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LINING METAL TUBES BY CREEP FORMING

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16. Abstract

A method of forming and bonding a metallic lining onto the inside surface of a metal tube is described and examples are given. The method uses the biaxial creep of an internally pressurized thin-wall tube as a means of applying force at high temperature to several layers of a thin metallic foil sandwiched between the inner tube and an outer metal tube. After bonding, the inner tube material is removed by either chemical or mechanical means.

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SUMMARY

A method of forming and bonding a metallic lining to the inside of a metal tube is described and examples are given. The method uses the biaxial creep of an internally pressurized thin-wall tube as a means of applying force at high temperature to several layers of a thin metallic foil. The foil layers are compressed together and against the inside surface of a concentric outer tube that encompasses the inner tube and foil. The lining may be bonded directly to the metal tube or it may be further processed into a free standing tube for use as a loose lining.

This method was developed for the specific purpose of bonding a thin layer of tungsten to the inside of a tantalum alloy (T-111) tube. The tungsten lining separates two incompatible materials that compose a fuel pin for use in liquid metal cooled nuclear reactors. Operating conditions for performing this specific process are given and evidence of the results are presented.

INTRODUCTION

The development of nuclear fuel pins for space power application is very dependent on material selection. The clad material is usually selected on the basis of mechanical properties while the fuel material selection is based on nuclear properties. Frequently the two materials are not chemically compatible and a physical barrier in the form of a lining is required to separate them. The lining may be loose or it may be bonded to one or both of the adjacent materials. Shape and physical and chemical properties of the materials are the important factors that determine how the lining can be formed. Some of the more common mechanical forming methods for metals include rolling, extrusion, spinning, and pressing while electrolysis and vapor deposition would represent chemical forming.

The method described herein uses biaxial creep of an internally pressurized thin-wall tube as a means of applying force at high temperature to form and bond a metallic lining to the inside of a metal tube. It was developed for the specific purpose of bonding a thin layer of tungsten to the inside surface of a tantalum alloy (T-111) tube. The tungsten lining separates two incompatible materials, T-111 and uranium nitride, which compose a fuel pin being investigated for space power nuclear reactor applications. Tungsten, in the form of several layers of a thin foil, is bonded to the inside of the T-111 tube by creeping a third material, in the form of a small inner tube, diametrically outward until it contacts and compresses the tungsten against the T-111. The combination of compression, temperature, and time bonds the tungsten layers together and to the T-111. The inner tube material is then removed by either chemical or mechanical means. This report discusses the procedures and describes the techniques and operating conditions necessary to form this type of lining.

SYMBOLS

A	constant, $hr^{-1}(Nm^{-2})^{-n}$
ΔH	activation energy for creep, $(J)(g\text{-mole}^{-1})$
n	stress-dependency constant
R	gas constant 8.3143 $(J)(g\text{-mole}^{-1})(K^{-1})$
T	absolute temperature, K
$\dot{\epsilon}$	creep rate, hr^{-1}
σ	stress, Nm^{-2}

CREEP FORMING PROCESS

The sequence of forming a lining onto the inside surface of a tube is (1) selection of the inner tube material, (2) fabrication and assembly, (3) creeping, (4) bonding, and (5) separation. The requirements for the inner tube material include a relatively low creep strength and availability in the form of thin-wall tubing of suitable dimensions. It should also be fabricable so that closures can be machined and welded.

The creep phase utilizes the biaxial creep characteristics of the internally pressurized thin-wall tube to close the diametral assembly gap and compress the liner against the outer tube. The operating conditions for this phase can be determined once the material and dimensions have been specified. Then, by choosing a creep rate that will close the gap within an acceptable period of time, the temperature and stress may be estimated from the creep properties of the material. The stress will usually be restricted by the available pressure at the test facility and therefore temperature is usually the controlling variable.

Since practically all creep data represent uniaxial conditions, it is necessary to convert the required tangential stress and strain rate of the thin-wall tube to a uniaxial stress and strain based on von Mises criterion for plastic flow (ref. 1). This relation is

$$\sigma_{tangential} = \frac{2}{\sqrt{3}} \sigma_{uniaxial}$$

$$\dot{\epsilon}_{tangential} = \frac{\sqrt{3}}{2} \dot{\epsilon}_{uniaxial}$$

Once the stress and strain rate are expressed in uniaxial terms the temperature may be estimated from creep data for the inner tube material. A convenient form for creep data is given in references 1 and 2 in which the correlating equation

$$\dot{\epsilon} = A\sigma^n \exp\left(\frac{-\Delta H}{RT}\right)$$

is used in conjunction with a statistical analysis of the data.

