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BURST TESTING OF TUNGSTEN TUBING AT TEMPERATURES FROM 3000° TO 4500° F (1650° TO 2480° C)

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

ABSTRACT

Thin-walled tungsten tubing, 3/8 inch (9.5 mm) or 1/2 inch (12.7 mm) in diameter, produced by three extrusion techniques, by two vapor-deposition processes, or by electroforming in a molten fluoride bath were internally pressurized to failure at temperatures from 3000° to 4500° F (1650° to 2480° C). The burst strength of the majority of the tungsten tubes was equal to or greater than the ultimate tensile strength of wrought tungsten. Test results showed that the tungsten hexachloride vapor-deposition process is capable of producing tubing as strong as wrought tungsten tubing. The failure propagation was intergranular in all tungsten tubes tested.

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SUMMARY

A test method was developed for determining the burst strength of 3/8-inch- (9.5-mm-) diameter and 1/2-inch- (12.7-mm-) diameter, thin-walled tungsten tubing. The method was used to evaluate the burst strength of tungsten tubing produced by several processes. These processes included direct extrusion to size by the use of a floating mandrel, a proprietary method of extrusion and processing, extrusion and drawing using the filled-billet technique, chemical vapor deposition from tungsten hexachloride, chemical vapor deposition from tungsten hexafluoride, and electroforming from a fluoride bath. Testing was accomplished at temperatures ranging from 3000° to 4500° F (1650° to 2480° C) using nitrogen gas as the internal pressurizing medium while the heated tube was in an argon atmosphere.

The burst strengths of the majority of the tungsten tubes were equal to or greater than the ultimate tensile strength of extruded or swaged-extruded tungsten rod. The burst strength results for tubes fabricated by chemical vapor deposition from tungsten hexachloride indicated that the process is capable of producing tubing which is as strong as wrought tungsten tubing.

INTRODUCTION

Tungsten tubing, a relatively new addition to the family of engineering materials, is being produced by various techniques. The production methods have included extrusion and drawing using the filled-billet technique (ref. 1), extrusion to size using a floating mandrel (ref. 2), chemical vapor deposition from tungsten hexafluoride (ref. 3) or tungsten hexachloride (ref. 4), and electroforming in a molten fluoride bath (ref. 5).

Some mechanical property data have been obtained for wrought tungsten tubing. Ring compression tests have been used to determine the ductile-brittle transition temperature of tubing fabricated by filled-billet extrusion (ref. 1). The strength of tubing produced

by the floating-mandrel technique has been determined under tensile and biaxial stress conditions at 3500° F (1930° C) in reference 2. Additional data are required for a complete evaluation of the high-temperature, mechanical properties of wrought tubing. These properties have not been determined for tubing fabricated by other methods.

This report presents the data obtained in a study of the effect of fabrication technique on the burst strength of tungsten tubing. Both wrought tubing and tubing produced by the deposition techniques are included. The test method used to evaluate thin-walled tubing of either 3/8-inch (9.5-mm) or 1/2-inch (12.7-mm) diameter is described. The burst strength of the tungsten tubing was determined at temperatures ranging from 3000° to 4500° F (1650° to 2480° C) and is compared to the ultimate strength of wrought tungsten rod. A metallographic evaluation of the tungsten tubing before and after testing is included.

MATERIALS AND PROCEDURE

Materials

The six tube materials included in this study are listed in table I along with their sources. The tungsten tubes used in this evaluation were some of the first produced by the various fabrication techniques and are not necessarily representative of more recently produced material.

TABLE I. - TYPES OF THIN-WALLED TUNGSTEN

TUBING EVALUATED

Fabrication process	Source
Extruded to size (floating-mandrel technique, ref. 2)	Lewis Research Center
Extruded (proprietary process)	General Electric Co.
Extruded and drawn (filled-billet technique, ref. 1)	Nuclear Metals, Inc.
Chemical-vapor-deposited from tungsten hexachloride (ref. 4)	Sylvania Electric Products, Inc.
Chemical-vapor-deposited from tunsten hexafluoride (ref. 3)	Oak Ridge National Laboratory
Electroformed (ref. 5)	Union Carbide Corp.

The extruded tubes made by the various fabrication techniques were produced to final size by metalworking operations. The hexachloride-vapor-deposited tubes were finish ground to size. The hexafluoride-vapor-deposited tubes were supplied in the as-formed condition. The electroformed tubes were made oversize (wall thickness, 0.030 to 0.040 in. (0.76 to 1.02 mm)) and ground or electrodischarge machined (EDM) to size (wall thickness, 0.020 in. (0.5 mm)). During grinding or EDM finishing, all of the electroformed tubes developed hairline cracks or were broken. However, one electroformed tube was electropolished to size and was tested. Prior to testing, all of the other tubes were electropolished. Typical appearance of the electropolished tubes is shown in figure 1.

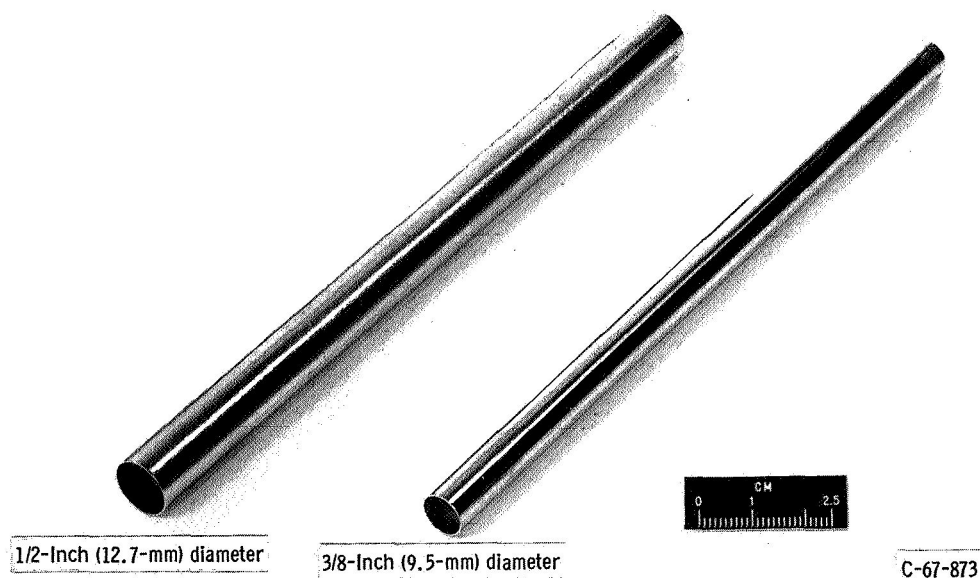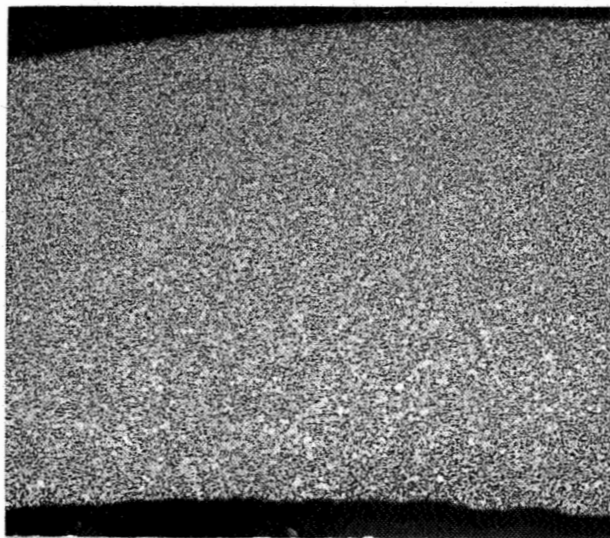| 1/2-Inch (12.7-mm) diameter | 3/8-Inch (9.5-mm) diameter | | C-67-873 |

