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RESEARCH AND DEVELOPMENT OF VACUUM
FOIL-TYPE INSULATION
FOR RADIOISOTOPE POWER SYSTEMS

December 1967 - February 1968

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VACUUM FOIL-TYPE INSULATION FOR
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by

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M. L. Paquin, and V. L. Poirier

ABSTRACT

This report is the second Phase II quarterly progress report on the development of high-temperature thermal insulation under Contract No. AT(30-1)-3634 with the U. S. Atomic Energy Commission and covers the work period December 1967 through February 1968.

Thirteen planar insulation samples have been fabricated and eight of these have been tested in the planar heat loss test apparatus. The purpose of these tests is to evaluate the effectiveness of various spray coatings.

Approximately 200 feet of tungsten foil has been chemically etched from a thickness of 1.3 to 1.4 mils to a thickness of .3 to .4 mil. Six-inch wide tungsten foil cannot normally be purchased in thicknesses less than 1 mil.

Slit-cup, overlap, and tapered corner assemblies have been fabricated and tested in the edge heat loss test apparatus. The tapered corner was the most effective.

The compatibility testing of aluminum, copper, nickel, and platinum foil in combination with alumina and zirconia is in progress. After 100 hours, zirconia appears to produce the least bonding between foil layers.

I. INTRODUCTION AND SUMMARY

This report is the second Phase II quarterly report on the development of high temperature thermal insulation under Contract No. AT(30-1)-3634 and covers the work period from December 1967 through February 1968.

The average applied oxide layer thickness has been measured as a function of the size of the oxide particles and the number of spray passes used to apply the coating. In addition, eight planar insulation samples have been fabricated and tested in the planar heat loss test apparatus. The purpose of these tests is to evaluate the effectiveness of various spray coatings. The performance of the samples is substantially better than the performance of the samples tested during Phase I. The improvement is due, in part, to modifications that have been incorporated into the test apparatus.

Approximately 200 feet of W foil has been chemically etched in-house to a thickness of .3 to .4 mil. The as-received tungsten foil varied in thickness between 1.3 and 1.4 mils. To date, it has not been possible to roll 6-inch wide tungsten foil to a thickness of less than .8 mil.

Slit-cup overlap, and tapered corner configurations have been fabricated and tested in the edge heat loss test apparatus. 40 layers of Ta-ThO₂ insulation were used in each assembly. Several attempts were made to improve the performance of the slit-cup corner assemblies but, in the final analysis, the overlap corner was equally as effective and far easier to fabricate. The tapered corner, which is only moderately difficult to fabricate, proved more effective than either the slit-cup or the overlap corner.

The compatibility testing of the foil-oxide combinations under consideration for low temperature insulation assemblies is in progress. Aluminum, copper, nickel, and platinum foil are being studied in combination with alumina and zirconia. The 100-hour samples have been examined and zirconia was found to produce less bonding between adjacent foil layers. The design of the test apparatus that will be used to measure the performance of the low temperature samples has been finalized and its fabrication will begin shortly.

II. COMPLETED SECOND QUARTER WORK

2.1 Task 2. Spray Coating

Subtask 2.1 was completed during the first quarter. Under Subtask 2.2 the average thickness of an oxide coating has been determined as a function of the size of the oxide particles and the number of spray passes used to apply the coating. Subtask 2.3 is investigating the effect of various oxide coatings on the performance of non-load bearing insulation samples while Subtask 2.4 is studying the performance of structurally loaded samples. Subtasks 2.2, 2.3, and 2.4 will each be discussed separately.

Subtask 2.2

The purpose of this subtask is to learn how to accurately predict the thickness of an oxide coating sprayed onto a foil surface. The thickness of a deposited oxide layer is a function of the size of the oxide particles leaving the spray gun and the number of spray passes used to coat the foil. Originally it was felt that, in addition to the original size of the oxide particles, the spray mixture rolling time and the spray gun pressure influenced the size of the particles leaving the gun nozzle. Subtask 2.1, however, established that neither rolling nor spraying changed the size of the oxide particles. Therefore, the only two parameters that influence the deposited oxide layer thickness are the original size distribution of the particles and the number of spray coatings.

Aluminum, molybdenum, and tungsten foils were used in the actual measurements. Three oxide powders were investigated; American Potash thoria Mixtures Nos. 112, 115, and 116. The previous

quarterly discusses each thorium powder and contains the photomicrographs that were used to establish the particle sizes. The following table summarizes the characteristics of each mixture.

American Potash Thorium Mixture	Most Common Particle Sizes	Largest Particle Size
Mixture 115	1 - 2 μ	2 μ
Mixture 116	5 - 10 μ	~20 μ
Mixture 112*	15 - 25 μ	~50 μ

* Slight preprocessing

Table 2.1 American Potash Thorium Powders

The test procedure was as follows. The thickness of a stack of bare foils is measured with an indicator. The spray coating is then applied to each foil and the thickness of the entire stack is then remeasured. The difference between the final stack thickness and the original stack thickness, divided by the number of foils in the stack, is the average oxide layer thickness. Sixteen stacks of foil were measured. Table 2.2 defines each stack and presents the results of the measurements. Figure 2.1 plots the average oxide layer thickness versus the number of spray passes for each of the three thorium powders. The results generally are in accord with the particle size determinations in Table 2.1.

