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Final Report

COEXTRUDED TANTALUM - 316 STAINLESS STEEL BIMETALLIC JOINTS AND TUBING

by

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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NASA Lewis Research Center
Cleveland, Ohio
P. Stone, Project Manager
Materials and Structures Division

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ABSTRACT

Techniques were established for the coextrusion of tandem and sleeve tantalum-stainless steel bimetallic joints. Large sleeve joints, 1.76 inches inside diameter with an unalloyed tantalum inner wall thickness of 0.10 inch and an AISI 316 stainless steel outer wall thickness of 0.22 inch, were produced. Extrusion procedures were also developed for the production of small sleeve joints, having the same wall thickness as the large sleeve joints but with an inside diameter of 0.76 inch. Conditions were established for the extrusion of tandem tantalum-stainless steel joints of 1.76 inches inside diameter and 0.12 inch wall thickness. An attempt was made to produce 25-foot long, 0.632-inch ID bimetallic tubing having a 0.020-inch tantalum thickness and a 0.060-inch stainless steel thickness.

The extrusion conditions for the production sleeve-type joints were an 8:1 reduction ratio at 1950°F, while the production tandem joints were made at a 5:1 reduction ratio at 1825°F. All joints -- both those produced in the developmental phase [with one exception] and those made for production -- were sound, with no evidence of non-bond or any metallurgical defects. Overall, the bimetallic tubing fabrication experiment was not successful, but it is believed that with further experimentation sound tubing could be produced.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES - continued

LIST OF TABLES

LIST OF TABLES - continued

COEXTRUDED TANTALUM - 316 STAINLESS STEEL

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SUMMARY

An experimental program was performed, the prime objective of which was to establish the optimum fabrication techniques for the production of extruded tantalum - 316 stainless steel tandem and sleeve joints. [A sleeve joint is a tantalum-lined stainless steel tube.] The design criteria were that all joints be metallurgically bonded and that after extrusion they be machinable to the following dimensions:

1. Tandem joint: 1.76 inches ID, 0.12 inch wall, 8 inches long
2. Large diameter sleeve joint: 1.76 inches ID, 0.10 inch tantalum, 0.22 inch stainless steel, 12 inches long
3. Small diameter sleeve joint: 0.76 inch ID, 0.10 inch tantalum, 0.22 inch stainless steel, 12 inches long.

The starting materials were forged, arc-melted, high-purity tantalum with a grain size finer than ASTM 5, and 316 stainless steel, both in the annealed condition. In the exploratory stage of the program, four tandem joints were extruded, at 5:1 and 7:1 extrusion ratios, at 1825°F and 1950°F. Following the selection of 1825°F/5:1 conditions for this type joint, one additional developmental tandem joint was made with a 0.4-inch beveled interface, to provide a longer interface length in the extruded joint.

The developmental large diameter sleeve joints were extruded at 1825°F and 1950°F, at 5:1 and 8:1 extrusion ratios. Since these joints could be satisfactorily evaluated without destroying them [from sample rings cut from the ends and center of the extruded tubes], it was possible to deliver to

NASA eight 8-inch lengths [two from each extrusion]. The developmental small diameter sleeve joints were extruded at an 8:1 reduction ratio at 1825°F and 1950°F. Four 8-inch lengths [two from each extrusion] of this joint type were delivered to NASA.

Evaluation of the developmental joints showed them all to be sound from both a metallurgical and mechanical point of view. All joints, with the exception of the 7:1/1950°F tandem joint, displayed continuous metallurgical bond lines, with no evidence of voids or inclusions at the tantalum - stainless steel interface. The 7:1/1950°F tandem joint showed a dark phase believed to be an oxide along the bond line. Tensile tests performed on large diameter sleeve joint samples normal to the bond line showed average ultimate strengths of 69,300 psi and 52,900 psi for joints extruded at 1825°F and 1950°F, respectively. The higher strengths of the joints extruded at lower temperature reflect the additional work-hardening imparted to the bimetal tube at the lower extrusion temperature. Tandem joints were tested after machining and revealed no defects when inspected by helium mass spectrometer and dye penetrant.

The production tandem joints were extruded at 1825°F at a 5:1 reduction ratio, since the microstructural examinations of the developmental joints showed this condition to be optimum. The developmental 7:1 extrusions produced interface lengths of 1.6 to 1.8 inches, while the corresponding length for the 5:1 joints was only 0.92 inch. All of these joints were made from square ended tantalum and stainless steel billet components. In order to produce the desired longer interface in the 5:1 joints, mating bevels were machined on the stainless steel and tantalum components. A trial extrusion at a 5:1 reduction and 1825°F extrusion temperature with a 0.4-inch bevel produced a joint with a satisfactory interface length of over 1.3 inches. Twelve production joints were subsequently made under these same conditions.

The production sleeve joints were extruded at 1950°F at an 8:1 reduction ratio. The choice of these conditions was based on the fact that the higher reduction ratio provides greater bonding assurance for a lined tube joint. The increased forces associated with the 8:1 reduction dictated the use of the 1950°F extrusion temperature. [The 8:1/1825°F large diameter sleeve extrusion required over 1300 tons on the 1400-ton extrusion press.] Bond strengths for all extrusions exceeded 43,000 psi.

A brief fabrication process development effort was conducted, the objective of which was to produce 0.652-inch ID bimetallic tubing having a 0.020-inch tantalum thickness, a 0.060-inch stainless steel thickness and a length of 25 feet. Although this effort was not successful, the indications were that with some further experimentation sound tubing could be produced.

INTRODUCTION

The primary objective of this program was to develop techniques for the extrusion of tandem and sleeve tantalum - 316 stainless steel joints. Starting materials were arc melted and forged tantalum, 99.98 percent pure of grain size ASTM 5 or finer, and 316 stainless steel purchased to QQ-S-763-C.

A tandem joint contains one metal situated axially behind the other, while a sleeve joint contains one component located radially inside the other; i.e., a sleeve joint is a lined tube. In this program the tandem joints were extruded with the stainless steel preceding the tantalum, while the sleeve joints comprised a tantalum lining inside a stainless steel tube.

Dimensions of the bimetallic joints were specified as follows:

	Number Required	ID (in.)	Thickness (in.) Ta	SS	Length (in.)
Large diameter sleeve	16	1.76	0.10	0.22	8 and 12
Small diameter sleeve	6	0.76	0.10	0.22	8 and 12
Tandem	12	1.76	0.12	0.12	8

In the second portion of the program the feasibility of the filled billet approach to long tantalum-lined stainless steel tubes of small diameter was investigated. Target dimensions were 0.652-inch ID, 0.020-inch tantalum, 0.060-inch stainless steel and a length of approximately 25 feet.

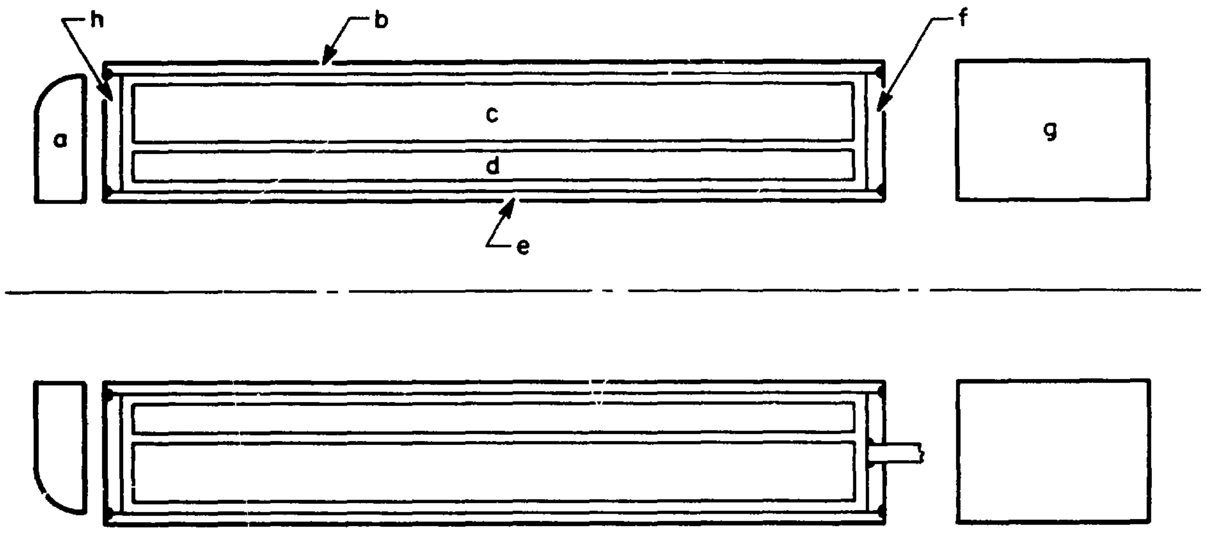
The manufacture of long tubes of small inside diameter requires different techniques from those used for sleeve joints. All of the sleeve joints were produced by extruding hollow composite billets over tool steel mandrels. The mandrel diameters were 1.720 and 0.720 inches respectively for the 1.76 and 0.76 inch inside diameter sleeve joints. For a tube with an inner diameter of 0.652 inch the required mandrel size of approximately 0.61 inch diameter is so small that there is a high probability it would overheat and fail in tension during the course of the extrusion. An additional impediment to a mandrel extrusion is the 25-foot length requirement, which dictates a long billet [increased contact time between mandrel and billet] and a high reduction ratio [increased unit extrusion pressures]. For these reasons it was decided that a filled billet extrusion technique would be investigated to manufacture a 25-foot length of 0.652-inch inner diameter tantalum-lined tubing. This prototype tube was to be preceded by two 5-foot long subscale tubes, each 0.325 inch ID, 0.012 inch tantalum, 0.030 inch stainless steel. The 28.5:1 reduction ratio required for the full size tube was also to be employed for the two subscale tubes.

In the filled billet technique, all components -- steel can, stainless steel outer tube, tantalum inner tube, and core -- are assembled into a billet that is a geometrical enlargement of the desired extruded product; e.g., a 5-inch diameter stainless steel billet component at a 25:1 reduction ratio produces a 1-inch diameter extruded tube [$5/\sqrt{25}$]. Compared to a tube made by conventional extrusion, a tube produced by the filled billet technique looses the benefit of having its inner surface "ironed" by the hardened steel mandrel; it therefore becomes especially important to utilize fine-grain starting material to produce a smooth inner surface in the extruded tube. For material such as tantalum in which the cast ingot size is not too much larger than the required extrusion blank, it is difficult to obtain fine-grain material for the filled billet. One way to solve this problem is to use tantalum sheet as starting stock, since the sheet manufacturing process results in a high degree of grain refinement. The sheet is converted into the desired cylindrical shape by wrapping it around a suitable billet component.

FABRICATION PROCEDURE

Large Diameter Sleeve Joints

Development -- Based upon prior experience in the extrusion of tantalum- and columbium-lined stainless steel tubing, four trial large diameter sleeve extrusions were performed at reduction ratios of 5:1 and 8:1, at 1825°F and 1950°F. A typical billet design for these extrusions is shown in Figure 1; the billet preparation procedure is included in Table 1. Extrusion con-



PART Nº	PART NAME	MATERIAL
a	Nose	Carbon Steel
b	Outer Can	Seamless Carbon Steel Tubing
c	Stainless Steel	
d	Tantalum	
e	Inner Can	Seamless Carbon Steel Tubing
f	Tail Plate	Carbon Steel
g	Cut-off	Graphite
h	Nose Plate	Carbon Steel

Figure 1. Billet design for sleeve joint extrusions.

TABLE I. PREPARATION OF TANTALUM - STAINLESS STEEL EXTRUSION BILLETS

A. CARBON STEEL

After inner and outer cans have been welded to nose plate, evacuation tubes welded to tail plate, and all welds leak checked.

1. Clean in detergent solution.
2. Water rinse: tap water followed by distilled water.
3. Rinse in acetone.
4. Rinse in ethanol; seal in polyethylene bag for shipment.
5. Outgas, 1950°F, 1×10^{-4} torr, 4 hours.
6. Store with dessicant in sealed polyethylene bag.

B. STAINLESS STEEL

Clean as in A-1 through A-4 above, and store in polyethylene bag.

