ALO 2832-46
UC-33, Propulsion Systems
and Energy Conversion

AEC Research And
Development Report

UNION CARBIDE CORPORATION
LINDE DIVISION

EVALUATION OF A MOLYBDENUM FOIL - QUARTZ CLOTH
INSULATION SYSTEM AFTER 18,000 HOURS AT A
SOURCE TEMPERATURE OF $\sim 1100^{\circ}\text{C}$

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LINDE DIVISION

EVALUATION OF A MOLYBDENUM FOIL - QUARTZ CLOTH
INSULATION SYSTEM AFTER 18,000 HOURS AT A
SOURCE TEMPERATURE OF $\sim 1100^{\circ}\text{C}$ (2012°F)

JUNE 1973

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A B S T R A C T

This report presents the results of the molybdenum foil-quartz cloth insulation system stability test performed by Linde Division of Union Carbide. The test and post test analysis were performed in the duration from December 1970 to May 1973.

The objectives of this program are to evaluate and demonstrate long term thermal performance stability of multi-layer insulation at source temperatures up to 1100°C (2012°F) for use in radioisotope power systems.

Two multi-layer insulation systems were selected for long term stability testing at a source temperature of 1100°C in high vacuum.

(1) Molybdenum Foil-Quartz Cloth

This test system was maintained at stability test conditions for 18,020 hours. Upon completion of this duration the system was disassembled for post test analysis. The results of the ensuing evaluation are documented in this report.

(2) Molybdenum Foil-Zirconia Cloth

This system was at stability test conditions for 10,000 hours, the results of the post test analysis are presented in report ALO 2832-45.

The information contained in this report should be supplemented with report ALO 2832-44 of May 1971.

INTRODUCTION

The objective of the current program is to demonstrate physical and thermal stability of multi-layer insulation systems at a maximum temperature of 2012°F for long term applications.

In order to provide stable long term thermal performance, insulation material, mechanical, chemical and physical stability becomes imperative. However, it is well known, that thermal and physical properties change with increase in temperature and also the reactivity between materials increases. The selection of insulation materials, thus, becomes quite critical. The materials chosen should not only be independently stable (low vapor pressure, etc.) but should also be compatible with other materials in the insulation system.

From the results of prior testing and a review of other materials available molybdenum foil-quartz cloth and molybdenum foil-zirconia cloth systems were selected for testing at the 2012°F source temperature.

The molybdenum foil-zirconia cloth insulation stability test was completed in September 1972. The results are presented in ALO 2832-45.

The molybdenum foil-quartz cloth insulation stability test and the post test evaluation have been completed. This report documents the results of the evaluation. The system had been subjected to a source temperature of ~ 2012°F and a cold side temperature of 230°F. The test duration objective was 20,000 hours however because of heater failure the test was terminated at 18,020 hours. The vacuum level ranged from an initial value of 3×10^{-7} torr to 2×10^{-9} torr at the completion of the test. As part of the post test analysis material samples were removed for metallurgical evaluation.

This effort is being performed under the direction of the United States Atomic Energy Commission Isotope Power Systems Branch, Division of Space Nuclear Systems. The AEC program director is P. A. O'Riordan at Washington, D.C.

The Linde project manager is D. J. Webster. Other Union Carbide personnel contributing to this test report are Dr. C. Angell, G.M. Faulring, Dr. W. Forgeng, J. Kern, D. H. Stellrecht and N. R. Wertz.

CONCLUSIONS

This test demonstrated thermal performance stability of a static molybdenum foil-quartz cloth insulation system operating between boundary temperatures of 2012°F and 230°F. A summary of the results of this test and the molybdenum foil-zirconia cloth system tests are tabulated in the table on top of the next page.

SYSTEM	% CHANGE FROM INITIAL VALUE	DURATION AT STABILITY TEST CONDITIONS	K $\left. \begin{array}{l} 2012^{\circ}\text{F} \\ 100^{\circ}\text{F} \end{array} \right\}$	TEST BOUNDARY TEMP.	
				HOT WALL	COLD WALL
	- DENOTES IMPROVEMENT	HOURS	BTU/HR FT ² F	°F	°F
Molybdenum- Quartz		0	2.12×10^{-3}	2007	233
	-7.1%	18,020	1.97×10^{-3}	2040	228
Molybdenum- Zirconia		546 *	3.07×10^{-3}	2006	396
	-3.6%	10,000	2.96×10^{-3}	2017	389

* System did not stabilize until 546 hours at test conditions.

