlet, right.

250X 0421

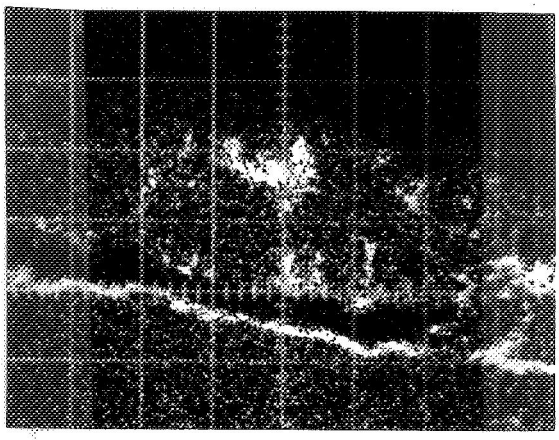
Figure 8 - Longitudinal cross-section through Ta tube weld 12½ feet from Hg inlet showing I.D. surface mass transfer deposited layer.



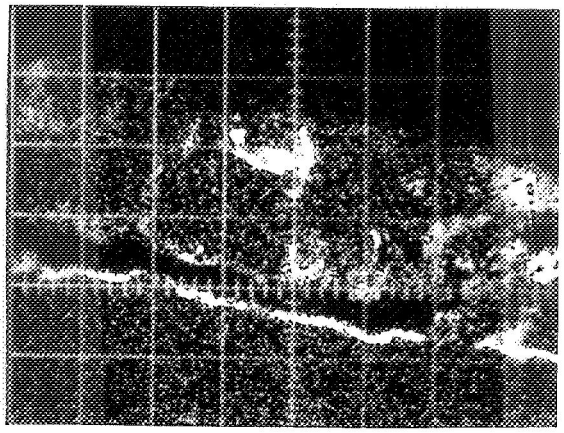
Visible light photograph. Image reversed from radiation displays.



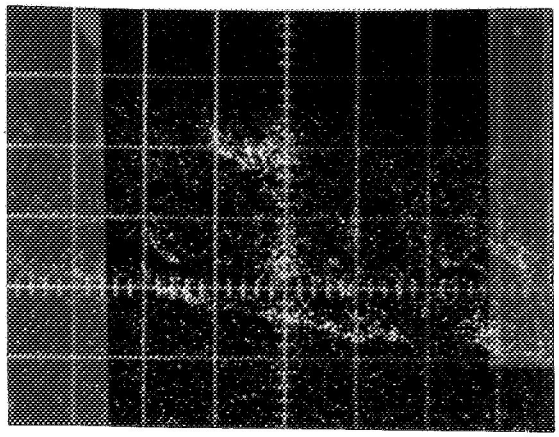
Tantalum



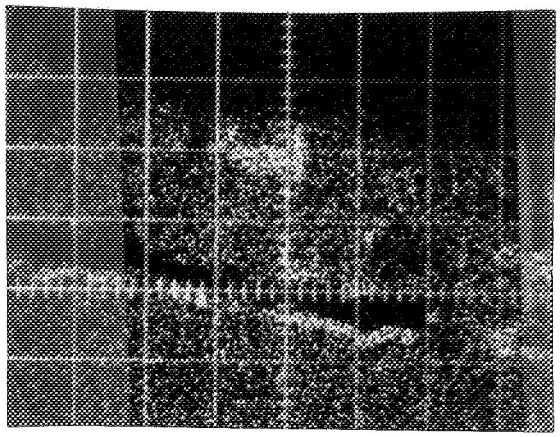
Nickel



Iron

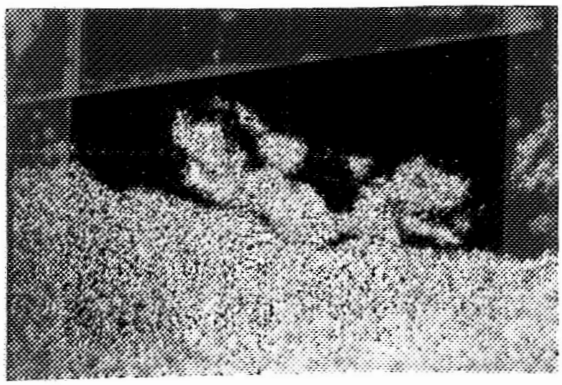


Chromium

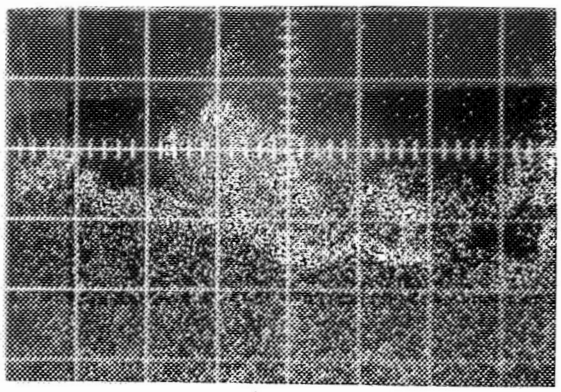


Cobalt

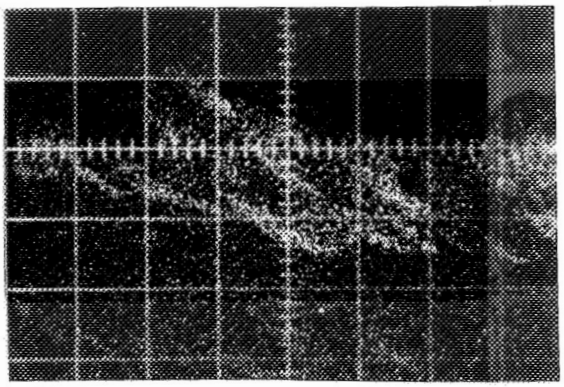
Figure 9 - Radiation displays of I.D. of Ta tube 15 feet from Hg inlet header showing distribution of Ta, Ni, Fe, Cr, and Co. 340X See Figure 10 for another display from another circumferential location on same tube cross-section.



