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

June 1968 - August 1968

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ABSTRACT

This report is the fourth 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 June 1968 through August 1968.

The zero-time performance measurements of the short-term life test samples have been completed and the 1000-hour soak period is now underway. A change in the appearance of tungsten foil exposed to high temperature was noted and a series of tests conducted to establish the cause of the change.

The tests designed to optimize the layer spacing in cylindrical samples have been completed and the minimum thermal conductivities were associated with the most tightly wrapped samples. By contrast, however, the minimum heat fluxes through a given number of layers were associated with layer spacings of 4.6 mils/layer.

The performance of Cu-ZrO₂, Al-ZrO₂, Ni-ZrO₂, and SS-ZrO₂ insulations have been evaluated at temperatures between approximately 200 and 700°C. Thermal conductivities on the order of 10⁻⁶ watts/cm. °C were measured.

I. INTRODUCTION AND SUMMARY

This report is the fourth 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 June 1968 through August 1968.

A new set of pressure measurements have been completed and an interesting result has emerged. The degree of sensitivity of performance to applied pressure appears to vary with particle size. A small particle sample was much more sensitive to applied pressure than was a large particle sample.

In the course of evaluating the zero-time performance of the short-term life test samples a change in the appearance of tungsten foil exposed to high temperatures was noted. A series of experiments sought to establish the cause of the observed change and a partially-substantiated explanation has been formulated.

The tests which sought to optimize the spacing between cylindrical layers of insulation have been completed. The minimum thermal conductivity values were associated with the most tightly wrapped samples but, for a given number of layers of insulation, the minimum heat fluxes penetrated insulation with layer spacings between 4 and 6 mils/layer. The optimum layer spacing therefore depends on the design requirements peculiar to each application.

The low temperature testing program is now well underway. Twenty layer samples of Ni-ZrO₂, Cu-ZrO₂, SS-ZrO₂, and Al-ZrO₂

insulation have been tested at source temperatures between 200 and 800°C. The thermal conductivity values were on the 10^{-6} watts/cm.°C scale. Twenty-layer Pt-ZrO₂ samples are now being fabricated for future testing, along with forty-layer Ni-ZrO₂, Cu-ZrO₂, SS-ZrO₂, and Al-ZrO₂ samples.

The 1000-hour extended soak tests (i. e., the short-term life tests) are now underway. The design of the 10,000-hour test vehicles has been completed and fabrication work is beginning.

II. COMPLETED FOURTH QUARTER WORK

2.1 Task 1. Program Management

A list of proposed revisions to the Phase II program plan has been submitted to the Atomic Energy Commission and Sandia Corporation. This list is currently being reviewed and a decision is expected during the next quarter. It is felt that the adoption of these changes will generate additional meaningful information about the properties of multi-foil insulation.

It has been proposed that two subtasks be deleted from the program and three subtasks and one full task be added. The specific changes recommended are as follows:

1. Delete Subtask 4.3--Non-Uniform Loading Cylindrical Heat Transfer Tests.
2. Delete Subtask 5.1--Edge Heat Losses - Analysis.
3. Add Subtask 2.5--Reproducibility Measurements.
4. Add Subtask 2.6--Layer Variations.
5. Add Subtask 5.3--Corner Fabrication Techniques.
6. Add Task 8--Emissivity Measurements.

The non-uniform loading cylindrical heat transfer tests would measure the additional thermal losses associated with loading a cylindrical insulation assembly along one element of the cylinder. These tests are considered expendable because recent experience

with insulation penetrations indicates that the insulation could be entirely unloaded without incurring excessive thermal losses.

The recommendation that the corner computer study be dropped is motivated largely by the requirement that additional resources be freed before the proposed new work can be pursued.

A brief description of the new subtasks follows.

Subtask 2.5 Reproducibility Measurements

The purpose of this subtask would be to evaluate the reproducibility of the performance of multi-foil insulation. Six or more planar insulation samples consisting of identical materials, an identical number of foil layers, and, hopefully, identical oxide coatings will be fabricated. The average oxide layer coating density and average oxide layer thickness of each sample will be measured. The thermal performance of each sample will then be evaluated in the planar heat transfer test apparatus and a comparison of the results will provide an indication of reproducibility.

