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MATERIALS TECHNOLOGY PROGRAMS IN SUPPORT OF A MERCURY RANKINE SPACE POWER SYSTEM

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16. Abstract

This report summarizes a large portion of the materials technology that was generated in support of the development of a mercury-rankine space power system (SNAP-8). The primary areas of investigation reported are (1) the compatibility of various construction materials with the liquid metals mercury and NaK, (2) the mechanical properties of unalloyed tantalum, and (3) the development of refractory metal/austenitic stainless steel tubing and transition joints. The primary results, conclusions, and state of technology at the completion of this effort for each of these areas are summarized in this report. Results of possible significance to other applications are highlighted.

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SUMMARY

During the development of the SNAP-8 mercury-rankine space power system, a significant amount of support materials technology was performed at the NASA-Lewis Research Center and at other laboratories under NASA contract. This technology was primarily concerned with (1) the compatibility of some conventional alloys and refractory metals with two liquid metals, mercury (Hg) and a sodium-potassium eutectic alloy (NaK), (2) the determination of the mechanical properties of unalloyed tantalum (Ta), and (3) the fabrication and evaluation of refractory metal/austenitic alloy (primarily Ta/316 stainless steel (316 SS)) bimetallic tubing and transition joints.

As a result of this materials support work, the following conclusions were made: (1) The refractory metals Ta, niobium-1-percent zirconium (Nb-1Zr), and the Ta alloy T-111 should not be attacked by liquid Hg at temperatures up to 650° C (1200° F). (2) The NaK corrosion rates of the major SNAP-8 primary reactor loop materials, Hastelloy N and 316 SS, appear to be acceptably low (<0.004 cm/10^4 hr or < 0.0015 in./10^4 hr) at temperatures up to 700° C (1300° F), providing that the oxygen level in the NaK is maintained at less than 30 ppm. (3) Tantalum having a uniformly distributed oxygen concentration of 115 ppm or less should not be attacked by purified NaK at temperatures up to 730° C (1350° F). (4) Recrystallized fine-grained Ta tubing is more creep resistant under uniaxial loading than large-grained material at 730° C (1350° F). (5) Recrystallized Ta bar has a low-cycle fatigue life in excess of 1000 cycles at a plastic strain range of 0.02 centimeter per centimeter (in./in.) at temperatures up to 730° C (1350° F). (6) Welded butt joints, tee joints, and tube-to-header joints for refractory metal/austenitic alloy tubing can be successfully produced. (7) The most suitable refractory metal/austenitic alloy bimetallic combination to be used as tubing for Hg containment at temperatures to 730° C (1350° F) is Ta/316 SS. (8) A successful fabrication method for producing Ta/316 SS bimetallic transition joints involves brazing and a tongue-in-groove design.

INTRODUCTION

This report summarizes a large portion of the materials technology that was developed in support of a previously planned nuclear, mercury-rankine space-power generation system termed SNAP-8. (A summary of the SNAP-8 program, since completed, can be found in ref. 1.) Most of this materials technology has been previously reported, but in so many diverse ways and places and under so many different titles, as to make it extremely difficult to develop total context or to reach overall conclusions. It is, therefore, the primary purpose of this report to summarize the major results of these many efforts and to give more comprehensive overall conclusions so that future investigators may have readily available information upon which to build. Although many other materials investigations in support of SNAP-8 were performed by Aerojet General Corporation, NASA's prime contractor for this system (ref. 1), the work described in this report was performed either at the NASA-Lewis Research Center or under other NASA contracts. More detailed discussion of each of the studies covered can be found in the references cited.

The original goal of the SNAP-8 mercury-rankine system was to provide 35 kilowatts of electrical power (kW_e) for space applications for at least 10 000 hours. This goal gradually evolved to a 90 kW_e requirement and a 40 000 hour life. In both cases, thermal energy for the system was provided by a uranium-hydride-fueled nuclear reactor, which was cooled by flowing liquid sodium-potassium eutectic alloy (NaK). The NaK was pumped to a heat exchanger (boiler) where it transmitted its thermal energy to the mercury (Hg) working fluid. (See fig. 1 for a SNAP-8 power conversion system sche-

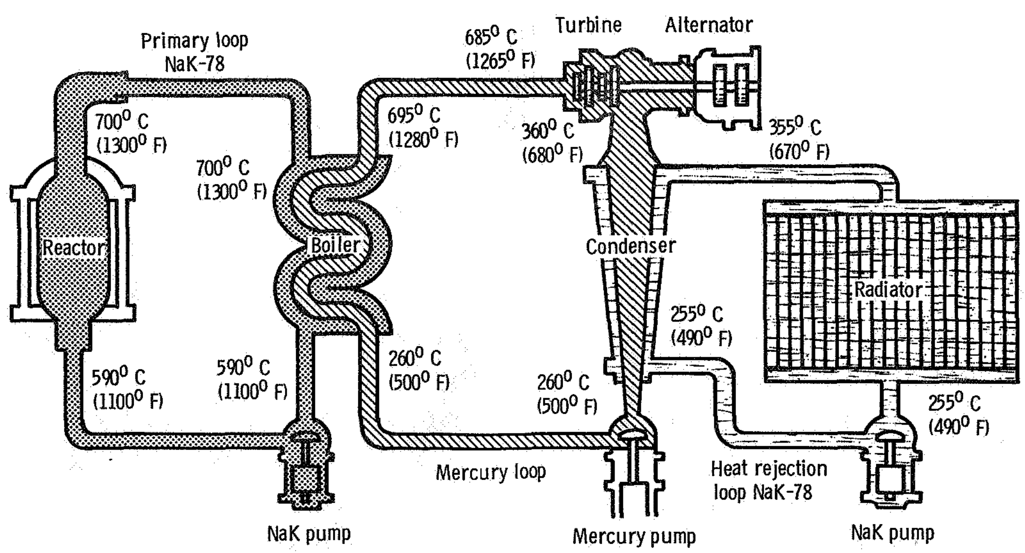


Figure 1. - SNAP-8 power conversion system schematic. CD-11490-17

matic.) The Hg was boiled and superheated, and flowed to a turbine alternator, which generated the electrical power. The Hg was then condensed and pumped back to the boiler. Cycle waste energy was removed from the condenser-heat exchanger to a radiator by means of another pumped-NaK loop for rejection to the space environment.

It was originally assumed that this SNAP-8 system could readily build on existing technology from the similar SNAP-2 system (mercury-rankine, 3 kW_e), and thus no significant effort would be required in the materials development area. But, shortly after the SNAP-8 program began, it was apparent that SNAP-2 technology was generally inadequate and that the materials area would involve a great amount of technology development. The most difficult materials problems were in the realm of Hg containment, particularly in the Hg boiler. The boiler was first constructed of the cobalt-base alloy L-605. When serious Hg corrosion of this alloy was observed, an interim change to 9-percent-chromium - 1-percent-molybdenum steel was made. Finally, it was determined that a refractory metal was required to reliably achieve the desired Hg corrosion resistance and system life. Unalloyed tantalum (Ta) was chosen. It was believed necessary to isolate the Ta from the flowing NaK stream to prevent it from gettering such ductility impairing elements as carbon and nitrogen. To this end, two boiler designs were initiated, both of which are described in more detail later in this report. One design used tantalum/austenitic stainless steel (SS) bimetallic tubing. The other design involved the use of a double-containment configuration in which several unalloyed Ta tubes, each surrounded successively by nonflowing NaK and a stainless steel tube, were further contained in a stainless steel outer boiler shell within which the primary loop NaK flowed. This design employed a Ta/316 SS transition joint at each end of the boiler.

The materials investigations discussed in this report were therefore primarily concerned with three areas (1) the compatibility of some conventional iron, nickel, and cobalt-base alloys and some refractory metals with Hg and NaK, (2) the determination of design-allowable mechanical properties of unalloyed Ta, and (3) the fabrication and evaluation of refractory metal/austenitic alloy bimetallic tubing and transition joints.

LIQUID METAL CORROSION STUDIES

At various times during the conduct of the SNAP-8 program it became necessary to determine the compatibility of certain candidate SNAP-8 construction materials with Hg and NaK. Many of these corrosion studies, concerned with conventional iron, nickel, and cobalt-base alloys and also refractory metals, were conducted either at NASA-Lewis or at other laboratories working under contract to Lewis. The studies included capsule tests, loop tests, and cavitation experiments. The highlights and prime conclusions of these studies are summarized in the following sections.

Mercury Corrosion

Conventional alloys. - Initially, a cobalt base alloy, L-605, was selected as the reference fabrication material for the SNAP-8 system because of its apparent successful use in the SNAP-2 system. In order to determine the Hg corrosion resistance of L-605 at system temperatures, reflux capsule tests and a pumped-loop test were conducted at Lewis (refs. 2 and 3). Several materials other than L-605 were also tested as reflux capsules. They were 9Cr-1Mo steel, the cobalt-base alloy H-8187, and the iron-base alloys AM 350 and AM 355. No nickel-base alloys were tested because of the poor Hg corrosion results previously obtained at TRW, Inc., in support of the SNAP-2 program (refs. 4 and 5).

The reflux capsule tests were conducted over the temperature range 540° to 700° C (1100° to 1300° F) for times up to 5000 hours. The results of the tests indicated gross Hg attack of all materials other than the 9Cr-1Mo steel. Comparative photomicrographs of L-605 and 9Cr-1Mo steel are shown in figure 2. The L-605 developed a deep porous layer, and the 9Cr-1Mo attack consisted of only a shallow surface effect.

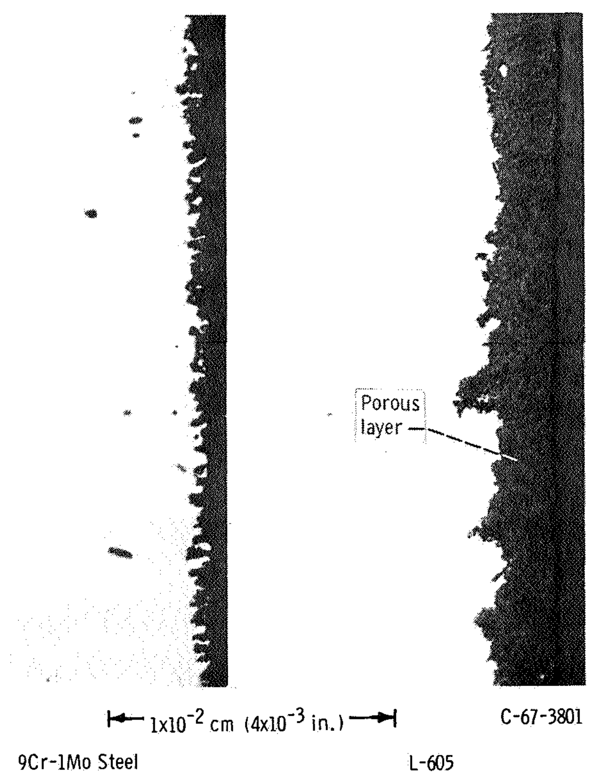


Figure 2. - Typical appearance of mercury-corroded areas of reflux capsules. Photomicrographs of longitudinal section of capsule wall. Capsule inner surface at far right of photomicrograph. Unetched; test temperature, 590° C (1100° F); test period, 1000 hours (ref. 2).

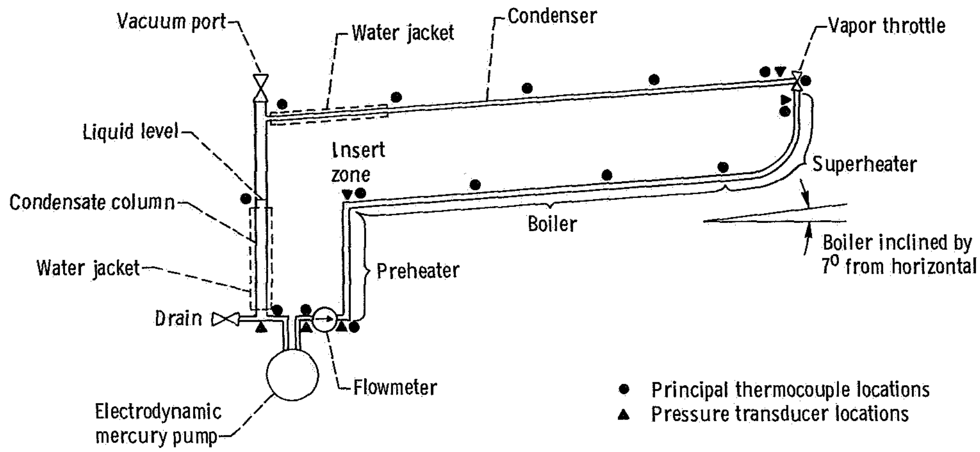


Figure 3. - NASA - Haynes 25 mercury corrosion loop (ref. 3).

The L-605 corrosion loop (fig. 3) was operated for 1147 hours at a peak liquid temperature of 580° C (1075° F) and at an average liquid velocity of 240 centimeters per second (8 ft/sec). The corroded porous layer thickness observed was 0.020 to 0.025 centimeter (0.008 to 0.010 in.). This was approximately 10 times that observed in the reflux capsule tests for approximately the same test time. The increased corrosion was attributed to the effect of the higher Hg liquid velocity in the loop test.

Based on the results of the capsule and loop tests, it was concluded that L-605 was not a good Hg containment material for SNAP-8 use. Thus, it was decided to substitute 9Cr-1Mo steel as the SNAP-8 boiler reference material.

Following the decision to change to 9Cr-1Mo steel as the boiler reference material, a contract was awarded to TRW, Inc., for the construction and operation of a Hg forced circulation loop to further study corrosion mechanisms in 9Cr-1Mo steel and also corrosion product separation techniques (ref. 6). This loop (fig. 4) was operated for 2918 hours at an average boiling temperature of 580° C (1075° F) and at an average liquid velocity of 0.61 centimeter per second (0.02 ft/sec). The corrosion of the 9Cr-1Mo steel was insignificant. But in retrospect, it was believed that the low liquid velocity in the loop, compared with the velocities newly being projected for SNAP-8, rendered the corrosion results to be of little direct applicability to the new system requirements. However, the corrosion product separators in the vapor portion of the system removed about 54 percent of the total corrosion products found in the system, and the separator in the liquid region removed about 25 percent. Based on this success, it was concluded that separators could also be effective in a larger system should the need arise.

Some Hg corrosion loop testing of 9Cr-1Mo steel was also conducted by Aerojet General Corporation under the scope of the SNAP-8 prime contract (ref. 7). This testing established conclusively that Hg corrosion of 9Cr-1Mo steel was velocity dependent. Also, at the velocities required in the boiler for stable Hg boiling, the corrosion rate

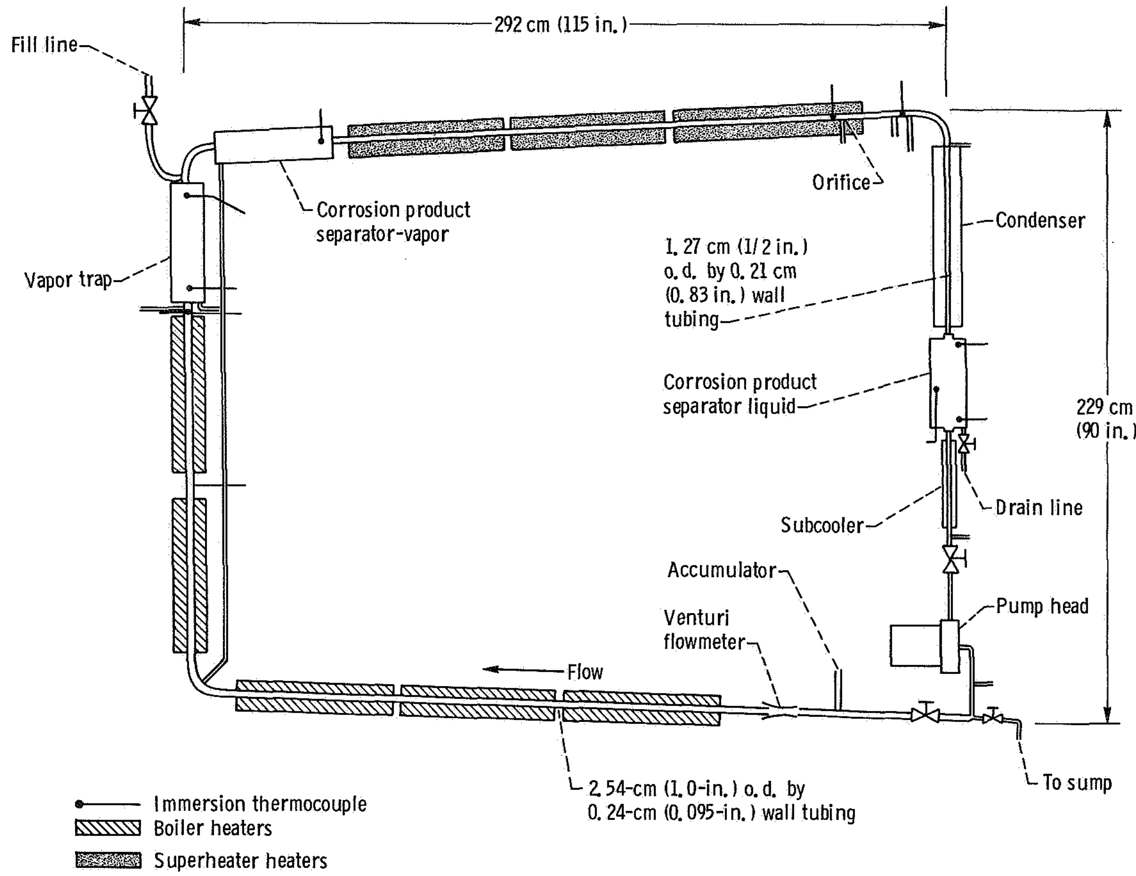


Figure 4. - TRW 9Cr-1Mo steel mercury corrosion loop (ref. 6).

was considered to be unacceptable for the required 10 000-hour system life (i.e., a uniform 0.0025 cm/100 hr or 0.001 in./100 hr).