Included are graphical presentations of data for some possible inner tube materials. This type of correlation gives a range of temperatures that could produce the desired creep rate. The choice of temperature could then be made on the basis of achieving the required diametral strain within some reasonable time period. After determining this temperature, the stress should be checked to determine whether or not it is above the yield point. As a general rule, the tangential stress should be maintained below that required for yielding until the inner tube walls are restrained by the outer tube. This condition ensures that the type of plastic flow will be primarily creep rather than rapid yielding which might produce local premature failure.

Following the creep part of the procedure, the operating conditions may be altered to enhance the bonding mechanism. The internal pressure should be maximized because this brings the metal surfaces more closely together. This pressure, of course, will be limited by the weakest part of the total assembly. Vunerable parts may include the end caps, welds, fill tube, and outer tube. Once the surfaces are in intimate contact and assuming they are relatively free of contaminants, the temperature will determine the type of bond that will result. If the temperature is high enough to cause significant diffusion of the metal atoms, solid state welding will occur. Lower temperatures will result in adhesive type bonds. Time at temperature is also very important, but because of the complexity of the bonding mechanism it can best be determined empirically. Times between 1 hour and 1 day appear reasonable.

Separation of the inner tube from the lined outer tube depends on the materials. If the inner tube becomes bonded to the lining material, selective chemical dissolution may be used. An inert coating may be applied to the inner tube to prevent bonding. Differences in thermal expansion of the materials may also be used as a method of separation.

APPARATUS AND PROCEDURE

Facility

A schematic of the facility is shown in figure 1. It consists of a high-temperature furnace capable of operating in either a vacuum or an inert gas environment, a gas pressurization system, and controls for maintaining temperature and pressure. The test assembly is suspended inside the furnace through the top cover plate, and the tubes are connected to the gas pressurization system. A detailed description of this facility is given in reference 1.

Procedure

The creep forming procedure described will be that used to apply a thin multilayered tungsten lining to the inside surface of a T-111 tube. Generalizing this procedure should make it applicable to other materials and dimensions. This, in fact, was done and

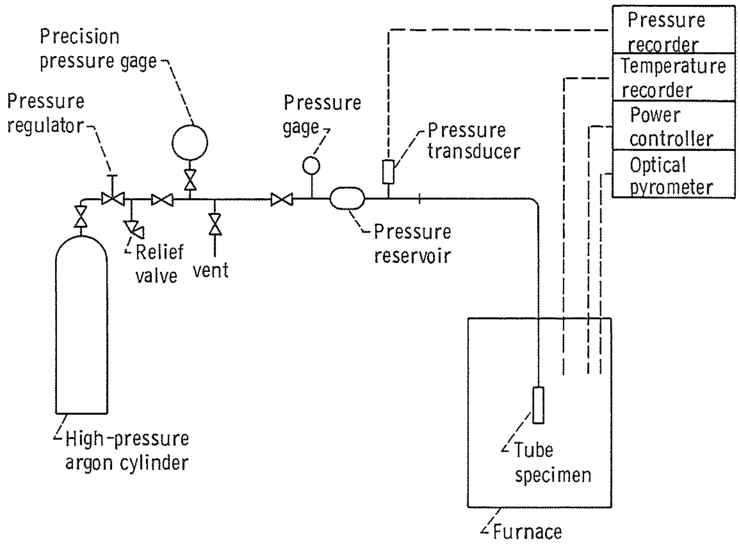

Figure 1. - Creep forming facility.

