JAN 8 1968

MASTER

ALO 3634-7
UC-33
AEC RESEARCH AND
DEVELOPMENT REPORT

THERMO ELECTRON CORPORATION

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

September 1967 - November 1967

Issued December 29, 1967

No objection from the patent
office to the publication or
distribution of this document:
Patent Group (Brookhaven)
By.....*CHL*.....
.....1/4.....1968

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

JAN 8 1968

MASTER

ALO 3634-7
UC-33
AEC RESEARCH AND
DEVELOPMENT REPORT
TE Report No. 4059-77-68

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

September 1967 - November 1967

J. B. Dunlay
J. Fronduto
E. Peredetto
M. L. Paquin
V. L. Poirier

Contract No. AT(30-1)-3634
U. S. ATOMIC ENERGY COMMISSION
Issued December 29, 1967

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

leg

ALO 3634-7

UC-33

AEC RESEARCH AND
DEVELOPMENT REPORT

DISTRIBUTION

L. W. Otski, Area Manager
Sandia Area Office
U. S. Atomic Energy Commission
Albuquerque Operations Office
Albuquerque, New Mexico 87115
(2 copies)

A. J. Clark, Manager
Isotope Power Department
Sandia Corporation
Post Office Box 5800
Albuquerque, New Mexico 87115
(2 copies)

R. T. Carpenter, Chief
Isotopic Power Branch
Division of Space Nuclear Systems
U. S. Atomic Energy Commission
Washington, D. C. 20545
Attn: E. Redden
(1 copy)

S. J. Seiken
Division of Reactor Development
and Technology
U. S. Atomic Energy Commission
Washington, D. C. 20524
(1 copy)

J. E. Konklin, Acting Chief
Technology Development Branch
Special Projects Division
U. S. Atomic Energy Commission
New York Operations Office
376 Hudson Street
New York, New York 10014
(1 copy)

J. F. O'Brien
Martin Marietta Corporation
Nuclear Projects
Baltimore, Maryland 21203
(1 copy)

Douglas G. Harvey
Martin Marietta Corporation
Nuclear Products
Baltimore, Maryland 21203
(1 copy)

E. Ray
General Electric Company
Missile and Space Development
Valley Forge Space Technology Center
Post Office Box 8555
Philadelphia, Pennsylvania
(2 copies)

William Botts
Atomics International
Division of North American Aviation
Post Office Box 309
Canoga Park, California 91304
(2 copies)

Paul Underhill, Project Manager
Large Milliwatt Program
Aerojet-General Nucleonics
Post Office Box 77
San Ramon, California
(1 copy)

ALO 3634-7
UC-33
AEC RESEARCH AND
DEVELOPMENT REPORT

DISTRIBUTION (Continued):

L. Sisler, Isotope Power Laboratory
Minnesota Mining & Manufacturing
Company
2501 Hudson Road
St. Paul, Minnesota 55119
(2 copies)

Joseph Kenney, Program Manager
Compact Thermoelectric Converter
Westinghouse Electric Corporation
Post Office Box 10864
Pittsburgh, Pennsylvania 15213
(1 copy)

W. Kortier
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201
(1 copy)

Joseph Epstein
Goddard Space Flight Center
Greenbelt, Maryland
(1 copy)

Captain Morrow
Wright-Patterson Air Force Base
Dayton, Ohio
(1 copy)

John Collins
Johns Manville Research and
Engineering Center
Manville, New Jersey
(2 copies)

Fred Hittman, General Manager
Hittman Associates, Inc.
3517 E. Wabash Avenue
Baltimore, Maryland 21215
(1 copy)

Harmon S. Potter, Chief
New York Patents Group
U. S. Atomic Energy Commission
Brookhaven Area Office
Upton, New York 11973
(1 copy)

John Dunlay
Thermo Electron Corporation
Post Office Box 482
Waltham, Massachusetts 02154
(2 copies)

U. S. Atomic Energy Commission
New York Operations Office
376 Hudson Avenue
New York, New York 10014
Attn: Reports Library
(1 copy)

U. S. Atomic Energy Commission
Division of Technical Information
Extension
Post Office Box 62
Oak Ridge, Tennessee 37831
(3 copies and 1 reproducible copy)

William DeWitt
Union Carbide Corporation
Linde Division
Tonawanda, New York 14152
(2 copies)

