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TOPICAL REPORT NO. 4
SNAP-8 REFRACTORY BOILER DEVELOPMENT PROGRAM

MERCURY THERMAL SHOCK TESTING OF 2½-INCH-DIAMETER BIMETALLIC JOINTS FOR SNAP-8 APPLICATIONS

By

S. R. Thompson

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-10610
Edward R. Furman, Project Manager

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (Cont.)

ABSTRACT

Coextruded and brazed tubular transition joints, between 2.5-inch-outside diameter tantalum and Type 316 stainless steel, were evaluated for usage in the SNAP-8 Mercury Power Conversion System. Representative joints were exposed to repeated thermal shocks by flowing 300°F mercury through the joints which were initially heated to 1300°F. Failure of the coextruded specimen occurred after 55 thermal shocks; no brazed joint failure was noted after testing to 100 and 155 cycles (two brazed samples). Posttest destructive and nondestructive examination of the sample assemblies indicated that the braze method produced assemblies which were essentially unaffected by the shock testing. The results of the above testing, and other severe thermal shock tests of this type of joint, show that the brazing method for effecting transitions between tantalum and stainless steel will produce a reliable joint for SNAP-8 applications.

FOREWORD

This report was prepared and the associated work performed in support of NASA program, "SNAP-8 Refractory Boiler Development," Contract No. NAS 3-10610, under the management of R. D. Brooks.

The author gratefully acknowledges the contributions of J. Holowach, responsible for performance of the thermal shock testing; W. F. Zimmerman, for assistance in interpretation and evaluation of posttest data; and W. R. Young, for reviewing the content of the report. In addition, valuable assistance was provided by H. J Bauer, I. A. Miller, and C. Asaud in the preparation and examination of the metallographic specimens.

MERCURY THERMAL SHOCK TESTING OF 2 1/2-INCH-DIAMETER BIMETALLIC JOINTS FOR SNAP-8 APPLICATIONS

I. Introduction

The Aerojet-General Corporation Specification AGC-10512, Power Conversion System, Ground Model (PSC-8), SNAP-8 requires that the mercury boiler be capable of a minimum of 100 restarts with a design life of 10,000 operational hours. The basic materials utilized in the system are unalloyed tantalum for the boiler tubes and Type 316 stainless steel for the remaining loop components. Both of these materials had been shown to have excellent individual resistance to mercury attack under conditions associated with the application.(1, 2) The startup sequence of the SNAP-8 power conversion system subjects the entire boiler section to a severe thermal shock when 70°F mercury is injected into the 1300°F boiler. Further, for the present boiler design, 2.50-inch-outside-diameter tantalum-to-Type 316 stainless steel tubular transition joints are required at both the inlet and exit of the boiler. Two candidate methods under consideration for fabrication of the bimetallic transition joints were coextrusion and brazing. Representative joints - coextruded, manufactured by Nuclear Metals, Inc., and brazed, fabricated by GE - NSP - were prepared for thermal shock testing to establish the relative reliability of the different designs and manufacturing processes. The testing parameters selected were more severe than those conditions associated with normal startup of the power conversion system. The mercury testing also provided a quantitative means for reaffirming the resistance to mercury attack of the tantalum - braze alloy - stainless steel materials combination under SNAP-8 operating conditions, which had been established earlier for smaller diameter brazed joints.(3)

II. Test Specimen Preparation

The basic design configuration for the 2.5-inch-OD brazed samples was a tongue-in-groove, as shown in Figure 1. The two brazed assemblies (Serial No's 14-14 and 16-16) discussed within this report, were vacuum brazed at 2160°F - 2180°F for five minutes, using the cobalt-base filler alloy designated J-8400 (B50T56-S1)*. After brazing, both brazed assemblies were nondestructively inspected using visual, dye penetrant, ultrasonic, and mass spectrometer techniques, and no rejectionable defects were observed. The Nuclear Metals, Inc. coextruded joint was purchased by the NASA - Lewis Research Laboratory and assigned to GE - NSP for thermal shock testing without performance of additional pretest inspection by GE - NSP. In addition to the different methods utilized in fabrication of the test specimens, the wall thickness of the coextruded joint was 0.020 inch less than that of either brazed assembly; i.e., 0.110 inch vs. 0.130 inch.

III. Thermal Shock Testing

For the initial shock testing, brazed sample S/N No. 14-14 and the coextruded specimen were assembled to form a test section by tungsten inert gas (TIG) welding the tantalum ends of each together. The welded assembly was positioned with the coextruded joint nearest the mercury inlet end for the forthcoming thermal testing. A 0.060-inch-diameter orifice plate was inserted downstream of the two bimetallic joints to restrict the flow of mercury through the test section and rapidly build up the inlet mercury pressure to suppress film boiling of the mercury in the joint areas.

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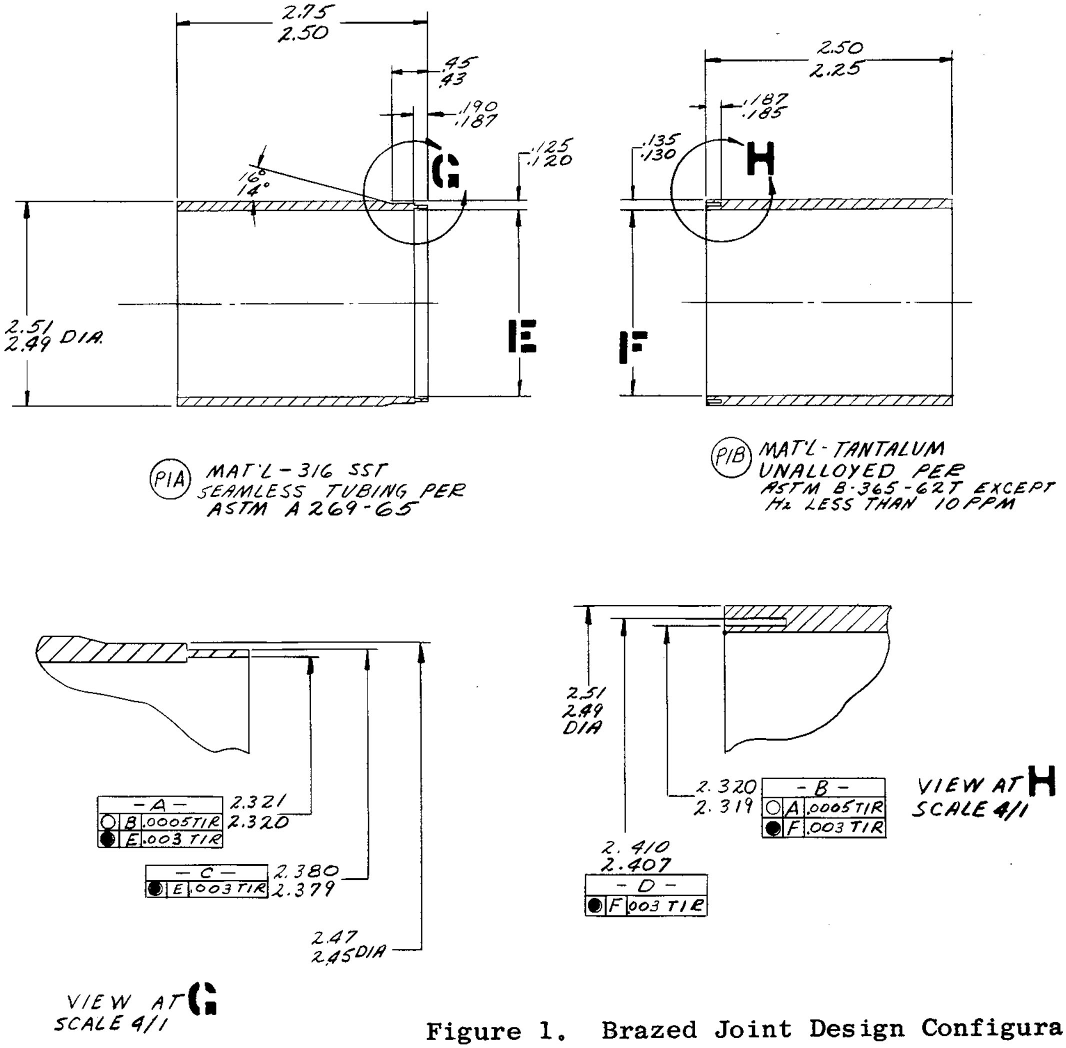
* Braze Alloy Composition: 21Cr-21Ni-3.5W-8.0Si-0.8B-0.04C-Bal. Co.