Although it was not expected to be more Hg corrosion resistant than 9Cr-1Mo steel, a modified 9Cr-1Mo steel was also used for SNAP-8 boiler construction. This material was considerably stronger than the standard alloy as a result of the addition of very small amounts of niobium, vanadium, boron, nitrogren, and zirconium and the use of a 1040° C (1900° F) normalize and 730° C (1350° F) temper heat treatment. The improved strength properties are clearly illustrated in figure 5 in which the modified alloy in the normalized and tempered condition is compared with standard 9Cr-1Mo steel, 304 SS, and 316 SS, all in the annealed condition. The modified alloy indicated no thermally induced instabilities even after 18 000 hours of stress-rupture testing at 650° C (1200° F).

Refractory metals. - As a result of the 9Cr-1Mo steel Hg corrosion testing, this material was replaced by unalloyed Ta as the SNAP-8 boiler reference material. The selection of Ta was based primarily on the minimum solubility of Ta in Hg at temperatures well above the 590° C (1100° F) Hg boiling temperature, as determined at the

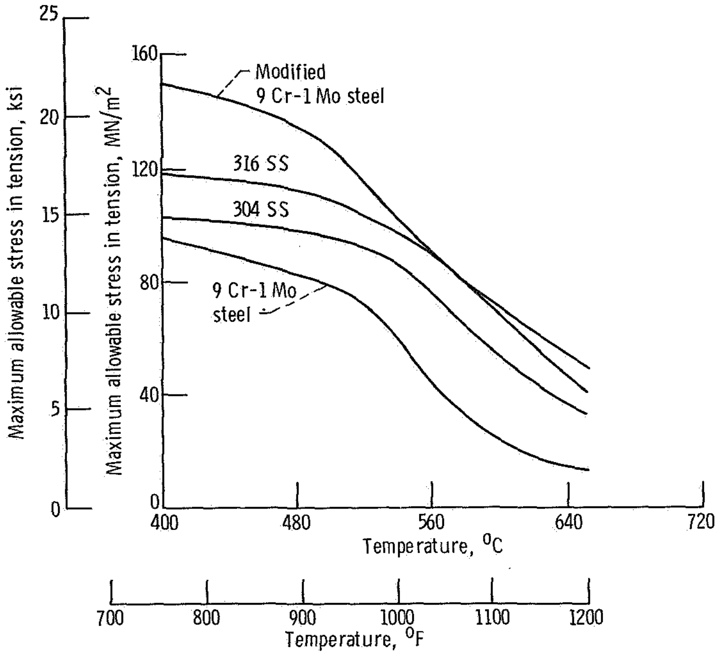


Figure 5. - Strength properties of modified 9 Cr-1 Mo steel and other conventional materials as function of temperature. Note: Allowable stress values are from the ASME boiler and pressure vessel code.

Brookhaven National Laboratory under an AEC sponsored program (ref. 8). The Brookhaven data are shown in figure 6. When Ta was selected as the SNAP-8 boiler reference material, it was recognized that other refractory alloys such as niobium-1-percent zirconium (Nb-1Zr) and T-111 (nominal composition, Ta-8W-2Hf) offered higher strengths and probably improved corrosion resistance to NaK. But the main deterrent to their use was the uncertainty of their resistance to Hg corrosion, particularly in the strained state. Data from reference 8 had indicated possible problems in that regard. Since it was possible, however, that it might become necessary to change to a higher strength refractory alloy, a test program was initiated to ascertain whether Hg corrosion and/or stress corrosion problems actually existed with Nb-1Zr and T-111.

The test program was conducted under NASA sponsorship by the General Electric Company (ref. 9). Sheet specimens of Ta, Nb-1Zr, and T-111 in the as-bent and in several as-bent-and-annealed conditions were exposed to liquid Hg isothermally in tantalum capsules at 650° C (1200° F) for 1000 hours. All specimens were totally unaffected by this exposure. It was therefore concluded that either Nb-1Zr or T-111 could be substituted for Ta in the SNAP-8 system should the need arise.

Some Hg corrosion testing of Ta was also conducted by Aerojet General Corporation under the scope of the SNAP-8 prime contract (refs. 1, 10, and 11). Included in the testing was an 8700-hour test of a full-size SNAP-8 Hg boiler. General Electric Com-

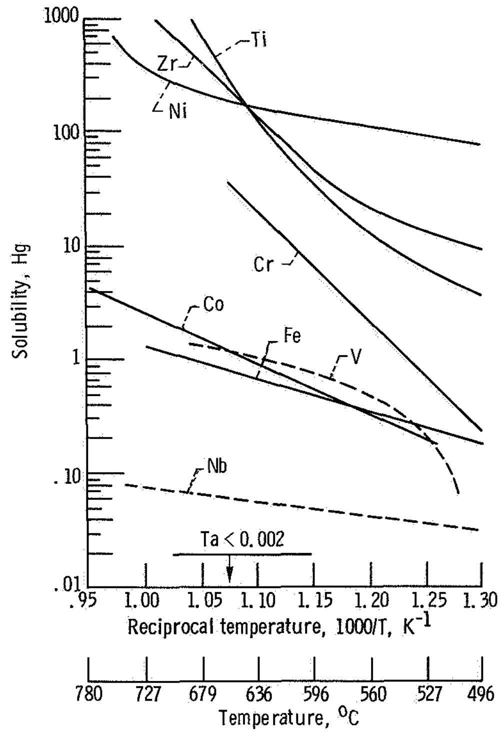


Figure 6. - Solubility of elements in mercury (ref. 8).

pany tested another full-size SNAP-8 boiler for 15 250 hours (ref. 12). In none of these tests did the Ta show any sign of Hg attack, thus verifying its acceptability for long-term service.

Cavitation damage. - Based on early 9Cr-1Mo steel corrosion loop testing (ref. 7), it appeared that certain mechanisms present during the Hg boiling process might resemble a cavitation situation. As a result, studies were initiated at Lewis (ref. 13) and at the University of Michigan (ref. 14) to determine simulated cavitation effects of Hg on various materials. At Lewis, 9Cr-1Mo steel was compared with three materials believed to have good resistance to cavitation damage due to their strength and/or hardness: L-605, Hastelloy X, and Stellite 6B. The test specimens were vibrated in 150° C (300° F) liquid Hg at 25 000 hertz at a peak-to-peak displacement amplitude of 0.0045 centimeter (0.00175 in.). The 9Cr-1Mo steel was the least cavitation resistant of the materials tested (see fig. 7). In the University of Michigan study, Ta was compared with 9Cr-1Mo steel and niobium in liquid Hg at 260° C (500° F). The frequency of vibration of the test specimens was 20 000 hertz, and the amplitude was 0.05 centimeter (0.002 in.). It was determined that Ta was considerably less resistant to cavitation damage than was the 9Cr-1Mo steel.

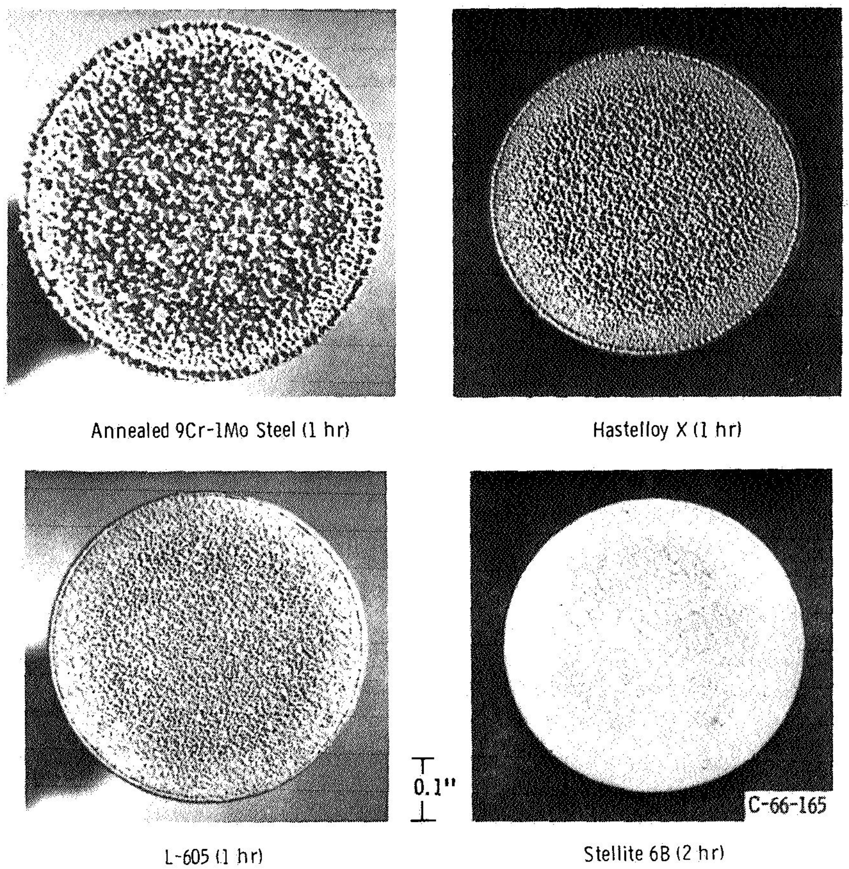


Figure 7. - Damaged surfaces of specimens after exposure to cavitation in mercury at 150° C (300° F) (from ref. 13).

Based on the results of these tests, it was concluded that neither 9Cr-1Mo steel nor Ta would be very resistant to cavitation should such a phenomenon actually be present in a SNAP-8 boiler. But the subsequent Ta corrosion testing revealed no evidence of cavitation damage (refs. 1 and 10 to 12). Thus it was concluded that either no cavitation situation actually existed in the boiler or that cavitation effects were greatly exaggerated in the laboratory simulation tests.

NaK Corrosion

Conventional iron- and nickel-base alloys. - While 9Cr-1Mo steel was the reference material for SNAP-8, an engineering evaluation of the compatibility of the primary loop constructional materials with the NaK reactor coolant was performed. The main goal was to determine where the maximum corrosive attack would occur and how severe it would be. As a result, a NaK corrosion loop program was contracted to the Oak Ridge National Laboratory (ORNL) (ref. 15). Eleven multimaterial loops, constructed of

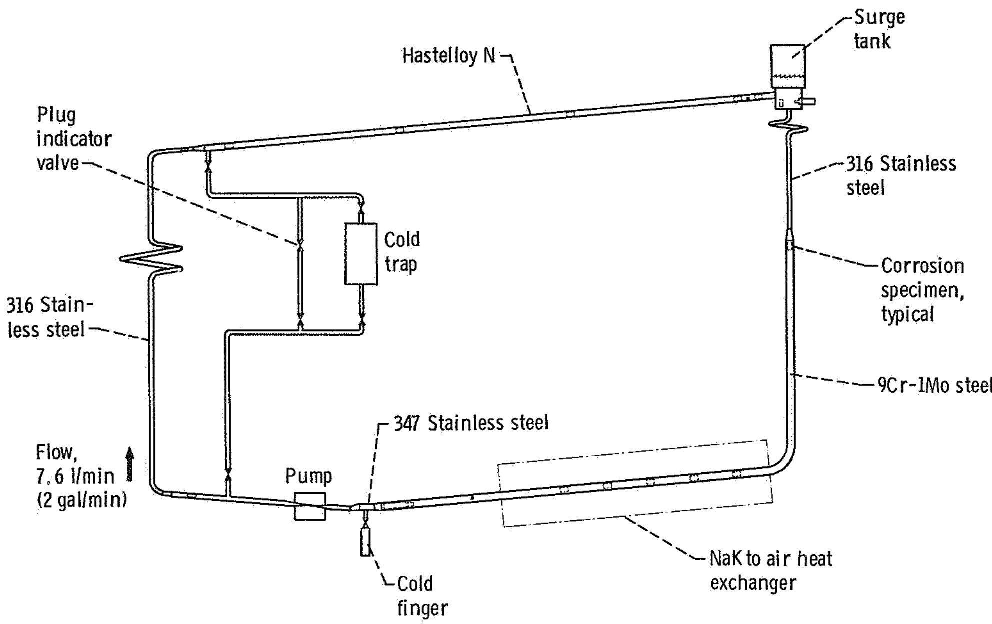


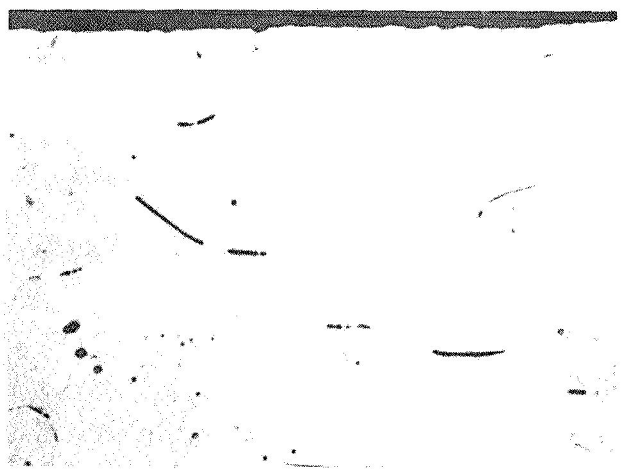
Figure 8. - Diagram of Oak Ridge National Laboratory NaK corrosion loops (ref. 15).

tubular sections of 316 SS, 9Cr-1Mo steel, the nickel-base alloy Hastelloy N, and 347 SS (fig. 8), were operated for times ranging between 700 and 5200 hours at maximum NaK temperatures of 700°, 760°, or 790° C (1300°, 1400°, or 1450° F). The 700° C (1300° F) loops were of the greatest interest because they were expected to be the most representative of the actual conditions at the SNAP-8 reactor outlet. The NaK used was reactor grade.

Corrosion of Hastelloy N (the material that was expected to be the most adversely affected in the SNAP-8 primary loop, based on previous ORNL data from sodium experiments) was less than 0.004 centimeter per 10^4 hours (0.0015 in./10^4 hr) at 700° C (1300° F) and at a NaK oxygen level of less than 30 ppm. This was believed to be acceptably low for a long-life system. At the same NaK oxygen level, the iron-base alloys exhibited minimal corrosive attack. A NaK oxygen level of 80 ppm did not noticeably change the corrosion rate of the Hastelloy N. The iron-base alloys, however, were significantly adversely affected at this higher oxygen level. One deleterious effect of NaK exposure was the decarburization of the 9Cr-1Mo steel and concurrent carburization of the 300-series stainless steels. This loss of carbon reduced the 1000-hour stress rupture strength of the 9Cr-1Mo steel by about 40 percent.

