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TE Report No. 4059-16-69

THERMO ELECTRON CORPORATION QUARTERLY PROGRESS REPORT OF RESEARCH AND DEVELOPMENT OF VACUUM FOIL-TYPE INSULATION FOR RADIOISOTOPE POWER SYSTEMS

March 1968 - May 1968

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Contract No. AT(30-1)-3634
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VACUUM FOIL-TYPE INSULATION FOR
RADIOISOTOPE POWER SYSTEMS

by

J. Carvalho, J. B. Dunlay, J. Fronduto
M. L. Paquin, and V. L. Poirier

ABSTRACT

This report is the third 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 from March 1968 through May 1968.

A new planar heat transfer test apparatus has been fabricated and is now in use. To date, twenty planar insulation samples have been fabricated and tested. These tests indicate that for a given particle size, the optimum oxide coating is the minimum amount consistent with complete foil separation. The performance of a planar insulation sample subjected to compressive stresses up to 5.11 psi is also presented.

The operating characteristics of a modified cylindrical heat transfer apparatus are discussed.

The low temperature material compatibility tests have been completed and a low temperature heat transfer apparatus has been designed and fabricated.

I. INTRODUCTION AND SUMMARY

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

A new planar heat transfer apparatus has been designed, fabricated, and placed in operation. The apparatus features a thermal flux transducer capable of measuring heat fluxes down to a few thousandths of a watt per square centimeter. To date, twenty planar insulation samples have been fabricated and tested. These tests indicate that, for a given particle size, the optimum oxide coating consists of the minimum number of particles consistent with complete foil separation.

The new planar apparatus permitted new, more accurate measurements of the effect of an applied compressive load on performance. Compressive stresses ranging from 0 to 5.14 psi were imposed on an insulation sample. An increase of nearly a factor of ten in the thermal losses was observed. The rate of bonding between the oxide particles and the foils increased markedly.

A considerable period was devoted to experimentation with the cylindrical heat transfer apparatus. "External" factors such as the support structure were found to influence the calorimeter measurements. Various schemes were tried in an effort to thermally isolate the calorimeter structure.

The low temperature material compatibility tests have been concluded and the results are tabulated in this report. Zirconia was

found to be more suitable than alumina for use with aluminum, copper, nickel, and platinum foil.

A low temperature heat transfer apparatus has been designed and fabricated. This apparatus will be the test vehicle for the performance measurements at source temperatures below 700°C.

The short-term life tests are about to begin. The Phase I "extended soak" apparatus has been extensively reconditioned and the test samples are now being prepared.

II. COMPLETED THIRD QUARTER WORK

2.1 Task 2. Spray Coating

Subtasks 2.1 and 2.2 have been completed and the results have been discussed in previous quarterlies. Subtask 2.3 is investigating the effect of various oxide coatings on the performance of non-load bearing insulation samples. Subtask 2.4 is studying the performance of structurally loaded samples. Subtasks 2.3 and 2.4 will be discussed separately.

Correction

Table 2.1 lists the characteristics of the three thorium powders under investigation. This table also appeared in the preceding quarterly report, but contained an error. The corrected table is as follows:

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

*Slight Preprocessing

TABLE 2.1 AMERICAN POTASH THORIUM POWDERS

Subtask 2.3

A new planar heat transfer test apparatus has been designed, fabricated, and placed in operation. The new apparatus uses a device called a thermal flux transducer to measure the heat fluxes through the insulation sample. The transducer was developed by Geonetics Corporation of San Diego and the early results are most promising. The new device can measure heat fluxes on the order of a few thousandths of a watt per square centimeter, whereas the older calorimeter apparatus can only measure fluxes down to about .01 watts/cm². The transducer is much easier to use than the calorimeter, as the output is converted from millivolts to watts/cm² by a calibration curve defined by the manufacturer.

Figure 2.1 presents the design of the new planar apparatus. The design is identical to the calorimeter apparatus on the hot side of the insulation sample. Only the heat rejection system and the measuring technique are different. Note that no guard ring is required. The diameter of the transducer matches the diameter of the insulation samples, but only a portion of the surface near the center actually measures the heat flux.

Figure 2.2 presents a photograph of the transducer as it was received from Geonetics Corporation. Figure 2.3 shows the transducer mounted on the heat rejection portion of the apparatus, and Figure 2.4 presents an overall view of the complete apparatus.

As Figures 2.5 and 2.6 illustrate, data obtained using the two test devices is in fairly good agreement. The only consistent difference

Figure 2.1 Thermal Flux Transducer Planar Heat Transfer Apparatus.

8480

Figure 2.2 The Transducer As Received from Geonetics Corporation.

8471

Figure 2.3 The Transducer Mounted on its
Heat Rejection System.

8483

Figure 2.4 Overall View - Transducer Planar
Heat Transfer Apparatus.

8488

Figure 2.5 A Comparison of Transducer and Calorimeter Data.

