

VIII. T-111 ALLOY CRACKING PROBLEMS  
DURING PROCESSING AND FABRICATION\*

R. A. Ekvall,<sup>†</sup> R. G. Frank,<sup>†</sup> and W. R. Young<sup>†</sup>

SUMMARY

A review is made of T-111 cracking encountered at General Electric Nuclear Systems Programs Department during fabrication of advanced space power Rankine cycle system components since 1965. Cracking has been observed after machining, forming, and welding. A majority of crack incidents have been associated with welding. Although tungsten inert gas welding is the primary joining technique used, cracks have also been found after electron beam and spot tack welding.

Cracking phenomena associated with each fabrication method are described with emphasis on weld related cracking. Preventative measures adopted for each type of cracking are reviewed. Possible causes for cracking, including attempts at correlating cracking incidents with material properties and processing history, are discussed. The frequency of crack occurrences and the requirements for highly reliable system components indicate the need for an investigation of the basic mechanism(s) underlying T-111 alloy cracking.

INTRODUCTION

The transition of a material from the laboratory to production mill products and then into machined, formed, and welded components, assemblies, and system configurations is normally accompanied by some technical difficulties. T-111 alloy is a material that is still in this stage of development. Laboratory testing of T-111 alloy has shown it to be a very ductile alloy, but numerous examples of brittle cracking of the alloy have been seen during mill processing and during secondary fabrication into system components. The observed cracking is intergranular in nature and appears to fall into two broad phenomenological categories: (1) cracking

\*Based on work conducted under NASA contracts NAS 3-6474 and NAS 3-9426.

<sup>†</sup>General Electric Company, Cincinnati, Ohio.

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

associated with elevated temperature grain boundary sliding and (2) brittle propagation of cracks at room temperature under applied stress.

Over the last 4 years, General Electric Nuclear Systems Programs (GE-NSP) has procured between 4000 and 4500 pounds of T-111 alloy in all mill forms for five NASA sponsored space electric power programs; however, the bulk of the alloy was procured for two current NASA sponsored programs, the advanced refractory alloy corrosion loop program (contract NAS 3-6474) and the potassium boiler development program (contract NAS 3-9426). Tables VIII-1 and VIII-2 summarize the sizes and quantities of the T-111 alloy procured for each program. Solids of round cross sections ranged from 0.062-inch-diameter wire to rods 4.625 inches in diameter. Solids of rectangular cross sections ranged from 0.005-inch-thick foil to 0.500-inch-thick plate. Tubing items have been as small as 0.25-inch outside diameter by 0.050-inch wall to 1.5-inch outside diameter by 0.100-inch wall. Tubular shapes larger than 1.5-inch outside diameter were made by machining solid rod. In processing T-111 alloy for these requirements, refractory metal producers encountered cracking during forging, extrusion, rolling, and tube reduction. However, the purpose of this paper is not to review the incidents of cracking associated with primary processing, many of which are of a proprietary nature. The primary emphasis of this paper is to review the cracking incidents that have been encountered at GE-NSP since its first major commitment (1965) to build a T-111 alloy system for NASA on the advanced refractory alloy corrosion loop program (ref. 1). Cracking has been observed during the cutting, machining, forming, and welding of T-111 alloy components.

NASA sponsored advanced Rankine cycle power system work at GE-NSP has been mainly concerned with the design, building, and testing of components and the determination of the alkali metal corrosion resistance of candidate advanced refractory alloys. Investigations of the mechanisms underlying the observed cracking in T-111 alloy were not included in the scope of this work. GE-NSP policy has been to (1) appraise NASA of the cracking problems encountered, (2) document the cracking encountered with informal letter reports and photographs, and (3) promptly determine means for solving the immediate cracking problem to minimize program delays. In a number of cases, the cracked parts were rather easily repaired, non-destructively evaluated to ensure their suitability for service, and used. In such cases, no destructive evaluation was performed. However, other cracking incidents required destructive evaluation before a plan for repair or replacement could be devised. In a few instances, even when a remedy was found for a particularly puzzling cracking problem or one likely to be encountered often, further but limited destructive evaluation was performed on similar parts in an attempt to identify the cause(s). For these reasons, each cracking event described in this paper has not

received equivalent evaluation effort, and the discussion may raise as many or more questions than are answered.

Prior to proceeding with the discussion of individual cracking events, some general remarks are in order regarding the quality and processing history of the material which exhibited cracking. It is a policy and contractual requirement at GE-NSP to maintain traceability back to the ingot for all T-111 alloy components. This capability enabled GE-NSP to review the quality and processing history of the as-received material used for each item that cracked to determine if a common material properties and/or processing history might be responsible for the observed cracking. The properties and processing histories of the cracked material were also compared with T-111 alloy mill products that did not crack. No property variation was found that was clearly associated with T-111 alloy that eventually cracked. Evaluating the contribution of processing history to cracking proved to be difficult. Some of the processing information was incomplete because the vendor supplying the T-111 alloy considered the information proprietary. Within the limits of available information, fair comparison of processing procedures was further complicated by the following processing differences among the vendors: number of heats melted, melting practice, size of ingots, primary conversion techniques, and secondary fabrication techniques. It can be said that some type of cracking was observed, during fabrication at GE-NSP, of material supplied by each vendor. No conclusive evidence could be found that showed that one processing approach was clearly superior to another. However, based on overall experience with T-111 alloy vendors, some general suggestions for improving the compositional control and the chances of successful ingot conversion are (1) Hf additions should be made by the vacuum arc melting process (two melts are preferred with the ingot being inverted between melts to improve Hf homogenization); (2) Hf content should be on the low side (<2.0 percent) of the specification; (3) initial conversion temperatures around 2000<sup>o</sup> F should be avoided because of possible low ductility (ref. 2); (4) reduction limits should be observed in extrusion (>3:1) and primary forging to avoid cracking; (5) proper cutting and grinding procedures should be used to avoid crack initiation and subsequent propagation; and (6) contact between T-111 alloy and copper or copper alloys should be avoided whenever possible.

The discussion of individual T-111 alloy cracking incidents at GE-NSP, which follows, is arbitrarily divided into three major categories: (1) cracks related to cutting and forming; (2) cracks related to machining and forming; and (3) cracks related to welding, including tungsten-inert-gas (TIG), electron beam (EB), and spot tack welding. In all cases, the material referred to is T-111 alloy unless otherwise specified.

## CRACKS RELATED TO CUTTING AND FORMING

Cutting of T-111 alloy mill products with an alumina abrasive (Allison VA 1202 MRA) cutoff wheel frequently produced cracks in the cut surfaces. The cracks induced by abrasive cutting always propagated in a brittle manner upon subsequent bending (fig. VIII-1). Although water cooling of the T-111 alloy and wheel was used in all cases, the cooling did not prevent cracking. Generally, slow feeding of the T-111 alloy mill products into the wheel resulted in more severe cracking than occurred with fast feeding. The effect of cutting T-111 alloy with an alumina abrasive wheel at varying speeds is illustrated in figure VIII-2, which shows transverse sections of a 0.375-inch-outside-diameter tube that were partially flattened after cutting. Removing 0.060 to 0.080 inch from the cut tube surfaces by grinding was sufficient to remove the cracks generated during cutting, and the tubing could then be bent without cracking (fig. VIII-3). Further evidence of the damage that can be caused by the use of an improper cutoff wheel is exhibited in figure VIII-4. Cracks were observed in T-111 alloy tube hollows during initial attempts to tube reduce the hollows. The deep cracks shown apparently propagated from shallow cracks resulting from cutting with an abrasive cutoff wheel. The sound portions of the tube hollows were used to successfully fabricate tubular bellows blanks (0.625-in. o.d. by 0.0085-in. wall). Alumina abrasive cutoff wheel induced cracking was not limited to tubing mill forms. Cracking also was observed in solid rods, as shown in figure VIII-5.

The cracking that occurs in both solid rod and tubing is intergranular in nature. However, the mechanism of the crack formation in T-111 alloy and, of even greater interest, the mechanism which causes the cracks to propagate in a brittle manner are not understood. However, embrittlement with nascent hydrogen from breakdown of coolant or abrasive binder is a possibility.

In limited testing to find a way to avoid crack initiation during cutting operations, it was found that cracking could be eliminated by the use of SiC cutoff wheels (Allison C120-K-RA), tube cutters, or band saws. Because of the limited testing and possible inadvertent use of the wrong wheels, GE-NSP has adopted the policy of using tube cutters and band saws for cutting operations.

## CRACKS RELATED TO MACHINING AND FORMING

Cracks related to machining have been observed in parts subsequently formed or welded; no cracking has been seen in as-machined parts at GE-NSP. Cracks in machined and welded parts are described later in this paper. Probably the most

striking example of failure of machined parts during forming is the brittle fracture of machined parts during forming is the brittle fracture of machined thermocouple wells during bending (fig. VIII-6(a)). Five wells were machined from 1.0-inch-diameter rod. The wells were jig bore drilled with a 0.156-inch-diameter hole to a depth of ~2.75 inches using Tap Magic as the cutting fluid. Then the part was turned on a lathe to final dimensions of 2.45- to 0.255-inch diameter to a 63 rms finish. After machining, the straight thermocouple well was placed between matched concave and convex aluminum blocks machined to a 1.01- to 0.99-inch radius with a 0.125-inch-radius groove in each block, and an attempt was made to form the machined T-111 alloy thermocouple well to an angle of 86° to 84°. Cracking began almost immediately and resulted in brittle fracture. The same type of failure occurred during initial attempts to bend a second machined thermocouple well. It was suspected that residual surface stresses present at the completion of the machining operations were responsible for the cracking during bending. In an attempt to alleviate this problem, subsequent thermocouple wells were given a stress relief heat treatment of 2400° F for 1 hour in a vacuum of  $\sim 10^{-5}$  torr after machining and prior to bending. The remaining three thermocouple wells were bent successfully without cracking, as determined by fluorescent penetrant inspection (fig. VIII-6(b)).

Metallographic and X-ray diffraction evaluations of the cracked thermocouple wells were performed. The metallographic results showed the major fracture to be intergranular and also revealed several areas of incipient intergranular cracking near the outside diameter of the tube on the tension side of the bend (fig. VIII-7). No cracking was observed in the inside diameter of the thermocouple wells. A comparison of the microstructure of an as-machined and a machined and stress relieved thermocouple well indicated the overall structures to be similar with the exception that recrystallization had occurred in a thin worked surface layer of the stress relieved thermocouple well. X-ray diffraction analyses qualitatively indicated that a reduction in residual stresses from machining and bending had occurred as a result of the 2400° F, 1-hour heat treatment.

Subsequent attempts to reproduce a brittle fracture in sheet samples (0.055-in. thick) after removal of 0.010 inch from each side using a shaping machine were unsuccessful. Neither samples machined to a surface finish similar to that of the thermocouple wells nor samples machined more severely to produce a very rough surface fractured when bent 180° over a 1/2 T bend radius at temperatures as low as -320° F.

Although stress relief treatments solved the immediate problem of fabricating thermocouple wells, the cause of the brittle intergranular fracture remains a matter for speculation. To reduce the possibility of cracking in other machined parts, a

specification (GE-NSP 03-0071-00-A) requiring that all machined parts of T-111 alloy be given a postmachining stress relief treatment at 2400<sup>o</sup> F for 1 hour was prepared and issued.

## CRACKS RELATED TO WELDING

The preponderance of cracking observed at GE-NSP has been related to welding. Selected incidents of cracking associated with each method of welding are discussed in this section.

### EB Welding

Welded and reworked tube. - To compare the biaxial creep properties of EB welded and seamless tubing, two 2.0-inch-outside-diameter by 0.25-inch-wall tube hollows (one welded and one seamless) made from the same heat of material were tube reduced and drawn to 1.5-inch-outside-diameter by 0.100-inch-wall tubing. The weld in the "welded" hollow was a simulated full penetration EB weld running the length of the hollow parallel to its longitudinal axis. The weld was formed by making two consecutive EB welding passes on a seamless hollow. The second pass was made to smooth the rough weld surface produced by the first pass. No defects were found in the "welded" tube hollow during ultrasonic and fluorescent penetrant inspections to NSP specifications 03-0001-00-C and 03-0027-00-A, respectively. After a 37-percent tube reduction, intermittent transverse cracks were observed in the heat-affected zones on either side of the weld for the entire length of the tube. Grinding the tube within allowable tolerances did not completely remove the cracks. Without further conditioning, the tube was annealed, processed to final size, and final annealed at 3000<sup>o</sup> F for 1 hour. The appearance of the finished tube is shown in figures VIII-8 and VIII-9. Because the quantity of the "welded" material required for the program was critical, no metallography was performed on the tubing after cracking was first observed. It is difficult to determine with certainty from the recrystallized structure of the finished tube whether or not the cracking was intergranular.

It is believed that residual stresses in the tube surface, promoted primarily by the second weld pass, were a major contributing factor to the observed cracking. Although a postweld stress relief treatment might have avoided the problem, it would seem wise to avoid multipass welds whenever possible. Details of the tube history, processing, and ultimate utilization have been reported (ref. 3).