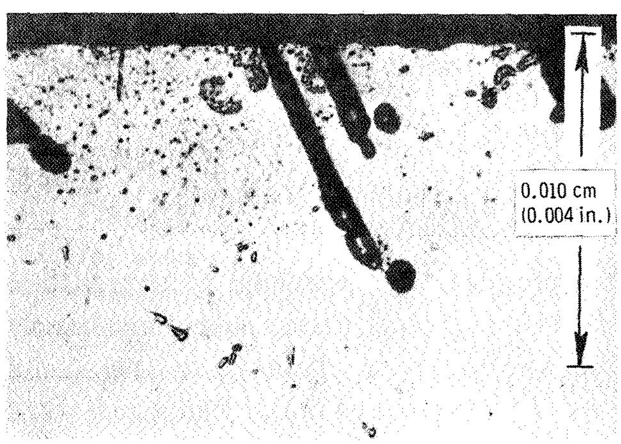
In addition to the corrosion portion of the program, ORNL was to provide information on the behavior and control of the hydrogen (from the reactor fuel) present in the NaK and on the diffusion of hydrogen from the primary loop into the power conversion Hg loop. From these hydrogen investigations, it was concluded that NaK hydride would precipitate in the NaK loop at a temperature of 160° C (320° F). It was also determined that the level of hydrogen in the NaK loop could be significantly reduced by means of a 2.5-percent bypass flow through a 130° C (260° F) cold trap, or by the use of a small quantity (about 0.1 wt. %) of lithium in the NaK to getter the hydrogen. The resultant hydride could then be effectively cold trapped at a higher temperature (i.e., 200° to 260° C or 400° to 500° F). The permeability of the primary loop materials to hydrogen was also experimentally determined.

Refractory metals. - It is known that oxygen-contaminated Ta is subject to corrosive attack by NaK at elevated temperatures. The extent and character of the attack is dependent both on the bulk oxygen level and on the distribution of the oxygen in the Ta. Since there was a considerable amount of Ta in contact with NaK (albeit nonflowing NaK) in the double containment boiler, it was important to determine quantitatively the extent



(a) Base metal (unwelded specimen).

(b) Heat affected zone (welded specimen).



(c) Weld.

Figure 9. - Corrosion in tantalum specimens contaminated with 270 ppm oxygen and exposed to NaK for 1000 hours at 730° C (1350° F) (ref. 16). (Etchant: 30 g NH_4F, 50 ml HNO_3, 20 ml H_2O.) X500.

and character of NaK corrosion over the probable range of oxygen levels and oxygen distributions in the Ta.

This contracted test program was conducted by the General Electric Company (ref. 16). Tantalum specimens were purposely contaminated to achieve homogeneous oxygen concentrations of 115, 220, 270, or 520 ppm. Additional contaminated and uncontaminated specimens were gas tungsten-arc (GTA) welded in pure helium or helium contaminated with air to evaluate the combined effects of welding, welding gas purity, and preweld oxygen concentration of the Ta on the corrosion resistance of the Ta to NaK. The specimens were subsequently exposed to reactor grade NaK at 730° C (1350° F) for 1000 hours in isothermal capsule tests to determine the threshold oxygen concentration for corrosion. Some specimens were also exposed at 650° C (1200° F) for 100 hours to determine the effects of temperature and time on corrosion.

The two major conclusions of this test program were as follows: (1) Tantalum having a uniformly distributed oxygen concentration of about 115 ppm or less will not be attacked by NaK at temperatures up to 730° C (1350° F), but attack on the Ta will definitely occur at a uniformly distributed oxygen concentration of 270 ppm and above (fig. 9(a)). (2) Gas tungsten arc welding of contaminated Ta specimens changed the morphology of the subsequent NaK corrosion; that is, the corrosion generally was worse in the weld and heat affected zone than in the base metal (fig. 9).

MECHANICAL PROPERTIES OF UNALLOYED TANTALUM

When the SNAP-8 double containment boiler was being designed, very little tensile data and virtually no long-time creep or low-cycle fatigue data were available for unalloyed Ta at 730° C (1350° F) and below. Therefore, a series of mechanical property tests were conducted to obtain these data. All tests were performed under contract to Lewis. The results are summarized in the following sections.

Tensile Properties

The Ta tensile testing program was conducted by Metcut Research Associates. Specimens from the actual lots of tubing, plate, sheet, and bar that were to be used in the fabrication of the boilers were tested. Uniaxial tensile specimens were machined from longitudinal elements of the seamless boiler tubing and were tested with special grips designed to accommodate the tube curvature. All elevated temperature tests were conducted in a vacuum of $<6.7\times10^{-4}$ newton per square meter (5×10^{-6} torr); most of the specimens were tested at 730° C (1350° F). Results of the tests are presented in references 17 and 18. Table I is a summary of these data.

TABLE I. - TENSILE PROPERTIES OF UNALLOYED TANTALUM IN THE

RECRYSTALLIZED CONDITION

Tantalum material	Temperature		Tensile strength		Yield strength		Elongation, percent	Reduction of area, percent
	°C	°F	MN/m^2	ksi	MN/m^2	ksi		
0.396 cm (0.156 in.) sheet	730	1350	119	17.3	48	7.0	56	93
			119	17.3	45	6.6	52	95
			121	17.5	41	5.9	61	92
			121	17.5	45	6.6	56	88
			121	17.6	52	7.6	51	95
			125	18.1	60	8.7	55	91
0.409 cm (0.161 in.) sheet	730	1350	122	17.7	45	6.5	68	93
	730	1350	118	17.1	44	6.4	61	89
	730	1350	119	17.3	46	6.7	74	86
0.635 cm (0.250 in.) plate	730	1350	107	15.5	43	6.2	64	90
			110	15.9	37	5.3	62	79
			110	16.0	34	6.0	44	79
			112	16.3	34	5.0	61	85
			116	16.8	53	7.7	52	73
			121	17.5	34	5.0	59	84
2.54 cm (1.00 in.) plate	730	1350	124	18.0	74	10.7	32	81
	730	1350	145	21.0	62	9.0	41	84
	730	1350	146	21.2	58	8.5	37	63
2.54 cm (1.00 in.) plate	730	1350	150	21.7	66	9.6	44	84
	730	1350	157	22.8	67	9.7	32	82
	730	1350	161	23.4	64	9.3	40	79
1.65-cm (0.652-in.) i.d. by 0.13-cm (0.051-in.) wall	730	1350	161	23.4	61	8.8	34	[a]63
			136	19.7	64	9.3	45	[b]71
			157	22.8	73	10.6	38	[c]67
			176	25.5	70	10.1	44	[c]59
			199	28.8	88	12.8	33	[d]65
			179	25.9	48	7.0	38	[e]70
			139	20.2	58	8.4	47	[f]74
			161	23.4	59	8.6	40	[f]67
			160	23.2	52	7.6	35	[f]67
			154	22.4	66	9.6	39	[f]68
	595	1100	181	26.2	68	9.9	27	73
	595	1100	179	26.0	56	8.2	25	74
	425	800	200	29.0	87	12.7	37	77
	425	800	211	30.6	87	11.8	33	76
	260	500	218	31.6	70	10.1	45	79
	260	500	212	30.8	68	9.9	45	77
	25	75	278	40.3	170	24.6	58	74
	25	75	267	38.8	125	18.1	54	80

[a]Heat A.
[b]Heat B.
[c]Heat C.
[d]Heat D.
[e]Heat E.
[f]Heat F.

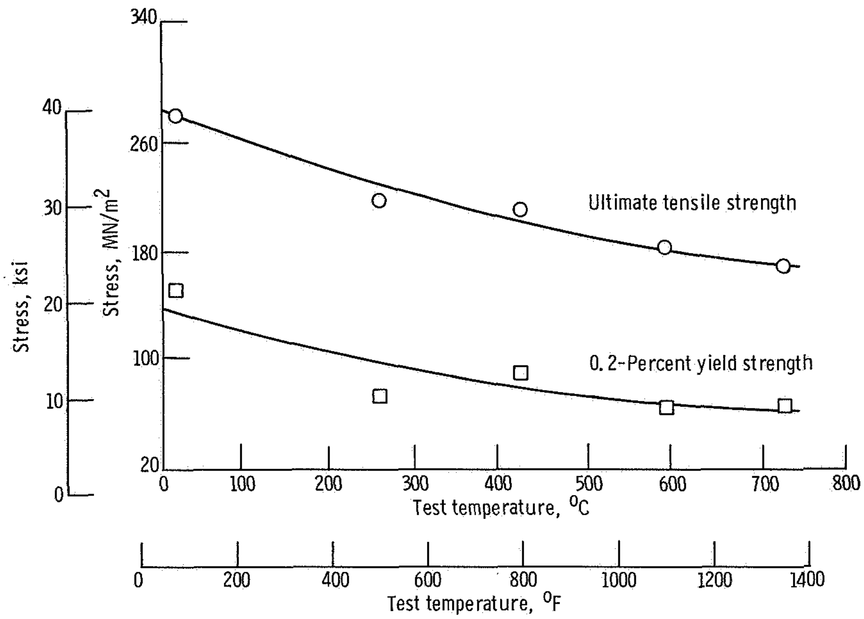


Figure 10. - Ultimate tensile and yield strength of unalloyed recrystallized tantalum tubing.

Figure 10 is a plot of the ultimate tensile and yield strength data for the tubing (averaged). At 730^{o} C (1350^{o} F) the most extensive scatter in the test results for a given shape was observed for the tubing specimens (see table I). This may have been the result of the slight chemistry variations from heat to heat and/or the grain size variations (ASTM grain size No. 3 to 7) from tube to tube. The greatest scatter in data occurred among the different shapes, that is, tubing, plate, sheet, and bar. This was expected because the thermomechanical history for each shape was different and this is known to noticeably affect the mechanical properties of most materials. All materials tested showed adequate tensile properties for their intended applications in the boiler. The goal for the tubing was 0.2-percent yield strength and an ultimate tensile strength about as high as 9Cr-1Mo steel at 730^{o} C (1350^{o} F), that is, 55 and 110 meganewtons per square meter (8.0 and 16.0 ksi), respectively.

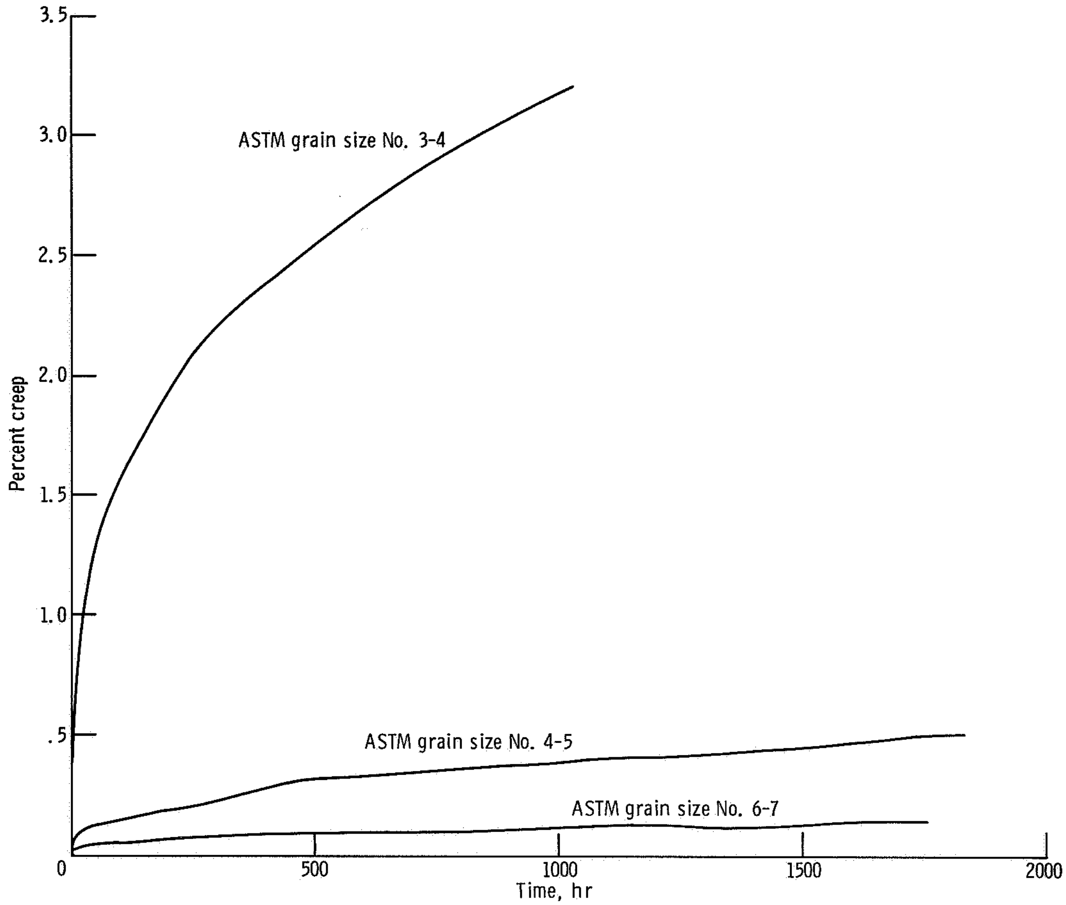
Creep Properties

A uniaxial creep testing program was conducted by TRW, Inc. (ref. 19). The highest stresses on the Ta, as determined by a stress analysis of the boiler, existed in the Ta tubing and in the Ta dome-shaped manifold. Therefore, most of the creep specimens were machined from the actual lots of tubing (1.65-cm (0.652-in.) i.d. and 0.13-cm (0.051-in.) wall thickness) to be used in the boilers or from the actual sheets (0.41-cm (0.16-in.) thick) from which the manifolds were to be formed. A sheet specimen was also tested in a prestrained condition to simulate the actual manifolds, which are strained 35 to 45 percent during fabrication. The tubing specimens were machined so as to concentrate the stress in the gage area. All tests were conducted in a vacuum of 1.3×10^{-6} newton per square meter (1×10^{-8} torr), and most were tested at 730° C (1350° F).

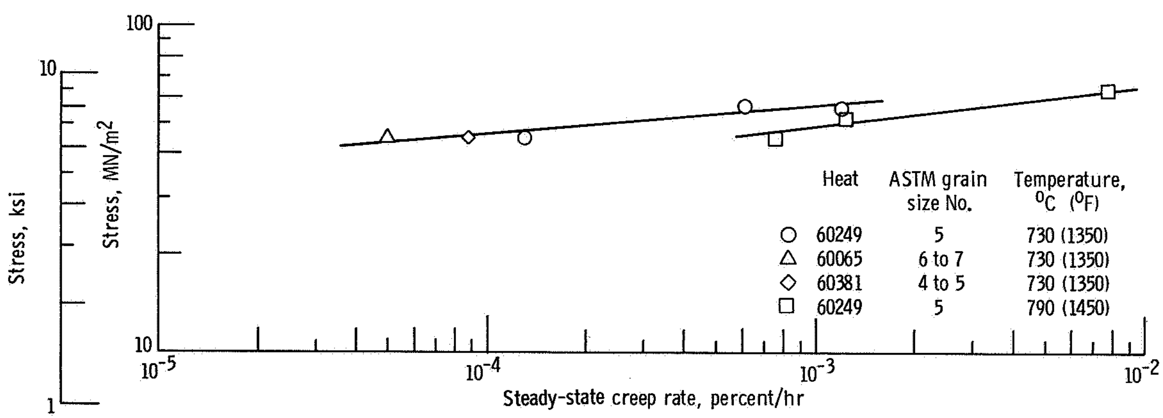
The results for the tubing specimens fell into two separate ranges, depending on the grain size of the test material. (Typical results are shown in fig. 11(a).) The larger grained specimens (ASTM grain size No. 3 to 4) tended to be weaker than the smaller grained specimens (ASTM grain size No. 4 to 7), which is typical of material tested below its equicohesive temperature. Steady-state creep rate for the small-grained specimens is shown in figure 11(b). The aforementioned prestrained sheet specimen was deformed 30 percent before testing, which was the maximum uniform elongation that could be achieved. This was considered to be an adequate first-order approximation of the condition of the material after forming into the dome-shaped manifold. The results of this test revealed a drastic difference between the recrystallized and prestrained material. The time to 1 percent creep at 44.8 meganewtons per square meter (6500 psi) and 730° C (1350° F) for the recrystallized material was 45 hours and for the prestrained material about 37 000 hours (extrapolated from a 4900-hr test). It is possible that in service some stress relieving might occur, but the actual stress on the manifold was not actually expected to be as high as the test point stress. It was concluded, therefore, that the Ta dome-shaped manifold would have adequate creep strength for its application in the boiler if used in the prestrained condition. It was also concluded that recrystallized fine-grained Ta tubing (ASTM grain size No. 5, or higher) would have adequate creep strength for its intended application in the boiler (i.e., <2 percent in 40 000 hr at 730° C (1350° F) for stresses of about 20.7 MN/m^2 (3000 psi)).