6 ULTRASONIC INSPECT PER GE SPPS SPEC 03-0001-00-B OR X-RAY PER AMS 2635 ASS'Y FOR INTERNAL DEFECTS

5 PENETRANT INSPECT PER AMS 2645

4 MASS SPECTROMETER LEAK TEST PER GE SPPS SPEC 03-0013-00-B

3 CLEAN PER GE SPPS SPEC 03-0021-00-A

2 PARTS P1A & P1B MUST BE BAGGED OR TAGGED AS A MATCHED ASS'Y WITH IDENT & SERIAL NO

1 INTERPRETATION OF DWG TERMS AND TOL. PER GE SPPS SPEC 06-0002-00-A

Figure 1. Brazed Joint Design Configuration, Dwg. No. 47C143236.

A vacuum jacket was assembled around the test section to protect the tantalum members of the bimetallic joints from surface contamination during the temperature cycles. The jacket was fabricated from standard vacuum components with either welded or copper gasket joints. A flexible bellows coupling was used to accommodate the differential thermal expansion between the test section and the vacuum jacket during the heating and cooling cycle. The bimetallic joints were heated with an electrical resistance heater attached to the vacuum jacket section surrounding the bimetallic joints. Heat was transferred from the vacuum jacket to the bimetallic joints by thermal radiation. A chromel/alumel thermocouple was attached to both the stainless steel and tantalum sections of each bimetallic joint. Test temperatures were recorded with a high-speed recording potentiometer which allowed the temperature transients of each joint to be compared. The test section assembly is shown in Figure 2.

The thermal shock test facility consisted of a mercury EM pump, the test section, and a water-cooled heat exchanger as shown in Figure 3. An evacuation and gas pressurization port was used to evacuate the loop before the test and to pressurize the test section with helium for mass spectrometer leak testing of the bimetallic joints at specified intervals during the test. The test section was placed in a vertical position to allow the mercury to gravity drain from the test section after each thermal shock.

The thermal shock cycle as shown in Figure 4 was as follows: the electric power to the heater was adjusted to heat the bimetallic joints to 1300°F in 30 minutes. This temperature was maintained at the joints

