NASA TECHNICAL MEMORANDUM

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VIEWING DEVICES FOR OBSERVING FLOW CONDITIONS IN HOT MERCURY SYSTEMS

by Lawrence A. Mueller and David W. Medwid

Lewis Research Center

Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • MAY 1968

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SUMMARY

Windows for both a mercury condensing tube and a mercury-boiler-outlet tube were investigated. A key factor in window design was proper matching of the thermal expansion coefficients of the window, the window sealants, and the window-support structure.

The window for the mercury-condensing tube was tested with flowing and condensing mercury at 670° F (628° K) and 15 psia (10^5 N/m^2). At room temperature, the window was also subjected to 20-g random vibration.

The boiler window was heated in a furnace to 1350° F (1005° K) with gas pressurization of 350 psig (2.4×10^6 N/m^2).

INTRODUCTION

Windows, or viewing devices, are used to observe and to help control chemical processes, metal manufacturing operations, and boiler operations (refs. 1 and 2). In space power systems that boil and condense liquid metals, there is a similar need to view the boiling and condensing processes. The use of liquid metals operating at high temperatures and pressures present some design difficulties.

Tests were conducted to investigate the capability of the design of viewing devices for studying the flow phenomena of a high-temperature, high-pressure mercury system. Various high-temperature sealants, window designs, and metal to glass brazing joints were tested at conditions that would be encountered in an experimental hot mercury system.

The first investigation was of window designs and high temperature sealants that would enable optically clear viewing of the mercury condensation process (ref. 3). The condensing tube viewing device was tested at a nominal temperature of 670° F (628° K) with pressures to 15 psia (10^5 N/m^2) and at 20-g random vibration to simulate the vibration levels obtainable during a rocket-propelled flight. A requirement in the design was that the viewing device would not cause a flow disturbance or obstruction to the condensing mercury; commercially available flexible epoxide sealants seemed feasible for use as the window sealant.

The second investigation was of a window for a mercury boiler having outlet conditions of 1350° F (1005° K) and 350 psig (2.4×10^{6} N/m^{2}). Because the operating temperature precluded the use of any known sealant, brazing the window into its supporting structure was investigated. The design and testing of the windows are described.

APPARATUS AND PROCEDURE

Condensing Tube Viewing Device

The viewing device design selected used an external metal support which limited the viewing area. The assembly (fig. 1) consisted of the steel Croloy 9M (Fe-9Cr-1Mo) tube having a 0. 50-inch (1. 27-cm) outside diameter and a 0. 035-inch (0. 088-cm) wall, a segment of borosilicate glass (Pyrex glass) of the same nominal dimensions as the 9M tubing, a sealant, and a clamping device to support the Pyrex glass. The 9M tubing and the Pyrex glass were selected because of their corrosion resistant properties to mercury at the condensing conditions (refs. 4 to 6). The 9M tubing was machined to accommodate the segment of Pyrex glass (fig. 1). The edges of the glass segment were chamfered and roughened to improve the bond with the sealant.

A number of sealants having suitable elevated thermal properties, thermal expansion coefficients, as well as bonding and sealing coefficients were selected and tested after a literature survey was conducted. The evaluation of these sealants is described in the appendix. The sealant selected for the condensing tube viewing device was Sample F which expands approximately 370 times more than the Pyrex glass and approximately 95 times more than the 9M tubing at a temperature of 670° F (628° K). More information on the thermal expansion coefficients are presented in table I.

The flanges of the viewing device (fig. 1) were made of cold rolled steel. They were used as clamps and as a tube support. The flange over the glass insert was designed to allow a sealant thickness of 0. 01- to 0. 020-inch (0. 025- to 0. 050-cm) clearance between the glass and flange. Grooves were machined in the flange to allow for the expansion of the sealout with increasing temperatures. The flange opposite the glass insert was similar in construction except that the machined grooves were excluded. The flanges were joined by machine screws. A photograph of the viewing device assembly is shown in figure 2.