Pressure transducer diaphragm. - Helium mass spectrometer leak checking and visual examination of the 0.005-inch-thick T-111 alloy diaphragm of a pressure transducer shown in figure VIII-10 revealed two diametrically opposed cracks. The cracks were approximately 0.040 inch from the fusion zone joining the diaphragm to the transducer housing. The transducer had been postweld annealed (2400° F for 1 hr) prior to the discovery of the leaks. Metallography indicated the cracks were intergranular. The microstructure appeared normal, and no significant variations in microhardness were found in the cracked sample. Bend tests of foil similar to that used to fabricate the diaphragms and heat treated similarly (2400° F for 1 hr) showed the material to be ductile. The cause of the cracking is not known, as numerous pressure transducers have been fabricated using the same procedure without cracking of the weld.

## TIG Welding

Since TIG welding is the most common welding technique used for joining T-111 alloy at GE-NSP, it is not surprising that most weld related cracks have been observed in TIG welds. Intergranular cracking was found in each cracking incident that was metallographically evaluated. Evidence indicates that the cracking occurring at elevated temperatures in T-111 alloy appears to be related to grain boundary sliding (ref. 4).

Butt weld joint. - During welding of a tube (0.375-in. o.d. by 0.065-in. wall) butt weld joint, one of the tubes was unsupported and sagged. In order to straighten the tube sections, the welded assembly was clamped between Mo plates and heated on one side of the weld nugget with a TIG torch. Intergranular cracking appeared on the side (tension side) of the weld nugget opposite the TIG torch. The cracks and grain boundary sliding observed in the weld nugget are exhibited in figure VIII-11. Similar grain boundary separation has been noted in welds inadvertently deformed at Oak Ridge National Laboratory (data obtained from B. Fleisher of ORNL).

Prevention, by providing adequate support for parts being welded, was the obvious solution to this problem.

Electromagnetic pump duct. - In repairing an electromagnetic (EM) pump duct, it was necessary to run a circumferential TIG weld approximately 0.8 inch (weld centerline distance) from a preexisting circumferential weld as illustrated in figure VIII-12. Cracking and grain boundary relief were noted in the machined duct surface between the welds after completion of the second weld. The location of the

cracking implies a critical temperature at which grain boundary sliding and separation occurs.

The cracks and grain boundary relief were removed by machining, benching, and hand polishing. Final visual, helium leak check, and X-ray inspection indicated the part was sound.

Absolute pressure transducers. - Cracks were discovered in three of six Taylor absolute pressure transducers after welding. Immediate leak testing with a helium mass spectrometer leak detector, according to NSP specification 03-0013-00-B, revealed that none of the transducers leaked. (A full section view drawing of a typical Taylor absolute transducer showing its functional relation to a test loop is given in fig. VIII-13.) The cracks were found near the circumferential TIG welds joining the top and bottom flanges. Photographs of the most severely cracked transducer (no. 14) are presented in figure VIII-14. The crack on the outer surfaces of the bottom flange of the transducer was approximately 0.5 inch deep, as can be seen in figure VIII-15, which shows the flange after removal of the weld nugget. Photomicrographs of end A of the crack are exhibited in figure VIII-16. The crack was quite wide where it intersected the outer circumferential surface of the flange, and it was not possible to definitely determine the mode of fracture. In figure VIII-16, however, cracking appears to be intergranular at end A.

It is interesting to note the amount of distortion in the grain shown by the arrow in figure VIII-16(b). It is possible that this distortion was not sufficient to relieve local stresses which resulted in grain boundary separation and subsequent propagation toward the surface. The fact that the crack width is larger at the surface than the center would tend to discount this theory by indicating the crack started at the surface. However, if residual stresses were induced at the surface during welding, there would be a considerable amount of spring back as the crack broke through the surface from the inside. This would cause a larger crack width at the surface, which would decrease as the spring back effects decreased from the edge to the center.

Cracks in all transducers (except the bottom flange of no. 14, which was metallographically sectioned) were removed by benching the material to a depth of approximately 0.030 inch. The shallowness of the cracks (except on transducer no. 14) indicates that the cracks may have been related to the surface condition of the machined transducers. The flanges were stress relieved at 2400° F for 1 hour following machining; however, X-ray diffraction studies have shown that this treatment does not completely relieve residual stresses. A longer time at 2400° F or a higher stress relief treatment might be necessary if residual stress from machining is the cause or contributes to the cause of the cracking.

Multipass tube welds. - One of the most reproducible incidents of cracking found in T-111 alloy is that which occurs in multipass welds (T-111 alloy filler material) of the type needed to join heavy sections (ref. 5). Grain boundary separation is first visible in the root pass after the second weld pass has been made. The grain boundary separation becomes more pronounced after the third pass and was found to extend into the second pass material after the fourth pass was complete. (Similar observations with multipass TIG welding of T-111 alloy have been made by R. Begley and W. Buckman of Westinghouse Electric Co.) Cracking and grain boundary separation were found predominantly in the periphery of the fusion zone (circled areas of fig. VIII-17(b)). Photomicrographs of a portion of one of these areas are presented in figure VIII-18. Although grain boundary separation was found primarily near the periphery of the fusion zone, some grain boundary separation was noted in the center of the fusion zone also. Occasionally grain boundary separation was found in the heat-affected zone near the edge of the fusion zone.

To determine the effect of filler wire composition on the cracking behavior of multipass welds, two 2.5-inch-outside-diameter by 0.375-inch-wall T-111 alloy tubes with solid ends were machined from 2.5-inch-diameter rod and subsequently butt welded using the TIG process with T-111, Ta-10W, and Cb-1Zr alloy filler wires. A J-shaped weld preparation was machined in each part prior to welding. The first weld pass, or root pass, was a T-111 alloy fusion pass except for a short region of the joint which was not welded to allow for the venting of the tube interior. Three additional passes were made using T-111 alloy filler for one 120° segment around the tube, Ta-10W alloy filler for another 120° segment, and finally Cb-1Zr alloy filler for the last 120° segment. A photograph of a section through the welded parts is shown in figure VIII-17(a). The welded tube was metallographically sectioned through each of the three segments representing the three filler materials. Photomicrographs of transverse sections through these welds are shown in figures VIII-19, VIII-20, and VIII-21. The major observations of this study are (1) grain boundary separation occurs in the T-111 alloy root pass for all three filler alloys, and (2) grain boundary separation is not observed in the Cb-1Zr or Ta-10W alloy portions of the corresponding weld segments. These observations indicate that hafnium, present only in T-111 alloy, is a significant factor contributing to the grain boundary separations in the T-111 alloy weldments.

Of the possible solutions to the multipass weld cracking in T-111 alloy, the most obvious solution is to design to avoid the use of heavy sections requiring multipass welds wherever possible; no weld cracking has been observed on TIG welds of parts having thin sections (~0.100 in.). Other suggested solutions include designing for single-pass EB instead of TIG welding, intermediate stress relieving, alloy modification, and alloy overlay.

Boiler weld failure. - During the initial startup of a T-111 alloy corrosion test loop, a leak developed between the lithium primary circuit and the potassium secondary circuit (refs. 6 and 7). The leak in the coaxial T-111 alloy tube boiler was traced to a crack in the weld nugget of the 0.375-inch-outside-diameter by 0.062-inch-wall inner tube, as shown in figure VIII-22. During operation of the boiler ( $\sim 2100^{\circ}$  F), lithium flowed in one direction in the annulus between the two tubes, and potassium flowed in the opposite direction within the inner tube. After the cracked segment of tube was cut from the boiler, stepwise removal of material from the surface of a longitudinal section through the crack, to a depth of 0.006 inch, clearly showed the crack to be intergranular (fig. VIII-23) with entire grains delineated as the result of grain boundary separation (fig. VIII-24). The appearance of grain boundary separation suggests some action of lithium which has penetrated into the crack. Although the intermittent voids in the grain boundaries are similar in appearance to those observed as a result of attack by lithium, the crack is not believed to have been initiated by a corrosion mechanism, since corrosion by lithium would not be expected in T-111 having an oxygen concentration as low as that (40 ppm) of the as-received boiler tube.

To determine the possibility of reusing the boiler tube (except for that section containing the leak), specimens from the boiler tube in the vicinity of the crack were removed and submitted for metallographic examination, microprobe evaluation, and chemical analyses for major metallic and interstitial elements. The ductility of the T-111 alloy was determined qualitatively by tube flattening tests.

Results of the various analyses (ref. 5) indicated no contamination of the T-111 alloy material had occurred, and no cracking was observed during flattening of the tubes. Further, interstitial analyses of the T-111 alloy weld quality assurance specimens welded before and after the welding of the boiler tube, as part of NSP specification 03-0025-00-A, indicated no contamination occurred during welding of the boiler tube.

Although the cause of the crack is not evident, it is believed that the most likely explanation would be that straining of the weld nugget while it was hot might have caused grain boundary separation similar to that which occurred during butt welding of tubes (described in the section Butt weld joint). Propagation of such a crack could have occurred during subsequent bending to form the helically shaped boiler.

### Spot Tack Welding

Prior to the postweld vacuum annealing of T-111 alloy at  $2400^{\circ}$  F for 1 hour, Cb-1Zr alloy foil was wrapped around two tubular T-111 alloy assemblies and

fastened in place by spot tack welding. After heat treatment, cracking was observed in the T-111 alloy in the vicinity of the tack welds. No cracking had been observed previously in spot tack welding of Cb-1Zr foil to T-111 alloy using a molybdenum (Mo) tipped welding electrode. The subassemblies exhibiting cracks, however, had been spot welded using a copper (Cu) electrode. The utilization of the Cu electrode was believed to be responsible for the observed failures. The highlights of an investigation to identify and eliminate the cause of cracking are summarized in the next paragraph (ref. 8).

A study was initiated to determine the effects of electrode materials (Cu, Mo, and Cb-1Zr), spot welding parameters, surface conditions and stresses in the tubing, and subsequent 2400<sup>o</sup> F, 1-hour vacuum exposures on the cracking tendencies of various sizes of T-111 alloy tubing. Preliminary results showed that only spot welds made with a Cu electrode produced areas in the T-111 alloy tubing that were subject to failure upon subsequent heat treatment. Metallographic examination of as-spot-welded samples revealed no microcracking or interdiffusion regardless of the electrode material, as shown in the sample in figure VIII-25(a), in which T-111 alloy was spot welded with a Cu electrode. Copper deposited on the T-111 alloy surface, from welding with that electrode, is shown in the photomicrograph. When the spot tack welded samples were heated to 2400<sup>o</sup> F for 1 hour, microcracking was seen only in those specimens which were spot tack welded using a copper electrode (figs. VIII-25(b) and VIII-26). (The light colored grain boundary phase was identified later in similar samples as a Cu/Hf phase.) The difference in the magnitude of the cracks between the 1.5- and 0.375-inch-diameter tubes may be due to differences in residual stresses resulting from straightening operations. Further tests in which Cu was simply placed on T-111 alloy and heated indicated that spot welding with a copper electrode only served to provide a source of copper for subsequent reaction with T-111 alloy in heating to 2400<sup>o</sup> F. The results of one such test are given in figure VIII-27. A Cb-1Zr foil boat (consisting of two layers of 0.002-in. -thick foil) containing copper was lightly spot tack welded (using a Mo electrode) to a 1.5-inch-diameter by 0.100-inch-wall T-111 alloy tube section. When the assembled ring section was heated to 2400<sup>o</sup> F, the Cu penetrated through the Cb-1Zr foil layers and completely through the tube wall producing the crack shown in figure VIII-27. Additional metallographic studies and microprobe analyses of the test specimens indicated that a Cu/Hf reaction at grain boundaries during heat treatment, in combination with some residual stress, caused intergranular failures in T-111 alloy. The one test sample in which microprobe analyses positively identified a Cu/Hf phase in the T-111 alloy grain boundaries is shown in figure VIII-28(a). A T-111 alloy sheet (0.063-in. -thick) sample was notched and bent (with the notched side in tension) and Cu placed in the notch. Then the sample was heated at 2400<sup>o</sup> F

for 1 hour. Microcracking was found at symmetrical locations on each side of the notch, as illustrated in figure VIII-28(b). An identical notched, bent, and heat treated sheet specimen without Cu did not exhibit microcracking. The notched sheet sample, heated to 2400<sup>0</sup> F, with Cu in the notch was examined with a microprobe at the failure location and at random positions throughout the specimen. The phase present at sample grain boundaries next to the failure contained Hf concentrations significantly higher than the matrix; Cu was also found in these grain boundaries. Cu was not present within the grains of the sample nor in grain boundaries remote from the failure. These results indicated that a Cu/Hf reaction, at grain boundaries originally high in Hf content, was the probable major factor causing Cu penetrations and the resultant failures.

### SUMMARY OF RESULTS

A summary of the cracking incidents observed during the fabrication of T-111 alloy loop components, their possible causes, and preventative measures is given in the following table:

Cracking problem during -	Probable cause or contributing cause(s)	Preventative measures
Cutting and forming	Possible nacent hydrogen embrittlement from coolant or binder in cutting wheel	Use bandsaw or tube cutters as appropriate. Use acceptable cutoff wheel and grind away cut surface to assure no cracks are present.
Machining and forming	Residual stresses and possible hydrogen embrittlement	Use appropriate vacuum stress relief anneal.
EB and TIG welding	Low strength of grain boundaries relative to matrix Residual stresses	Design to avoid multipass welds. Use alloy overlay technique or intermediate stress relief for multipass welds when necessary. Use EB welding as much as possible. Stay on low Hf side of specification and use double arc melted alloy. Stress relieve machined parts before welding. Provide adequate mechanical support for welding.
Spot tack welding	Cu High Hf concentration in grain boundaries	Avoid use of Cu electrode; use Mo electrode

An examination of this table clearly shows that the problems of T-111 alloy cracking are remediable. Although nondestructive evaluation of machined parts or welded joints is not mentioned in the table, X-ray, fluorescent penetrant, helium mass spectrometer leak checking, and visual inspection are recommended and were performed, as appropriate, to prevent installation of cracked components into test systems. GE-NSP performs visual inspections (at magnifications up to 30) of all welded joints. Also, thermal cyclic testing has been used as a proof test of critical components or assemblies containing welds.