Subtask 2.6 Layer Variations

All the planar insulation samples fabricated to date have consisted of 40 layers of foil. The purpose of this subtask would be to generate design data for 80, 120, 160, 200, and 240 layers of multi-foil insulation. Multi-layered insulations differ from bulk insulations in that the heat fluxes through the insulation are not directly related to thickness (i. e., the number of layers). Therefore, it is important that the relationship between performance and the number of layers be established.

Subtask 5.3 Corner Fabrication Techniques

The purpose of this subtask would be to investigate the possibility of using a laser to cut multi-foil insulation. Frequently, the optimum multi-foil insulation configuration cannot be fabricated because of limited foil-shaping capabilities. This problem would arise less frequently if techniques could be developed to cut the most difficult multi-foil edges with a laser.

There is little doubt that a laser can cut multi-foil insulation. A question that remains to be answered is whether adjacent layers of foil can be kept from fusing at the plane of the cut. A laser, because it imparts less energy to the work piece, appears to have a better chance of preventing fusion than an electron beam. Other questions to be resolved are how many layers of each foil can be cut simultaneously, and whether a smooth edge can be obtained.

Task 8. Emissivity Measurements

The objectives of this task would be three-fold. The emissivities of the bare metal foils used in multi-foil insulation would be measured, the emissivities of the same foils after spraying would be measured, and the influence of emissivity on insulation performance would be determined. Whereas Task 2 is devoted to optimizing the oxide coatings, this task represents a corresponding effort directed at the only other component of multi-foil insulation, i. e., the radiation shields.

When the original Phase II program plan was written the oxide coatings were much heavier than those in use now. The foils were completely covered and the emissivity of the substrate was irrelevant. The current spray coatings, however, are very sparse and in all likelihood the foil emissivity now directly influences insulation performance.

2.2 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 insulation samples subjected to uniform compressive stresses. The progress in each area will be discussed separately.

Subtask 2.3

The planar heat transfer test apparatus was used to evaluate the so-called "zero-time" performance characteristics of the insulation samples for the short-term life tests. The zero-time performances of these samples will be compared to the performances after 1000 hours at 1800°C.

The defining characteristics of the first four short-term life test samples are presented in Table 2.1. These samples were fabricated and tested and the curves labeled "FIRST" in Figures 2.1, 2.2, 2.3, and 2.4 present the results.

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Figure 2.1. STLT 1 Zero-Time Performance.

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Figure 2.2. STLT 2 Zero-Time Performance.

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Figure 2.3. STLT 3 Zero-Time Performance.

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Figure 2.4. STLT 4 Zero-Time Performance.

American Potash Thoria 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 THORIA POWDERS

After the samples were tested in the planar apparatus, they were examined and a disturbing result was noted. The once-shiny tungsten foil in each sample had developed a cloudy, murky appearance. The condition of the tungsten suggested that some sort of physical or chemical attack had taken place. The 1000-hour life tests were therefore postponed, rather than test what appeared to be defective samples, and a series of experiments were initiated to discover the cause of the change in appearance.

A more thorough examination of the test samples revealed that the outer surface of the top tungsten foil had not been affected, nor had the bottom surface of the tungsten foil adjacent to the top tantalum foil. The tantalum also was not affected. Only the interior tungsten surfaces in the insulation samples had lost their shiny appearance. Two other pieces of evidence were also noted.

A narrow band approximately $1/8$ of an inch wide at the outer edge of each interior circular foil shield was still shiny, and not all interior surfaces were equally affected. Some were less cloudy than others. This evidence indicates that tungsten surfaces freely exposed to the surrounding volume are not affected, while those buried in the interior portions of the insulation assembly are affected. The variations in degree of cloudiness observed between interior foils probably means that slight differences in spacing existed between layers.