Figure 1. - Typical appearance of tungsten tubes electropolished in 10 percent solution of sodium hydroxide.

Even though the electropolished tubes appeared to be similar, gross microstructural differences existed among them, as illustrated in figure 2. These differences resulted from the fabrication techniques used in tube production. All tubes produced by extrusion techniques had similar fine-grained structures that exhibited evidence of cold work (figs. 2(a) to (c)). Conversely, the tubes produced by chemical vapor deposition or electroforming generally exhibited a columnar grain structure, as shown in figures 2(d) and (e). In these tubes, the microstructures in the transverse and longitudinal directions were quite similar; hence, only the transverse sections are shown.

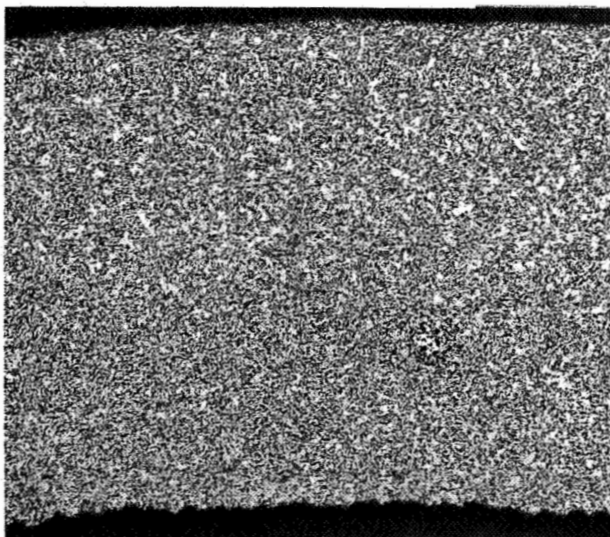The chemical-vapor-deposited tubes produced by the hexafluoride process had the
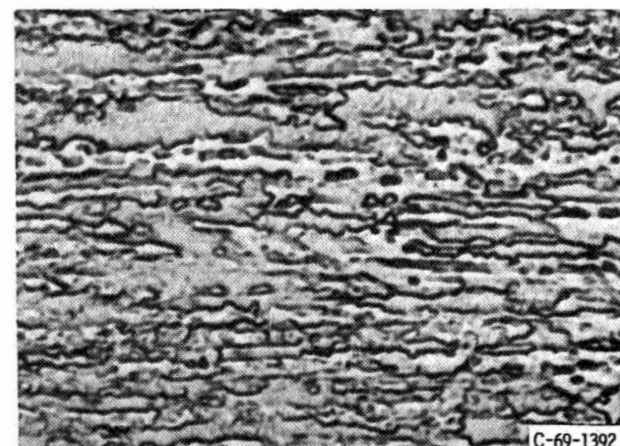
(a-1) Cross section. X100.

(a-2) Longitudinal section. X750.

(a) Extruded (floating-mandrel technique).
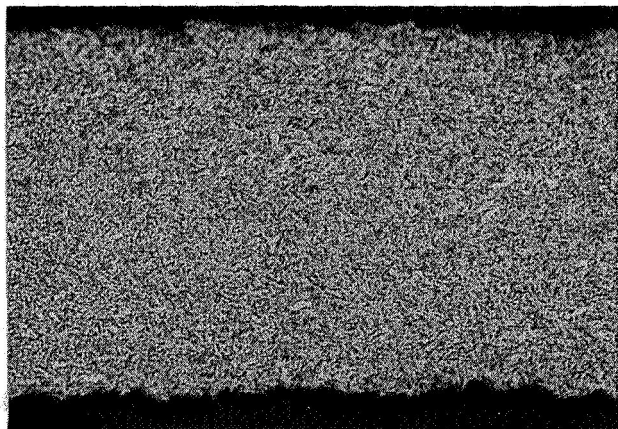
(b-1) Cross section. X100.

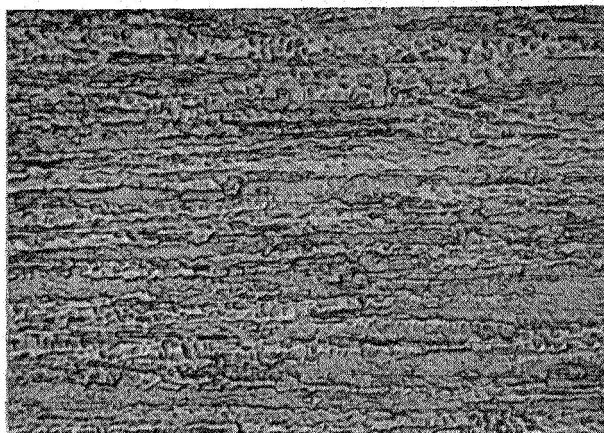(b-2) Longitudinal section. X750.

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(b) Extruded and drawn (filled-billet technique).