C. TANTALUM

To be prepared immediately prior to billet assembly.

1. Degrease in trichlorethylene vapor degreaser.
2. Wash in acetone.
3. Bright etch, 2 mils/surface [4 mils from thickness]:
   1 part HF [49% Assay]
   2 parts H_2SO_4 [96% Assay]
   2 parts HNO_3 [70% Assay]
4. Rinse thoroughly in tap water.
5. Rinse in distilled water.
6. Rinse in acetone.
7. Rinse in ethanol.

D. BILLET ASSEMBLY

1. Refer to billet drawings for location of tantalum and stainless steel components. Wear nylon gloves.
2.
   a. Sleeve-type joints shall have a 5-mil tantalum foil getter at <u>both ends</u> of the extrusion. These getters will be donut-shaped, with OD and ID corresponding to the stainless steel and tantalum, respectively.
   b. Tandem joints shall have a 5-mil tantalum sheet wrapped once around the outside of the tantalum - stainless steel combined length.
3. Weld tail plate to billet immediately after assembly.
4. Leak check each billet using helium mass spectrometer.
5. Evacuate each billet for 2 hours at room temperature and then heat slowly to 1200°F. Hold at temperature for at least 4 hours.
6. Cool billets to room temperature and seal off.

ditions are summarized in Table II. All extrusions were performed on a 1400-ton Loewy accumulator-driven extrusion press.

TABLE II. EXTRUSION CONDITIONS FOR DEVELOPMENTAL LARGE DIAMETER SLEEVE JOINTS

Extrusion Number	R*	Temp. (°F)	Speed (in./min)	Upset Force (tons)	Running Force (tons)
4370-1	8	1950	100	1225	1200
4370-2	8	1825	100	1325	1300
4371-1	5	1950	100	650	600
4371-2	5	1825	100	675	700

$$\text{Reduction Ratio, } R = \frac{\text{Area of billet}}{\text{Area of extruded tube}}$$

After cropping, the extruded joints were 27 to 31 inches long and were bowed 1/16 to 1/8 inch. The bow was reduced to less than 1/32 inch by cold straightening in a specially designed gag press. The carbon steel inner and outer jackets were then removed by immersion of the joints in a 50 percent nitric acid solution, after which the joints were ground to an outside diameter of 2.400 inches. However, short sections at the ends of some tubes did not completely clean up at this dimension. An 8-inch length of joint was cut from the front and from the rear of each extruded tube. One joint from each tube was bored out sufficiently to remove completely the as-extruded surface, while the insides of the remaining four joints were lightly etched [removing approximately 0.002 inch per surface] in the same hydrofluoric acid/sulfuric acid/nitric acid solution used in the billet preparation procedure. Tables III and IV list the dimensions of these joints before and after machining and etching. The eight joints were shipped to the Westinghouse Astronuclear Laboratories for ultrasonic inspection.

The center and end sections of the extruded sleeves were used for evaluation of the bond quality. Testing consisted of bend, stud-tensile, and notch-fracture tests and metallographic examination of the tantalum - stainless steel interface.

Bend Test -- Longitudinal strips of the entire tube wall were cut from the joints. The edges were polished and inspected by fluorescent dye penetrant, and no indications of voids or cracks were found. The specimens were then bent cold around a 1-inch pin and reinspected by fluorescent dye penetrant. Again, no signs of separation were detected.

TABLE III. DIMENSIONS OF DEVELOPMENTAL LARGE DIAMETER SLEEVE JOINTS AFTER REMOVAL OF CARBON STEEL JACKETS

Joint Number	Outside Diameter			Length
	Front (in.)	Middle (in.)	Rear (in.)	(in.)
S-8-1950	2.491 - 2.422	2.417 - 2.422	2.423 - 2.424	25-1/2
S-8-1825	2.393 - 2.395	2.410 - 2.414	2.440 - 2.441	25-3/4
S-5-1950	2.401 - 2.401	2.424 - 2.428	2.431 - 2.432	30-1/2
S-5-1825	2.432 - 2.435	2.417 - 2.421	2.396 - 2.399	30-5/8

TABLE IV. OUTER AND INNER DIAMETERS OF DEVELOPMENTAL LARGE DIAMETER SLEEVE JOINTS

Joint Number	Condition	Outside Diameter (in.)		Inside Diameter (in.)	
S-8-1950	Machined	2.386-2.386	2.386-2.386	1.743-1.744	1.745-1.745
S-8-1825	Machined	2.376-2.376	2.373-2.373	1.756-1.757	1.760-1.761
S-5-1950	Machined	2.404-2.405	2.404-2.405	1.775-1.776	1.771-1.771
S-5-1825	Machined	2.400-2.402	2.401-2.401	1.750-1.751	1.749-1.751
S-8-1950	Etched	2.402-2.403	2.403-2.403	1.732-1.735	1.732-1.734
S-8-1825	Etched	2.400-2.401	2.401-2.401	1.733-1.736	1.734-1.736
S-5-1950	Etched	2.402-2.403	2.404-2.405	1.742-1.742	1.742-1.742
S-5-1825	Etched	2.401-2.402	2.403-2.403	1.736-1.739	1.740-1.743

Stud Tests -- Stud test specimens as shown in Figure 2 were machined from all sleeve joints. These were tested to failure, and the results of these tests are shown in Table V. One specimen was destroyed accidentally. All other specimens failed in the joint area at ultimate strengths between 42,000 psi and 69,000 psi. One specimen failed in the threads at 74,000 psi. In this specimen the bond was located very close to the base of the stud, which may explain the higher strength on this test. The generally high values for the strength of the bond is a further indication of the high quality of the joints.

TABLE V. STRENGTH OF TANTALUM - STAINLESS STEEL INTERFACE DETERMINED BY STUD TENSILE TESTS

Joint Number	Front*		Rear *	
	Stress at Failure (psi)	Location	Stress at Failure (psi)	Location
S-8-1950	42,200	joint	61,400	joint
S-8-1825	broken before test		64,100	joint
S-5-1950	49,200	joint	58,600	joint
S-5-1825	74,700**	threads	69,000	joint

*specimen taken from front or rear of extruded sleeve joints

**Failed in the tantalum, at root of stud

Notch-Fracture Test -- Notch-fracture tests, as shown schematically in Figure 3, were carried out on strips cut from all extrusions. Because of the thickness and the extreme degree of ductility of the tantalum, it was possible to bend these specimens extensively without fracturing of the material under the notch, which normally occurs in the testing of most other materials. In each case, after considerable deformation, it was possible to separate the stainless steel from the tantalum and to propagate the interface. This was neither alarming nor entirely unexpected, since this test eventually forces a crack to propagate through the base material or the bond. In the case of the stainless steel - tantalum bond at the thicknesses tested, the bond line apparently offers the easiest path for this crack propagation.

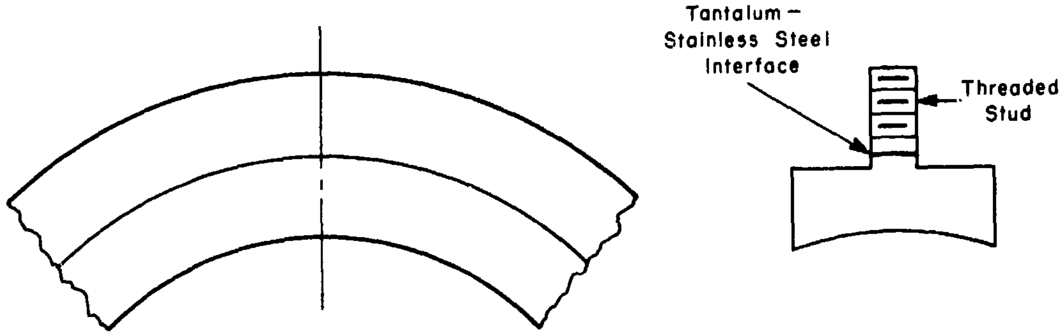


Figure 2. Stud test specimen for sleeve joints.

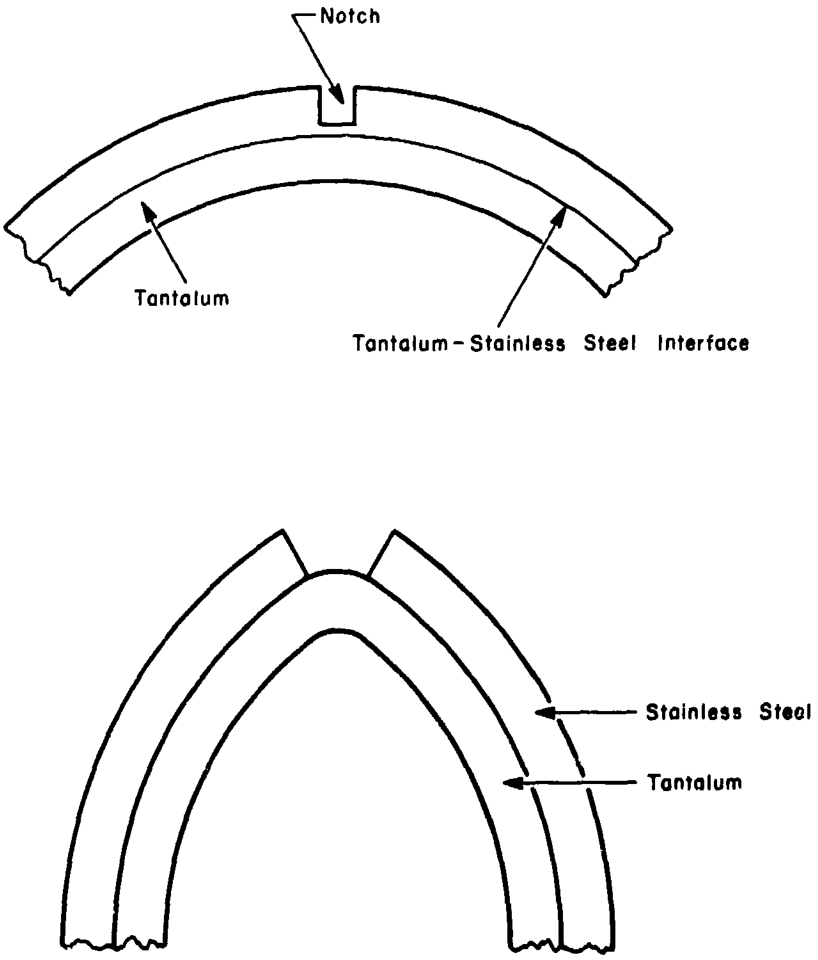


Figure 3. Notch-fracture test, sleeve joint.

Metallography -- Metallographic specimens were taken from all extrusions in order to measure the joint geometry and to determine the nature and thickness of the bond layer. Figure 4 shows rings cut from the sleeve joints. These rings were used to measure tantalum and stainless steel thickness using a toolmaker's microscope, and the results are given in Table VI. [These measurements were taken across a single arbitrary radial line.] Examination of the irregularity of the stainless steel - tantalum line leads to the tentative conclusion that the higher temperature extrusions produced a more regular interface [or a more uniform tantalum and stainless steel wall]. The appearance of the tantalum - stainless steel interface is illustrated in Figure 5. The thickness of the visible intermetallic layer in these joints is tabulated in Table VII, where it is seen that in all cases this layer is less than 0.000039 inch [0.001 mm] thick. The tantalum in all joints is in a cold-worked condition, with grains aligned in the extrusion direction.

Joint evaluation thus indicated high bond quality in all cases. The 8:1 reduction at 1950°F was chosen for the production phase because of the greater bonding assurance of the high reduction extrusion and because of the strong possibility of obtaining a more regular interface.