One of the first possibilities that was considered was that the tungsten had reacted either with the thoria or with one of the minor impurities in the thoria, or with a left-over component of the organic binder used to spray the thoria. These possibilities were quickly eliminated by a test which involved placing bare tungsten foil in the same environment by a test which involved placing bare tungsten foil in the same environment (1700°C in vacuum) that had produced the cloudy tungsten foil in the insulation samples. The bare tungsten, which originally was shiny, also became cloudy, indicating that the change was not associated with the thoria or the spraying process.

Since the phenomena, at this point, appeared to originate with the tungsten, a fresh supply of tungsten foil was ordered from a new manufacturer. When the new tungsten arrived it too was exposed to 1700°C in vacuum. This time the foil did not lose its shiny appearance after the high temperature exposure. The problem, although not solved, appeared to have been successfully avoided and the new tungsten foil was used to fabricate replacements for the four original

short-term life test samples. The zero-time performance of the new samples was measured in the planar heat transfer apparatus, and the curves labeled "SECOND" in Figures 2.1, 2.2, 2.3, and 2.4 present the results.

The post-test examination unfortunately revealed that the original problem had reoccurred. Apparently when the replacement tungsten foil was fired at 1700°C in vacuum it was not held at temperature long enough to lose its shiny appearance. The time scale of the reaction seems to be on the order of a few hours rather than a few minutes.

Figure 2.5 presents a photomicrograph of one of the dull-appearing tungsten surfaces. By way of contrast, Figure 2.6 is a photomicrograph of a tungsten surface that also had been at 1700°C in vacuum for several hours, but had remained shiny. The lines that are visible are grain boundaries and at least a hundred-fold difference in grain size exists between the dull and shiny foil areas.

Figure 2.7 presents a photomicrograph of a shiny, as-received tungsten foil that was chemically etched to reveal the grain size. It is interesting to note that the as-received foil is shiny and has a small grain size, while the foil with the small grain size that has been exposed to 1700°C has a very dull appearance. The grain boundaries are believed to be the key to this apparent contradiction. The as-received tungsten foil with the small grain size has a very smooth, shiny surface because the grain boundaries are shallow. If the grain size does not increase when the foil is exposed to high temperature, thermal grooving deepens the grain boundaries, thereby

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Figure 2. 5. Photomicrograph of a Dull Portion of Tungsten Foil Exposed to 1700°C in Vacuum.

Figure 2. 6. Photomicrograph of a Shiny Portion of Tungsten Foil Exposed to 1700°C in Vacuum.

roughening the surface and accounting for the dull appearance. The tungsten foil with the very large grain size appears shiny because the total length of grain boundaries is vastly reduced and the overall surface is therefore very smooth.

The next important question is why does the grain size increase in foil areas immediately adjacent to the vacuum surrounding the insulation and not increase in the interior of the insulation where there is relatively poor access to the surrounding vacuum. This is a formidable question and, although a plausible explanation is at hand, more tests are necessary to either support or reject the various possibilities. It has been observed in refractory metals that minute impurities at the grain boundaries can retard, or totally impede, grain growth. If an impurity such as carbon were present at the tungsten foil grain boundaries, and if the carbon could be removed from the boundaries only in those foil regions near the edges of the insulation, the observed phenomena would indeed take place. If a partial oxygen pressure of 10^{-6} torr existed in the surrounding volume, carbon at the grain boundaries near the edges could react with the oxygen to form carbon dioxide and vaporize, freeing the grain boundaries of what might be the controlling impurity. (The vapor pressure of pure carbon is too low to expect it to vaporize in its elemental state.)

Some support for this theory is presented in Figure 2.8. Figure 2.12 is a photomicrograph of a tungsten foil surface from the interior of a packet of foils that had been exposed to 1700°C for

Figure 2.7. Photomicrograph of the As-Received Tungsten Foil (Chemically Etched to Reveal the Grain Boundaries).

Figure 2.8. Photomicrograph of Shiny Tungsten Foil Exposed to 1700°C in Vacuum After Pre-Firing in Dry Hydrogen at 1700°C.

several hours. The foil appeared shiny, and the photomicrograph confirms the expectation that the grain size would be larger. The reason the individual grains are large rather than small, as has been the usual case in the interior of a foil packet, is that the individual layers had been pre-fired at 1500°C in hydrogen. This prior exposure to high temperature, without neighboring foils impeding outgassing, apparently provided the opportunity for the removal of impurities that might later have impeded grain growth. The fact that the foils were fired in hydrogen is not considered important.