2. The primary factor which facilitated stable thermal conductivity in the insulation package was that foil reflectance values of the molybdenum layers did not change significantly.

There was indication of deterioration in the molybdenum foil mechanical strength. This was evidenced by recrystallization in the hottest layers and hence some embrittlement⁽¹⁾. These layers also exhibited thermal etching and grooving at the grain boundaries.

The hottest quartz layers also indicated deterioration in mechanical strength. This was noted in the form of embrittlement and loss of strength. This occurs because at sustained temperatures of 1900°F or above the amorphous quartz begins to change into a crystalline form known as Cristobalite. Apparently this change did not degrade the system thermal performance.

PROCEDURE

A number of techniques were utilized in the investigation of the vacuum insulation test package. They included:

1. Determination of the effective thermal conductivity of the vacuum insulation.
2. Visual examination of molybdenum and quartz insulation layers at low magnifications (to 60X).
3. Micro-examination of samples of molybdenum and quartz layers at magnifications to 1240X.

4. X-ray diffraction analyses of selected layers of quartz.
5. Electron probe scans of selected quartz layers.
6. Examination of selected quartz layers via X-ray image.
7. Reflectance measurements on various molybdenum layers from the test package.

RESULTS/OBSERVATIONS

1. Temperature measurements which permitted calculation of the effective thermal conductivity of the insulation test package were made while the test was in progress. Figure 1 (3) shows a plot of the effective thermal conductivity as a function of source temperature. As source temperature was increased thermal conductivity increases smoothly until the stability test hot face temperature of $\sim 2012^{\circ}\text{F}$ was reached. Performance was stable for the entire 18,000 hour duration. After the testing was concluded, a calibration of the hot wall thermocouple indicated that it was reading 26°F low. Based on this calibration, hot wall temperature at the end of the stability test was 2040°F . The effective thermal conductivity at the 18,000 hour point was:

$$K \int_{100^{\circ}\text{F}}^{2012^{\circ}\text{F}} = 1.97 \times 10^{-3} \text{ Btu/Hr-Ft-}^{\circ}\text{F} \quad (3)$$

The above value is corrected for a 100°F cold wall. This value is 7.1% lower than indicated at the beginning of the test. (3) However, it may be unrealistic to report performance changes of less than $\pm 10\%$ since the test error band is probably of the same order. To minimize testing error thermocouples were calibrated prior to obtaining the initial data and immediately after recording the final data. After completing the stability test, post test conductivity data was obtained at three source temperatures from 2000°F down to 1600°F as shown in Table IV and Figure 1. Except for some slight improvements the performance at source temperature below 2000°F was similar to the results noted prior to the stability test exposure.

2. Sections were cut from the side of the test package for metallurgical examination. The locations of these sections are shown in Figure 2. The arrow numbered "1" in this figure indicates the position of the set removed for microscopic examination, and the arrow numbered "2" indicates the position of the set removed for visual examination. Set 1 was also given a cursory visual examination, and no significant difference was noted between the two sets.

A visual examination of the sample set was performed at magnifications up to 60X. Various trends were observed in the layers of the test package. The results of the visual examination are tabulated in Table I.

Figure 3 shows the sample set that was cut out of the test package. Figure 3a shows the condition of layer no. 1, the hottest Mo layer. The surface is clean and the foil is intact. Very few of the Mo foil layers showed any signs of discoloration due to oxidation, and on those that did there were only a few small spots. In Figures 3b and 3c it can be seen that the quartz cloth is brittle and fragmented. This was the general condition of the hottest quartz layers. Figure 4 shows the difference between the hottest and coldest quartz cloth layers and an intermediate layer. The fragmentation of the hottest layer can be seen in Figure 4a, and in 4b and 4c it can be seen that there was virtually no difference in the appearance of the intermediate and cooler layers.

3. Following the visual examination of the molybdenum foils and quartz cloths at low magnifications, selected layers were subjected to metallographic examination at magnifications up to 1240X. Examination of the quartz cloth layers revealed that the surface of the hottest layers had small particles and pieces of broken fiber on them, as shown in Figures 5a and 5b. The pieces of fiber are from the cloth, having broken off due to handling after high temperature embrittlement. Figures 5c and 5d show cooler layers of the package, with layer 108, shown in 5d, being the coldest layer of the quartz cloth. Both of these layers are very clean.