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
ABSTRACT	9
I. INTRODUCTION AND SUMMARY	10
II. COMPLETED FIRST QUARTER WORK	12
Task 2. Spray Coating	12
Task 3. W and W(25)Re Foils	55
Task 4. Cylindrical Insulation	56
Task 5. Edge Heat Losses	57
Task 6. Lower Temperature Applications	101
Task 7. Life Tests	101
III. PLANNED SECOND QUARTER WORK	103

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
2.1	Mo foil sprayed with Fisher ThO_2	14
2.2	Fisher ThO_2 straight out of the bottle	15
2.3	Mo foil sprayed with Fisher ThO_2 before and after firing	17
2.4	Sifting pan, sieve, and cover	18
2.5	10, 20, and 45 μ sieves	19
2.6	Mo foil sprayed with ThO_2 that had accumulated between 10 and 20 μ sieves	20
2.7	ThO_2 particles which on three separate occasions failed to drop through the 20 μ sieve	22
2.8	Sieve pores clogged with ThO_2	23
2.9	Results of an independent spectroscopic analysis of American Potash ThO_2	25
2.10	American Potash ThO_2 Mixture 116 after grinding from the as-receiver "nugget" form to a relatively coarse powder form	26
2.11	American Potash ThO_2 particles (Mixtures 112 and 115); out-of-the bottle	27
2.12	Mo foils sprayed with American Potash ThO_2 Mixture 112; many particles larger than 4 μ are clearly visible	29
2.13	Mo foils sprayed with American Potash ThO_2 Mixture 115; roll time 24 hours, spray pressure 10 psi.	31
2.14	Mo foil sprayed with American Potash ThO_2 Mixture 115; a more dilute spray mixture was used. Roll time 26 hours, spray pressure 10 psi . .	32
2.15	Mo foil sprayed with American Potash ThO_2 Mix- ture 115; roll time 24 hours, spray pressure 50 psi	33

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
2.16	Mo foil sprayed with American Potash ThO ₂ Mixture 115, a comparison of foils sprayed at 10 and 50 psi. Roll time 24 hours.....	34
2.17	Mo foil sprayed with American Potash ThO ₂ Mixture 112; roll time 24 hours, spray pressure 10 psi .	35
2.18	Mo foil sprayed with American Potash ThO ₂ Mixture 116; roll time 24 hours, spray pressure 10 psi, extra nitrocellulose in binder (307X).....	37
2.19	Mo foil sprayed with American Potash ThO ₂ Mixture 116; roll time 24 hours, spray pressure 10 psi, extra nitrocellulose in binder (1085X).	38
2.20	Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO ₂ ; roll time 24 hours	40
2.21	Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO ₂ ; roll time 24 hours, spray pressure 10 psi, 1085X.....	41
2.22	Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO ₂ ; roll time 24 hours, spray pressure 30 psi, 1085X.....	42
2.23	Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO ₂ ; roll time 24 hours, spray pressure 50 psi, 1085X.....	43
2.24	Spray mixture rolling mill.....	45
2.25	Mo foil sprayed with American Potash ThO ₂ Mixture 112; spray pressure 10 psi, 307X.....	46
2.26	Mo foil sprayed with American Potash ThO ₂ Mixture 112; spray pressure 10 psi, roll time 5 hours, 1085X	47
2.27	Mo foil sprayed with American Potash ThO ₂ Mixture 112; spray pressure 10 psi, roll time 24 hours, 1085X	48

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
2.28	Mo foil sprayed with American Potash ThO ₂ Mixture 112; spray pressure 10 psi, roll time 100 hours, 1085X	49
2.29	Automated Foil Spraying Apparatus. Planar foil sample - front view	52
2.30	Automated Foil Spraying Apparatus. Cylindrical insulation sample	53
2.31	Automated Foil Spraying Apparatus. Planar foil sample - side view	54
5.1	Alternate Edge Configurations	58
5.2	Corner Regions - Regions I and III are identical in both analyses.	59
5.3	Concentric Cup Corner Nodal Network	61
5.4	A typical cup drawing punch and die combination	81
5.5	Deep drawn cup attempts - 1 mil Ni and Cu.	83
5.6	Deep drawn cup attempts - 5 mil Ni and Mo	84
5.7	Deep drawn cup attempts - 3 mil Mo	85
5.8	Deep drawn cup attempts - 3 mil Mo	86
5.9	Deep drawn cup attempts - 1 mil Mo	87
5.10	Deep drawn cup attempts - 1 mil Mo	88
5.11	Deep drawn cup attempts - 1 mil Mo	89
5.12	Deep drawn cup attempts - 1 mil Mo and 15 mil Cu	90
5.13	Deep drawn cup attempts - 1/2 mil Mo.	91
5.14	Deep drawn cup attempts - 1/2 mil Mo.	92
5.15	Deep drawn cup attempts - 1/2 mil Mo (slit cups)	93
5.16	Alternate Corner Configurations	95
5.17	Edge Heat Loss Data.	96