It is obvious from the table that there is still much to be learned about the basic mechanisms underlying cracking in T-111 alloy. Some general observations that are particularly noteworthy when considering cracking mechanisms are (1) whenever metallography has been performed, cracking has been found to be intergranular in nature; (2) only multipass weld cracking has been found to be readily reproducible; (3) cracking related to cutting and machining has been particularly difficult to reproduce; and (4) the intermittent appearance of cracking may indicate local contamination (e. g., with copper). It should also be noted that the mechanism of crack initiation and brittle crack propagation at room temperature has not been explained. Until the underlying crack-provoking mechanisms can be found and corrective action taken, those working with T-111 alloy should avoid the obvious pitfalls described in this paper and be alert to potential problems not yet uncovered.

In conclusion, there are three major points to be kept in mind, particularly by those who hasten to draw pessimistic conclusions concerning the use of T-111 alloy in space systems: (1) T-111 alloy is still in the development stage with respect to manufacturing complex hardware, and some difficulties and a learning period should be expected; (2) cracking observed to date is remediable; and (3) the T-111 alloy Rankine system corrosion test loop has now completed over 3500 hours of continuous, stable, trouble-free operation at a maximum lithium temperature of 2250° F.

## REFERENCES

1. Harrison, R. W.: Advanced Refractory Alloy Corrosion Loop Program. Rep. GESP-258, General Electric Co. (NASA CR-72560), May 16, 1969.
2. Turner, F. S.: Tantalum Alloy Tubing Development Program. Rep. RTD-8-109(1), Allegheny Ludlum Steel Corp., Oct. 1963.
3. Engel, L. B., Jr.: Determination of Biaxial Creep Strength of T-111 Tantalum Alloy. Rep. GESP-237, General Electric Co. (NASA CR-72541), June 17, 1969.

4. Begley, R. T.; and Godshall, J. L.: Some Observations on the Role of Grain Boundaries in High Temperature Deformation and Fracture of Refractory Alloys. Presented at the AIME Symposium on the Physical Metallurgy of Refractory Metals, French Lick, Ind., Oct. 3-5, 1965.
5. Brandenburg, G. P.: Evaluation of T-111, Ta-10W, and Cb-1Zr Alloy Filler Materials in T-111 Alloy Multipass Tube Welds. Rep. GESP-385, General Electric Co., 1970.
6. Harrison, R. W.: Advanced Refractory Alloy Corrosion Loop Program. Rep. GESP-182, General Electric Co. (NASA CR-72483), 1968.
7. Harrison, R. W.: Advanced Refractory Alloy Corrosion Loop Program. Rep. GESP-189, General Electric Co. (NASA CR-72505), Mar. 20, 1969.
8. Thompson, S. R.: Investigation of the Cracking in T-111 Alloy Tubing Associated with Spot Tack Welding. Rep. GESP-384, General Electric Co., 1970.



TABLE VIII-1. - T-111 ALLOY PROCURED BY  
GE-NSP FOR NASA CONTRACT NAS 3-6474,  
ADVANCED REFRACTORY ALLOY

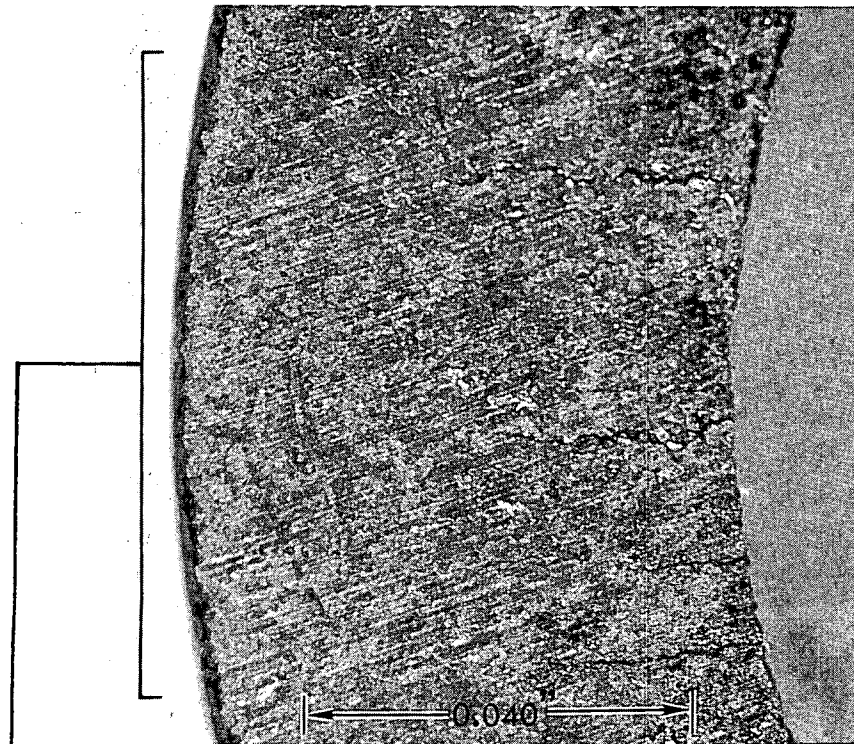
CORROSION LOOP

Item and size, in.	Weight, lb
<b>Rod</b>	
0.250 diam	1
0.500 diam	11
0.625 diam	5
1.000 diam	40
1.125 diam	10
1.500 diam	13
2.000 diam	85
2.500 diam	93
3.125 diam	<u>74</u>
	332
<b>Bar</b>	
1.0 by 1.0	30
1.0 by 2.0	<u>115</u>
	145
<b>Wire</b>	
0.062 diam	13
0.094 diam	8
0.125 diam	<u>31</u>
	52
<b>Foil, sheet, or plate</b>	
0.005 by 3.5	2
0.009 by 3.5	1
0.035 by 1.0	1
0.040 by 12.0	29
0.125 by 6.0	5
0.500 by 6.125	<u>41</u>
	79
<b>Tube</b>	
0.375 o.d. by 0.065 wall	66
1.00 o.d. by 0.065 wall	104
2.25 o.d. by 0.375 wall	40
2.50 o.d. by 0.450 wall	46
3.00 o.d. by 0.375 wall	50
3.25 o.d. by 0.250 wall	40
3.25 o.d. by 0.500 wall	<u>73</u>
	419
<b>Total</b>	<b>1027</b>

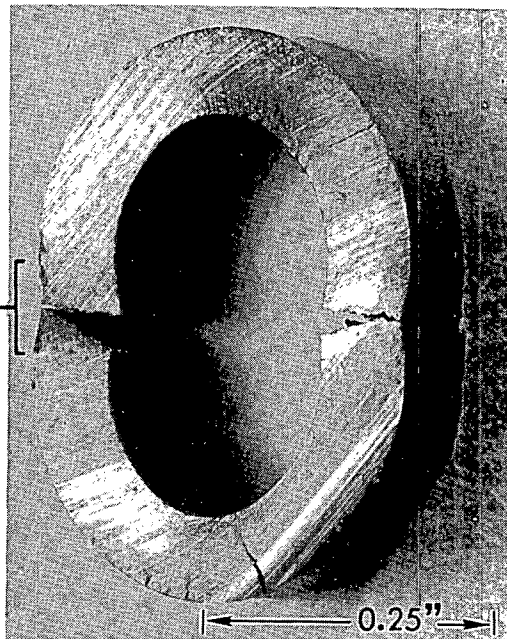
TABLE VIII-2. - T-111 ALLOY PROCURED BY  
GE-NSP FOR NASA CONTRACT NAS 3-9426,  
DEVELOPMENT OF A SINGLE TUBE

POTASSIUM BOILER

Item and size, in.	Weight, lb
<b>Rod</b>	
0.125 diam	4
0.250 diam	4
0.690 diam	10
0.750 diam	9
1.000 diam	38
1.375 diam	128
1.500 diam	34
2.000 diam	38
2.250 diam	77
2.500 diam	303
3.063 diam	389
3.688 diam	121
4.438 diam	163
4.625 diam	<u>176</u>
	1494
<b>Wire</b>	
0.062 diam	10
0.094 diam	15
0.125 diam	<u>26</u>
	51
<b>Foil, sheet, or plate</b>	
0.005 by 3.5	1
0.005 by 5.5	1
0.040 by 20.5	55
0.063 by 6.0	3
0.100 by 9.0	23
0.125 by 22.0	51
0.250 by 8.0	105
0.400 by 6.0	<u>44</u>
	283
<b>Tube</b>	
0.250 o.d. by 0.050 wall	16
0.375 o.d. by 0.065 wall	14
0.500 o.d. by 0.075 wall	18
0.625 o.d. by 0.008 wall	8
0.690 o.d. by 0.045 wall	13
0.750 o.d. by 0.04 wall	17
0.875 o.d. by 0.100 wall	363
1.325 o.d. by 0.100 wall	70
1.500 o.d. by 0.100 wall	190
2.375 o.d. by 0.220 wall	79
4.386 o.d. by 1.343 wall	180
4.420 o.d. by 0.537 wall	92
4.424 o.d. by 1.362 wall	184
4.625 o.d. by 0.225 wall	45
4.625 o.d. by 0.275 wall	55
4.625 o.d. by 0.409 wall	<u>79</u>
	1423
<b>Total</b>	<b>3251</b>

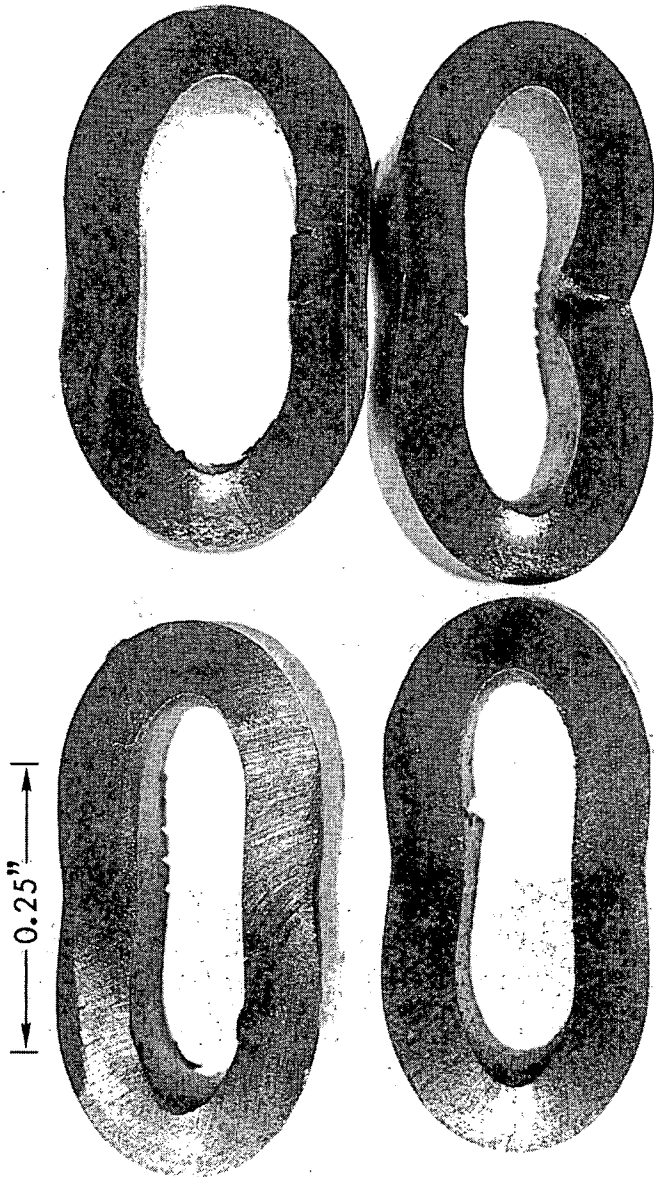


(a) Before bending.



(b) After bending.

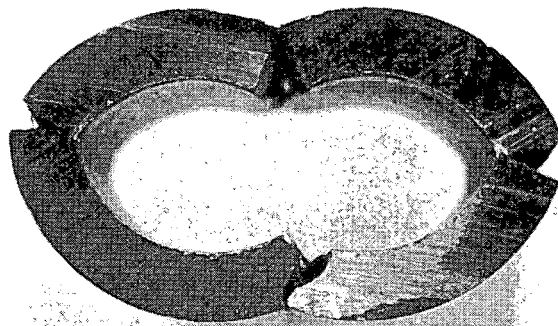
Figure VIII-1. - T-111 alloy tubing (0.375-in. by 0.062-in. wall) after cutting with water-cooled alumina abrasive cutoff wheel.



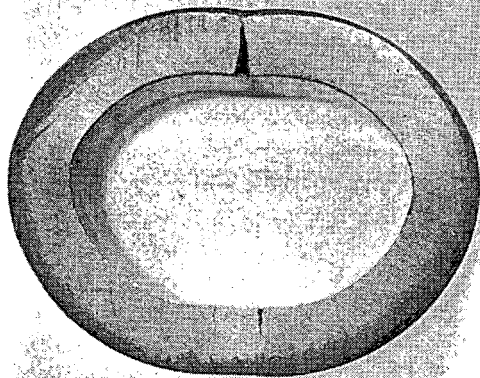
(a) Bent following "slow" cutting.