8500

Figure 2.6 A Comparison of Transducer and Calorimeter Data.

that has arisen is that the transducer data are generally lower at the lower temperatures (800 to 1100°C). The heat fluxes at these temperatures are usually very close to the limit of the sensitivity of the calorimeter apparatus.

Table 2.2 contains an up-to-date list of the planar insulation samples that have been fabricated and tested. Each sample has been tested in essentially the same fashion. The heat flux through the insulation sample was measured as a function of the source temperature for a sink temperature of approximately 30°C. To date, every insulation sample has consisted of forty layers of foil. By holding the total number of radiation shields constant, the differences in performance can be attributed to the different materials and oxide coatings being tested.

At the time the preceeding quarterly was being written, there was some confusion about the optimum number of thoria coatings per foil layer. Results obtained with one thoria powder seemed to contradict the results obtained with another powder. A sample that had been sprayed with seven coats of the very firm Mixture 115 thoria powder did not perform as well as a sample that had been sprayed with only one coat. With the relatively coarse Mixture 116 thoria powder the opposite was true. Subsequent tests have shown that the number of spray coatings is a poor correlation parameter because successive spray coatings are not 100% reproducible, especially in the case of the large thoria particles. By using a new parameter that is a more direct measure of the amount of thoria on a foil surface, a more consistent pattern has emerged from recent measurements.

8502

Table 2.2 Planar Insulation Samples Fabricated and Tested.

Sample Number	Foil Material	Foil Thickness	Number of Layers	Oxide	Number of Coats	Avg. Oxide Layer Thickness
1	Mo	.001"	40	Mixture 115 ThO ₂	1	1.3μ
2	Mo	.001"	40	Mixture 112 ThO ₂	1	7.3μ
3	Mo	.001"	40	Mixture 116 ThO ₂	1	15.2μ
4	Mo	.001"	40	Mixture 116 ThO ₂	3	24.5μ
5	Mo	.001"	40	Mixture 116 ThO ₂	7	23.8μ
6	Mo	.001"	40	Mixture 115 ThO ₂	7	5.1μ
7	W	.001"	40	Mixture 116 ThO ₂	1	20.3μ
8	W	.001"	40	Mixture 116 ThO ₂	7	27.0μ
9	Mo	.001"	40	Mixture 116 ThO ₂	1	21.6μ
10	W	.001"	40	Mixture 116 ThO ₂	1	16.5μ
11	W	.001"	40	Mixture 116 ThO ₂	7	33.7μ
12	Ta	.0005"	40	Mixture 116 ThO ₂	1	11.4μ
13	Ta	.0005"	40	ZrO ₂	1	10.8μ
14	Mo	.001"	40	Mixture 115 ThO ₂	1	1.6μ
15	Mo	.001"	40	Mixture 112 ThO ₂	1	10.2μ
16	Mo	.001"	40	ZrO ₂	1	9.5μ
17	W	.001"	40	ZrO ₂	1	4.5μ
18	W	.001"	40	Mixture 115 ThO ₂	1	2.5μ
19	W	.001"	40	Mixture 112 ThO ₂	1	1.2μ
20	W	.001"	40	Mixture 116 ThO ₂	1	8.3μ

The new parameter is the average thickness of an oxide layer. This number is determined by measuring the thickness of the foil layers before and after spraying, taking the difference, and dividing by the number of layers. Figures 2.7, 2.8 and 2.9 all compare the performance of samples fabricated using the same foil material and thorium mixture. The samples differ from one another in that they have different average oxide layer thicknesses. In all cases, the samples with the higher average oxide layer thickness did not perform as well as the more compact samples. If one assumes that for a given particle size the oxide layer thickness increases as the quantity of thorium increases, then a lower heat flux can be achieved by depositing less thorium on each foil surface.

This result is consistent with an analysis of the heat transfer mechanisms through multi-foil insulation. The sole purpose of the oxide particles is to separate the radiation shields. The goal is to achieve this separation with as few particles as possible, because each particle represents a conduction path from one radiation shield to the next. Therefore the higher heat fluxes observed with the more heavily coated samples probably can be attributed to increased conduction losses. It is worth noting that an optimum coating must exist because if too few particles cover the foil surfaces the foils will occasionally touch, effectively "short-circuiting" the insulation locally. It is doubtful that any of the samples prepared to date have been coated too lightly.

One of the objectives of Task 2.3 is to determine whether the size of the oxide particles influences performance. Figure 2.10

8501

Figure 2.7 A Comparison of Oxide Layer Thicknesses.

8484

Figure 2.8 A Comparison of Oxide Layer Thicknesses.

8490

Figure 2.9 A Comparison of Oxide Layer Thicknesses.

compares the performance of three 40-layer Mo-ThO₂ insulation samples. Sample 14 received one coat of the small Mixture 115 thoria particles, Sample 15 received one coat of the medium-size Mixture 112 thoria particles and Sample 9 received one coat of the coarse Mixture 116 thoria particles. As Figure 2.10 shows, the performance of the samples improved as the particles increased in size. If each foil layer was covered by the same quantity of thoria, a fair conclusion would be that the larger thoria particles produce better multi-foil insulation. However, one might expect that the size of the particles strongly influences the quantity deposited on a foil surface by one pass of the spray gun. A series of tests was therefore planned to establish whether this is, in fact, the case.