Figures 6a through 6e show the quartz cloth at a higher magnification. These photomicrographs indicate the presence of microscopic particles. The particles were later identified as being molybdenum bearing. At temperatures above 1300°F to 1400°F, molybdenum forms a volatile, non protective oxide film.⁽¹⁾ The volatile oxide may then condense on the quartz cloth, and on the cooler layers of molybdenum. A comparison of Figures 6a through 6e also shows a difference in the physical appearance of the fibers in the hottest layers and the intermediate layers.

Particles were also observed in the cooler layers, as indicated in Figures 7a through 7c which show quartz cloth layer number 83 of the test package. These particles are also molybdenum oxide, however they are much smaller than those found in the hotter layer.

As was mentioned previously, there was evidence of only very small quantities of oxides present. This was because of the care taken during the test to prevent inadvertent introduction of oxygen into the test package. The source of the oxides was probably from surface oxides already present on the molybdenum foil.

4. Various layers of the molybdenum foil were also subjected to micro-examination. Figure 8 shows layer number 1 of the foil, which was the hottest layer. Extensive grain nucleation can be seen and thermal etching is apparent. Molybdenum layer number 3, which is shown in Figure 9, also exhibited extensive grain nucleation and some grain growth.

The occurrence of recrystallization in these and subsequent "hot" layers of molybdenum caused a loss in ductility. This is because upon recrystallization, oxides in the metal segregate at the grain boundary. The loss in ductility is particularly apparent with bending.

Figure 10 shows molybdenum layer number 7. Grain nucleation is still apparent, and there is a "terracing" of the surface also evident. This is a result of the volatilization of oxides, which vary in thickness, from the molybdenum foil surface. These oxide films may vary in thickness because of any one of the following reasons: accessibility, grain orientation, topographical variations or local differences in chemistry. The terracing occurred on what appear to be large grains. However, these are more likely part of the original wrought structure. This is most clear in the area indicated by the arrow. The presence of surface deposits is also evident in this figure.

Figures 11 and 12 show layers 9 and 17 of the molybdenum foil respectively. It can be seen that there is an increasingly smaller amount of recrystallization than in the previous layers of the foil. A relatively large amount of terracing is again evident in Figure 11; in Figure 12 there is relatively little. Very few surface deposits are evident.

Figure 13 shows molybdenum layer number 25 from the test package. There is evidence of only small amounts of the nucleation stage of recrystallization, as indicated by the arrows. These nuclei occur at mechanical twins, which are sites of high strain energy. Mechanical twins are lattice deformations resulting from plastic deformation processes; energy has to be supplied to form a twin. It is apparent that the degree of thermal etching and the amount of recrystallization decreases as the layers get cooler.

Some signs of grain nucleation can also be seen in Figures 14 and 15, which show molybdenum layers 33 and 56 respectively. Also evident in these figures are small surface deposits. These are probably molybdenum oxides which volatilized from hotter layers and condensed on these layers.

5. In Figure 16, which shows molybdenum layer number 80, there is no evidence of any recrystallization. Surface deposits, probably of the same type and source as above, are apparent. Figure 17 shows molybdenum layer number 105, which is one of the coldest layers of the package. There are only a few small surface deposits, and the appearance is very similar to that of an unused foil.

Figure 18 shows molybdenum layer number 109, the coldest layer in the package. Compare this to Figure 19, which shows an unused sample of the foil. It is apparent that the cooler layers of foil in the test package are very similar to the unused foil. The condition of the molybdenum foil, and also the quartz cloth, in the colder layers was very good. The molybdenum layers remained shiny and ductile, and the quartz cloth remained flexible and strong, exhibiting no apparent change to Cristobalite.

6. After micro-examination, selected layers of quartz cloth were subjected to electron microprobe analysis in order to detect and identify any molybdenum-bearing deposits on the fibers. The results of these scans are shown in Figures 20a through 20f. The first picture in each pair is an electron image of the cloth, and the second is an X-ray scan of the same area for molybdenum. As can be seen from the figures, most of the particles are molybdenum-bearing deposits. Some of the particles are not molybdenum-bearing, as indicated by the arrows in Figure 20c. These are probably dust particles.