One coat of Mixture 115 thorium particles resulted in an average oxide layer thickness of approximately $1\frac{1}{2}$ microns. Three coats

TABLE 2.2

Sample Number	Foil Material	Thoria Mixture	Number of Layers	Number of Spray Passes	Total Sample Thickness Before Spraying	Total Sample Thickness After Spraying	Total Sample Thickness After Firing	Final Average Oxide Layer Thickness
1	Al	116	50	1	0.072"	0.089"	0.088"	7.9 μ
2	Al	115	50	1	0.071"	0.076"	0.074"	1.5 μ
3	Al	116	50	3	0.072"	0.092"	0.084"	6.1 μ
4	Al	115	50	3	0.073"	0.087"	0.077"	2.0 μ
5	Al	115	50	7	0.071"	0.085"	0.081"	5.1 μ
6	Al	112	50	1	0.072"	0.128"	0.109"	18.8 μ
7	Al	112	50	3	0.074"	0.125"	0.106"	16.3 μ
8	Al	112	50	7	0.073"	0.142"	0.142"	34.0 μ
9	Al	116	50	7	0.072"	0.098"	0.092"	10.0 μ
10	Mo	115	40	1	0.0415"	0.0455"	0.0435"	1.3 μ
11	Mo	116	40	1	0.043"	0.0555"	0.0545"	7.3 μ
12	Mo	112	40	1	0.045"	0.073"	0.069"	15.2 μ
13	Mo	112	40	3	0.0425"	0.0825"	0.081"	24.5 μ
14	Mo	112	40	7	0.045"	0.086"	0.0825"	23.8 μ
15	W	112	40	1	0.044"	—	0.076"	20.3 μ
16	W	112	40	7	0.044"	—	0.0865"	26.9 μ

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Figure 2.1 Average Oxide Layer Thickness vs Number of Spray Passes, American Potash ThO_2 Mixtures 112, 115, and 116

increased this number to only 2 microns, but this is because the applied particle density after only one coat is relatively sparse and even after three coats most particles haven't yet begun to pile on top of one another. In contrast after seven coats the particles apparently have become stacked because the average spacing was 5.1 microns.

A very similar situation was observed with the Mixture 116 thoria particles. The actual data suggest a 1 micron decrease in the average oxide layer thickness when three coats rather than one are applied to each foil surface. If all spray coatings were identical this wouldn't be possible. The observed decrease therefore indicates that slight variations still exist between spray coatings. The average oxide layer thickness resulting from one coat of Mixture 116 particles was approximately $6\frac{1}{2}$ microns. After seven coats the average thickness was 10 microns.

The data obtained after spraying the Mixture 112 thoria particles was somewhat scattered and inconsistent. In one case the average oxide layer thickness after three spray passes was 16 microns while in another case the thickness was 24 microns. This scatter is a direct result of the difficulty associated with spraying the large particles in the Mixture 112 thoria powder. The spray gun clogs more frequently when the binder contains these particles and the composition of the spray jet varies with the total elapsed spraying time. When spraying first starts the spray jet is mostly binder, but gradually the supply of binder is exhausted and a much "drier" jet results. Both effects are caused by the rapid settling of the large, and therefore heavy, thoria particles in the binder. The mixture of binder and particles is contained in a reservoir positioned above the spray gun (Figure 2.2), and

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Figure 2.2 Spray Gun and Binder Reservoir

an electric motor stirs the binder constantly while spraying. The stirring rod extends even into the reservoir sump in an effort to keep the particles in suspension until they enter the spray gun. Apparently, once inside the gun the large particles tend to separate because large deposits of packed thoria accumulate in the gun while spraying.

In an effort to minimize the problems associated with spraying the large thoria particles, up to five times more nitrocellulose was added to the binder mixture. This increased the viscosity of the binder and, to some extent, helped keep the particles in suspension. The clogging and settling problems were alleviated but unfortunately not eliminated. Fuel-air calculations indicated that it would have been dangerous to add even more nitrocellulose to the binder because of the danger of explosion in the spray booth.

Efforts to reach a more basic solution are now in progress. After consultation with the spray gun manufacturer a new nozzle with a larger orifice has been ordered. In addition several new binder ingredients have been ordered in an attempt to blend a more viscous but non-explosive binder.

Subtask 2.3

The purpose of Subtask 2.3 is to investigate the effect the oxide coating has on the performance of unloaded insulation samples. The first two subtasks have established that the size of the oxide particles and the number of spray coatings per layer define the oxide coating. Table 2.3 lists thirteen 40-layer insulation samples that have been fabricated during the second quarter. Samples 1 through 8 were tested in an improved version of the planar heat loss test apparatus that was

TABLE 2.3

Sample Number	Foil Material	Foil Thickness	Number of Layers	Thoria Mixture	Number of Coats	Sample Thickness
1	Mo	.001"	40	115	1	.0435"
2	Mo	.001"	40	116	1	.0545"
3	Mo	.001"	40	112	1	.069"
4	Mo	.001"	40	112	3	.081"
5	Mo	.001"	40	112	7	.0825"
6	Mo	.001"	40	115	7	.0505"
7	W	.001"	40	112	1	.076"
8	W	.001"	40	112	7	.0865"
9	Mo	.001"	40	112	1	.073"
10	W	.001"	40	112	1	.070"
11	W	.001"	40	112	7	NA
12	Ta	.0005"	40	112	1	NA
13	Ta	.0005"	40	*	1	NA

* Zirconia particles were used, not thoria.