Three new 40-layer insulation samples were fabricated, this time using tungsten foil. In addition to the normal thickness measurements, the unsprayed foils, and later the sprayed foils were accurately weighed with a balance capable of reading $\pm .1$ milligram (Figure 2.11). The difference in weight before and after spraying (and firing) represents the weight of the thoria deposited on the foil surfaces. This number was used to compute the average oxide coating density (mg./cm²) deposited on each insulation sample. These densities were as follows:

	THORIA POWDER	AVG. THORIA COATING DENSITY
Sample No. 18	Mixture 115	1.346 mg/cm ²
Sample No. 19	Mixture 112	.130 mg/cm ²
Sample No. 20	Mixture 116	.005 mg/cm ²

TABLE 2.3 OXIDE LAYER COATING DENSITIES

8489

Figure 2.10 A Particle Size Comparison:
Mo-ThO₂ Insulation.

8469

Figure 2.11 Weighing Multi-Foil Insulation Samples.

For these three samples, the small particle and large particle coating densities differed by a factor of twenty-five. The performance of the three samples is compared in Figure 2.12. As was the case with the molybdenum samples, the larger particles produced the best insulation, the medium-size particles the next best, and the small particles the worst insulation. It is impossible, at this point, to attribute X% of the performance differences to the different particle sizes and Y% to the different coating densities. Future testing will seek to make this resolution.

An attempt was made to remove a portion of the thoria coating on Sample 18 to ascertain whether this would improve performance. Sample 18 was disassembled and each layer was brushed, but only about 5% by weight of the thoria was removed. The sample was then rubbed with emory cloth, but again only about 5% of the total thoria coating could be removed. This was not considered a sufficient difference to warrant retesting the sample.

The adherence of the thoria particles to the foil surface was undoubtedly closely related to the testing history of the sample. Having been at elevated temperatures for several hours bonds apparently formed between the thoria and the tungsten. The fact that the layers were easily separated, however, indicates that these bonds formed only between the particles and the surface onto which they were sprayed, and not between the particles and the backside of the adjacent layer. This evidence indicates that multi-foil insulation very likely can be made vibration-resistant.

8499

Figure 2.12 A Particle Size Comparison:
W-ThO₂ Insulation.

Table 2.2 indicates that three multi-foil insulation samples were fabricated using zirconia rather than thoria as a separator. A substitute for thoria is being sought because of the inconvenience caused by the mild radioactivity of thoria. To be acceptable, a substitute oxide must be compatible with the foil materials and the resulting thermal insulation must be effective. Zirconia is considered a likely candidate for a number of reasons. The melting point of zirconia is lower than that of thoria (2770°C vs 3300°C), but not by enough to disqualify zirconia. The Zirconium Corporation of America, supplier of the zirconia used for these tests, lists the vapor pressure of its zirconia as 4×10^{-5} torr at 1925°C. This vapor pressure is adequate for some applications and inadequate for others. Figure 2.13 compares the thermal conductivity of zirconia to that of thoria. Zirconia has a much lower thermal conductivity at low temperatures, but the two are very similar above 1000°C. The zirconia particles that were used had a particle size distribution very similar to the Mixture 112 thoria.

Figures 2.14, 2.15, and 2.16 compare zirconia insulation samples to thoria samples. The results are inconclusive since with tantalum the zirconia sample was better, but with molybdenum and tungsten the thoria samples were better. The comparison in Figure 2.16 could be the most meaningful since the average oxide coating density of both samples is known. The small differences in the previous two figures could be caused by excessive oxide. The average oxide coating density will be measured for all future test samples.

8491

Figure 2.13 The Thermal Conductivity of Zirconia and Thoria.

8492

Figure 2.14 A Comparison of Ta-ThO₂ Insulation to Ta-ZrO₂ Insulation.

8493

Figure 2.15 A Comparison of Mo-ThO₂ Insulation to Mo-ZrO₂ Insulation.

8496

Figure 2.16 A Comparison of W-ThO₂ Insulation to W-ZrO₂ Insulation.

Subtask 2.4

The preceeding quarterly report contains data specifying the effect of an applied compressive load on the heat transfer characteristics of an insulation sample. This data was obtained using the Phase I planar heat transfer test apparatus. The report states that some uncertainties were associated with the test results and it contains a detailed discussion of the problem. Briefly, there was some question about whether the applied load was distributed uniformly over the test specimen. It was felt that the separate guard and collector pieces might be at different heights, which means that one or the other would support the applied load. The intervening insulation would then be unevenly loaded.

