NASA-CR-72394

WANL-PR-(ZZ)-003

DETERMINATION OF OPTIMUM FABRICATION TECHNIQUES FOR THE PRODUCTION OF AUSTENITIC REFRACTORY BIMETALLIC TUBING

MIDPOINT REPORT

By

R. W. Buckman

J. N. Kass

Prepared for

National Aeronautics and Space Administration

Lewis Research Center

Space Power Systems Division

Under Contract NAS 3-10601

Astronuclear Laboratory

Westinghouse Electric Corporation

NASA-CR-72394

WANL-PR-(ZZ)-003

DETERMINATION OF OPTIMUM FABRICATION TECHNIQUES FOR THE PRODUCTION OF AUSTENITIC REFRACTORY BIMETALLIC TUBING

MIDPOINT REPORT

By

R. W. Buckman

J. N. Kass

Prepared for

National Aeronautics and Space Administration

Lewis Research Center

Space Power Systems Division

Under Contract NAS 3-10601

INFORMATION CATEGORY

Unclassified

R. O. Bagley 5/3/68

AUTHORIZED CLASSIFIER DATE

Astronuclear Laboratory

Westinghouse Electric Corporation

FOREWORD

This report describes progress at the Westinghouse Astronuclear Laboratory under NASA Contract NAS 3-10601 during the period from March 29, 1967 to October 23, 1967. This work is administered by the Nuclear Power Technology Branch of NASA, Cleveland, Ohio with Mr. P. Stone acting as Technical Manager.

The investigation is being conducted by R. W. Buckman, Jr. and J. N. Kass of the Astronuclear Laboratory.

Astronuclear Laboratory

PRECEDING PAGE BLANK NOT FILMED.

TABLE OF CONTENTS

Section		Page
	FOREWORD	iii
1.0	INTRODUCTION	1-1
2.0	BIMETAL TUBING FABRICATION	2-1
3.0	TEST PROGRAM	3-1
4.0	PROGRAM STATUS	4-1
5.0	EXPERIMENTAL TECHNIQUES AND RESULTS	5-1
	5.1 Dimensional Measurements	5-1
	5.2 Dye Penetrant Testing	5-1
	5.3 Ultrasonic Testing	5-4
	5.4 Metallography	5-11
	5.5 Thermal Expansion Measurements	5-17
	5.6 Creep Burst Testing	5-17
	5.7 Thermal Cycle Testing	5-17
6.0	FUTURE WORK	6-1
7.0	REFERENCES	7-1

LIST OF ILLUSTRATIONS

Figure		Page
1	Extrusion Billet Design Used by Metalonics Corp.	2-2
2	Extrusion Billet Design Used by Nuclear Metals	2-5
3	Nuclear Metals Extrusion Setup	2-6
4	Inspection and Test Schedule for Ta/316SS Bimetal Tubing	3-2
5	Aerojet Downey Bimetal Tubing	5-5
6	As-Received Metalonics Ta/316SS Bimetal Tubing	5-6
7	Schematic Illustration Showing Pulser and Receiver Signals	5-7
8	Ultrasonic Test Apparatus	5-9
9	Unbonded Area in Metalonics Tubing Identified By Ultrasonic Testing 400X	5-13
10	Typical Interface in As-Explosively Bonded to Size Tubing	5-15
11	Longitudinal Section of Metalonics Tubing	5-16
12a.	Thermal Expansion Apparatus	5-18
12b.	Thermal Expansion Schematic	5-18
13	10,000 psi Pressure Test Rig Schematic	5-19
14	Schematic of Thermal Cycle Test Apparatus	5-20

LIST OF TABLES

Tables		Page
1	Starting Material Chemical Analysis for Metalonics Ta/316SS Bimetal Tubing (Vendor Furnished)	2-3
2	Extrusion Temperature for Metalonics Tubing	2-4
3	Chemical Analysis for Nuclear Metals Starting Material (Vendor Certifications)	2-7
4	Extrusion Data for Nuclear Metals Tubing	2-9
5	Dimensional Test Results	5-2
6	Dye Penetrant Results	5-3
7	Ultrasonic Test Results	5-12

1.0 INTRODUCTION

This report describes the work accomplished on Contract NAS 3-10601, "Determination of the Optimum Fabrication Techniques for the Production of Austenitic/Refractory Bimetallic Tubing", from March 29, 1967 to October 23, 1967. The purpose of this investigation is to evaluate and characterize three lots of refractory (tantalum)/austenitic (Type 316SS) bimetal tubing using both destructive and non-destructive testing. The tubing dimensions are nominally 0.650 inch ID x 0.080 inch wall and 15 feet minimum length. Tantalum, 0.020 inch thick comprises the inner liner of the tube which is clad with 0.060 inch thick type 316SS. After evaluation, a portion of the tubing will be used to fabricate a prototype tube-in-tube boiler for the SNAP-8 system. The SNAP-8, a liquid metal Rankine cycle power conversion system uses mercury as the working fluid which is heated by NaK. A detailed description of the SNAP-8 system can be found elsewhere [1].

The material selected initially for containing the mercury did not provide adequate corrosion resistance in critical areas of the boiler and it became apparent that a substitute material would have to be used [1]. Both columbium and tantalum exhibit very excellent corrosion resistance to mercury and the austenitic stainless steel is resistant to NaK under the environmental conditions predicted for the SNAP-8 boiler [1]. Work by Buckman and Goodspeed [3] showed that tantalum exhibited excellent compatibility with the stabilized 300 series stainless steels at temperatures up to 1400°F which is 50-100°F higher than the peak operating temperature of the SNAP-8 boiler.

The Ta-316SS combination was selected as the optimum combination based in part on prior work at WANL [2, 3]. The bimetal tubing to be evaluated was furnished by NASA and consisted of three lots of tubing. Each lot was to consist of approximately 300 feet total length with the minimum tube length to be 15 (fifteen) feet. Two lots, fabricated by extrusion to size, were produced by the Nuclear Metals Division of Whittaker Corp. and Metalonics Corp., respectively. The third lot of tubing was fabricated by explosive bonding to size by AGC, Downey.

2.0 BIMETAL TUBING FABRICATION

The Ta/316SS bimetal tubing was fabricated using two different techniques; explosive bonding to size, and co-extrusion to size.

The explosively bonded to size bimetal tubing, produced by AGC, Downey, was manufactured by placing a tantalum tube inside a 316SS tube. The tubes are sized such that a small uniform standoff distance is maintained between the tubes. The tantalum tube is filled with a solid medium, to prevent collapse during forming, and a layer explosive charge is placed around the outer 316SS tube. The explosive charge is ignited at one end and the travelling shock wave collapses and bonds the 316SS tube to the tantalum tube. Other details of the fabrication procedure are unavailable.

Co-extruded to size 316/Ta bimetal tubing was produced by the Metalonics Corp., Boston, Massachusetts and the Nuclear Metals Div. of Whittaker Corp., W. Concord, Massachusetts.

The fabrication technique used by Metalonics to produce Ta/316SS bimetal tubing consisted of extruding a filled billet and subsequently removing the core. The extrusion billet design used is shown in Figure 1. Starting material chemical analyses are given in Table 1. The billet was evacuated, heated to 1000°F in a vacuum of 10^{-6} torr and then sealed. The extrusion billets were heated for 2-1/2 hours in an argon atmosphere (Table 2) and then extruded through a Zircoa die, 0.830 inches in diameter. After extrusion the rods were straightened while hot and air cooled. The ends were cropped to yield about 16 foot lengths and the C1018 mild steel cores were leached out with nitric acid at about 216°F. Finally, the OD was sized by belt sanding and the ID by pulling a torpedo mandrel through followed by alumina honing.