Low Cycle Fatigue Tests

Since the SNAP-8 boiler had to be capable of 100 startups and shutdowns for ground testing, it was necessary to obtain data on the low-cycle fatigue behavior of unalloyed Ta. This testing program was conducted at the General Electric Research Laboratory (ref. 20). Mechanical strain was used to simulate the strain effected by the temperature cycles of startup and shutdown. Recrystallized 1.27-centimeter (0.5-in.) diameter bar specimens were used. Because of the extreme sensitivity of Ta to environmental contamination at high temperature, a special titanium susceptor-heater in combination with a highly purified flowing argon gas was used during the testing. Tests were conducted in a tightly sealed chamber over the temperature range 20° to 760° C (70° to 1400° F) with emphasis on tests at 320°, 590°, and 730° C (600°, 1100°, and 1350° F). The resulting



(a) Effect of grain size on creep rate of unalloyed tantalum tubing. Temperature, 730° C (1350° F); stress, 44.8 meganewtons per square meter (6500 psi).



(b) Steady-state creep rate for unalloyed tantalum tubing.

Figure 11. - Creep behavior of unalloyed tantalum tubing.

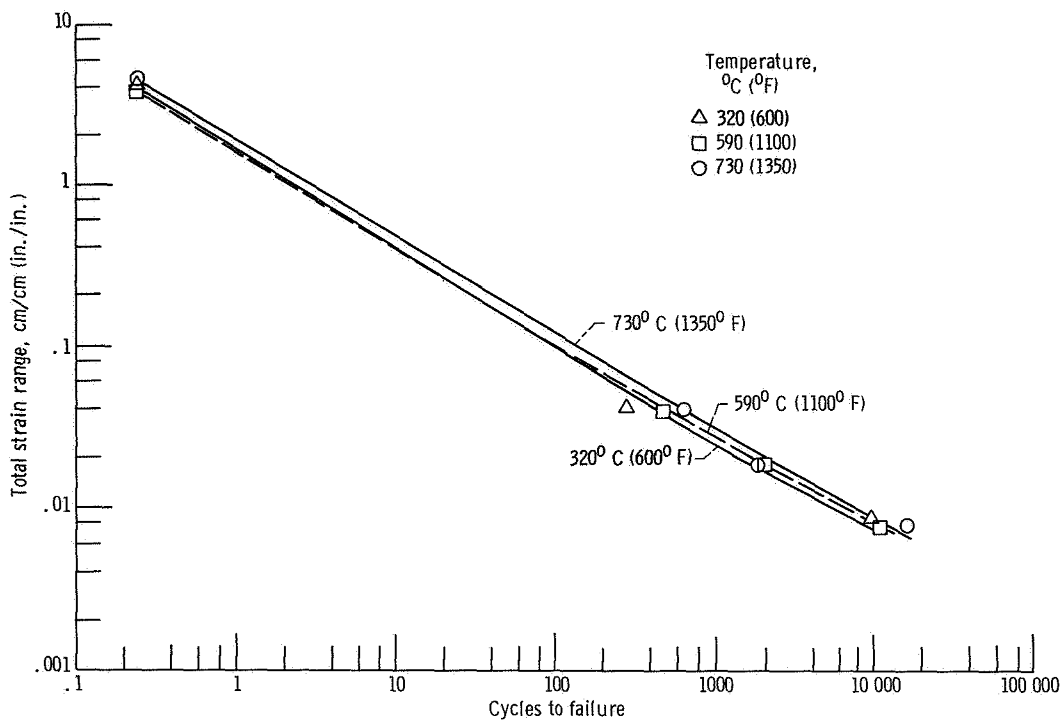


Figure 12. - Low cycle fatigue behavior of unalloyed tantalum bar.

data are shown in figure 12. Analysis of the data indicated that the low-cycle fatigue life of unalloyed Ta at the strain ranges to which it would be exposed in the SNAP-8 boiler (maximum of 0.02 cm/cm (in./in.)) should be in excess of 1000 cycles. Also, fatigue life was not very temperature dependent over the temperature range used in these tests.

FABRICATION AND EVALUATION OF BIMETALLIC TUBING AND JOINTS

As mentioned previously, there was two SNAP-8 boiler designs. The bimetal tube boiler was a counterflow design (fig. 13) using seven Ta/316 SS tubes from one boiler header to the other. The double containment boiler design (fig. 14) consisted of seven unalloyed Ta boiler tubes, each within a flattened-oval 321 SS tube, which was required to accommodate the difference in thermal expansion between the Ta and the 321 SS. The annulus between each Ta and 321 SS tube was filled with nonflowing NaK. The seven tubular assemblies were further contained in a 316 SS boiler outer shell within which the primary loop NaK flowed in a counterflow direction. The Ta mercury containment tubes were interconnected by Ta header-manifolds at the boiler inlet and outlet. To avoid continuation of Ta outside the 316 SS boiler outer shell, the Ta header-manifolds were joined within the shell to 316 SS at the boiler inlet and outlet by means of Ta/316 SS

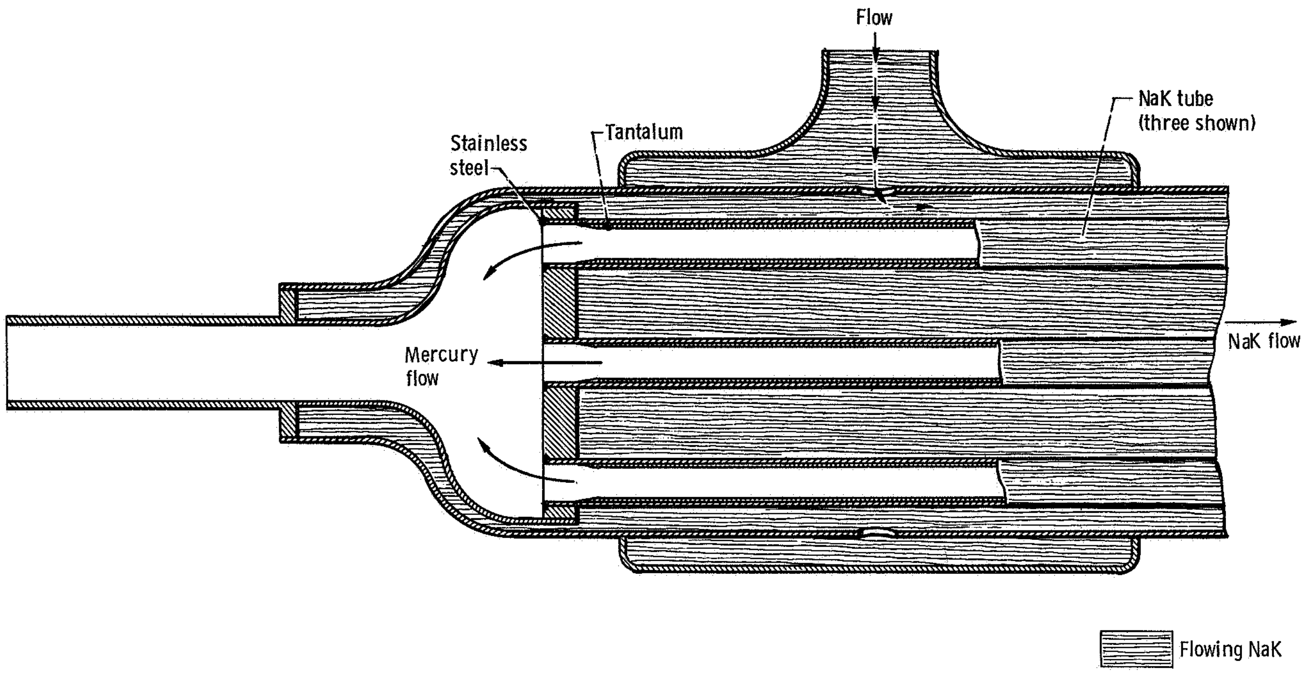


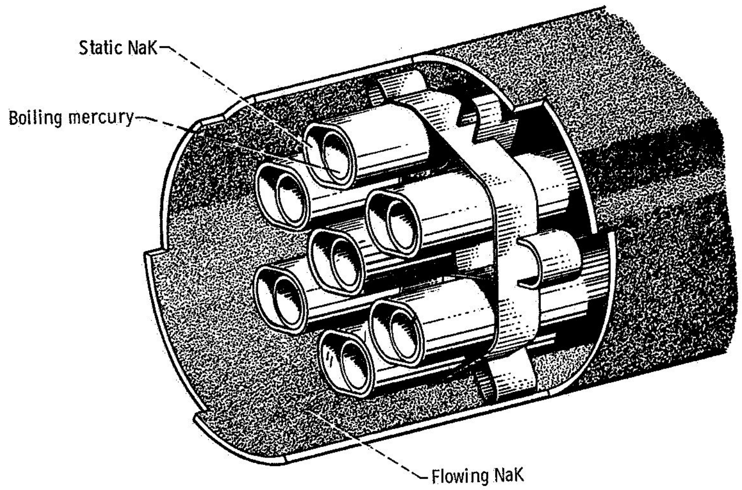
Figure 13. - SNAP-8 bimetallic tubing boiler configuration.

transition joints. Zirconium foil was wrapped around the joints in order to protect them from embrittlement by any carbon and/or nitrogen that might be in the static NaK.

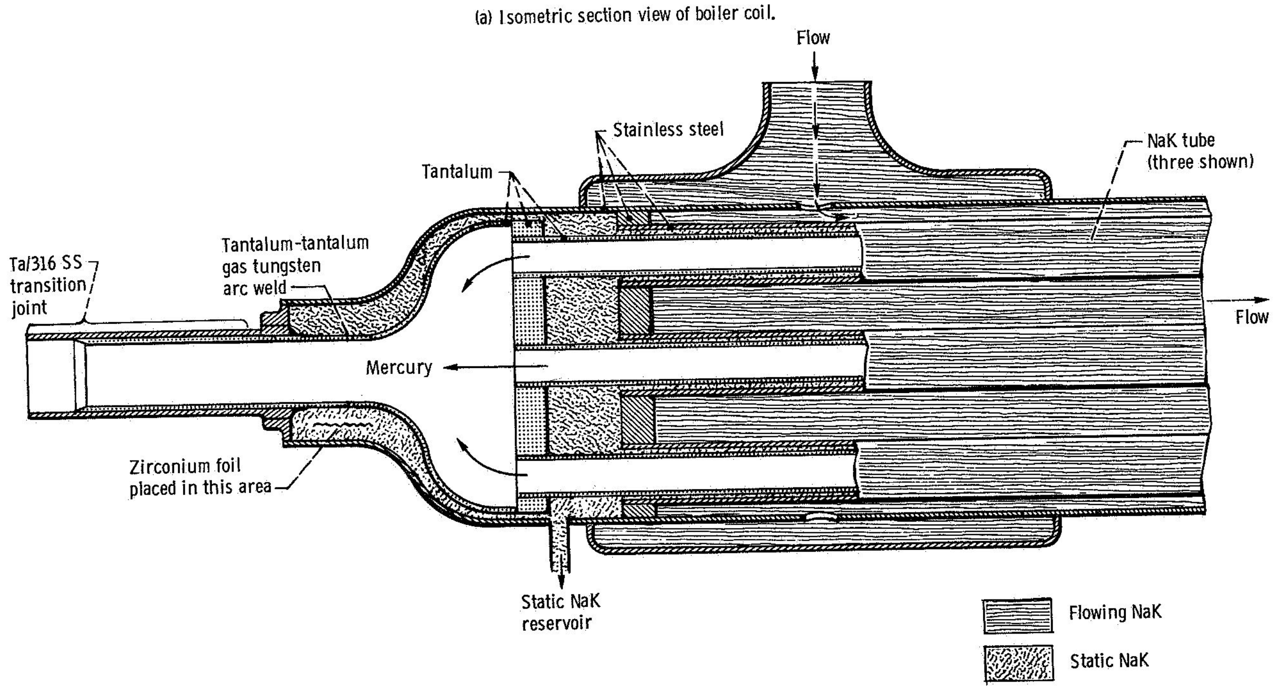
Both the bimetallic tubing and bimetallic joints required considerable fabrication process development and evaluation. This was performed under several NASA contracts and the results are summarized in the following sections.

Initial Investigation

Early in the SNAP-8 program, consideration had been given to the possibility of using refractory metals such as Nb or Ta to contain the Hg working fluid in the boiler. It was believed at that time that steel-clad bimetallic tubing would be required to prevent the refractory metal from gettering embrittling elements, such as carbon and nitrogen, from the flowing NaK stream. Experimental quantities of Nb or Ta/SS tubing were produced by several vendors using the following fabrication processes: hot coextrusion and drawing, explosion welding and drawing, and explosion welding to size. A program to evaluate the various typesof bimetallic tubing and to develop welded joints using this tubing was conducted at the Westinghouse Astronuclear Laboratory (refs. 21 and 22). The various types of tubing were compared on the basis of bond integrity, dimensional control, and surface condition. On this basis, the Nb/316 SS tubing produced by hot coextrusion and drawing was considered to be the best. The weld-joint investiga-



(a) Isometric section view of boiler coil.



(b) Boiler end.

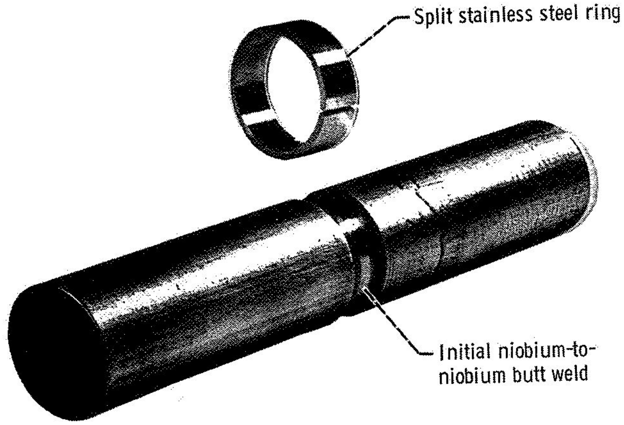
Figure 14. - SNAP-8 double containment boiler configuration.

tion was concerned with three basic configurations: a butt joint, a tee joint, and a tube-to-header joint. Coextruded and drawn Nb/316 SS tubing was used. After considerable experimentation, a successful design for each of the three basic configurations was developed (figs. 15 to 17). Several of each type joint were produced and tested primarily to determine their ability to withstand thermal cycling between 320° and 730° C (600° and 1350° F) and an internal pressure of 3.9 meganewtons per square meter (565 psia) at 730° C (1350° F). The test results indicated that the joints would withstand the SNAP-8 system operating conditions (370° C (1350° F) and 1.9 MN/m² (275 psia)). The details of the tubing evaluation and joint development efforts are described in references 21 and 22.

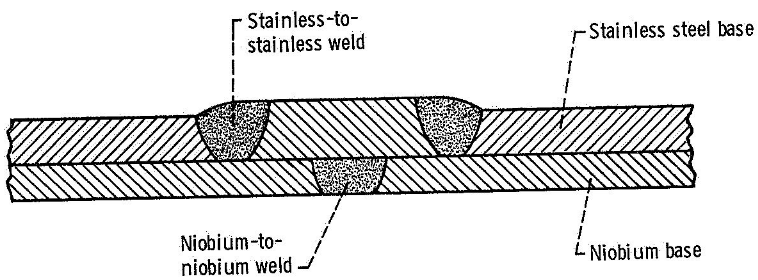
Concurrent with these programs, an investigation was conducted to determine the optimum refractory metal/austenitic alloy combination for use in the SNAP-8 system. This program was also conducted at the Westinghouse Astronuclear Laboratory (ref. 23). Sixteen bimetallic combinations, produced by explosion welding, were evaluated by interdiffusion experiments at 760°, 820°, and 870° C (1400°, 1500°, and 1600° F), room temperature tensile tests, creep-rupture tests at 730° C (1350° F), and thermal cyclic testing between 320° and 730° C (600° and 1350° F). The bimetals consisted of Nb, Ta, Nb-1Zr, FS-85, or T-222 in combination with 321 and 347 SS or the nickel-base alloys Inconel 600 and Hastelloy N. A major finding of the program was that the bimetallic combinations having an interdiffusion zone thickness of less than 12.7×10^{-4} centimeter (5×10^{-4} in.) would withstand a minimum of 20 thermal cycles between 320° and 730° C (600° and 1350° F) without degradation of the interface weld. It was concluded that the optimum refractory metal/austenitic alloy combination was tantalum/300 series stainless steel, primarily on the basis of minimum interdiffusion. The details of the evaluation can be found in reference 23.