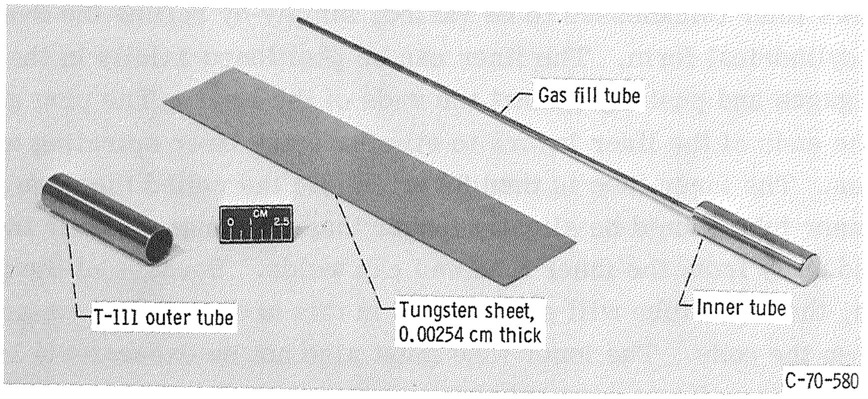

Figure 2. - Creep forming components.

examples are given in the RESULTS AND DISCUSSION section.

Typical creep forming components, shown in figure 2, are a T-111 tube, a sheet of 0.00254 centimeter (0.001 in.) thick tungsten foil, and a molybdenum inner tube. The inner tube was fabricated from a length of seamless tubing. End caps were electron beam welded onto the tubing. A 0.318 centimeter (1/8 in.) outside diameter with 0.0635 centimeter (0.025 in.) wall fill tube for pressurization was electron beam welded to one end cap. The T-111 tube was open on the ends and had a 32 rms finish on the inside surface. The tungsten sheet served as the liner. The tungsten was sheared to the proper size, and both sides were lightly sanded to remove oxidation. Each component was cleaned before assembly. The tungsten foil and the T-111 cylinder were immersed in an acid solution of 1/3 H_2O, 1/3 HNO_3, and 1/3 H_2SO_4 by volume for 10 minutes and rinsed in distilled water. The molybdenum inner tube was cleaned first in acetone then rinsed in ethyl alcohol. The assembly of the components is seen in figure 3. Clean

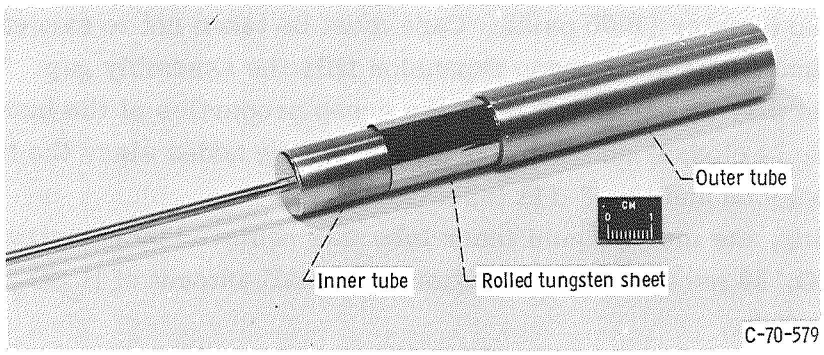

Figure 3. - Assembly of components.

nylon gloves were used to roll the tungsten and insert it in the T-111 tube. Using tungsten sheet allows liner thicknesses to be varied, simply by rolling the desired number of layers into cylindrical form. The liner can be positioned axially in the T-111 tube by inserting plug gages and pushing against the ends of the liner. The plug gage will also tend to aline the ends of the liner layers to eliminate any liner spiraling which occurred during insertion. The inner tube is then inserted into the rolled tungsten sheet. The length of the inner tube should be slightly longer than the tungsten liner to allow the liner to be positioned away from the inner tube end cap welds. Because of the added support near the welds, the inner tube will expand less in this area and thus cause the liner to be poorly bonded on the ends. The inner tube must also not be excessively longer than the liner or end ballooning effects may cause premature failure or make inner tube removal difficult.

The clearance required for assembly varies with tube diameter. It is desirable to use a minimum clearance to limit the amount of inner tube creeping required to press the tungsten liner against the walls of the T-111 tube. Enough clearance must be maintained, however, to provide ease of assembly. For a tube diameter between 1.27 and 1.91 centimeters (1/2 and 3/4 in.), an assembly clearance of 0.0127 to 0.0178 centimeter (0.005 to 0.007 in.) is sufficient.