Production -- Eight large sleeve joint extrusions were performed at 8:1 and 1950°F, at a speed of 100 to 150 inches per minute. Each extruded tube was then cropped front and rear to remove the "extrusion defect." The rear end of each extruded tube displayed a tantalum thickness of approximately 0.200 inch, with a correspondingly thinner stainless steel wall. [Inner and outer diameters of the tubes were approximately 1.730 and 2.430 inches, respectively.] The tubes were then radiographed to indicate the tantalum wall thickness in each tube. Examination of the films showed that in each extruded tube the inner [tantalum] wall gradually thickened over the last 2-1/2 inches of the tube's length. This effect is due to the gradual chilling of the tantalum because of heat loss of the billet to the cooler tooling at the rear of the billet. The length of each tube and the length of the outward-tapering tantalum inner wall of the tubes' end is listed below:

Tube Number	1	2	3	4	5	6	7	8
Overall Length (in.)	$23\frac{7}{8}$	$24\frac{13}{16}$	$26\frac{1}{16}$	$25\frac{1}{8}$	$24\frac{15}{16}$	$24\frac{7}{8}$	$24\frac{7}{16}$	$24\frac{3}{8}$
Taper Length (in.)	$1\frac{1}{2}$	$1\frac{9}{16}$	$2\frac{3}{8}$	$2\frac{3}{8}$	$2\frac{1}{2}$	$2\frac{9}{16}$	$2\frac{1}{2}$	$2\frac{5}{16}$

At the request of the NASA Project Manager, all tubes with the exception of Tubes 1 and 3 were cut to yield one 8-inch and one longer length. Tube 1 was cut into two equal lengths, and Tube 3 was not cut at all. The lengths to which each tube was cut and dimensions after a light clean-up machining

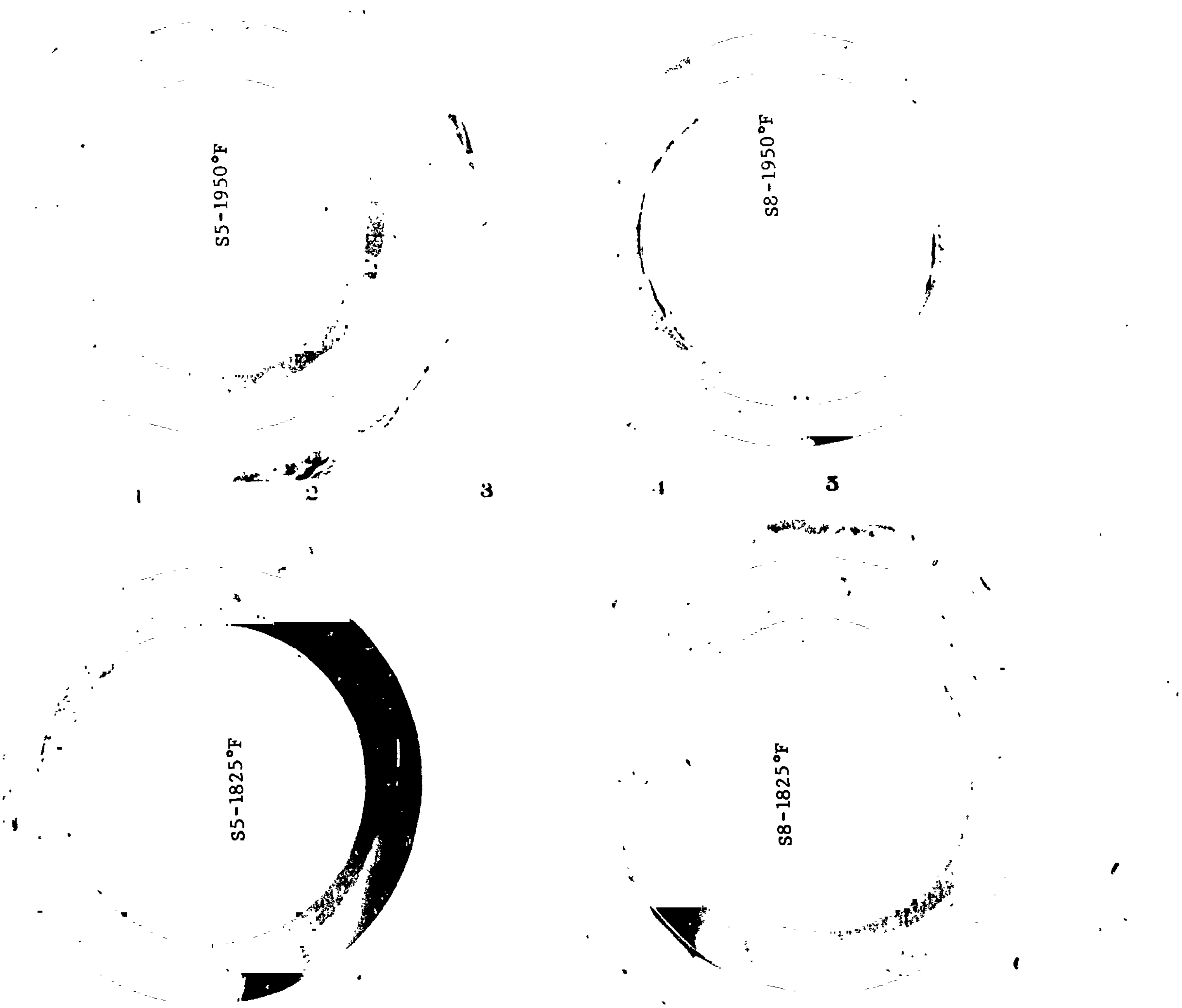


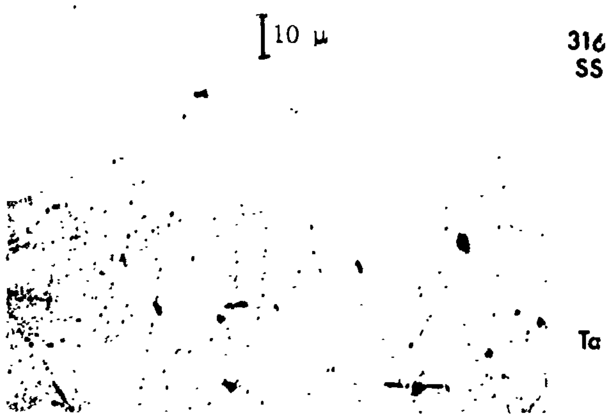
Figure 4. Cross section of developmental large diameter sleeve joints.

TABLE VI. TANTALUM AND STAINLESS STEEL WALL THICKNESS FOR DEVELOPMENTAL LARGE DIAMETER JOINTS

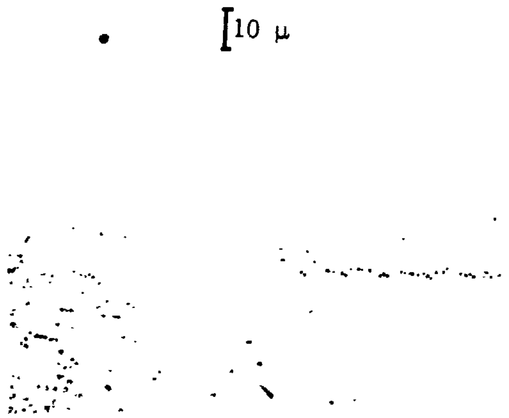
Joint Number	Wall Thickness (in.)	
	Tantalum	Stainless Steel
S-8-1950	.1083	.2275
S-8-1825	.1002	.1982
S-5-1950	.1122	.2191
S-5-1825	.1130	.2202

TABLE VII. THICKNESS OF THE TANTALUM - STAINLESS STEEL INTERMETALLIC LAYER FOR DEVELOPMENTAL LARGE DIAMETER JOINTS

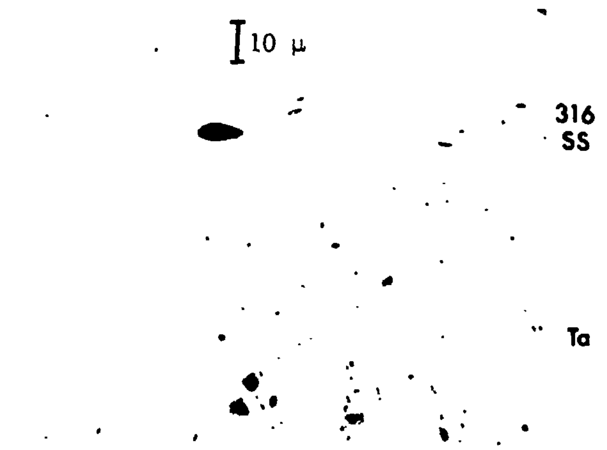
Joint Number	Layer Thickness (10^{-5} in.)
S-8-1950	3.6
S-8-1825	2.4
S-5-1950	3.6
S-5-1825	2.4



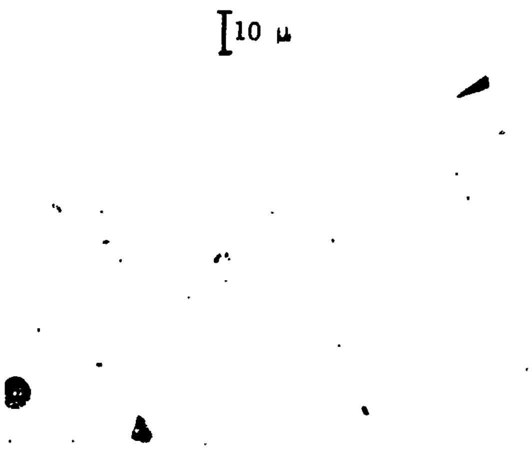
S-8-1950 Plate 260C



S-8-1825 Plate 260B



S-5-1950 Plate 260A



S-5-1825 Plate 260

Figure 5. Transverse sections of developmental large diameter sleeve joints, unetched, bright light.

cut are given in Table VIII. The joints were then forwarded to Westinghouse Astronuclear Laboratory for non-destructive evaluation.

Bend, notch-fracture, and stud tests were performed as on the developmental joints. All test results were equally positive in that no defects of any type were indicated. No flaws were detected by fluorescent penetrant techniques on the bend test samples. As in the case of the prototype joints, the notch-fracture tests all produced eventual separation of the tantalum and stainless steel components. The stud tensile tests [two samples from each tube] yielded a minimum tensile strength of 44,400 psi and an average strength of over 49,000 psi, excluding the unexplained high strength [73,600 psi] of the samples from Tube 1. Test results are tabulated in Table IX.

Photographs of the tantalum - stainless steel interface for the eight joints are shown in Figure 6. Table X lists the respective thicknesses of the stainless steel, tantalum, and intermetallic layers.

TABLE VIII. DIMENSIONS OF PRODUCTION LARGE DIAMETER SLEEVE JOINTS

Tube No.	Length (in.)	OD (in.)	ID (in.) Front*	ID (in.) Middle (at cut)	ID (in.) Rear
1-Front*	11-1/2	2.260	1.716	1.735	-----
Rear	11-1/2	2.370	-----	1.736	1.767
2-Front	8	2.364	1.721	1.727	-----
Rear	15-3/8	2.307	-----	1.734	1.763
3	24-1/4	2.414	1.721	-----	1.763
4-Front	15-3/8	2.369	1.730	1.735	-----
Rear	8	2.361	-----	1.735	1.761
5-Front	8	2.297	1.724	1.732	-----
Rear	14-1/2	2.333	-----	1.731	1.764
6-Front	14-3/4	2.253	1.725	1.737	-----
Rear	8	2.395	-----	1.733	1.752
7-Front	14-1/2	2.285	1.734	1.739	-----
Rear	8	2.308	-----	1.730	1.761
8-Front	8	2.351	1.721	1.732	-----
Rear	14-1/4	2.371	-----	1.734	1.760

*Front, Middle, and Rear refer to positions along the extruded tube; e.g., Middle is at the rear of the front section, or the front of a rear section.

TABLE IX. BOND STRENGTH OF PRODUCTION LARGE DIAMETER SLEEVE JOINTS

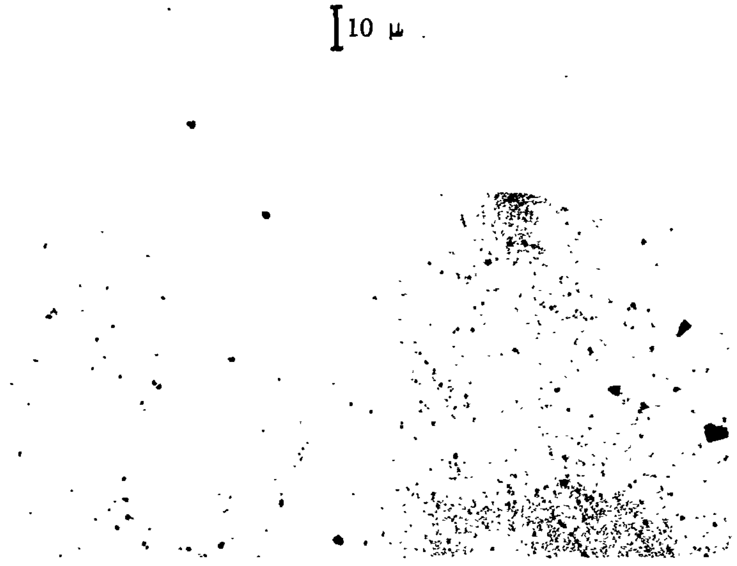
Tube Number	1	2	3	4	5	6	7	8
Ultimate Tensile Strength* (psi x 1000)	73.6	48.7	43.6	54.0	52.8	44.4	53.0	49.7

*Average of two test samples.