The fact that duplicates of each of the first four short-term life test samples were fabricated provides an opportunity for further coating density comparison. Figure 2.1 shows that approximately a 40% improvement in performance was realized by lightening the average coating density from 2.21 mg/cm.² to .17 mg/cm.². The situation in Figure 2.2 is confusing and will be put aside for the moment. The two curves in Figure 2.3 are believed to represent the first evidence of degradation in performance as a result of an oxide coating being too light, especially when these curves are compared to the curves in Figure 2.1 which also are for Mixture 115 ThO₂ particles. (The proportions of tungsten and tantalum foil are different but this is believed to be a small affect compared to the layer variations). In Figure 2.4 a slight improvement in performance is noted when the coating density decreased from .11 mg/cm.² to .08 mg/cm.². The "FIRST" curve in Figure 2.2 indicates a slight degradation in performance with a coating density of .037 mg/cm.²,

which implies that the optimum coating density is probably about .06 mg/cm². The "SECOND" curve in Figure 2.2 is very difficult to explain unless an error was made while measuring the coating density.

Subtask 2.4

Figure 2.9 is reprinted from the previous quarterly progress report. The performance of a forty-layer Mo-ThO₂ insulation sample is plotted for various applied compressive loads. The thorium particles used for this sample were the large, Mixture 116 particles. Figure 2.10 presents a new set of pressure data obtained by testing a sample coated with the very small, Mixture 115 particles. Both figures illustrate a strong relationship between applied load and performance, but a careful comparison reveals that the small particle test sample was even more sensitive to the applied load than was the large particle sample. This result, if it is valid, indicates that an insulation sample that will be subjected to an applied pressure should be sprayed with larger oxide particles.

This result might have been predicted without any testing. It is reasonable to expect the larger particles to more effectively separate the radiation shields under an applied load. Furthermore, the total contact area between particles and foil is larger when small particles are used, which means there are more contact resistances to be lowered by an applied load.

Further tests will seek to confirm or correct these results.

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Figure 2.9. Planar Pressure Loading Data - Large Particle Sample.

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Figure 2.10. Planar Pressure Loading Data - Small Particle Sample.

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

No work scheduled.

2.4 Task 4. Cylindrical Insulation

The cylindrical insulation testing program encompasses three subtasks. Subtask 4.1 has sought to optimize the spacing between adjacent layers of cylindrical insulation, Subtask 4.2 will study the performance of a cylindrical insulation sample subjected to uniform compressive stresses, and Subtask 4.3 is scheduled to investigate the effects of non-uniform compressive stresses.

Subtask 4.1 has recently been completed, Subtask 4.2 is about to begin, and Subtask 4.3 is being delayed pending a decision on the proposed program changes listed in Section 2.1. A discussion of the Subtask 4.1 test results follows.

Subtask 4.1

Table 4.1 lists the defining characteristics of the seven cylindrical insulation samples fabricated for this subtask. Each sample consisted of forty layers of insulation, and the outer diameter of each sample was 1.494 inches. The average layer spacings were varied from sample to sample by varying the inner diameter. Samples C1, C2, C3, and C7 were fabricated from tantalum foil, while Samples C4, C5, and C6 were of tungsten.