After assembly the T-111 cylinder with tungsten foil and inner tube in position is suspended in a furnace. Reference 1 gives a detailed description of this procedure. The 0.318 centimeter (1/8 in.) diameter fill tube extending from the inner tube is inserted through the furnace plate by means of a tube fitting providing outside access for pressurization. Both the tube and the furnace are purged several times with high purity argon. The high purity argon also serves as the furnace atmosphere during operation. Pressure in the furnace is slightly greater than 1 atmosphere. The furnace temperature is raised to the desired level after purging and back filling the furnace. Operating temperature is predetermined by the creep properties of the inner tube and the type of bond desired. The inner tube is pressurized after the furnace is up to temperature. Pressurization was in ten equal increments up to a maximum final pressure of 6.89×10^{6} newtons per square meter (1000 psia). Care must be taken not to exceed the yield strength of the inner tube until creep expansion fills the assembly gap. The time and pressure for this may be estimated from the creep properties of the inner tube. When the assembly gap is closed, additional pressure can be added since the inner tube is backed by the tungsten and the T-111 tube.

After forming, the molybdenum inner tube was removed by chemical dissolution with a 50 percent H_2O, 50 percent HNO_3 solution. A small amount of H_2SO_4 was added to catalyze the reaction.

If the inner tube material is chosen so there is sufficient differential expansion between it and the outer tube, the leaching step can be eliminated. A steel tube used with a T-111 outer tube can be easily removed mechanically after cooldown. The steel inner tube, however, will adhere to the tungsten liner. A coating of high purity alumina oxide or an additional layer of tungsten covering the inner tube eliminated the problem.

A free standing tungsten cylinder can also be creep formed. A procedure like that outlined previously is followed with the exception that the T-111 outer tube is replaced with a molybdenum outer tube. After creep forming and the inner and outer molybdenum is dissolved, only the layered tungsten cylinder remains.

RESULTS AND DISCUSSION

The operating conditions for creep forming various linings are presented in table I. Figures 4 and 5 illustrate the different sizes and lengths that were processed and tested, indicating no length to diameter effect. Some of the problems associated with the creep, bonding, and separation phases of this process are now described.

TABLE I. - OPERATING CONDITIONS FOR CREEP FORMING

Assembly details	Test							
	1	2	3	4	5	6	7	8
Outer tube:								
Material	T-111	T-111	T-111	Molybdenum	Molybdenum	Molybdenum	Molybdenum	T-111
Outside diameter, cm	1.91	1.91	1.91	1.91	2.54	2.54	2.54	1.91
Wall thickness, cm	0.102	0.102	0.102	0.102	0.279	0.279	0.279	0.157
Inner tube:								
Material	Molybdenum	TZM	TZM	TZM	316 SS	316 SS	316 SS	316 SS
Outside diameter, cm	1.59	1.59	1.59	1.59	1.91	1.91	1.91	1.54
Wall thickness, cm	0.102	0.102	0.051	0.051	0.089	0.089	0.089	0.089
Lining:								
Material	Tungsten	Tungsten	Tungsten	Tungsten	Tungsten	Tungsten	Tungsten	Tungsten
Thickness, cm	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025	0.0025
Length by width, cm	5.1 by 32.3	5.1 by 26.9	5.1 by 26.9	1.3 by 16.3	5.1 by 31.5	5.1 by 31.5	5.1 by 31.5	15.2 by 31.5
Number of layers	6	5	5	3	5	5	5	5
Clearance, cm	0.041	0.046	0.046	0.051	0.025	0.025	0.025	0.015
Creep process:								
Temperature, K	1922	1811	1811	1811	1144	1144	1172	1172
Internal pressure, N/m^2	1.03×10^6	7.00×10^6	3.45×10^6	3.45×10^6	6.90×10^6	6.90×10^6	6.90×10^6	6.90×10^6
Tangential stress, N/m^2	7.58×10^6	51.0×10^6	52.40×10^6	52.40×10^6	70.33×10^6	70.33×10^6	70.33×10^6	55.64×10^6
Diametral creep rate, hr^{-1}	2.4×10^{-3}	6.6×10^{-3}	7.6×10^{-3}	7.6×10^{-3}	2.0×10^{-2}	2.0×10^{-2}	2.0×10^{-2}	5.0×10^{-3}
Time, hr	23	9	8	9	2	2	2	3
Bonding process:								
Temperature, K	1922	1922	1811	1811	1144	1144	1172	1172
Internal pressure, N/m^2	2.07×10^6	7.00×10^6	3.45×10^6	3.45×10^6	6.90×10^6	6.90×10^6	6.90×10^6	6.90×10^6
Time, hr	15	41	28	27	3	3	1/4	1
Separation process - method	Chemical	Chemical	Chemical	Chemical	Mechanical	Mechanical	Mechanical	Mechanical