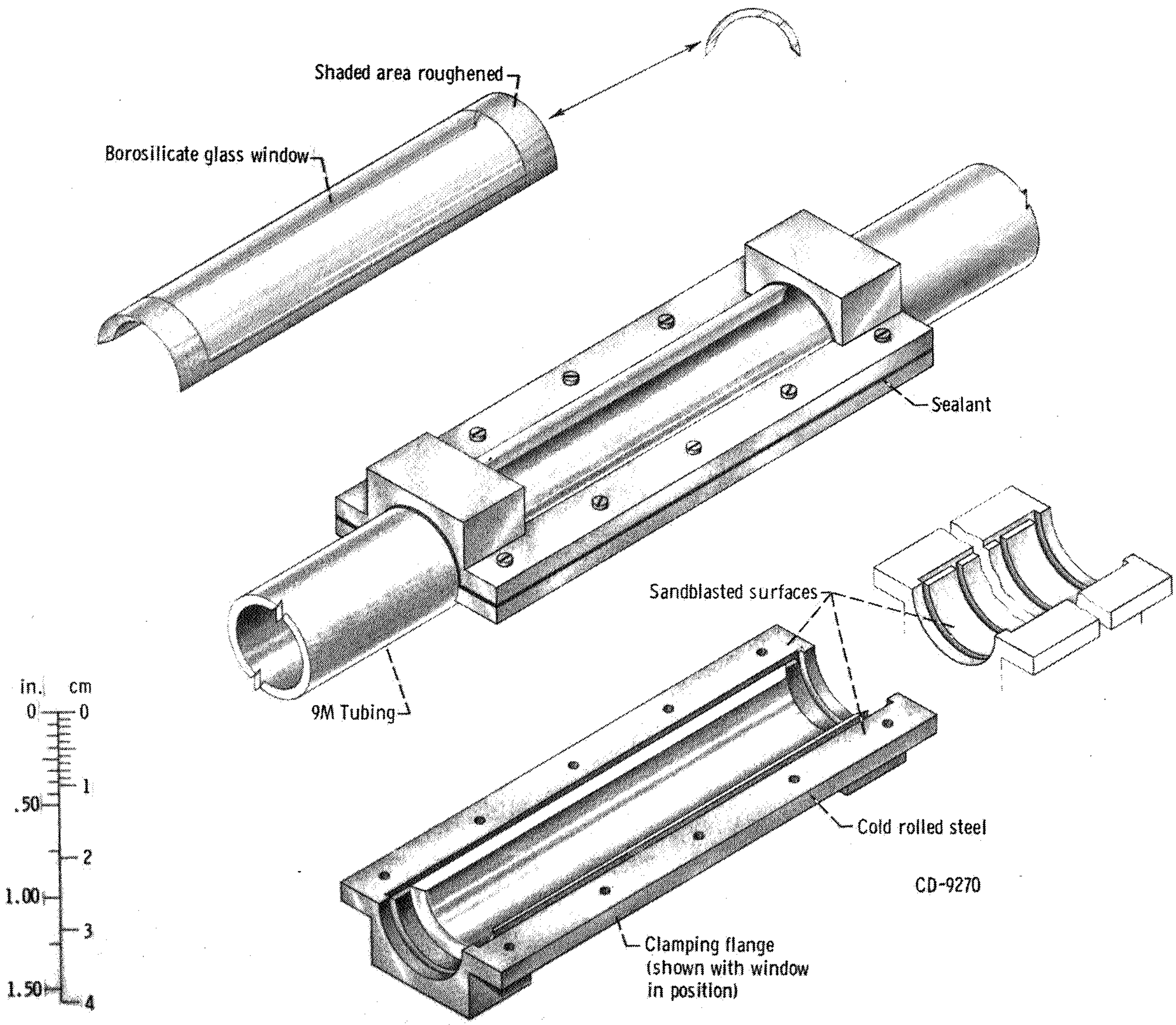


Figure 1. - Viewing device assembly.

TABLE I. - THERMAL EXPANSION COEFFICIENTS

Item	Linear coefficient of thermal expansion		Valid temperature range of expansion coefficient	
	(in./in.)/^{o}F	(cm/cm)/^{o}C	^{o}F	^{o}C
Sample F	670.0×10^{-6}	1206.0×10^{-6}	$^{a}>32$	$^{a}>0$
Pyrex	1.8	3.24	32 to 572	0 to 300
9M	7.0	12.6	70 to 900	21 to 480

[a]Upper limit is not known, but it has been operated at 600^{o} F (588^{o} K).

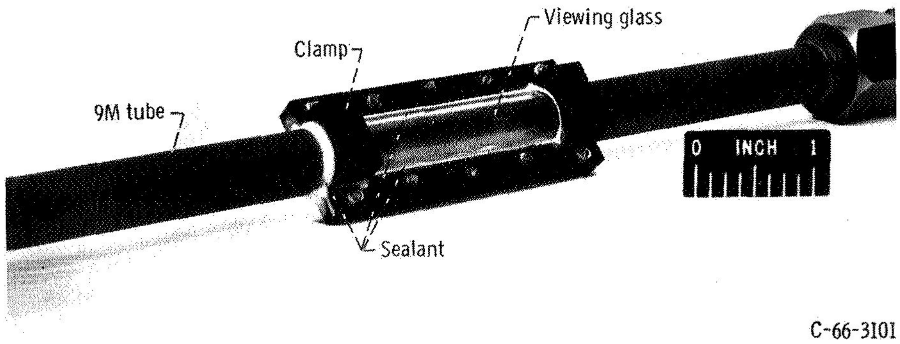
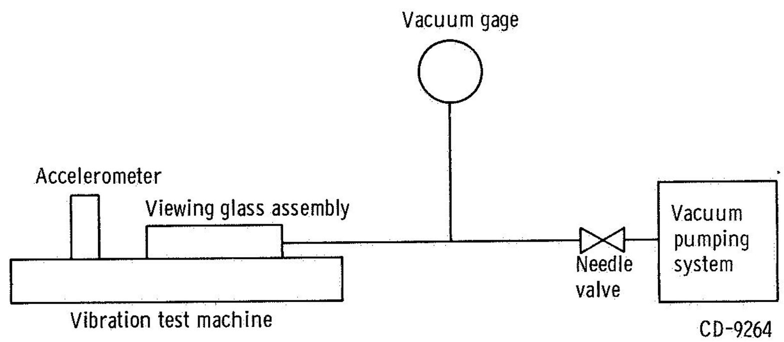


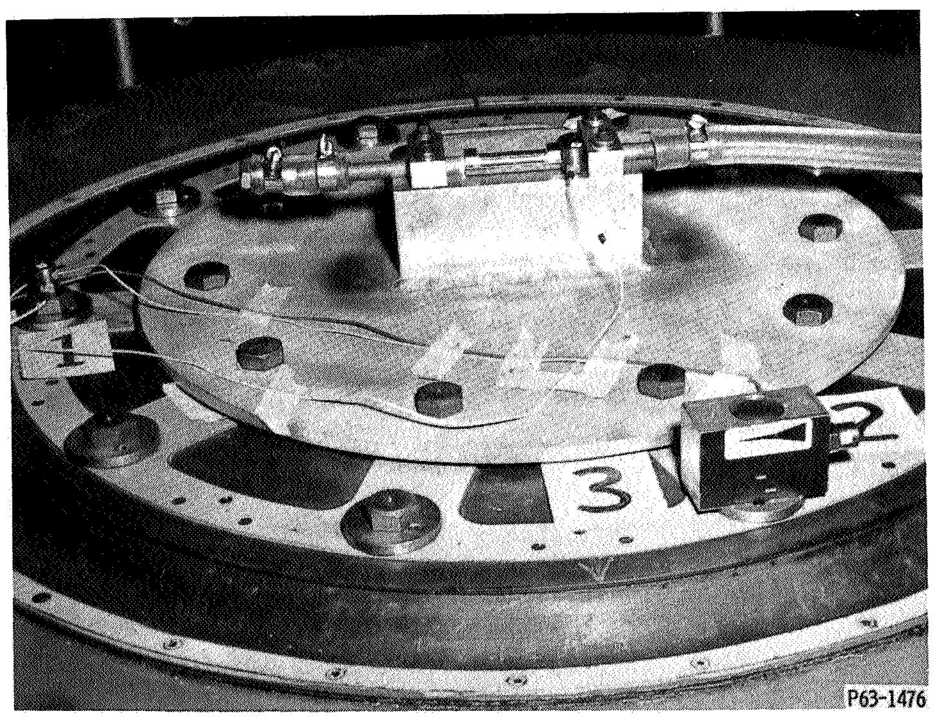
Figure 2. - Condensing tube viewing device.

Viewing device vacuum-vibration. - The condensing tube viewing device assembly was subjected to a series of vacuum-vibration tests to determine its leakage and vibration limitations during a rocket launch. The vacuum-vibration system shown in figure 3(a) consisted of the viewing device, a vacuum system, and a shake table which is capable of producing 20 g's of random vibration. A photograph of the viewing device mounted on the shaker is shown in figure 3(b). The glass assembly was evacuated to 5 microns (0.6 N/m^2) and was vibrated at 10, 15, and 20 g's of random vibration. Each vibration level was maintained for 90 seconds duration. Instrumentation consisted of a vacuum gage and three accelerometers mounted on the shake table (fig. 3(b)).