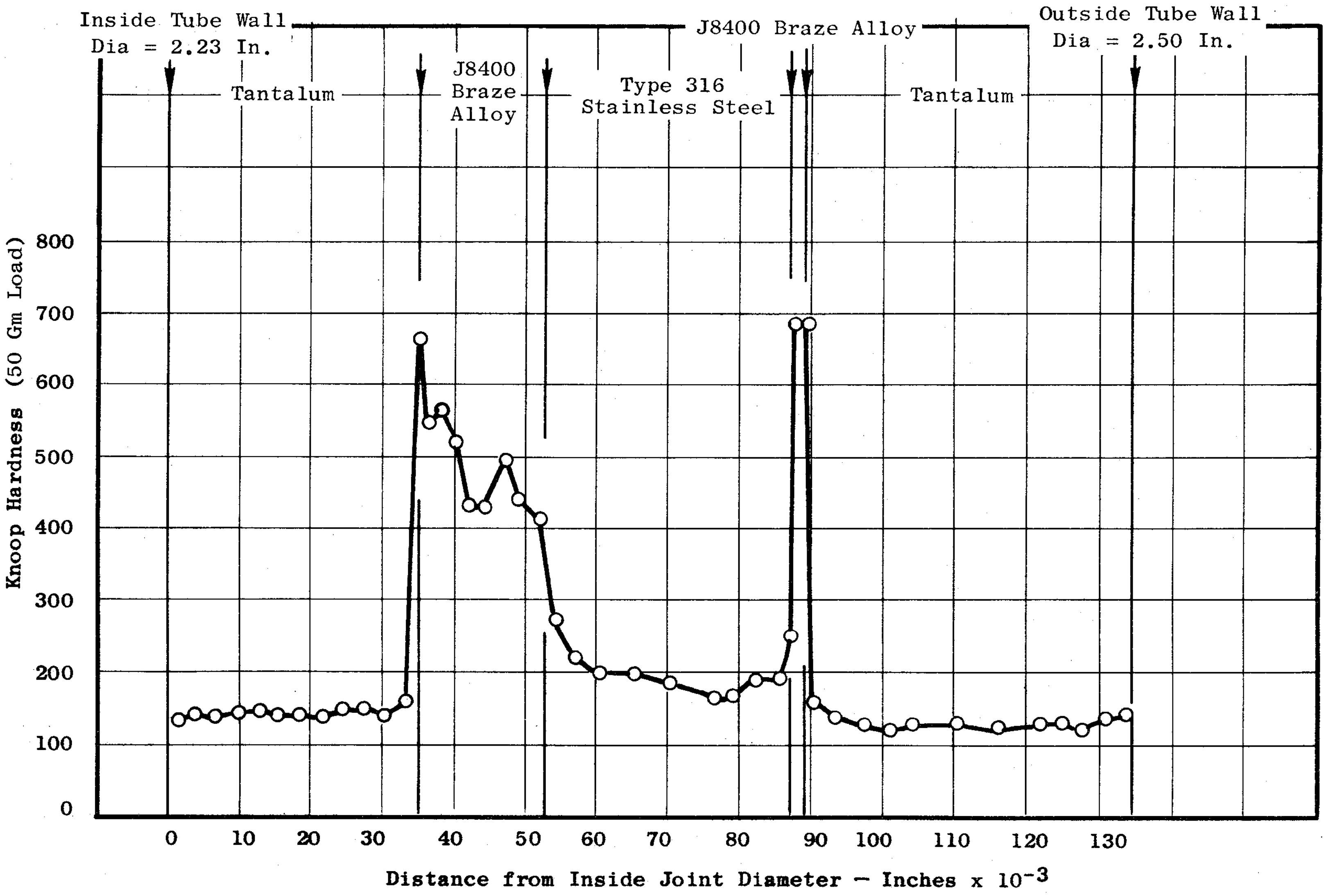
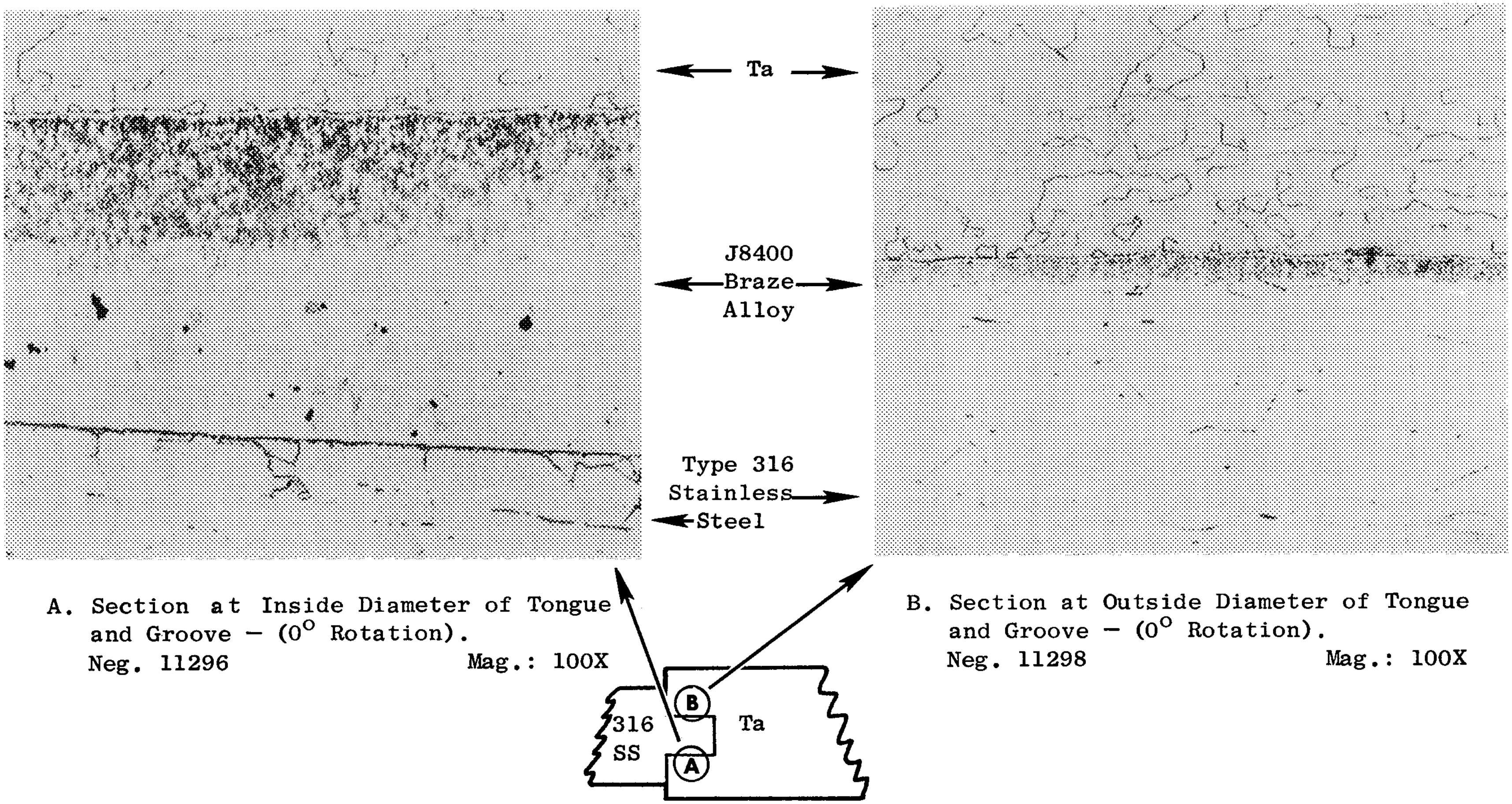