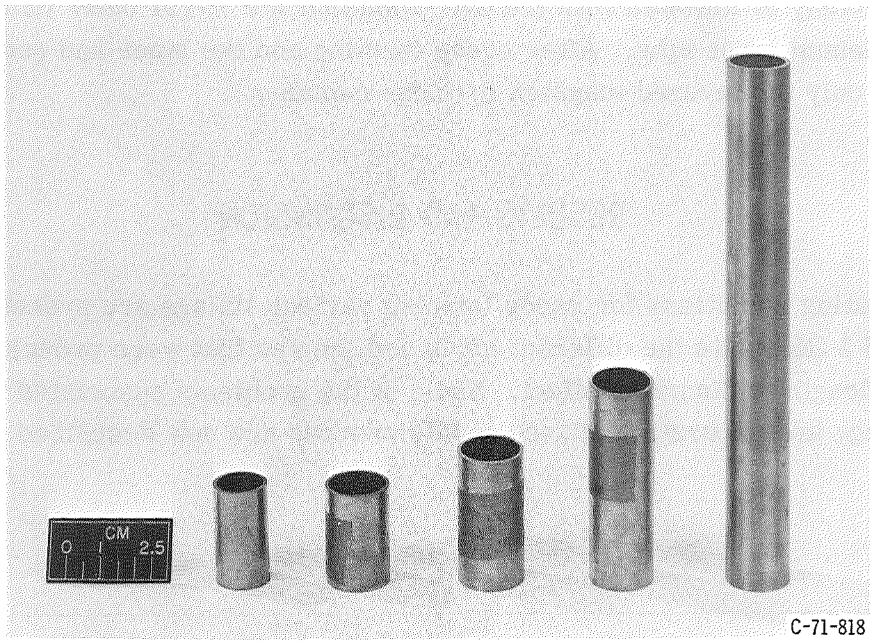

Figure 4. - Various sizes of lined tubes.

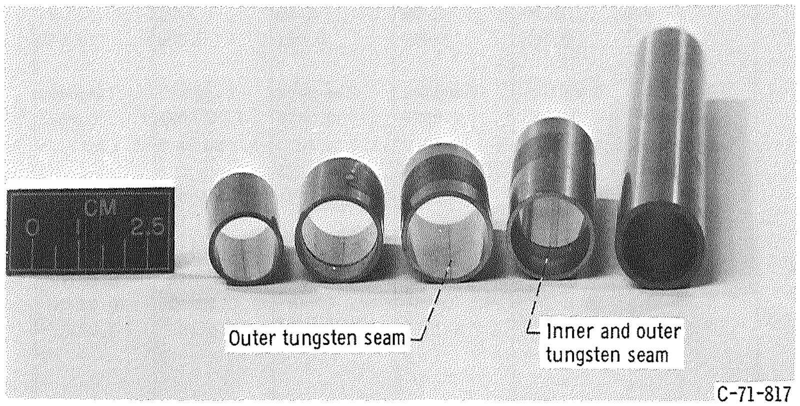

Figure 5. - Lined tubes showing liner seam.

Creep

Although the temperature and stress for creep can be calculated and controlled, the time required to complete the creep phase, reported in table I, is an estimate that depends on the accuracy of the available creep data. Variations of factors of two or more are entirely possible. This can be resolved by either extending the calculated creep time to include the uncertainty factor or by creep testing a representative sample of the inner tube and experimentally determining its biaxial creep properties.