Viewing device dynamic tests. - The condensing tube viewing device was also subjected to a flow test in a mercury condensing tube facility shown schematically in figure 4 and described in detail in reference 7. The mercury facility consisted of a mercury expulsion unit, a liquid flow measuring system, a preheater, a high-flux boiler, a main boiler, a vapor flow measuring venturi, a horizontal condensing tube, the viewing device, and a receiver to collect the condensed mercury. The mercury vapor was condensed by flowing cooling air over the entire tube length. The system was operated so that the mercury liquid-vapor interface was positioned in the center of the viewing device assembly.



(a) Test setup.



(b) View glass assembly mounted on vibration test machine.

Figure 3. - Viewing glass assembly vibration test system equipment.

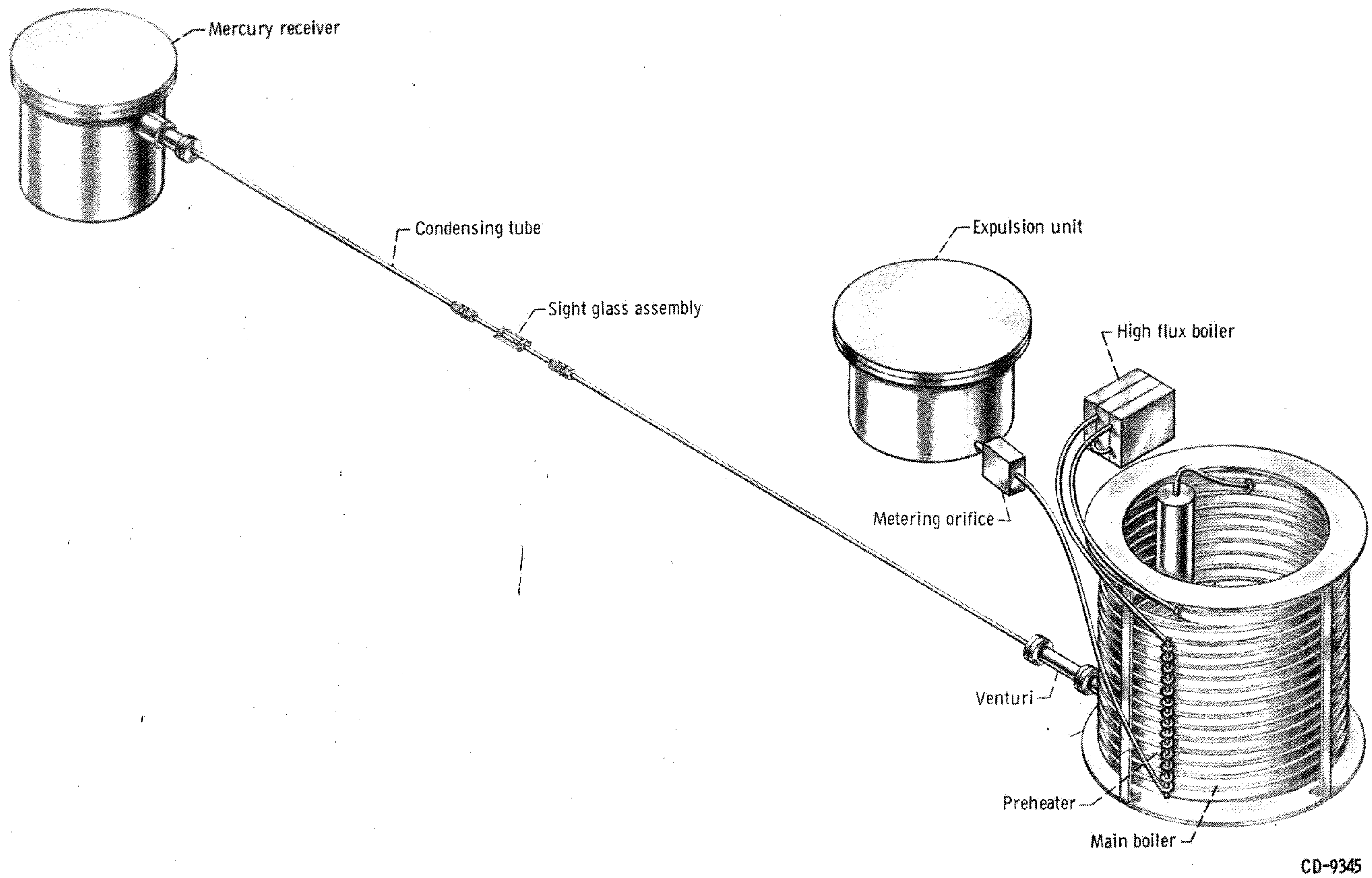


Figure 4. - Mercury condensing tube, sight glass assembly, and components.

Mercury Boiler Outlet Viewing Device

Basic materials selected in the construction of the viewing device at the boiler outlet were suited to a mercury environment of 1350° F (1005° K) and at a pressure of 350 psig (2.4×10^{6} N/m^{2}). The materials chosen were AISI 316 stainless steel, molybdenum, Kovar, and synthetic sapphire. The synthetic sapphire was selected as the window material because it transmits all wavelengths of light from the ultraviolet to the infrared and is especially impervious to mercury vapor. A schematic of the sapphire window assembly is presented in figure 5. The 0.125-inch (0.317-cm) thick sapphire window was metallized with tungsten around the outer perimeter where it was mated with and brazed to the flared molybdenum tubing. A length of Kovar tubing, which provided compatibility and weldability to the AISI 316 stainless steel used in the mercury system, was brazed to the molybdenum tubing. A schematic of the viewing device is shown in figure 6. It consisted of a AISI 316 stainless-steel body, which replaces a part of the mercury process piping, and two sapphire window assemblies, which were surrounded by AISI 316 stainless-steel housings, located 90° apart. One of the sapphire window assemblies was used for viewing purposes; the other to allow light from an external source to enter the viewing device. The surfaces of the sapphire window assemblies were exposed

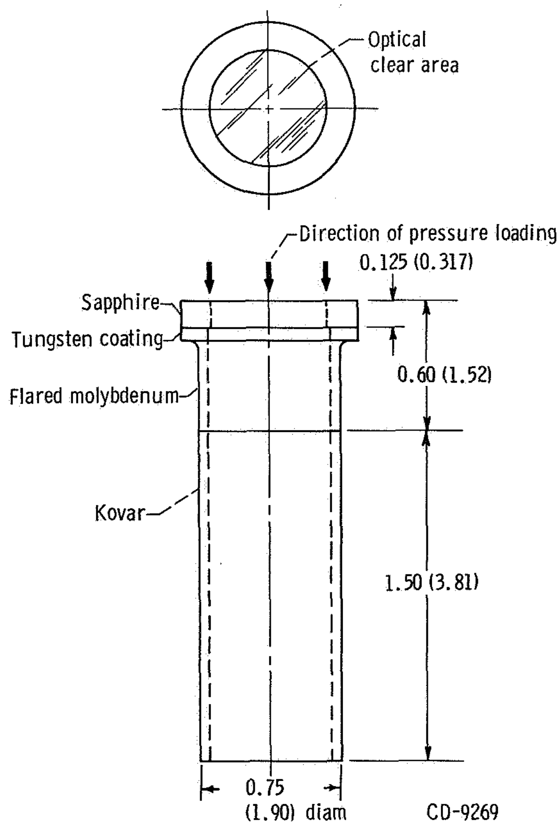


Figure 5. - Schematic of sapphire window assembly (all dimensions are in inches (cm)).