NA - Not available as yet.

used throughout Phase I. Samples 9 through 13 have not, as yet, been tested but will be during the next quarter.

The first five samples all consisted of 40 layers of Mo-ThO₂ insulation. The spray coatings, however, differed from sample to sample. Sample Nos. 1, 2, and 3 varied the particle size while holding the number of spray passes constant, while Sample Nos. 3, 4, and 5 varied the number of spray passes while holding the particle size constant.

Figure 2.3 plots the heat flux through Sample Nos. 1, 2, and 3 as a function of the source temperature. Also appearing in Figure 2.3 as a dashed line is the 40-layer Mo-ThO₂ data reported in the Phase I Final Report. A significant improvement in performance is apparent and this is attributed, in part, to the improvements in the test apparatus. Experimentation at the outset of Phase II with a Phase I insulation sample indicated that some heat was flowing radially inward along the foils of the insulation sample. This heat eventually reached the calorimeter, indicating that the heat flux through the insulation was higher than it actually was. The radial heat flow has now been eliminated, accounting for a great deal of the improvement apparent in Figure 2.3. The performance of the sample sprayed with Mixture 116 thoria was slightly better than that of the sample sprayed with the Mixture 115 particles. In view of this result the Mixture 112 sample was expected to be even more effective than the Mixture 116 sample, but this did not prove to be the case. The overall differences in performance from sample to sample were very slight after only one spray pass.

Figure 2.4 compares the performance of Sample Nos. 3, 4, and 5. All three samples consisted of 40 layers of molybdenum foil sprayed

Figure 2.3 Heat Flux vs Source Temperature, Mo-ThO₂ Insulation

Figure 2.4 Heat Flux vs Source Temperature, Mo-ThO₂ Insulation

with the large Mixture 112 thoria particles. Sample No. 3 received one coat per layer, Sample No. 4 received three coats per layer, and Sample No. 5 received seven coats per layer. The performance of Sample No. 5 was consistently better than the performance of Sample Nos. 3 and 4, which were very similar to one another. Therefore, whereas no clearcut advantage was gained by using one coat of any one of the three different size thoria powders, a significant improvement in performance was obtained by applying seven coatings of the large thoria particles to each foil surface rather than one or three coatings.

At this point Sample No. 6 was fabricated, tested, and compared with Sample No. 1. Once again, both samples consisted of 40 layers of molybdenum foil, in this case sprayed with the smallest thoria particles. Sample No. 1 received one coating per layer and Sample No. 6 received seven coatings per layer. Figure 2.5 shows that Sample No. 6 permitted a higher heat flux to reach the calorimeter at every source temperature. In the case of the largest thoria particles seven spray passes per layer proved more effective than one spray pass. In contrast, the opposite was true with the very fine thoria powder. The optimum number of spray passes appears to be a function of the size of the thoria particles.

The remaining samples that have been tested, Sample Nos. 7 and 8, consist of 40 layers of tungsten foil sprayed with the large thoria particles, Mixture 112. Sample No. 7 received one spray pass per layer, and Sample No. 8 received seven spray passes per layer. A comparison of the performance of these two samples appear in Figure 2.6.

Figure 2.5 Heat Flux vs Source Temperature
Mo-ThO₂ Insulation

Figure 2.6 Heat Flux vs Source Temperature
W-ThO₂ Insulation

In contrast to the results obtained with molybdenum, the seven spray pass sample was less effective than the one spray pass sample. It is very unlikely that the optimum number of spray passes is a function of the foil material, and the contradiction that exists between the molybdenum and tungsten data can be attributed to the spray coatings; spray coatings which are expected to be identical with respect to thickness and particle density in reality are not identical. This lack of reproducibility is associated solely with the Mixture 112 thoria powder, and is a further manifestation of the difficulty associated with spraying the large particles. Sample Nos. 9, 10, and 11, along with other samples yet to be fabricated, hopefully will resolve the inconsistencies that currently exist. When the Mixture 112 thoria powder can be sprayed in a reproducible manner, the performance of the insulation samples will be consistent and meaningful.

Subtask 2.4

The purpose of this subtask is to determine the effect of an applied compressive load on the heat transfer characteristics of selected insulation samples. To date, the load bearing characteristics of only one insulation sample have been tested and the data that has been obtained may change when adjustments are made to the planar test apparatus. The vast majority of the work associated with this task will take place in the next two quarters.