The planar heat transfer test apparatus that was built to accommodate the thermal flux transducer avoids this problem entirely. As shown in Figure 2.1, the heat rejection structure is a one piece unit. The rigid heater block on the hot side, and the rigid transducer on the cold side, insure that the applied load is distributed uniformly over the insulation sample.

The test specimen that was used for the pressure loading tests was Sample 9. Sample 9 consists of forty layers of .0010" molybdenum foil coated with the large Mixture 116 thorium powder. Measurements were taken with applied load of 0.00 psi, 0.67 psi, 1.57 psi, 2.00 psi, and 5.14 psi acting on the insulation sample. The test results appear in Figure 2.17 with 0.67 psi acting on the test specimen. The heat flux was very nearly identical with the unloaded values. 1.57 psi, 2.00 psi and 5.14 psi produced the anticipated increases in heat flux.

8503

Figure 2.17 Planar Pressure Loading Data.

An interesting phenomenon was observed at 5.14 psi. After approximately 16 hours at temperature a sizable increase in the heat flux was noted. This was believed due to the accelerated formation of bonds between layers under pressure. At the completion of the test the sample was removed from the apparatus and the subsequent examination confirmed that moderately strong bonds had formed between layers. In the course of breaking these bonds layer-by-layer, a significant fraction of the thoria particles became detached from the surfaces. This reduction in thoria is believed responsible for the observed improvement in the performance of the sample under zero load. The next sample to be tested under pressure will be left intact throughout all the tests.

2.2 Task 3. Tungsten and Tungsten 25% Rhenium Foil

No work scheduled.

2.3 Task 4. Cylindrical Insulation

The cylindrical insulation testing program encompasses three subtasks. Subtask 4.1 seeks to optimize the spacing between adjacent layers of a cylindrical insulation sample, Subtask 4.2 studies the performance of a cylindrical insulation sample subjected

to uniform compressive stresses, and Subtask 4.3 investigates the effect of non-uniform compressive stresses. Each subtask will be discussed individually.

Subtask 4.1

Table 4.1 lists the defining characteristics of the six cylindrical insulation samples that have been fabricated to date. Two foil materials, tantalum and tungsten, are being tested because it was felt that the optimum layer spacing may depend on the foil material. The relatively soft, ductile nature of tantalum contrasted with stiff tungsten should establish whether this is, in fact, the case. Note that all the insulation samples consist of 40 wraps of foil. If the layer spacing were changed by varying the number of wraps between fixed inner and outer restraining cylinders, the measured differences in performance would be due largely to the variable number of radiation shields. Therefore, Samples C1 through C6 vary the layer spacing by holding the outer diameter and the number of wraps constant and varying the inner diameter. The original intent was to build samples with average layer thicknesses of .0010", .0020", and .0040". During fabrication it became apparent that the minimum average layer thickness attainable was .0016".

Test Sample	Foil Material	Oxide	Number of Layers	O. D.	I. D.	Average Layer Spacing
C1	.0005 ¹⁵ Ta	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.367 ¹⁵	.0016 ¹⁵
C2	.0005 ¹⁵ Ta	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.334 ¹⁵	.0020 ¹⁵
C3	.0005 ¹⁵ Ta	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.174 ¹⁵	.0040 ¹⁵
C4	.0004 ¹⁵ W	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.367 ¹⁵	.0016 ¹⁵
C5	.0004 ¹⁵ W	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.334 ¹⁵	.0020
C6	.0004 ¹⁵ W	Mixture 115 ThO ₂	40	1.494 ¹⁵	1.174 ¹⁵	.0040 ¹⁵

TABLE 4.1
CYLINDRICAL TEST SAMPLES

The cylindrical heat transfer test apparatus being used for these tests was designed and fabricated during Phase I. Figure 4.1 presents a drawing of this apparatus. At the outset of this new series of tests the apparatus was thoroughly reconditioned and the Phase I performance data were reviewed in an effort to locate any potential design improvements. This review established that much of the Phase I data had been obtained while the guard temperatures were as much as 65°C hotter than the collector temperature. Conceivably, such an axial temperature gradient could direct heat along the foils to the center of the insulation sample, and then out through the collector to the calorimeter. The overall effect would be to make the heat flux appear larger than it actually was.

The importance of eliminating axial temperature gradients was recognized during the design stage. The guards, however, were expected to run cooler, not hotter, than the collector because of end losses from the test samples. The auxillary heaters that had been brazed to the guards were therefore powerless to correct the axial gradient. The calibration heater on the collector could have been used to raise the collector temperature, thereby eliminating the axial gradient, but the associated thermal input would then be superimposed on the normal calorimeter heat flow.

At the outset of the Phase II testing, one series of measurements was taken to see if the axial temperature gradient would reappear. Figure 4.2 presents a typical set of temperature measurements and essentially reproduces the situation encountered throughout the Phase I testing. The water-cooling coils on the guard rings are attached by copper straps, whereas on the calorimeter the water-cooling coil is

8497

Figure 4.1 Cylindrical Heat Transfer Test Apparatus.