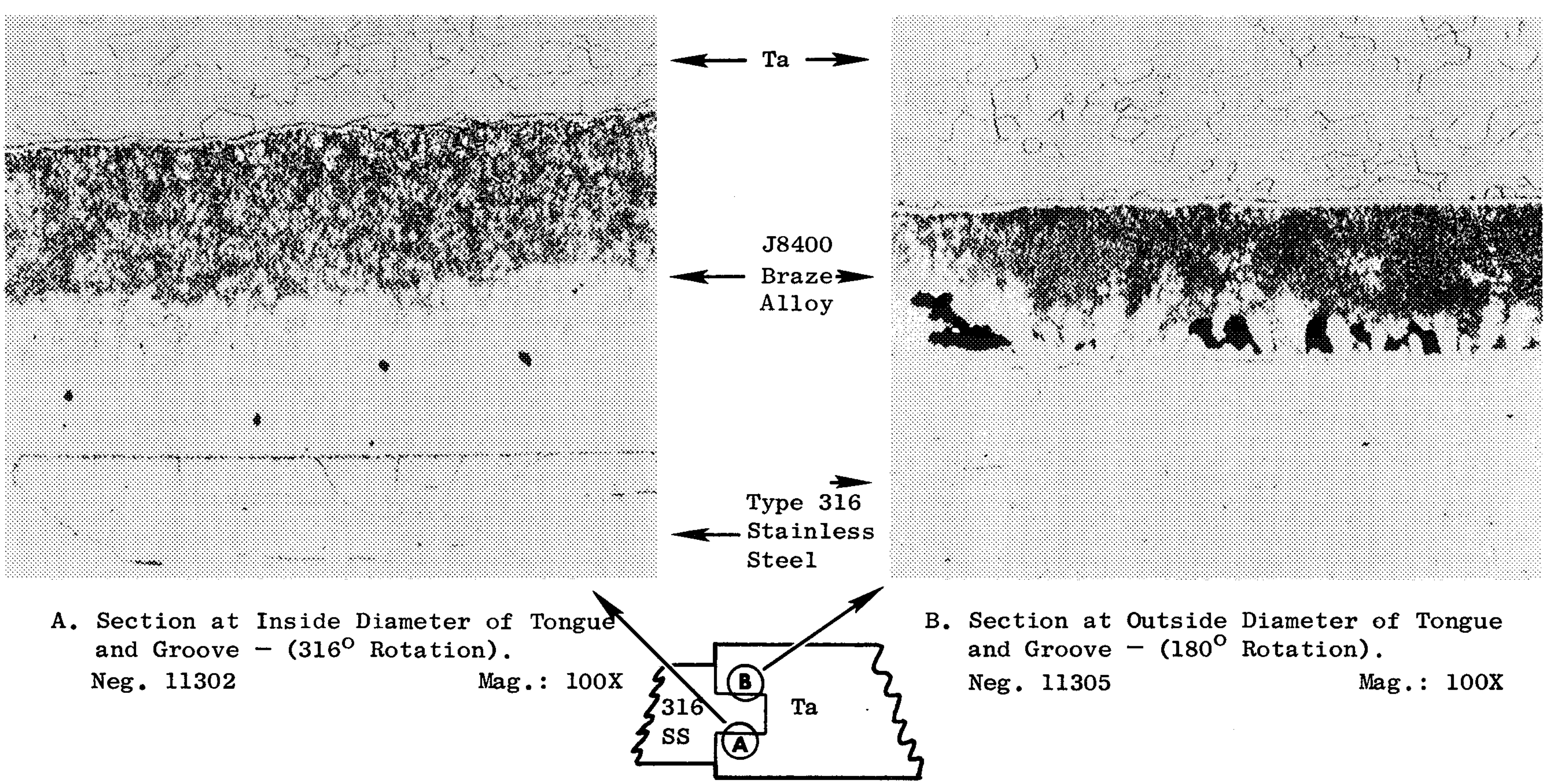
Figure 10. Hardness Traverse Across Wall of 2 1/2-Inch Diameter Tantalum/Type 316 Stainless Steel Transition Joint (S/N 14-14 at 0°) After Mercury Thermal Shock Test (155 Cycles).



A. Section at Inside Diameter of Tongue and Groove — (0° Rotation).
Neg. 11296 Mag.: 100X

B. Section at Outside Diameter of Tongue and Groove — (0° Rotation).
Neg. 11298 Mag.: 100X

Figure 11. Representative Microstructures of Brazed Transition Joint, S/N No. 14-14, After Mercury Thermal Shock Test (155 Cycles).



A. Section at Inside Diameter of Tongue and Groove — (316° Rotation).
Neg. 11302 Mag.: 100X

B. Section at Outside Diameter of Tongue and Groove — (180° Rotation).
Neg. 11305 Mag.: 100X

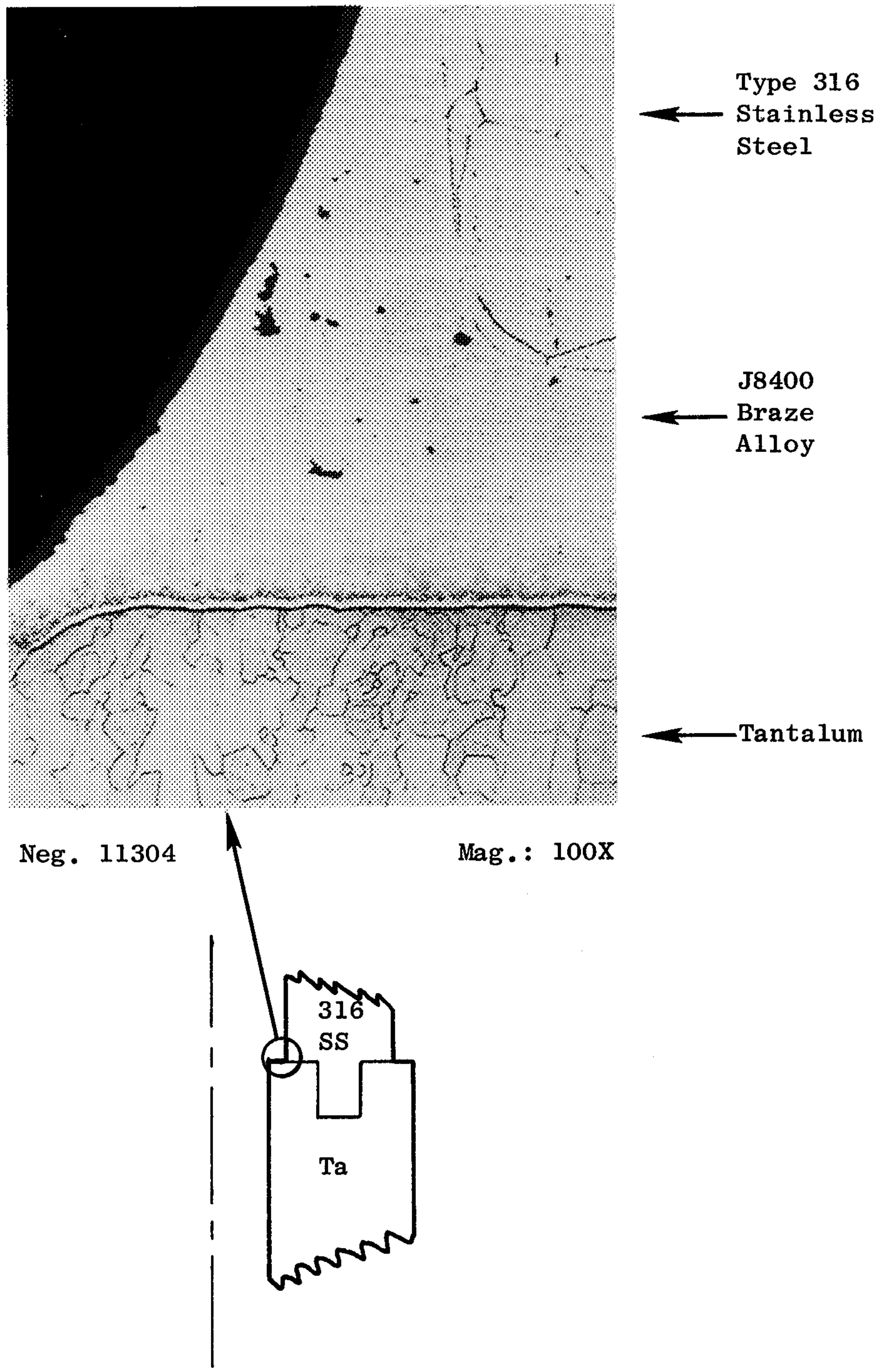
Figure 12. Representative Microstructures of Brazed Transition Joint, S/N No. 16-16, After Mercury Thermal Shock Test (100 Cycles).

entrance braze flow side (outermost tantalum section). The reaction zone in the stainless steel was largest on the inside surfaces of the tongue (Kn 270 at 0.002 inch from the interface and Kn 200, 0.006 inch away); the lower hardness readings were encountered less than 0.002 inch away from the outside surfaces of the stainless steel tongue. The width of the diffusion zone in the outermost tantalum areas was approximately 0.007 inch to 0.011 inch; the hardness decreased in that area from Kn 180, 0.0005 inch from the braze interface to the base hardness of Kn 125.