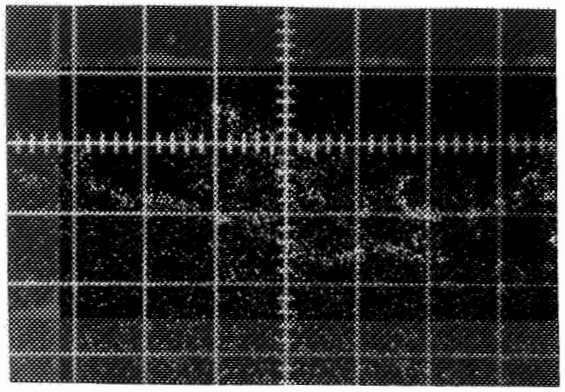
Tantalum



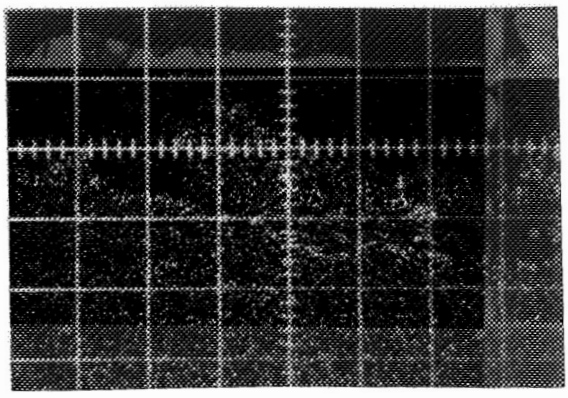
Nickel



Iron



Chromium



Cobalt

Figure 10 - Radiation displays of I.D. of Ta tube 15 feet from Hg inlet header showing distribution of Ta, Ni, Fe, Cr, and Co. 340X

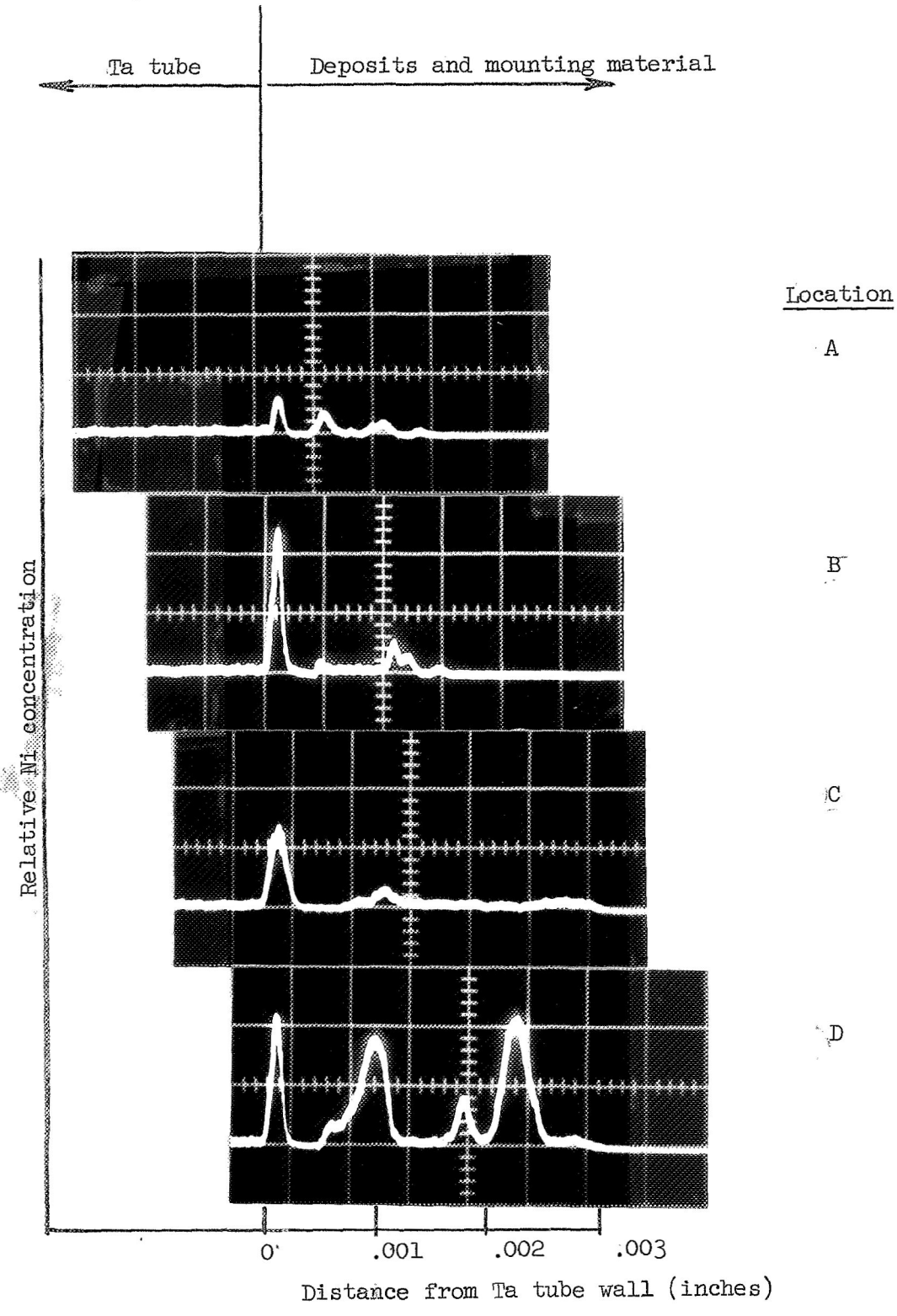


Figure 11 - Nickel concentration radiation traces at four random I.D. locations on cross-section of Ta tube 15 feet from Hg inlet.