The sample tested was Sample No. 8 in Table 2.3. Sample No. 8 consists of 40 layers of tungsten foil sprayed with seven coats of the Mixture 112 thoria particles. Figure No. 2.7 plots the heat flux through the insulation sample as a function of the source temperature for

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Figure 2.7 Heat Flux vs Source Temperature
 W-ThO₂ Insulation, Applied Compressive Load

applied loads of 0 psi, 1.21 psi, and 2.10 psi. In the 0 psi case the weight of the tungsten heater block was supported by the 15-mil thick tantalum sleeve in Figure 2.8. No load rested on the insulation. To exert 1.21 psi on the insulation sample the tantalum sleeve was removed and the tungsten heater block was supported by the insulation, as in Figure 2.9. Finally, to exert 2.10 psi on the insulation sample, small weights were placed on top of the tungsten heater block, thereby increasing the load on the insulation sample.

The sensitivity of performance to applied load that was reported in Phase I is far less pronounced in Figure 2.7. Table 2.4 compares Phase I and Phase II heat fluxes for 40-layer W-ThO₂ insulation samples.

Temperature	Phase I			Phase II		
	Zero Load	1.21 psi	Percent Increase	Zero Load	1.21 psi	Percent Increase
1000°C	$.05 \frac{\text{watts}}{\text{cm}^2}$	$.48 \frac{\text{watts}}{\text{cm}^2}$	860%	$.021 \frac{\text{watts}}{\text{cm}^2}$	$.032 \frac{\text{watts}}{\text{cm}^2}$	52%
1200°C	$.10 \frac{\text{watts}}{\text{cm}^2}$	$.72 \frac{\text{watts}}{\text{cm}^2}$	620%	$.037 \frac{\text{watts}}{\text{cm}^2}$	$.055 \frac{\text{watts}}{\text{cm}^2}$	49%
1400°C	$.17 \frac{\text{watts}}{\text{cm}^2}$	$1.00 \frac{\text{watts}}{\text{cm}^2}$	488%	$.062 \frac{\text{watts}}{\text{cm}^2}$	$.106 \frac{\text{watts}}{\text{cm}^2}$	71%

Table 2.4
Comparison of Phase I and Phase II Heat Fluxes
Through 40 Layers of W-ThO₂ Insulation

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Figure 2.8 Planar Heat Loss Test Apparatus-
Ta Sleeve Support Heater Block

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Figure 2.9 Planar Heat Loss Test Apparatus-
Ta Sleeve Missing

This drastic improvement in performance, however, may only be a mirage and must be considered only as a preliminary result. The tungsten block rests on the insulation sample which in turn rests on both the guard ring and the collector (Figure 2.9). Every effort is made to position the tops of the guard and collector at exactly the same height so that the applied compressive load will exert a uniform pressure on the insulation sample. However, suppose the guard ring is slightly higher than the collector during the testing, due to differential thermal expansion. In this case the applied load would be supported only by a band near the outer circumference of the insulation sample and the heat reaching the calorimeter would not truly correspond to the calculated average compressive load. This may or may not have been the situation when the data in Figure 2.7 was taken. This situation will be investigated during the next quarter by raising and lowering the entire collector-calorimeter assembly and studying the variations in performance. Once this question of the relative position of the guard to the collector is resolved, the testing will proceed promptly.

2.2 Task 3. W and W(25)Re Foil

During the first quarter 400 feet of 1-mil thick tungsten foil was purchased. One half of the 400 feet of foil was 6 inches wide and the other half was 4 inches wide. The thickness of this foil was to be reduced to $\frac{1}{2}$ mil or less by chemical etching. At the end of the first quarter, it was undecided whether to etch the foil at Thermo Electron or at GE-NMPO.

During the second quarter approximately 150 feet of the 6-inch wide foil and 50 feet of the 4-inch wide foil was chemically etched, in-house,

at Thermo Electron. The etchant consisted of the following acids diluted with water to reduce the rate of etching.

3 parts Hydrofluoric Acid

2 parts Nitric Acid

2 parts Sulphuric Acid

The actual thickness of a typical sheet of the as-received tungsten foil varied from spot to spot on the sheet between approximately 1.2 and 1.3 mils. After etching, the thickness of the foil sheets varied between .3 and .4 mils, indicating that the slight non-uniformities in the thickness of the as-received foil had not vanished during the etching process. The existing variations, however, are too small to influence the fabrication of insulation samples using etched foil. After a foil sheet was dipped in acid it was transferred to a water bath (Figure 2.10) and then to an acetone bath. The foil sheets were then wiped and hung up to dry.

Additional quantities of one mil tungsten foil will be etched as needed.

2.3 Task 4. Cylindrical Insulation

Nothing was done in this area during the past quarter.

2.4 Task 5. Edge Heat Losses

Edge heat loss work during the second quarter concentrated on the production and acquisition of thin metal foil cups and the testing of complete corner assemblies.

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Figure 2.10 Etching of Tungsten Foil

Deep-Drawn Cups

Since the problem of how to join a cylindrical insulation sample to a planar insulation sample was first considered, the thin metal foil cup has been sought as a means of avoiding small gaps in the insulation at the cylindrical-planar interface. At the end of the first quarter a technique had been developed to deep-draw molybdenum cups from metal foil disks as thin as 1.0 mil. Every attempt to draw molybdenum cups using $\frac{1}{2}$ -mil disks resulted in brittle fracture, but it was possible to produce molybdenum cups only a fraction of a mil thick by chemically etching cups drawn from 1-mil foil. Unfortunately, no more than one or two different size cups can be produced from a punch and die of a given size. A prohibitive number of punch and die combinations would therefore be required to fabricate a multi-layer insulation corner assembly. Nevertheless, even individual foil cups properly placed in a corner can significantly reduce edge losses and the cup drawing work therefore has continued.