Previous studies on Cb-1Zr/Type 316 stainless steel brazed transitions, using the J-8400 braze alloy(5) have demonstrated that the formation of an intermetallic phase at the Cb-1Zr-braze alloy interface during brazing limited the amount of interdiffusion occurring during subsequent 1600°F/1000-hour exposures in vacuum. Microstructural examination of the tantalum/Type 316 stainless steel, 2.5-inch-OD joints, indicated the presence of a similar phase, as shown in Figures 11 and 12. If equivalent reaction rates are assumed between the braze alloy and both Cb-1Zr and tantalum, then the above indicated diffusion in the tantalum probably occurred during brazing.

Microstructural examination at high magnification (to 1000X) of the internal piping surfaces and the internal braze fillets produced no significant evidence of mass transfer, mercury corrosion, or joint deterioration caused by the repeated thermal/mercury shock cycles. Figure 13 shows a typical appearance of the inner braze fillet area for joint S/N No. 16-16; similar structures were also present at the inner fillet areas of joint S/N No. 14-14.

Microshrinkage voids were discovered to varying degrees in the



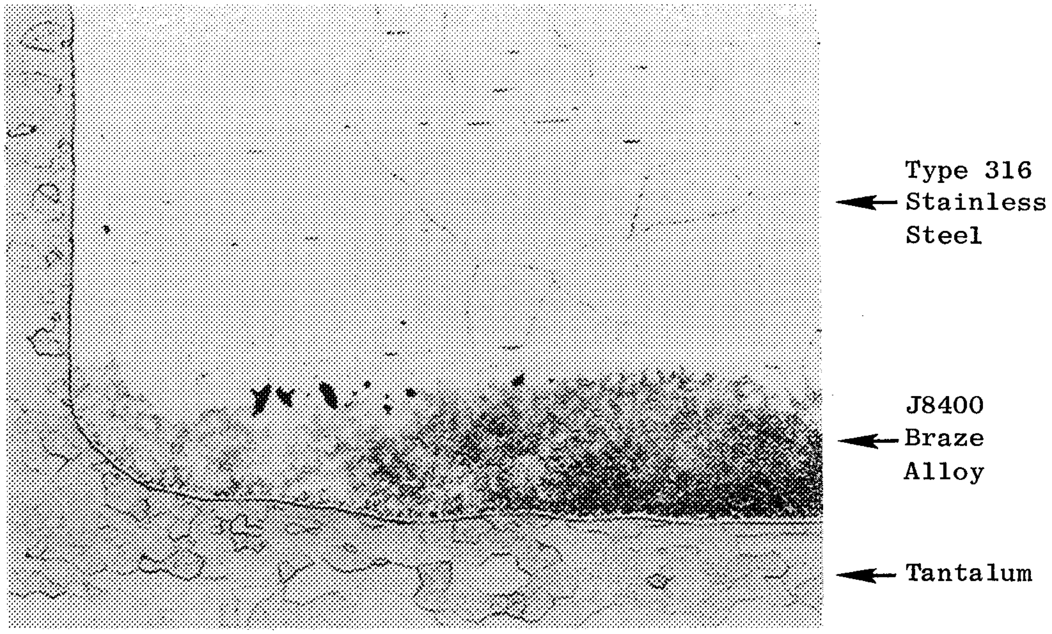
Neg. 11304

Mag.: 100X

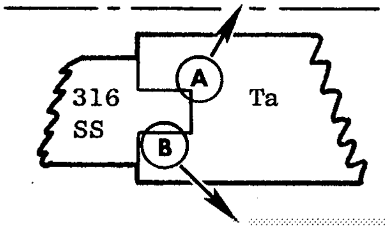
Figure 13. Typical Microstructure at Inside Braze Fillet of Brazed Joint, S/N No. 16-16, After Mercury Thermal Shock Test (100 Cycles).

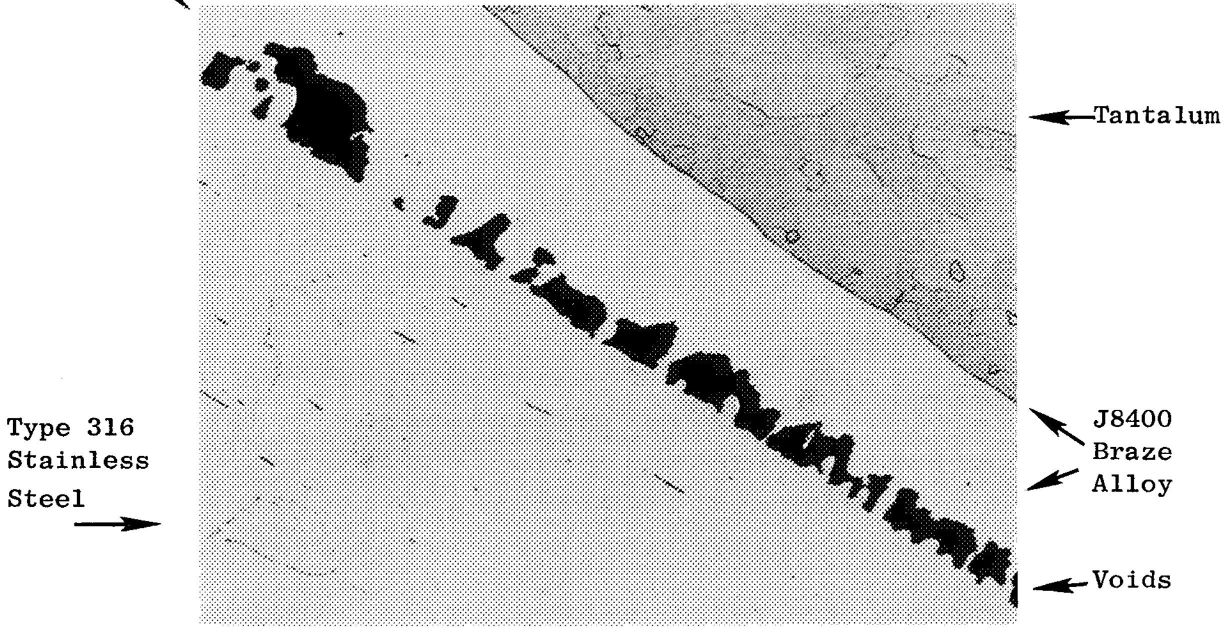
brazed flow paths of both assemblies. These voids were formed on cooling from the original brazing thermal cycle - they occur in the last portions of the braze alloy which solidify, and their occurrence is a normal brazing by-product if freezing of the alloy at a location between the entrance feed fillet and the discussed area blocks the supply of liquid braze. As shown in Figures 12B and 14B, the largest zones of microshrinkage were found in joint S/N No. 16-16, near the base of the tongue and groove in the sections taken at 180° and 226° rotation from the ultrasonic inspection reference index. As indicated previously, these sections represented (ultrasonic) intermediate and best-bonded areas of that assembly, and their presence to the extent shown in the microphotographs was not anticipated. Further, the extent of their size and agglomeration, while greater than desired, was not prohibitive from a brazing or structural standpoint. Microstructural examination of the most suspect area (at 0° rotation) of Specimen S/N No. 16-16 revealed a completely sound brazed section, in direct contradiction to the ultrasonic data. The lack of agreement, between the ultrasonic indications and the actual microstructures present, may possibly be explained by realizing that in cutting the assemblies, and subsequent metallographic preparation, material removal may be somewhat different than expected. Thus, the sections examined may not have coincided exactly with the desired locales, and the joint characteristics could be completely different at the positions examined.