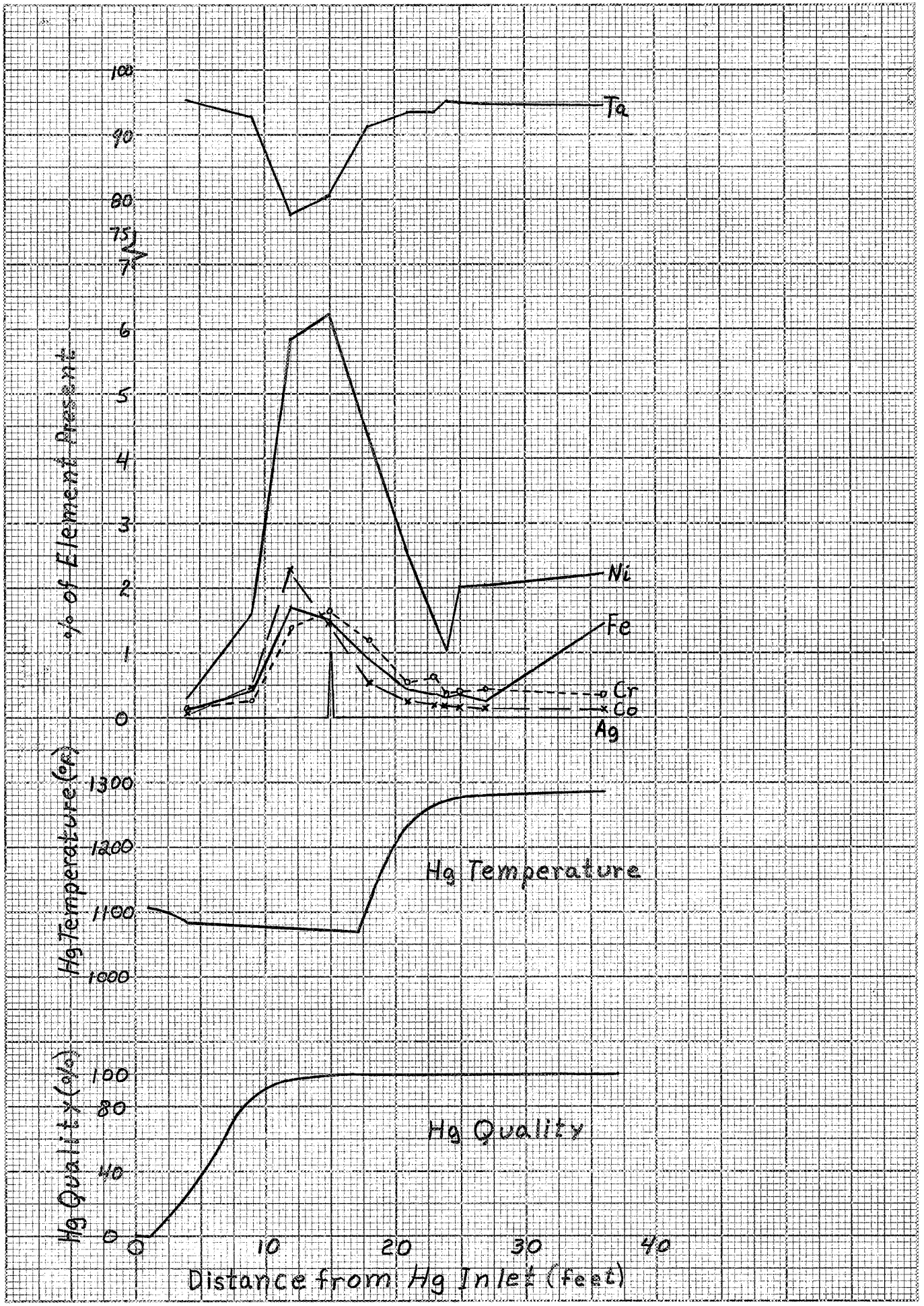
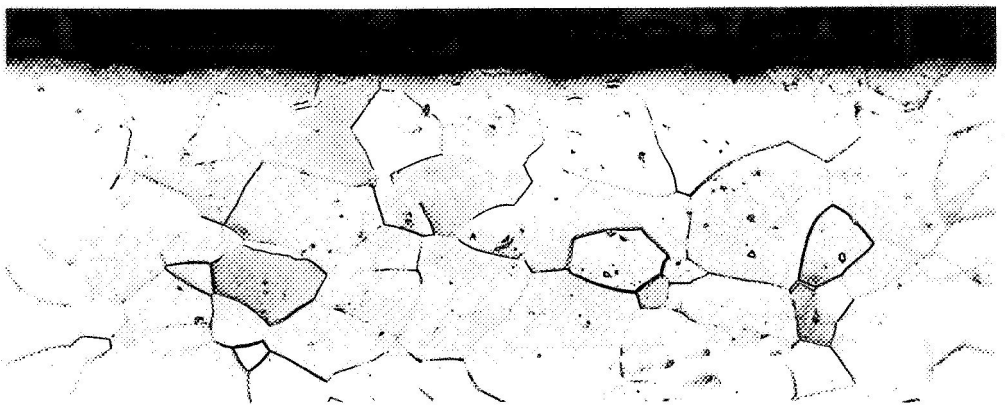


Fig. 12. Composition of I.D. of Ta Tube as Determined By X-Ray Fluorescence Analysis.