8504

Figure 4.2 Typical Cylindrical Test Situation - Axial Temperature Gradient.

brazed directly to the surface. To improve the guard water-cooling, the straps were removed and replaced by a continuous braze. Figure 4.3 shows that the axial temperature gradients were essentially eliminated by this change. Figure 4.4 indicates, however, that even though the axial gradients were eliminated, the performance data were virtually unchanged.

Figure 4.4 also illustrates that the overall magnitude of the cylindrical losses is well above the magnitude of the losses measured in the planar heat transfer test apparatus. Higher layer densities can be obtained with planar samples, which means that planar samples can have a lower effective thermal conductivity. Nevertheless, the absolute thermal losses through n planar layers are expected to be very nearly the same as the absolute thermal losses through n cylindrical layers. This has not been observed to date, perhaps because the cylindrical test apparatus is not fully debugged.

As Figure 4.1 shows, the exterior portion of the cylindrical apparatus is wrapped in Micro-Quartz. This insulation was added in an effort to channel as much of the heat that has passed through the multi-foil as possible through the guard and collector rings. In an effort to establish whether the Micro-Quartz influenced the test results, the Micro-Quartz was removed, the apparatus recalibrated, and one test point obtained. Figure 4.5 shows that for a given input power to the calibration heater, a smaller ΔT was observed in the calorimeter without the Micro Quartz in place, and Figure 4.6 shows that at a given source temperature a larger heat flux was observed without the Micro-Quartz.

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Figure 4.3 Axial Temperature Gradient Eliminated.

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Figure 4.4 Cylindrical Performance Data
Independent of Axial Gradient.

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Figure 4.5 Cylindrical Calibration Curve - With and Without Micro-Quartz.

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Figure 4.6 Cylindrical Heat Fluxes - With and Without Micro-Quartz.

Figure 4.7 attempts to explain why this was the case. The Micro-Quartz can conduct some heat away from the calibration heater, but without the Micro-Quartz even more heat can radiate from the collector. These tests indicate that it is desirable to use as much insulation as possible next to the collector and guards to minimize the extraneous thermal losses.

Figure 4.1 shows that two planar multi-foil insulation assemblies are used to plug the ends of the cylindrical test sample. At the bottom of the test sample, the planar insulation rests in a molybdenum cup with a hole in it for the heater lead. It was felt that the high flux could be caused, in part, by thermal short-circuiting of the insulation in the manner depicted in Figure 4.8. To combat this effect, the molybdenum cup was removed and replaced by a small piece of molybdenum screen (Figure 4.9). The screen is able to support the end plug without conducting a great deal of heat around to the back of the test sample. The differences in performance with the cup and the screen were as follows:

Test	Source Temperature	Calorimeter ΔT	Calorimeter Q	Heat Flux
1. With Mo Cup	1510°C	.425 mv	13.7 watts	.307 $\frac{\text{watts}}{\text{cm}^2}$
2. With Mo Screen	1550°C	.285 mv*	9.2 watts	.205 $\frac{\text{watts}}{\text{cm}^2}$

*recalibrated

TABLE 4.2 CYLINDRICAL PERFORMANCE COMPARISON:
Mo CUP VS Mo SCREEN

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CONDUCTION THROUGH MICRO-QUARTZ

$$(q/A)_{\text{cond.}} = k \frac{\Delta T}{t} \approx .006 \text{ watts/cm}^2 \text{ (TYPICALLY)}$$

RADIATION FROM THE COLLECTOR

$$(q/A)_{\text{rad.}} = \epsilon \sigma (T_1^4 - T_2^4) \approx .020 \text{ watts/cm}^2 \text{ (TYPICALLY)}$$

Figure 4.7 Radiation Without Micro-Quartz
vs. Conduction With Micro-Quartz.

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Figure 4.8 Mo Cup Effectively "Short-Circuits" Insulation.

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Figure 4.9 Mo Screen Replaces Mo Cup.

Apparently heat was flowing from the center of the cup to the edges, into the foil at the back of the insulation sample, and ultimately into the collector-calorimeter structure.

In conclusion, a number of factors appear to be influencing the operation of the cylindrical apparatus. Current efforts are directed towards thermally isolating the calorimeter from the remainder of the apparatus. Once this is achieved the testing of Samples C1 through C6 should be very straightforward.

Subtask 4. 2

This work will commence when Subtask 4. 1 is completed.

Subtask 4. 3

Figure 4. 10 presents the preliminary design of the non-uniform loading cylindrical heat transfer test apparatus. The design is very similar to that of the unloaded cylindrical heat transfer test apparatus. The heater block is encased in a metal sleeve which will be an interchangeable part. Both molybdenum and tungsten sleeves will be used to obtain two different weights for the sleeve-heater block subassembly. This subassembly rests, in theory, along an element of the cylindrical insulation sample. In actual practice, a small sector of the insulation cross-section will support this load.