(b) Bent following "fast" cutting.

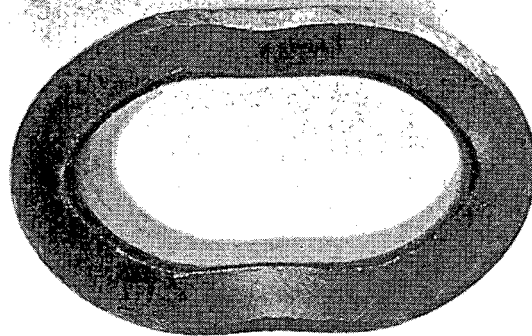
Figure VIII-2. - T-111 alloy tubing (0.375-in. by 0.062-in. wall) showing effect of cutting speed on cracking. Tubing was cut using alumina abrasive cutoff wheel.



(a) Bent as cut.



(b) 0.040 Inch ground off cut surfaces prior to bending.



(c) 0.10 Inch ground off cut surfaces prior to bending.

0.25"

Figure VIII-3. - Effect of removing cracked surface prior to bending of T-111 alloy tubing (0.375-in.) cut with alumina cutoff wheel. Tubing was supplied by ORNL from tubing described in Westinghouse Report WANL-PR(N)-004.

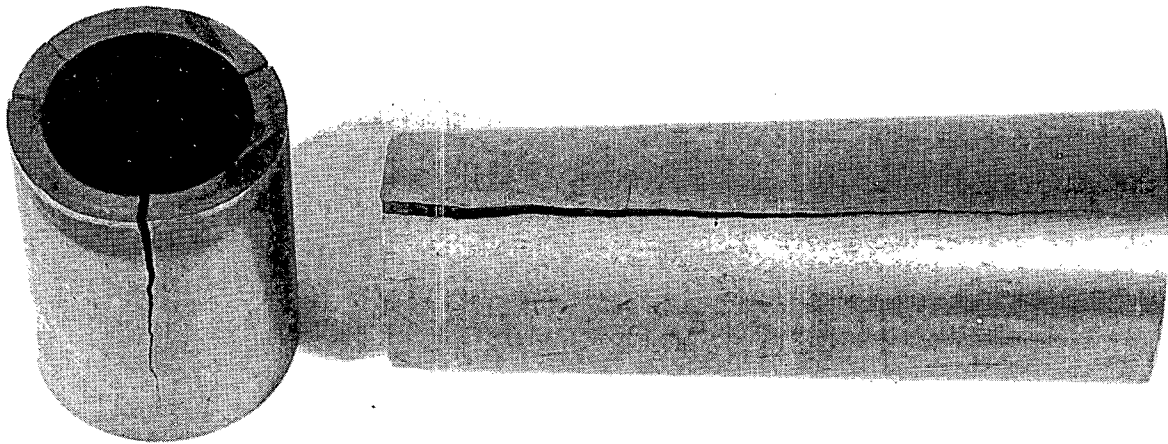
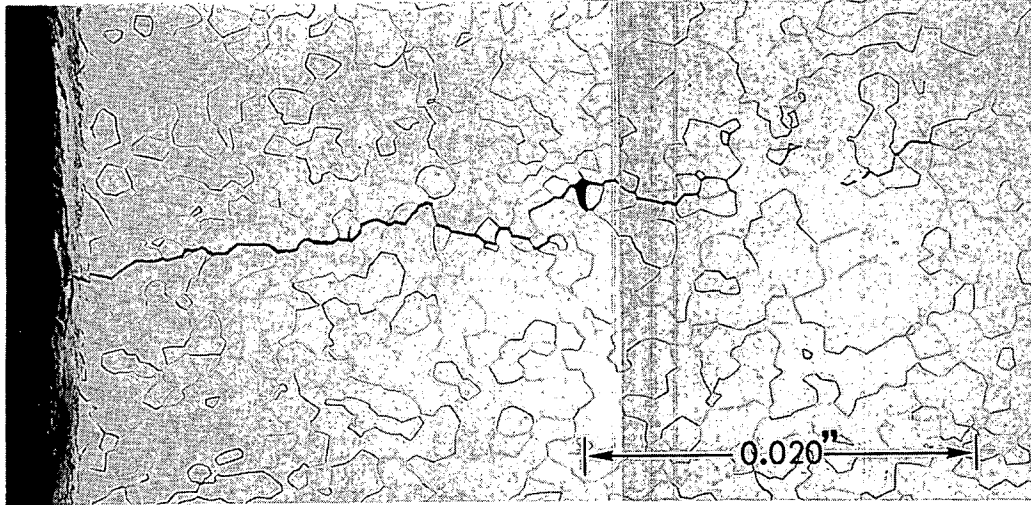


Figure VIII-4. - Cracks in T-111 alloy tube hollows (2-in. by 0.25-in. wall) observed during initial tube reducing. The cracks apparently propagated from shallow cracks introduced when hollows were cut with abrasive cutoff wheel.



(a) 1.5-Inch-diameter rod.



(b) 0.5-Inch-diameter rod.

Figure VIII-5. - Intergranular cracks in T-111 alloy rod cut with water-cooled alumina abrasive cutoff wheel. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .

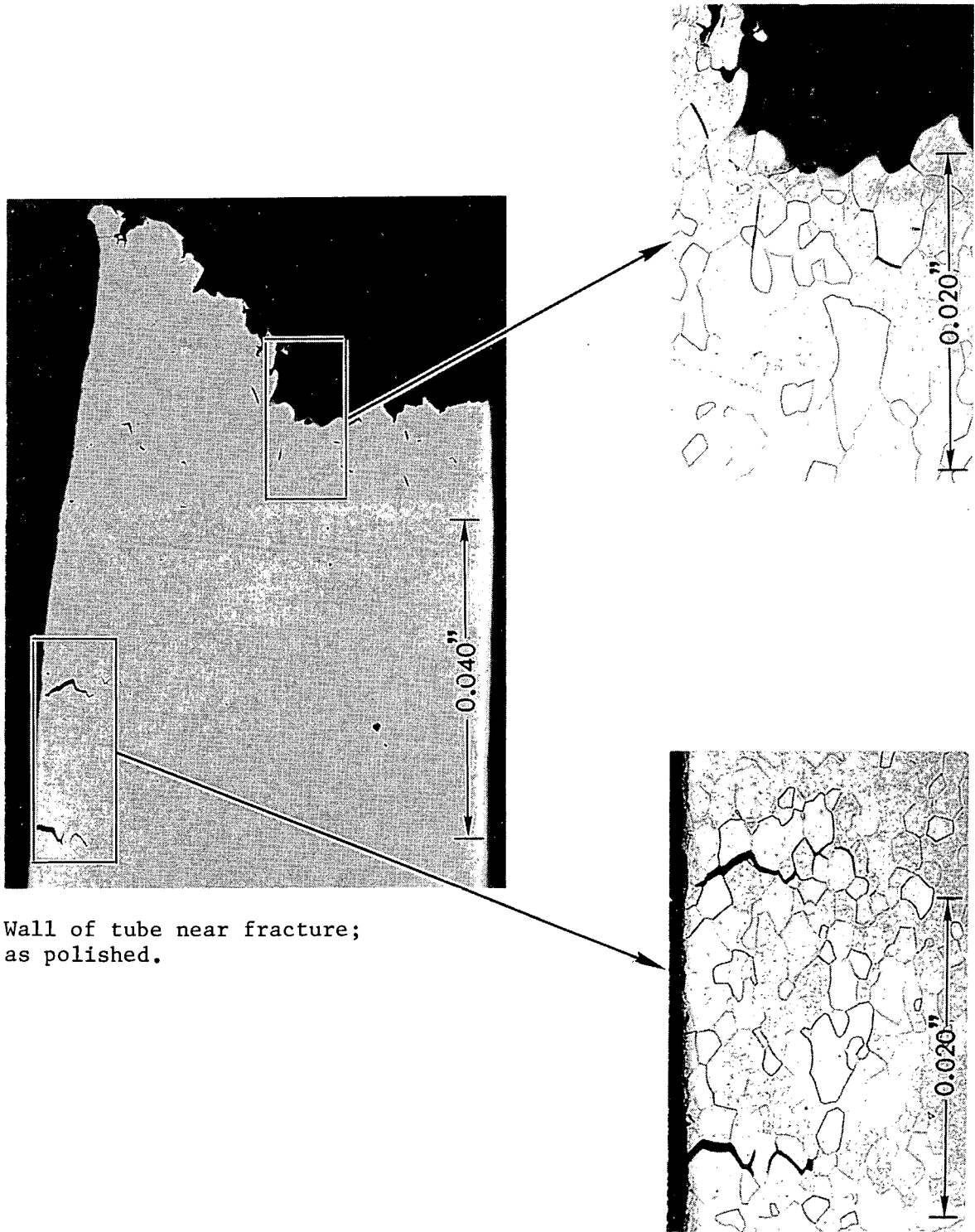


(a) No stress relief before bending.



(b) 2400<sup>o</sup> F, 1-hour stress relief before bending.

Figure VIII-6. - Machined T-111 alloy thermocouple wells after bending.



Wall of tube near fracture;  
as polished.

Figure VIII-7. - Longitudinal sections of fractured T-111 alloy thermocouple well after bending showing intergranular cracking in the wall on tension side of bend. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .



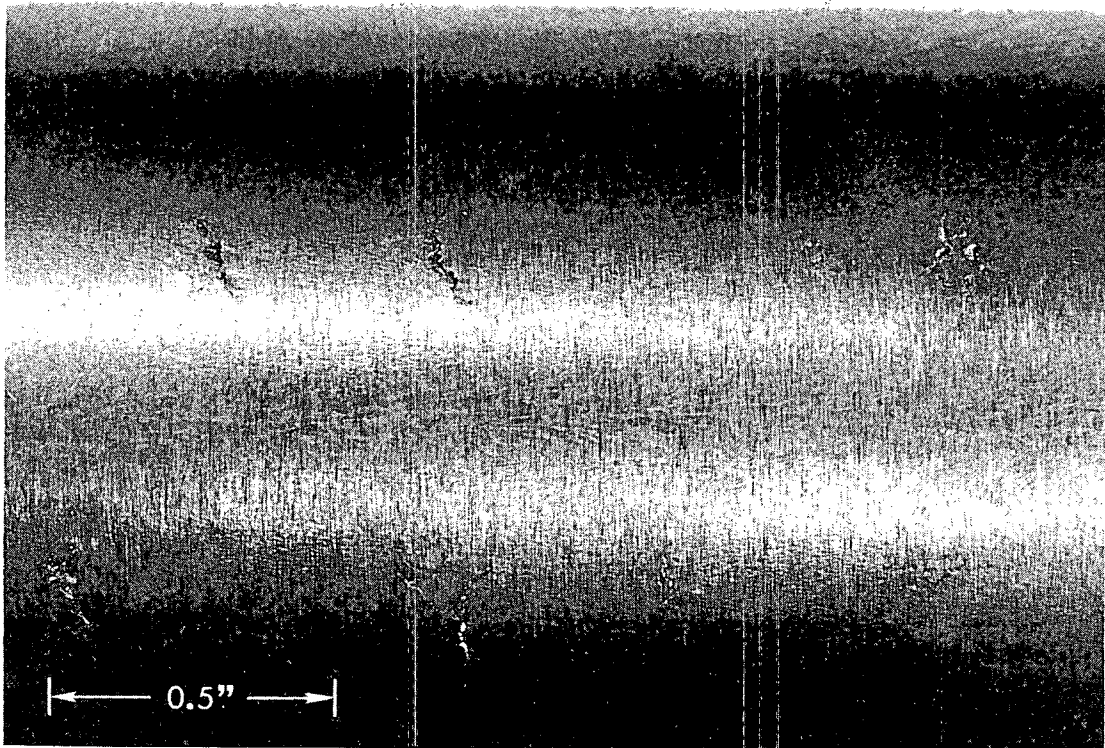
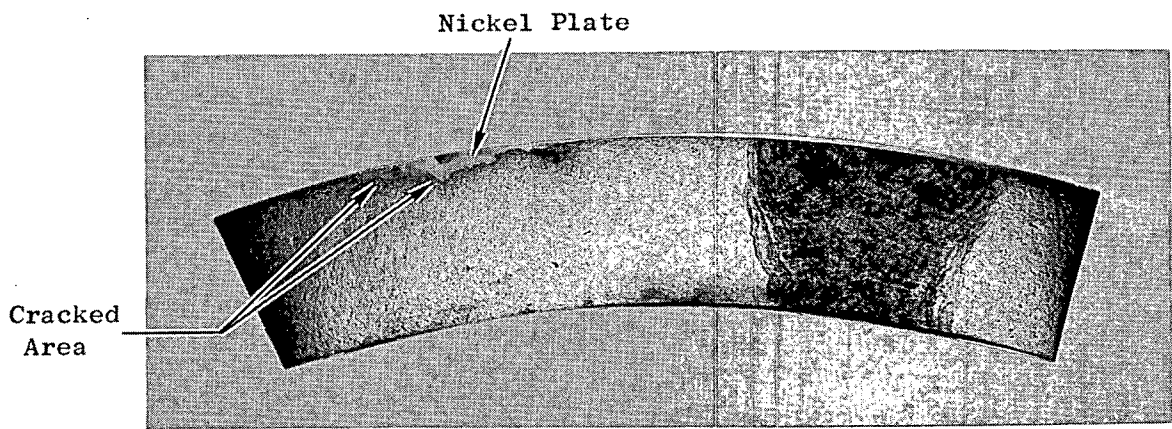
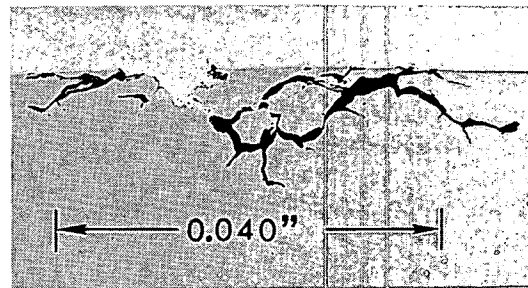


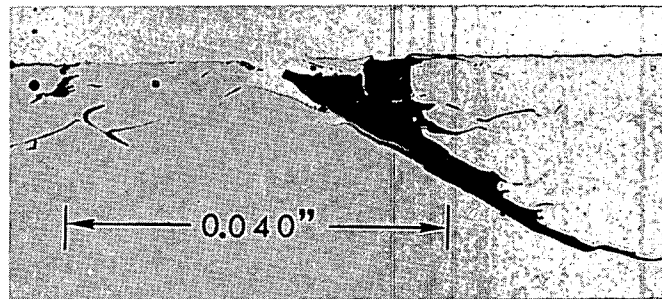
Figure VIII-8. - Transverse cracks in the heat-affected zone adjacent to weld on outside diameter of 1.5-inch-outside-diameter by 0.100-inch-wall EB welded and reworked T-111 alloy tube.