7. Several layers of quartz were also subjected to X-ray diffraction analysis at the UCC - Mining and Metals Division. The results of this analysis are tabulated in Table II, and also shown in Figures 21a through 21i. The crystalline nature of the hottest layers is evident from Figures 21a, 21b, and 21d, which are from layers 2, 4, and 12 respectively. The basically amorphous nature of the intermediate and cooler layers is shown by Figures 21e through 21i. Layer number 8, corresponding to figure 21c, is one of the hotter layers, but is only slightly crystallized which is unusual.

8. The surfaces of the molybdenum layers were similar throughout the package. Some of the hotter foils had a dull or frosty look, or dull spots. Thermal etching and thermal effects on the rolling texture left many of the hotter layers quite wrinkled. Reflectance measurements were performed on selected layers of molybdenum from the test package. Table III tabulates the results of these measurements. It should be noted that the values listed in Table III are all relative to an as received molybdenum foil sample which was assumed to have a reflectance of 100%. There was little decrease in the reflectivity of any of the foils, with the maximum decrease being 4%.

9. The test package was heated using a platinum-10% rhodium wire heater, which is shown in Figures 22a and 22b. The discoloration, which can be seen running lengthwise on the heater in both figures, was a blue stain. This stain is probably due to deposits of molybdenum oxides. In Figure 22b, bright spots can be seen on the wire. These are the result of coarsening of the grains in the platinum wire, and led to failure of the wire after 18,020 hours of the proposed 20,000 hour test.

DISCUSSION

In previous studies, involving a molybdenum-zirconia vacuum insulation system, large scale oxidation of the molybdenum and deposition of these oxides on the surface of cooler molybdenum surfaces led to deterioration of the insulation system. The source of the oxygen was the zirconia cloth which becomes oxygen deficient at conditions of high temperature and vacuum. No problems of this type were encountered with the molybdenum-quartz vacuum insulation system, and the thermal conductivity of this system was about 30% lower than that of the molybdenum-zirconia system.

The molybdenum in general remained ductile, which was desirable. In the hottest layers there was some recrystallization, but not enough to cause severe embrittlement as occurred in some previous studies.

The hottest layers of the quartz cloth became embrittled due to a transformation to Cristobalite. This did not affect their thermal insulating properties however.

The static performance of the molybdenum-quartz vacuum insulation system was considered to be very satisfactory. The thermal conductivity of the system was stable over the duration of the 18,000 hour test.

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3. Experimental Data

TABLE I
RESULTS OF VISUAL EXAMINATION OF INSULATOR
LAYERS FROM SIDE OF TEST PACKAGE

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
1	Mo foil	Clean, dull, wrinkled, creases when bent
2	SiO ₂ Cloth	Very brittle, fractured in many pieces
3	Mo foil	Same as for 1, slightly more brittle
4	SiO ₂ Cloth	Very brittle, several large pieces
5	Mo foil	Clean, brittle, dull with some shiny spots, weave texture of SiO ₂ cloth visible in some areas
6	SiO ₂ Cloth	Brittle, one piece
7	Mo foil	Creases when bent, texture of SiO ₂ cloth visible. Clean "hot" side mostly shiny with some dull areas, "cold" side shiny. Wrinkled texture also evident.
8	SiO ₂ Cloth	Brittle, two pieces
9	Mo foil	Similar to No. 7, SiO ₂ Cloth texture not visible
10	SiO ₂ Cloth	Same as for No. 8
11	Mo foil	Creases when bent in rolling direction, SiO ₂ cloth texture slightly visible in some areas. Clean and shiny, with some dull spots on "hot" side
12	SiO ₂ Cloth	One piece, less brittle
13	Mo foil	Similar to No. 11, more brittle, SiO ₂ cloth texture more visible