TABLE X. CROSS SECTIONAL DIMENSIONS OF PRODUCTION LARGE DIAMETER SLEEVE JOINTS

Tube Number	Ta Thickness (in.)	SS Thickness (in.)	Intermetallic Thickness (10^{-5} in.)
1	.169*	.174*	7.2
2	.099	.226	3.6
3	.118	.249	7.2
4	.113	.230	3.6
5	.137	.228	7.2
6	.108	.230	7.2
7	.110	.230	7.2
8	.101	.247	14.5

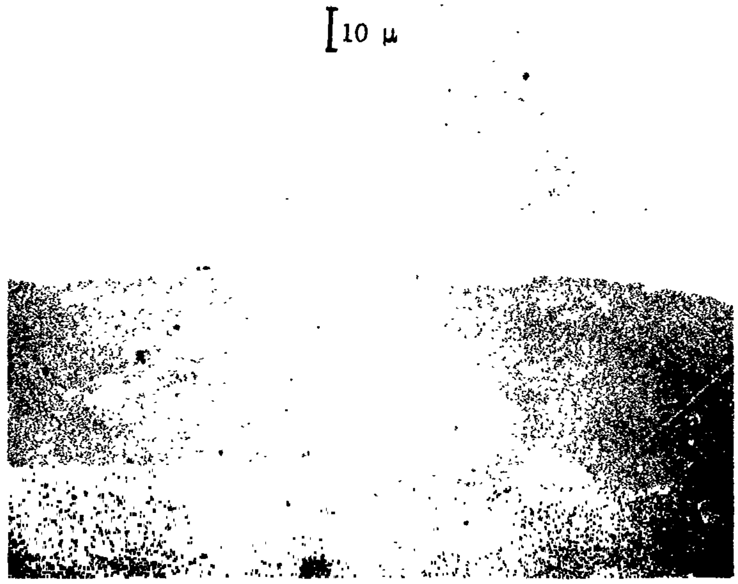
*Sample taken in region of extrusion defect.



316SS

Ta

Tube No. 1 Plate 300

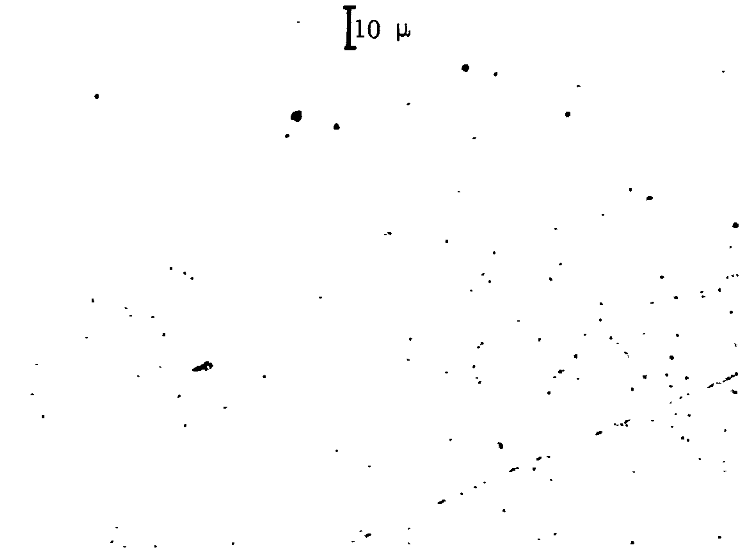


316 SS

Ta

Tube No. 2 Plate 300A

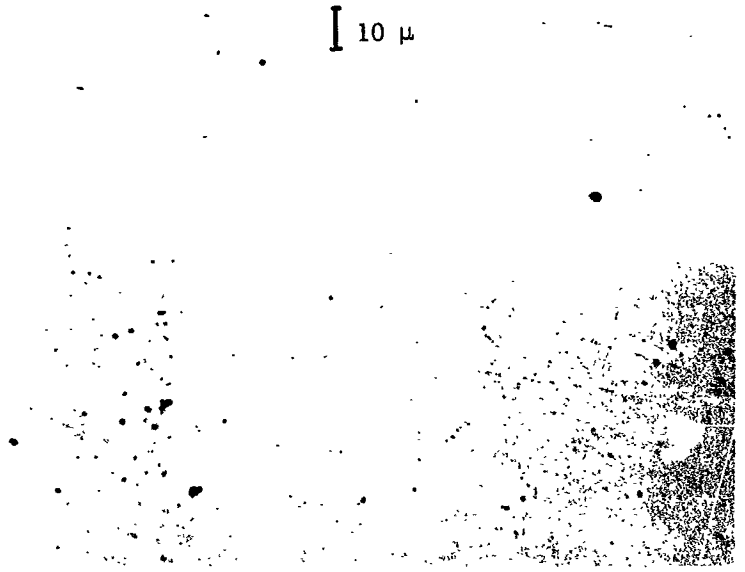
Figure 6. Transverse section of eight production large diameter sleeve joints, unetched.



316 SS

Ta

Tube No. 3 Plate 300B

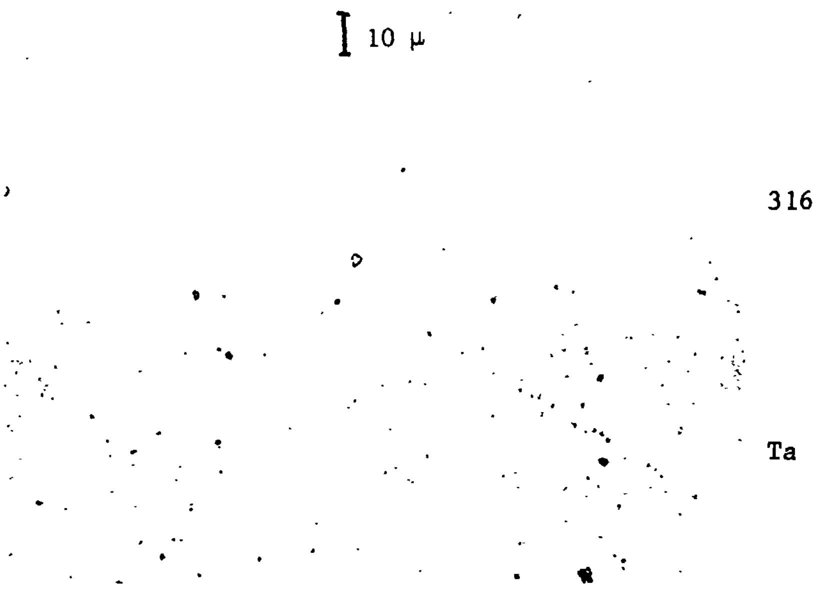


316 SS

Ta

Tube No. 4 Plate 300C

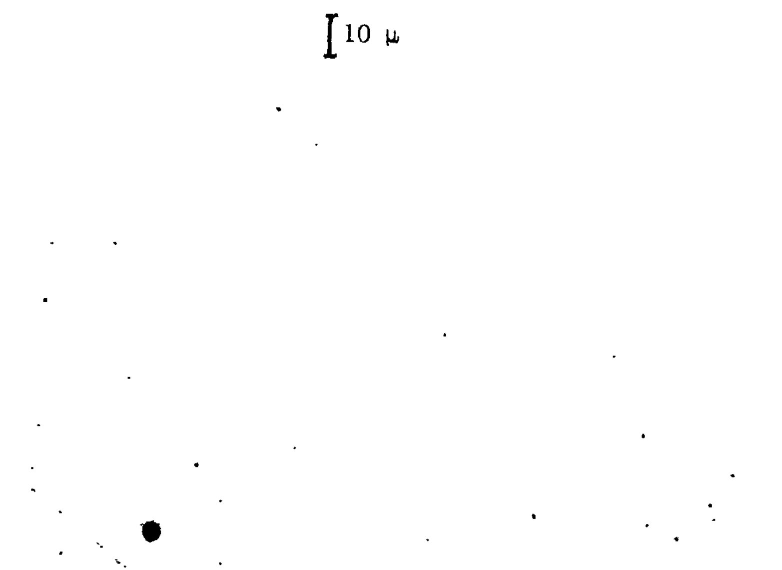
Figure 6. Continued.



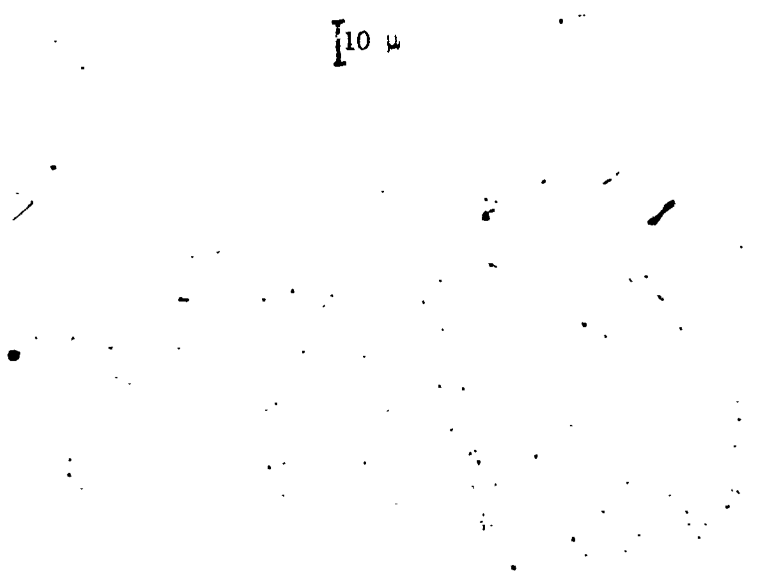
Tube No. 5 Plate 300D

Tube No. 6 Plate 300E

Figure 6. Continued.



Tube No. 7 Plate 300F



Tube No. 8 Plate 300G

Figure 6. Continued.

The discrepancy between the component dimensions for Tube 1 and the other tubes may explain the higher tensile values recorded in stud testing Tube 1. The thicker tantalum layer in this sample indicates that the ring from which the sample was cut was still part of the non-uniform rear extrusion defect, and as such would not be expected to give typical results.

Small Diameter Sleeve Joints

Development -- A pair of small diameter sleeve joint billets were assembled and extruded using the same procedures described for the large diameter sleeve joints. A reduction ratio of 8:1 was used for both billets, one of which was extruded at 1825°F and one at 1950°F [Table XI].

TABLE XI. EXTRUSION CONDITIONS FOR DEVELOPMENTAL SMALL DIAMETER SLEEVE JOINTS

Extrusion Number	R	T (°F)	Speed (in/min)	Upset Force (tons)	Running Force (tons)
4541-1	8	1950	100	1000	875
4541-2	8	1825	100	1050	975

The tool steel mandrels failed during both of these extrusions. Examination of cross sections taken from both tubes [Table XII] after they were cut in two, indicated a partial explanation for the tool failure: in both tubes the tantalum thickness was approximately twice and the stainless steel thickness one-half of design dimensions. This was due to a miscalculation of billet dimensions. Despite their dimensional variances, the four joints were of quality equal to that of the developmental large diameter sleeve joints. Figure 7 shows the appearance of these joints in cross section; the microstructure of the joint interfaces is illustrated in Figure 8.

Based upon the extrusion data and the appearance of the two trial joints, it was decided to extrude the six production joints at 1950°F to minimize stresses on the tool steel mandrel. To further guard against failure, the mandrels were preheated to only 400°F, rather than the usual 900°F, and the extrusion speed increased from 100 inches per minute to 300 - 400 inches per minute, thus keeping the small diameter [0.720-inch] mandrels from overheating.