Nuclear Metals produced the Ta/316SS bimetal tubing by extruding a hollow billet over a mandrel. The billet design and extrusion set up used are shown in Figures 2 and 3 respectively. Starting material chemical analyses are listed in Table 3. After outgassing at 800°F and a 10^{-4} mm H_9 vacuum, the billets were heated in graphite containers

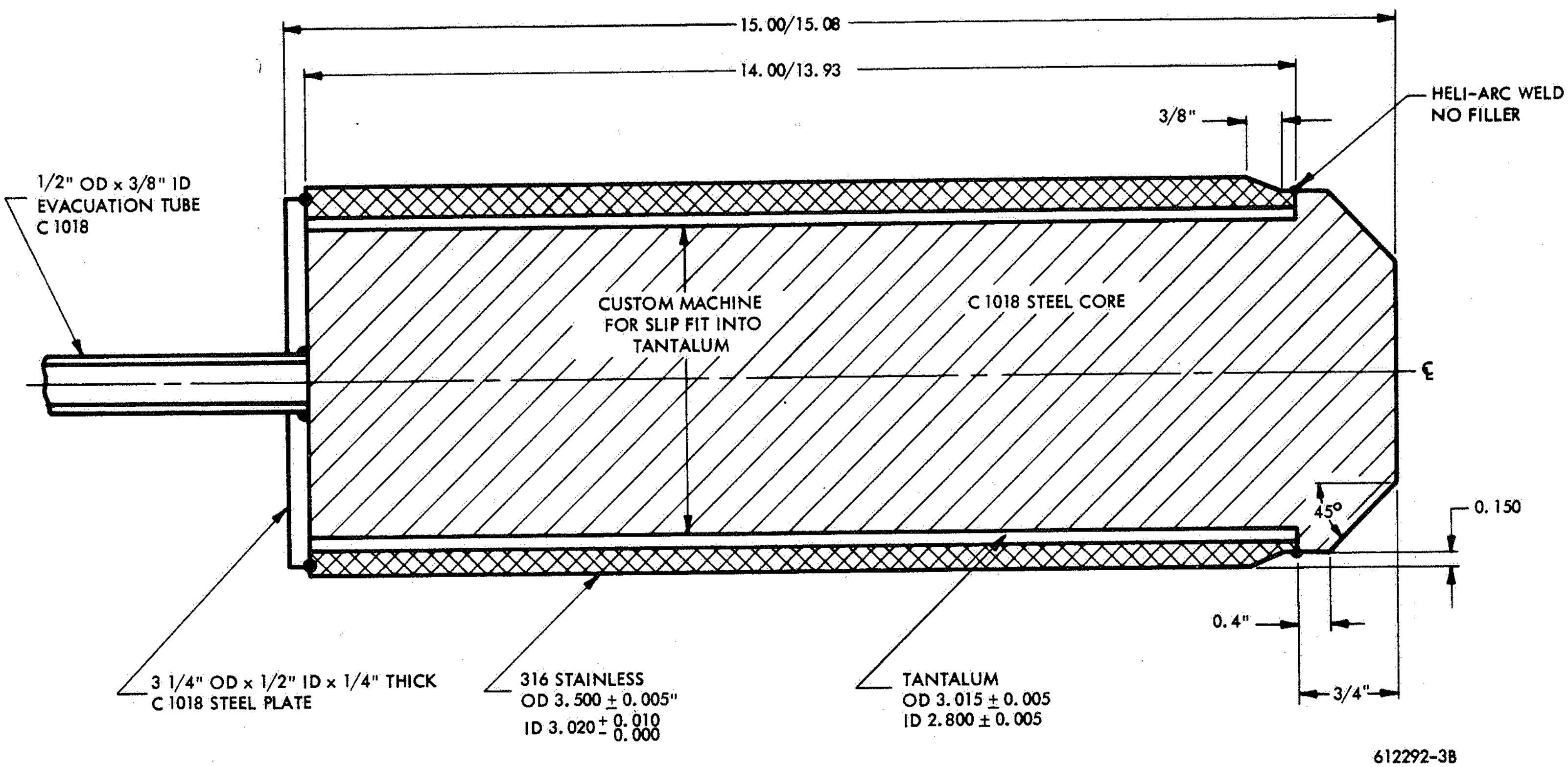


Figure 1. Extrusion Billet Design Used by Metalonics Corp.

TABLE 1

STARTING MATERIAL CHEMICAL ANALYSIS FOR METALONICS Ta/316SS BIMETAL TUBING (VENDOR FURNISHED)

Tantalum Tubing 3.015" OD x 2.80" ID Lot 6717 (Kawecki Chemical Co.)			
Analysis - ppm			
C	25	O_2	<50
H_2	10	N_2	1.1
Cb	25	Ti	<5
Fe	<5	Mn	<5
Si	<5	Sn	N. D.
Ni	<5	Cr	<5
Ca	5	Na	N. D.
Al	<5	Mo	<5
Cu	<5	Zr	<5
Co	N. D.	Mg	<5
B	<5	W	<100

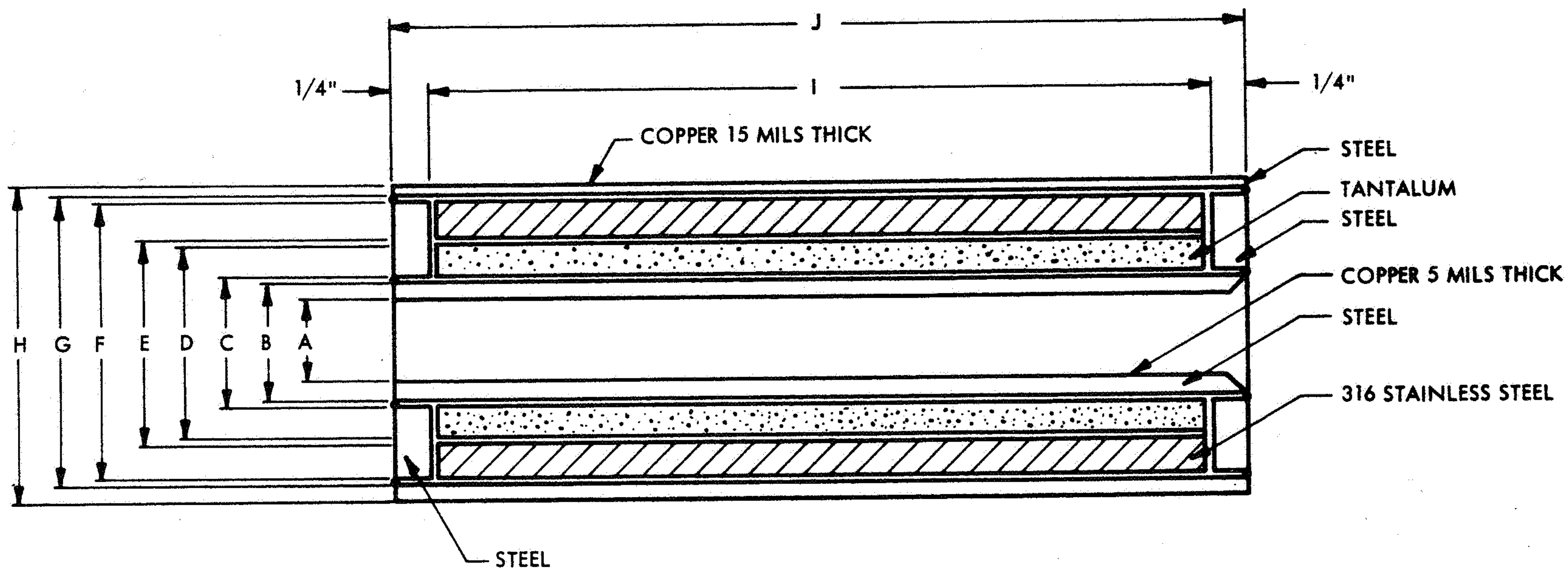
316 Pipe, 3" Sch 80 (3-1/2" OD x 0.300" Wall) Heat No. 2P1300 (USS)			
Analysis - weight percent			
C	0.065	Ni	13.77
Mn	1.62	Cr	16.97
S	0.019	Mo	2.21
Si	0.58		

TABLE 2 [5]

EXTRUSION TEMPERATURE FOR METALONICS TUBING

Indicated Furnace Temperature*	Tube Number
1850°F	2, X-12
1900°F	M-4, M-5, M-7, M-9, X-13
1920°F	M-15, M-16, M-17, M-18, M-19, M-20, M-21, M-22, M-6, M-10
1980°F	M-3, M-11, M-12, M-13

* Temperatures listed are furnace temperatures and are 30-50°F higher than the actual billet temperature as determined by calibrated thermocouple readings.



DIMENSIONS (INCHES)									
A	B	C	D	E	F	G	H	I	J
.657	1.063	1.073	1.662	1.670	2.750	2.760	2.970	8.00	8.50

612292-1B

Figure 2. Extrusion Billet Design Used by Nuclear Metals

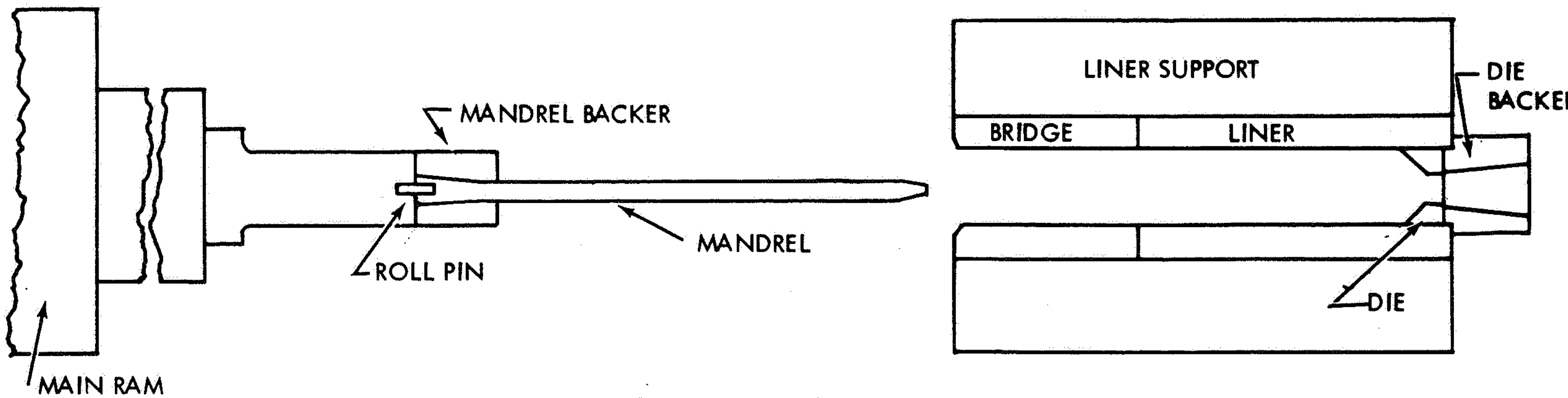


Figure 3. Nuclear Metals Extrusion Setup

TABLE 3

CHEMICAL ANALYSIS FOR NUCLEAR METALS STARTING MATERIAL (VENDOR CERTIFICATIONS)