LIST OF FIGURES (Continued)

<u>Figure</u>		<u>Page</u>
5.18	Insulation test configuration.....	98
5.19	Edge Heat Loss Data - Improved Experimental Configuration.....	100
6.1	Material Compatibility Constant Temperature Oven...	102

ALO-3634-7
UC-33
AEC RESEARCH AND
DEVELOPMENT REPORT

VACUUM FOIL-TYPE INSULATION FOR
RADIOISOTOPE POWER SYSTEMS

by

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

ABSTRACT

This report is the first 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 September 1967 through November 1967.

Thoria powders containing small ($1/\mu$), medium ($\sim 20 \mu$), and large ($\sim 50 \mu$) particles were purchased. Spraying experiments established that the deposited particle size is independent of the spray mixture rolling time and the spray gun pressure. The deposited particles were the same size as the original particles. In order to standardize the spray pass, an automated foil spraying apparatus has been designed and fabricated.

400 feet of 1 mil tungsten foil and 50 feet of 1/2 mil tungsten 25% rhenium foil has been purchased. The tungsten will be chemically etched to 1/2 mil either in-house or at GE-NMPO.

The ability to deep draw thin molybdenum foil cups has been developed. These cups can be used to fabricate insulation corner assemblies. The Phase I computer program is being revamped to analyze a concentric cup corner.

I. INTRODUCTION AND SUMMARY

This report is the first 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 September 1967 through November 1967.

Thoria powders were purchased containing small ($1/\mu$), medium ($\sim 20\mu$), and large ($\sim 50\mu$) particles. The sizes of the particles deposited on a foil surface were compared with the particle sizes used in the corresponding spray mixtures. The deposited particles were the same size as the original particles. Specifically, the spray gun pressure and the elapsed spray mixture rolling time did not affect the deposited particle sizes. An automated foil spraying apparatus was designed and fabricated specifically to reproduce spray coatings. The total oxide layer thickness can now be determined as a function of the number of standard spray passes used to apply the coating, and the size of the particles in the spray mixture.

Sources of 1 mil tungsten foil and 1/2 mil tungsten 25% rhenium foil were located. Quantities of each were ordered and delivered. A decision will be made shortly about whether to chemically etch the 1 mil tungsten foil to 1/2 mil in-house or at GE-NMPO.

The ability to deep draw thin metal foil cups has been developed. These cups can be used in an insulation corner assembly. Perfect 1 mil thick molybdenum foil cups were drawn to a depth of approximately 1/2 inch. Thicknesses as small as 1/5 of a mil were also obtained, but only by chemically etching 1 mil cups. No more than two cups could simultaneously be drawn in a given punch and die combination. At present, this means a prohibitive number of dies would be required to fabricate a concentric cup insulation corner assembly. No attempts have been made yet to draw tungsten cups.

The Phase I computer program is being modified to analyze a concentric cup corner.

The edge heat losses through a 40-layer overlap corner were compared with those through a 40-layer concentric cup corner. The Phase I edge heat loss test assembly was used. The concentric cup corner was fabricated using foil cups that were slit radially inward near the outer circumference. These slits enabled several cups to be simultaneously drawn in one punch. Surprisingly, the overlap corner was approximately 15% better than the slit cup corner. The test configuration was then carefully scrutinized in an effort to locate any extraneous heat paths to the calorimeter. Several, rather subtle changes were made to the test configuration and when the 40-layer overlap tests were rerun, the heat losses were significantly reduced. The slit cup corner tests have not been rerun yet, but will be in the next quarter.

In preparation for the lower temperature materials compatibility tests four constant temperature ovens are now being fabricated. These ovens, and the Phase I compatibility fixtures, will restrict the total compatibility testing period to less than two months.

II. COMPLETED FIRST QUARTER WORK

2.1 Task 2. Spray Coating

Subtasks 2.1 and 2.2 received the most attention during the first quarter. The purpose of Subtask 2.1 is to determine the effect of the spray gun pressure, the spray mixture rolling time, and the size of the original thoria particles on the size of the particles deposited on the foil surface. Subtask 2.2 will determine the overall thickness of the oxide coating as a function of the number of spray passes, the spray gun pressure, the spray mixture rolling time, and the size of the original thoria particles. Each subtask will be discussed separately.