Figure 2. – Typical microstructures of tungsten tubing used in burst strength evaluation. Etchant, Murikami's.
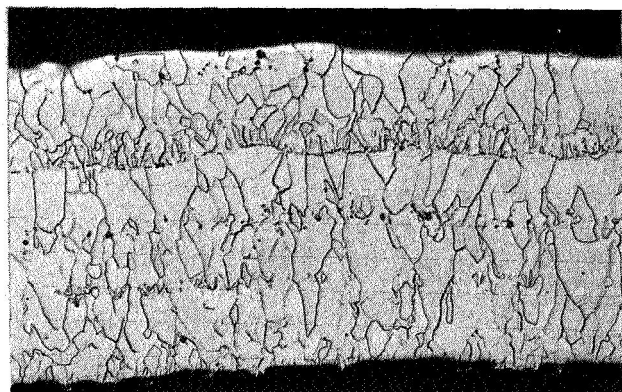
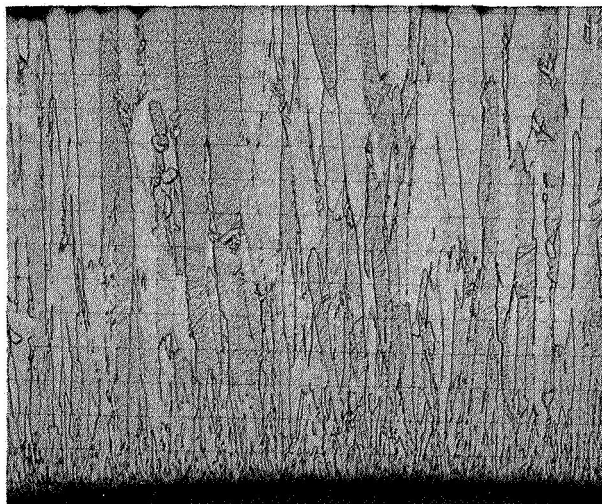(c-1) Cross section. X100.

(c-2) Longitudinal section. X750.
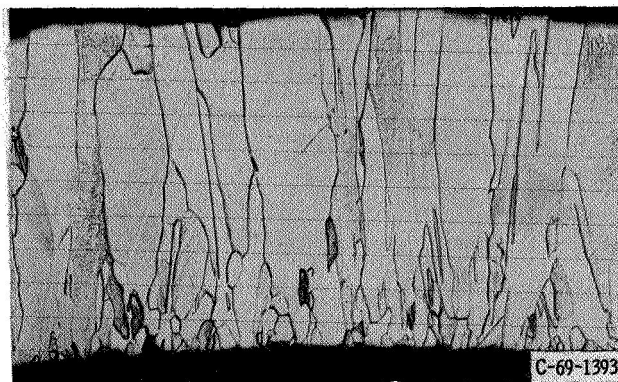
(c) Extruded (proprietary process).

(d-1) Tungsten hexachloride process.

(d-2) Tungsten hexafluoride process.

(d) Chemical vapor deposited. Cross section. X100.

C-69-1393

(e) Electroformed. Cross section. X100.

Figure 2. - Concluded.

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most columnar grains. The grains of the electroformed tungsten tubes were less columnar, while the grains of chemical-vapor-deposited tubes produced by the hexachloride process were least columnar. The interrupted deposition technique employed in the hexachloride process was designed to produce essentially equiaxed grains.

Apparatus

The test apparatus, shown schematically in figure 3, was designed to test 7-inch-(18-cm-) long test specimens in an inert atmosphere chamber. The apparatus was essentially a set of water-cooled connectors to which the tungsten tube specimens were brazed. The brazed assembly was provided with thermocouples, placed inside an induction coil, and connected to nitrogen gas and water terminals located in an inert atmosphere chamber.

A peak load indicator, located downstream from the nitrogen pressurizing gas inlet,
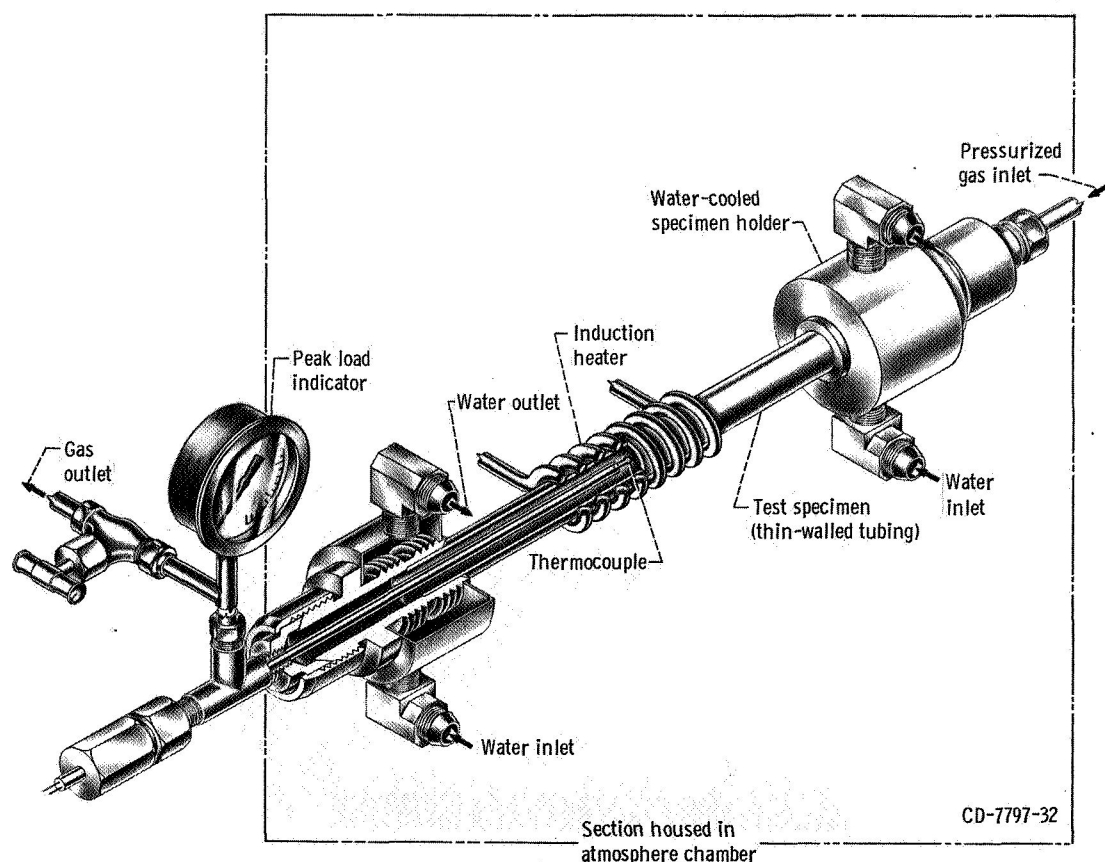

Figure 3. - Schematic of high-temperature tube bursting apparatus.

automatically maintained the maximum applied pressure reading. The pressurizing nitrogen gas was passed through a bubbler just before venting to atmosphere. This provided a pressurizing train which was isolated from the atmosphere during purging of the lines and heating of the tubular specimen.