Tantalum/316 Stainless Steel Tubing Development

General. - As mentioned previously, evaluation of a small quantity of several similar types of experimental bimetallic tubing had indicated that the piece of tubing produced by hot coextrusion followed by cold drawing was the best. Other sections of tubing from this same lot, however, were found in other investigations to have a significant amount of nonwelded areas. It was therefore considered necessary to further improve the fabrication procedures for bimetallic tubing before tantalum/300 series stainless steel could be seriously considered for SNAP-8 use. Since cold drawing was apparently responsible for the unwelded areas observed, it was decided to investigate processes that excluded this step. The processes investigated were hot coextrusion to final size and explosion welding to final size. The stainless steel selected was type 316. Hot coextrusion to size was attempted by two suppliers under NASA contract: the Nuclear

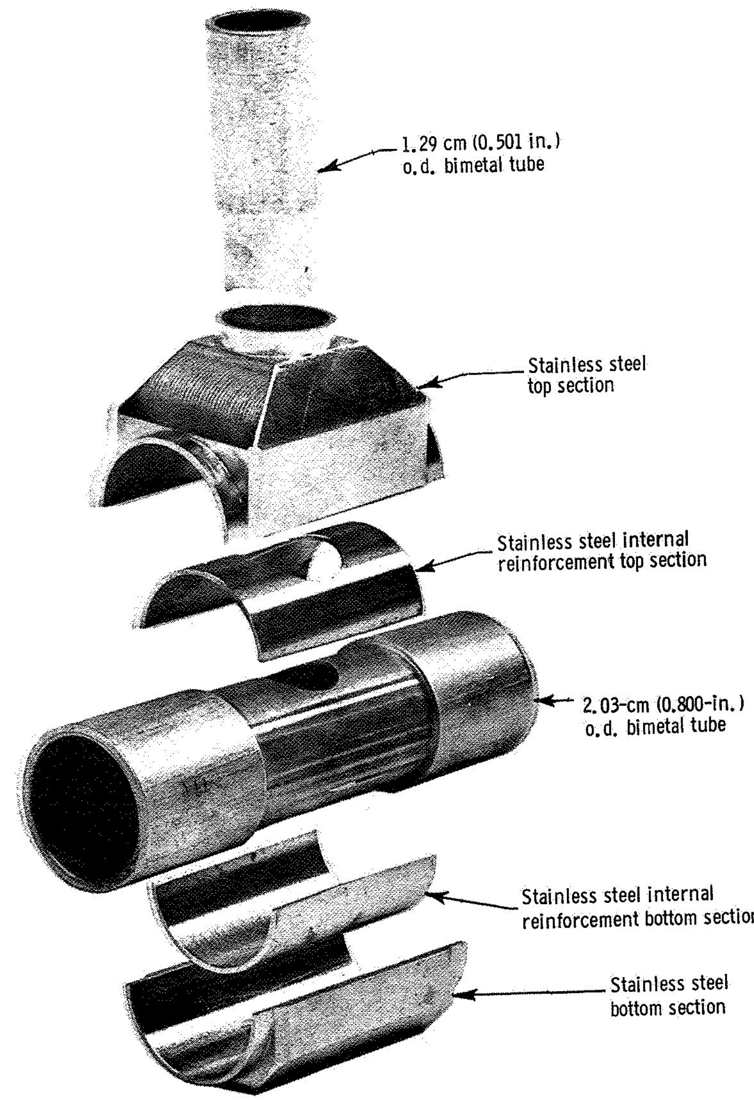


(a) Assembly view.



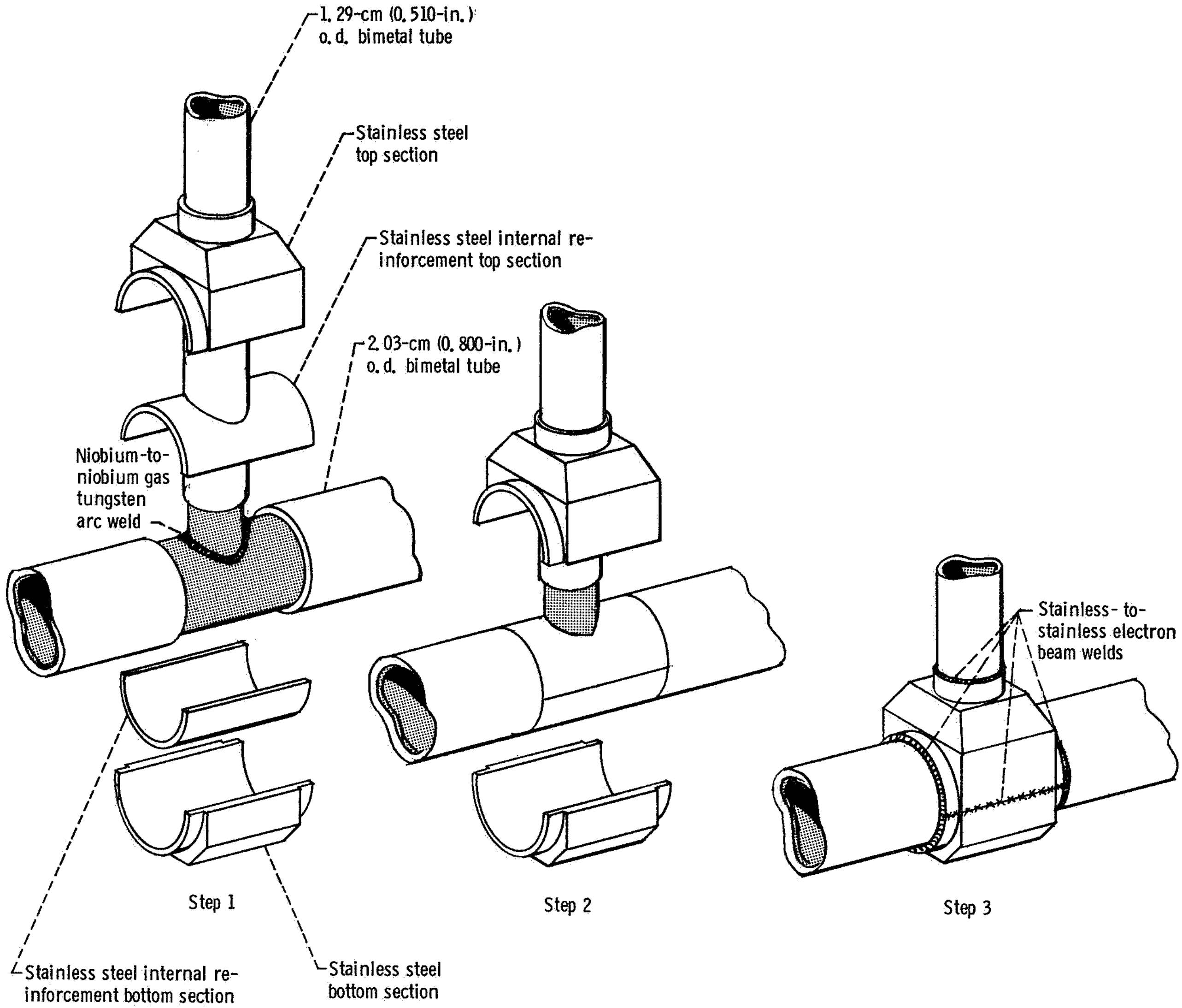
(b) Joint cross section.

Figure 15. - Niobium/316 stainless steel bimetallic tubing butt joint produced by gas tungsten-arc welding (ref. 21).



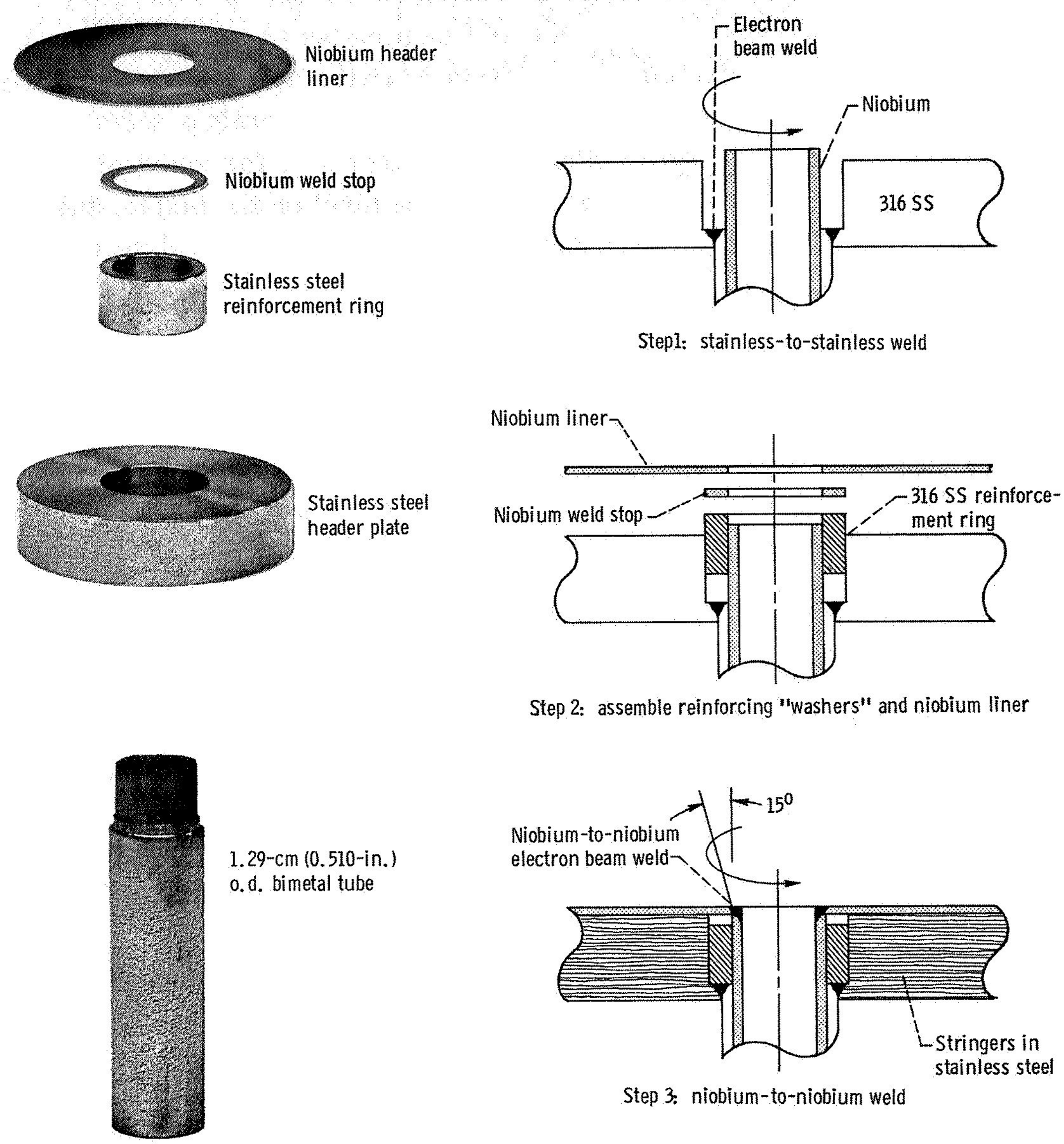
(a) Assembly view.

Figure 16. - Niobium/316 stainless steel bimetalic tubing tee joint (ref. 21).



(b) Sequence of welding.

Figure 16. - Concluded.



(a) Assembly view. (b) Sequence of welding.

Figure 17. - Niobium/316 stainless steel bimetallic tubing tube-to-header joint (ref. 21).

Metals Division of the Whittaker Corporation and the Metalonics Division of the Kawecki Chemical Corporation. Nuclear Metals extruded over a tool steel mandrel; Metalonics used a filled-billet technique. Explosion welding to size was attempted by Aerojet-Downey, under subcontract to Aerojet-General. The tubing, in each case, was to have a 1.65-centimeter (0.652-in.) inside diameter and be longer than 4.6 meters (15 ft). The Ta wall thickness in all cases was to be 0.051 centimeter (0.020 in.). The 316 SS wall thickness was to be 0.152 centimeter (0.060 in.) for the coextruded tubing and 0.203 centimeter (0.080 in.) for the explosion welded tubing (readily available stock).

Hot coextruded tubing - mandrel. - The billet configuration for mandrel extrusion (fig. 18) consisted of four concentric cylinders: carbon steel on the inside, then Ta, stainless steel, and carbon steel on the outside. Carbon steel front and rear plates and

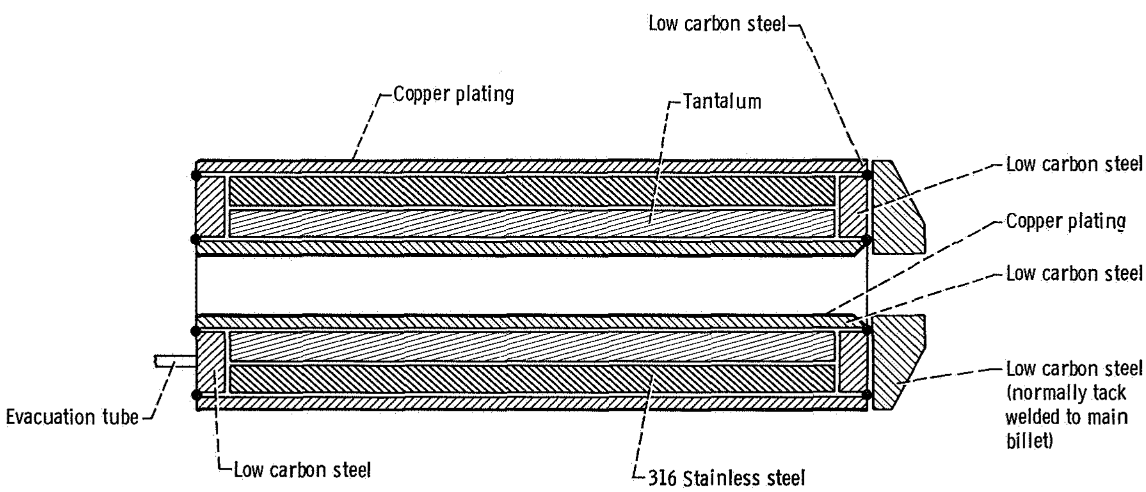


Figure 18. - Extrusion billet design for producing tantalum/316 stainless steel tubing using a mandrel extrusion method.

a carbon steel nosepiece were also used. The assembly was welded as shown, then outgassed and sealed at an elevated temperature. The original overall billet size was calculated to produce a tube about 6.1 meters (~20 ft) long at a reduction ratio of 32:1. The final extrusion conditions selected were 1000° C (1830° F) and a reduction ratio of 25:1. The particulars of the billet assembly process and the extensive development program required to produce sound tubing, heretofore unpublished, are presented in the appendix. Thirteen tubes, approximately 4.6 meters (15 ft) long, were successfully produced and prepared for subsequent evaluation (to be discussed in a later section).

Hot coextruded tubing - filled billet. - The filled-billet configuration (fig. 19) consisted of a plain carbon steel core over which was tightly fit, in turn, a Ta cylinder and a stainless-steel cylinder. A nose piece and tail piece, also of carbon steel, were

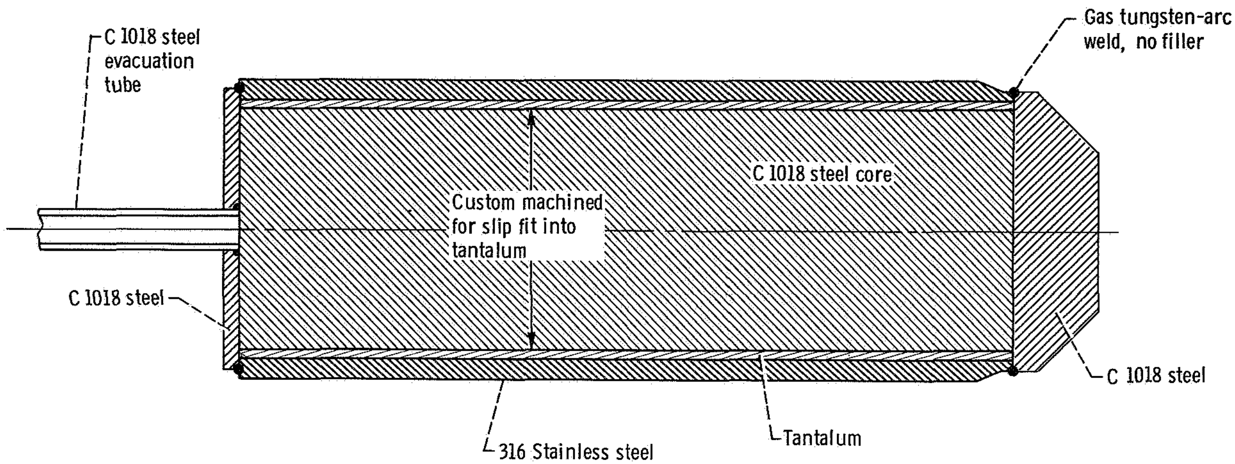


Figure 19. - Extrusion billet design for producing tantalum/316 stainless steel tubing using a filled billet method.

welded to the 316 SS outer cyliner. The assembly was then outgassed and sealed at an elevated temperature. The overall billet size was calculated to produce a tube about 6.1 meters (20 ft) long at a reduction ratio of 20:1. The nominal extrusion temperature was about 1030° C (1880° F). The particulars of the billet assembly process and the extrusion process, heretofore unpublished, are presented in the appendix. Twenty tubes, greater than 4.6 meters (15 ft) long, were successfully produced and prepared for subsequent evaluation (to be discussed in a later section).