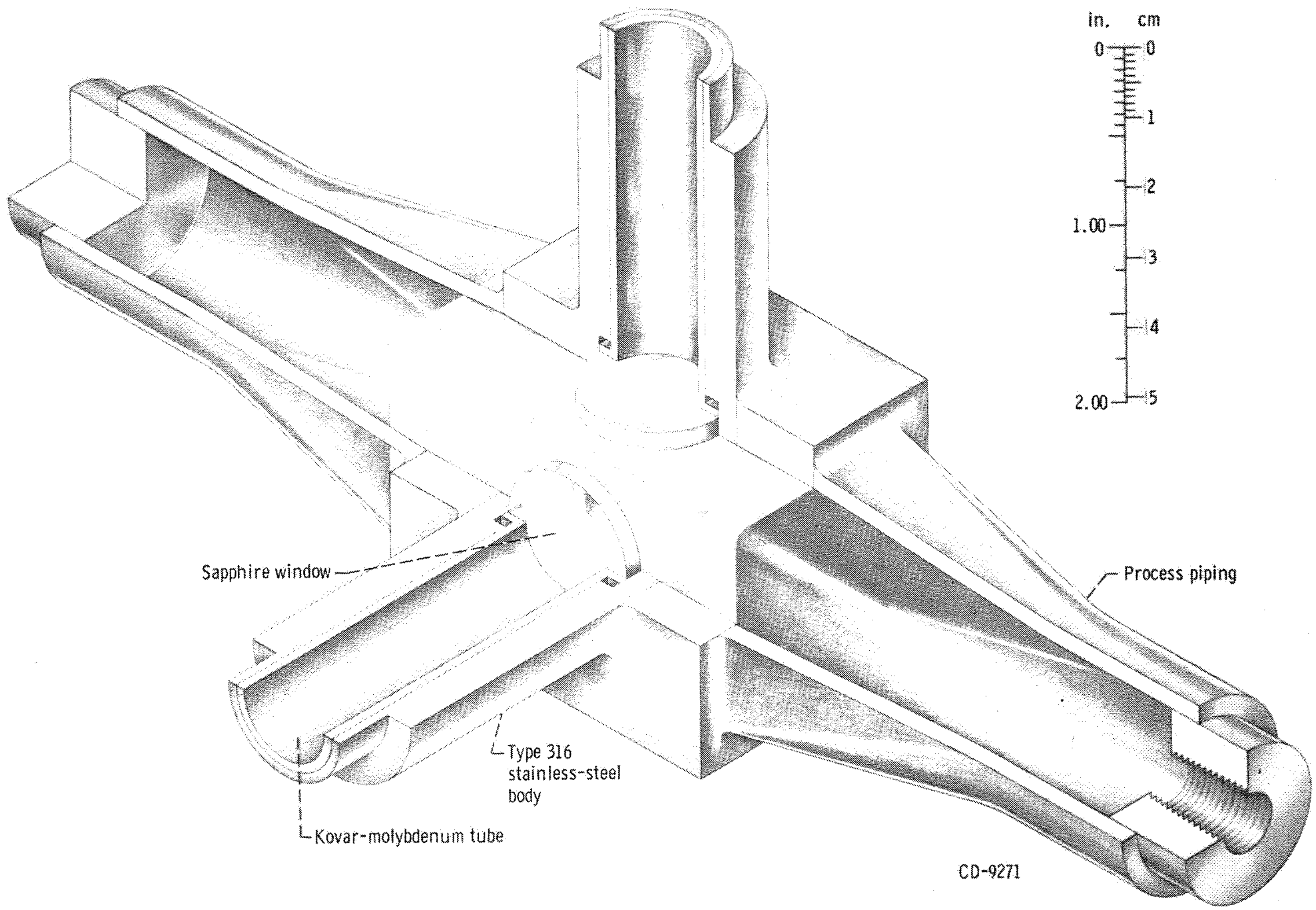
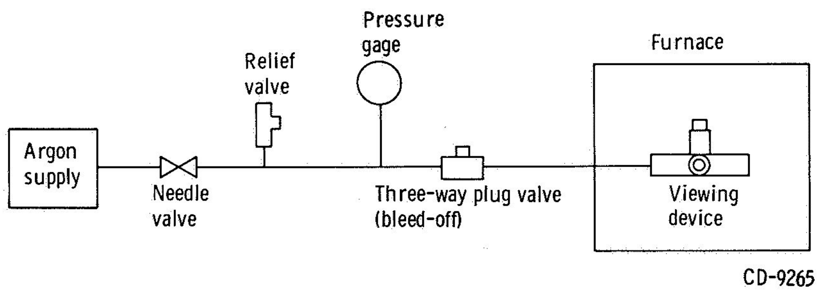


Figure 6. - Boiler outlet viewing device.

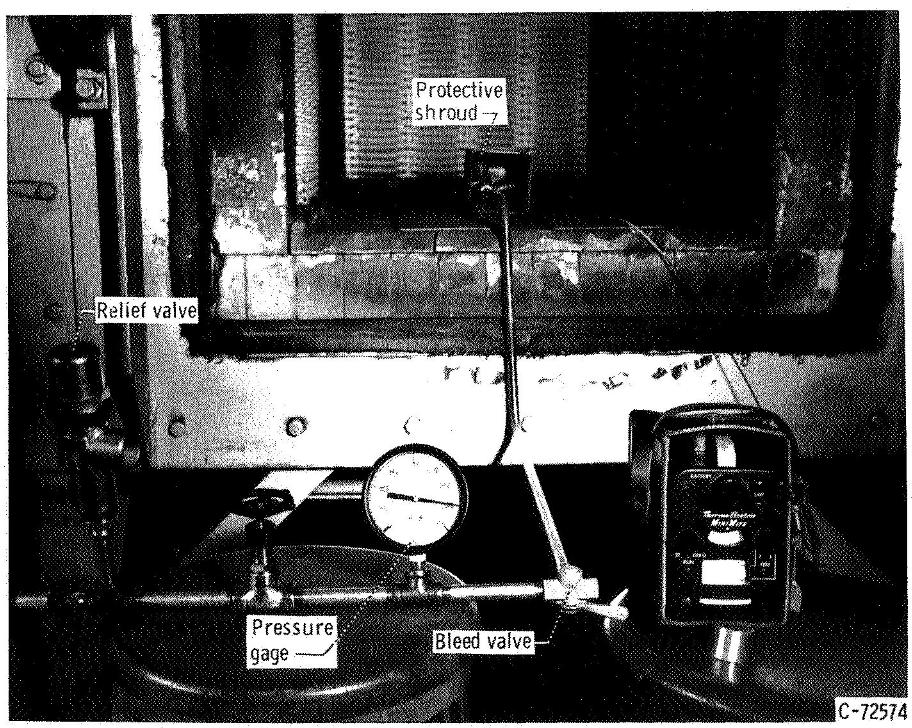
to the process fluid through holes in the stainless-steel body. A clearance of 0.03-inch (0.076-cm) was maintained between the sapphire window surface and the stainless-steel body.