The apparatus will operate horizontally rather than vertically, and the ends of the cylindrical insulation sample are plugged by planar insulation subassemblies. Whereas the existing cylindrical apparatus now employs tapered corners, it will be necessary to build overlap

Figure 4.10 Non-Uniform Loading Cylindrical Heat Transfer Test Apparatus.

corners for this device. The cylindrical sample will deform slightly under the applied load and the anticipated deformation would not be compatible with a tapered corner.

With the exception of the outermost support structure, the remainder of the apparatus is identical with the existing design. The central collector-calorimeter structure is flanked by guard subassemblies. The guards will be operated at the same temperature as the collector to eliminate axial temperature gradients. A calibration heater is brazed to the exterior of the collector, and a thermocouple will monitor the heater block temperature.

2.4 Task 5. Edge Heat Losses

Activity on this task has centered around assembling a complete set of the .0010" thick vapor-deposited tungsten cups that will be used to fabricate a concentric cup corner. The cups are being supplied by San Fernando Laboratories of Pacoima, California. Table 5.1 lists the dimensions of each of the forty cups, and specifies the delivery status and condition of each. Five cups have yet to be delivered, five have been delivered but are broken, and three are coated with a gel to protect the cups during shipment. The gel was very protective, but has proven very difficult to remove from the cup surface.

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

Cup No.	Dia. in.	Height in.	Status	Cup No.	Dia. in.	Height in.	Status
1	1.000	.600	In-house, intact	21	1.100	.640	In-house, intact
2	1.005	.602	In-house, intact	22	1.105	.642	In-house, intact
3	1.010	.604	In-house, broken	23	1.110	.644	In-house, intact
4	1.015	.606	In-house, intact	24	1.115	.646	In-house, intact
5	1.020	.608	In-house, intact	25	1.120	.648	In-house, intact
6	1.025	.610	In-house, broken	26	1.125	.650	In-house, intact
7	1.030	.612	In-house, intact	27	1.130	.652	In-house, intact
8	1.035	.614	In-house, intact	28	1.135	.654	In-house, intact
9	1.040	.616	In-house, intact	29	1.140	.656	In-house, intact
10	1.045	.618	In-house, broken	30	1.145	.658	In-house, intact
11	1.050	.620	In-house, intact	31	1.150	.660	In-house, intact
12	1.055	.622	In-house, intact	32	1.155	.662	Not delivered
13	1.060	.624	In-house, intact	33	1.160	.664	Not delivered
14	1.065	.626	In-house, broken	34	1.165	.666	In-house, intact
15	1.070	.628	In-house, intact	35	1.170	.668	Not delivered
16	1.075	.630	In-house, intact	36	1.175	.670	In-house, gel coating
17	1.080	.632	In-house, intact	37	1.180	.672	Not delivered
18	1.085	.634	In-house, broken	38	1.185	.674	In-house, packed in gel
19	1.090	.636	In-house, broken	39	1.190	.676	Not delivered
20	1.095	.638	In-house, intact	40	1.195	.678	In-house, gel coated

TABLE 5.1 VAPOR-DEPOSITED TUNGSTEN CUPS

aluminum, copper, nickel, and platinum foil in combination with zirconia and alumina. (Platinum, although expensive, has been included because of oxidation resistance at elevated temperatures.) The investigation is following the sequence established during Phase I; material compatibility tests have established which foil-oxide combinations are practical, and only these combinations are being used to fabricate the samples that will be tested.

Subtask 6.1 Material Compatibility Testing

The material compatibility tests, which were in progress when the preceeding quarterly was written, have now been completed. Aluminum, copper, nickel, and platinum foil were evaluated in combination with alumina and zirconia. The test temperatures for each combination of materials were as follows:

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

Samples of each material combination at each test temperature were examined after 100 hours, 500 hours, and 1000 hours. Each sample was carefully disassembled and examined with the help of a binocular microscope. The appearance of the oxide layers and the degree of bonding between the metal foils were noted for each sample. Two important generalizations can be made about the observations. First, as Table 6.1 indicates, the ZrO_2 samples consistently showed less bonding than the corresponding Al_2O_3 samples. Specifically, of all the ZrO_2 samples tested, the only ones which displayed moderate foil adhesion were the Pt samples heated for 100, 500 and 1000 hours at $1200^\circ C$. By contrast, the only Al_2O_3 samples that did not display at least moderate adhesion after 1000 hours were the Al samples heated at $300^\circ C$ and the Cu samples heated at $600^\circ C$.

The second generalization is that the effects of the heat treatments appeared, in general, to be more temperature-dependent than time-dependent. Typically, the condition of samples heated for 1000 hours was very similar to the condition of similar samples heated for only 100 hours. For each of the four metals investigated (Al, Cu, Ni, and Pt) the degree of bonding was stronger at the higher of the two temperatures tested, except in the case of Ni where the bonding was virtually the same at $800^\circ C$ and $1000^\circ C$. The Ni samples were the only examples of stronger adhesion resulting from longer exposure but not from an increase in temperature.