Specimens were heated with an induction coil powered by two 30-kilowatt, 10-kilohertz motor generators connected in parallel. Heating was manually controlled. The temperatures developed in the tungsten tubes were recorded on a strip-chart recorder. The initial heating rate, from room temperature to 2400° F (1320° C) was governed by the idling characteristics of the induction heater. The average heating rate over this temperature range was 20° F (11° C) per second. The average heating rate, from 2400° F (1320° C) to test temperature, was maintained at 5° F (3° C) per second.

The short, 7-inch- (18-cm-) long, specimens caused a large temperature gradient to exist in the test samples. The magnitude of this gradient was determined with four equally spaced tungsten - 3-percent rhenium against tungsten - 25 percent rhenium thermocouples. Temperature profiles for specimens heated to different maximum temperatures are shown in figure 4. The zone of maximum temperature was relatively short (approx 1/2 in. (12.7 mm)), and failure did not always occur here. However, the re-
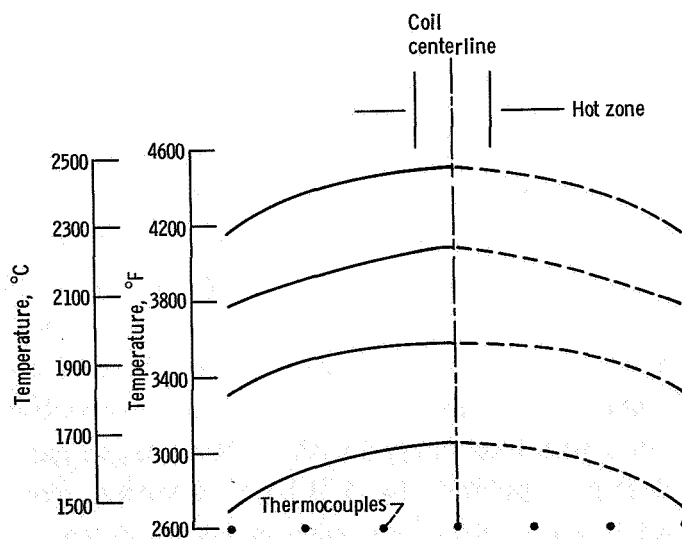

Figure 4. - Typical temperature profiles of test section of tungsten tubes. Distance between thermocouples, 1/2 inch (1.27 cm).

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ported failure temperature was always the maximum maintained during testing and not necessarily the temperature that prevailed at the failure site.

Procedure

All tubes subjected to the burst test were electropolished to remove at least 0.002 inch (0.005 cm) from the diameter. The electropolishing was accomplished in an aqueous 10-percent sodium hydroxide solution. After electropolishing, the tubes were checked for integrity by using a mass spectrometer (helium leak check). The tubes that were leaktight were incorporated into the apparatus shown schematically in figure 3 for burst testing.
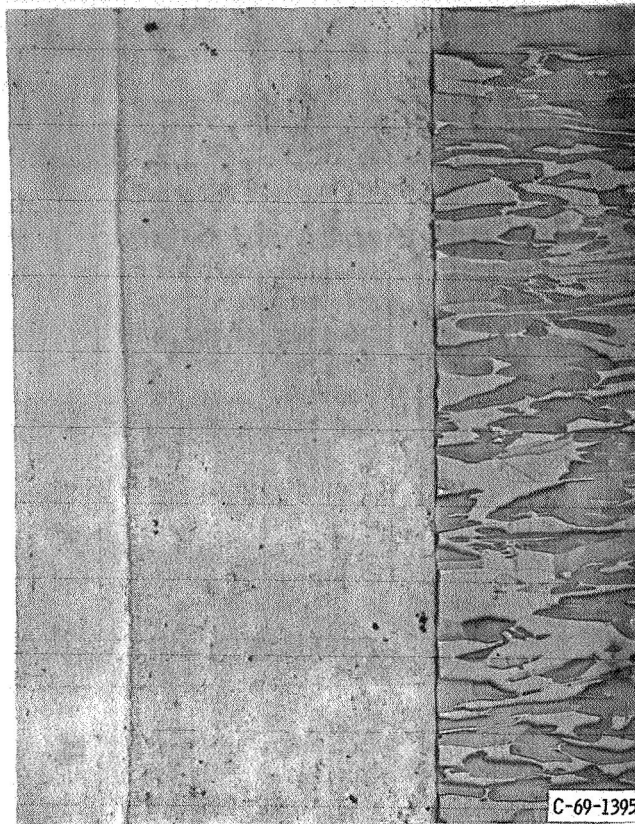
To produce a pressure-tight system, the tungsten tubing was brazed to adapters of stainless steel or copper. Brazing was accomplished in a hydrogen atmosphere furnace using a low-melting-point braze alloy having the following nominal composition (in percent): 45 Ag, 24 Cd, 16 Zn, 15 Cu, 0.15 other (max.). All braze joints were of good quality. Metallographic study of the braze area indicated that good wetting was obtained between the braze alloy, the adapter, and the tungsten tube. The photomicrograph shown in figure 5 is typical of the excellent braze joints obtained.

After the adapters were brazed to the tungsten tube specimen, the assembly was subjected to a pressure test. In this test, the tube-adapter assembly was pressurized with air while being submerged in a water bath. If no indication of a leak was detected during pressurization at 40 psia (0.276 MN/m^2) for a period of 3 minutes, the assembly was considered to be pressure tight. Pressure-tight assemblies were provided with water cooling jackets, thermocoupled, and incorporated into the pressure train. The specimen section was centered in an induction coil housed in an atmosphere chamber. Prior to sealing the chamber, the pressure train was checked for leaks by pressurizing with nitrogen gas at 40 psia (0.276 MN/m^2). The train was considered to be leaktight if no pressure drop was experienced in 1/2 hour. Then the train was continuously flushed with purified nitrogen until start of pressurization-to-failure of the heated tube.