The Kovar tubing of the sapphire window assembly was welded to the stainless-steel housing maintaining a 0.005-inch (0.012-cm) radial clearance between the window assembly and the stainless-steel housing. This clearance allowed the process fluid to contact the surfaces of the window assembly and the housing. The exposure of the sapphire window surface to the internal pressures of the process fluid and the thermal expansion of the various materials placed the metallized sapphire window and window assembly in compression.

The boiler outlet tube viewing device was statically tested in the system shown schematically in figure 7(a). The viewing device was placed within a protective shroud as



(a) Schematic of test setup.



(b) Boiler outlet tube viewing device test section in furnace.

Figure 7. - Static test equipment for boiler outlet tube viewing device.

a safety precaution against shattering due to a failure. In addition, the shroud stabilized the radiant heat from the furnace walls. A photograph of the viewing device with its protective shroud in the furnace is presented in figure 7(b).

With one end of the viewing device plug welded close, a constant argon pressure through a range of 90 to 350 psig (6.2×10^5 to 2.4×10^6 N/m^2) was supplied to the viewing device. The temperature of the viewing device was controlled by means of an oven temperature controller and was recorded by means of a Chromel-Alumel thermocouple located on the viewing device. Pressures were recorded by a pressure-gage installed in the argon gas system. The furnace temperature was increased to 1350° F (1005° K) during a period of $3\frac{1}{2}$ hours and then allowed to stabilize for approximately 1/2 hour. At the end of the stabilization period, valves in the argon system were closed, the electrical power to the furnace turned off, and the door to the furnace opened. This permitted rapid cooling and provided a thermal shock to the viewing device. Pressure and temperature were recorded at intervals during the heat-up, temperature stabilization, and cool-down periods.

RESULTS AND DISCUSSION

Condensing Tube Viewing Device

Results of the vacuum-vibrations tests in which the viewing device was subjected to 10 to 20 g's of random vibration (equivalent to vibrations recorded in rocket launches) at a 100-micron (13-N/m^2) vacuum level showed that the viewing device could withstand the launch vibration levels without inducing leakage across the seal. The dynamic tests of the condensing tube viewing device were conducted at pressure levels from 12 to 15 psia (8.2×10^4 to 1×10^5 N/m^2) and temperature levels from 660° to 670° F (622° to 628° K).

No evidence of leakage across the condensing tube viewing device was found during the dynamic tests. The relatively high expansion coefficient of the sealant, Sample F, was beneficial in preventing mercury leakage. The sealant was the only one tested that had properties similar to silicone rubber and endured the thermal cycles imposed on it without resulting in leakage (see the appendix). The portion of the 9M tubing machined to receive the Pyrex glass was calculated to expand 4 mils (0.010 cm) and the Pyrex glass, 1 mill (0.002 cm) at a temperature of 670° F (628° K). The theoretical gap of 3 mils (0.007 cm) between the insert and the 9M tubing was filled with the sealant, which, in an unrestrained condition, could expand 6 mils (0.015 cm) (3 mils (0.007 cm) or greater than required to close the theoretical gap) at a temperature of 670° F (628° K). The expansion conditions of the 9M to glass joint are shown schematically in figure 8. The ex-

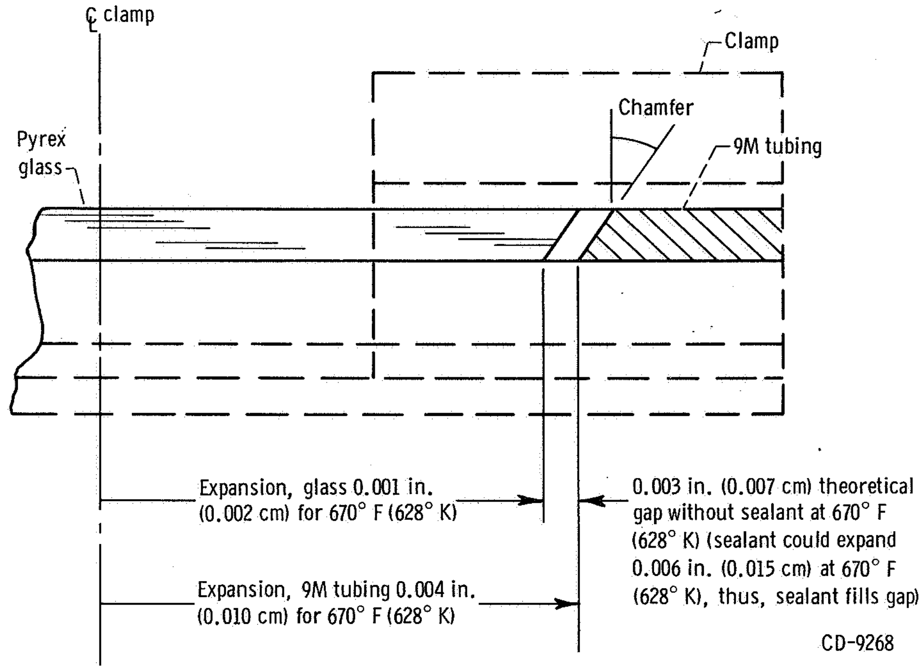


Figure 8. - Theoretical expansion conditions for end joint of Pyrex glass and 9M tubing for condensing tube viewing device.

pansion qualities of the sealant, therefore, produced a sealing pressure in the gap section. Additional pressure from the clamp screws was placed on the sealing surfaces such that the machine clearance between the two clamp halves and the grooves were filled with sealant. When the clamping screws are fastened with a load of 103 pounds (457 N) per screw, the bearing pressure on the hold-down clamp flanges was 800 psi (5.5×10^{6} N/m^2).

Visual observation of the condensing process was excellent with distinct detail.

Inspection of the viewing device, after removal from the mercury system, indicated a slight discoloration and a coating on the inner surface of the Pyrex glass insert. However, optical qualities of the glass were not affected by the coating.