Explosion welded tubing. - The billet configuration used for explosion welding is shown in figure 20. The Ta tubing was dimpled by means of a special hydraulic tool. After degreasing, the inside of each tube was filled with Cerrobend-A using a vertical casting technique. Each tube was inspected to insure a void-free Cerrobend core. The bonding was performed with the Ta and 316 SS tubing fixed vertically in a hole in the ground. The explosive used was nitroguanadine powder packed around the outside diameter of the 316 SS tube and held in place by a 8.9-centimeter (3.5-in.) diameter surrounding cardboard tube. The explosion welding technique was successfully developed to the point that 4.6-meter (15-ft) long tubes could be produced. The process, however, had an inherent, undesirable characteristic, which was the lack of welding at the dimple locations. A more detailed description of the explosion welding procedure can be found in reference 24. Thermal cyclic testing of both the explosion welded tubing and the coextruded tubing is described in reference 1.

Evaluation of tantalum/316 stainless steel bimetallic tubing. - Specimens of all three types of coextruded tubing were thoroughly evaluated at Westinghouse Astronuclear Laboratory under NASA contract (ref. 24). All of the tubing was carefully dimensionally inspected: outside diameter, inside diameter, length, and straightness. In addition, each tube was inspected by dye penetrant and ultrasonic techniques.

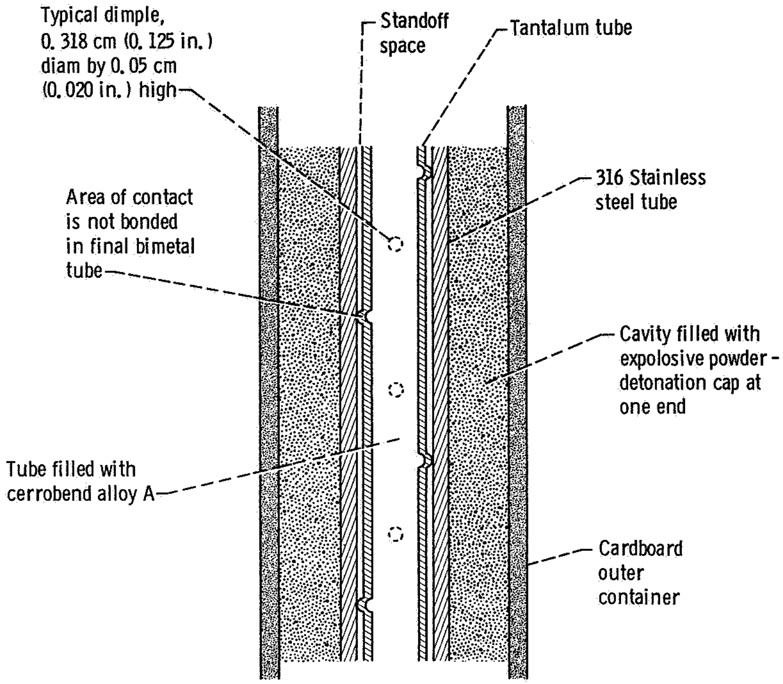


Figure 20. - Configuration and setup for explosion welding of tantalum/316 stainless steel bimetallic tubing.

All three types of tubing had good dimensional control (figs. 21 to 23), although the inside diameter of the coextruded filled-billet tubing was extremely rough (fig. 21). The dye penetrant inspection revealed numerous inside and outside diameter surface defects on much of the coextruded filled-billet tubing. But the mandrel extruded and explosion welded tubing showed no surface defects. Ultrasonic inspection showed the filled-billet tubing to have a few nonwelded spots. And, as expected, the explosion welded tubing was nonwelded at the dimple locations, again, as determined by ultrasonic inspection. The mandrel-extruded tubing had no nonwelded defects. Metallography revealed that both of the coextruded types of tubing had continuous welds and no significant intermetallic layers were observed at the bimetal interfaces. Conversely, the explosion welded tubing showed a nearly continuous hard intermetallic layer at the bimetal interface resulting from the incipient melting that occurs during the explosion welding of the two surfaces.

The test program consisted of thermal cycling between 320° and 730° C (600° and 1350° F) for 100 cycles to determine the effect of thermal stress on the bimetal weld interfaces. Biaxial creep burst tests at 730° C (1350° F) were also conducted using internal gas pressurization. It was observed that the nonwelded spots on the explosion welded tubing propagated along the weld interface during thermal cycling. The coex-

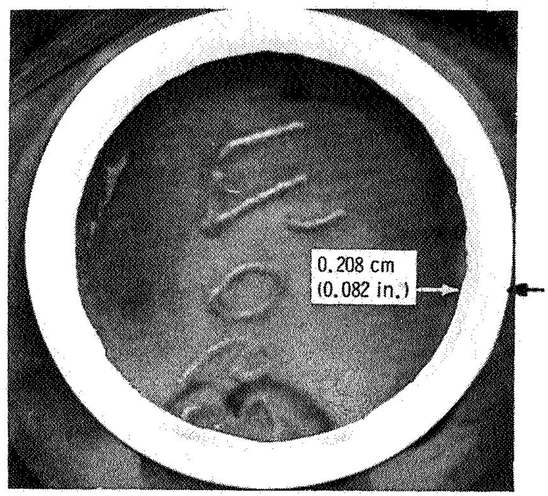


Figure 21. - Transverse section of Tantalum/316 stainless steel tubing produced by a filled billet method (ref. 24). Unetched.

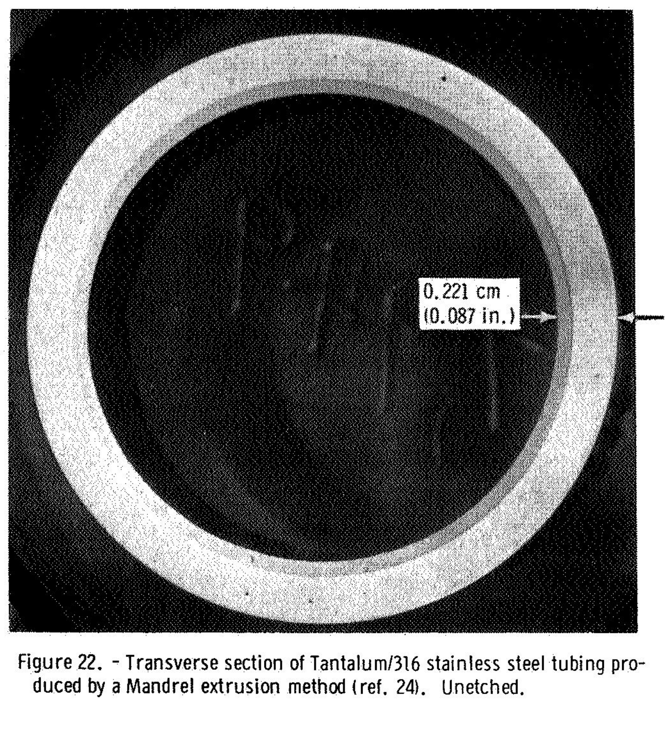


Figure 22. - Transverse section of Tantalum/316 stainless steel tubing produced by a Mandrel extrusion method (ref. 24). Unetched.

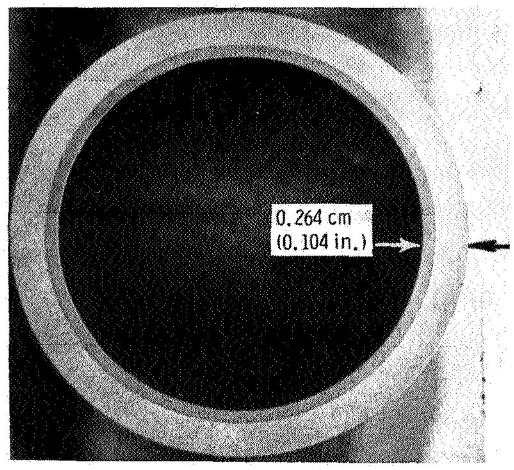


Figure 23. - Transverse section of Tantalum/316 stainless steel tubing produced by explosion welding (ref. 24). Unetched.

truded types showed no noticeable change. After considering all test results for the three types of tubing, it was concluded that the tubing coextruded over a mandrel was the best primarily because of the combination of good surface finish, dimensional control, and general integrity during testing. The details of the evaluation program can be found in reference 24.

Tantalum/316 Stainless Steel Transition Joint Development

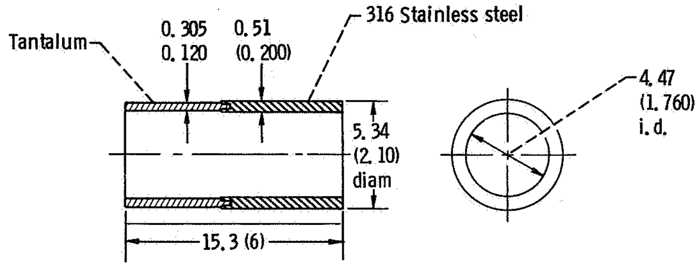
General. - As stated previously, the double containment boiler design required Ta/316 SS inlet and outlet transition joints. To satisfy initial requirements, trial joints of this type were procured from two suppliers: the General Electric Company and the Nuclear Metals Division of the Whittaker Corporation. The General Electric joints were produced using a brazed tongue-in-groove design. The braze alloy used was J-8400 (Co-21Cr-21Ni-8Si-2.5W-0.8B-0.4C). The Nuclear Metals joints were hot coextruded over a mandrel and had a tandem, tapered interface design. Several joints of each type were produced and evaluated. Both types of joints were operated in three full-scale SNAP-8 boilers that were built at Lewis. Also, testing of several joints of each type was performed by Aerojet General Corporation. Results of these tests indicated that both joint types required additional fabrication optimization before being considered adequate for 100 startup cycles and 5 years of service at 730° C (1350° F). During the Aerojet testing, some of the brazed joints showed a serious lack of braze filling. Some of the coextruded tandem joints showed flaws at the interface between the Ta and 316 SS on the outer circumference, which propagated during test. As a result of these determinations, programs were initiated to optimize the fabrication procedure for each joint.

A third joint design, best described as a sleeve joint, was added to this development effort. Basically, the new joint was a large diameter, heavy-wall bimetallic tube machined to a joint configuration, and thus it was basically an extension of the technology acquired during the development and testing of the coextruded bimetallic tubing. It offered several potential advantages over the other types of joint. For example, the sleeve joint, as a bimetallic tube, could be extended toward the SNAP-8 turbine or Hg pump if corrosion effects in the Hg vapor line to the turbine, or liquid Hg line from the pump, became significant. This extension was, of course, not possible with the other two joints. Another advantage would be the long interface length. If interfacial separation occurred, it would have to propagate a much longer distance with the sleeve joint than with the tandem (3.81 cm (1.50 in.) long interface) or brazed joint (1.90 cm (0.75 in.) long interface). Also, a sleeve joint would be much easier to inspect by ultrasonic techniques than either of the other two. Another important advantage would be that a piece of the actual joint could be destructively, as well as nondestructively, examined in the as-fabricated condition by merely removing a ring from either end. It was obviously not possible to destructively examine either the tandem or brazed joint in the as-fabricated condition.

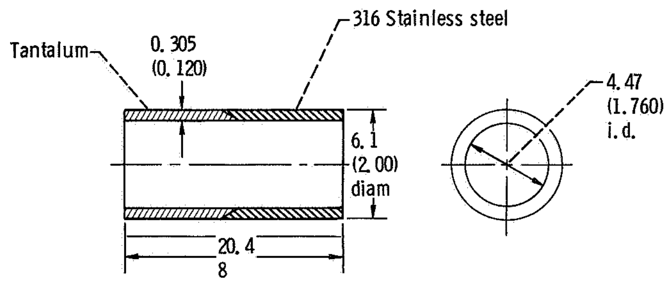
The brazed joint fabrication optimization program was conducted by the General Electric Company. The fabrication optimization program for the two coextruded joints was conducted by Nuclear Metals. The three types of joint and their dimensions are shown in figure 24.

Brazed joints. - A major change to the brazed joint configuration was made for this program. In the early brazed joint design, the tongue of the tongue-in-groove design was stainless steel and the groove was Ta. For the joints produced under this program, this was reversed: the Ta became the tongue and the stainless steel the groove. This arrangement was considered more satisfactory from several standpoints, which included lower joint stress, better braze filling, and improved cleaning efficiency before brazing. The brazing process consisted of several steps. The components were assembled in a vertical position, and the braze alloy (J-8400) was applied as a slurry. The assembly was then positioned in a vacuum furnace, and the furnace was evacuated to a pressure of less than 6.7×10^{-3} newton per square meter (0.5×10^{-5} torr). The assembly was heated to the brazing temperature, held at temperature for a brief time, and then cooled slowly. Extensive experimentation revealed that minimum braze microshrinkage could be achieved by brazing either at 1180° C (2160° F) for 5 minutes or at 1230° C (2250° F) for 1 minute and cooling at a rate of 15° C (25° F) per minute during braze solidification.

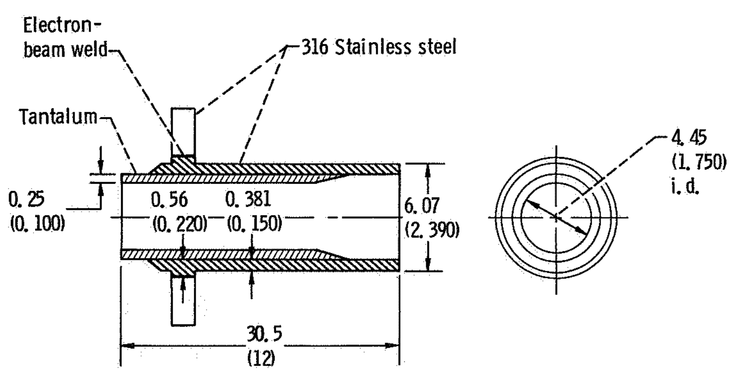
Another program goal, in addition to determining an optimum fabrication procedure, was to ascertain a reliable ultrasonic inspection method for determining the quality of the joints. The capability of ultrasonics to accurately delineate braze integrity was demonstrated by correlating inspection data with physical microstructures of actual prototype joints.



Brazed joint



Tandem tapered joint



Sleeve joint

Figure 24. - Tantalum/316 stainless steel transition joint configuration. (All dimensions are in cm (in.).)

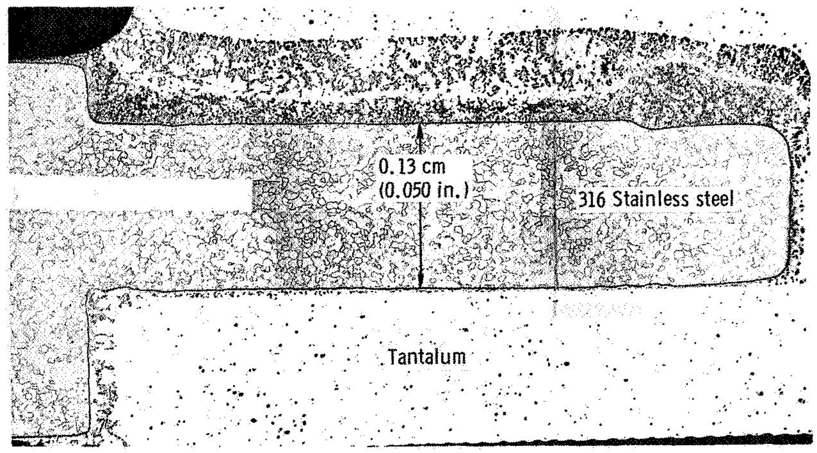


Figure 25. - Longitudinal section of brazed joint in the as-brazed condition (ref. 27).