A problem area that was encountered in some of the tests was premature failure of the inner tube assembly. One source of failure was in the end cap welds. These welds should be as strong or stronger than the tube walls. Failures were attributed to poor penetration, thinning of the tube walls adjacent to the weld, and improper postweld heat treatment of the heat affected zone. A second source of premature failure was the tube walls. Inner tube wall thickness was the primary variable in these tests. Wall thicknesses in the 0.0254 to 0.0508 centimeter (0.010 to 0.020 in.) range generally resulted in premature creep failure, probably because it is more difficult to maintain good dimensional tolerances and produce good welds with the thinner wall tubes. Tube wall thicknesses of 0.0762 to 0.1016 centimeter (0.030 to 0.040 in.) generally produce good, reproducible creep results and fewer weld failures.

Clearance was also an important factor because it determined the strain required of the inner tube. As a general rule, strains greater than 10 percent resulted in creep failure while strains less than 5 percent were usually successful.

Bonding

The time at which the creep phase is completed and the bonding phase begins cannot be accurately determined because of the uncertainty in the creep data. The bonding times reported in table I may include creep time and vice-versa. Therefore, experimental results are the only reliable means of determining the degree of bonding. The photomicrographs of figures 6 to 8 illustrate the degree of bonding that was achieved. Figure 6 shows the bond achieved by test 1 conditions. The tungsten layers are clearly visible and there is little, if any, grain growth between layers. Figure 7, representing test 2, shows the effect of increasing pressure and time. The separation between layers is less and there is considerable grain growth across tungsten layers. Figure 8 represents test 3 and shows the effect of reducing temperature, pressure, and time compared to test 2. The separation between tungsten layers is slightly greater but there is still grain growth across layers. In all of these tests there was no evidence of diffusion between the tungsten and the T-111. No photomicrographs of the low temperature tests

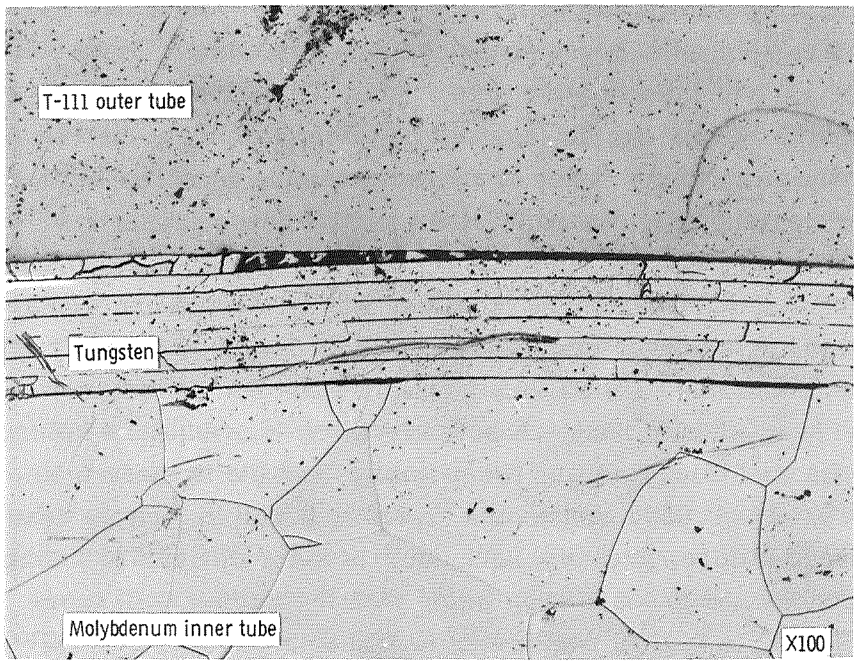

Figure 6. - Microstructure of specimen from creep-forming test 1. Etchant; Murakami's reagent.