(a) Transverse section; etched.



(b) Transverse section; as polished.

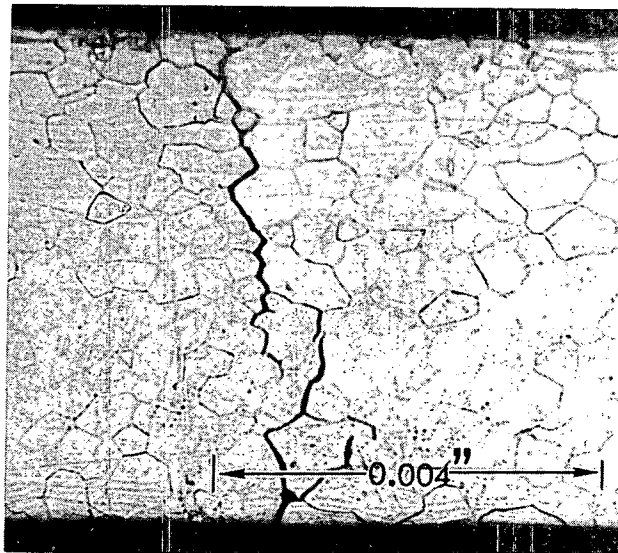


(c) Longitudinal section; as polished.

Figure VIII-9. - Sections through cracks in 1.5-inch-outside-diameter by 0.100-inch-wall EB welded and reworked T-111 alloy tube after final anneal at 3000<sup>o</sup> F for 1 hour. Etchant: 30 grams NH<sub>4</sub>F, 50 milliliters HNO<sub>3</sub>, 20 milliliters H<sub>2</sub>O.

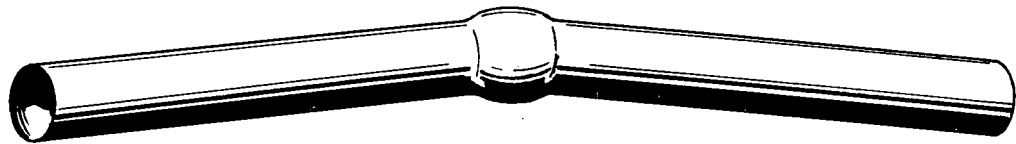


(a) Cracks (arrows).



(b) Transverse section through one cracked area of T-111 alloy diaphragm; etched.

Figure VIII-10. - Intergranular cracks in T-111 alloy pressure transducer diaphragm. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .



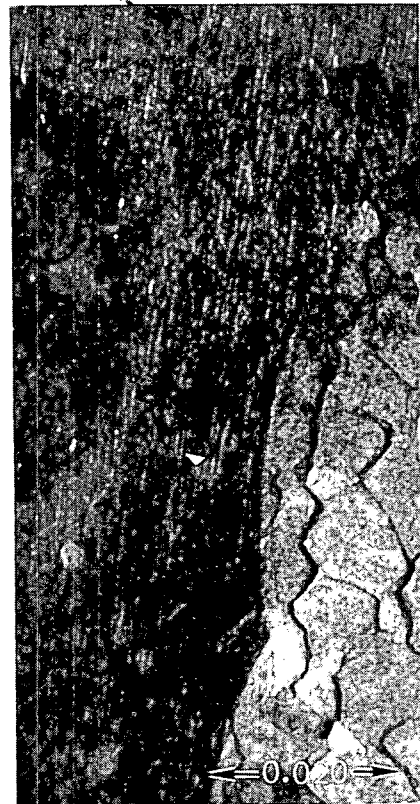
(a) Sagged butt weld joint.



(b) Joint after straightening.



(c) Intergranular cracking.



(d) Grain boundary sliding.

Figure VIII-11. - Cracking in T-111 alloy tube-to-tube (0.375-in. by 0.065-in. wall) butt weld joint. Cracking occurred during attempts to straighten tube after it had sagged during welding.

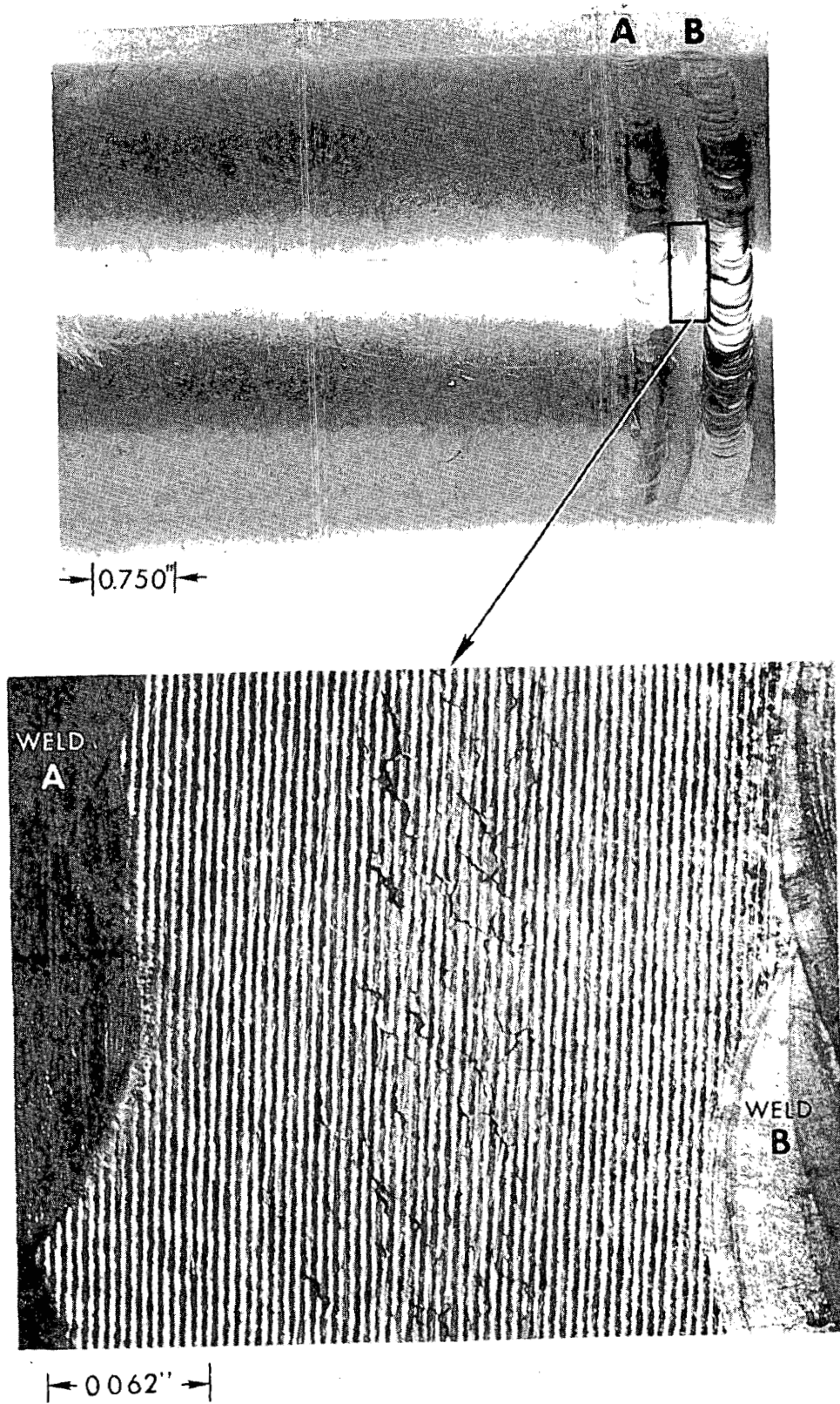


Figure VIII-12. - Typical area of cracking between GTA welds of T-111 alloy EM pump duct.

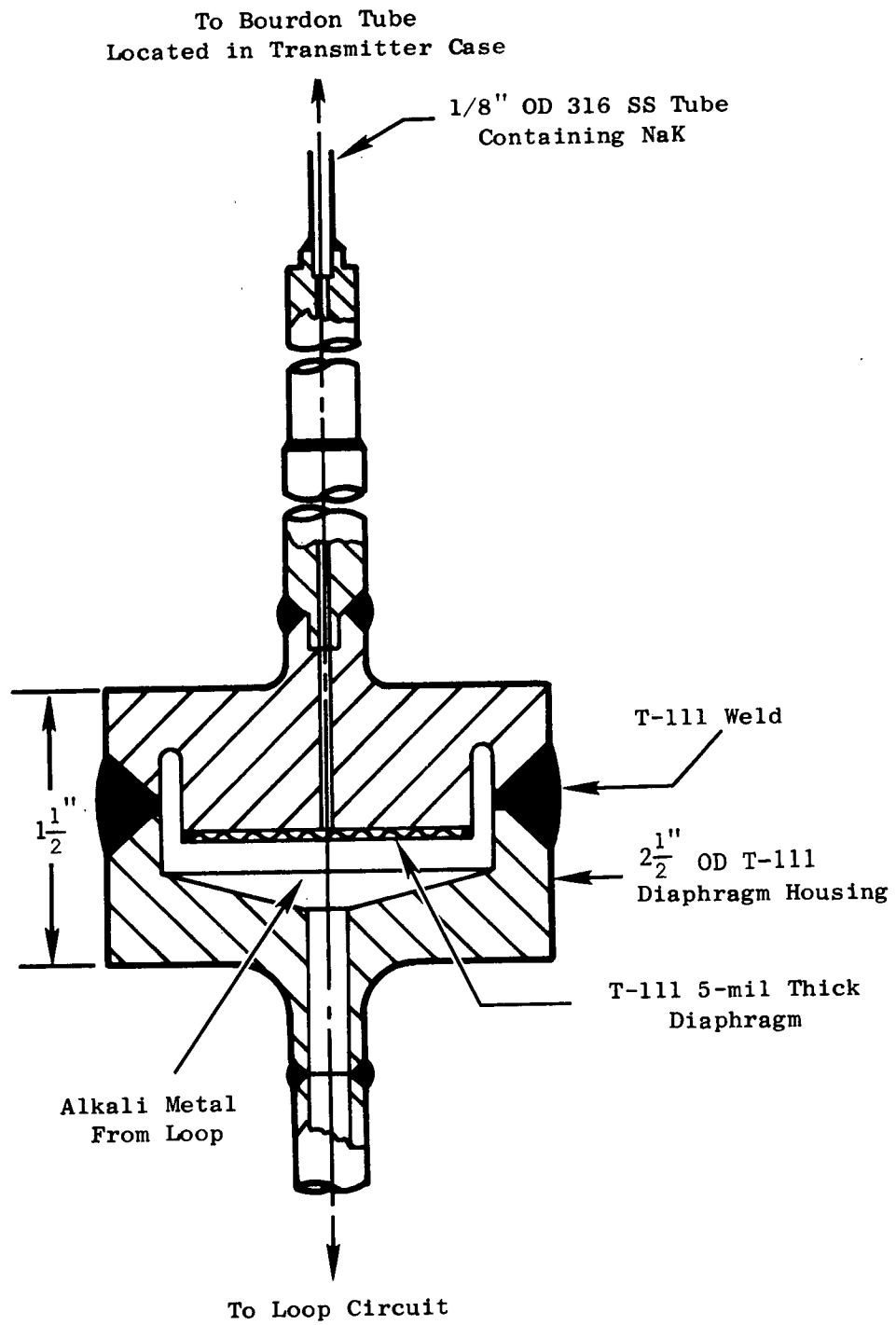


Figure VIII-13. - Full section view of Taylor pressure transducer showing its functional relation to loops of boiler test rig.