Twelve 5.1-centimeter (2-in.) outside diameter tubular joints were successfully brazed, and their quality verified by ultrasonic inspection. A typical brazed joint longitudinal cross section is shown in figure 25. The details of this fabrication optimization program can be found in reference 25.

Hot coextruded joints. - The extrusion optimization program for the coextruded tandem and sleeve joints included scrupulous control of prebillet assembly cleaning procedures and careful outgassing and sealing procedures. Also, Ta foil was used in the billet assembly to getter any entrapped air. Several trial billets of each type of joint were extruded at different temperatures and extrusion ratios. Based on the examination of the trial extrusions, the extrusion conditions for the final joints were decided upon. The tandem joints were extruded at 995° C (1825° F) at a 5:1 extrusion ratio; the sleeve joints were extruded at 1070° C (1950° F) at an 8:1 extrusion ratio. Twelve tandem joints were produced. A longitudinal cross section of a typical joint is shown in figure 26. Eight sleeve extrusions were made from which 16 sleeve joints were obtained, since each extrusion was over 61.0 centimeters (24 in.) long. The sleeve cross section was similar to that for coextruded tubing (fig. 22) except that the total wall thickness was 0.89 centimeter (0.35 in.).

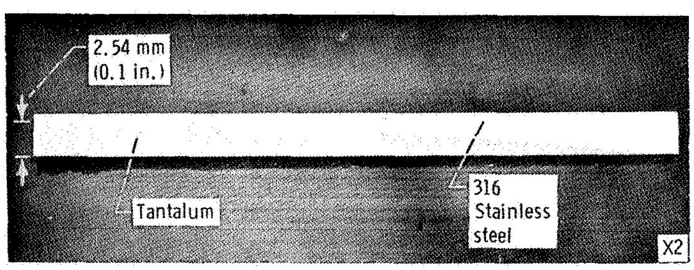


Figure 26. - Longitudinal section of coextruded joint in the as-extruded condition (ref. 27). Unetched.

In addition, two smaller sleeve extrusions were produced with 1.91-centimeter (0.75-in.) inside diameters and the same Ta and 316 SS thicknesses as the large sleeve extrusions. Because these extrusions were over 1.0 meter (40 in.) long, three sleeve joints were obtained from each. These joints were designed to be used at the Hg inlet of the double containment boiler. The details of this fabrication optimization program can be found in reference 26.[1]

Evaluation of joints. - Several joints of each of the three joint types were tested and thoroughly evaluated by Westinghouse Astronuclear Laboratory under Lewis contract (ref. 27). In order to simulate the stress imposed by the double-containment boiler end flange, a stainless-steel collar was attached to the sleeve joints by means of electron beam welding. Each joint was carefully dimensionally inspected (outside and inside diameters, length, and straightness). In addition, each joint was inspected by helium leak, dye penetrant, and ultrasonic techniques. The wall thickness of the sleeve joints varied considerably because the as-extruded sleeves had been insufficiently straightened before machining. The taper lengths of the tandem joints varied from 2.56 to 4.24 centimeters (1.01 to 1.67 in.). The dimensions of the brazed joints showed no significant variation. A few minor surface defects were discovered in each group of joints by the dye penetrant technique. But helium leak checking and the ultrasonic inspection showed no significant potential problem areas.

The joint testing program consisted of thermal cycling four of each type of joint between 120° and 730° C (250° and 1350° F); two of each type of joint were unpressurized, and two were pressurized to 1.83 meganewtons per square meter (265 psia) with argon to simulate boiler operation. Each joint was cycled 100 times; the holding times at 730° C (1350° F) varied from 2 to 10 hours, but two 100-hour soaks were included. In addition, several of the joints were subjected to a 1000-hour soak at 730° C (1350° F) prior to their final 10 cycles. The joints were thoroughly nondestructively inspected periodically by the aforementioned techniques as the thermal cycling progressed.

None of the joints tested showed leaks as determined by the helium leak check. But the dye penetrant and ultrasonic inspection revealed that the condition of all joints deteriorated somewhat during testing. The coextruded sleeve and tandem joints were considerably worse than the brazed joints in this regard. The sleeve and tandem joint deterioration consisted primarily of fissures at or near the Ta/316 SS interface. The brazed joints displayed a small amount of microcracking in the braze. An illustration of the fissuring is shown in figure 27. In addition, the sleeve joints bowed somewhat during test, and both the sleeve and tandem joints displayed diametral contraction in the

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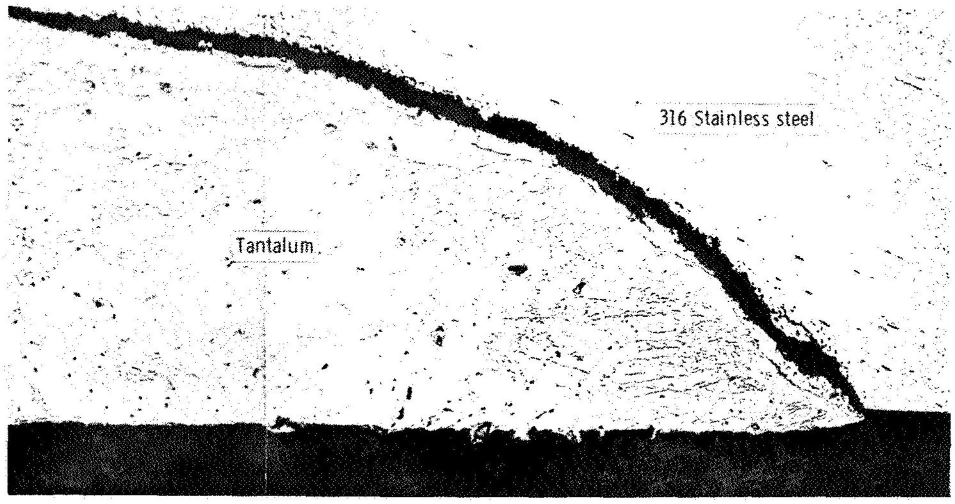
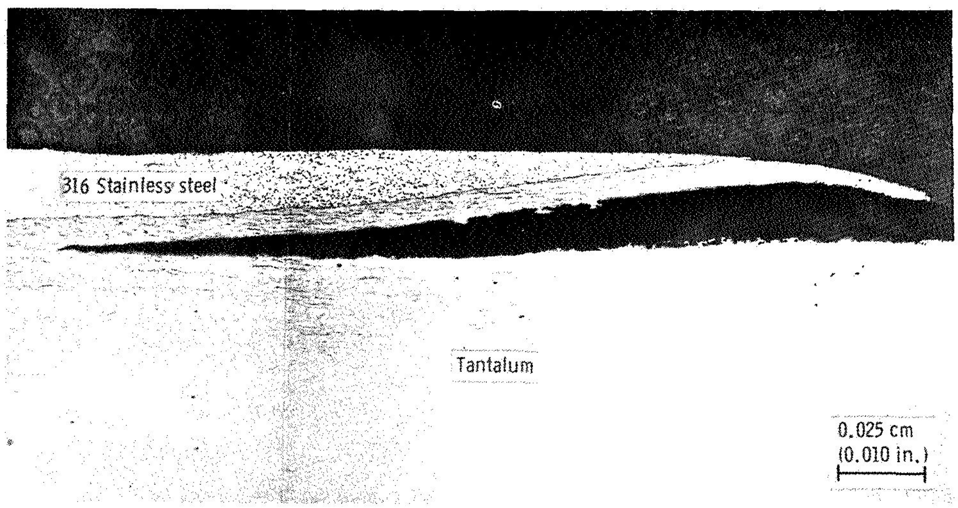
[1]Also in this program, an attempt was made to produce 1.65-cm (0.652-in.) i.d. bimetallic tubing using thin, rolled-up fine-grained Ta sheet in place of seamless Ta tubing and a filled-billet technique. This effort was not successful.



(a) Inside-diameter fissure in stainless steel area.



(b) Outside-diameter fissure in Tantalum area.

Figure 27. - Longitudinal sections of coextruded joint after thermal cycling, showing bond line fissures.

bimetal transition area. No significant interdiffusion between the Ta and 316 SS or Ta, braze, and 316 SS was observed as a result of temperature exposure, and none of any significance would be expected over a 5-year period, based on calculated diffusion rates. It was concluded that, although none of the joints actually failed in test, the sleeve and tandem joints indicated a need for future development before they could be considered acceptable for SNAP-8 service. The brazed joint, having demonstrated good dimensional stability and joint durability was concluded to be the best candidate for SNAP-8 use (at the current state of development).

This conclusion was reinforced by a review of some earlier testing of similar Ta/316 SS brazed joints. A 10-centimeter (2.5-in.) outside diameter brazed joint had survived 150 severe thermal shocks in a high flow velocity Hg test system without apparent damage (ref. 28). Also, failures during 730° C (1350° F) tensile tests of brazed joint configurations had never occurred in the braze itself (ref. 17). Finally, several brazed joints had been used in Hg boiler tests with good results from both a corrosion and structural standpoint.

APPLICABILITY OF RESULTS

Although work on the SNAP-8 program has been terminated, it should be recognized that much of the materials technology that was developed could well be applicable to other systems. For example, a mercury-rankine system is being developed to power an artificial heart. Investigators involved in this development are using corrosion information generated under the SNAP-8 program. Also, it is likely that some of the NaK corrosion information could be used in nuclear reactor systems for land-based power (e.g., breeder reactors).

The Ta tensile, creep, and low-cycle fatigue testing has thoroughly described this material's properties in a temperature range where previously little data were available. Also, basic designs for refractory metal/stainless steel bimetallic welds were developed; these could easily be applied to other dissimilar bimetallic systems. Various types of refractory metal/stainless steel bimetallic tubing produced by techniques developed under the SNAP-8 program could be used in heat-pipe applications. Or, with a reduced amount of refractory metal, it could be used in commercial chemical systems where the corrosion resistance of refractory metals would extend tubing life greatly. Refractory metal/stainless steel bimetallic transition joints produced by techniques developed under the SNAP-8 program are being utilized in thermoelectric modules for space power systems and could be used in other advanced power systems. Many other applications of the technology developed are, of course, possible.

SUMMARY OF RESULTS

Several major conclusions can be drawn based on the materials technology efforts summarized in this report:

1. Tantalum, niobium - 1-percent zirconium, and alloy T-111 (Ta-8W-2Hf), unlike more conventional cobalt and iron-base alloy containment materials such as L-605 and 9 chromium-1 molybdenum steel, should not be affected by liquid Hg exposure at temperatures at least up to 650° C (1200° F).

2. The sodium-potassium eutectic alloy (NaK) corrosion rates of the major SNAP-8 primary reactor loop materials Hastelloy N and 316 SS appear to be acceptably low (less

than 0.004 cm/10^4 hr or 0.0015 in./10^4 hr) at temperatures up to 700° C (1300° F) providing the oxygen level in the NaK is maintained at less than 30 ppm.

3. Tantalum having a uniformly distributed oxygen concentration of 115 ppm or less will not be attacked by NaK at temperatures up to 730° C (1350° F).

4. Uniaxial creep testing of Ta tubing at 730° C (1350° F) revealed a strong dependence of creep strength on grain size, the fine-grained tantalum being considerably more creep resistant.

5. Low-cycle fatigue testing of unalloyed Ta bar at temperatures up to 730° C (1350° F) revealed that the Ta low-cycle fatigue life at the maximum plastic strain range (0.02 cm/cm (in./in.)) to which it would be exposed in the SNAP-8 system should be in excess of 1000 cycles at temperatures up to 730° C (1350° F).

6. Several different welded joint designs using refractory metal/austenitic alloy bimetallic tubing can be successfully produced. The three basic configurations produced in this program were a straight butt joint, a tee joint, and a tube-to-header joint.

7. The most suitable refractory metal/austenitic alloy bimetallic couple for fabrication into tubing for mercury containment service at temperatures up to 730° C (1350° F) was determined to be tantalum/type 316 stainless steel (Ta/316 SS).

8. The preferred fabrication method for producing Ta/316 SS bimetallic tubing was determined to be hot coextrusion over a mandrel. The two other techniques attempted, hot filled-billet coextrusion and explosion welding, had significant deficiencies.

9. A brazed Ta/316 SS tubular bimetallic transition joint is considered to be the best candidate for SNAP-8 use at the current state of development. The other two types of joint, the hot-coextruded sleeve and tandem, require further development before they could be considered acceptable for SNAP-8 service.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 7, 1973,
501-21.

APPENDIX - PROCEDURES USED TO COEXTRUDE TANTALUM/316 STAINLESS STEEL BIMETALLIC TUBING

In order to provide the SNAP-8 system with reliable Ta/316 SS bimetallic tubing for use in the mercury boiler, it was necessary to optimize tubing fabrication techniques. A program was conducted by the Nuclear Metals Division of the Whittaker Corporation that utilized hot extrusion over a mandrel to produce the tubing, and it involved a fairly extensive study of the extrusion variables associated with this process. The other program, conducted by the Metalonics Division of the Kawecki Chemical Corporation, involved hot extrusion of a filled billet. This study was much less extensive and was basically limited to applying extrusion conditions previously developed for other applications. The purpose of this appendix is to describe these two extrusion programs since neither of them have been reported previously.

MANDREL EXTRUSION TECHNIQUE

Billet Component Preparation

After machining to the sizes indicated in figure 28, the seamless mild-steel components were outgassed at about 1010° C (1850° F) for 24 hours in a heated vacuum retort. Following cooling and removal from the retort, they were stored in plastic bags with a dehumidifying agent until required for assembly.

Similarly, the machined 316 SS sleeves were scrubbed with a detergent solution, and rinsed with tap water, distilled water, acetone, and ethanol.

The Ta sleeve was machined to the size needed for the first extrusions. It was degreased with trichloroethylene and then acetone. Then it was chemically etched in a solution composed of one volume hydrofluoric acid (49 percent assay), two volumes sulfuric acid (96 percent assay), and two volumes nitric acid (70 percent assay). This solution was used to remove a 0.0025- to 0.0051-centimeter (0.001- to 0.002-in.) thick surface layer from the Ta. The chemical etch was followed by a thorough tap water rinse, a distilled water rinse, an acetone rinse, and an alcohol rinse.

Immediately after the Ta was cleaned, the billets were assembled into the configuration shown in figure 28. First the steel end closures (one with an evacuation tube) were welded, and then the shaped nose piece was attached. After helium leak checking, the billet was ready for copper plating, outgassing, and sealoff.