Tests were run on more than one metal at only two of the six furnace temperatures: both Cu and Ni at $800^\circ C$ and Ni and Pt at $1000^\circ C$. At each of these temperatures there was less bonding between the Ni foils

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	None	None
Al-Al ₂ O ₃	500	Moderate	Moderate	Moderate
Al-ZrO ₂	300	None	None	None
Al-ZrO ₂	500	Very Weak	Very Weak	Very Weak
Cu-Al ₂ O ₃	600	Weak	Weak	Weak
Cu-Al ₂ O ₃	800	Moderate	Moderate	Strong
Cu-ZrO ₂	600	Very Weak	Very Weak	Very Weak
Cu-ZrO ₂	800	Weak	Weak	Weak
Ni-Al ₂ O ₃	800	Weak	Weak	Moderate
Ni-Al ₂ O ₃	1000	Weak	Weak	Moderate
Ni-ZrO ₂	800	Very Weak	Very Weak	Weak
Ni-ZrO ₂	1000	Very Weak	Very Weak	Weak
Pt-Al ₂ O ₃	1000	Moderate	Strong	Strong
Pt-Al ₂ O ₃	1200	Strong	Very Strong	Very Strong
Pt-ZrO ₂	1000	Very Weak	Weak	Weak
Pt-ZrO ₂	1200	Moderate	Moderate	Moderate

TABLE 6.1
LOW TEMPERATURE MATERIAL COMPATIBILITY TEST RESULTS

than between the foils of the other test metal. (Note that Pt has a higher melting temperature than Ni, by 317°C.) The foils in all of the Pt-Al₂O₃ samples were virtually impossible to separate intact, and it should be noted that this was due not only to adhesion but also to the fact that the individual Pt foils were only .00025" thick and therefore exceptionally fragile.

As indicated already, the ZrO₂ layers were consistently much better at preventing adhesion between the metal foils than were the Al₂O₃ layers. In addition, the ZrO₂ layers were much more uniform and continuous, and this fact probably contributed to their superior performance. The ZrO₂ formed a cohesive layer which adhered well to the surface of each foil. The ZrO₂ came off in large flakes if it fell off at all. By contrast, the Al₂O₃ was always agglomerated, was very loosely adherent to the metal foils, and tended to fall out from between the foils as the sandwiches were undone, particularly from samples which had been heated to only 600°C or less. In spite of these differences, the color of the Al₂O₃ and ZrO₂ layers were almost identical for similar runs, and these colors were virtually the same after 1000 hours as after 100 hours. In the Pt samples the oxide layers were still white and in the Al and Ni samples they were only slightly discolored. In the 800°C Cu samples the oxides were again only slightly discolored, but in the 600°C Cu samples the Al₂O₃ was reddish brown in color and the ZrO₂ was purplish. When these oxides were observed under a microscope they were found to consist of aggregates of grains, most of which were still white, while the colored material coated the outside of the aggregate. From this microscopic

appearance it was inferred that the oxides had not been changed chemically and had merely been coated with Cu metal by surface diffusion. The fact that the oxides in the higher temperature Cu samples (800°C) were not similarly coated might perhaps be explained by a lower driving force for surface diffusion at 800°C, owing to differential changes in surface energy with temperature.

One final observation was that the foils were often bonded together more strongly around the edges than elsewhere. This stronger bonding was associated with a lack of oxide coating at the edges of the foils, and indicates the effectiveness of the coating in inhibiting adhesion.

Even highly adherent samples showed no signs of chemical reaction at the points of contact between the foils and the oxide particles. This is consistent with predictions from thermodynamic calculations, and indicates that physical processes such as diffusion are chiefly involved in the changes which occur during testing.

Subtask 6.2 Lower Temperature Heat Transfer

The purpose of this subtask is to measure the heat transfer characteristics of insulation samples fabricated from the foil-oxide combinations that the previous subtask established as being compatible. These include Al-ZrO₂, Cu-ZrO₂, Ni-ZrO₂, and Pt-ZrO₂. Al-Al₂O₃, Cu-Al₂O₃, Ni-Al₂O₃, and Pt-Al₂O₃ have been eliminated from further consideration.

The previous quarterly report contained detailed drawings of the test apparatus that has been designed to measure the anticipated

low heat fluxes. Also appearing in that report is an explanation of the test procedure that will be followed.

The low temperature heat transfer test apparatus has now been fabricated and a test station established in the insulation laboratory. Figure 6.1 presents a photograph of the test apparatus with a 12-inch long cylindrical sample of Ni-ZrO₂ insulation in place. Checkout testing of the apparatus is now in progress, and the lengthy low temperature test program is expected to begin shortly.

2.6 Task 7. Life Tests

Both short-term and long-term life tests are planned. The short-term life tests will last for 1000 hours and the long-term life tests are scheduled for 10,000 hours. In both cases, the test temperature will be 1800°C.