Subtask 2.1

The first step was to separate thoria particles according to their size. Three mixtures were sought. The first mixture was to consist of particles on the order of 1μ in size. The second mixture was to consist of particles of intermediate size ($\sim 20\mu$), and the third and coarsest mixture was to consist of particles on the order of 40μ in size.

ORIGINAL PLANS - The original plans were as follows. The Fisher Scientific Company manufactures a thoria powder which they claim contains particles principally between .5 and 2μ in size. This thoria mixture appeared to be an ideal source of very fine thoria powder. The intermediate and coarse thoria mixtures were to be extracted from a 325-mesh mixture manufactured by the Gallard-Schlesinger Chemical Corporation. A 325-mesh mixture contains particles ranging in size up to 44μ . 10, 20, and 45μ sieves were

purchased and sifting of the Gallard-Schlesinger thoria began. With these sieves three thoria mixtures were envisioned. The first was to consist of particles between 10 and 20 μ , the second was to consist of particles between 20 and 45 μ , and the last was to consist of particles larger than 45 μ . Unfortunately, the grouping of thoria particles by size became unexpectedly complicated.

FISHER THORIA - The first problem arose with the Fisher thoria. Figure 2.1 presents four photomicrographs of molybdenum foils sprayed with the Fisher thoria. The size of the deposited particles does not fall between .5 and 2 μ . Figures 2.1a and 2.1c clearly display many particles as large as 10 μ in size. The foil surface and the very small particles deposited on it are in focus in Figures 2.1b and 2.1d.*

Where did the 15 μ thoria particles come from? The foil surfaces had been sprayed with the Fisher thoria mixture which allegedly contained only particles between .5 and 2 μ in size. There were two possibilities. Either the spray gun, despite extensive cleaning, was partially clogged with larger particles from previous spray mixtures, or the Fisher thoria mixture did, in fact, contain these larger particles from the outset. Figure 2.2 proves that the latter was the case. Many particles much larger than 2 μ were present in the original mixture.

* The photomicrographs which appear throughout this report were taken at magnifications ranging from 307X to 1085X. At such high magnifications the depth of field is exceptionally shallow and only selected portions of most photomicrographs are in focus.

8009

(a)

Focused on larger ThO₂ particles.
Many particles are $\approx 10\mu$ in size.

(b)

Same as (a), except the smaller
particles are now in focus.

(c)

A 16μ particle is clearly visible

(d)

Focused on the foil surface

Figure 2.1 Mo Foil sprayed with Fisher ThO₂
Roll time - 24 hours. Spray pressure - 10psi

8008

(a)

Individual ThO_2 particles are clearly visible

(b)

Several 20μ particles. The two irregularly shaped particles in the center are probably many smaller particles on top of one another.

Figure 2.2 Fisher ThO_2 straight out of the bottle

While working with the Fisher thoria, photomicrographs were taken of molybdenum foils before and after the foils were fired to remove the spray binder. These photomicrographs, which appear in Figure 2.3, show that no noticeable cracking of the thoria particles occurs during firing. The particles are as large after firing as they are before firing.

GALLARD-SCHLESINGER THORIA - The cover, the pan, and one of the sieves used to separate the Gallard-Schlesinger thoria are shown in Figure 2.4. Figure 2.5 presents photomicrographs of the 10, 20, and 45 μ sieve screens. The sieves were stacked in a vibrator with the largest sieve on top and piles of thoria were sifted. After several hours, about 10% of the original thoria remained above the 45 μ sieve. Approximately 70% of the thoria was between the 20 and 45 μ sieves and the remaining 10% was just above the 10 μ sieve. This indicates that the bulk of the 325-mesh thoria consists of particles between 20 and 45 μ in size which, at the time, seemed reasonable. Figure 2.6 presents two photomicrographs of molybdenum foil sprayed with thoria that had accumulated between the 10 and 20 μ sieves. A scale marking off 10 and 20 μ appears on each photomicrograph. Clearly, the deposited particles do not fall between 10 and 20 μ in size. Most of the particles are 3 or 4 μ in diameter.

Similar results were obtained with spray mixtures assembled using thoria that had accumulated between the 20 and 45 μ sieves, and thoria that had failed to pass through the 45 μ sieve. The particles photographed on the foil surface were very similar to the particles in

8010

(a)

Before firing

(b)

Before firing, the large particles are out of focus; the small particles are in focus.

(c)

After firing. Focused on larger particles.

(d)

After firing. Same as (c) except the smallest particles are now in focus

Figure 2.3 Mo Foil sprayed with Fisher ThO_2 . Before and after firing.
Roll time - 24 hours. Spray pressure - 10 psi.