Production -- Two billets, both lengthened sufficiently over the prototype length to provide material for three 12-inch joints, were assembled and extruded [Table XIII]. The extrusion procedure modifications were successful in that mandrel failures did not occur.

TABLE XII. AS-EXTRUDED DIMENSIONS OF DEVELOPMENTAL SMALL DIAMETER SLEEVE JOINTS

	Front	Middle	Rear
1825°F			
ID (in.)	0.733	0.737	0.736
Ta thickness (in.)	0.215	0.220	0.202
SS thickness (in.)	0.131	0.124	0.133
OD (in.)	1.425	1.422	1.422
SS - Ta intermetallic (10^{-5} in.)	3.6	3.6	3.6
1950°F			
ID (in.)	0.720	0.758	0.767
Ta thickness (in.)	0.223	0.206	0.237
SS thickness (in.)	0.140	0.131	0.109
OD (in.)	1.425	1.436	1.440
SS - Ta intermetallic (10^{-5} in.)	2.4	2.4	2.4

TABLE XIII. EXTRUSION CONDITIONS FOR PRODUCTION SMALL DIAMETER SLEEVE JOINTS

Extrusion Number	R	T (°F)	Speed (in./min)	Upset Force (tons)	Running Force (tons)
4599-1	8	1950	400	900	800
4634-1	8	1950	300	825	750

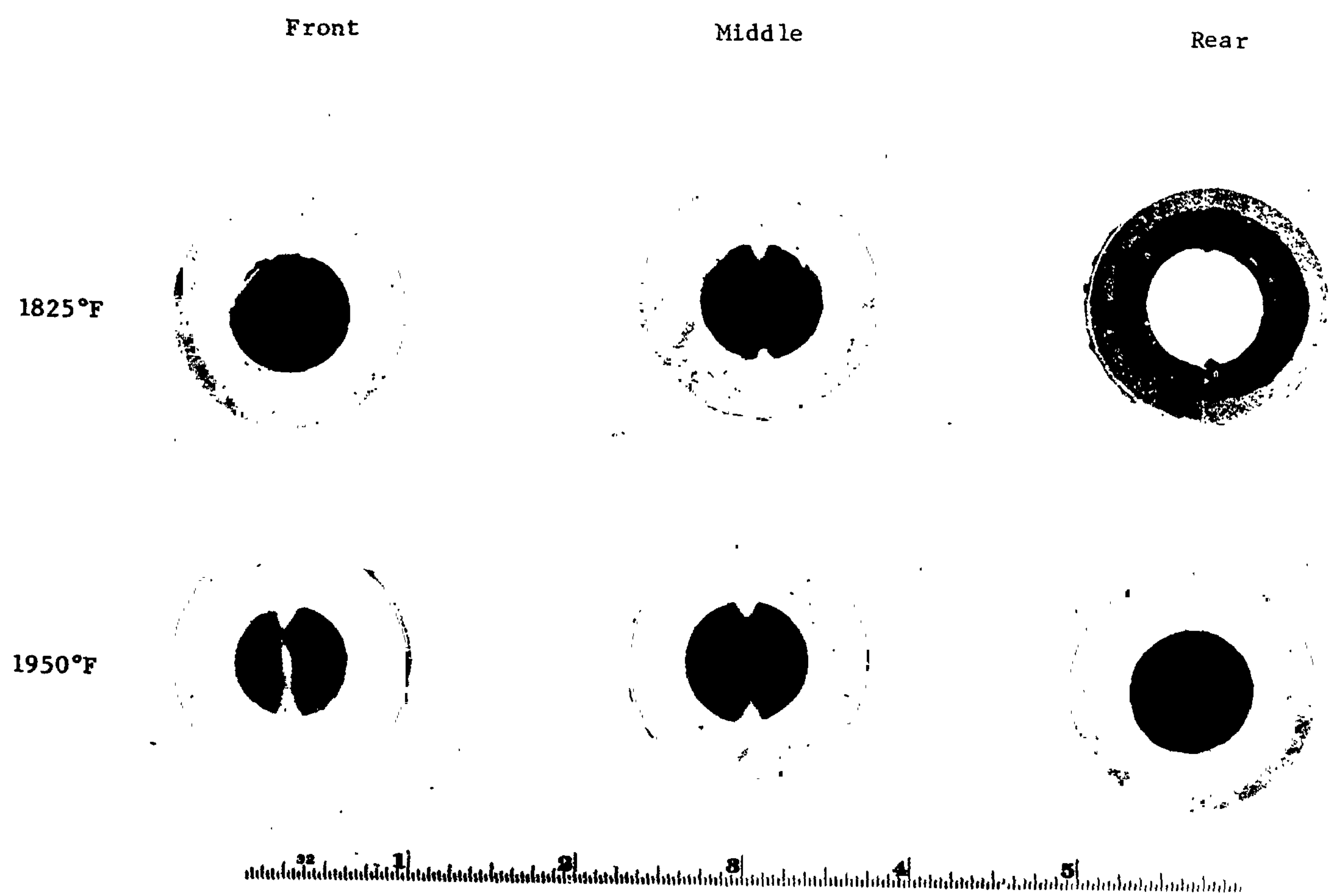


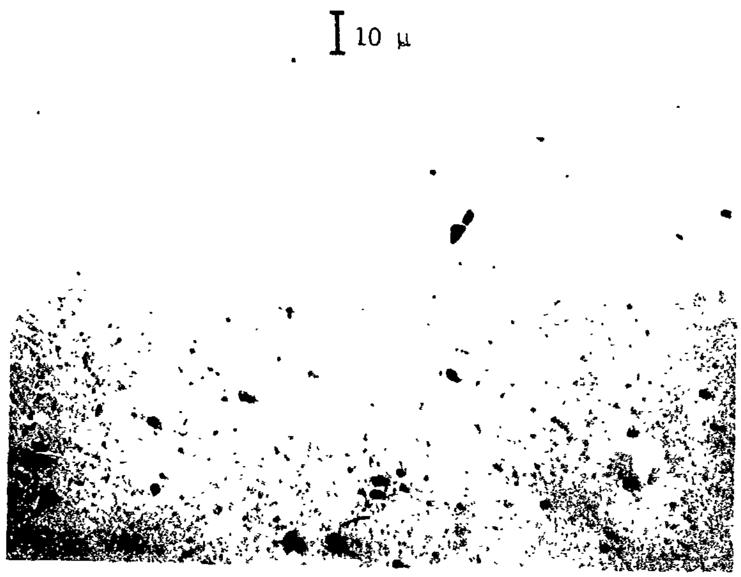
Figure 7. Cross sections of developmental small diameter sleeve joints.

316 SS

Ta

1825°F

Plate 286



316 SS

Ta

1950°F

Plate 286A

Figure 8. Transverse section of developmental small diameter sleeve joints, unetched.

The first tube, after cropping and removal of the carbon steel extrusion can, had the dimensions shown in Table XIV. This tube was cut into three joints, each 10-1/2 inches long, plus two 4-inch evaluation lengths. Examination with a toolmaker's microscope of the samples cut from the front and rear of the tube yielded the dimensional data [wall thickness and filar or intermetallic layer measurements] shown in Table XV. The greater apparent thickness of the tantalum [and correspondingly thinner stainless steel] shown at the rear of the extrusion is again only an indication that this sample is located within the extrusion defect region of the tube. The microstructure of the tantalum - stainless steel interface at the front and rear of this extrusion is shown in Figure 9. Bend and notch-fracture tests yielded results similar to all other sleeve joints, with separation occuring in notch-fracture but not in bending.

The second small diameter sleeve joint extrusion was shipped to NASA in one piece, after acid removal of the steel and a bright etch.

Tandem Joints

Development -- Four tandem joints were extruded under the conditions listed in Table XVI. Figure 10 illustrates a typical billet design for tandem joints. Dimensions of these eight joints after acid removal of the carbon steel cans

TABLE XIV. DIMENSIONS OF FIRST PRODUCTION SMALL DIAMETER SLEEVE EXTRUSION

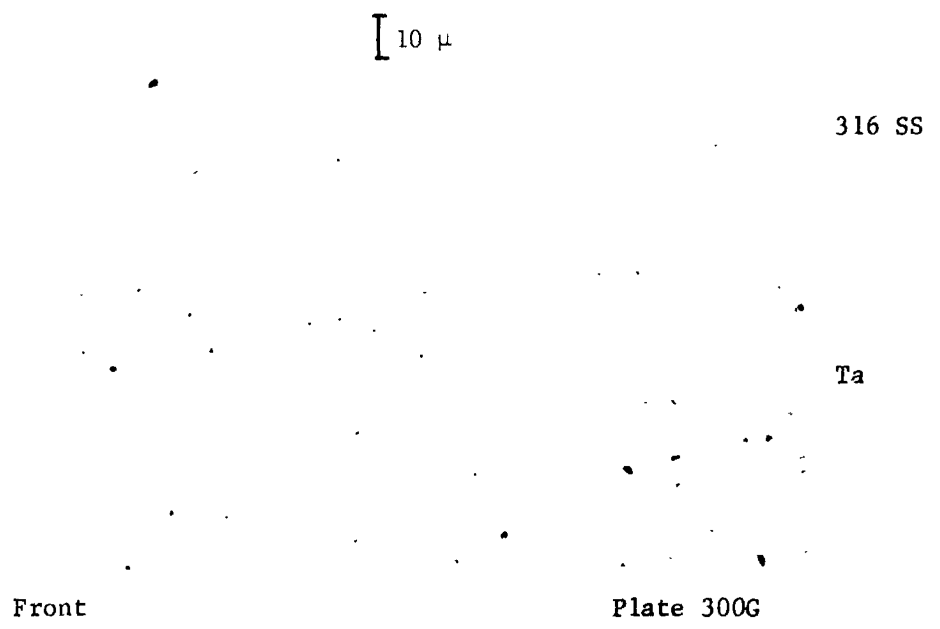
	Front	Middle	Rear
Outside diameter (in.)	1.423	1.428	1.450
Total wall thickness (in.)	0.332	-----	0.332
Tantalum thickness (in.)	0.10	-----	0.12
Stainless steel thickness (in.)	0.23	-----	0.21
Length (in.)	41-7/8		

TABLE XV. CROSS SECTIONAL DIMENSIONS OF FIRST PRODUCTION SMALL DIAMETER SLEEVE JOINT

	Ta Thickness (in.)	SS Thickness (in.)	Intermetallic Thickness (10^{-5} in.)
Front	0.100	0.288	3.6
Rear	0.175	0.165	3.6

TABLE XVI. EXTRUSION CONDITIONS FOR DEVELOPMENTAL TANDEM JOINTS

Extrusion Number	R	T (°F)	Speed (in./min)	Upset Force (tons)	Running Force (tons) SS	Running Force (tons) Ta
4358-1	5	1950	100	525	500	550
4358-2	5	1825	100	550	525	575
4359-1	7	1950	100	775	725	750
4359-2	7	1825	100	850	800	825