10.
TABLE I - continued

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
14	SiO ₂ Cloth	Similar to No. 12
15	Mo foil	Shiny, still slightly brittle, wrinkled texture
16	SiO ₂ Cloth	Slightly brittle
17	Mo foil	Similar to No. 15
18	SiO ₂ Cloth	Similar to No. 16
19	Mo foil	Similar to No. 15
20	SiO ₂ Cloth	Similar to No. 16, slightly less brittle
21	Mo foil	Clean, shiny, slightly brittle, wrinkled
22	SiO ₂ Cloth	Similar to No. 20
23	Mo Foil	Similar to No. 21
24	SiO ₂ Cloth	Slightly more flexible
25	Mo foil	Some SiO ₂ fibers on surface, dull spots on "hot" side, creases when bent
26	SiO ₂ Cloth	More flexible, but still slightly brittle, some powder on surface
27	Mo foil	Shiny, slightly more brittle than No. 25
28	SiO ₂ Cloth	Similar to No. 26
29	Mo foil	Ductile, shiny with some dull spots, some small, discolored spots visible at 15 X (MoO ₂)
30	SiO ₂ Cloth	Flexible
31	Mo foil	Shiny, creases when bent, SiO ₂ fibers on surface
32	SiO ₂ Cloth	Flexible

TABLE I - continued

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
33	Mo foil	Shiny, some dull spots on "hot" side
34	SiO ₂ Cloth	Similar to No. 32
35	Mo foil	Similar to No. 33, fairly ductile, some small spots of discoloration (MoO ₂)
36	SiO ₂ Cloth	Flexible, powder on surface, tears easily
37	Mo foil	Shiny, some dull spots on "hot" side, dull spots show SiO ₂ weave, fibers on surface
38	SiO ₂ Cloth	Flexible
39	Mo foil	Similar to No. 37, no fibers, creases when bent
40	SiO ₂ Cloth	Similar to No. 38
41	Mo foil	Similar to No. 39, slightly more ductile
42	SiO ₂ Cloth	Similar to No. 38
43	SiO ₂ Cloth	Similar to No. 38, improved strength
44	Mo foil	Ductile, shiny, small spots of discoloration (MoO ₂), clean
45	SiO ₂ Cloth	Similar to No. 43
46	Mo foil	Shiny, clean, ductile
47	SiO ₂ Cloth	Flexible, good strength
48	Mo foil	Similar to No. 46, a few small fibers on surface
49	SiO ₂ Cloth	Flexible, good strength
50	Mo foil	Shiny, ductile, small dull spots on surface (60X)

TABLE I - continued

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
51	SiO ₂ Cloth	Similar to No. 49
52	Mo foil	Shiny, ductile, a few small SiO ₂ fibers on surface
53	SiO ₂ Cloth	Similar to No. 49
54	Mo foil	Shiny, clean, ductile
55	SiO ₂ Cloth	Flexible, even better strength
56	Mo foil	Similar to No. 54
57	SiO ₂ Cloth	Similar to No. 55
58	Mo foil	Similar to No. 54
59	SiO ₂ Cloth	Similar to No. 55
60	Mo foil	Similar to No. 54, very clean
61	SiO ₂ Cloth	Similar to No. 55
62	Mo foil	Similar to No. 60
63	SiO ₂ Cloth	Slight bunching of fibers in some spots, good strength, flexible
64	Mo foil	Ductile, shiny, clean. There is an additional small piece of foil here due to overlapping. Small holes in overlap correspond to spots of fiber bunching in layer No. 63. Wrinkling becoming less pronounced
65	SiO ₂ Cloth	Flexible, good strength
66	Mo foil	Similar to No. 60
67	SiO ₂ Cloth	Flexible, good strength
68	Mo foil	Ductile, clean, shiny
69	SiO ₂ Cloth	Similar to No. 67

TABLE I - continued

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
70	Mo foil	Similar to No. 68
71	SiO ₂ Cloth	Similar to No. 67
72	Mo foil	Similar to No. 68
73	SiO ₂ Cloth	Similar to No. 67
74	Mo foil	Ductile, very clean, shiny
75	SiO ₂ Cloth	Flexible, very strong
76	Mo foil	Similar to No. 74
77	SiO ₂ Cloth	Similar to No. 75
78	Mo foil	Similar to No. 74
79	SiO ₂ Cloth	Similar to No. 75
80	Mo foil	Similar to No. 74
81	SiO ₂ Cloth	Similar to No. 75
82	Mo foil	Shiny, ductile, very clean
83	SiO ₂ Cloth	Flexible, very strong
84	Mo foil	Similar to No. 82
85	SiO ₂ Cloth	Similar to No. 83
86	SiO ₂ Cloth	Similar to No. 83
87	Mo foil	Shiny, ductile
88	SiO ₂ Cloth	Flexible, good strength
89	Mo foil	Similar to No. 87
90	SiO ₂ Cloth	Similar to No. 88
91	Mo foil	Similar to No. 87
92	SiO ₂ Cloth	Flexible, good strength