The metallographic sections prepared from joint S/N No. 14-14, at 0°, 160°, and 316° rotation from the ultrasonic index, yielded essentially identical microstructures. Again, complete agreement between the actual



A. Inside of Tongue and Groove.
Neg. 11307 Mag.: 100X





B. Outside of Tongue and Groove.
Neg. 11306 Mag.: 100X

Figure 14. Microstructures Near Base of Tongue and Groove of Brazed Joint, S/N No. 16-16 at 226° After Rotation Mercury Thermal Shock Test (100 Cycles).

structures and the ultrasonic indications was not achieved, although generally better correlation was evident than with sample S/N No. 16-16. The microshrinkage present in sample S/N No. 14-14 (Figure 11A) was uniformly widespread, and individual voids were very small (0.0001 inch). It was possible that these voids were the cause for the ultrasonic indications at the above specified locations if the inspection equipment sensitivity was set too high. Also, the spacing between each void (approximately 0.010 inch) may have been small enough that the sensing transducer could not resolve their separation, since the transducer does sweep a finite distance. One additional factor that should be pointed out, since it could have influenced the ultrasonic data, was that the braze alloy on the inside of the tongue and groove (both brazed assemblies) was two-phase in nature, as shown in Figures 11A and 12A. This zoning effect essentially represented another possible interface, and the chemistry changes present therein could have altered the speed of the sound waves passing through, which in turn could cause suspect areas to appear on the ultrasonic "A" scans. Because none of the described phenomena for joint S/N No. 14-14, as well as joint S/N No. 16-16, were unacceptable from a structural standpoint, it was obvious that the ultrasonic inspection technique required refinement to more clearly define objectionable defects. The bulk of the difficulties associated with ultrasonic inspection of tongue-in-groove brazed specimens were contingent on the lack of a satisfactory "standard" brazed specimen. To alleviate the problem, "standard" specimens containing good and bad brazed areas at prespecified locations, are presently being prepared for future applications.

Other pertinent facts established by the microstructural examination and microhardness testing of the brazed specimens were as follows:

a. The capillary spacing between the outside of the tongue and groove for the two specimens was different; 0.0015 inch for joint S/N No. 14-14, 0.005 inch for joint S/N No. 16-16.

b. The previously mentioned microshrinkage voids in both brazed samples were prevalent in the wide gap side of the tongue and groove, in the light etching areas of the two-zone braze material. The braze material in the outside of the tongue and groove etched darker and was essentially free of voids.

c. The two-zone nature of the braze in S/N No. 16-16 extended around the base of the tongue from the wide gap side and into the 0.005 inch capillary side for approximately 0.030 inch. Thus, some microshrinkage was also noted on the capillary side of that joint (see Figure 12B).

d. The grain size of the stainless steel in the tongue areas of the brazed joints was larger than that determined in the remainder of that component. Two possible reasons for this larger grain size were stresses associated with (1) the original machining operation and/or (2) differential thermal expansion/contraction induced during testing. These stresses could have caused plastic deformation which coupled with the elevated test temperatures resulted in the increased grain size.

e. The reaction zones identified in the stainless steel tongues by the microhardness testing resulted primarily from interdiffusion with the J-8400 braze alloy. This was substantiated further by the fact that the grains in that area were equiaxed, implying that the thermal/mechanical stresses induced during machining or testing were completely relieved.

f. As shown in Figure 14B, the corners at the base of the tongue and groove of joint S/N No. 16-16 were rounded (approximately 0.001-inch radius). This effect could have been produced by (1) interalloying of the base metals with the braze alloy during the brazing cycle or (2) the initial machining operation. In either event, the effect was beneficial, since the stress concentration factors are reduced; thus, the assembly can more readily withstand the stresses associated with thermal cycling. A similar corner rounding was observed for joint S/N No. 14-14.

No detrimental reactions between the joint components and mercury were noted; nor did the thermal stresses during testing cause degradation of the brazement with the possible exception of the stainless steel variation in grain size. Also, the fact that the ultrasonic inspection results before and after the thermal shock test were identical was meaningful because any significant degradation would no doubt have been identified by that technique. Since no change was observed in that data, the metallurgical phenomena observed were attributed primarily to the brazing operations and not to the thermal shock testing.