During the second quarter attention turned to the more difficult problem of drawing cups from the less ductile materials, tungsten and tungsten 25% rhenium. At the outset 7-mil and then 5-mil thick tungsten cups were sought. The essential features of the punch and die used in this work appear on Page 81 of the preceeding quarterly. To successfully deep draw a thin foil cup the slippage between the foil blank and the punch and die must be optimized. Too little slippage will result in brittle fracture and too much slippage produces a cup with deeply creased side walls. After some experimentation an effective compromise was reached and smooth, well-formed, 7-mil and 5-mil thick tungsten cups were drawn.

This procedure was repeated with 4-mil, 3-mil, and 2-mil tungsten foil. The optimization of the slippage became more difficult as the thickness of the foil blanks decreased, but after some experimentation perfect 3-mil and 4-mil thick cups were obtained. Unfortunately, every attempt to draw 2-mil cups resulted in failure and this work was eventually abandoned. Given the ability to draw 3-mil tungsten cups, a thinner cup can always be obtained by chemical etching because the thickness of the cups drawn from the 3-mil foil exhibited the necessary uniformity. The etchant would be identical to that used to reduce the thickness of the tungsten foil in Task 3. Figure 2.11 presents a photograph of perfect 5-mil and 3-mil cups and a broken 2-mil cup.

After tungsten, tungsten 25% rhenium foil was expected to present a lesser problem because of its increased ductility. A successful tungsten 25% rhenium cup was drawn but the project was more difficult than was anticipated because only 1 mil foil was available. A tungsten 25% rhenium cup appears on the right in Figure 2.12. The cup on the left was drawn from a 3-mil tungsten blank. The uneven height of the cups can be corrected by trimming.

Vapor-Deposited Cups

Since it is impractical to build a different size punch and die combination for each foil cup that is required an alternate method was sought to produce cups. To fabricate a true concentric cup corner assembly, approximately forty cups are required that fit together concentrically with a 1-mil gap between cups. It is essential that very precise tolerances be maintained. San Fernando Laboratories of Pacoima,

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Figure 2.11 5-Mil, 3-Mil and 2-Mil Deep-Drawn
Tungsten Cups

8263

Figure 2.12 A 1-Mil Tungsten 25% Rhenium Cup and
a 3-Mil Tungsten Cup (Deep-Drawn)

California, was commissioned to investigate the feasibility of vapor depositing a 1-mil thick layer of tungsten on an appropriately sized molybdenum mandrel. It was felt that vapor deposition was ideally suited to maintaining the precise tolerances required, but it was not clear whether the resulting cups would have any strength or whether they could be removed in tact from the mandrels. San Fernando Laboratories produced six 1-inch in diameter, 1-inch deep, 1-mil thick tungsten cups. It was not easy, but it was possible to remove the tungsten cups in tact from the mandrels by etching away the molybdenum. The resulting cups were about twice as deep as any that had been drawn with a punch and die but, as expected, they were very fragile. Figure 2.13 presents a photograph of two cups, one that broke after handling and one that is in tact.

Based on these results an order has been placed with San Fernando Laboratories for forty 1-mil thick vapor-deposited tungsten cups sized to permit fabrication of a concentric cup corner assembly. The assembly will be thermally tested in the edge heat loss test apparatus. The order specifies that no cup can be cracked or chipped and an assembly procedure has been established that will minimize the possibility of damaging the cups when they are sprayed with the oxide particles. The cups are scheduled for delivery late in April and the testing of the corner should take place in May.

Slit-Cup Corners

During the first quarter a 40-layer overlap corner and a 40-layer slit-cup corner were fabricated using Ta-ThO₂ insulation. They were tested in the edge heat loss test apparatus. Certain improvements were

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Figure 2.13 1-Mil Vapor-Deposited Tungsten Cups
(One broken, one intact)

then made in the test apparatus and when the overlap corner was retested it became apparent that this corner was more effective than was originally thought. At the end of the first quarter the slit-cup corner had not been retested.

Intuitively, the slit-cup corner was expected to improve on the performance of the overlap corner and after the initial testing indicated that the opposite was true. The slit-cup corner was disassembled in an effort to identify possible improvements. Figure 2.14a presents a cross-section of the slit-cup corner assembly and Figure 2.14b shows a top view of a circular tantalum foil blank with wedge-shaped slits equally spaced around the circumference. Two groups of twenty such blanks were placed in the punch and die and drawn into cups. The "cups," however, were very springy and exhibited a tendency to flatten out, thereby opening up gaps in the sidewalls at the slits so the actual corner assembly more closely resembled the situation in Figure 2.14c than that in Figure 2.14a. The cups also appeared to be packed too tightly together.