Boiler Outlet Tube Viewing Device

A series of 8 runs was conducted on the boiler outlet tube viewing device in the static test rig. The thermal cycle experienced by the viewing device is shown in figure 9.

The data plotted in figure 9 are tabulated in table II. Runs 1 to 6 were successfully terminated. No leakage across the sapphire-molybdenum brazed joint was noted even though the joint had experienced 6 severe thermal cycles as well as pressure cycles (90, 150, 250, and 300 psig or 6.2×10^{5}, 1.0×10^{6}, 1.7×10^{6}, and 2.0×10^{6} N/m^2) to atmospheric pressure). During run 7, however, at 350 psig (2.4×10^{6} N/m^2), the rated pressure decayed to zero within four minutes after closing the valve at the termination of

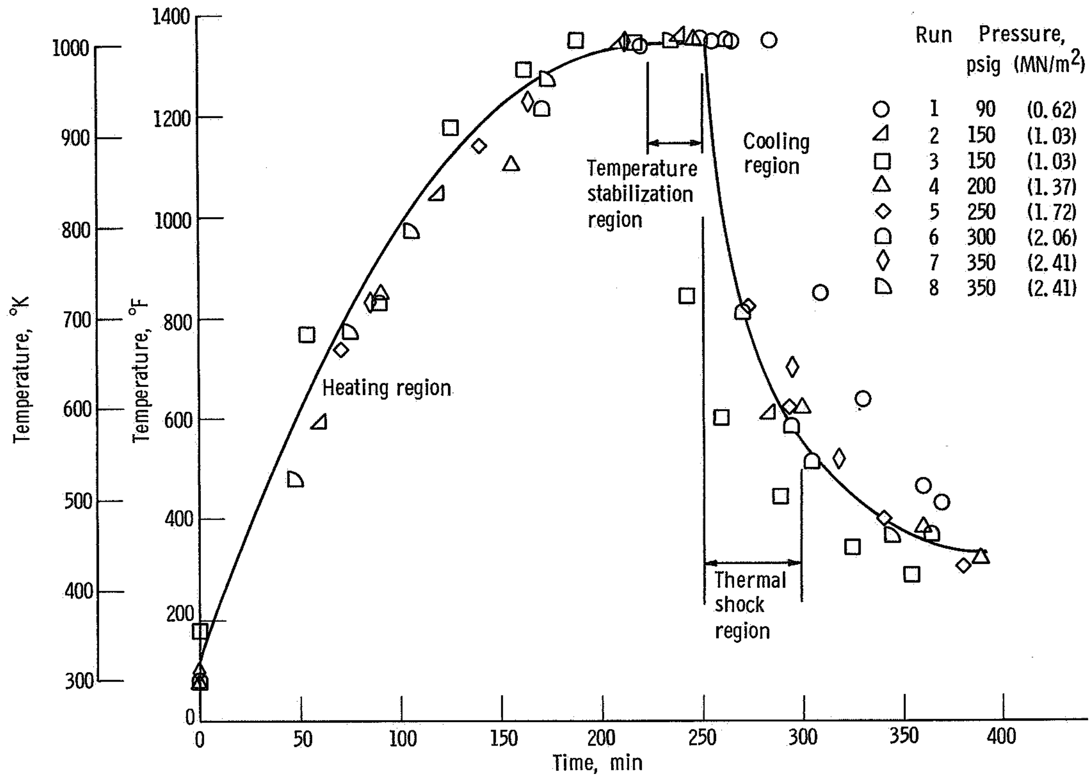


Figure 9. - Thermal cycling of boiler outlet tube viewing device.

the 1350° F (1005° K) temperature stabilization period. The leakage was caused by a failure of the brazing alloy at the sapphire window-molybdenum joint.

Run 8 was conducted with the argon pressure maintained at 350 psig (2.4×10^6 N/m²) by use of a regulator throughout the complete thermal cycle. A photograph of the viewing device at 1350° F (1005° K) and 350 psig (2.4×10^6 N/m²), prior to the thermal cycling is presented in figure 10.

Examination of the viewing device after the tests were completed, indicated that the sapphire windows were discolored. A layer of molybdenum oxide found on the window surface exposed to the furnace atmosphere (not the process fluid side) did not drastically impair the optical quality of the viewing device.

To prevent the molybdenum oxidation, it is recommended that the internal surfaces of the sapphire window assembly (the surface exposed to the furnace atmosphere) be plated initially with nickel followed by an overlay of chromium. The failure of the brazing alloy at the sapphire window-molybdenum joint was attributed to an overloaded brazed joint. To eliminate this defect the thickness of the sapphire window must be increased to lower the flexural stress in the window and reduce the tension load in the brazed joint. The cleaning and assembling of all parts of the viewing device in a clean room and the brazing, accomplished in a vacuum furnace, are necessary.

Since the braze did not fail at run 6 at a pressure of 300 psig (2.0×10^6 N/m²) after

TABLE II. - STATIC TEST RESULTS OF BOILER OUTLET TUBE VIEWING DEVICE

(a) U.S. customary

Run															
1		2		3		4		5		6		7		8	
Pressure, psig															
90		150		150		200		250		300		350		350	
Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F	Time, min	Temperature, °F
0	70	0	85	0	175	0	70	0	95	0	78	0	85	0	87
255	1350	29	228	16	345	91	850	70	738	90	833	83	837	47	480
285	[a]1350	59	588	34	508	120	1022	140	1145	169	1213	163	1232	75	777
294	1173	89	863	53	770	155	1105	215	1345	210	1340	220	1342	105	973
299	1035	119	1042	77	940	179	1263	250	[a]1353	248	[a]1352	265	[a]1350	173	1273
304	932	179	1281	105	1090	205	1362	273	828	271	812	295	705	235	1342
309	852	209	1348	125	1177	220	1350	293	622	294	587	319	520		
314	788	239	[a]1355	161	1288	245	[a]1355	337	413	305	518	364	372		
319	732	254	940	188	1350	271	840	380	307						
329	635	284	613	218	[a]1348	300	625								
339	568	314	455	238	840	330	475								
349	510	344	362	260	607	360	387								
359	467	374	312	288	450	388	325								
367	437			325	345										
				354	298										

[a]Furnace heating controls turned off; door opened to room atmosphere.