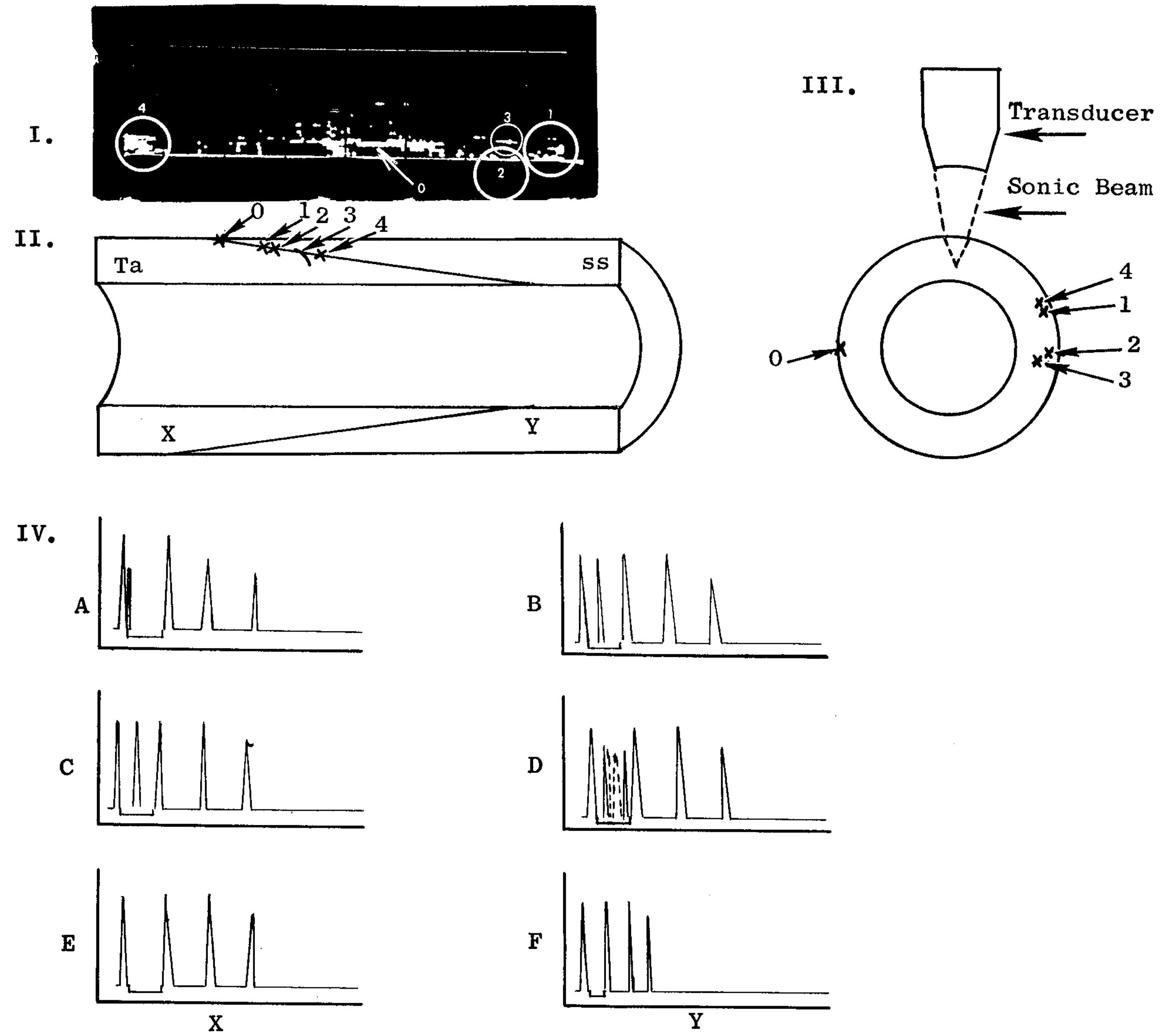
V. Summary of Evaluation Data from Failed Coextruded Joint

The 2 1/2-inch-diameter tantalum/Type 316 stainless steel coextruded joint was examined, after testing, nondestructively by ultrasonic and dye penetrant techniques and then sectioned for metallographic examination.

The ultrasonic inspection was performed using a focused transducer and a longitudinal wave mode through the total joint wall thickness; scans were made circumferentially along the outside diameter of the joint area. A "C"scan ultrasonic trace was prepared, from repeated "A" scans, by means of appropriate gating techniques, to differentiate and record any electronic signals different from those obtained for the outside and inside tube wall reflections and those found for a completely bonded cross section. The nature of the ultrasonic indications is shown in Figure 15.

The locations of the principal ultrasonic suspect areas were marked on the outside of the tested joint; after which the assembly was dye penetrant ("Zyglo") inspected. Continuous indications were noted around the "feather" edge of the joint area on the inside of the assembly and at several locations on the outside feather edge.

After "Zyglo" inspection, the joint was sectioned for microstructural study at the principal ultrasonic and "Zyglo" suspect areas. Several microstructures obtained at these locations are presented in Figure 16. The correlation between the inspection indications and the actual microstructures was excellent; i.e., at every suspect location examined, separation of the interface was observed metallographically. No evidence of any significant intermetallic phase formation at the interface was observed during examinations to 1000X. Further, no microstructural



I. C-scan recording depicting areas of interface discontinuity. White indicates response areas.

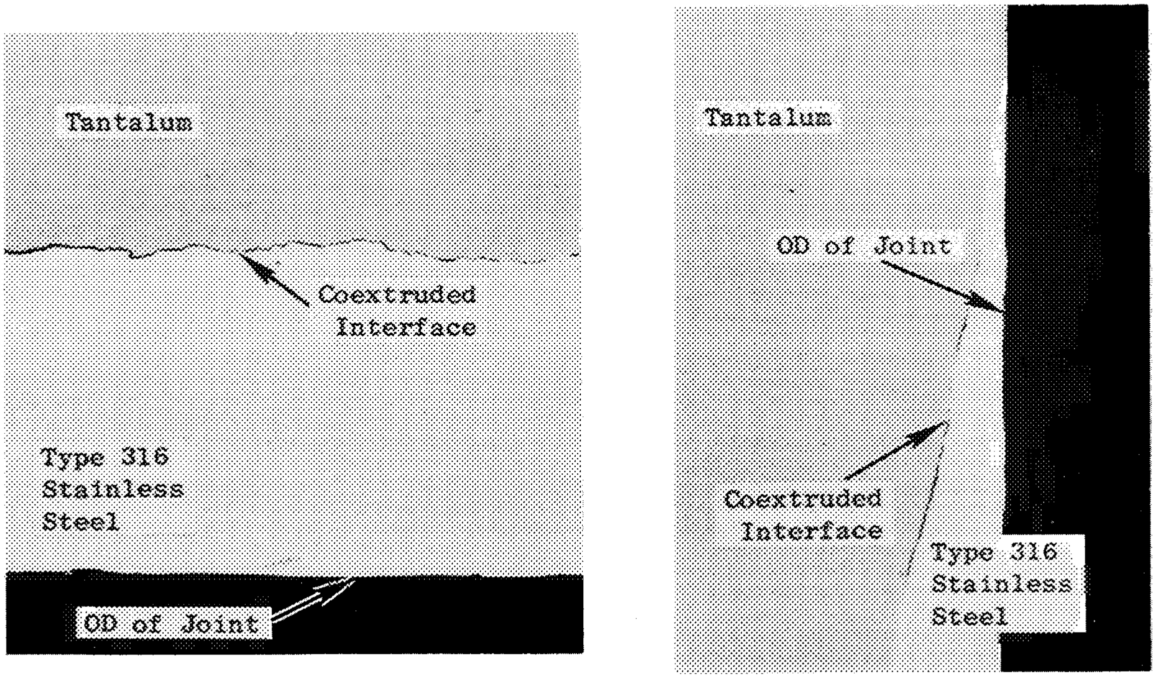
II. Longitudinal cross section indicating depth of indications relative and wall thickness. Note depth of #3