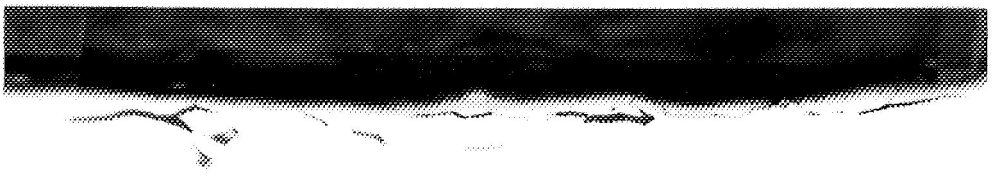
250X, Glyc + HF + HCl + HNO_3 etc. A3703

Figure 13 - O.D. surface of Hg inlet Ta dome showing effect of static NaK removal of oxides produced in the weld heat affected zone during repair welding.



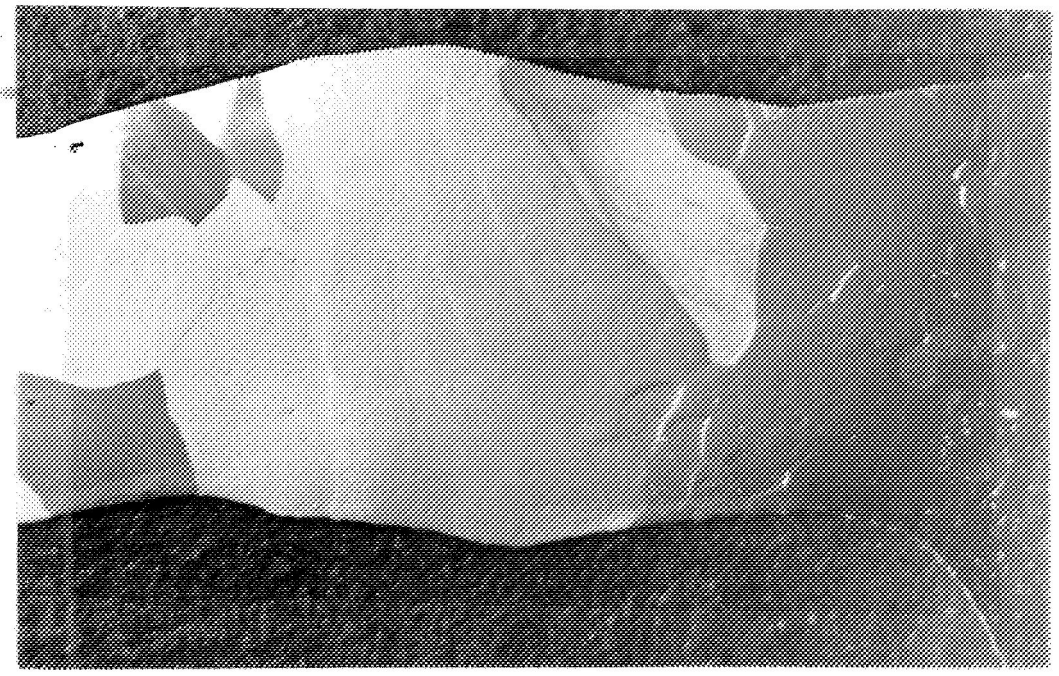
250X, Glyc + HF + HCl + HNO_3 etch. 0425

Figure 14 - I.D. surface of Hg inlet Ta dome.



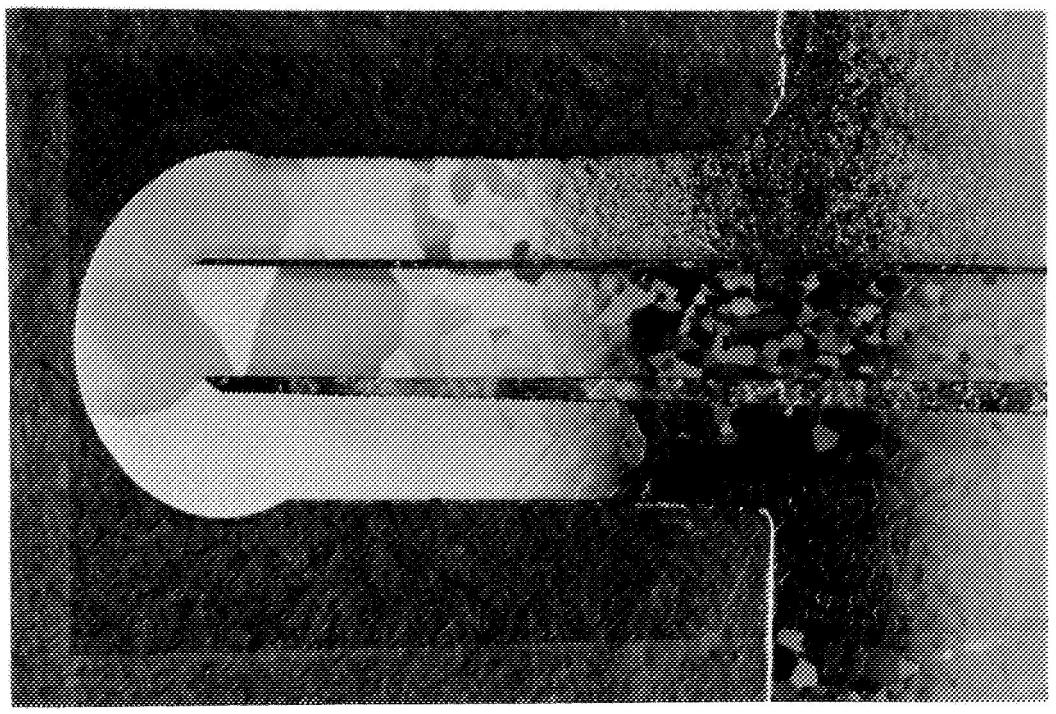
500X 0418

Figure 15 - O.D. surface of Hg inlet Ta dome exhibiting surface tears produced during fabrication.