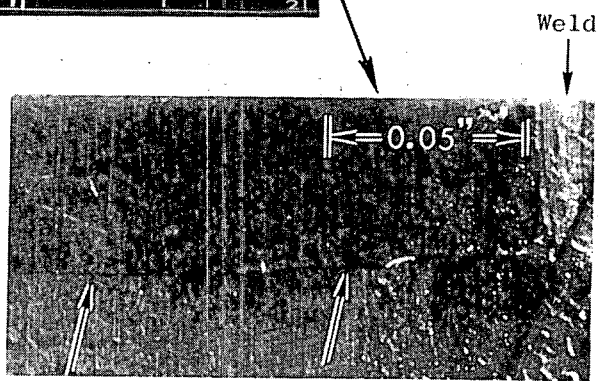
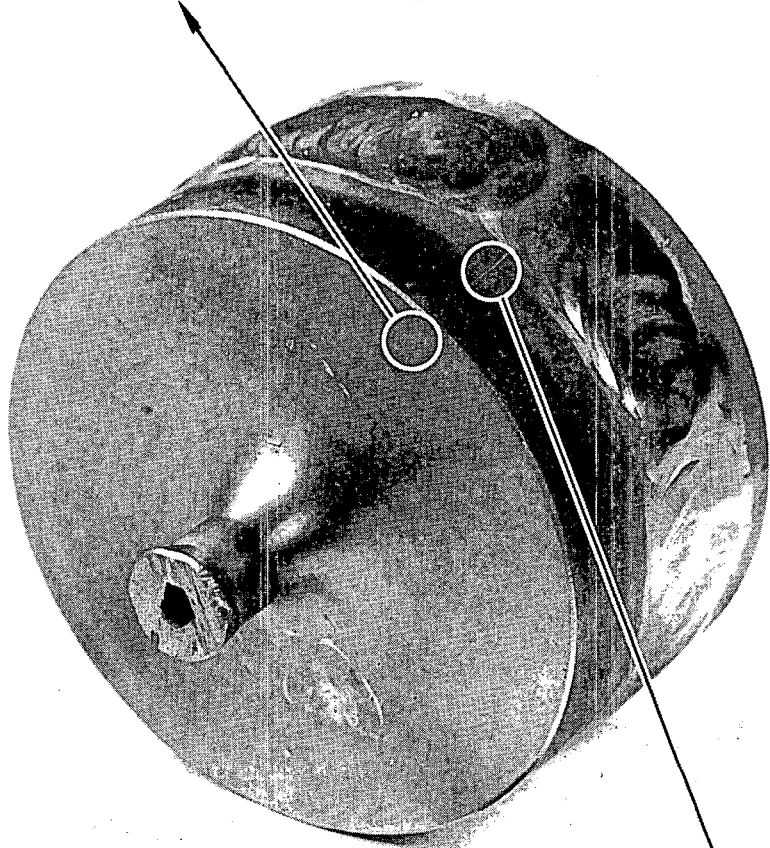
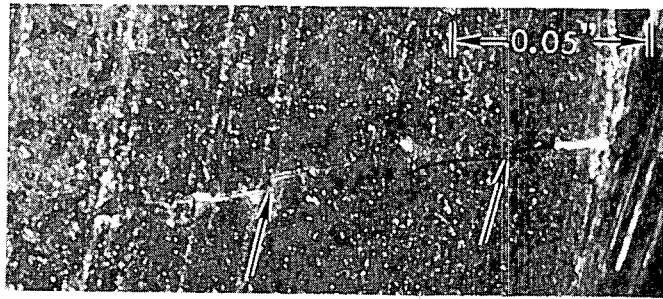


Figure VIII-14. - Taylor absolute pressure transducer 14 showing large crack (see arrows in enlargements of cracked areas) extending from edge of weld nugget to flat surface of bottom flange.

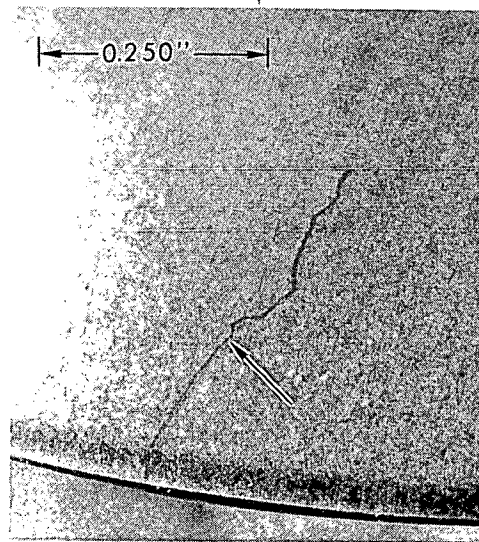
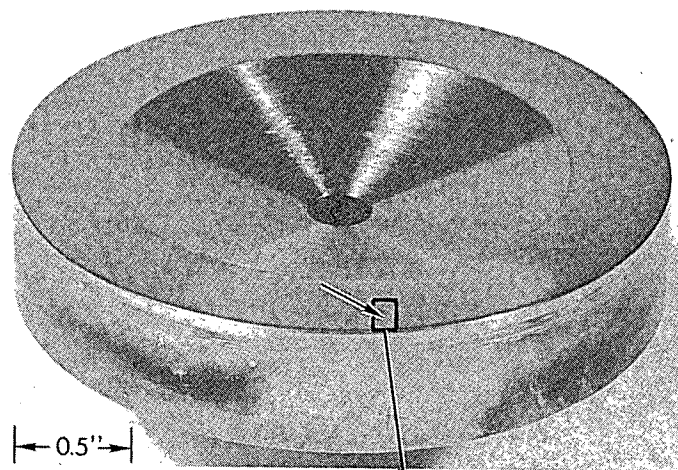
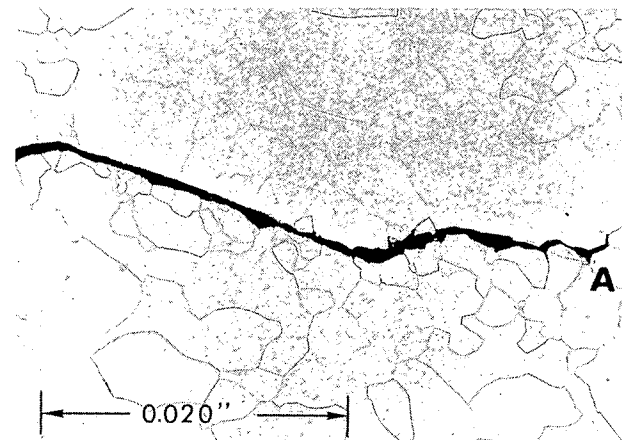
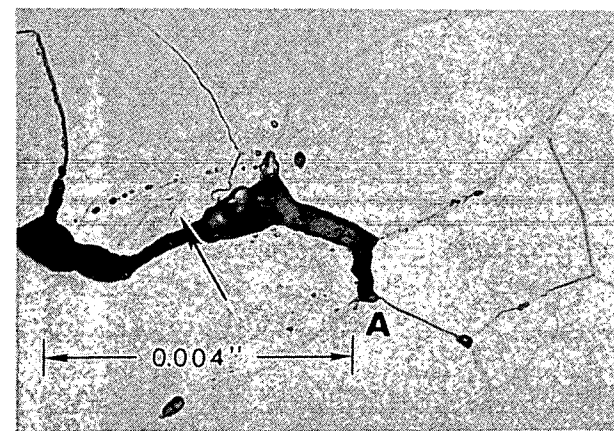


Figure VIII-15. - Bottom flange of pressure transducer 14 after removal of weld nugget. Enlargement of inside facing flange shows depth of crack propagation.



(a)



(b)

Figure VIII-16. - Photomicrographs of end A of crack shown in Figure VIII-15 at two magnifications. The arrow in photomicrograph (b) shows presence of highly distorted grain near end of crack. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .



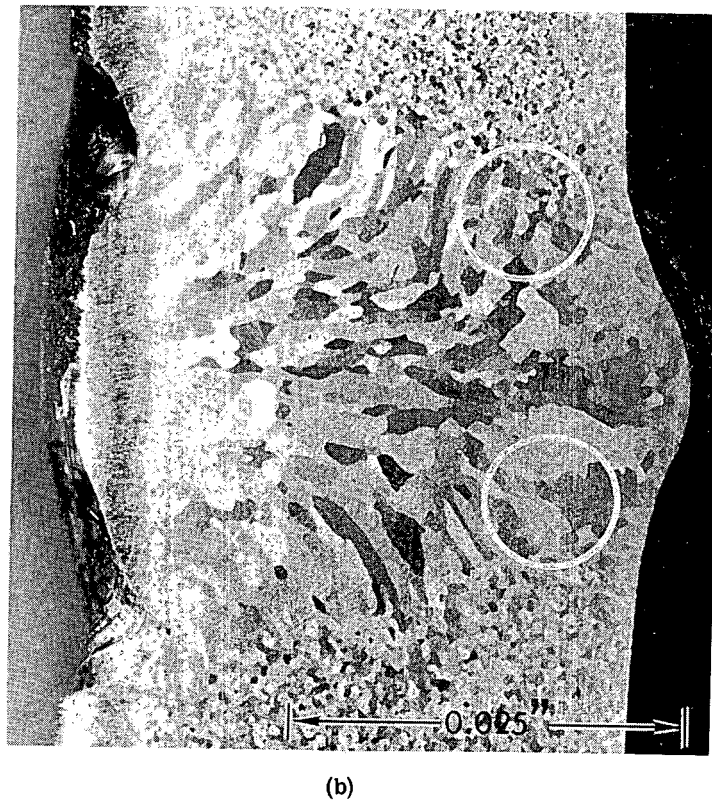
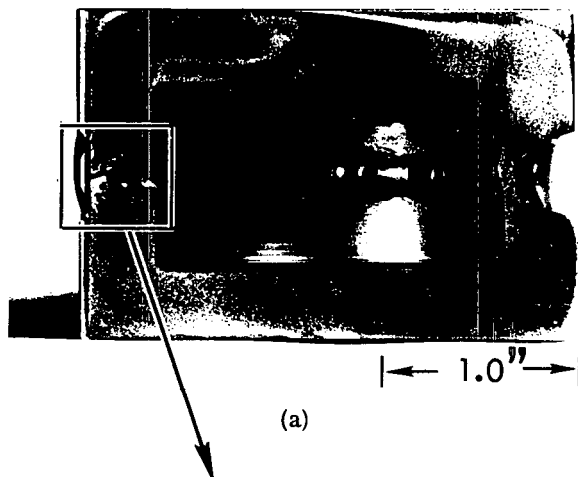
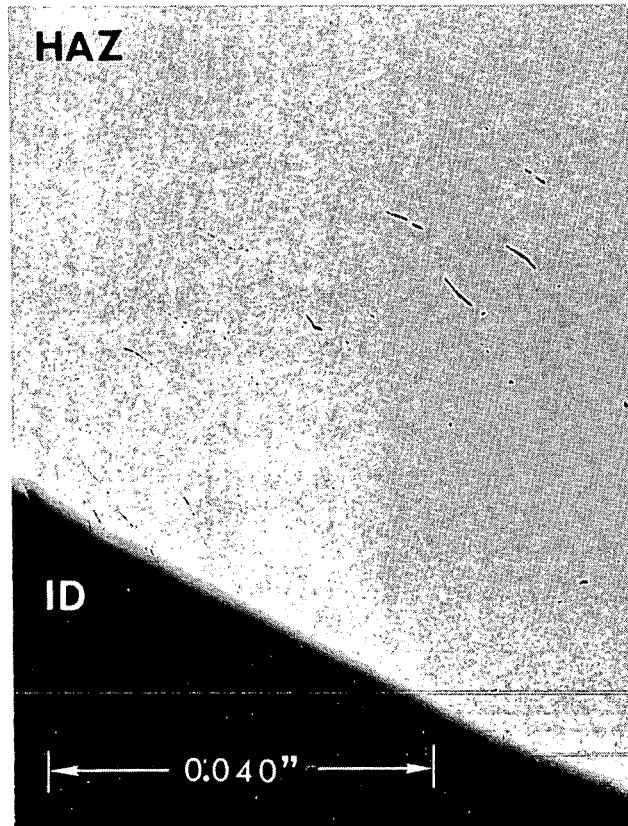
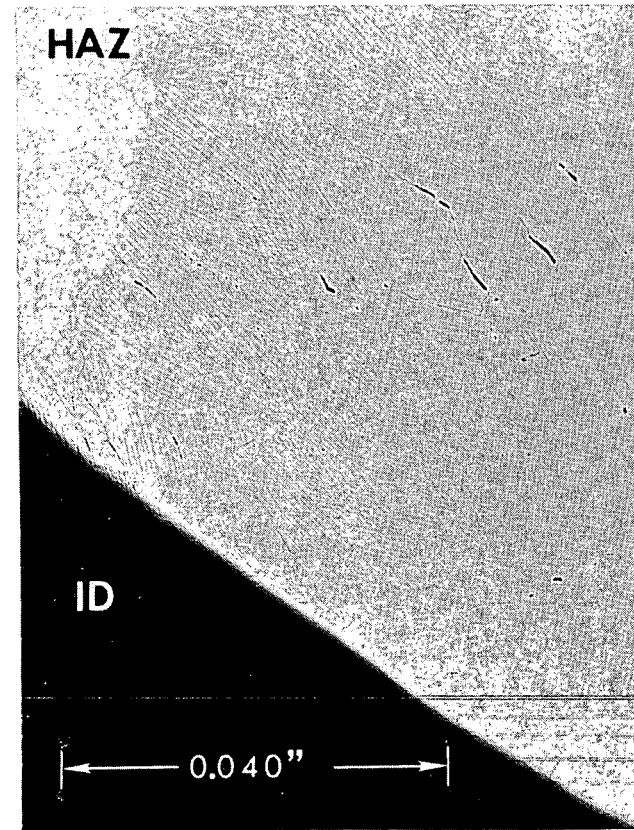


Figure VIII-17. - Multipass GTA weld sample formed by joining two 2.5-inch-outside-diameter by 0.375-inch-wall T-111 alloy closed end tubes with three different filler wires, T-111 alloy, Cb-1Zr, and Ta-10W. The circled areas in (b) indicate approximate locations of most cracking observed.