Figure 4.1 compares the thermal performance of Samples C1, C2, and C3. As the average layer spacing increased from 1.6 to 2.0

Test Sample	Foil Material	Oxide	Number of Layers	O. D.	I. D.	Average Layer Spacing
C1	.0005" Ta	Mixture 115 ThO ₂	40	1.494"	1.367"	.0016"
C2	.0005" Ta	Mixture 115 ThO ₂	40	1.494"	1.334"	.0020"
C3	.0005" Ta	Mixture 115 ThO ₂	40	1.494"	1.174"	.0040"
C4	.0004" W	Mixture 115 ThO ₂	40	1.494"	1.367"	.0016"
C5	.0004" W	Mixture 115 ThO ₂	40	1.494"	1.334"	.0020"
C6	.0004" W	Mixture 115 ThO ₂	40	1.494"	1.174"	.0040"
C7	.0005" Ta	Mixture 115 ThO ₂	40	1.494"	1.000"	.0062"

TABLE 4.1
CYLINDRICAL TEST SAMPLES

Figure 4.1. Performance Comparison - Samples C1, C2, and C3.

to 4.0 mils/layer, the thermal performance improved. Figure 4.2, however, shows that the performance of Sample C7 (6.2 mils/layer) was virtually the same as that of Sample C3 (4.0 mils/layer), indicating that the heat flux cannot be lowered still further by loosening the wraps beyond 4.0 mils/layer.

Figure 4.3 presents the corresponding results obtained by testing the tungsten foil insulation samples. Once again, the lower heat fluxes are associated with the more loosely wrapped insulation samples.

A somewhat different picture emerges when the thermal conductivities of the various samples are compared. The increases in sample thickness overshadow the decreases in heat flux, the net effect being that the mean thermal conductivities of the looser samples are higher than those of the tighter samples. Figures 4.4 and 4.5 illustrate this point.

These tests indicate that to minimize the thermal conductivity a cylindrical insulation sample should be wrapped as tightly as possible. With 0.5-mil foil the tightest assemblies fabricated to date allow 1.5 - 2.0 mils/layer. With 1-mil foil these numbers would increase accordingly. The minimum heat fluxes, however, result from a layer spacing of 4 - 6 mils/layer. The choice of whether to wrap a cylindrical assembly tightly or loosely depends on the intended application. If cost and weight are the dominant considerations, a loosely wrapped assembly minimizes the thermal losses for a given number of layers. If it is more important to minimize the volume of the insulation, a tightly wrapped assembly would be best. By the same token, a tightly

Figure 4.2. Performance Comparison - Samples C3 and C7.

Figure 4.3. Performance Comparison - Samples C4, C5, and C6.

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Figure 4.4. Thermal Conductivity Comparison - Samples C1, C2, and C3.

8548

Figure 4.5. Thermal Conductivity Comparison - Samples C4, C5, and C6.

wrapped assembly can squeeze more layers into a given thickness, thereby reducing the thermal losses to an absolute minimum, but at the expense of consuming additional foil and adding weight.

One final comparison can be made with the data collected for this subtask. Figures 4.6, 4.7, and 4.8 compare the thermal performances of the similarly spaced W-ThO₂ and Ta-ThO₂ insulation samples. In each case, the Ta-ThO₂ sample outperformed the W-ThO₂ sample. There are two possible reasons for this. Tungsten is much stiffer than tantalum and it is possible that, even though space was available, the tungsten effectively acted as a coiled spring and exerted a load on itself. This is reasonable at room temperature; it is less clear whether this is also possible at elevated temperatures where the foil would be less rigid. The other possible explanation for the apparent superiority of tantalum over tungsten involves the emissivities of the two materials. The tungsten foil used for these samples was .0004 inch thick. This thickness cannot be rolled in any significant quantity so it was necessary, as part of Task 3, to chemically etch the tungsten to the desired thickness. Before etching, the tungsten was bright and shiny with a highly reflective surface. After etching the bright finish had been replaced by a consistently dull finish. Although no measurements were taken, it is likely that the emissivity of the etched tungsten was higher than that of the tantalum. If foil emissivity influences insulation performance, and it is reasonable to expect that it does, an emissivity difference could have contributed to the superiority of Ta-ThO₂ insulation over W-ThO₂ insulation.

Figure 4.6. W-ThO₂ vs. Ta-ThO₂ - 1.6 mils/layer.

Figure 4.7. W-ThO₂ vs. Ta-ThO₂ - 20 mils/layer.

Figure 4.8. W-ThO₂ vs. Ta-ThO₂ - 4.0 mils/layer.