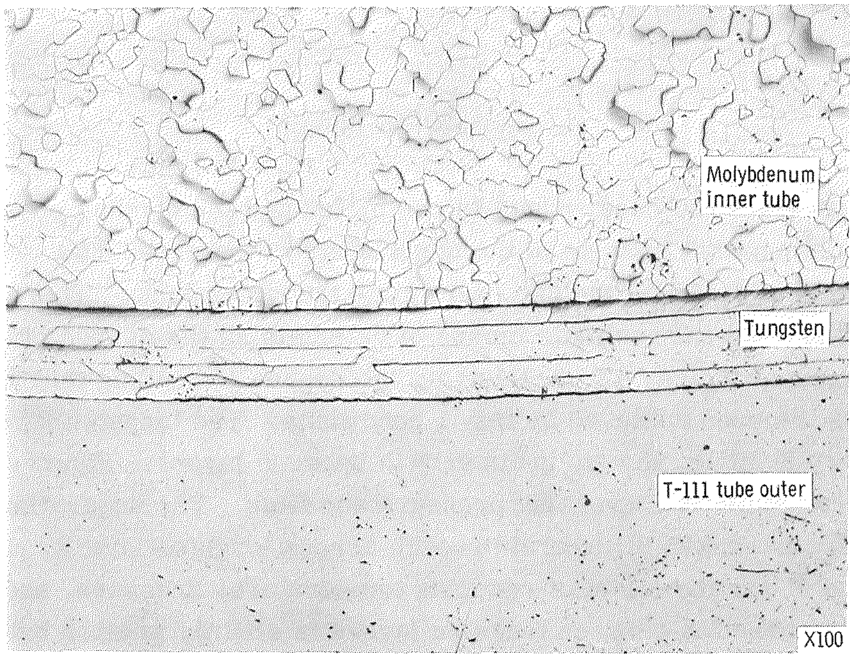

Figure 7. - Microstructure of specimen from creep-forming test 2. Etchant; Murakami's reagent.

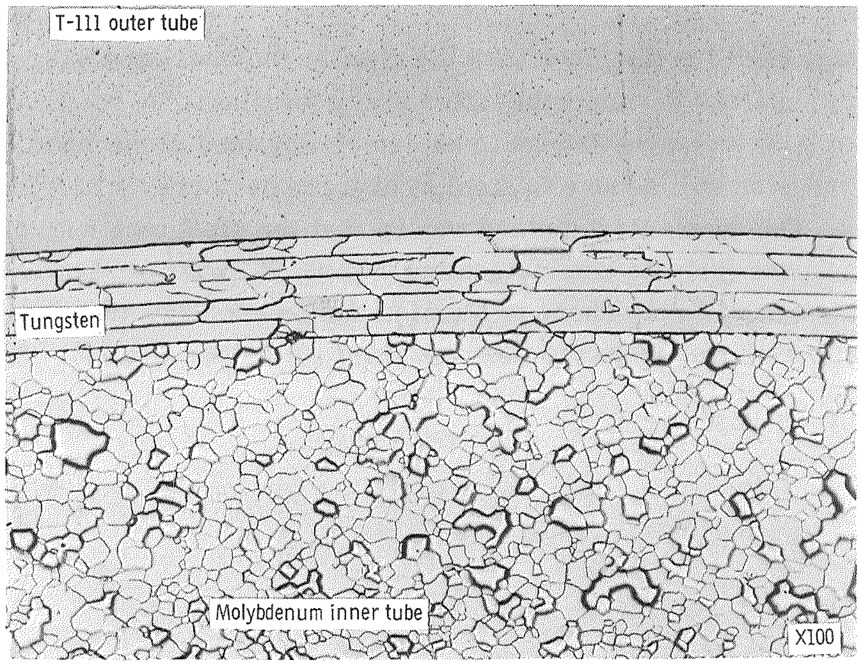

Figure 8. - Microstructure of specimen from creep-forming test 3. Etchant; Murakami's reagent.

(5 to 8) were taken because a satisfactory metallographic specimen could not be prepared without the inner tube to serve as a backing material. However, there would be no tungsten grain growth at the lower temperatures and therefore figure 6 would be representative of these tests.

In all tests the tungsten was firmly attached to the outer tube and there was no indication of looseness or blistering. Measurements made on the inside diameter of the tungsten layers showed that there was no measurable clearance between layers or the outer tube. In fact, the compression was such that the tungsten seam is clearly visible in several tests (fig. 5).

Samples from tests 1, 2, and 3 were thermal-cycled 25 times between room temperature and 1922 K (3000° F) to determine any detrimental effects on the tungsten lining. No changes in appearance or microstructure was evident.

Separation

Removal of the inner cylinder material was accomplished by both chemical and mechanical methods. For the molybdenum and molybdenum alloy (TZM) materials a solution of nitric and sulfuric acids was used to dissolve the inner tube away from the chemically inert tungsten and T-111. This reaction required about 30 minutes to complete.