TABLE II. - Concluded. STATIC TEST RESULTS OF BOILER OUTLET TUBE VIEWING DEVICE

(b) SI units

Run															
1		2		3		4		5		6		7		8	
Pressure, MN/m^2															
0.6		1.0		1.0		1.3		1.7		2.0		2.4		2.4	
Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time, min	Temperature, ^oK	Time min	Temperature, ^oK
0	294	0	302	0	352	0	294	0	308	0	298	0	302	0	303
255	1005	29	382	16	447	91	727	70	665	90	718	83	720	47	522
285	[a]1005	59	582	34	537	120	823	140	891	169	929	163	940	75	687
294	907	89	735	53	683	155	869	215	1002	210	1000	220	1001	105	796
299	830	119	834	77	777	179	957	250	[a]1007	248	[a]1006	265	[a]1005	173	962
304	773	179	967	105	861	205	1012	273	715	271	706	295	647	235	1001
309	728	209	1004	125	909	220	1005	293	601	294	581	319	544		
314	693	239	[a]1008	161	971	245	[a]1008	337	485	305	543	364	462		
319	662	254	777	188	1005	271	722	380	426						
329	608	284	596	218	[a]1004	300	602								
339	571	314	508	238	722	330	519								
349	538	344	456	260	592	360	470								
359	515	374	428	288	505	388	436								
367	498			325	447										
				354	421										

[a]Furnace heating controls turned off; door opened to room atmosphere.

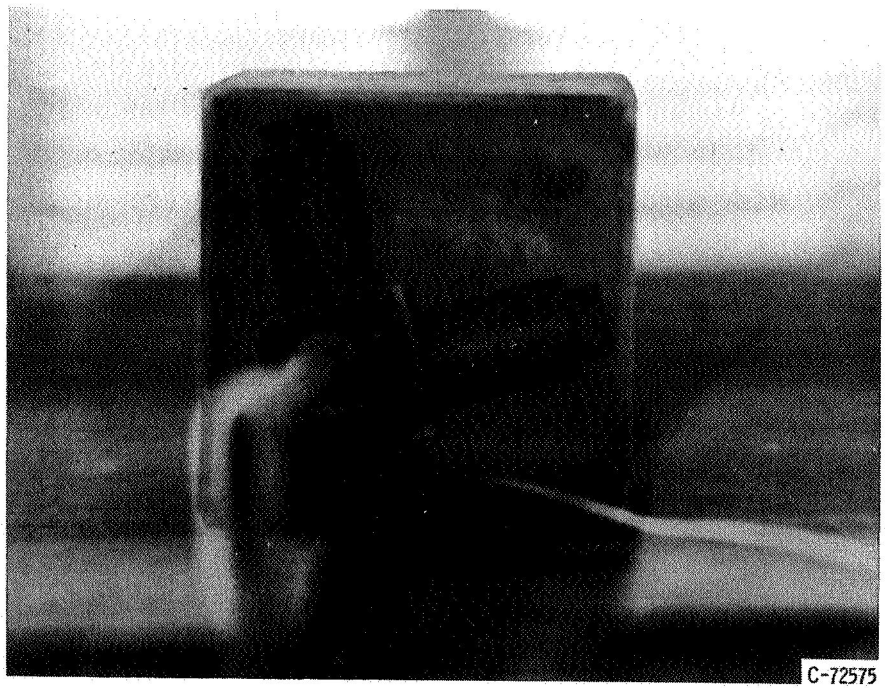


Figure 10. - Test section at 1350° F (1005° K) prior to thermal shock.

6 thermal and pressure cycles, the basic design concept of the viewing device is believed to be adequate for use in liquid-metal systems having boilers requiring outlet pressures and temperatures of approximately 300 psia (2.0×10^6 N/m^2) and 1300° F (1005° K), respectively.

CONCLUDING REMARKS

Tests were conducted to determine the feasibility of the use of viewing devices in both a mercury condensing tube and a mercury boiler outlet tube. Operation conditions for the mercury condensing tube were 15 psia (10^5 N/m^2) pressure at 670° F (628° K) and, for the mercury boiler outlet tube, 90 to 350 psig (6.2×10^5 to 2.4×10^6 N/m^2) pressure at 1350° F (1005° K).

Vacuum-vibration tests on the condensing tube viewing device indicated its ability to withstand 20 g's random vibration typical of a rocket launch. The condensing tube viewing device also withstood dynamic tests consisting of a mercury environment at a temperature of 670° F (628° K) and a pressure of approximately 15 psia (10^5 N/m^2). No leakage was detected, a result attributed to the proper selection of the sealant between the metal tube and its borosilicate glass insert.

The boiler outlet viewing device was successfully tested statically up to pressures of 300 psig (2.0×10^6 N/m^2) for 6 severe thermal cycles. Leakage across the sapphire window-molybdenum tubing brazed joint occurred during the seventh cycle at a test pressure of 350 psig (2.4×10^6 N/m^2). Recommendations to eliminate this leakage include using a thicker sapphire window and a thicker flange section of the molybdenum tubing at the brazed joint. A slight discoloration and a molybdenum oxide deposit was found on the surface of the sapphire window. This problem might be overcome by first plating the tubular sections with nickel and then with a layer of chromium.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 20, 1967,
170-04-00-02-22.

APPENDIX - EVALUATION OF SEALANTS FOR DYNAMIC TESTS

After a literature survey six commercial sealants were selected as being most promising for the selected test conditions. Selection criteria were elevated temperature properties, thermal expansion coefficients, and bonding and sealing coefficients. All sealants selected for test were epoxide based compounds. Other sealants equally suitable may exist, but were not revealed by the literature search.

Ceramic-base sealants were not considered in this evaluation, because they might not withstand the vibrations during dynamic testing. In addition, excessive strains would occur in the ceramic-base sealants during cooling from elevated temperatures. These strains, due to differential thermal contraction of the viewing glass, sealing joint, and viewing glass case, would cause the ceramic-base sealant to leak.

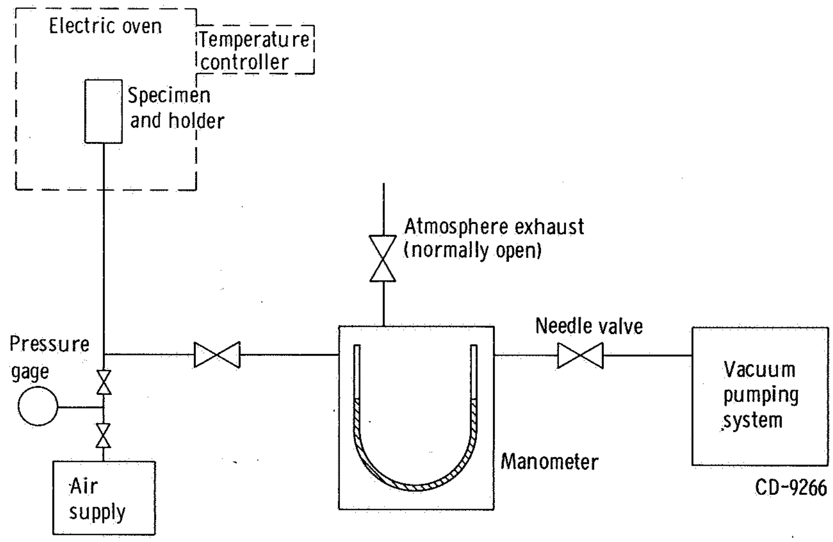
Static Test Apparatus

A schematic of the static test apparatus used for a sealant evaluation testing is shown in figure 11(a). The system consisted of a specimen holder, an electric oven, and a vacuum system with associated valving and gages. Test instrumentation consisted of a mercury U-tube manometer, a compound pressure-vacuum gage, and an oven temperature controller. Specimen holders and borosilicated glass inserts were fabricated for each of the six sealants (see fig. 11(b)).