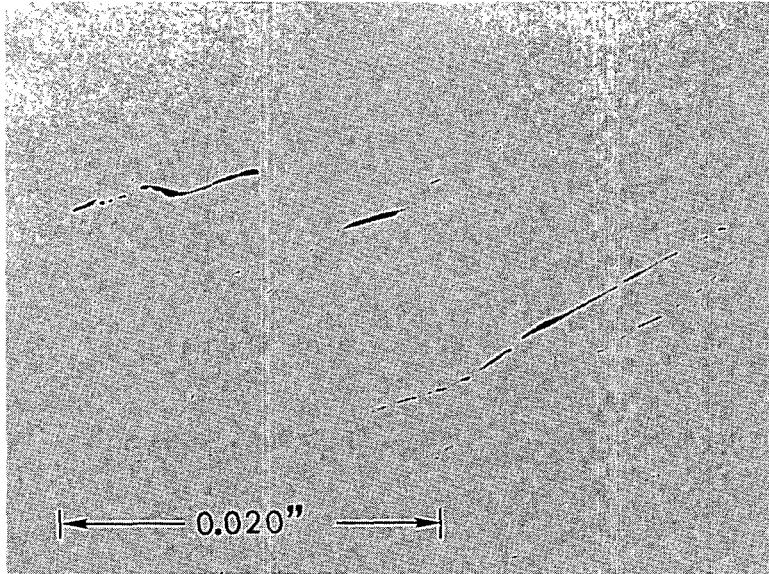


As Polished

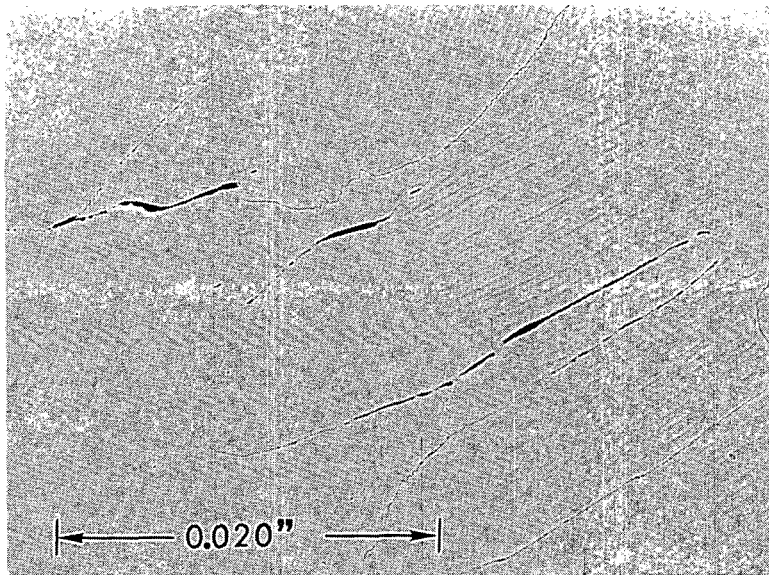


Lightly Etched

Figure VIII-18. - Transverse microstructure near edge of weld nugget in three-pass T-111 alloy weld showing cracking typical of this area. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .

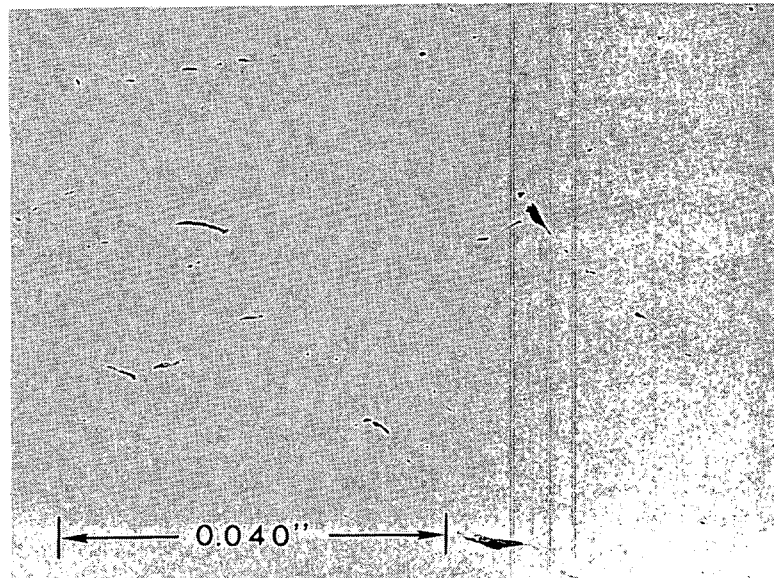


As Polished

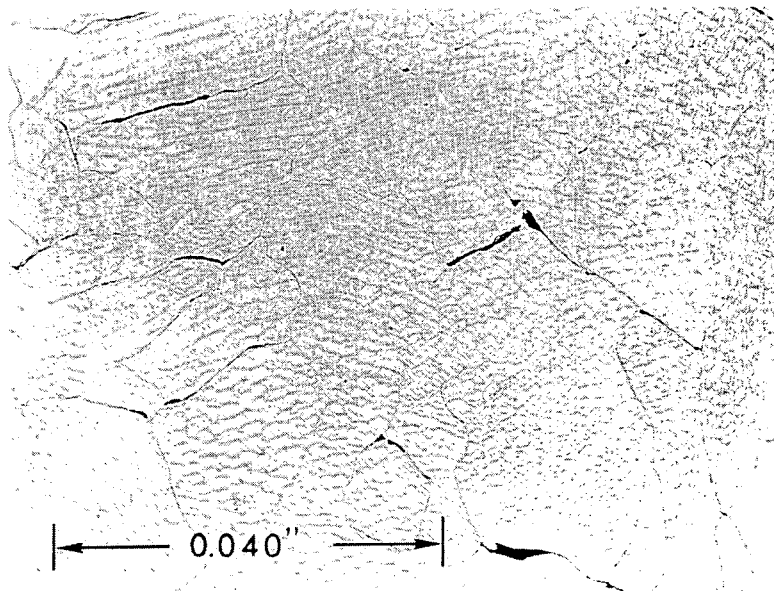


Lightly Etched

Figure VIII-19. - Transverse microstructure of T-111 alloy fusion pass of multipass T-111 alloy tube weld using T-111 alloy filler showing grain boundary separation. Etchant: 30 grams  $\text{NH}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .

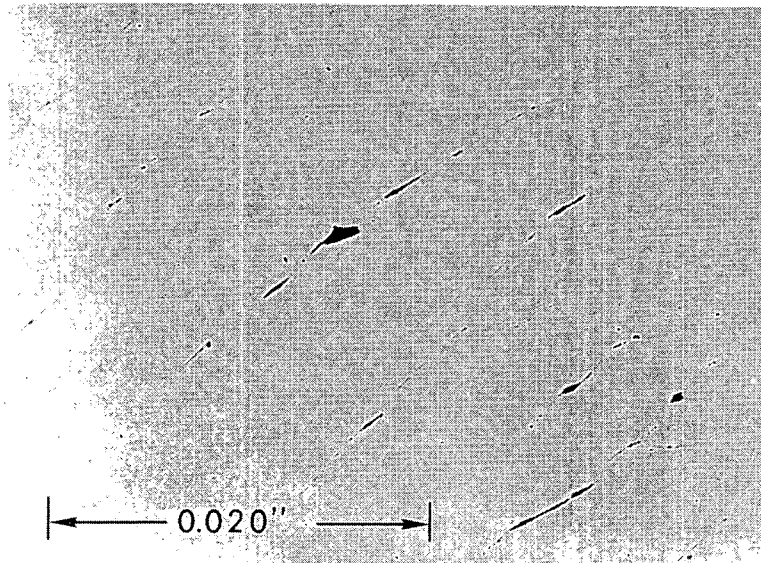


As Polished

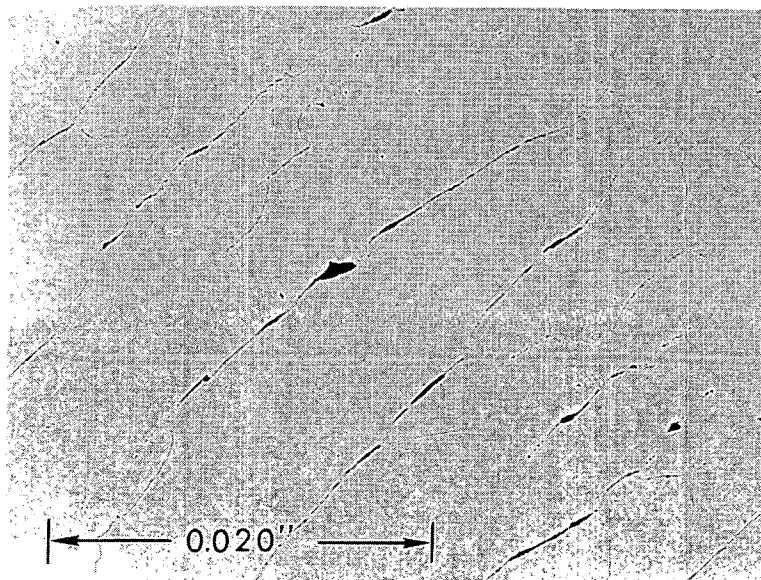


Lightly Etched

Figure VIII-20. - Transverse microstructure of T-111 alloy fusion pass of multipass T-111 alloy tube weld using Cb-1Zr alloy filler showing grain boundary separation. Etchant: 30 grams  $\text{HN}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ . X100.



**As Polished**



**Lightly Etched**

**Figure VIII-21. - Transverse microstructure of T-111 alloy fusion pass of multipass T-111 alloy tube weld using Ta-10W alloy filler showing grain boundary separation. Etchant: 30 grams  $\text{HN}_4\text{F}$ , 50 milliliters  $\text{HNO}_3$ , 20 milliliters  $\text{H}_2\text{O}$ .**

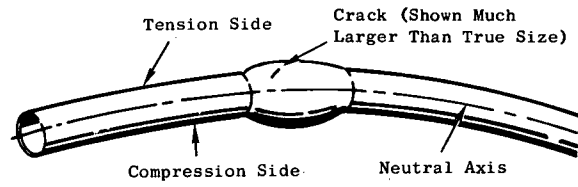


Figure VIII-22. - Sketch showing location of crack in weld nugget of T-111 alloy tube removed from corrosion test loop boiler.

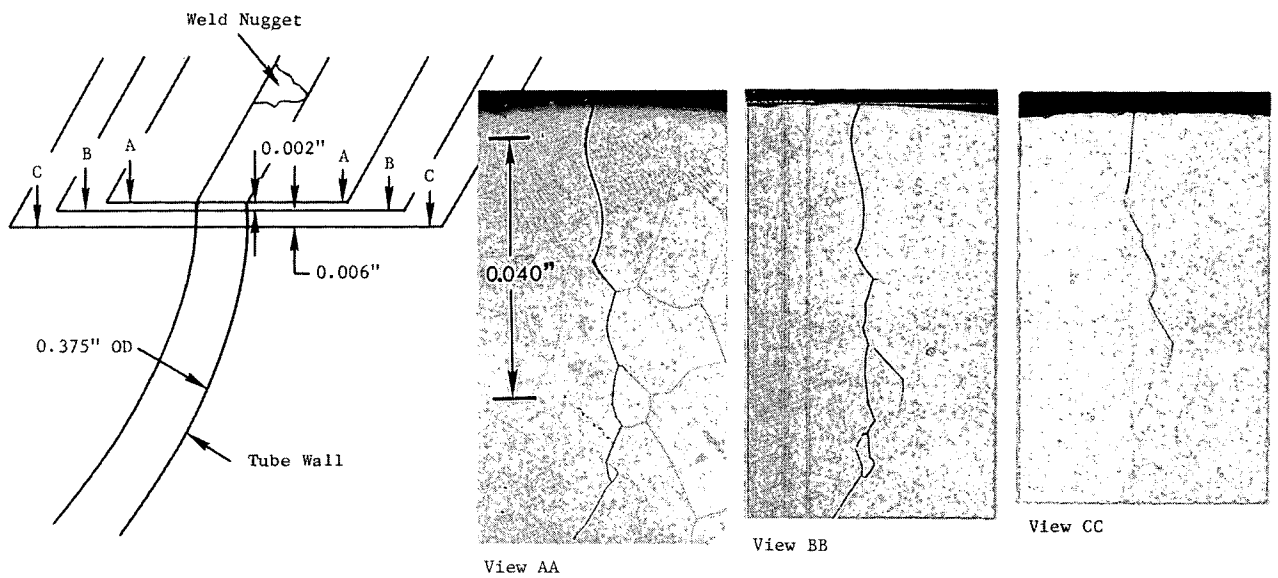


Figure VIII-23. - Intergranular crack in weld nugget of 0.375-inch-diameter T-111 boiler tube removed from T-111 Rankine system corrosion test loop.