Tantalum, Lot 7106 (NRC)			
Analysis - ppm			
C	22	Cu	<1
O_2	28	Fe	11
N_2	26	Mo	<10
H_2	2	Nb	63
Al	<10	Ni	<1
Cr	<1	Si	<10
Ti	<5	W	170
316 Stainless Bar Heat No. 16240 (ARMCO)			
Analysis - weight percent			
C	0.055	Cr	16.96
Mn	1.60	Ni	13.24
P	0.029	Mo	2.74
S	0.021	Cu	0.31
Si	0.46	Co	0.19

under a nitrogen atmosphere. Each billet was soaked at 1825°F for 3-1/2 hours prior to extrusion over a fixed mandrel. Extrusion conditions are outlined in Table 4. After extrusion, the cladding was leached off with nitric acid and aqua regia. Stretcher straightening, chemical polishing of the tantalum, and centerless grinding of the stainless OD completed the fabrication process.

TABLE 4

EXTRUSION DATA FOR NUCLEAR METALS TUBING

Tube No.	Extrusion Speed (ipm)	Extrusion Forces (Tons)	
		Upset	Run
14	400	600	560
20	550	550	675
21	600	675	600
22	500	725	650
23	500	700	650
24	500	700	650
25	500	675	650
26	500	725	675
27	500	700	650
28	500	700	650
29	500	700	650
30	500	700	650
31	500	725	650

Note - Other Extrusion Conditions
Extrusion Reduction - 25/1
Lubrication

Billet - sprayed dry film graphite
Liner - Necrolene
Die - Necrolene
Mandrel - Light coat of Lube-a-Tube

Liner ID - 3.050"
Mandrel Diameter - 0.630"
Die Size - 0. 857"

3.0 TEST PROGRAM

Each lot of tubing is to be non-destructively and destructively inspected to completely characterize the degree of bonding, dimensional control, and the size and location of defects. The tubing is to be inspected per the schedule outlined in Figure 4.

Selected lengths of tubing will be subjected to extensive destructive inspection and testing. This is necessary to evaluate ID surface condition, cladding thickness, and bond strength. Bond integrity will be evaluated by thermal cycling. This will consist of heating the specimen to 1350°F, holding for one hour at temperature, cooling to 600°F in 30 seconds and reheating to 1350°F. One hundred thermal cycles constitutes the test. Specimens will be tested in the as-received condition and also after having been thermally treated to produce a 0.2 inch interdiffusion zone between the tantalum and 316SS. Creep-burst at 1350°F and thermal expansion characteristics between room temperature and 1400°F will also be determined.

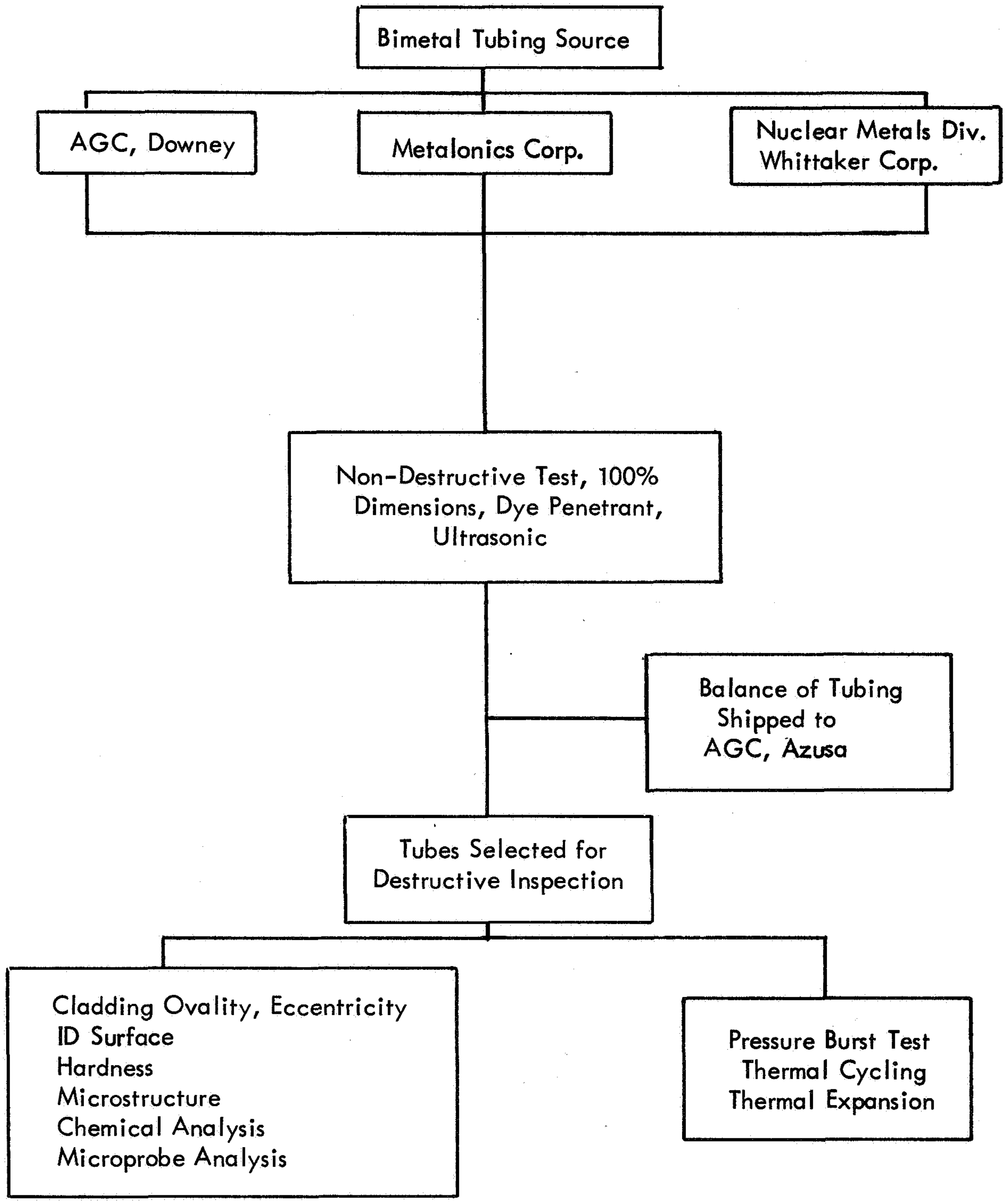


Figure 4. Inspection and Test Schedule for Ta/316SS Bimetal Tubing

4.0 PROGRAM STATUS

The following work has been accomplished during the first six months of this program.

The three lots of tubing have been received for evaluation, and consist of:

1) Aerojet Downey tubing, fabricated by explosive bonding to size. This lot consisted of two tubes, one 43 inches long and the other 96 inches long.

2) Metalonics Corp. tubing, fabricated by co-extrusion to size. This lot consisted of 21 tubes. One of these was 24 inches long; and the remaining 20 pieces were each about 15 feet long.

3) Nuclear Metals Division, Whittaker Corp. tubing, fabricated by extrusion to size. This lot consisted of 14 tubes, one was 24 inches long and the remaining 13 tubes were each about 15 feet long.

The Aerojet Downey tubing was received in July, 1967, the Metalonics tubing in two shipments during July and August, 1967 and the Nuclear Metals tubing in September, 1967.

Dimensional measurements, dye penetrant testing, and ultrasonic testing have been completed.

Upon completion of the testing described above:

1) All the Aerojet Downey tubing was retained for further testing.

2) Metalonics tubes M-9, M-10, and X-13 were retained for further testing and the remainder of the lot was sent to Aerojet General Corporation, Azusa, California.

Destructive inspection of Aerojet Downey and Metalonics tubing to determine ovality and eccentricity of layers, straightness, ID surface condition, and microstructural features has been completed. The destructive inspection of Nuclear Metals tubing has been initiated. Testing for hardness variation across the wall, interstitial content and microprobe analysis for all three lots of tubing has been initiated. On completion of testing, the sound Nuclear Metals tubing will also be sent to Aerojet Azusa.