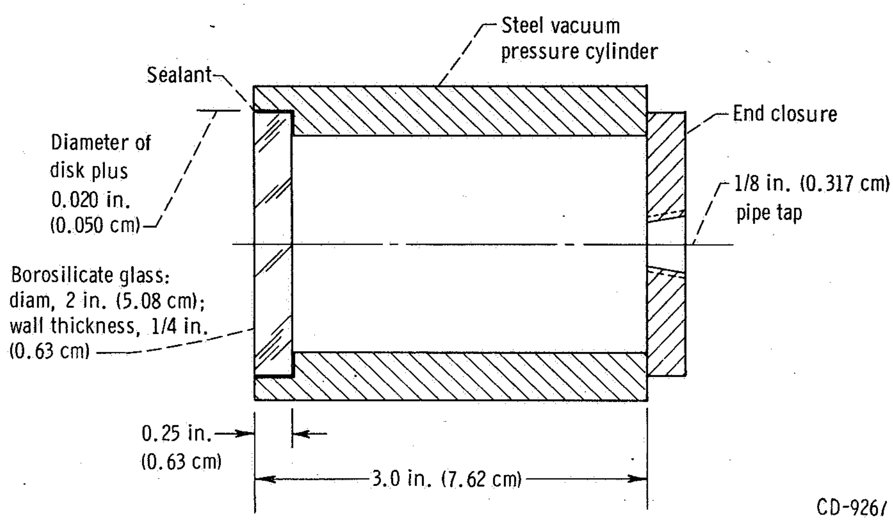
Static Test Procedure

The six sealant specimens were installed in their respective holders in accordance with the manufacturer's instructions. These instructions required that the bonding surfaces be free of oil, grease, and foreign matter. Initial abrasive roughening of the bonding surfaces to enable a better bond and a final rinse with a solvent were also required. The required curing conditions varied from 16 hours at ambient temperature to several increments of 3 hours each from the recommended elevated temperature down to ambient temperature.

After each specimen was placed in the test rig, the vacuum pump was activated before oven temperature was increased to a design temperature of 670^{o} F (628^{o} K). However, the rate of temperature increase was not controlled. As the temperature was increased, vacuum level readings were taken every 10 minutes to determine leakage rate if any. The oven temperature was maintained at 670^{o} F (628^{o} K) for approximately 20 minutes before decreasing the temperature to ambient condition. Vacuum level readings were



(a) Schematic of test setup.



(b) Specimen holder used for preliminary sealant evaluation.

Figure 11. - Vacuum pressure heat system for sealant test evaluation.

also taken during the cooldown period to detect leakage due to differential contraction between the sealant and the bonded parts. A final vacuum reading was taken at room temperature.

The sealants were also subjected to a pressure test. The system shown in figure 11(a) was pressurized with air to approximately 20 to 30 psig (1.3×10^5 to 2.0×10^5 N/m^2). The system was maintained at the desired pressure level to determine if leakage would develop as a result of internal pressurization (reverse loading).

Seal Test Results

The sealants tested in this investigation and their general properties are as follows:

Test 1 - Sample A: A one-component conductive epoxide-based adhesive useful in bonding applications at temperatures up to 500° F (533° K).

Test 2 - Sample B: An epoxide adhesive capable of bonding many materials. Continuous use at temperatures up to 500° F (533° K) and short period usage at 600° F (588° K).

Test 3 - Sample C: A two-part high temperature epoxide adhesive useful for bonding metals, ceramics, and plastics at temperatures in excess of 500° F (533° K).

Test 4 - Sample D: A high-temperature epoxide compound for adhesive to metals, glass, and plastic materials for temperatures up to 500° F (533° K) and for short periods to 600° F (588° K).

Test 5 - Sample E: An epoxy resin useful for patch or repair in common household and automotive hardware. When properly cured, this epoxy resin has a bond shear strength in excess of 1000 psi (6.8×10^{6} N/m^{2}).

Tests 6A and 6B - Sample F: A pourable resin that, when primed with an additive recommended by the manufacturer, can adhere to many materials for prolonged periods at temperatures to 600° F (588° K).

Results of the sealant tests are summarized in table III. All sealant specimens, with the exception of Sample F, failed to maintain the initial vacuum during the first thermal cycle. The pressurization tests performed after the vacuum-temperature tests confirmed the leakage across the sealants.

Test 6B (a rerun of test 6A) on the Sample F sealant was conducted to establish the reliability of the sealant. This sealant specimen maintained the initial vacuum level from room temperature to 720° F (655° K) which is 50° F (27° K) above the design level. A slight leakage (lowest rate obtained) developed as the temperature was raised to 740° F (666° K).

TABLE III. - SEALANT TEST RESULTS

Test number	Sealant	Vacuum-heat run								Pressure run			Remarks
		Vacuum		Recommended maximum temperature		Failure temperature		Leak rate		Measurement		Leakage	
		torr	kN/m^2	^oF	^oK	^oF	^oK	torr/min	$(N/m^2)/min$	psig	kN/m^2		
1	Sample A one-component epoxide	731.5	97.5	595	586	610 - 630	594 - 605	2.28	3.0×10^2	20	137	Yes	Vacuum held at room temperature; minute leak and visible crack in bond
2	Sample B one-component epoxide	734.0	97.8	440	500	480 - 530	522 - 550	40.64	5.4×10^3	30	206	Yes	Minute leak in bond
3	Sample C two-component epoxide	-----	----	---	---	---------	--------	------	--------	--	---	---	Sample failed in curing cycle; glass cracked around edges
4	Sample D epoxide resin	736.6	98.2	470	516	510 - 550	538 - 561	152.4	2.0×10^4	20	137	Yes	Large crack adjacent to glass; bond material pulled from disk
5	Sample E epoxide resin	739.1	98.5	625	602	650 - 660	616 - 622	2.54	3.3×10^2	30	206	Yes	No visible crack
6a	Sample F RTV silicone rubber	736.6	98.2	670	627	---------	--------	------	--------	20	137	No	No leakage; specimen untouched
6b	Sample F RTV silicone rubber	734.0	97.8	670	627	720 - 740	655 - 666	1.01	1.3×10^2	20	137	Yes	Minute leakage through bond

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