TABLE I - continued

<u>Layer Number</u>	<u>Description</u>	<u>Observations</u>
93	Mo foil	Shiny, ductile, very clean
94	SiO ₂ Cloth	Similar to No. 92
95	Mo foil	Similar to No. 93
96	SiO ₂ Cloth	Similar to No. 92
97	Mo foil	Shiny, ductile
98	SiO ₂ Cloth	Flexible, strong
99	Mo foil	Similar to No. 97
100	SiO ₂ Cloth	Similar to No. 98
101	Mo foil	Shiny, clean, ductile, wrinkles less prominent
102	SiO ₂ Cloth	Similar to No. 98
103	Mo foil	Similar to No. 101
104	SiO ₂ Cloth	Strong, flexible
105	Mo foil	Similar to No. 101
106	SiO ₂ Cloth	Similar to No. 104
107	Mo foil	Shiny, ductile, clean, wrinkled texture barely evident
108	SiO ₂ Cloth	Similar to No. 104
109	Mo foil	Similar to No. 107

NOTES: (1) See Figure 2 for location of this set of samples in the test package

(2) Visual examination was performed at magnifications up to 60X

TABLE II
RESULTS OF X-RAY DIFFRACTION ANALYSIS OF SAMPLE
LAYERS OF SiO_2

<u>Layer Number</u>	<u>Results</u>	<u>Comments</u>
2	Primarily cristobalite* (well crystallized)	Hottest layer
4	Primarily cristobalite (not as well crystallized as No. 12)**	
8	Primarily amorphous, small amount of cristobalite	
12	Primarily cristobalite (well crystallized)**	
20	Primarily amorphous, indication of cristobalite	
36	Primarily amorphous indication of cristobalite	
57	Primarily amorphous, indication of cristobalite	
83	Primarily amorphous, indication of cristobalite	"Hot" side
108	Primarily amorphous, indication of cristobalite	Coldest layer

* The degree of crystallinity, refers to the crystallite size or periodicity of the SiO_2 atoms.

** The fact that layer #12 is more crystallized than layer #8 may indicate different lots of SiO_2 .

TABLE III

Results of Reflectance Measurements on Samples of Mo Layers

<u>Layer Number</u>	<u>Side</u>	<u>Absorbance</u>	<u>Reflectance %</u>
1	Hot	.005	99
1	Cold	.008	98.2
3	Hot	< 0	100
3	Cold	< 0	100
7	Hot	< 0	100
7	Cold	< 0	100
11	Hot	.015	96.6
11	Cold	0.10	97.6
19	Hot	.008	98.2
19	Cold	.018	96
35	Hot	< 0	100
35	Cold	< 0	100
58	Hot	.009	98
58	Cold	< 0	100
82	Hot	< 0	100
82	Cold	< 0	100
107	Hot	< 0	100
107	Cold	< 0	100

NOTES: (1) The samples were measured on a Lang 14 Spectrophotometer with Lang 1411 Reflectance attachment.

(2) Data:

IR source: tungsten lamp
 IR detector: PbS cell
 Wavelength: 1.2 microns (12,000 Å)
 Slit 2.2 mm

(3) Zero adjustment was made so that an unused sample of Mo foil versus an unused sample of Mo foil read zero. All other samples were measured against the unused Mo sample as a reference. Therefore, all values tabulated in the Table are relative to an unused Mo foil, which was assumed to have adequate surface properties prior to thermal treatment.

TABLE IV

INSULATION SYSTEM CONDUCTIVITY TEST RESULTS
AFTER 18,000 HOUR STABILITY TEST

TEMPERATURE, °F		Q, BTU/HR				EFFECTIVE THERMAL CONDUCTIVITY ** BTU/HR FT°R X 10 ⁻³
HOT SIDE	COLD SIDE	TOTAL INPUT	LOSSES *		Q THROUGH INSULATION	
			ELECTRICAL	THERMAL		
End of 18,000 hour 2040	228	180.3	3.9	24.75	151.7	2.24
System Calibration After 18,000 hour						
a) 2031	235	181.0	3.95	24.25	152.8	2.27
b) 1922	213	140.7	2.35	22.75	115.6	1.81
c) 1649	171	81.3	1.46	19.0	60.8	1.10

17.