III. Transverse cross section of joint indicating depth of indication and method of ultrasonic inspection.

IV. A-scan presentation of A. defect #0 near OD B. defect #2 depth C. defect #1 & 4 depth D. defect #3 change of depth E. & F. change of time base due to velocity change created by change of ratio of Cb-1Zr to S.S.

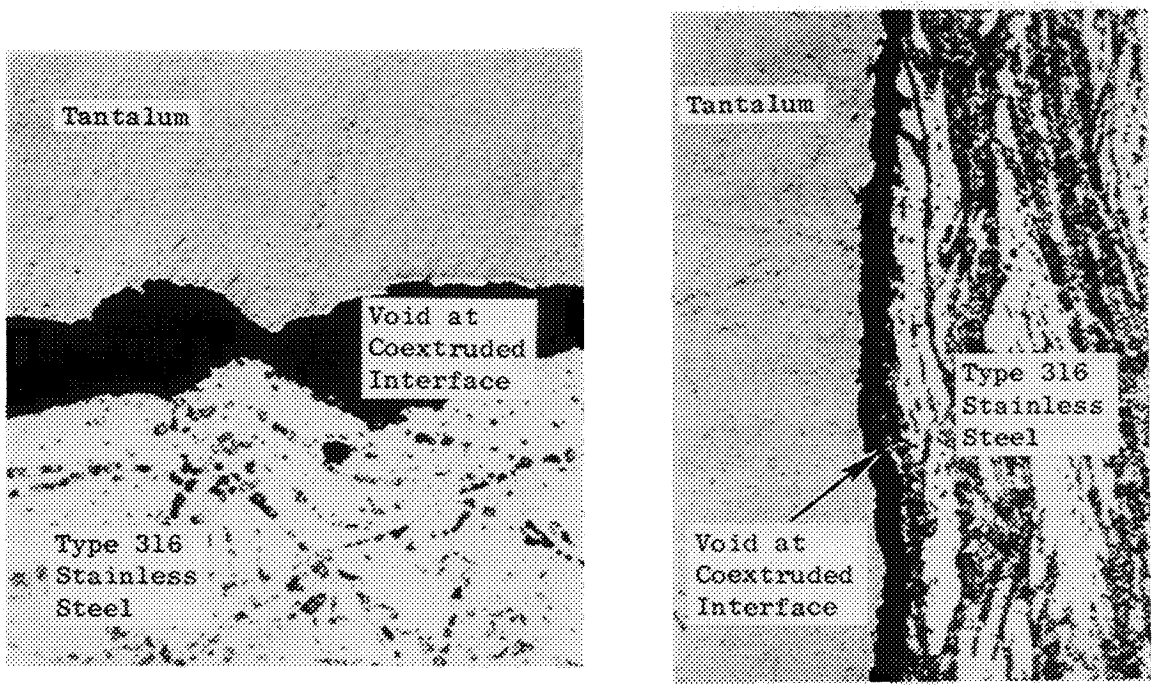
Figure 15. Presentations from Ultrasonic Inspection of Coextruded Tantalum/ Type 316 Stainless Steel Joint After Mercury Thermal Shock Test (55 Cycles).

Transverse Section Longitudinal Section



Unetched Mag.: 50X
F900311

Unetched Mag.: 50X
F900611



Etchant: 10 Oxalic Mag.: 1000X
100 H_2O
Electrolytic
F900313

Etchant: 10 Oxalic Mag.: 1000X
100 H_2O
Electrolytic
F900614

Figure 16. Typical Microstructures in Joint Area Coextruded Tantalum/Type 316 Stainless Steel Joint After Mercury Thermal Shock Test (55 Cycles).

indications of mercury corrosion or erosion were found along the inside of the assembly. Microhardness traverse data were determined across the wall of the coextruded specimen. As shown in Table I, essentially constant hardness values were obtained throughout the individual components, tantalum and stainless steel, in the joint area. The data also verified the visual observations that no intermetallic phases were present at the joint interfaces.

The susceptibility of the coextruded joint to failure under severe thermal shock conditions was clearly indicated. Further, good agreement was realized between ultrasonically detected flaw locations and actual separation points of the joint.

TABLE I

TYPICAL MICROHARDNESS VALUES ACROSS WALL OF 2 1/2-INCH-DIAMETER COEXTRUDED SPECIMEN AFTER MERCURY THERMAL SHOCK TEST

Impression Location (Inches from Interface)		Hardness (Knoop-100 gm Load)
0.002	In Stainless Steel	251
0.004		258
0.006		258
0.008		272
0.010		247
0.012		259
0.014		259
0.016		259
0.018		263
0.002	In Tantalum	159
0.004		146
0.006		154
0.008		166
0.010		156
0.012		151
0.014		156
0.016		147
0.018		138
0.020		140
0.022		159

VI. Summary

Tubular transition joints between 2.5-inch-OD tantalum and Type 316 stainless steel are required as components in the SNAP-8 Power Conversion System which utilizes mercury as the working fluid. Two candidate fabrication methods, coextrusion and brazing, were considered and both subsequently evaluated by exposing representative joints to repeated thermal shocks in the 1300°F to 300°F temperature range. Each individual thermal cycle was achieved by flowing mercury at 300°F through the joints which were initially heated to 1300°F. These test parameters were selected to simulate those conditions expected at the joint transitions during start-up of the SNAP-8 system. Failure of the coextruded joint was observed after 55 thermal cycles. The failed joint was subsequently replaced by another brazed joint and the testing continued until the initial brazed assembly had accumulated a total of 155 thermal shocks. Subsequent posttest examination of the brazed joints, using both destructive and nondestructive inspection techniques, indicated that the braze method (1) produced assemblies which were essentially unaffected by the mercury exposure and (2) could be used with a high degree of reliability to fabricate the transition joints necessary for the power conversion system. This report summarizes the results of the posttest evaluation of the brazed joints which support the previous statement regarding the suitability of the brazed technique for fabrication of the tantalum/stainless steel SNAP-8 bimetallic transitions.

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