The atmosphere chamber was sealed after all water connections and gas connections were tight. The chamber was then purged with purified argon gas until an exposed filament of a 40-watt bulb burned brightly for 1/4 hour. Average purge time was approximately 1 hour. When the purge had progressed to the above specifications, the tubes were inductively heated to the test temperature.

Temperature was manually controlled, and all thermocouples were monitored on a strip-chart recorder. When equilibrium test temperature was obtained, the tube train was internally pressurized with purified nitrogen gas.

The tubes were pressurized at a rate of 115±10 psia (0.785±0.069 MN/m^2) per minute until failure occurred. The pressure rate was manually controlled, and the maximum

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Figure 5. – Typical braze joint between 304 stainless-steel tubing and tungsten tubing (electrodeposited). X100.

pressure reached was determined on a peak load indicator. The hoop stress at failure (burst strength) was calculated from the dimensions of the electropolished tubes and the maximum pressure developed within the tube using thin-wall criteria $\sigma_t = PD/2t$ where

σ_t hoop stress (burst strength)

P   internal pressure

D   average diameter

t   minimum wall thickness

These stress data were compared to the ultimate tensile strength of extruded powder-metallurgy tungsten rods and extruded arc-melted tungsten rods tested at a strain rate of 0.05 inch per inch per minute (0.05 mm/mm/min).

The failed tubes were evaluated visually as to failure type and location of failure with respect to the hot zone. The failure area was sectioned for metallographic study.

TABLE II. - BURST STRENGTH OF TUNGSTEN TUBING SPECIMENS

Speci-men	Nominal diameter		Nominal wall thickness		Test temperature		Burst strength		Failure type
	in.	mm	in.	mm	°F	°C	ksi	MN/m²	
Extruded (floating-mandrel technique)									
1	0.375	9.53	0.020	0.51	3420	1880	7.53	51.9	Pinhole
2	.375	9.53			3440	1890	8.60	59.3	Ductile
3	.375	9.53			3740	2060	10.20	70.3	
4	.500	12.7			3400	1870	11.30	77.9	
5					3500	1930	13.30	91.7	
6					3520	1940	9.82	67.7	
7					3520	1940	7.65	52.7	
8					3550	1950	10.10	69.6	
9					3600	1980	10.90	75.2	
10					3650	2010	9.47	65.3	
11					3820	2100	1.26	8.7	Seam
12					3940	2170	5.29	36.5	Ductile
13					4010	2210	4.57	31.5	Ductile
Extruded (proprietary process)									
1	0.375	9.53	0.020	0.51	3480	1920	12.20	84.1	Ductile
2	.375	9.53			4250	2340	9.07	62.5	Ductile
3	.375	9.53			4400	2430	5.45	37.6	Pinhole, cold zone
4	.500	12.7			3050	1680	14.10	97.2	Ductile, cold zone
5	.500	12.7			3550	1950	6.49	44.7	Ductile
6	.500	12.7			4020	2220	5.60	38.6	Pinhole
Extruded (filled-billet technique)									
1	0.375	9.53	0.020	0.51	3000	1650	8.45	58.3	Pinhole, cold zone
2			.020	.51	3380	1860	5.37	37.0	Pinhole, cold zone
3			.020	.51	4000	2200	5.29	36.5	Ductile, cold zone
4			.040	1.02	3500	1930	9.10	62.7	Ductile
5			.040	1.02	3960	2180	7.65	52.7	Ductile
6			.040	1.02	4500	2480	4.71	32.5	Ductile
Chemical vapor deposited (hexachloride process)									
1	0.375	9.53	0.020	0.51	3000	1650	6.35	43.8	Pinhole
2	.375	9.53			3560	1960	8.13	56.1	Ductile
3	.375	9.53			3960	2180	7.01	48.3	
4	.500	12.7			3000	1650	8.89	61.3	
5					3520	1940	9.35	64.5	
6					3960	2180	5.53	38.1	Ductile, cold zone
7					4080	2250	5.35	36.9	Ductile
Chemical vapor deposited (hexafluoride process)									
1	0.500	12.7	0.040	1.02	3250	1790	3.30	22.8	Ductile
Electroformed									
1	0.400	10.2	0.020	0.51	3490	1920	8.66	59.7	Brittle