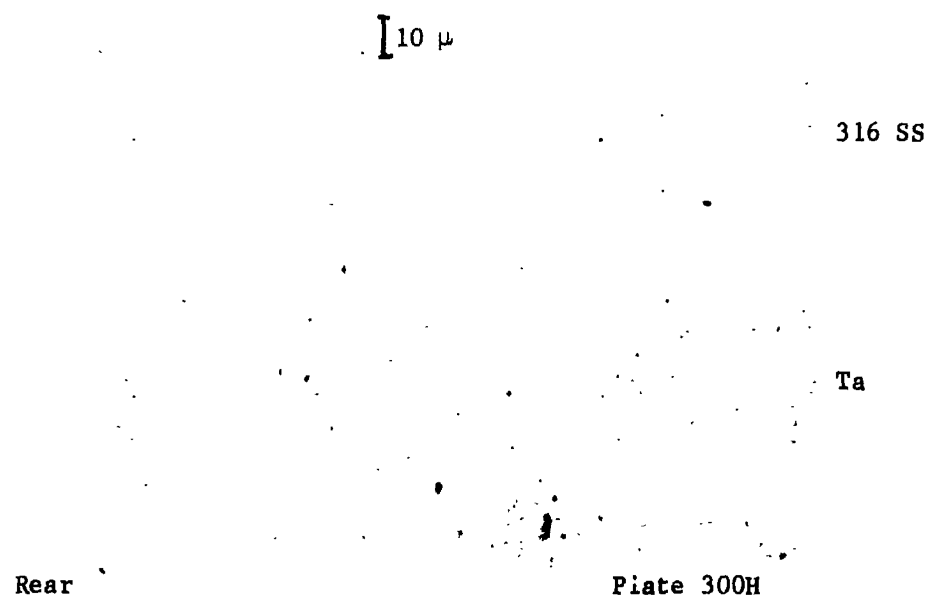
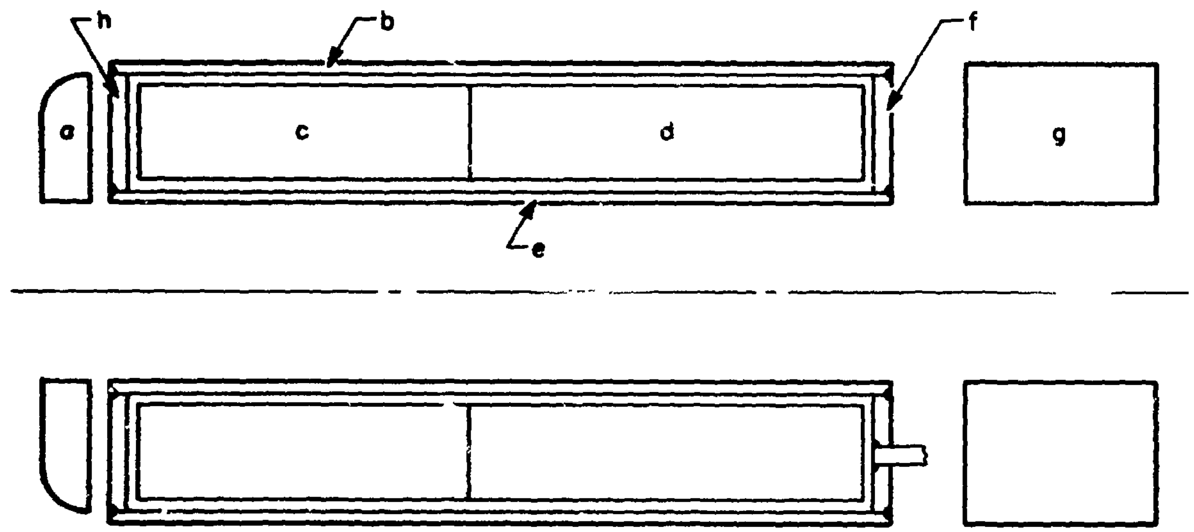


Figure 9. Transverse section of production small diameter sleeve joint, unetched.



PART Nº	PART NAME	MATERIAL
a	Nose	Carbon Steel
b	Outer Can	Seamless Carbon Steel Tubing
c	316 Stainless Steel	
d	Tantalum	
e	Inner Can	Seamless Carbon Steel Tubing
f	Tail Plate	Carbon Steel
g	Cut-off	Graphite
h	Nose Plate	Carbon Steel

Figure 10. Billet design for tandem joint extrusions.

are listed in Table XVII. The joints were then machined to 2.000-inch outside diameter by 1.760-inch inside diameter by approximately 8 inches long. [Figure 11].

The four joints showed no defects when inspected for leaks by the helium mass spectrometer and for bond separation by fluorescent dye penetrant. Longitudinal test specimens were then cut from each joint. No sign of joint separation occurred in any of the bend test samples. Notch fracture tests were performed by cutting through the stainless steel to the bond line and then bending the joints, with the cut on the outside of the bend. As in the case of the sleeve joints, the tantalum and stainless steel components separated at the notch, in all cases.

Measurement of the tantalum - stainless steel intermetallic [Table XVIII] showed values similar to those obtained for the sleeve joints. The length of each joint interface was measured under the microscope:

Joint:	5-1950	5-1825	7-1950	7-1825
Interface Length (in)	0.93	0.92	1.62	1.83

Figure 12 illustrates the microstructure of the interface region for the four joints. It can be seen that, with the exception of the 7:1/1950°F joint, all interfaces are clean and free of voids or inclusions. In the case of the 7:1/1950°F joint, a thin layer [tentatively identified as an oxide] separated the two components in an almost continuous layer. Considering the data presented above for the sleeve joints and for the other three tandem joints, it is fair to say that this black layer was not inherent to the extrusion process, but rather represented foreign material initially present on either the tantalum or stainless steel surface. Its thickness was no greater than the intermetallic layer present on the 5:1 tandem joint extruded at the same temperature.

Since both of the joints extruded at 1825°F were completely free of defects and contained intermetallic layers only half as thick as the 1950°F joints, it was decided to extrude the production joints at 1825°F and 5:1 reduction ratio. The 5:1 reduction ratio was chosen because the interface over the entire length appeared better than that extruded at 7:1.

It is considered desirable for the interface length in a tandem joint to be approximately 1-1/2 inches long. As indicated above, the 5:1 tandem joints had interfaces less than 1 inch long. Therefore, to lengthen the interface, it was necessary to preshape the mating tantalum and stainless steel interface surfaces in the billet. [This is frequently done, but in the opposite manner, in the extrusion of nuclear fuel elements where it is desired to attain

TABLE XVII. AS-EXTRUDED DIMENSIONS OF DEVELOPMENTAL TANDEM JOINTS

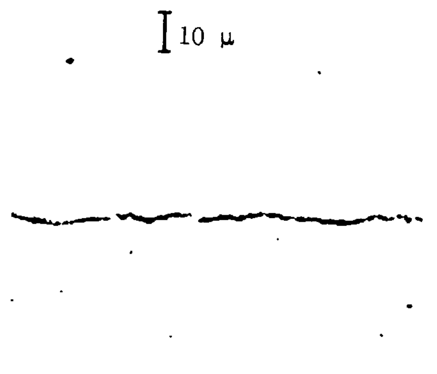
Joint	Outside Diameter (in.)			Inside Diameter (in.)	
	SS	Joint Line	Ta	SS	Ta
5-1825	2.198	2.215	2.225	1.555	1.580
5-1950	2.200	2.226	2.228	1.560	1.585
7-1825	2.278	2.184	2.204	---Not measured---	
7-1950	2.180	2.200	2.190	---Not measured---	

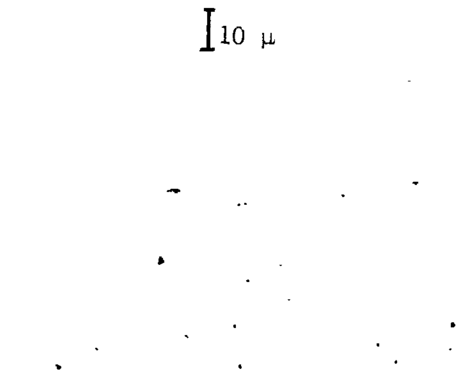
TABLE XVIII. THICKNESS OF TANTALUM - STAINLESS STEEL INTERMETALLIC LAYER

Joint Number	Intermetallic Thickness (10^{-5} in.)
T-5-1825	3.6
T-5-1950	7.2
T-7-1825	3.6
T-7-1950	7.2

5:1 / 1825°F   7:1 / 1825°F   5:1 / 1950°F   7:1 / 1950°F

Figure 11. Prototype tandem joints.



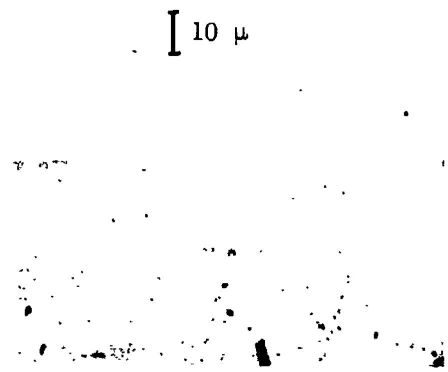


SS

Ta

T-7-1950 Plate 261C

T-7-1825 Plate 261B



SS

Ta

T-5-1950 Plate 261A

T-5-1825 Plate 261

Figure 12. Tandem joints -- tantalum-stainless steel interface, unetched, bright light.

a short, sharply defined interface between the end of the fuel and the end closures.] In the present situation, it was calculated that a simple bevel, 0.4 inch long, would satisfactorily lengthen the joint interface. A single tandem joint billet was therefore assembled in which the stainless steel was machined so that it was 0.4 inch shorter on the inside than on the outside. A corresponding bevel was machined on the mating end of the tantalum component. The billet was extruded at 1825°F at a 5:1 reduction ratio and machined to 2.000 inches outside diameter by 1.760 inches inside diameter. The joint passed mass spectrometer inspection and fluorescent penetrant inspection and was then sectioned. Figure 13 illustrates the bond line which was clean and sound. The interface length had been increased from 0.93 inch, in the case of the 5:1 joints with plane ends, to 1.225 inches in the present case. This interface length was acceptable to NASA, and the decision was made to produce the production joints by the same procedure.

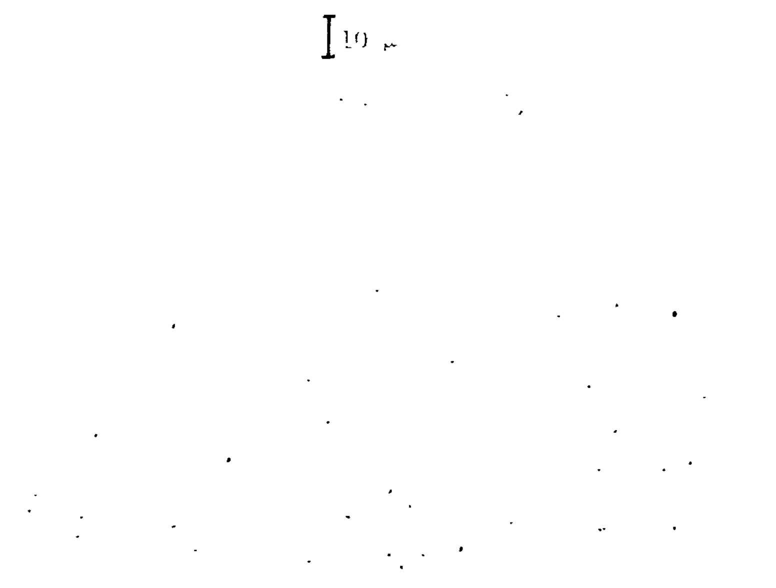
Production -- Twelve tandem joints were extruded at 1825°F at a 5:1 reduction ratio. Extrusion speed for all joints was 100 inches per minute; the upset force was 675 tons; the running force was 750 tons. After acid removal of the carbon steel jackets, the joints were machined to 2.000 inches outside diameter by 1.760 inches inside diameter by 8 inches long [Figure 14]. All joints passed helium mass spectrometer and fluorescent penetrant inspection.

Bimetallic Tubing

Development -- The first of two subscale billets was machined and assembled as shown in Figure 15. The manganese steel core piece served as the solid central filler during extrusion and was to be removed after extrusion by utilizing its exceptional strain hardening characteristics. The core was separated from its containment can by a thin layer of parting agent. The core/parting agent/containment can assembly was separate, inside the hermetically sealed stainless steel/titanium package.

Tantalum foil 0.011 inch thick by 3 inches wide was wrapped seven and one-half times around the outside of the inner steel can to form the cylindrical tantalum billet component. The stainless steel and tantalum were purchased to the same specifications used for the sleeve and tandem joints. Since 316 stainless steel of the proper size was not readily available, 304 stainless steel was used. Billet preparation procedure was the same as used for the joints except that the tantalum was not etched.