The assembled billets were electroplated with copper by a standard procedure that began with a thorough degreasing of the exterior in a trichloroethylene bath. The clean

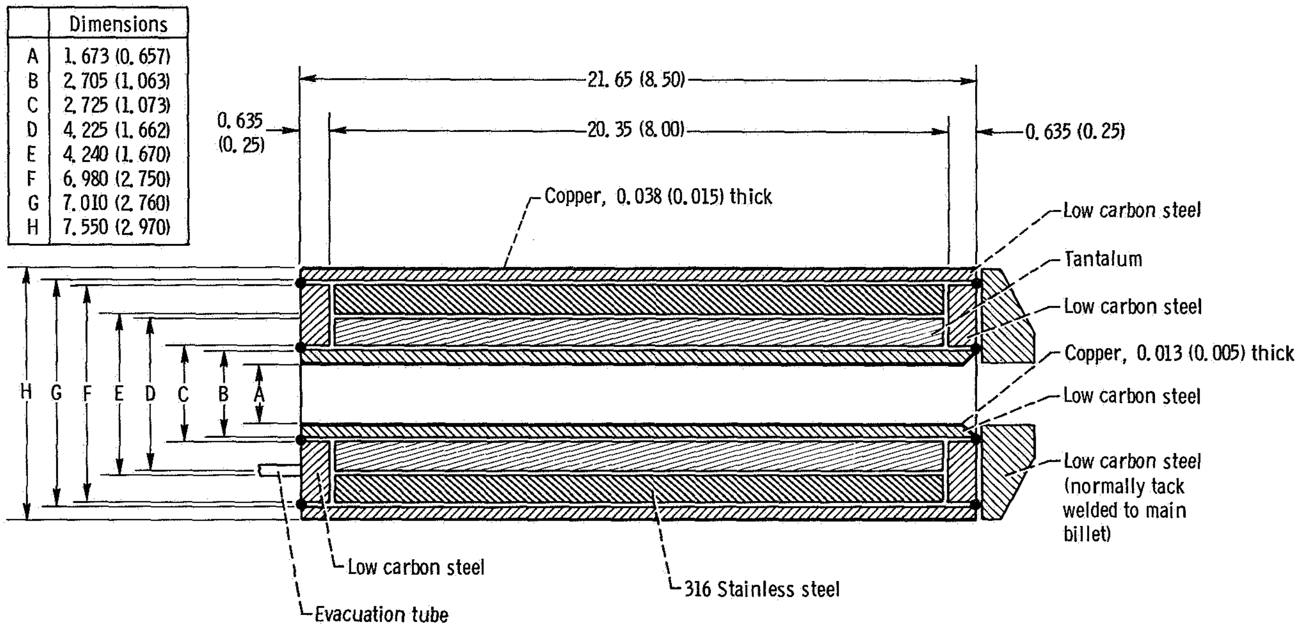


Figure 28. - Extrusion billet design for producing tantalum/316 stainless steel tubing using a mandrel extrusion method. Extrusion ratio, 25:1. (All dimensions are in cm (in.).)

billet was then dipped in a hydrochloric acid solution, rinsed in tap water, and transferred to a copper cyanide plating bath where it received a flash coating of copper. The final coating of copper, 0.038 centimeter (0.015 in.) thick on the outer surface of the tubular billet and 0.0076 to 0.0127 centimeter (0.003 to 0.005 in.) thick on the interior of the billet, was applied in an acidified copper sulfate bath. The evacuation tube was protected during plating by a wrapping of electrical tape.

The billets were attached to a vacuum system and evacuated to 1.33×10^{-2} newton per square meter (10^{-4} torr) and then slowly heated to 430° C (800° F). After at least 4 hours at 430° C (800° F) or until outgassing was completed, the billets were slowly cooled to room temperature. Sealoff was accomplished by torch heating the evacuation tube to bright red heat, then hammering it flat close to the end plate, and melting off the excess. The copper on the sealed-off billets was abraded to remove oxide formed during outgassing, washed with trichloroethylene, then spray-coated with a dry graphite film lubricant, after which the billets were ready for loading into the furnace.

Extrusion Procedures

The tools, 18-4-1 steel (hardened to Rockwell C 58 to 60 for the backers and mandrels) and H-21 or M-2 steel for the dies, were degreased with trichloroethylene, then heated in 480° C (900° F) furnaces for several hours until coated with an oxide film.

They were then cooled and sprayed with a graphite lubricant. In addition, the dies were coated with Necrolene. The liner was wire brushed and blown free of any debris.

The billets were heated in stainless steel retorts flooded with flowing argon. The retorts containing the billets were heated for a minimum of $3\frac{1}{2}$ hours in a resistance furnace.

Extrusions were made in a 1.25-meganewton (1400-ton) hydropress, using a 7.72-centimeter (3.050-in.) inside diameter liner. The maximum force available to the liner was 6.8 meganewtons (770 tons).

Overheating and softening of mandrels was minimized by attaching them to the stem, which was attached to the main ram of the press. A roll pin was generally adequate to maintain proper alinement of the mandrel. Further assurance of proper alinement was provided by a graphite sleeve, which tightly fit both the stem and the mandrel backer.

The sequence of operations was as follows:

(1) Attach mandrel to stem.

(2) Insert die in liner 7 to 10 minutes before extruding.

(3) Lubricate mandrel and liner.

(4) Remove billet from furnace and hand load until nose enters liner.

(5) Extrude at a reduction ratio of 32:1.

Several tubes were extruded over the temperature range 940° to 1090° C (1725° to 2000° F). Extrusion speeds varied over the range 2.5 to 12.7 meters per minute (100 to 500 in./min). A basic process was demonstrated in that tubing was successfully extruded nearly to the specified dimensions. Based on the tube surface condition, the optimum extrusion temperature was determined to be 995° C (1825° F). The extrusion speed selected was 7.6 to 12.7 meters per minute (300 to 500 in./min).

Initial Results

Samples of the tubing subjected to microscopic study generally revealed a bondline layer less than 5×10^{-5} centimeter (2×10^{-5} in.) thick, but with protuberances as thick as 1×10^{-4} centimeter (4×10^{-5} in.).

When split lenghtwise, 5.1-centimeter (2-in.) long samples removed from the middles and the ends of several tubes revealed that the Ta surface was smooth and apparently defect free. A more complete examination of these and later tubes with a borescope revealed surface defects that appeared to be incipient tears. These appeared to be random in location in the tube, but they were alined longitudinally quite frequently. Although these tears appeared to be only about 0.005 centimeter (0.002 in.) deep, they were considered to be symptomatic of a potentially serious deficiency in the basic process. A detailed investigation into the causes of these tears was therefore conducted.

Process Improvement

The random occurrence of relatively localized defects suggested some defect in the materials composing the billet rather than in the extrusion technique. The Ta, which was rather large grained, was especially suspect. Unfortunately, all of the Ta stock was procured at the start of the program, hence, that parameter could not be varied. The tears could, however, also have been attributed to many other process variables and these were investigated.

Honing of the Ta and heavy etching was first tried, but this produced no improvement. Then, the basic mismatch in stiffness of the components was checked by extruding sheathed rods of Ta in tandem with stainless steel and comparing the extrusion constants. No serious mismatch problems were apparent between the materials used in the first billets; moreover, it did not appear possible to obtain a better match by adjusting the temperature within the range of extrusion temperatures available.

Stiffness of the inner steel extrusion sheath was investigated by varying three parameters. The first change from the basic process was to increase the thickness of the sheath to offset chilling from contact with the cold mandrel and to thereby provide less of a stiffness gradient through the sheath to the tantalum. Thick sheathing also provided assurance that the sheath was not tearing because of being extruded too thin. Secondly, a warm mandrel was used to further counteract chilling of the sheath. A third variation involved changing to a sheath material with a different stiffness than the low carbon steel.

The first trial extrusion showed that roughly double the inner sheath thickness was not beneficial. The next extrusion indicated that a double sheath thickness with a warm mandrel might be a step in the right direction. Continued improvement of the inside-diameter surface of succeeding extrusions occurred as the inner sheath was increased in thickness. Other attempts to adjust the stiffness of the inner sheath by using materials other than low carbon steel or by increasing the thickness of the copper outside of the steel resulted in no marked improvement. Unsuccessfully used as inner sheath materials were copper, copper - 30-percent nickel, and Monel.

Lowering the extrusion reduction, which frequently alleviates tearing problems, was explored as another alternative. This appeared to result in a better Ta surface.

Other changes were also incorporated into the process. The first billets were heated on their sides in argon-flooded stainless steel retorts. In this position, forced contact of billet components might permit localized interaction of the components, and this could conceivably produce embrittlement of the Ta surface, which would then tear while being extruded. Opportunity for such interaction was minimized by heating subsequent billets vertically (standing on their noses) either inside graphite cans or directly exposed to the nitrogen furnace atmosphere.

All but one vertically heated tube appeared to be tear-free. Therefore, although the conclusion that vertical heating prevented tears could not be drawn, it was decided to include it in the process. Heating inside graphite cans or directly in nitrogen did not appear to make a difference in the quality of the extrusions.

The results of this study led to the following conclusions:

(1) Changing temperatures in the range 940° to 1090° C (1725° to 2000° F) with relatively thin inner steel extrusion sheaths did not appear to prevent tears in the Ta.

(2) Softer (all copper) or stiffer (Monel) inner extrusion sheaths did not produce a good Ta surface.

(3) The effect of various inner sheathing materials merits further investigation, although both copper (a soft material) and Monel (a stiff material) permitted surface tears. One possibility is the use of 316 SS for the inner sheath. This could be readily dissolved by aqua regia without affecting the external 316 SS.

(4) The use of a warm mandrel in combination with a low reduction ratio and a thick steel inner sleeve (0.51 cm (0.2 in.) wall) minimized or eliminated tearing.

Postextrusion Processing

Some of the early tubes were stretch-straightened with less than 1.5 percent permanent strain. The straightening was done on a hydraulic draw bench equipped with two sets of grips, one on the ram and one attached to the rear of the bench. The ends of the tube were fitted with steel plugs to prevent the tube from collapsing. It was found that a 1.2- to 1.5-percent permanent strain was adequate to straighten the tubes.

Removal of the bulk of the extrusion jacket was accomplished by use of a nitric acid solution. Concentrations of 30 to 50 volume percent of nitric acid (70 percent assay) with water were suitable. Frequently, pinhead-size pieces of steel became passivated. These were attacked by numerous methods such as heating the nitric acid solution to boiling. Hydrochloric acid appeared to be capable of removing the traces of steel if given enough time. Aqua regia seemed to perform most rapidly; however, there was some indication that the steel could also become passivated to this. All of the methods for removing the steel were intermingled for most of the tubing made in this program. The tubes were subjected to successive treatments until nothing that could be suspected of being steel could be seen on the Ta. Further study of techniques for the removal of passivated steel in such tubes is desirable.

A final chemical polish of the Ta was obtained using a solution of the same composition as that used to prepare the Ta billet components for extrusion. About 10 minutes in this solution at 40° C (110° F) cleaned the tubes and appeared to remove a layer of Ta about 0.0025 centimeter (0.001 in.) thick. This procedure also needs improvement. More uniform attack would probably occur if the solution were slowly circulated through the tube by an air lift or by an acid pump. During the program the tubes were rolled and occasionally partially drained and then refilled by raising and lowering their ends.

The chemical polish was completed by transferring the tubes to a cold water rinse, followed by a hot water rinse. The tubes were then raised to a vertical position to drain. Acetone poured through the tube hastened the drying.

The straightened tubes were centerless polished on an abrasive-belt, centerless polishing machine. A 10.2-centimeter (4-in.) wide, 320-grit silicon carbide belt was adequate to remove about a 0.0025- to 0.0076-centimeter (0.001- to 0.003-in.) thick surface layer in one pass and leave a 16 rms surface finish. A water based coolant was used. A 4.6-meter (15-ft) tube required about 10 minutes to pass through the machine.

Final Processing Conditions

The development program resulted in procedures that could produce straight tubing with the tantalum - stainless steel well welded and with polished stainless steel surfaces. The surface of the Ta was not as smooth as desired, but no tears were observable. Since both a heavy inner steel extrusion sheath and a lower reduction produced tear-free Ta, these two techniques were combined to form the basis of the subsequent processing.

In order to verify the final process, a trial batch of three tubes was made and the interiors were carefully examined for tears. About 3.7 meters (12 ft) of one tube was split lengthwise to preclude any confusion due to borescope inspection. All tubes were tear-free; some heavy ripples were apparent near the ends of two of the tubes and near the middle of the third tube.

The quality of the three tubes was considered an adequate verification of the extrusion process so that the remaining stock could be extruded. The ripples in the Ta surface indicated that the process was not, however, fully optimized.

The billets for the final tubing utilized a 0.51-centimeter (0.2-in.) thick inner steel extrusion sheath. The billets were extruded at 995° C (1825° F) at a reduction ratio of 25:1 and an extrusion speed of 12.7 meters per minute (500 in./min). The tool steel mandrels were heated to 480° C (900° F).

After straightening, removal of the extrusion jacket by pickling, and chemical and mechanical polishing, the tubes were given a final inspection and found to be acceptable.

FILLED-BILLET EXTRUSION TECHNIQUE

All components were machined to the billet design shown in figure 29. No machining of the outside diameter of the stainless was necessary. Following all the machining operations, each steel component was vapor degreased with trichloroethylene and then degassed at 1065° C (1950° F) in a vacuum of 1.33×10^{-2} newton per square

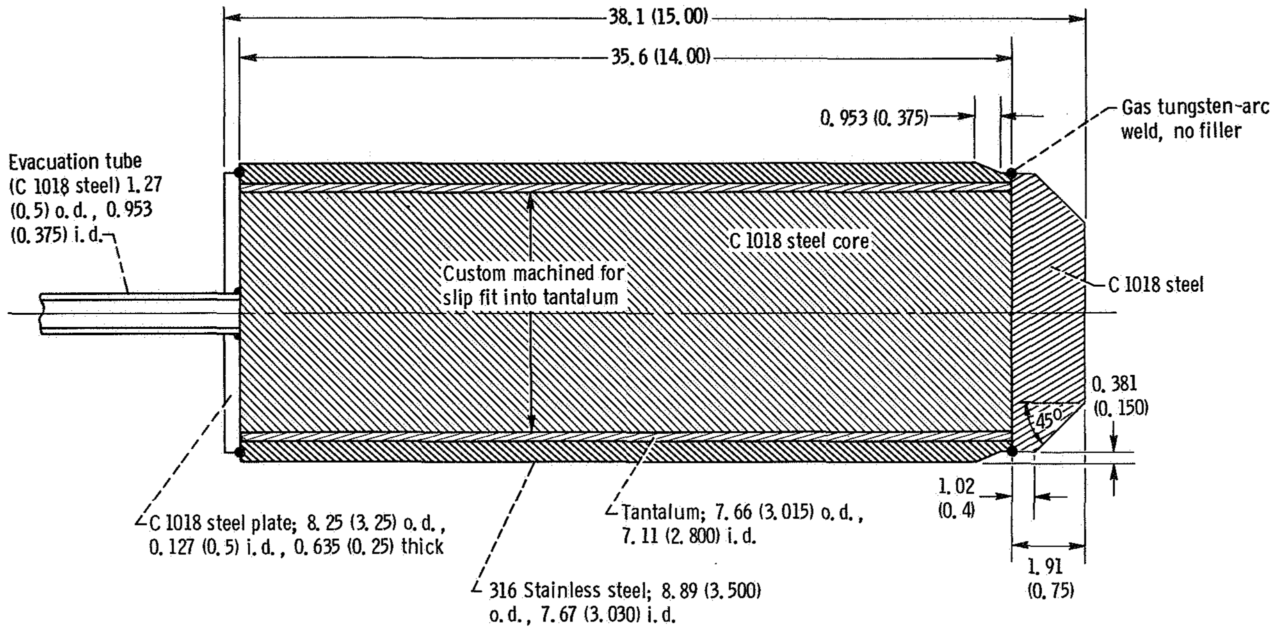


Figure 29. - Extrusion billet design for producing tantalum/316 stainless steel tubing using a filled billet method. (All dimensions are in cm (in.).)

meter (10^{-4} torr). After the vacuum degassing operation, each part was cleaned in acetone and assembled. Careful attention was paid not to get contaminants on any of the pieces before assembly. The unit was then welded together. Sealing was performed at 540° C (1000° F) in a vacuum of 1.33×10^{-4} newton per square meter (10^{-6} torr).

After evacuation and sealing, the billets were inserted into a furnace which was at the prescribed extrusion temperature. The furnace door was sealed, and argon was pumped into the furnace. The heating time was approximately $2\frac{1}{2}$ hours. Graphite cutoffs were inserted into the furnace with the billets. The billets were then extruded at 1.0 meter per minute (40 in./min) through zirconium oxide coated die (2.1 cm (0.0830 in.) diam) at a 45° approach angle. The force required to start the push was 5.9 meganewtons (660 tons) rising to a peak of 6.8 meganewtons (770 tons). A 9.1-centimeter (3.600-in.) liner, and a graphite lubricant were used. The billet extrusion temperatures ranged between 990° and 1060° C (1810° and 1940° F) although most were extruded at 1030° C (1880° F). Use of the higher extrusion temperature appeared to result in an overall better surface finish.

After extrusion, the rods were straightened while they were still hot and allowed to cool in air to room temperature. The ends were trimmed to leave approximately 4.9-meter (16-ft) lengths, and the cores were removed. Core removal was accomplished by nitric acid leaching at approximately 90° C (200° F). The acid was pumped into the tubes.

The outside diameter was belt ground to a 2.07-centimeter (0.815-in.) diameter following a cold hand straightening operation. The inside diameter was sized by pulling a torpedo type of mandrel through the tube. About four to five passes were necessary to achieve the size required. Only protruding hills of Ta (0.0127 to 0.0203 cm (0.005 to 0.008 in.)) on the inside diameter were displaced. The tubes were thoroughly vapor degreased following the sizing operations.

The oxide formed in the core removal operation was removed from the inside surface by a honing operation. Specifically, 80-mesh aluminum oxide was blown through the tube using tank nitrogen.

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