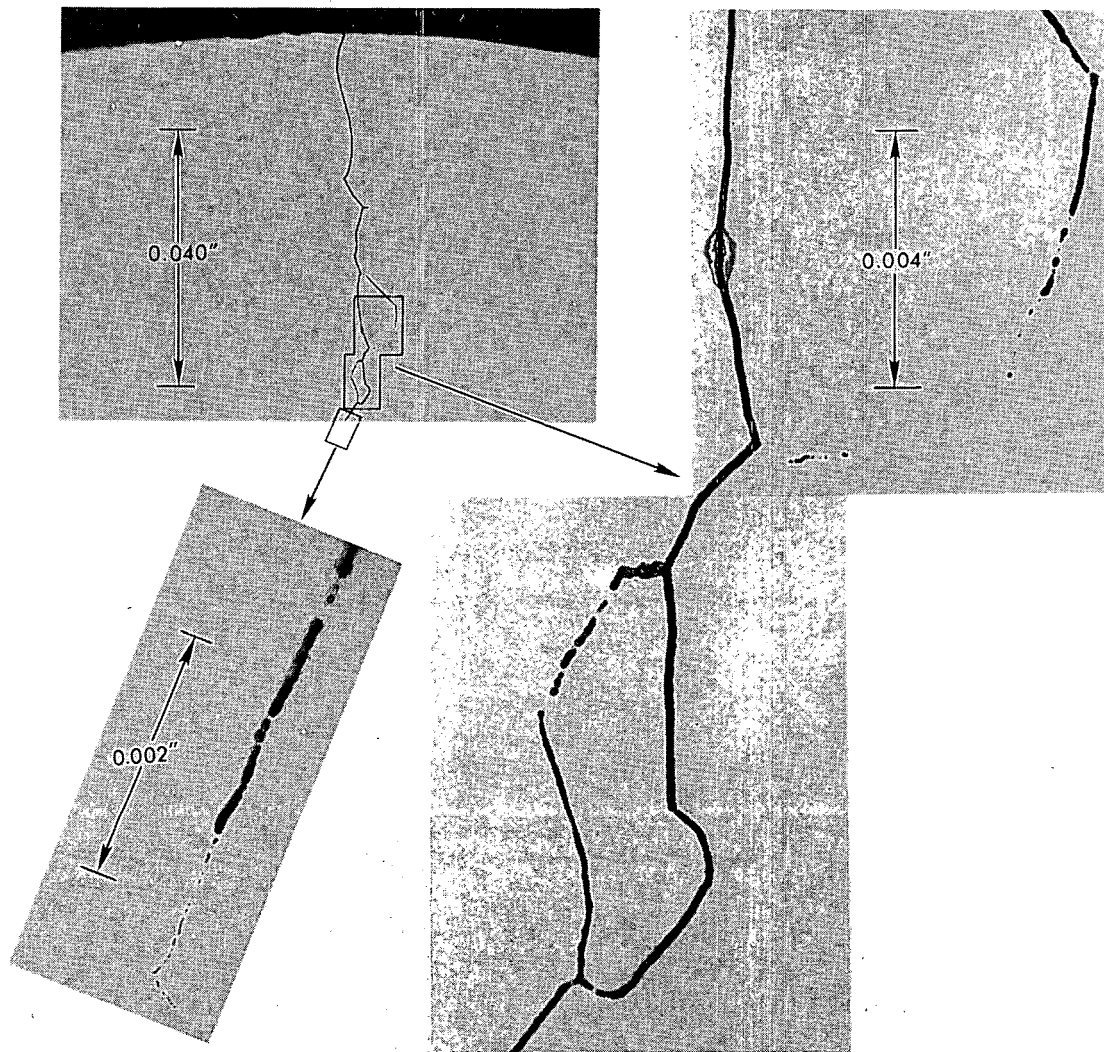
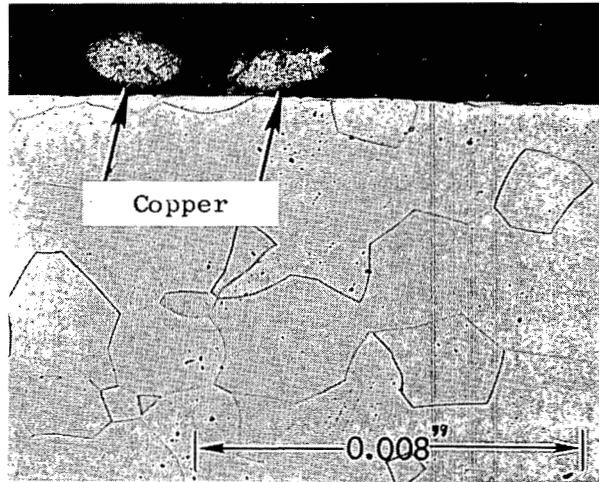
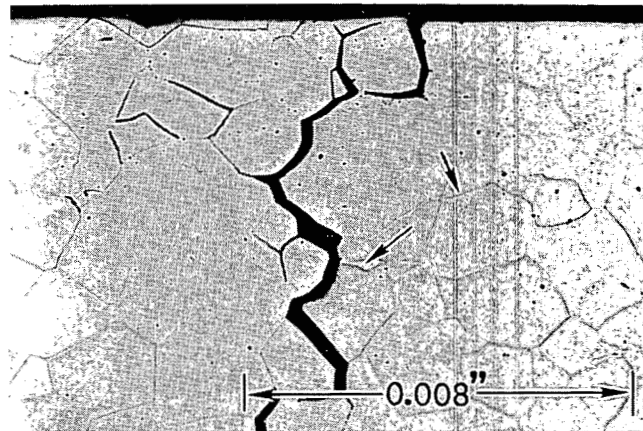


Figure VIII-24. - Intergranular crack in weld nugget of 0.375-inch-diameter T-111 boiler tube removed from T-111 Rankine system corrosion-test loop.



(a) As tack welded.



(b) Tack welded followed by 2400° F, 1-hour heat treatment.

Figure VIII-25. - T-111 alloy tubing (1.5-in. by 0.100-in. wall) tack welded with copper electrode. Residual copper shown by arrows in (a). Etchant: equal volumes of  $H_2O$ ,  $H_2O_2$ ,  $H_2SO_4$ , HF.



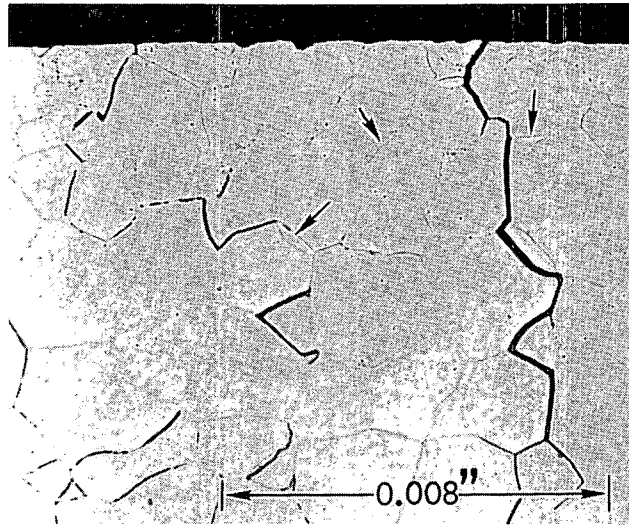


Figure VIII-26. - Intergranular cracking of T-111 alloy tubing (0.375-in. by 0.065-in. wall) tack welded using copper electrode and heated to 2400° F for 1 hour. Etchant: equal volumes of H<sub>2</sub>O, H<sub>2</sub>O<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>, HF.

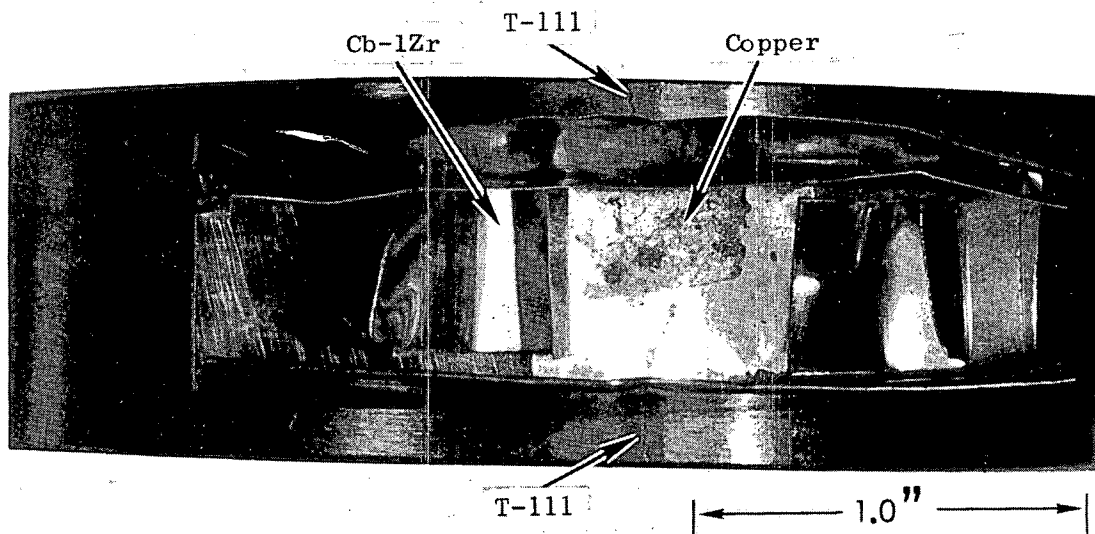


Figure VIII-27. - Crack (see arrows) in T-111 alloy tube (1.5-in. by 0.100-in. wall) produced by copper diffusion into tube through multilayers of 0.002-inch-thick Cb-1Zr foil during heat treatment at 2400° F for 1 hour.

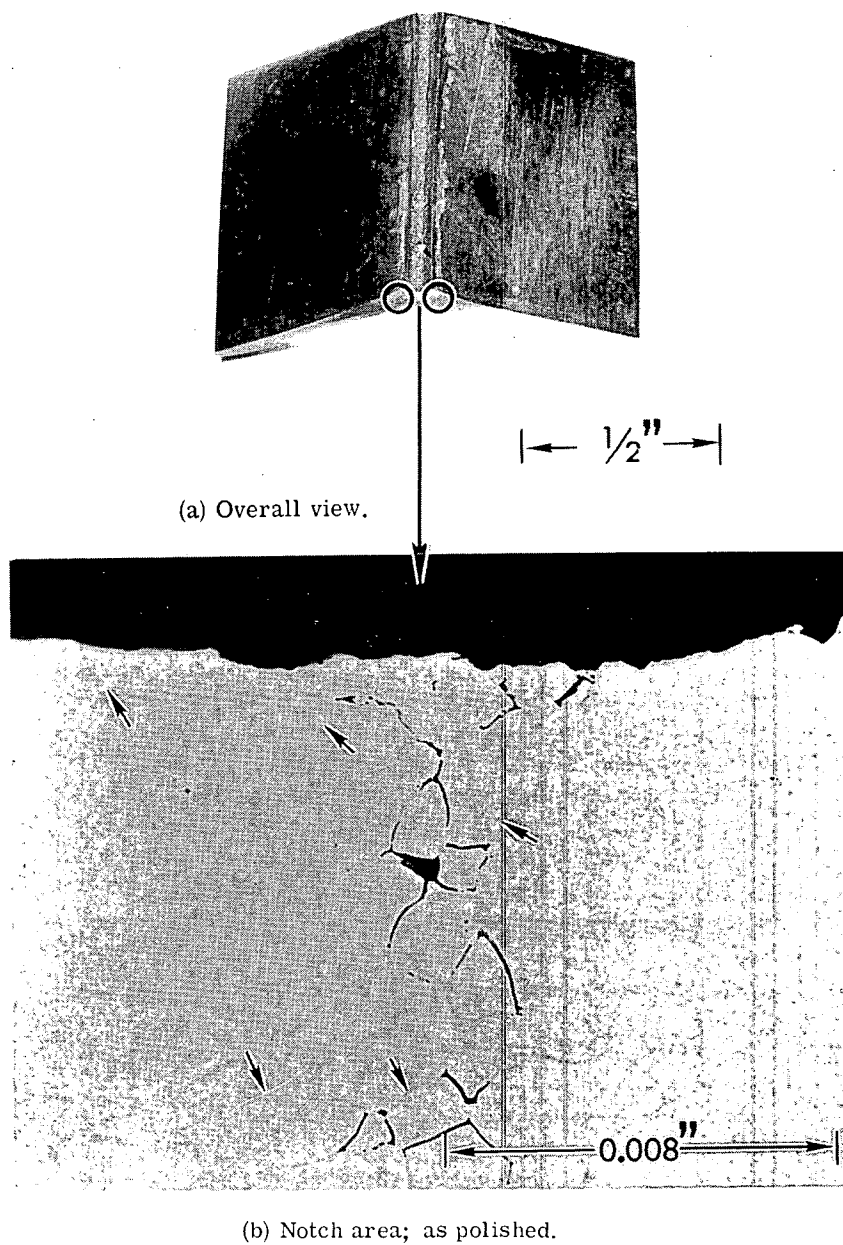


Figure VIII-28. - Copper filled notch in T-111 alloy bend sample (0.063 in. thick) after heat treatment at 2400° F for 1 hour. Light colored grain boundary phase in (b) is rich in Cu and Hf.