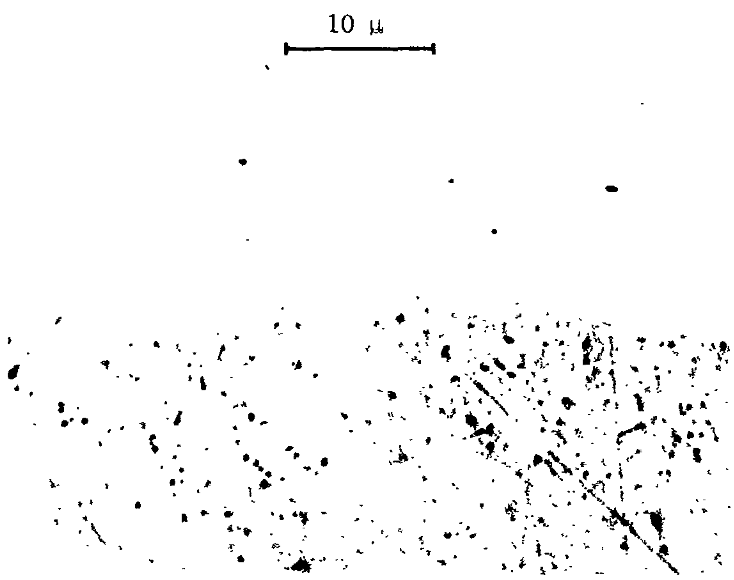
The billet was evacuated, outgassed, sealed off, and heated following standard procedures. The composite rod/tube was extruded satisfactorily at 1950°F through a 0.565-inch die and after cropping was 69 inches long.



316 SS

Ta

500X Plate 286B



316 SS

Ta

2000X Plate 268C

Figure 13. Interface of 5:1/1825°F tandem joint containing beveled interface, unetched.

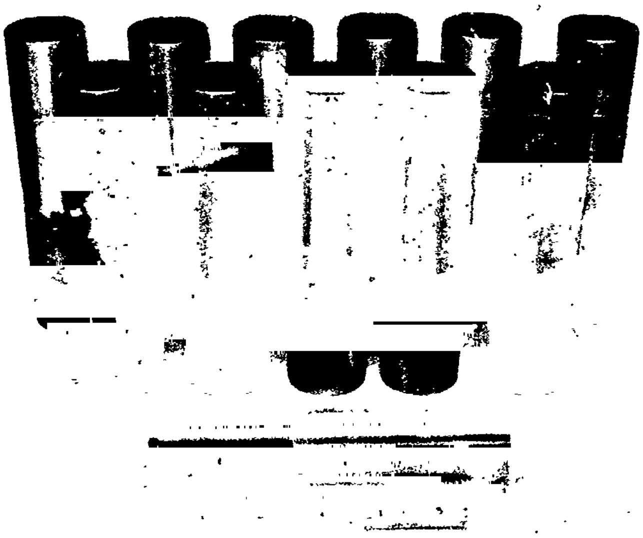


Figure 14. Production tandem joints.

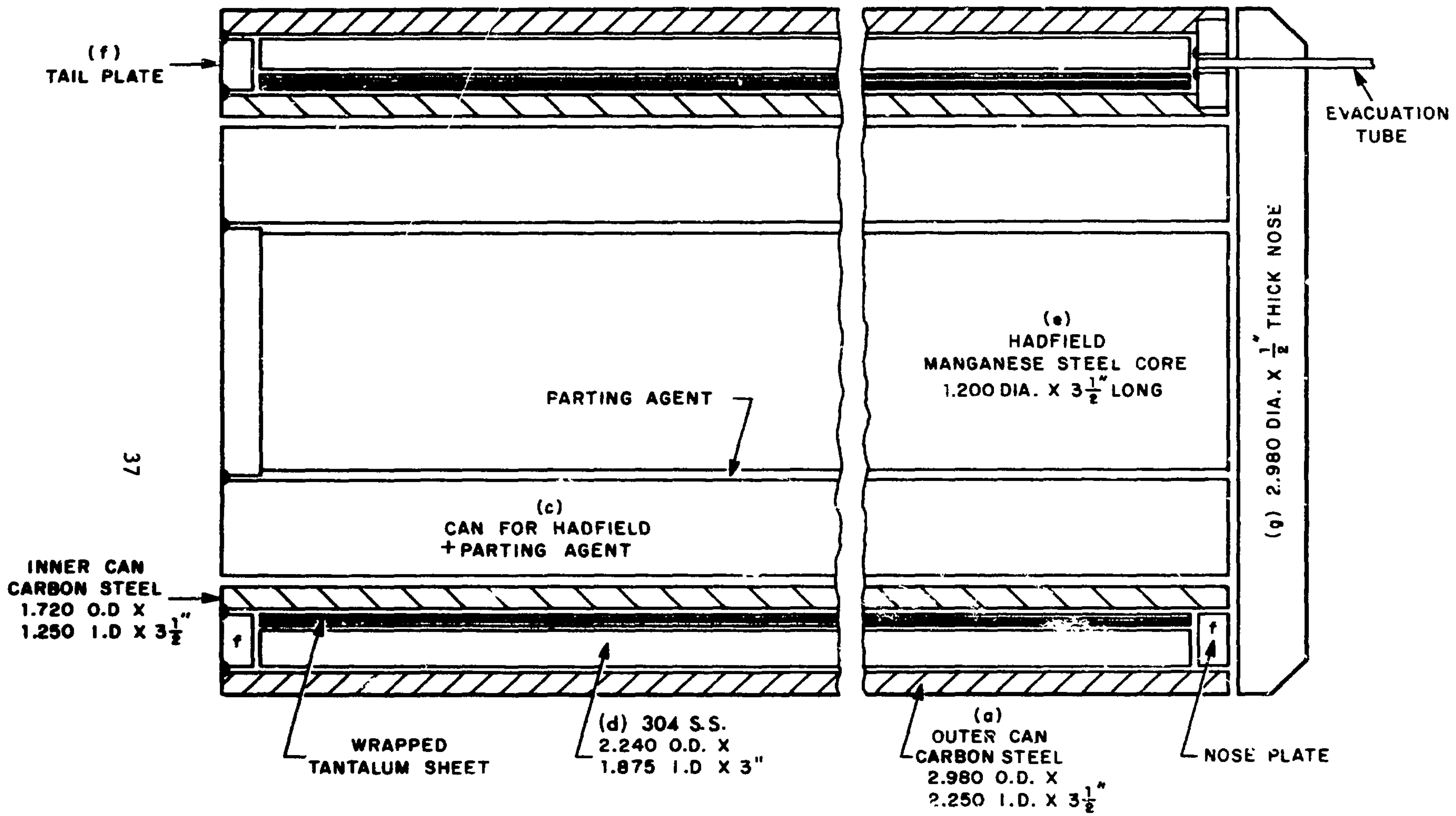


Figure 15. Tantalum-lined 304 stainless steel tube from sheet. Billet details for subscale Tube No. 1.

A tube cutter was used to remove the ends from the outer steel can, the stainless steel/tantalum tube, the inner steel can, and the core containment can. The core rod was then gripped in a drawbench and pulled, resulting in necking and hardening of successive sections such that the core became small enough to be pulled out. The remaining tube was then immersed in a 30 volume percent aqueous nitric acid solution to dissolve the inner and outer carbon steel cans. Dimensions of this tube are given in Table XIX.

TABLE XIX, CROSS SECTIONAL DIMENSIONS OF TANTALUM-LINED 304 STAINLESS STEEL TUBE NO. 1 MADE FROM WRAPPED TANTALUM SHEET

Position	OD (in.)	ID (in.)	Ta Thickness (in.)	SS Thickness (in.)
Front	.430	.310	.014 - .019	.042 - .045
Middle	.407	.306	.011 - .017	.033 - .035
Rear	.407	.304	.012 - .017	.035 - .037
Objective	.416	.326	.0125	.032

The greater thickness of the tantalum and stainless steel components at the front end of the tube indicate that this sample is still in the "extrusion defect" region.

Figure 16 illustrates the microstructure of the tantalum lining where it contacts the stainless steel. Interfaces can be seen between the stainless steel and tantalum and between most of the tantalum layers. As the photograph indicates, the inner tube surface is smooth.

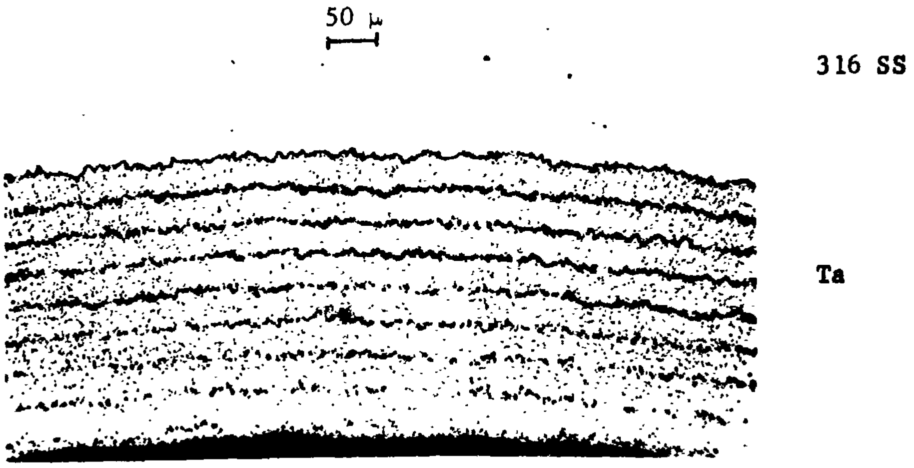


Figure 16. Cross section of subscale tantalum-lined stainless steel Tube 1, made from 0.010 inch tantalum sheet, 100X, Plate 314B.

A second billet was assembled, similar to the first in all respects except that the tantalum sheet was 0.015 inch thick to allow for acid pickling prior to the assembly process. The tantalum was degreased and etched in a solution of hydrofluoric acid, sulfuric acid, and nitric acid to remove approximately 1 mil of metal from the sheet surfaces. The tube was processed in the same manner as Tube 1 and resulted in similar dimensions. The photomicrographs of the tube (Figure 17) show that an interface is still visible between the tantalum and stainless steel and between the tantalum layers at the front and middle of the tube but that no tantalum/tantalum interfaces can be seen at the rear of the tube. The rough inner surface of the tube may have been caused by the pre-extrusion etching of the tantalum.

Because the acid cleaning of the tantalum produced such variable bonding results and because the 0.015-inch tantalum was more difficult to work with, it was decided to produce the full size tube from 0.010-inch sheet, cleaned but not etched.

Production -- The production billet was assembled as shown in Figure 18, utilizing 10 feet of 12-inch wide 0.010-inch thick tantalum sheet. Assembly and extrusion were performed as before, producing a composite rod 0.861 inch in diameter by 20 feet long. Attempts to pull the manganese steel core from the tube were unsuccessful due to surface cracks in the core rod. It is believed that the longer heating time [as compared to the subscale tubes] required for this larger billet caused a surface reaction between the manganese steel and parting agent, transforming the tough austenite phase to the weaker and more brittle martensite structure. The core was removed by an acid drilling technique. Dimensions at the center of this tube are given in Table XX.