Two steps were taken to improve the performance of slit-cup corners. Work started on the design of an entirely new slit-cup corner and, in addition, the complete 3-mil cups that constrained the slit cups in the existing corner were reduced in thickness to 1 mil in an effort to alleviate the crowding and therefore reduce the conduction losses. With these modifications the old corner was retested in the improved edge loss apparatus and the results appear in Figure 2.15. The overall level of performance of both the overlap and slit-cup corners improved in the revamped test apparatus, plus the performance gap between the two narrowed indicating that the reduced thickness of the constraining cups did help.

Figure 2.14

Figure 2.15 Edge Area Heat Flux vs Source Temperature
Comparison of Slit-Cup and Overlap Corners

The new slit-cup corner that was designed appears in Figure 2.16a. To facilitate a comparison between the new and the old designs, 40 layers of tantalum foil were again used to fabricate the new corner. Instead of removing wedge-shaped pieces from the circumference of the blanks, simple slits directed radially inward were cut in the new blanks (Figure 2.16b). After the foil blanks were drawn into cups the slits formed tabs that overlapped each other slightly. The cups still had a tendency to flatten out, but additional complete cups (in this case 2 mils thick) were stamped to help the slit cups hold their shape.

The performance of the new slit-cup corner is compared to that of the old corner in Figure 2.17. Surprisingly, the new slit-cup corner wasn't as good as the earlier version and once again the corner was disassembled. The slit cups were still packed together too tightly despite the fact that extra room had been left for them in the new design. The crowding came about because the edges of adjacent tabs occasionally hooked one another forming a bulge instead of overlapping and forming a continuous, smoothly curved side wall. Figure 2.18 attempts to illustrate why each slit cup therefore occupied more room than was expected. In an effort to alleviate the crowding four of the forty slit cups and two of the stamped 2-mil cups were permanently removed from the assembly. The corner was then reassembled and retested and the results are included in Figure 2.17. The performance was very nearly identical with that of the improved version of the original slit-cup corner.

The experience gained by fabricating and testing slit-cup corners indicates that they do not offer any advantage over overlap corners

a. Second Slit-Cup Corner Design

b. Individual Foil Blank Before Deep-Drawing Slit-Cup

Figure 2.16

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Figure 2.17 Edge Area Heat Flux vs Source Temperature
Comparison of First and Second Slit-Cup Corners

a. EXPECTED SHAPE. LESS
SPACE IS REQUIRED

b. ACTUAL SHAPE. MORE
SPACE IS REQUIRED

Figure 2.18 Slit-Cups: Top View

and since they are much more difficult to fabricate the slit-cup concept has been abandoned.

Packed Zirconia Corner

The next corner that was tested was a very simple one to design and fabricate. 325-mesh zirconia powder was packed into the annular region between two concentric cups fabricated from 3-mil tantalum sheet (Figure 2.19). Zirconia has the lowest thermal conductivity among the ceramics and, in view of the relative simplicity of the corner, it was interesting to study its performance in the edge heat loss test apparatus. The results of the tests appear in Figure 2.20. Clearly such a corner is not competitive with multi-foil thermal insulation corners, but it is interesting to note that the apparent thermal conductivity of the powdered zirconia was less than 5% of the conductivity of solid zirconia.

Source Temperature	Thermal Conductivity		$\frac{k_m}{k_t} \times 100$
	Measured Value k_m	Handbook Value (100% theoretical density) k_t	
1180°C	.00037 watts/cm°K	.0237 watts/cm°K	1.56
1350°C	.00055 watts/cm°K	.00243 watts/cm°K	2.27
1510°C	.00095 watts/cm°K	.0246 watts/cm°K	3.86

Table 2.5 Zirconia Corner Thermal Conductivity Comparison

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Figure 2.19 Packed Zirconia Corner

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Figure 2.20 Edge Area Heat Flux vs Source Temperature
Packed Zirconia Corner

Tapered Corner

The next corner configuration to be tested was a butt-joint between tapered cylindrical and planar insulation samples (Figure 2.21). Forty layers of Ta-ThO₂ insulation were used to construct this tapered corner. The cylindrical portion of the corner is fabricated by taking a long rectangular strip of foil and removing a triangular piece from one edge, as in Figure 2.22a. Figure 2.22b shows how the tapered edge results when, after spraying, the foil is rolled into a cylinder. The tapered edge of the planar section of the corner is machined on a lathe. Circular foil disks are stacked one on top of another and then squeezed together very tightly (Figure 2.23a) by thick aluminum plates. This assembly is then mounted on a lathe and a truncated cone is machined as in Figure 2.23b.

The tapered corner test results appear in Figure 2.24. Included for comparison purposes are dashed lines representing the best slit-cup and overlap corner performance data. At every source temperature the tapered corner minimized the heat flux reaching the calorimeter.

None of the edge heat loss data appearing in this quarterly has been reduced to isolate the corner effect because up-to-date 40-layer Ta-ThO₂ data is not yet available. Sample No. 12 in Table 2.3 will provide the necessary data during the next quarter. The Phase I Ta-ThO₂ data is no longer applicable. Nevertheless, a comparison such as that in Figure 2.24 is meaningful because each of the three corners were fabricated using 40 layers of Ta-ThO₂ insulation. Whatever correction is applied to the data will apply equally to the three corners and the tapered corner will still be the best after the data is reduced.