Pressure testing and thermal cycle testing of Aerojet Downey and Metalonics tube specimens has been initiated.

The apparatus and equipment necessary for thermal expansion testing have been assembled and qualified.

5.0 EXPERIMENTAL TECHNIQUES AND RESULTS

5.1 DIMENSIONAL MEASUREMENTS

Upon receipt of the tubing, detailed measurements were made of the tubing dimensions. Measurement of the inside diameter of the tubes was accomplished by use of a diagauge with an air operated readout. The apparatus was attached to a long boom which permitted measurement along the entire length of tubing. The Metalonics tubing in the as-received condition was very badly bent and kinked. A typical runout value was 3/16 in/ft. This prevented accurate measurement of the inside diameters because the boom could not fit through the entire length of tubing. In order to complete dimensional measurement as well as ultrasonic testing the tubing was roll straightened. In order to insure that the straightening operation did not introduce any defects into the tubing, two pieces were first straightened and evaluated. Prior to straightening, the tubes were ultrasonically inspected on the ends to determine the integrity of the bond at the bimetal interface. They were also dye penetrant inspected and the size and position of each defect found was recorded. After straightening the tubes were then re-ultrasonic and dye penetrant tested. No additional unbonded areas were found nor were the OD defects aggravated. Since roll straightening did not have any deleterious effect, the remaining tubes were then straightened.

The results of the dimensional measurements are shown in Table 5. All three lots of tubing exhibited good dimensional control.

5.2 DYE PENETRANT TESTING

Dye penetrant inspection test results, for the Metalonics tubing, are summarized in Table 6. The Nuclear Metals tubing and the Aerojet tubing were essentially free of OD dye penetrant indications.

TABLE 5
DIMENSIONAL TEST RESULTS

Metalonics Tubing

Tube No.	ID ave	σ ID	ID max/ave	ID min/ave	OD ave	σ OD	OD max/ave	OD min/ave	As-Received Length
	ID Measurements				OD Measurements				
M-3	.6552	.0004	.6566	.6537	.8188	.0003	.8197	.8180	15' - 9-1/2"[(a)]
M-4	.6526	.0002	.6532	.6508	.8177	.0004	.8193	.8162	14' - 10-1/2"
M-5	.6517	.0004	.6535	.6499	.8131	.0003	.8137	.8125	15' - 10-3/4"
M-6	.6533	.0004	.6552	.6514	.8194	.0002	.8200	.8187	15' - 9"
M-7	.6517	.0003	.6534	.6501	.8145	.0002	.8151	.8138	15' - 8-1/2"
M-9	.6524	.0006	.6545	.6503	.8158	.0009	.8168	.8147	15' - 5"
M-10	.6516	.0003	.6531	.6501	.8187	.0001	.8190	.8183	15' - 9-3/4"
M-11	.6504	.0003	.6516	.6492	.8156	.0001	.8164	.8147	15' - 10-1/4"
M-12	.6548	.0005	.6568	.6528	.8196	.0004	.8209	.8184	15' - 5"
M-13	.6516	.0003	.6529	.6502	.8170	.0002	.8177	.8162	15' - 10-1/4"[(b)]
M-15	.6531	.0002	.6542	.6520	.8184	.0001	.8186	.8182	15' - 10-1/4"
M-16	.6532	.0004	.6553	.6512	.8187	.0003	.8189	.8183	15' - 10-1/4"
M-17	.6526	.0003	.6542	.6511	.8191	.0004	.8197	.8186	15' - 5"
M-18	.6526	.0004	.6542	.6511	.8203	.0003	.8208	.8199	15' - 10-1/4"
M-19	.6530	.0007	.6550	.6509	.8184	.0002	.8189	.8178	15' - 10-1/4"
M-20	.6525	.0003	.6532	.6517	.8195	.0003	.8197	.8192	15' - 10-1/4"
M-21	.6535	.0006	.6564	.6506	.8211	.0002	.8220	.8201	15' - 10-1/4"
M-22	.6568	.0010	.6546	.6489	.8193	.0003	.8203	.8182	15' - 5-1/2"
X-12	.6530	.0010	.6556	.6504	.8143	.0010	.8145	.8140	16'
X-13	.6555	.0011	.6578	.6533	.7965	.0007	.7972	.7959	2'

(a) 11" cut out to remove unbond and OD crack
(b) 6-1/2" cut for dye penetrant check

Aerojet Downey Tubing

Tube No.	ID ave	σ ID	ID max/ave	ID min/ave	OD ave	σ OD	OD max/ave	OD min/ave	
	ID Measurements				OD Measurements				
96"	.6611	.0007	.6619	.6603	.8687	.0012	.8712	.8699	

Nuclear Metals Tubing

Tube No.	ID ave	σ ID	ID max/ave	ID min/ave	OD ave	σ OD	OD max/ave	OD min/ave	As-Received Length
	ID Measurements				OD Measurements				
14	.6447	.00029	.6461	.6433	.8001	.00039	.8028	.7995	16' - 4-3/4"
20	.6443	.00039	.6456	.6429	.8192	.00041	.8202	.8181	14' - 1-1/2"
21	.6424	.00048	.6435	.6412	.8164	.00075	.8177	.8180	14' - 4-1/2"
22	.6454	.00034	.6464	.6443	.8219	.00032	.8230	.8207	14' - 6-3/4"
23	.6449	.00029	.6458	.6440	.8168	.00042	.8177	.8159	14' - 7-3/4"
24	.6435	.00081	.6444	.6425	.8146	.00050	.8158	.8133	14' - 4-1/4"
25	.6428	.00041	.6441	.6414	.8161	.00043	.8170	.8151	14' - 9"
26	.6451	.00035	.6459	.6442	.8209	.00049	.8218	.8199	14' - 1-1/2"
27	.6472	.00051	.6484	.6459	.8231	.00076	.8248	.8214	14' - 5"[(a)]
28	.6440	.00028	.6450	.6430	.8124	.00053	.8155	.8128	14' - 7-1/2"
29	.6445	.00031	.6453	.6437	.8180	.00039	.8189	.8171	14' - 7-1/2"
30	.6452	.00035	.6464	.6440	.8204	.00051	.8223	.8184	14' - 4-1/2"
31	.6454	.00023	.6460	.6448	.8141	.00048	.8150	.8132	14' - 2-3/8"

(a) 6" cropped for dye penetrant check

TABLE 6

DYE PENETRANT RESULTS

Tube No.	Metalonics Tubing — Dye Penetrant: Defects[a]	Metalonics Tubing — Dye Penetrant: Comments
M-3	32" longitudinal	all heavy indications
M-4	55" longitudinal 23" transverse	most are light indications
M-5	16" longitudinal	all heavy indications
M-6	11" longitudinal	all light indications
M-7	20" longitudinal	all light indications
M-9	41" longitudinal	some light some heavy indications
M-10	3" longitudinal	all light indications
M-11	32" longitudinal	all heavy indications
M-12	16" longitudinal 41" transverse	all light indications
M-13	40" longitudinal	most are light indications
M-15	23" longitudinal	all heavy indications
M-16	29" longitudinal	most are light indications
M-17	25" longitudinal	most are light indications
M-18	49" longitudinal	most are light indications
M-19	59" longitudinal	most are light indications
M-20	70" longitudinal	most are light indications
M-21	59" longitudinal	most are light indications
M-22	22" longitudinal	most are light indications
X-12	29" longitudinal	some are light; few heavy
X-13	7" longitudinal	all light indications
2	3" longitudinal	all light indications

(a) Numbers denote linear inches of longitudinal defects and transverse defects.

5.3 ULTRASONIC TESTING

Ultrasonic testing was used to determine the bond integrity between the austenitic and refractory metal components. In order to help determine the most sensitive procedure to be used for each lot of tubing and also the test limitations, a metallographic investigation was performed on each lot of tubing.

5.3.1 Aerojet Downey Tubing

The salient features of this tubing which are illustrated in Figure 5 consist of a smooth interface disturbed by the presence of dimples which cause out of roundness.

5.3.2 Metalonics Tubing

The tubing produced by Metalonics was characterized by:

a) a rough interface

b) an ID surface roughened bylongitudinal striations about 2-3 mils deep and occasional cracks of varying depth

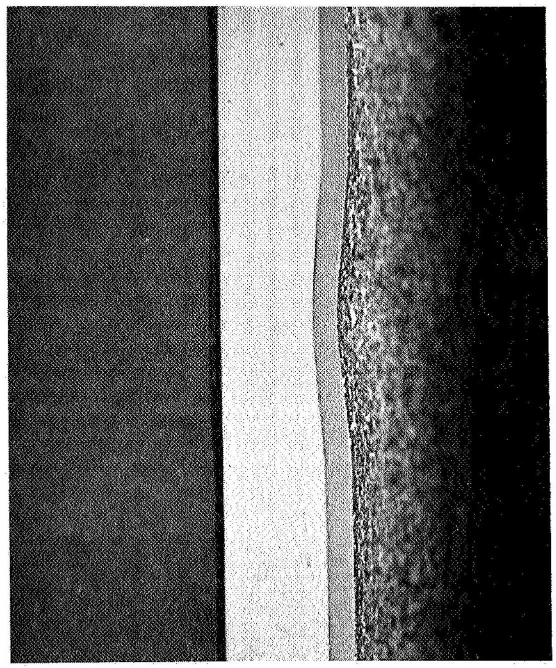
c) a smooth OD surface containing several cracks of varying depth.