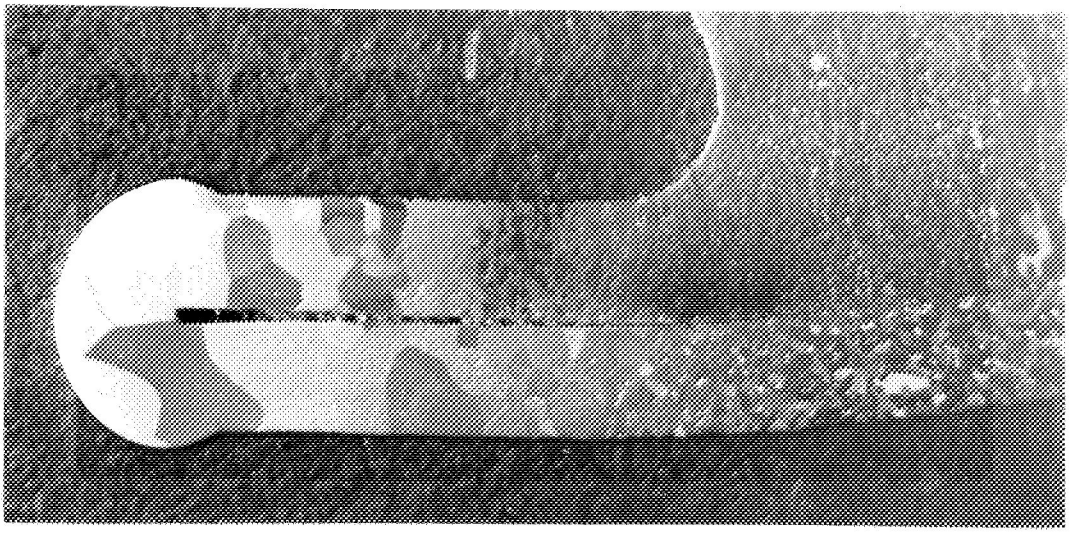
10X 0431

Figure 16 - Weld of Ta dome to header at Hg outlet.



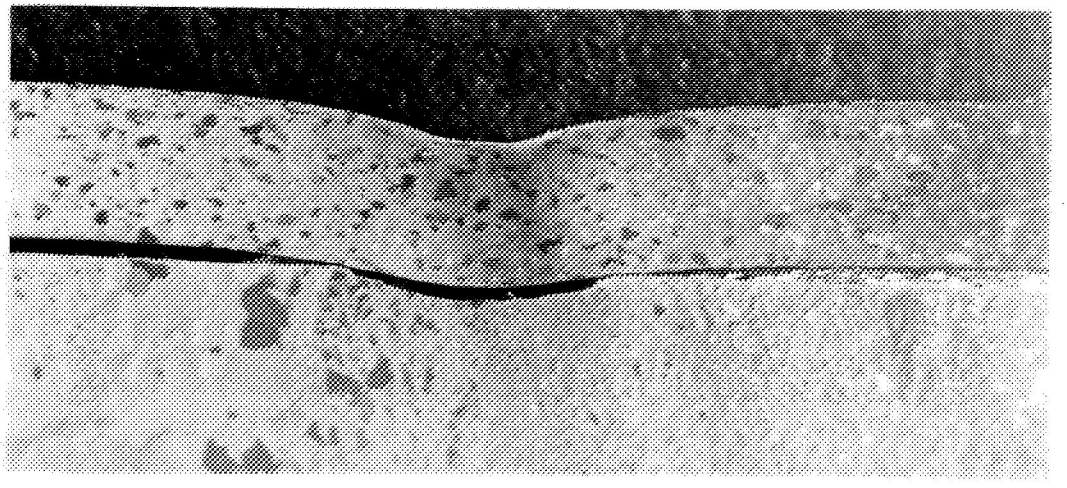
15X, Glyc + HF + HCl + HNO_3 etch. 3707

Figure 17 - Ta Tube/Orifice/Header weld at Hg inlet



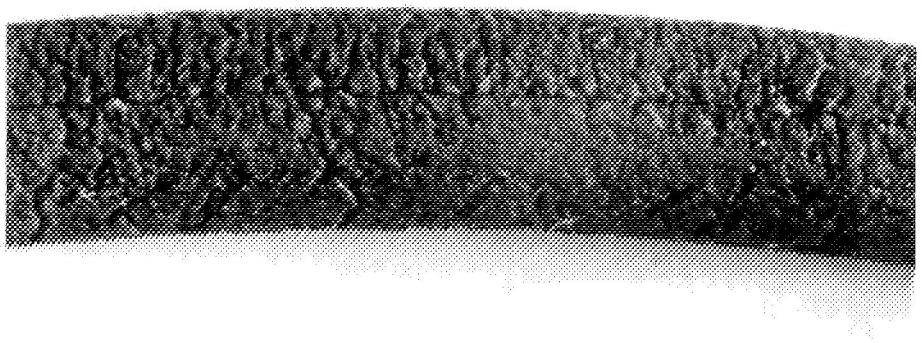
15X 0429

Figure 18 - Ta tube to header weld at Hg outlet.