Subtask 7.1 Short-Term Tests

The test vehicle for the short-term tests will be the so-called "extended soak" test apparatus that was designed, fabricated, and extensively used throughout Phase I. Essentially, this apparatus consists of four high temperature ovens, each capable of accommodating two planar insulation test samples. The defining characteristics of the eight insulation samples that will be tested are tabulated in Table 7.1. Samples STLT 1, STLT 2, STLT 3, and STLT 4 will test the durability of the two spray coatings selected for the long-term life tests. Samples STLT 5, STLT 6 and STLT 7 will test the high temperature compatibility of zirconia with tungsten, molybdenum, and tantalum. Sample STLT 8 will contain both a pyrolytic graphite penetration and a zirconia

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Figure 6.1 Low Temperature Heat Transfer Test Apparatus.

Test Sample	No., Type of Foils	Oxide	Notes
STLT 1	20 layers Ta 20 layers W	Mixture 115 ThO ₂ Powder	One spray pass. Small (~ 1μ) particles.
STLT 2	20 layers Ta 20 layers W	Mixture 112 ThO ₂ Powder	One spray pass. Medium (0 - 10μ) particles.
STLT 3	10 layers Ta 30 layers W	Mixture 115 ThO ₂ Powder	One spray pass. Small (~ 1μ) particles.
STLT 4	10 layers Ta 30 layers W	Mixture 112 ThO ₂ Powder	One spray pass. Medium (0 - 10μ) particles.
STLT 5	40 layers Ta	Zirconium Corporation of America ZrO ₂	Tests compatibility of tantalum with zirconia.
STLT 6	40 layers Mo	Zirconium Corporation of America ZrO ₂	Tests compatibility of molybde- num with zirconia.
STLT 7	40 layers W	Zirconium Corporation of America ZrO ₂	Tests compatibility of tungsten with zirconia
STLT 8	13 layers Ta 13 layers W 13 layers Mo	Zirconium Corporation of America ZrO ₂	Tests compatibility of Ta, W, and Mo with zirconia and pyrolytic graphite.

TABLE 7.1 SHORT TERM LIFE TEST (STLT) SAMPLES

penetration. 1000 hours at 1800°C will provide an indication of whether any compatibility problems exist between the insulation and these penetrations.

It is worth noting that these compatibility tests were not originally part of Subtask 7.1. However, four empty chambers in the test apparatus were available and the opportunity was therefore taken to perform these necessary tests.

The test samples are 3.360-inches in diameter, which is the exact size required by the planar heat transfer test apparatus. The thermal performance of the eight test samples will be evaluated in the planar apparatus before and after the 1000-hour test. Differences in before and after performance, if any, will then be a measure of the degradation of the insulation.

Figure 7.1 presents a drawing of the "extended soak" test apparatus. A photograph of one of the individual constant temperature ovens during fabrication appears in Figure 7.2. The foil blanks for Samples STLT 1, STLT 2, STLT 3, and STLT 4 have been machined into disks of the proper diameter, but they have not as yet been sprayed. Samples STLT 5, STLT 6, and STLT 7 are completely fabricated and their zero-time performance has been evaluated in the planar apparatus. Sample STLT 8 remains to be fabricated.

Subtask 7.2 Long-Term Tests

The long-term life tests require construction of four conduction heater blocks of the general type built for the Phase I cylindrical and edge heat transfer test devices. These heater blocks contain very

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Figure 7.1 **Extended Soak Test Apparatus.**

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Figure 7.2 Test Oven.

reliable high temperature resistance heaters ideally suited for 10,000 hours operation. Manufacturing these heaters involves gun-drilling molybdenum rod, obtaining BeO tubing, and pressure bonding a complicated lamination. The heaters are definitely a "long lead-time" item and, in anticipation of this latter need, work has started on preparation of the four heater blocks.

III. PLANNED FOURTH QUARTER WORK

Task 2. Spray Coating

Planar insulation samples will be fabricated with lighter small- and medium-size particle coatings. Comparison of the data generated by testing these samples with existing data should help establish whether the large particles actually do produce better insulation, or whether the small-particle samples are simply coated too heavily.

Additional tests are planned with an applied compressive load acting on the insulation. Tests run to date indicate that approximately 5 psi is the maximum test load.

Task 3. W and W(25)Re

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

Task 4. Cylindrical Insulation

Cylindrical samples C1 through C6 will be evaluated in the cylindrical heat transfer test apparatus.

Task 5. Edge Heat Losses

The 40-layer concentric cup corner will be fabricated from the vapor-deposited tungsten cups that are being supplied by San Fernando Laboratories. The edge heat loss test apparatus will then be mounted on one of the vacuum systems and the performance of the corner evaluated.

Task 6. Lower Temperature Applications

Checkout testing of the low temperature heat transfer test apparatus will begin. If all goes well the actual low temperature tests will begin.

Task 7. Life Tests

The so-called "short-term" life tests will begin in the reconditioned Phase I extended soak test apparatus.

The four conduction heater block assemblies required for the 10,000-hour life tests will be designed and the necessary parts ordered. Depending on delivery, fabrication may also begin.