These features are shown in Figure 6.

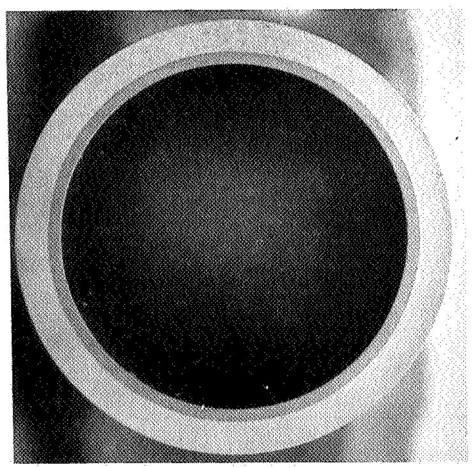
5.3.3 Nuclear Metals

The metallographic examination of the Nuclear Metals tubing has not been completed but preliminary results indicate generally a smooth interface and smooth ID and OD similar to that exhibited by the explosively bonded to size tubing.

The differences between the surface condition of the lots of tubing required the use of two ultrasonic test procedures in order to inspect for debonding at the bimetal interface. A "pulse-echo" technique satisfactorily deliniated debonded areas in the exploded to size tubing. However, the rough ID surface of the Metalonics extruded to size tubing required a "pulse-echo-thickness measurement" technique in order to detect unbounded areas at the bi-metal interface. A schematic illustration of both procedures is shown in Figure 7.

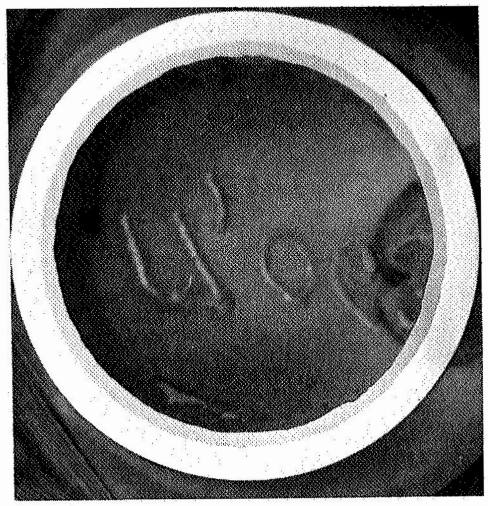


(a) Dimpled Area in As-Received Aerojet Downey Tubing (100 X)



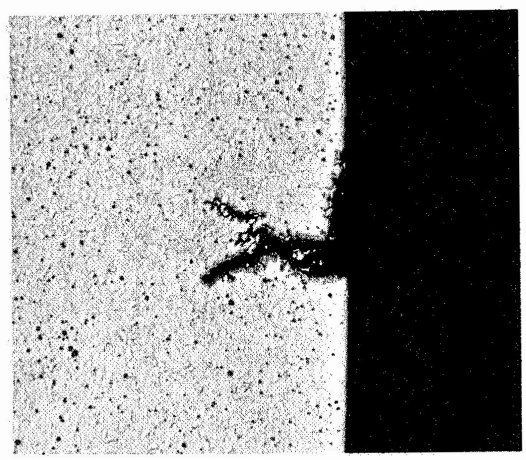
(b) Transverse Section, Away from Dimple (3 X)

Figure 5. Aerojet Downey Bimetal Tubing



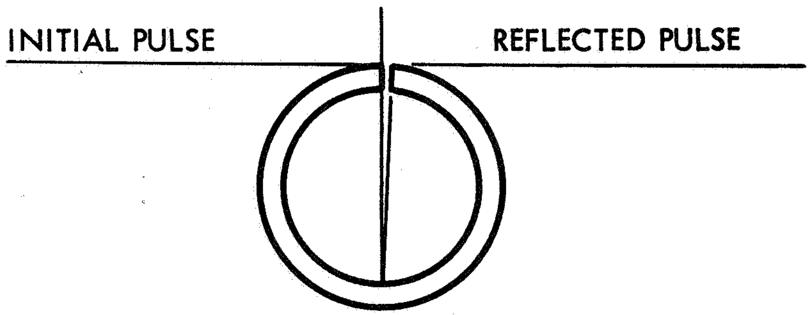
(a) Transverse Section 3X

(b) ID Crack, ~2 Mils Deep 100X

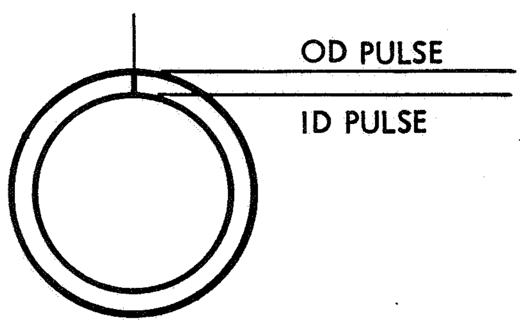


(c) OD Crack ~7-1/2 Mils Deep 100X

Figure 6. As-Received Metalonics Ta/316SS Bimetal Tubing



(a) SCHEMATIC OF PULSE ECHO TECHNIQUE USED TO ULTRASONICALLY INSPECT AEROJET DOWNEY TUBING



(b) SCHEMATIC OF PULSE ECHO THICKNESS MEASUREMENT TECHNIQUE USED TO ULTRASONICALLY INSPECT METALONICS TUBING

612292-4B

Figure 7. Schematic Illustration Showing Pulser and Receiver Signals

In order to inspect the fifteen foot lengths of tubing in a semi-automatic fashion, certain special pieces of equipment were designed and fabricated. Since focused transducers were to be used in the inspection, immersion of the tubes in a fluid was required. Both the tubing transport and immersion requirements were met by the use of a specially designed "stuffing box".

The tubes were fed into long slightly bowed booms, through seals and into a water filled box. Here a rotating device moved the tubing past the test crystal in a helical fashion. The pitch of the helix and longitudinal speed through the box can be varied through a fairly wide range of values. The longitudinal speed can be varied from 0 ipm to 4 ipm and the pitch from $0''/360^{\circ}$ to $1/2''/360^{\circ}$. A spring loaded teflon vee block was used to maintain alignment between the crystal transducer and the tubing as well as a fixed distance between the crystal and the tube.

The recording device used for both test techniques was a light operated oscilloscope, a high resolution galvanometer, and a strip chart recorder. The ultrasonic test setup is shown in Figure 8. It was also assumed that any surface defects which occurred during extrusion and not completely removed during subsequent conditioning operations would interfere, thus a high resolution short pulser was deemed necessary.

A Sperry Products Style 57A3621 crystal was used to inspect the Aeroject Downey tubing and a Sperry Products Style SIL 57A2753 crystal and a Sperry Products Style 50E568 thickness read out module were used to inspect the co-extruded Metalonics tubing. Since no ultrasonic test standards were available it was necessary to manufacture a specimen from each type of tubing containing an unbond. This was necessary for two reasons: first to insure the differentiation between metallurgical bonding and intimate mechanical bonds; and second to fix the intensity of the pulses generated and the nature of the gating for the ID pulse. This gating was necessary to aid in distinguishing between varying degrees of bonding. The diameter of the test crystal was about 1/8 inch and so at any instant an area 1/8 inch in diameter of tubing was under inspection. Unbonded areas 1/8 inch or larger can be positively