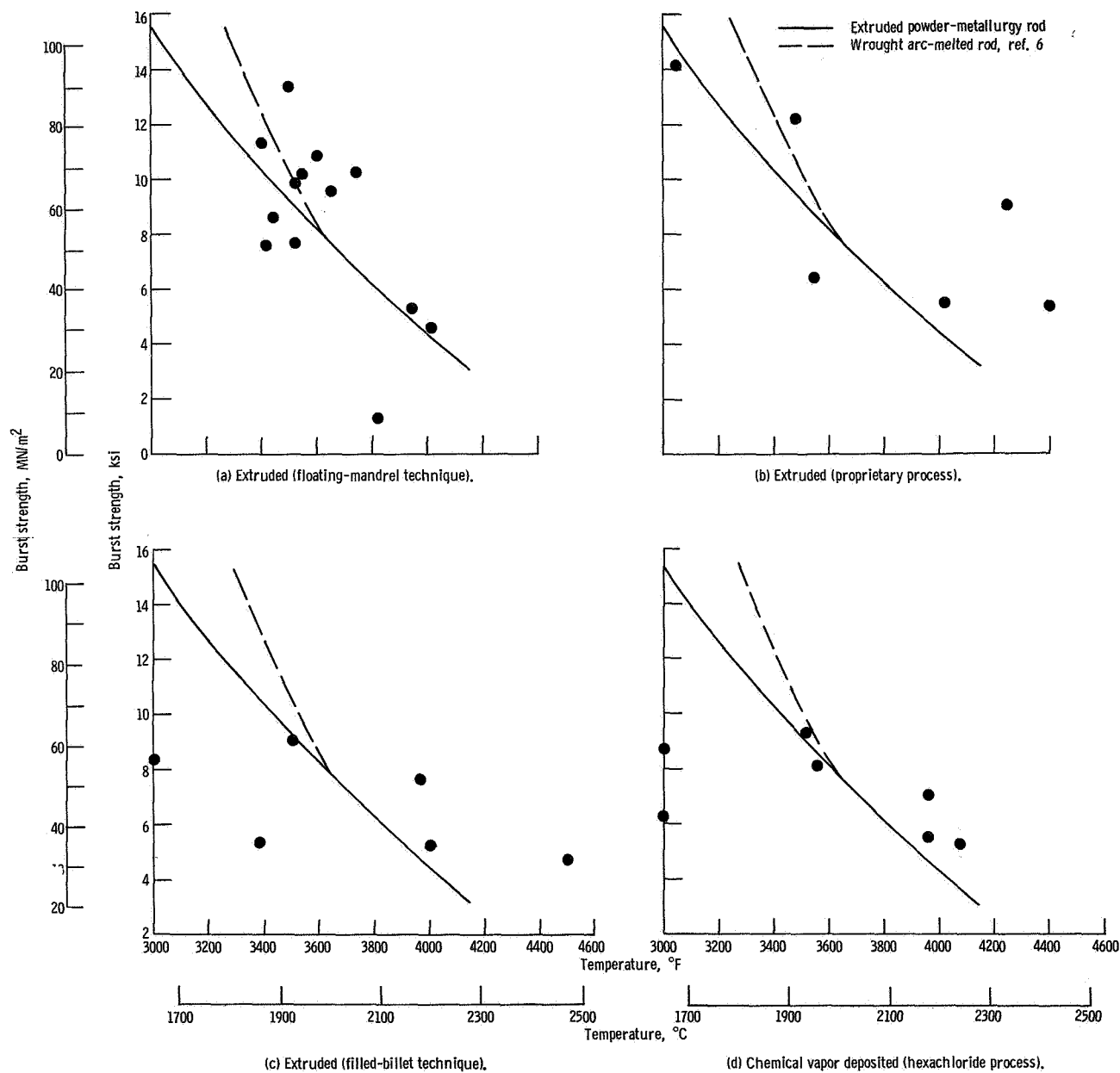Figure 6. - Burst strength of tungsten tubing compared to ultimate tensile strength of tungsten rod.

RESULTS AND DISCUSSION

Burst Strength of Tungsten Tubing

The burst strength data from samples of six fabrication techniques are presented in table II. These data, with the exception of the data from electroformed tubing and hexafluoride-vapor-deposited tubing, are plotted in figures 6(a) to (d) with the burst strength as a function of temperature.

To serve as a reference, extruded powder-metallurgy tungsten rods were tested in tension. The extrusions were made above the recrystallization temperature, and the rods contained small (fine) equiaxed grains. The strength of these rods compares favorably with that of recrystallized tungsten fabricated by other methods. The ultimate tensile strengths of the extruded rods are given in table III. These data coupled with the

TABLE III. - ELEVATED-TEMPERATURE TENSILE STRENGTH OF

POWDER-METALLURGY, EXTRUDED TUNGSTEN[a]

Specimen	Extrusion temperature		Test temperature		Ultimate tensile strength	
	^{o}F	^{o}C	^{o}F	^{o}C	ksi	MN/m^2
1	3000	1650	2900	1590	17.2	119
2	3540	1950	2920	1600	16.5	114
3	4000	2200	2980	1640	15.6	108
4	3000	1650	3420	1880	10.5	72.4
5	4000	2200	3420	1880	9.9	68.3
6	3540	1950	3430	1890	10.2	70.3
7	3000	1650	3930	2170	4.8	33.1
8	3540	1950	3950	2180	5.1	35.2
9	4000	2200	3950	2180	5.2	35.8

[a]Extruded at a 12 to 1 reduction ratio and tested at a strain rate of 0.05 in./in./min (0.05 mm/mm/min).

ultimate tensile strength data for swaged-extruded arc-melted tungsten (ref. 6) are plotted in figures 6(a) to (d) as a reference scatterband for comparison with the burst strengths of the tungsten tubing.

Extruded tubing (floating-mandrel technique). - The tubes produced by the floating-mandrel technique showed burst strengths that were, in most cases, greater than the tensile strength of tungsten (fig. 6(a)). Two of the four failures occurring at values less than the ultimate tensile strength of tungsten were not typical failures; one was a striation-induced seam failure and the other a pinhole-type failure. The other two low-

strength tubes may have contained defects that were not detected. Some of the tubes exhibited exceptional strength, up to 45 percent greater strength than the rod strength. Metallographically, these high-strength tubes appeared to be similar to the lower-strength tubes.

Extruded tubing (proprietary process). - The tungsten tubing extruded by the proprietary technique (fig. 6(b)) showed burst strengths similar to those of the tungsten tubes produced by the floating-mandrel technique (fig. 6(a)). Two tubes failed at strengths less than the ultimate tensile strength of tungsten rod. One tube failed in the cold zone, and the other was a typical hot-zone failure. In this group two pinhole-type failures occurred (one in the hot zone and the other in the cold zone) at burst strengths appreciably greater than the ultimate tensile strength of tungsten. The reason for the high strength obtained for the pinhole failures was not discernible by metallographic techniques.