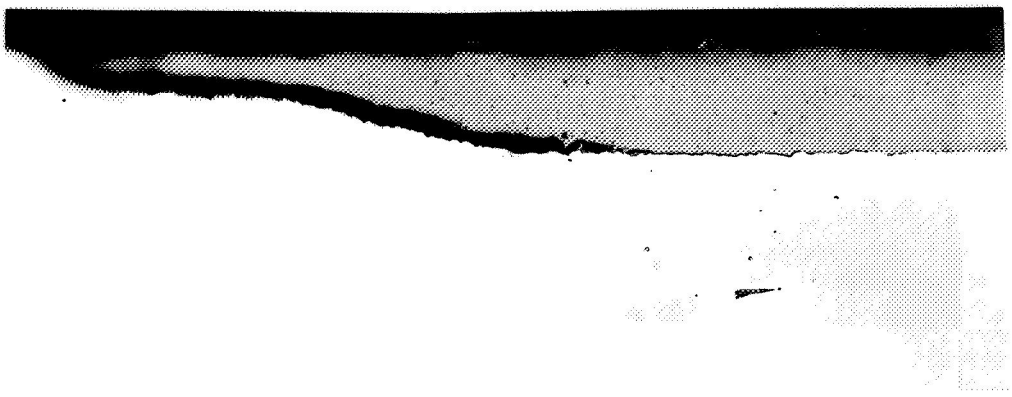
15X 0428

Figure 19 - Configuration of Ta tube where it was rolled into the Hg outlet header during boiler fabrication.



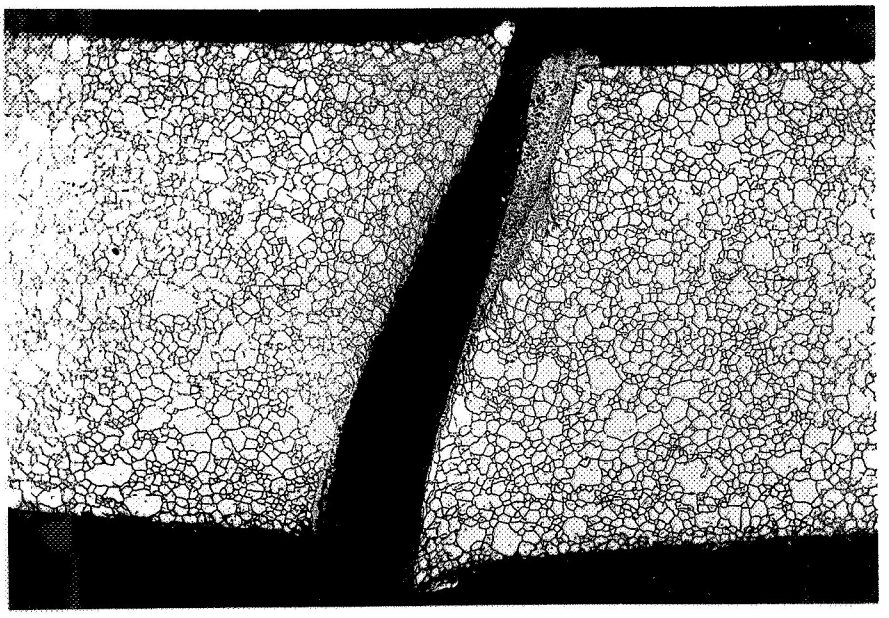
20X 3603

Figure 20 - Macrophoto of section of Ta-10w swirl wire which was recovered from turbine inlet filter.



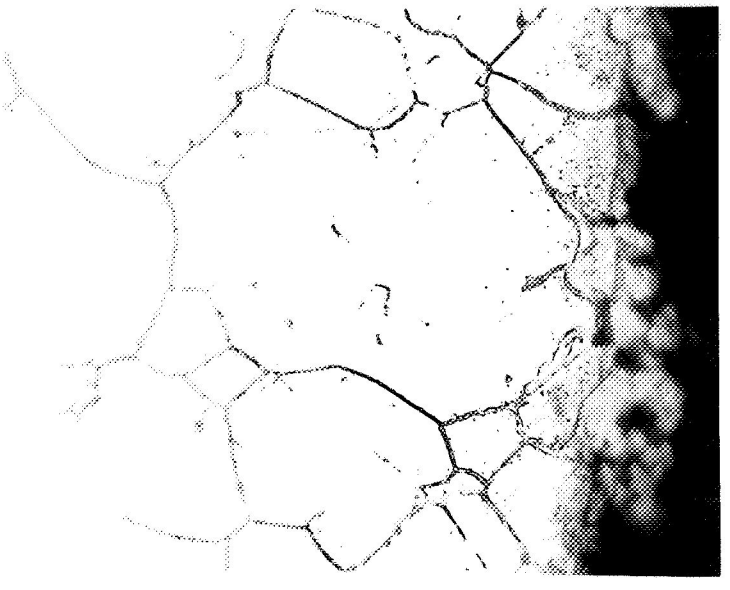
250X, unetched 3739

Figure 21 - Cross-section through Hg inlet, hot coextruded Ta/316SS transition joint showing debonding.