2.5 Task 5. Edge Heat Losses

The computer study of a concentric cup corner assembly (Subtask 5.1) has been delayed pending a decision on the recommended revisions discussed in Section 2.1.

The purpose of Subtask 5.2 is to measure the thermal performance of various insulation corner configurations. Early in the Phase II work the performances of overlap, tapered, and slit-cup corners were measured. The next objective was to evaluate the performance of a concentric-cup corner assembly. At the outset, an attempt was made to produce the required concentric foil cups by deep-drawing planar foil disks. A technique was developed that could produce one suitable cup at a time but it was not economically feasible to produce an entire set of concentric cups in this manner. Exploratory tests then established that 1-mil thick vapor-deposited tungsten cups were fabricable at a reasonable cost and an order was placed with San Fernando Laboratories for forty cups sized to fit together to form the desired concentric cup corner assembly.

A number of factors have contributed to a lengthy delay in the delivery of these cups. The vapor-deposited cups are as fragile as egg shell because the tungsten has never been worked. As a result, several cups broke in the course of normal handling and extreme measures were required to ship those still intact across the country. Originally the cups were packed in a gel which very effectively supported the side walls of the cups, but which proved to be very difficult to remove. Despite careful handling several cups cracked while the gel was being removed. Molded styrofoam

containers therefore replaced the gel, but the first cups that arrived packed in this manner were bonded to the styrofoam. Two cups were broken separating the cups from the styrofoam before tests established that styrofoam could be dissolved by acetone.

Table 5.1 presents an up-to-date status report on each of the forty cups. Five cups (Nos. 10, 35, 36, 38, and 40) are required to complete a workable set. Recent conversations with San Fernando Laboratories indicate that the missing cups will be available in mid-October. Five other cups are listed as being in-house, but broken. These cups are cracked, but not to the extent that they can't be used in their present state.

It should be noted that this experience with vapor-deposited tungsten cups has clearly established the fact that such cups would be highly unsuited for a real insulation application. Nevertheless, by exercising extreme care it will be possible to fabricate a 40-layer concentric cup corner assembly which, when tested, will establish whether concentric cup corners are worth pursuing further. If the scheduled tests do indicate that there are definite advantages associated with concentric cup corners, other techniques and materials will be investigated. For example, there is a small group of alloys that exhibit a property called "super-plasticity" that might enable cups to be deep-drawn economically. Similarly, vapor-deposited tungsten 25% rhenium or tantalum 10% tungsten might not exhibit the brittle characteristics of vapor-deposited tungsten.

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	In-house, intact
13	1.060	.624	In-house, intact	33	1.160	.664	In-house, intact
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	In-house, intact
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	In-house, intact
20	1.095	.638	In-house, intact	40*	1.195	.678	In-house, gel coated

*Returned to San Fernando, replacements due.

TABLE 5.1 VAPOR-DEPOSITED TUNGSTEN CUPS

2.6 Task 6. Lower Temperature Applications

The purpose of this task is to develop special types of multi-foil insulation for applications at temperatures roughly between 100°C and 1000°C. At these temperatures the expensive refractory foil materials such as tungsten and molybdenum can be replaced by less exotic metals such as nickel or copper.

The development of lower temperature insulation has been divided into two subtasks. Subtask 6.1 was devoted to establishing which foil materials and oxides were compatible with one another for extended periods at temperature. The results of this investigation were presented in the preceeding quarterly report. Subtask 6.2 involves measuring the thermal performance of insulation samples fabricated using compatible foil/oxide combinations, as established by Subtask 6.1.

Subtask 6.2 Lower Temperature Heat Transfer Tests

Detailed drawings and photographs of the test apparatus have appeared in previous quarterly reports. A particular insulation material combination such as Ni-ZrO₂ is evaluated by fabricating two cylindrical test samples that are identical except for their length. The difference between the input powers required to raise the interior cavities of a 6-inch sample and a 12-inch sample to the same temperature, divided by the difference in lateral areas between the two samples, represents the heat flux through the insulation at that source temperature. The same planar insulation subassemblies are used to control the end losses from the two cylinders. As a result, the end losses need not be measured; as long as they are the same for the 6-inch and 12-inch cases the technique is valid.