Extruded tubing (filled-billet technique). - The burst strengths of extruded and drawn tubing produced by the filled-billet technique are shown in figure 6(c). In this group, only 3/8-inch- (9.5-mm-) diameter tubes were available for testing. These tubes were supplied in various wall thicknesses. The heavy-walled (0.040 in., 1.02 mm) specimens were generally stronger than the thin-walled (0.020 in., 0.51 mm) specimens. Two of the thin-walled tubes failed with pinhole-type failures at strengths less than the ultimate tensile strength of tungsten, while the third thin-walled specimen failed in the cold zone at a relatively high strength. One of the thick-walled specimens failed at a stress less than the ultimate tensile strength of tungsten. In general, the burst strengths of these tubes were similar to those of the floating-mandrel tubes.
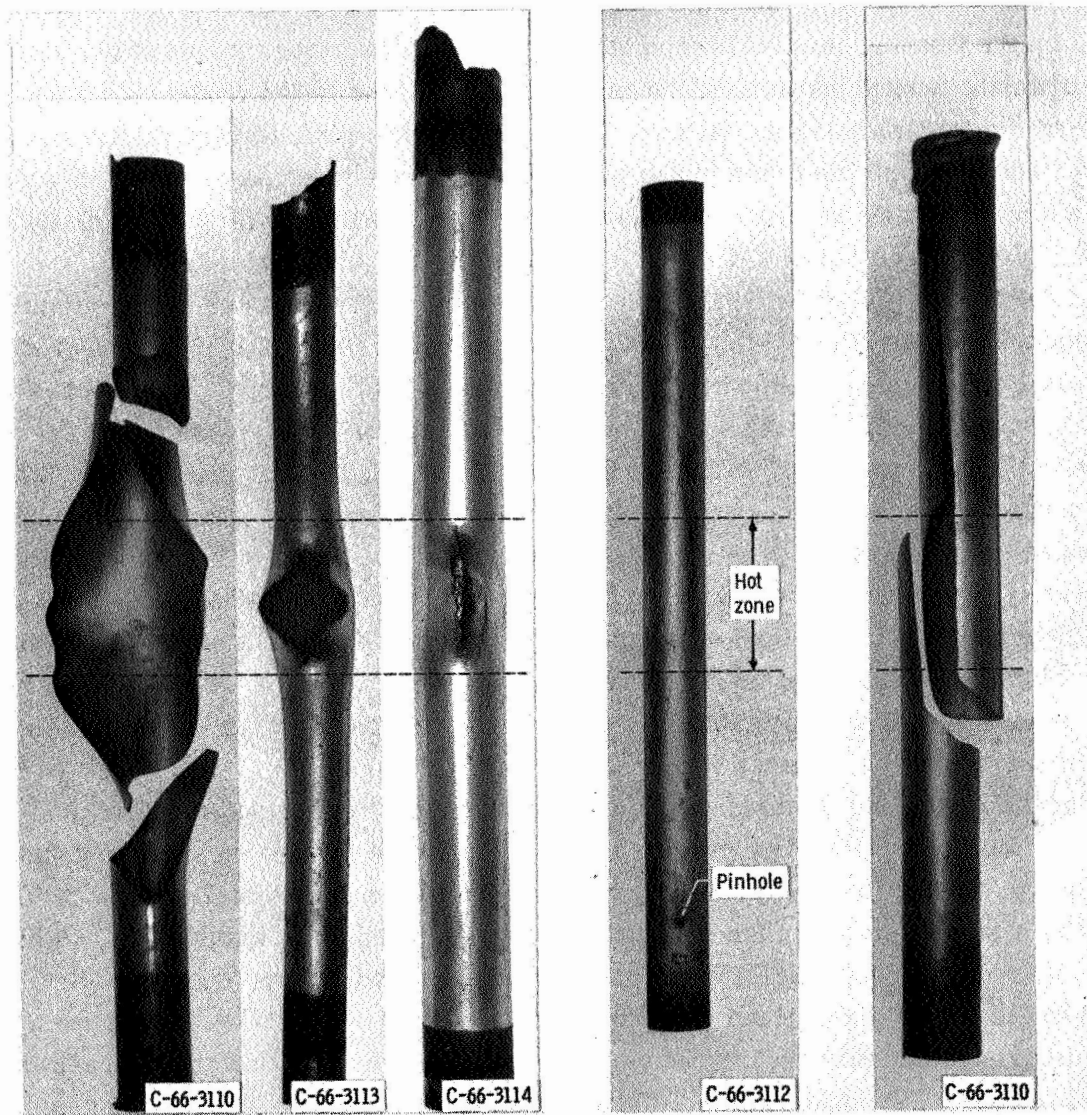
Chemical-vapor-deposited tubing (hexachloride process). - With the exception of the extremely low strengths obtained for the tubes tested at 3000° F (1650° C), the burst strength of the remaining tubes chemical vapor deposited from tungsten hexachloride were approximately equal to, or greater than, the ultimate tensile strength of tungsten rod (fig. 6(d)). A ductile cold-zone failure occurred in the specimen tested at 3960° F (2180° C) at a stress greater than the ultimate tensile strength of tungsten rod (table II). The reason for this atypical failure and the low strengths of the two tubes at 3000° F (1650° C) could not be determined metallographically.

A comparison of figure 6(d) with figures 6(a) to (c) indicates that the process is capable of producing vapor-deposited tungsten tubing as strong as wrought tungsten tubing of like size.

Chemical-vapor-deposited tubing (hexafluoride process) and electroformed tubing. - The burst strength of the only hexafluoride tube tested, 3300 psi (22.8 MN/m^2) at 3250° F (1790° C), was too low to be considered representative of tungsten. Thus, the single test precludes the drawing of any conclusion on the properties of tubing produced by the hexafluoride process.

The burst strength of the only electroformed tungsten tube tested was somewhat

(a) Ductile.   (b) Pinhole.   (c) Seam.

Figure 7. - Types of failures occurring during burst testing of tungsten tubes.

better than that obtained from the hexafluoride tube, but it was still less than the tensile strength of tungsten at the test temperature, 8660 psi (59.7 MN/m^2) at 3490° F (1920° C) against 9400 psi (64.9 MN/m^2). Again, this single test should not be considered representative of the burst strength of electroformed tungsten tubing.

Evaluation of Failed Tubes

Tube failures. - Most of the failures showed some evidence of ductility. Only the electroformed specimen failed in a truly brittle manner. The heated section broke into shrapnel during the failure, and the origin of failure could not be determined.

Failure types, except the brittle failure, are shown in figure 7. In ductile-type failures, the measure of ductility, as indicated by the movement of metal during failure in figure 7(a), appeared to be more closely related to the internal pressure at time of failure than to temperature. In other words, tubes that failed at the highest temperature did not always have the greatest displacement of metal.

Pinhole failures were considered to be nontypical because they indicated a very localized weak spot in the tube; however, metallographic examination of the failure site did not substantiate this. Location of a pinhole failure did not appear to be a function of temperature, for failure locations ranged from the hot zone to the cold zone outside of the heating coil (as shown in fig. 7(b)).
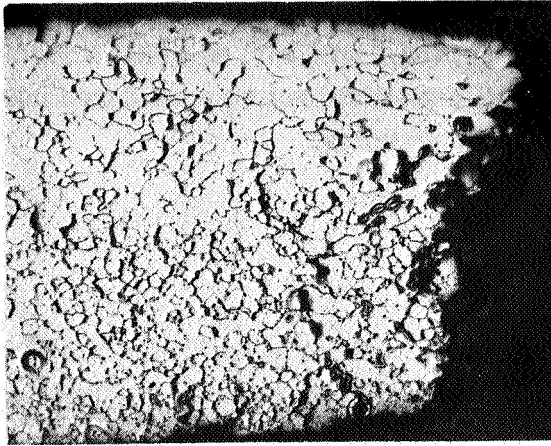
The seam failure (fig. 7(c)) showed no evidence of ductility. Failure propagated along a striation produced in the tube during extrusion. This failure was considered to be nontypical because of the presence of the obvious flaw.