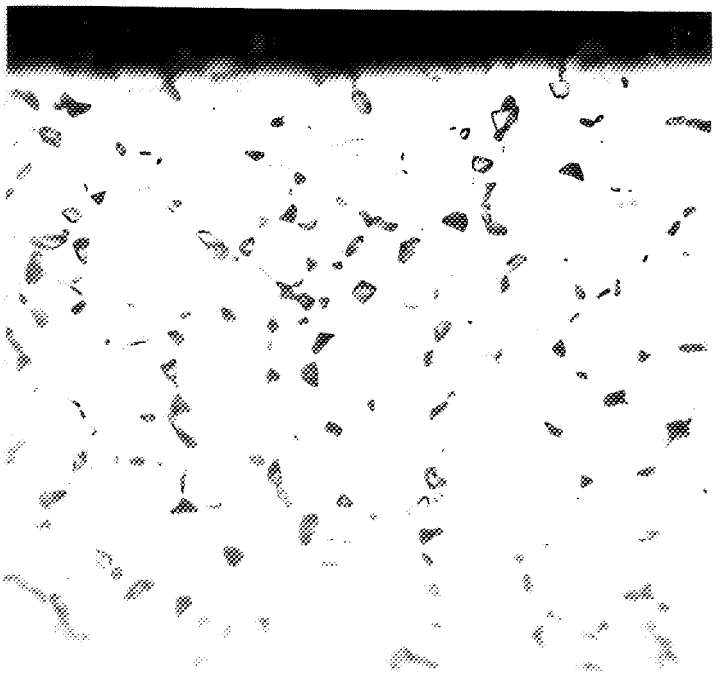
75X, Ammonium persulfate electrolytic etch 3662

Figure 22 - Longitudinal cross-section through crack in 321SS static NaK tube near Hg inlet.

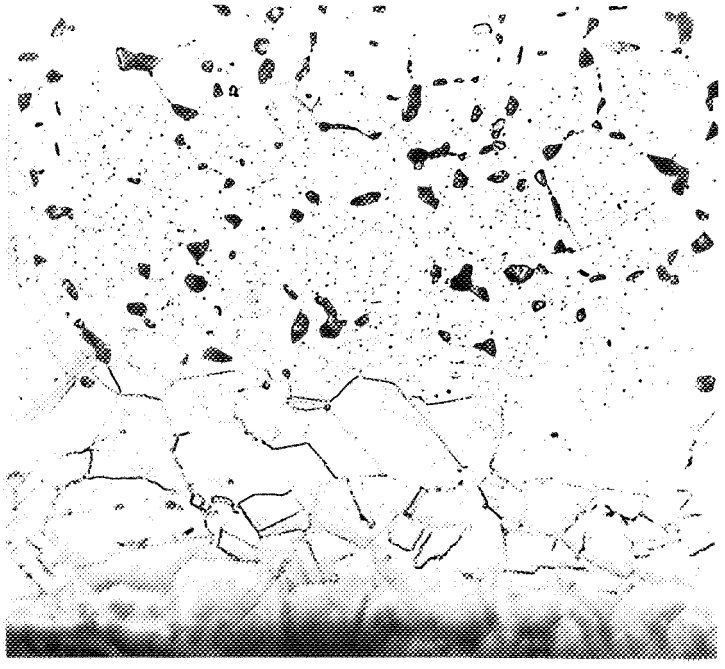


500X, Ammonium persulfate electrolytic etch 3714

Figure 23 - Section through 316SS boiler shell at primary NaK outlet showing mass transfer deposition I.D. and absence of sigma phase in structure.

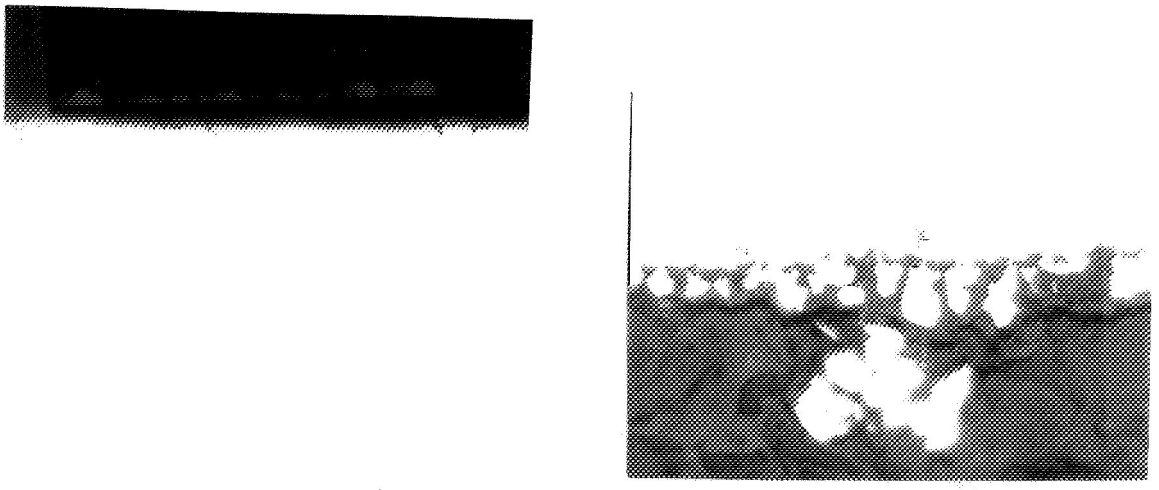


500X 0456



500X 0457

Figure 24 - Cross-section through static NaK tube at Hg outlet (top), I.D. exposed to static NaK (bottom), O.D. surface exposed to flowing NaK. 10% $(NH_y)_2$, S_2, O_8 electrolytic etch.