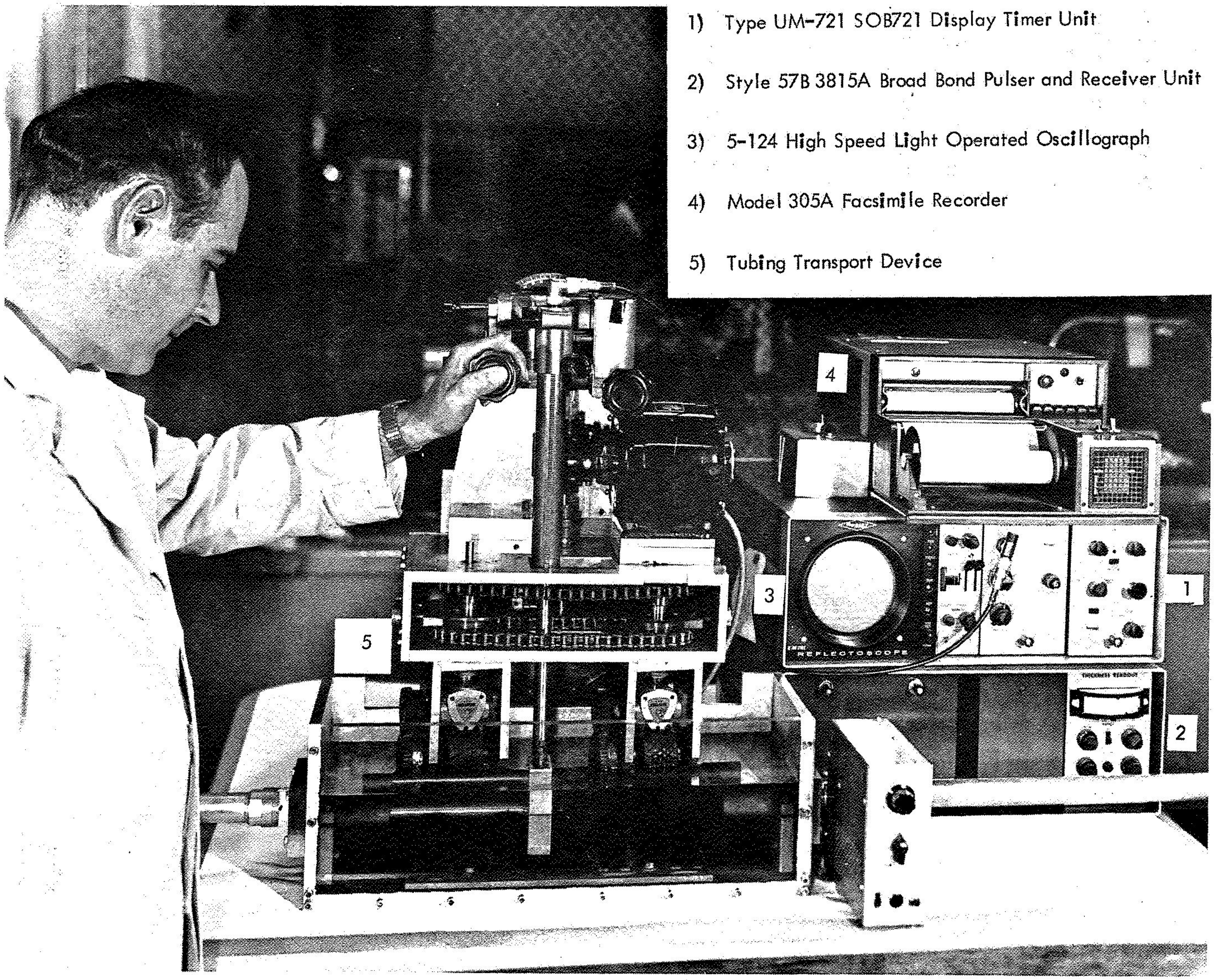


Figure 8. Ultrasonic Test Apparatus

identified because the "echo pulse" will be completely attenuated. Unbonded areas smaller than 1/8 inch in diameter are examined by means of the "gating system". Recalling that, at any instant an area 1/8 inch in diameter is under inspection, if only part of this area is unbonded then only part of the echo pulse is attenuated. The gating system serves to completely attenuate the echo pulse if its intensity falls below a given value, thus giving an unbond signal in the recording apparatus. It is important to note that despite the use of gating, the determination of unbonded areas smaller than 1/8 inch in diameter where "partial bonding exists" is quite difficult and may be considered as the limit in resolution of the test technique.

However, previous experience at WANL has shown that debonding at the interfaces in drawn refractory/Austenitic bimetal tubing occurs generally over a continuous longitudinal length rather than as isolated discontinuous defects. The exception, of course, would be the "dimples" in exploded to size tubing.

The standards were manufactured by the following techniques:

1) Aerojet Downey - an interdiffusion zone approximately 0.2 mils thick was grown in a specimen 6 inches long. Thermal exposure was at 1650°F for 300 hours. The specimen was then cycled 20 times between 1350 and 600°F. Cooling takes place in about 30 seconds. This debonded the tubing in both the dimpled and undimpled portions. In addition to this manufactured standard, the 96 inch long piece had previously been ultrasonically tested by Automation Industries and a copy of the trace generated was sent to WANL. This trace showed the existence of an unbonded area which was corroborated by the WANL results.

2) Metalonics - several attempts to unbond this tubing by thermal cycling testing specimens with 0.2 inch thick interdiffusion zones were unsuccessful.

It is interesting to note the severity of testing which the sample withstood. The attempts to unbond were:

a) Thermal cycle testing the 6 inch piece in the as-received condition.

b) Growing a 0.6 mil diffusion zone and quenching from 1900°F in water.

c) Heating localized areas to 2000°F by use of an acetylene torch while protecting the ID with helium then quenching in water.

d) Heating the entire tube to 2000°F by use of an acetylene torch while protecting the ID with helium and then quenching the ID with water.

A standard was successfully prepared by the following procedure:

a) A piece 1/2 inch long was cut from a 6 inch bimetallic tube.

b) The stainless layer was etched from the 1/2 inch piece.

c) A 1/2 inch long cylinder of Ta was machined out from the remaining 5-1/2 inch piece.

d) The 1/2 inch Ta cylinder resulting from steps a) and b) was then shrink fitted into the cavity in the 5-1/2 inch piece left by the machined out Ta.

The resulting specimen had a 1/2 inch long unbonded portion and a 5-1/2 inch long bonded portion. The unbonded portion meets the requirement of intimate mechanical but not metallurgical bonding.

An interesting complication due to the OD defects became evident during the testing of this lot of tubing. The OD defects generated pulses somewhat similar in intensity and position to those produced by an unbonded area. Careful examination of both the tubing and the pulses displayed on the oscilloscope however permitted differentiation. The pulses displayed on the strip chart however did not allow this differentiation. The results of the ultrasonic testing are summarized in Table 7.

The Aerojet Downey lot was tested by use of the pulse echo procedure. This procedure was used because of the smooth nature of the ID and OD surfaces. All of the dimples, both in the 24 inch and 96 inch tubes were identified. In addition, an unbonded area was found in the 96 inch piece whose size and configuration matched that found by Automation Industries.

The Metalonics lot was tested by the "pulse echo thickness measurement" procedure. This procedure was used because of the nature of the ID surface. Unbonded areas 3/4 inch and 1/4 inch were found in two of the 21 tubes tested. These two tubes were identified as M-3 and M-10. The unbond in tube M-3 was near an end and so it was removed and the tube salvaged. This was impossible in tube M-10 because the unbond was two feet from one end. The defect areas were sectioned and metallographically identified in both tubes. Figure 9 shows metallographical confirmation of the existence of the unbonding in tube M-10.

TABLE 7

ULTRASONIC TEST RESULTS

Metallonics Tubing

Tube No.	Comments
M-3	Unbond at end 1/4" x 3/4", cut out
M-10	Unbond at 2 ft. from end 1/4" x 3/4"
X-13	Wall thickness fluctuations

All the other Metalonics tubes were fully bonded.

Aerojet Tubing

Tube No.	Comments
96"	Unbond formed at one end
43"	OK

NOTE: All dimples in the exploded tubing were found.

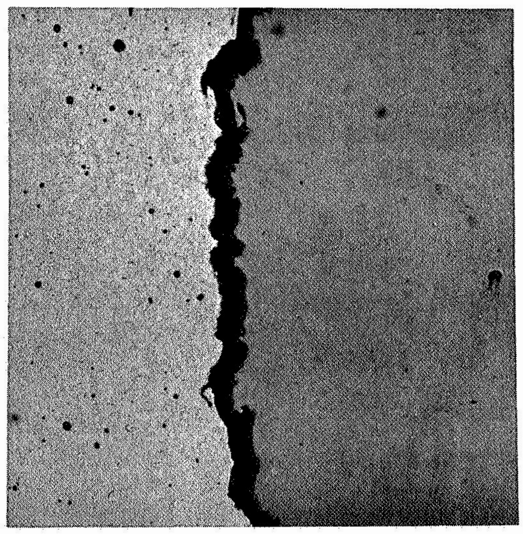


Figure 9. Unbonded Area In Metalonics Tubing Identified By Ultrasonic Testing 400X

Generally, the co-extruded tubing exhibited excellent metallurgical bonding between the 316 and tantalum as discriminated by the ultrasonic test. The explosively bonded to size tubing has the disadvantage of having unavailable unbond areas which are present because of the nature of the process.

5.4 METALLOGRAPHY

In addition to the preliminary metallography performed to help determine the appropriate ultrasonic test techniques for each lot of tubing, extensive metallographic examinations have been carried out on Aerojet Downey and Metalonics tubing in order to define the nature of the interface and determine the uniformity of the layers. Some preliminary work has also been completed on Nuclear Metals tubing.