* Includes the electrical losses through the heater leads and the thermal losses down the heater and thermocouple leads.

** The effective thermal conductivity is based on the equation;

$$[K] \text{ effective} = \frac{[Q] \text{ through insulation}}{S [T_{\text{hot side}} - T_{\text{cold side}}]}$$

$$\text{where } [S] \text{ Insulation Shape Factor} = \left[\frac{\text{Area}}{\text{Thickness}} \right] \text{ Insulation}$$

FIGURE 1

STABILITY TEST THERMAL CONDUCTIVITY DATA
CYLINDRICAL TESTING

Figure 2. Shown above is the test package which was stability tested at a source temperature of approximately 2000°F for 18,000 hours. The arrows indicate locations where the test package was sectioned for metallurgical examination. The section numbered 1 was used to provide samples for photomicrographs, the section numbered 2 provided samples for macroexamination. The white areas are the high purity alumina tube which was used as a core for the package.

Mag. Approx. 1/10X

Figure 3a

Mag. 1X

Figure 3b

Mag. 1X

Figure 3c

Mag. 1X

The above photomicrographs show the section that was removed from the test package. Figure 3a shows layer 1, the hottest layer of Molybdenum foil. Note that the foil is intact and has a rather good surface appearance. Figures 3b and 3c show layers 2 and 4 respectively, the hottest and next-hottest layers of quartz cloth. It can be seen that in layer 2 (3b), the cloth is fragmented quite badly. In layer 4 (3c) the cloth is again fragmented, but to a lesser extent.

Figure 4. The above photomicrograph shows samples of the quartz cloth after thermal treatment for 18,000 hours. These samples were obtained after sectioning of the test package at position 2 shown in Figure 2. The hottest layers become very brittle and fragmented, while the cooler layers change little in appearance.

Mag. .25X

Figure 5a. This is a photomicrograph of layer 2 of the quartz cloth. Note the light particles on the strands and the broken individual fibers. The square weave of the cloth can also be seen.

Mag. 40X

Figure 5b. This shows layer 8, quartz cloth. Note broken fibers and pieces of broken fiber on the strand (arrows).

Mag. 40X

Figure 5c. This is layer 36 of the quartz cloth. Note the generally clean surface and few broken fibers.

Mag. 40X

Figure 5d. This is layer 108 of the package, the coldest layer of quartz cloth. Note the clean surface and few broken fibers.

Mag. 40X

Figure 6a. Layer No. 2 quartz cloth
Mag. 300X

Figure 6b. Layer No. 4, quartz cloth
Mag. 300X

Figure 6c. Layer No. 8, quartz cloth
Mag. 300X

Figure 6d. Layer No. 12, quartz cloth
Mag. 300X

Figure 6e. Layer No. 36, quartz cloth
Mag. 300X

Shown are photomicrographs of various layers of the quartz cloth which was removed from the hotter end of the package after stability test exposure. Particles are evident on the fibers of the cloth. Note also the change in appearance of the fibers themselves, from hottest to cooler layers.

Figure 7a. Layer No. 83, quartz cloth.
Mag: 550X

Figure 7b. Same as 7a, different position.
Mag: 550X

Figure 7c. Same as 7a, higher magnification.
Mag: 1240X

These photomicrographs are of the hot face of Layer No. 83, from the test package. Note the particles adhering to the fibers of the quartz cloth. These particles were shown to be mainly Molybdenum oxide particles.

26.

Figure 8. Mo layer no. 1 Nucleation stage of recrystallization and thermal etching are apparent.

Mag: 500X

Figure 9. Mo layer no. 3. Grain growth has occurred and again, heavy thermal etching is apparent.

Mag: 500X

Figure 10. Mo layer no. 7. Terracing due to surface volatilization is evident. What appear to be large grains where terracing has occurred are more likely part of the original wrought structure (arrow).

Mag: 500X

27.

Figure 11. Mo layer No. 9

Mag: 500X

Figure 12. Mo layer No. 17

Mag: 500X

Figure 13. Mo layer No. 25. Very early stages of grain nucleation can be seen along what were mechanical twins. The arrows in the photo point towards two such areas. Numerous others particularly in the left corner of the photo are visible.