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Figure 2.21 Tapered Corner

Cut rectangular strip of foil along dotted line and remove shaded area.

a. Foil Preparation for Cylindrical Subassembly

b. Cylindrical Subassembly with Tapered Corner (at top)

Figure 2.22

a. Planar Disks Between Aluminum Plates on Lathe for Machining

b. Planar Subassembly with Tapered Corner

Figure 2.23

8299

Figure 2.24 Edge Area Heat Flux vs Source Temperature Tapered Corner

2.5 Task 6. Lower Temperature Applications

This task is devoted to the development of thermal insulation for application at temperatures below 1200°C. The expensive refractory foil materials required at higher temperatures are unnecessary at the more modest intermediate temperatures. Therefore, this task is investigating the characteristics of multi-foil thermal insulation fabricated using aluminum, copper, nickel, and platinum foil in combination with zirconia and alumina. (Platinum, although expensive, has been included because of its oxidation resistance at elevated temperatures.) The investigation follows the sequence established during Phase I; material compatibility tests establish which foil-oxide combinations are practical, and then only those combinations are used to fabricate the samples that are tested.

Subtask 6.1 Material Compatibility Testing

Six constant temperature ovens in vacuum are required. One bell jar contains ovens that operate at 300, 500, and 1000°C, while a second bell jar contains 600, 800, and 1200°C ovens. Each material combination is tested at two temperatures and, at each temperature, samples are examined after 100, 500, and 1000 hours. Six insulation samples are therefore required to completely test each foil-oxide material combination. Photographs of the material compatibility test stations appear in Figures 2.25 and 2.26. The test temperature material combinations are as follows:

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Figure 2.25 Material Compatibility Test Station

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Figure 2.26 From top to bottom, the 1200°C, the 800°C, and the 600°C constant temperature ovens for the material compatibility tests

<u>Insulation Material Combination</u>	<u>Test Temperatures °C</u>
Al-Al ₂ O ₃	300, 500
Al-ZrO ₂	300, 500
Cu-Al ₂ O ₃	600, 800
Cu-ZrO ₂	600, 800
Ni-Al ₂ O ₃	800, 1000
Ni-ZrO ₂	800, 1000
Pt-Al ₂ O ₃	1000, 1200
Pt-ZrO ₂	1000, 1200

The 100-hour checkpoint was reached during the second quarter. As was the case during the Phase I compatibility studies, no gross chemical reactions of any kind were apparent. However, varying amounts of bonding did occur between the oxide particles and the adjacent foil surfaces. If the bonding was severe, the foils would tear before separating. Such bonding lowers the contact resistance to heat conduction between the foils and the particles, which implies that the best material combinations are those that form the weakest bonds. During Phase I the strength of foil-oxide bonds increased with time; if a "weak" bond had formed after 100 hours a "moderate" bond generally followed after 1000 hours. The 100-hour results contained in Table 2.6 therefore should be interpreted as "tendencies" that will probably become more pronounced as the testing proceeds.

TABLE 2.6

METAL- OXIDE COMBINATION	TEST TEMPERATURE °C	EXTENT OF BONDING BETWEEN LAYERS		
		After 100 Hours	After 500 Hours	After 1000 Hours
Al-Al ₂ O ₃	300	None		
Al-Al ₂ O ₃	500	Moderate		
Al-ZrO ₂	300	None		
Al-ZrO ₂	500	Very Weak		
Cu-Al ₂ O ₃	600	Weak		
Cu-Al ₂ O ₃	800	Moderate		
Cu-ZrO ₂	600	Very Weak		
Cu-ZrO ₂	800	Weak		
Ni-Al ₂ O ₃	800	Weak		
Ni-Al ₂ O ₃	1000	Weak		
Ni-ZrO ₂	800	Very Weak		
Ni-ZrO ₂	1000	Very Weak		
Pt-Al ₂ O ₃	1000	Moderate		
Pt-Al ₂ O ₃	1200	Strong		
Pt-ZrO ₂	1000	Very Weak		
Pt-ZrO ₂	1200	Moderate

With every foil material at every temperature zirconia was as good as, or better than, alumina. At 1000°C the bonding between platinum and alumina was classified as "moderate," while that between platinum and zirconia was "very weak." At 1200°C the platinum-alumina bonds were again stronger than the platinum-zirconia bonds. After 1000 hours the tests may indicate that 1200°C is too hot for platinum in combination with either oxide.

In some cases there was a slight discoloration of the oxide particles. The 500-hour and 1000-hour samples will be examined carefully to ascertain whether these discolorations become more pronounced as the tests proceed. Both alumina and zirconia particles exhibited a slight tendency to form conglomerates, thereby decreasing their surface free energy. This particle migration is very slight and it is extremely doubtful that the performance of an insulation sample would be adversely affected.