0451 0450

Figure 25 - Cross-section through 321SS static NaK containment tube at Hg inlet (left). I.D. exposed to static NaK; (right) O.D. exposed to flowing NaK. Unetched, 500X

TABLE I

SEMI-QUANTITATIVE CHEMICAL ANALYSIS OF RESIDUE
ON O.D. OF Ta TUBE AT Hg INLET END OF BRDC #2 BOILER

ELEMENT	WEIGHT PERCENT PRESENT IN RESIDUE
Fe	8.9
Cr	1.9
Ni	2.1
Mn	1.8
Si	0.7
Ta	8.6
V	4.6
Mo	5.5
Cu	0.07
Mg	Trace
Na	32.5
K	13.2
C	3.7

TABLE II

KNOOP HARDNESS OF Ta TUBE AT VARIOUS LOCATIONS

Distance from Tube O.D. (inches x 10^3)	Distance from Hg Inlet Header								
	1 inch (Unswaged)	14 inches (swaged over plug) at plug land	14 inches (swaged over plug) between lands	4 ft.	9 ft.	15 ft.	21 ft.	27 ft.	36 ft.
1		296	274	154	137	158	141	158	151
2	108	244	244	154	145	158	145	154	154
3	-	-	198	-	-	-	-	-	-
4	111	191	163	154	141	154	141	154	148
6	111	130	141	145	149	148	145	154	141
8	111	123	130	149	158	154	137	158	134
10	108	-	-	-	-	-	-	-	-
12	111	111	141	149	141	154	141	154	149
14	-	-	-	-	-	-	-	-	154
16	113	126	137	154	130	154	137	158	137
20	106	117	130	158	137	149	137	154	141
24	110	130	123	158	141	149	145	149	141
28	108	117	114	145	141	149	130	145	145
32	104	120	120	145	145	141	141	142	145
34	104	123	123	141	141	149	137	142	142
36	110	120	130	147	137	154	141	149	149
38	102	-	120	147	145	145	137	149	142
Ave.	108	150	152	150	142	151	140	151	145

TABLE III

KNOOP HARDNESS Ta TUBE-TUBE WELDS AND HEAT AFFECTED ZONES (HAZ)

Distance from O.D. (inches x 10^3)	Distance from Hg Inlet									
	$36\frac{1}{2}$ ft.		$28\frac{1}{2}$ ft.		$18\frac{1}{2}$ ft.		$12\frac{1}{2}$ ft.		$4\frac{1}{2}$ ft.	
	Weld	HAZ	Weld	HAZ	Weld	HAZ	Weld	HAZ	Weld	HAZ
2	116	111	151	127	127	113	142	124	228	188
4	118								192	145
6	116	122	140	118	134	118	148	127	154	122
8	120								145	
12	118	127	137		122		122	122	134	113
18	120	127	137	122	116	110	120		127	118
24	111	116	134		111		116	118	127	
32	110	122	132	132	110	116	118	116	110	102
38	116	122	142	134	111	120	120	124	120	113
40		137				127		127	122	
48	122		148		120				120	
50							129			
52	113				124					
56							142			
Ave.	116	123	140	127	119	117	129		140	128

TABLE IV

INTERSTITIAL ELEMENTS CONTENT OF SAMPLES FROM BRDC #2 BOILER

Material and Location in Boiler		PPM of Element Present			
		C	H	N	O
Zr Foil NaK Inlet	Inner-wrap/ Outer-wrap	311/1335	84/184	380/1160	4850/19500
Zr Foil NaK Outlet		170/2414	4600/8000	<50/<50	6800/7000
Zr Foil Original Condition		26	25	150	780
Ta-10w wire from boiler but found in TAA inlet filter		20	36	50	550
Ta-10w wire unused but not the wire that was used in BRDC #2 boiler		63	25	100	630
Ta Tube - Distance from Hg Inlet Header					
	1 inch	29	12	<50	110
	18 feet	78	7	<50	50
	36 feet	49	22	50	180

TABLE V

KNOOP HARDNESS OF Ta WELDS AT VARIOUS LOCATIONS

Distance From O.D. (inches x 10^3)	Dome to Transition Joint Weld at Hg Inlet	Dome to Transition Joint Weld at Hg Outlet	Tube-Orifice Header Weld at Hg Inlet	Tube-header Weld at Hg Outlet
2	94	104	129	99
4	106	111	134	118
6	110	111	129	124
8		111	134	129
10		108	140	129
18	116		129	120
30		97	127	120
38	120		127	113
50		97	118	110
58	116			102
70		99		
78	127			
90		106		
98	129			
110		116		
118	127			
130		127		
138	137			
150		104		
158	164			
170		124		
178	192			
182	192	134		
184	197	134		
186	180	137		
188		134		
190		137		
Ave.	144	116	130	116

TABLE VI

KNOOP MICROHARDNESSES OF Ta FROM VARIOUS LOCATIONS IN BRDC #2 BOILER

Distance from O.D. (inches x 10^3)	Ta Dome at Hg Inlet, PM	Ta Dome at Hg Outlet, PM	Dist. from I.D.	Hg Orifice	Dist. From O.D.	Thinned Wall Ta Tube	1" from Thinned Wall	Dist. From O.D.	Ta Plug			
									16" from Inlet		42" from Inlet	
									Land	Valley	Land	Valley
2	132	134	2	134	2	102	99	1	179	126	137	126
6	180	142	4	122	4	104	102	2	173	120	145	120
10	176	151	6	127	6	104		4	168	120	141	120
20	172	164	8	118	8	108	111	6	168	117	141	117
30	168	134	10	137	10	110	113	8	163	111	141	111
40	161	124	20	132	14	111	106	12	158	111	145	111
60	158	127	30	145	16	118		16	163	126	137	126
80	154	132	40	137	18	118		20	158	126	137	126
100	140	118	60	121	20	111	111	24	173	126	134	126
120	140	116	80	118	24		104	28	-	-		
140	142	124	100	128	28		100	32	158	117	145	117
146		104			32		106	34	-	-		
150	122				36		111	36	158	-		
153	134				38		102	40	145	114	149	114
								50	123	114	134	114
Ave.	152	131		127		109		60	123	130	117	130
								80	114		114	117
								100	117		114	
								140	114		117	
								Core	129			