Mag: 500X

Figure 14. Mo layer No. 33. Early stages of grain nucleation are evident. There are also deposits on the surface which are probably oxides.
Mag: 500X

Figure 15. Mo layer No. 56. Early stages of grain nucleation (arrow) and surface deposits are again apparent.
Mag: 500X

Figure 16. Mo layer No. 80. Large surface deposits are apparent.

Mag: 500X

Figure 17. Mo layer No. 105

Mag: 500X

30.

Figure 18.

Mo layer No. 109

Mag: 500X

Figure 19. Sample of unused Mo foil. Surface appearance was bright and shiny.

Mag: 500X

Electron image of quartz
cloth surface

Mo electron probe scan of quartz
cloth surface. Light spots are
Mo deposits.

Figure 20a. Quartz cloth layer No. 2, electron microprobe

Mag: 1250X

Electron image of quartz cloth
surface

Mo electron probe scan of quartz
cloth surface. Light spots are
Mo deposits.

Figure 20b. Quartz cloth layer No. 8, electron microprobe

Mag: 1250X

Electron image of quartz cloth surface

Mo electron probe scan on quartz cloth surface. Light spots are Mo concentrations.

Figure 20c. Quartz cloth layer No. 12, electron microprobe

Mag: 1250X

Electron image of quartz cloth surface

Mo electron probe scan quartz cloth surface. Light spots are Mo concentrations.

Figure 20d. Quartz cloth layer No. 36, electron microprobe.

Mag: 1250X

Electron image of quartz cloth surface.

Mo electron probe scan on quartz cloth surface. Light spots are Mo deposits.

Figure 20e. Quartz cloth layer No. 57, electron microprobe.

Mag: 1250X

Electron image of quartz cloth surface.

Mo electron probe scan on quartz cloth surface. Light spots are Mo deposits.

Figure 20f. Quartz cloth layer No. 83, electron microprobe.

Mag: 1250X

Shown above are the results of electron microprobes of various layers of the quartz cloth from the test package. The first photo of each pair is an electron image of the quartz cloth. Particles can be seen on the fibers as indicated by the arrows in Figure 20a. The second photo of each pair is an X-ray image of the same area which indicates Mo deposits on the quartz fibers. Note that not all of the particles are Molybdenum bearing (arrows, Figure 20c). These could be dust particles or SiO_2 fragments.

Figure 2la. Quartz cloth layer No. 2, X-ray diffraction.

Figure 2lb. Quartz cloth layer No. 4, X-ray diffraction.

Figure 2lc. Quartz cloth layer No. 8, X-ray diffraction.

Figure 2ld. Quartz cloth layer No. 12, X-ray diffraction.

Figure 2le. Quartz cloth layer No. 20, X-ray diffraction.

Figure 2lf. Quartz cloth layer No. 36, X-ray diffraction.

Figure 2lg. Quartz cloth layer No. 57, X-ray diffraction.

Figure 2lh. Quartz cloth layer No. 83, X-ray diffraction.

Figure 2li. Quartz cloth layer No. 108, X-ray diffraction.

Figure 22a. Platinum -10% Rhodium wire heater which was used for thermal treatment of the insulation package.

Mag: 1/4X

Figure 22b. Same as 21a., higher magnification. The discoloration is a blue stain which is probably due to deposits of MoO_2 . Note also the bright spots on the wire which are reflections from large grains.

ATTACHMENT ITYPICAL MOLYBDENUM FOIL*CHEMICAL ANALYSIS

(Hamilton Job No. 6980)

	<u>Impurities, ppm</u>
Al	4
Ca	2
Cr	1
Cu	4
Fe	9
K	15
Mg	1
Mn	1
N	7
Ni	1
Si	6
Sn	4
W	145

* Foil was manufactured by a powder metallurgy process.

ATTACHMENT IITYPICAL ASTROQUARTZ CLOTH COMPOSITION

(Exclusive of Binder)

TYPICAL CHEMICAL ANALYSISSiO₂ - 99.95% MinImpurities - ppm

FeO	3
Ti	12
Al	100
Mg	2.5
Ca	23
Li	1
Na	5
K	9
P	3
Mn	2
Cu	.1
Sb	.5
Cd	.5
B	10