5.4.1 Aerojet Downey

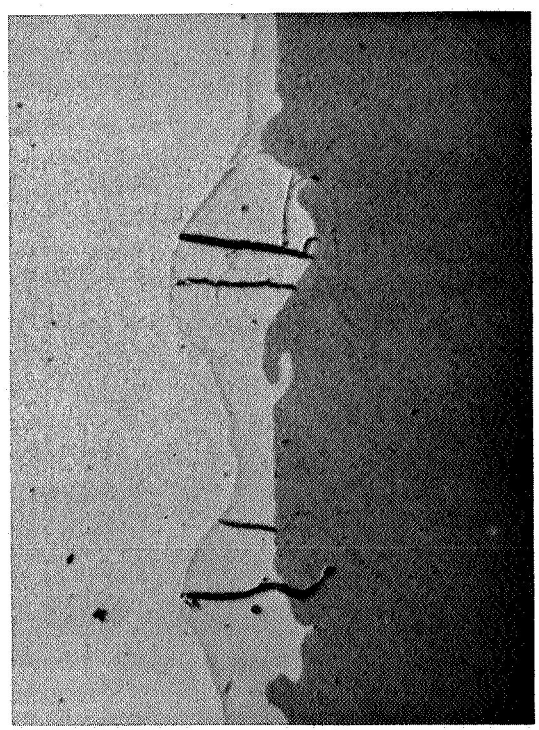
Transverse sections were taken every 12 inches along the length of the 96 inch length of explosively bonded tubing and examined metallographically. As shown in Figure 10, an intermetallic layer was formed at the interface during bonding. The layer appears to be continuous and microprobe analysis will be used to identify its composition. Transverse cracks were also observed in this intermetallic layer which in some instances propagated into the tantalum liner. The intermetallic layer was observed on 75 percent of the samples examined and was formed almost continuously around the diameter. Longitudinal sectioning (see Figure 5a) showed the thickness of each of the components to be uniform except in the area where a stand off dimple was made. Here the dimple protuberance is reflected in both layers by a kink.

5.4.2 Metalonics Tubing

As previously discussed the interface of this lot of tubing is extremely rough. Figure 11 shows a typical longitudinal section which illustrates the thickness of the individual bimetal layers to be quite uniform.

Extensive sectioning of the type of tubing seems to indicate that the majority of the cracks identified by dye penetrant testing of the OD are about 5 mils deep and also that the majority of ID cracks are about 2-3 mils deep. (See Figure 6)

316 SS



Ta

Figure 10. Typical Interface in As-Explosively Bonded to Size Tubing

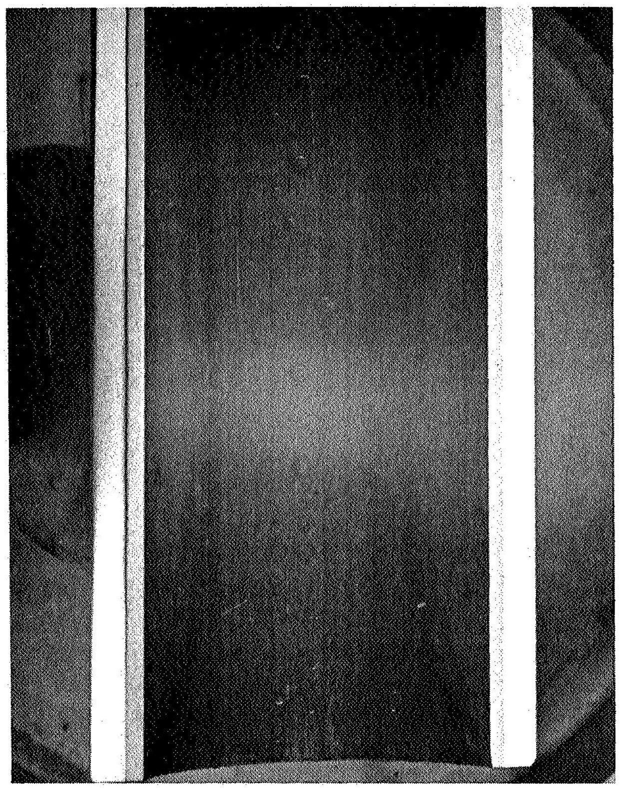


Figure 11. Longitudinal Section of Metalonics Tubing

5.4.3 Nuclear Metals Tubing

The limited amount of metallography done thus far on the Nuclear Metals tubing has shown that; there is no evidence of OD or ID cracks, the ID and OD surfaces are smooth, the thickness of each layer is quite uniform, and there is no evidence of unbonding.

5.5 THERMAL EXPANSION MEASUREMENTS

All the apparatus and equipment necessary for testing have been assembled and qualified. The apparatus used to perform the measurements is shown in Figure 12. Since the thermal expansion is measured over a range of from room temperature to 1400°F the apparatus is enclosed in a vacuum chamber to prevent atmospheric contamination of the refractory metal. The diffusion pumped metal bell jar operates normally at 10^{-6} torr.

The unit utilizes a modified form of the standard quartz pushrod and tube technique. The quartz pushrod is formed into a ring to surround and contact the tubular specimen opposite the pedestal. The change in specimen diameter is thus transferred directly to pushrod motion. Pushrod motion is measured and recorded by an electro-mechanical transducer.

A vertical quartz tube with a flat polished upper end and a slit forms the pedestal. Figure 12a shows the apparatus with a dimetal specimen, Figure 12b shows schematically the pushrod, tabs and transducer measuring a longitudinal specimen. Due to the fact that the tubing is not perfectly round, longitudinal measurements are a better characteristic of the thermal expansion behavior.

5.6 CREEP BURST TESTING

The apparatus used for creep-burst testing of the tubing is shown schematically in Figure 13. A detailed description of the equipment and operation can be found elsewhere [3]. Basically, the specimen, which is contained in an evacuated cylinder, is pressurized with high purity helium gas and heated to the test temperature by using a hot wall furnace. A bimetal specimen was prepared and tested at 1350°F and 6300 psi. The test was terminated because a leak developed at the pressurizing gas entrance fixture. The primary purpose of this test was checkout of the pressurization system which performed perfectly.