Metallography evaluation. - Failure propagation was intergranular for each fabrication technique, as can be noted in figure 8. A tendency for grain growth as the test temperature increased was noted.
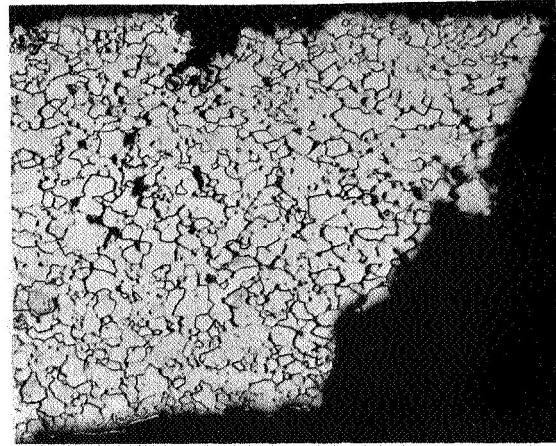
Metallographic study of tube specimens before and after testing showed that recrystallization and grain growth occurred in the wrought tubes during testing. Only minor changes were noted in specimens tested at 3000° F (1650° C), but complete recrystallization occurred in specimens tested at 3400° F (1870° C) or higher.

Little or no grain growth was noted in the vapor-deposited tube tested below 3960° F (2180° C). Some grain growth was apparent in the specimens tested at 3960° or 4080° F (2180° or 2250° C) (compare figs. 2(d-1) and 8(d)).
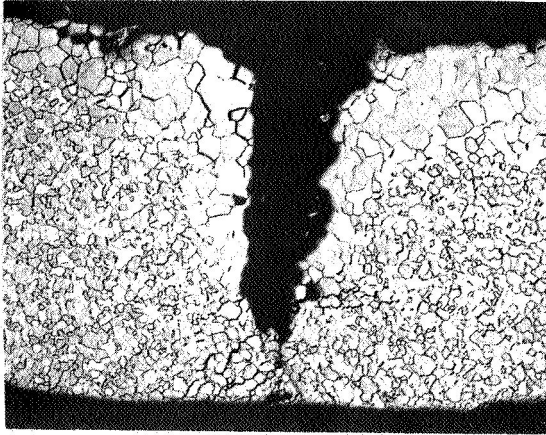
Evidence of void formation was noted in the grain boundaries of the chemical-vapor-deposited hexafluoride-process tube (fig. 8(e)). This void formation is apparently a function of the amount of fluoride trapped in the tube wall during the deposition process (ref. 7).
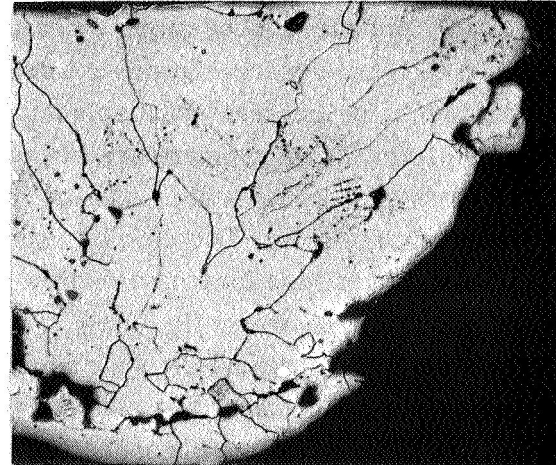
(a) Extruded (floating-mandrel technique); tested at 3400° F (1870° C); burst strength, 11.3 ksi (77.9 MN/m^2). X250.
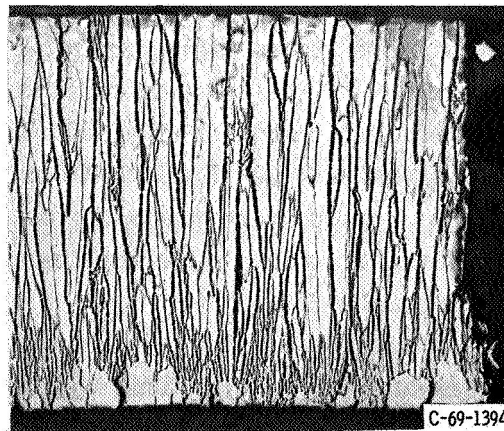
(b) Extruded (proprietary process); tested at 3480° F (1920° C); burst strength, 12.2 ksi (84.1 MN/m^2). X180.

(c) Extruded (filled-billet technique); tested at 4000° F (2200° C); burst strength, 4.7 ksi (32.4 MN/m^2). X100.

(d) Vapor deposited (hexachloride process); tested at 3960° F (2180° C); burst strength, 7.0 ksi (48.3 MN/m^2). X180.

(e) Vapor deposited (hexafluoride process); tested at 3250° F (1790° C); burst strength, 3.3 ksi (22.8 MN/m^2). X75.

Figure 8. - Comparison of failure sections of pressure-tested tungsten tubing.

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SUMMARY OF RESULTS

A test method developed to determine the effect of fabrication technique on the burst strength of 3/8- and 1/2-inch- (9.5- and 12.7-mm-) diameter thin-walled tungsten tubing at temperatures from 3000° to 4500° F (1650° to 2480° C) has had the following results:

1. The majority of the tungsten tubes tested exhibited burst strengths equivalent to, or greater than, the ultimate tensile strength of tungsten rod, at all test temperatures.

2. The tungsten hexachloride vapor-deposition process is capable of producing tubes which are as strong as wrought tungsten tubes of similar size.

3. Failure propagation in the tungsten tubing throughout this temperature range was intergranular for all fabrication processes.

Lewis Research Center,
    National Aeronautics and Space Administration,
        Cleveland, Ohio, April 8, 1969,
           129-03-14-03-22.

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