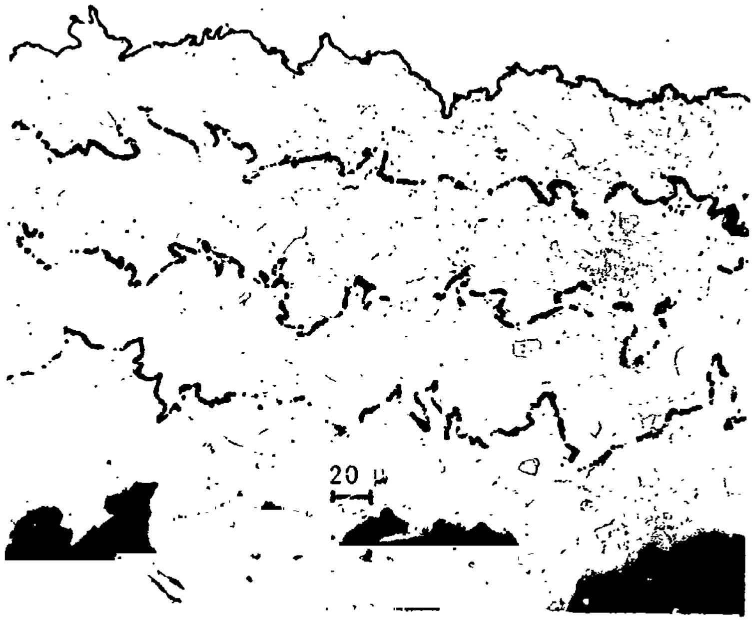
TABLE XX. CROSS SECTIONAL DIMENSIONS AT THE CENTER OF FULL SIZE TANTALUM-LINED STAINLESS STEEL TUBE FROM WRAPPED SHEET

	OD (in.)	ID (in.)	Ta Thickness (in.)	SS Thickness (in.)
Actual*	.805	.632 - .634	.025 - .026	.065
Objective		.0625	.020	.060

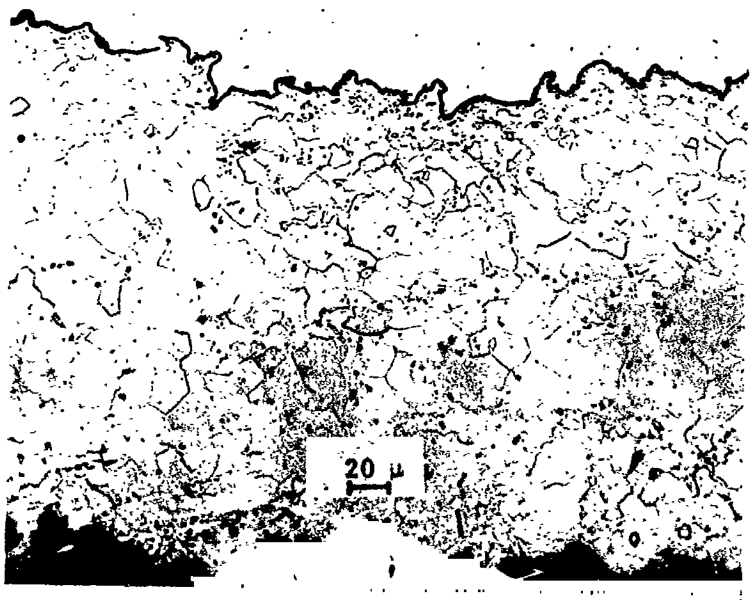
*After belt sanding

The oversize wall dimensions are a result of the cumulative clearances in the billet [which were larger than for the 3-inch long subscale billets]. This also resulted in the tube not achieving the target length of 25 feet.

Figure 19 illustrates the tube microstructure at the front, center, and rear. It can be seen that the tantalum bonding is of variable quality: The innermost tantalum layer [relative to the tube ID] is unbonded at the front, and the outermost layer has separated from the tantalum and the stainless steel at the middle and rear of the tube.



a) Center of tube (Plate 322A)



b) Rear of tube (Plate 322B)

Figure 17. Cross-sections of subscale tantalum-lined stainless steel Tube No. 2, made from 0.015-inch tantalum sheet.

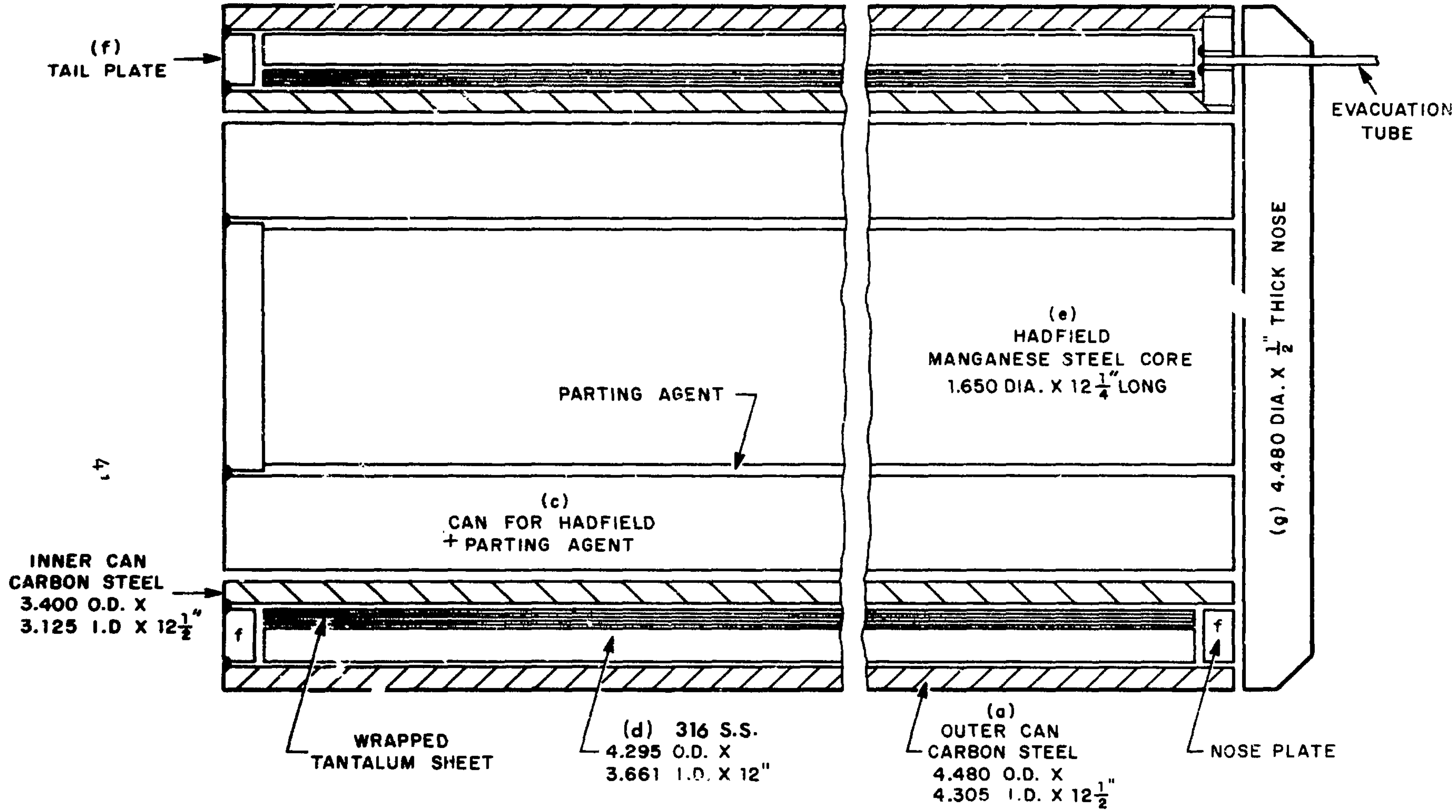
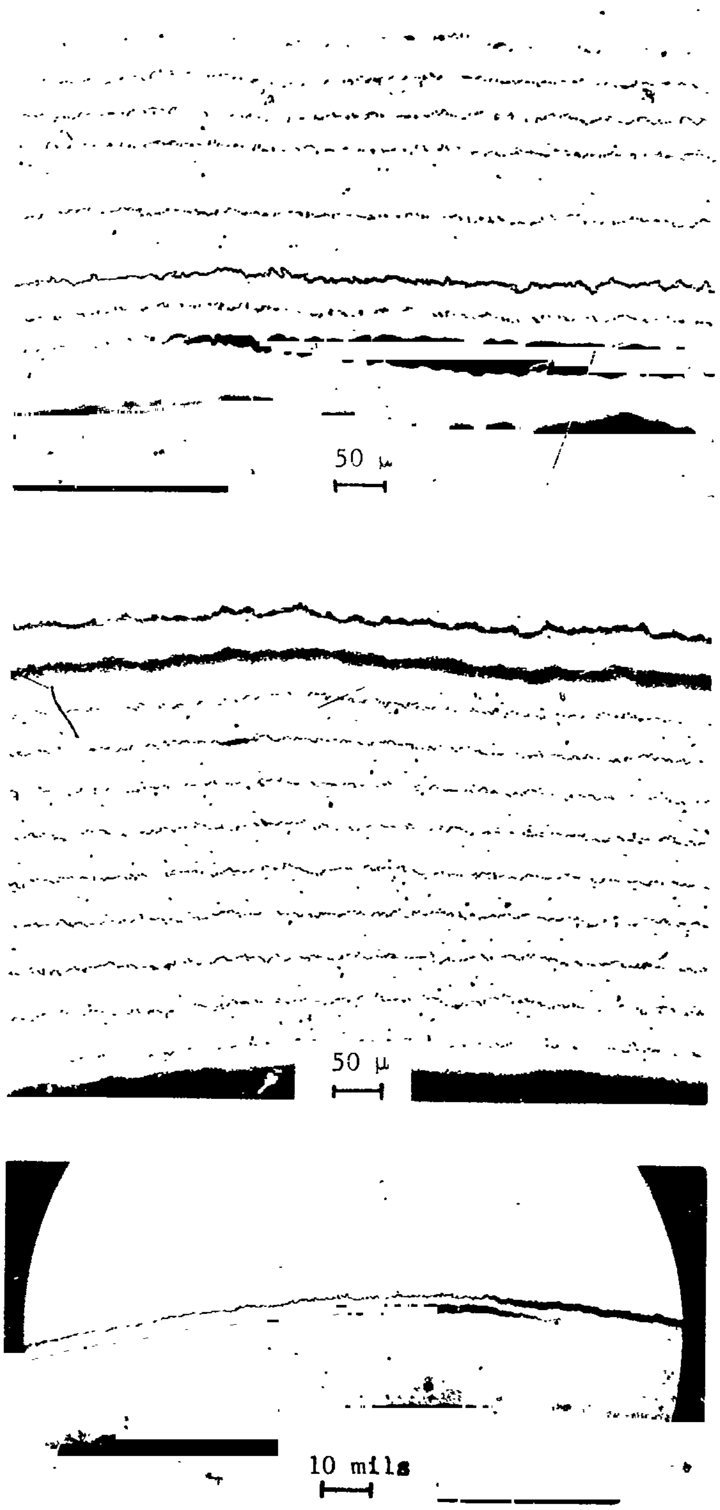


Figure 18. Tantalum-lined 316 stainless steel tube from sheet. Billet details for full-size tube.



316 SS

Ta

a) Front, 100X

316 SS

Ta

b) Center, 100X

316 SS

Ta

c) Rear, 25X

Figure 19. Cross sections of full-size tantalum-lined stainless steel tube made from 0.010-inch tantalum sheet.

CONCLUSIONS

Techniques were developed for the fabrication by hot extrusion of sleeve and tandem tantalum - 316 stainless steel joints. The sleeve joints were 0.76 and 1.76 inches inside diameter. The tandem joints were 1.76 inches inside diameter. The tested joints were all leak tight and showed no signs of non-bond. Tensile strengths of over 40,000 psi transverse to the bond line were obtained for the large sleeve joints.

It was demonstrated that high quality tandem joints can be made at 1825°F at a reduction ratio of 5:1. High quality sleeve joints can be made at 1950°F at a reduction ratio of 8:1. The sleeve joints offer the quality assurance advantage that samples may be taken from regions adjacent to the joint without affecting the joint itself.

The brief composite tubing investigation indicates that tubing can be made by a wrapped sheet filled billet technique but that the procedure used cannot produce uniformly satisfactory tantalum/tantalum or tantalum/stainless steel bonds for a 0.652-inch, 25-foot long tube. It is possible that further work could establish the precise nature of the bonding difficulties which would result in a technique for producing tubes with the same bond quality as the sleeve and tandem joints.

An alternate and more reliable route for introducing fine grain tantalum into the extrusion billet would be to use pressed-and-sintered tantalum powder as starting material. The powder billet would produce even smoother inner surfaces in the extruded tube, based on its very fine grain size and isotropic condition.

A second, though more costly, alternative would be to refine the grain size of wrought tantalum by a redundant working scheme in which the cast bar is reduced in size by forging or extrusion at a low temperature, upset back to its original size, and then annealed at a low temperature. The cycle can be repeated as often as is necessary to achieve the desired grain size. A single cycle consisting of 75 percent cold work [total] plus a one-hour anneal at 2100°F has been used to refine the as-cast grain size of a 3-3/4 inch dimaeter bar to ASTM 5 - 6.

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Robert G. Jenkins, Whittaker Corporation/Nuclear Metals Division, "Process Development and Pilot Production of Tantalum Lined 316 Stainless Steel Tubing," Contract NAS 3-9435 for National Aeronautics and Space Administration/Lewis Research Center, 30 October 1967.