To date, the performances of 20-layer samples of Ni-ZrO₂, Cu-ZrO₂, Al-ZrO₂, and SS-ZrO₂ (stainless steel-zirconia) insulation have been measured. Figures 6.1, 6.2, 6.3, 6.4, and 6.5 present the unreduced test data, i. e., plots of the input powers required to reach various interior cavity temperatures for the 6-inch and 12-inch cylindrical test samples. Also appearing in these figures are plots of the input power differences.

The data presented in Figure 6.5 was obtained by retesting the same SS-ZrO₂ insulation samples used to generate the data in Figure 6.4. These samples were retested for two reasons. The thermocouple measuring the cavity temperature burned out before the last 12-inch data point could be obtained, and an indication of the reproducibility of the basic measurements was sought. A comparison of the data in Figures 6.4 and 6.5 indicates very close agreement between the corresponding test runs.

The accuracy of this technique decreases as the source temperatures drop below 200-300°C because the analysis depends upon the difference between two small, very nearly equal, numbers. This point is illustrated by Figure 6.6, which plots the heat fluxes based on the power differences for the various insulations. Above approximately 300°C the heat fluxes bear a reasonable relationship to one another. Below 300°C some inconsistencies are apparent. The relatively high heat flux through the Ni-ZrO₂ insulation may be a result of some experimental problems encountered in the first series of tests. These samples will be retested to either confirm or correct the early results.

Figure 6.1. Unreduced Low Temperature Ni-ZrO₂ Test Data.

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Figure 6.2. Unreduced Low Temperature Cu-ZrO_2 Test Data.

Figure 6.3. Unreduced Low Temperature Al-ZrO₂ Test Data.

Figure 6.4. Unreduced Low Temperature SS-ZrO₂ Test Data (Run #1)

Figure 6.4. Unreduced Low Temperature SS-ZrO₂ Test Data (Run #1)

Figure 6.5. Unreduced Low Temperature SS-ZrO₂ Test Data (Run #2).

Figure 6.6. Low Temperature Heat Flux Data.

Table 6.1 presents thermal conductivity values for the various insulations. All values fall on the 10^{-6} watts/cm. °C scale.

2.7 Task 7. Life Tests

The life test program consists of a preliminary stage (Subtask 7.1) and a final stage (Subtask 7.2). The preliminary tests will bake different types of multi-foil insulation for 1000 hours in vacuum at approximately 1800°C. The performance of the test insulations before and after high temperature exposure will be measured and compared. The long-term life tests will last for 10,000 hours at test temperatures of 1700°C and 1800°C. The insulation chosen for the long-term life tests will depend upon the results of the preliminary evaluation. A summary of the status of each subtask follows.

Subtask 7.1

The so-called "short-term" life tests are now underway. Figure 7.1 presents a photograph of the test apparatus in operation. The 1000-hour mark will be reached in late October..

The only incident that has occurred to date involved Sample STLT-8. This is the sample that contains the pyrolytic graphite and zirconia pins. The portion of the bell jar immediately opposite the pyrometer ports became coated with a black substance that may or may not be carbon. This is somewhat surprising since the tabulated vapor pressure of carbon at 1800°C is 10^{-6} torr. Regardless of the source of the deposit, it was no longer possible to obtain valid pyrometer measurements of the temperature in this oven. In an attempt to correct this situation, the ovens were cooled to room temperature,

Insulation	Mean Thermal Conductivity, watts/cm. °C x 10 ⁻⁶						
	200°C	300°C	400°C	500°C	600°C	700°C	800°C
Al-ZrO ₂	1.68	1.69	2.12	2.59	—	—	—
Cu-ZrO ₂	1.25	—	1.91	—	3.37	—	4.92
SS-ZrO ₂ ⁽¹⁾	1.09	1.09	1.28	1.88	—	—	—
SS-ZrO ₂ ⁽²⁾	.52	.69	1.29	1.96	2.91	4.44	—
Ni-ZrO ₂	.46	1.61	2.96	4.12	5.39	7.12	—

TABLE 6.1 MEAN THERMAL CONDUCTION

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Figure 7.1. Short-Term Life Test Apparatus.

the vacuum seal was broken, and the bell jar was rotated to a position which made the pyrometer ports visible again. Unfortunately, the outgassing, vapor-deposition, or whatever, was continued and, as a result, there is no direct measurement of the temperature of that oven. It is possible, however, to obtain an indication of the temperature by inference from the known temperatures of the other ovens, and the measured power inputs to the source heaters. The relationship between input power and temperature in each oven was very nearly the same before this problem arose.