Subtask 6.2 Lower Temperature Heat Transfer

The purpose of this subtask is to evaluate the effectiveness of multi-foil insulation samples fabricated using the non-refractory foil materials currently under test in Subtask 6.1. For each foil-oxide combination tested, the source temperature will range from approximately 100°C up to whatever maximum is established by the compatibility tests. The current planar heat loss test apparatus cannot be used at source temperatures below 800°C because the heat fluxes through the calorimeter become too small to measure with any accuracy. Given the area of the heat collector and the size of the copper calorimeter the smallest heat flux that the planar apparatus

can measure is approximately $.01 \text{ watts/cm}^2$. At a source temperature of 100°C , however, a typical heat flux might be $.0005 \text{ watts/cm}^2$. A far more sensitive heat loss apparatus is therefore required.

The designs of the current planar and cylindrical heat loss test devices were examined in an effort to determine whether the necessary increase in sensitivity could be achieved by simply resizing some of the components. However, the anticipated heat fluxes are so low that radiation losses from either calorimeter would destroy all accuracy. An entirely new approach, one that eliminates calorimetry with all its associated problems, is required.

The basic procedure for the new low temperature heat loss measurements will be as follows:

1. Two cylindrical multi-foil insulation samples will be fabricated that are identical except for their height. One cylinder will be 6 inches high and the other will be 12 inches high.
2. An electric heater will be placed inside the 6-inch cylinder and the ends of the cylinder will be blocked by specially sized planar multi-foil end plugs. This assembly will be placed inside a vacuum system.
3. The power input to the electric heater, P_6 , required to reach a given temperature, T_1 , in the interior cavity will be measured.
4. Steps 1, 2, and 3 will be repeated using the 12-inch high cylindrical multi-foil insulation sample. The same planar end plugs used with the 6-inch cylinder will be used with the 12-inch cylinder.
5. The difference in the power inputs, $P_{12} - P_6$, required to reach T_1 , divided by the difference in the lateral areas of the 6-inch and 12-inch cylinders, will be the heat flux through the insulation at source temperature T_1 .

Note that no attempt is made to measure the losses through the multi-foil planar end plugs. If the same end plugs are used with the two different cylindrical samples the thermal losses from the ends of each cylinder will be identical at a given source temperature, i. e., the thermal losses at the ends will be independent of the height of the cylindrical samples at a given source temperature.

This technique will be applied at temperatures up to 800°C. The nickel and platinum insulation samples are capable of operation at temperatures above 800°C and these measurements will be made in the existing cylindrical heat loss test apparatus. The same 6-inch high cylindrical insulation sample fabricated for the low temperature tests can be used in the high-temperature device.

The power input to the electric heater will be determined by measuring the voltage drop across the heater and the current through the heater. The voltage taps will be just outside the end plug where the heater leads penetrate. The source and sink temperatures will be measured by thermocouples and heat rejection will be by radiation from the cold side of the cylindrical samples.

The lower temperature heat loss test apparatus with the 6-inch cylindrical sample in place appears in Figure 2.27. Figure 2.28 presents the same test configuration incorporating the 12-inch sample. The electric heater is fabricated in two sections. The top section fits over a collar onto the bottom section when testing the 12-inch insulation sample, but only the bottom is required to test the 6-inch cylinder. The cylindrical insulation samples fit tightly over a 2-mil thick nickel cylinder which also is fabricated in sections. The apparatus can therefore be lengthened or shortened to accommodate either sample.

8301

Figure 2.27 Low Temperature Heat Loss Test Apparatus
Six-Inch Configuration

8300

Figure 2.28 Low Temperature Heat Loss Test Apparatus
Twelve-Inch Configuration

The end plugs are fabricated using 100 layers of Mo-ZrO₂ insulation. Molybdenum foil was chosen because of its strength, and zirconia because it avoids the radioactivity problem associated with thorium. A tapered corner will be fabricated at both ends, thereby minimizing the possibility of thermally short-circuiting the cylindrical foils. It is not essential that the end plugs be manufactured from multi-foil insulation. Fibrous insulations such as MIN-K 2000 were considered but, since these insulations are many times less effective than multi-foil insulation, the end losses would be far higher and it is desirable to avoid this situation if possible.

2.6 Task 7. Life Tests

No work scheduled.

III. PLANNED THIRD QUARTER WORK

Task 2. Spray Coating

The successful completion of Subtask 2.2 and 2.3 hinges on solving the problem of spraying the large thoria particles. A new nozzle with a larger orifice and new binder ingredients have been ordered. In addition, in all future work the spray gun will be disassembled and thoroughly cleaned after every forty spray passes. This problem will be resolved in the next quarter and Subtasks 2.2 and 2.3 will then be completed.

The study of the variation of performance with applied compressive load (Subtask 2.4) will continue.

Task 3. W and W(25)Re Foil

The chemical etching of tungsten foil will resume if additional etched foil is required.

Task 4. Cylindrical Insulation

The cylindrical test apparatus used throughout Phase I will be reconditioned and work will start on the optimization of the layer spacing.

Task 5. Edge Heat Losses

The 40-layer concentric cup corner will be fabricated from the vapor-deposited tungsten cups on order from San Fernando Laboratory.

Task 6. Lower Temperature Applications

The material compatibility tests will be completed and the lower temperature heat transfer apparatus fabricated.

Task 7. Life Tests

The four planar insulation samples that will be tested in the Phase I extended soak test apparatus will be fabricated and subsequently tested in the planar heat loss test apparatus. These tests will establish the zero-time performance characteristics of the samples.