When steel was used for the inner tube the greater thermal expansion of this material was used to separate the assembly after creep forming. On cooling to room temperature the steel tube contracts enough to be completely free of the lined outer cylinder. However, since tungsten will adhere to steel at high temperature, a barrier between the two must be used. One successful method was to coat the steel tube with alumina by a flame spraying process (private communication with G. K. Watson, Lewis Research Center). The tungsten will not adhere to alumina and on cooldown the alumina coated inner tube is easily removed from the tungsten lined outer tube (fig. 9). A second

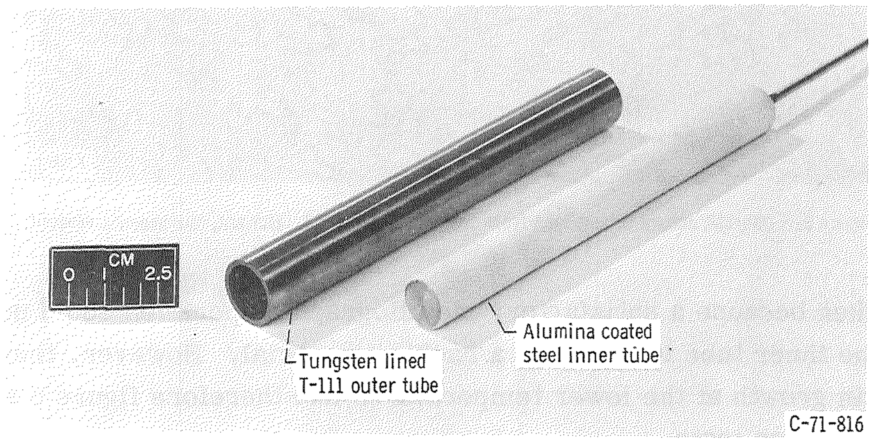

Figure 9. - Alumina (Al_2O_3) coated steel inner tube with 6 inch (15.2-cm) lined tube.

successful method used a single complete wrap of tungsten foil positioned adjacent to the steel in addition to the normal multilayered wrap for bonding. On cooldown the greater adherence of tungsten to steel will pull the single tungsten wrap away from the multilayered tungsten wrap that adheres to itself and to the outer cylinder. The breakaway point is between the first inner tungsten interface since the shearing forces caused by the contracting steel are greatest at this point.

The free standing lining shown in figure 10 was made by conditions similar to test 4 and then the molybdenum outer tube was dissolved away with an acid solution. This lining was flexible and could be handled without undue fear of breaking or cracking.

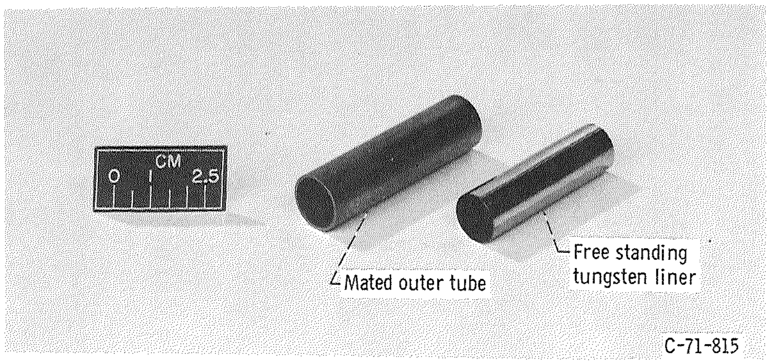

Figure 10. - Free standing liner with mating tube.

SUMMARY OF RESULTS

A method of creep forming a metallic lining onto the inside of a metal tube has been described. Specific examples and operating conditions for lining a tantalum alloy (T-111) tube with several layers of tungsten foil were presented. The process consists of three phases (i.e., creep, bonding, and separation). The operating conditions for the creep phase can be determined from the biaxial creep properties of the inner tube material. Bonding is achieved by maximizing temperature and pressure for a given time, but the complexity of the bonding mechanism requires that the operating conditions be determined experimentally. Separation of the inner tube material from the lined outer tube has been accomplished by both chemical and mechanical methods. The linings achieved by this process were firmly attached with no measurable clearance between layers and/or the outer tube.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 14, 1971,
120-27.

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