Subtask 7.2

Figure 7.2 presents the final design of the four test vehicles for the long-term life tests. These devices face the exacting goal of operating for 10,000 hours at 1700 and 1800°C. Prior experience at Thermo Electron with such tests has made it clear that every possible precaution must be taken to insure the stable operation of these vehicles.

Eighty layers of mult-foil insulation will surround a heater block 2 inches long and 1 inch in diameter. The exact choice of an insulation will depend on the Subtask 7.1 results. Four identical test vehicles will be fabricated. Two will operate at 1700°C and two at 1800°C. The duplicate systems will provide an indication of the level of reproducibility that can be expected from complete insulation systems.

The conduction heater blocks specified in the design are similar to those fabricated for the cylindrical and edge loss devices. Eight conduction heating elements are embedded in a molybdenum block. This type of heater appears to have an excellent chance of operating without failure for 10,000 hours. Single elements have operated

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Figure 7.2. Final Design - 10,000 Hour Life Test Vehicle.

satisfactorily for comparable periods, and the multi-element heater blocks already in use in the program have never been a cause for concern.

The source temperatures will be measured by an optical pyrometer. This approach was chosen rather than a thermocouple approach because it is doubtful that a high temperature thermocouple would operate reliably for the entire test period. The outer containment vessel contains a sapphire window opposite a black body hole in the heater block. A hinged stainless steel panel shields the sapphire window from any deposits that might otherwise interfere with the required temperature measurements. The position of the panel will be controlled by the motion of a magnet outside the vacuum enclosure.

A titanium getter surrounds each insulation system. The getter will help maintain a static vacuum of at least 10^{-5} torr. The pressure in the vacuum chambers will be periodically measured and a series of vac-ion pumps will be available to lower the pressure if necessary. The titanium contains a number of holes to facilitate insulation outgassing.

The heater block is supported along its axis by a rigid coaxial heater lead. The weight of the block therefore does not rest on the insulation assembly. Nickel cable connects the coaxial heater lead with the terminals that penetrate the outermost stainless steel vacuum enclosure. The end of the cable is welded to a nickel ring which, in turn, is brazed to a nickel rod which serves as the lead-through.

Figure 7.3 illustrates how each test device will be connected through a series of valves to a roughing pump and a vac-ion pump. Figure 7.4 shows all four systems mounted in a common sheet metal structure with a frontal control panel.

Figure 7.3. Final Design - 10,000 Hour Life Test Vehicle.

Figure 7.4. Final Design - 10,000 Hour Life Test Vehicle.

III. PLANNED FIFTH QUARTER WORK

Task 1. Program Management

A decision will be reached on the proposed revisions to the Phase II Program Plan. This decision will influence the course of some work scheduled for the fifth quarter.

Task 2. Spray Coating

The spray coating optimization studies will continue as will the pressure testing program. The pressure tests will seek to establish whether the magnitude of the pressure dependency varies with particle size.

Task 3. W and W(25) Re Foil

No work scheduled.

Task 4. Cylindrical Insulation

The uniform loading cylindrical heat transfer tests will be completed.

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 installed on one of the vacuum systems and the performance of the corner evaluated.

Task 6. Lower Temperature Applications

20-layer Pt-ZrO₂ samples will be evaluated. 40-layer Cu-ZrO₂ Ni-ZrO₂, Al-ZrO₂, and SS-ZrO₂ samples will be fabricated and tested.

Task 7. Life Tests

The short term life tests will be completed and the fabrication of the four 10,000 hour test devices will be underway.