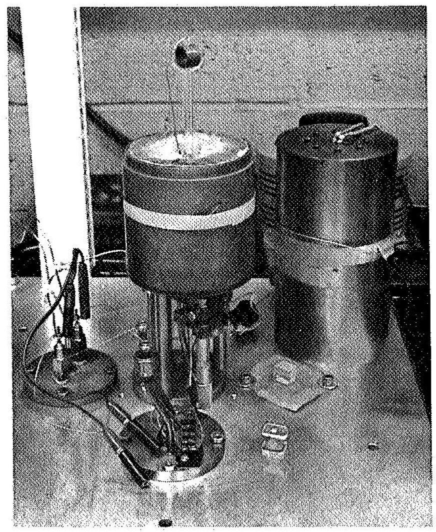


Figure 12a. Thermal Expansion Apparatus

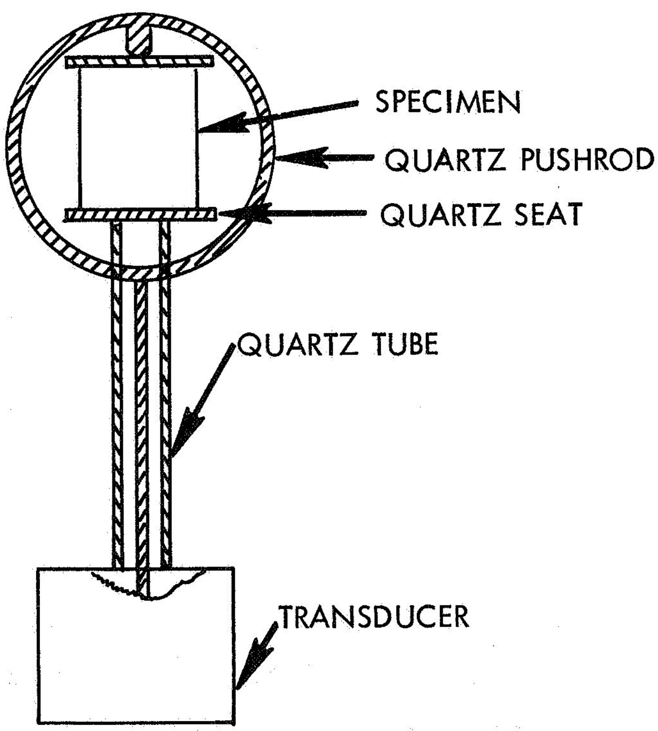


Figure 12b. Thermal Expansion Schematic

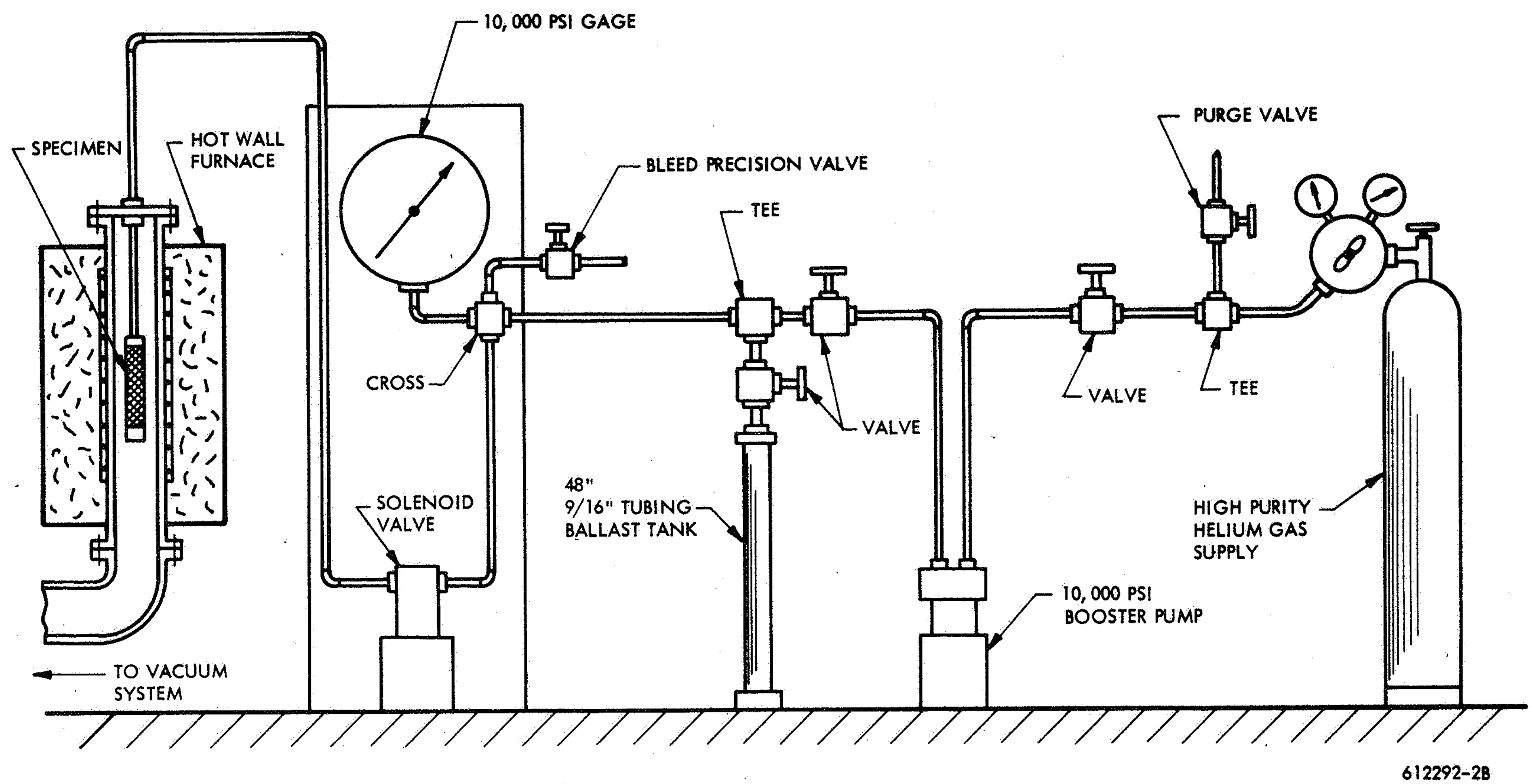


Figure 13. 10,000 psi Pressure Test Rig Schematic

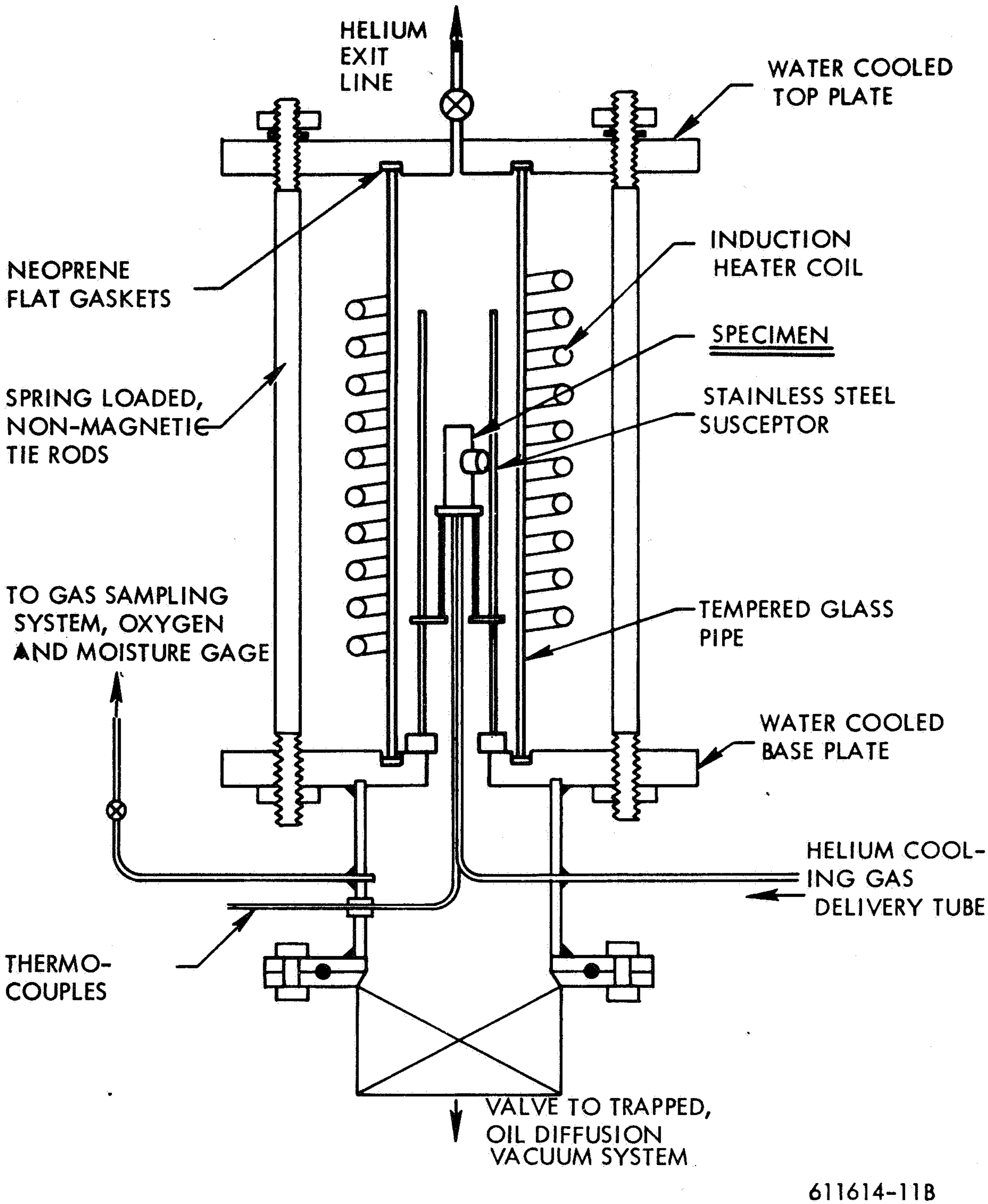


Figure 14. Schematic of Thermal Cycle Test Apparatus

5.7 THERMAL CYCLE TESTING

The apparatus used for thermal cycle testing is shown schematically in Figure 14 and a detailed description is given in Reference 3. Initially, the thermal cycle was to consist of heating the specimen to 1350°F, holding for five minutes and cooling to 600°F within thirty seconds. This cycle was to be repeated a total of twenty times. The requirements have been changed in that the holding time at 1350°F has been increased to sixty minutes for each cycle, and the total number of cycles has been increased to one hundred. The equipment is currently being adapted to automatic operation.

6.0 FUTURE WORK

The following work will be done between now and the completion of the contract.

1) Chemical analysis and comparison to the chemical analysis of the starting material.
2) Microprobe analysis
3) Pressure testing - selection of specimens, end cap design, load selection for long time tests, long time tests, and post test analysis.
4) Thermal cycle testing - selection of specimens and post test analysis.
5) Thermal expansion measurements - specimen selection, and testing.

7.0 REFERENCES

1. Slone, Henry O., SNAP-8 Development Status, Space Power Systems Advanced Technology Conference, Lewis Research Center, Cleveland, Ohio, pp 147.

2. Buckman, R. W. Jr. and Goodspeed, R. C., "Evaluation of Refractory/Austenitic Bimetal Combination", Final Report under Contract NAS 6-7634, Astronuclear Laboratory.

3. Stoner, D. R., "Joining Refractory/Austenitic Bimetal Tubing", Final Report under Contract NAS 3-7621, Westinghouse Astronuclear Laboratory.

4. Gardner, Norman R., "Fabrication History for the Production of NASA Stainless Clad Ta Tubing, Metalonics Div. Kawecki Chemical Company, Prepared under Contract NAS 3-9436.

5. Jenkins, Robert S., Process Development and Pilot Production of Tantalum Lined 316 Stainless Steel Tubing, Nuclear Metals Div., Whittaker Corp., Prepared under Contract NAS 3-9435.