8005

Figure 2.4 Sifting pan, sieve and cover.

8007

(a)

10μ sieve

(b)

20μ sieve

(c)

45μ sieve

Figure 2.5 10, 20 and 45μ sieves

8006

(a)

No particles as large as 10μ are present.
Note the scale marking off 10 and 20μ .

(b)

Again, none of the particles are as big
as expected.

Figure 2.6 Mo Foil sprayed with ThO_2 that had accumulated
between the 10 and 20μ sieve.

Figure 2.6. Their sizes definitely were not the sizes expected, considering the ultimate position of the thoria particles in the stratified sieve structure.

Two factors appear to have contributed to the ineffective particle separation. The vast majority of particles in the Gallard-Schlesinger 325-mesh thoria mixture were about 5μ in size. 20, 30, and 40μ particles were very rare. Figure 2.7 presents two photomicrographs of thoria particles which, on three separate occasions, failed to fall through the 20μ sieve. The particles are not all larger than 20μ ; in fact, only a tiny percentage of the particles were larger than 20μ . The vast majority of the particles are between 2 and 5μ in size. Figure 2.8 explains why these particles failed to drop through the 20μ sieve. The 20μ sieve (and also the 10μ sieve) becomes clogged almost immediately with thoria particles which have fallen into the vacant pores. The clogging is particularly noticeable when one compares the clogged sieves of Figure 2.8 with the unclogged sieves shown in Figure 2.5.

AMERICAN POTASH THORIA - Since neither the Fisher thoria nor the Gallard-Schlesinger thoria contained particles in the exact size ranges required by this program, new sources of thoria powders were sought. The Rare Earth Division of the American Potash and Chemical Corporation was among those contacted. American Potash sells three thoria powders which differ by the sizes of the particles contained in each. They say that their Mixture 115 thoria contains particles all less than 1μ in size. Their Mixture 112, on the other hand, is said to contain particles exclusively between 1 and 4μ . 90% of the mass of their last

8023

(a)

No particles are as large as the 10μ scale

(b)

Most of the particles are $2-4\mu$ in size

Figure 2.7 ThO_2 particles which on three separate occasions failed to drop through the 20μ sieve

thoria powder, Mixture 116, is allegedly contained in particles larger than 44μ in size. Samples of each mixture were requested and received.

A few grams of each mixture were sent to the Kent Analytic Laboratories in Newton, Massachusetts. The results of their semi-quantitative spectroscopic analysis are shown in Figure 2.9. The coarse thoria, Mixture 116, contained somewhere between .1 and 1.0% each of magnesium and cobalt. This is a slightly higher level of contamination than those encountered during the Phase I material compatibility tests. It is difficult to predict what effect, if any, this will have on the compatibility of the thoria with the various foil materials. The other American Potash thoria powders, Mixtures 112 and 115, were at least 99.90% thoria.

The Mixture 116 thoria, as-received, was practically in what one could call a "nugget" form. Many particles were one-eighth of an inch or more in diameter which, of course, was too coarse. The mixture was therefore ground, by hand, in the thoria booth into a less coarse, relatively homogeneous, powder. The powder was then examined under a microscope and the photomicrographs which appear in Figure 2.10 were taken. The 80μ particle in Figure 2.10c was the largest observed. Generally speaking, the majority of the mass of the mixture was concentrated in particles between 40 and 70μ . The inevitable tiny thoria particles were still present, but they account for only a small fraction of the total mass of the mixture.

Figure 2.11 presents photomicrographs of the thoria particles taken from the 112 and 115 mixtures. Mixture 115 was supposed to contain particles less than 1μ in size and, as Figure 2.11a shows, this does

8022

(a)

10 μ sieve. The dark pores are unclogged.
The others are clogged.

(b)

20 μ sieve. One pore is unclogged.

Figure 2.8 Sieve pores clogged with ThO₂

8021

Laboratory No. A-4885

Report from

Kent Laboratories
box 626 • 2276 Washington Street
Newton Lower Falls • Mass • 02162

*Spectrographic and
Chemical Analysis
Tel. 244-8366*

TO Thermo Electron Corporation
65 First Avenue
Waltham, Massachusetts 02154

DATE 12/6/67

TELEPHONED

CONCERNING

ORDER NO. 28311

Thorium Oxides, Specimens #61

	Th	Mg	Co	Cu	Si	Mn	Fe	Ni	Zn	B	Al	Cr	Sn
Amer. Pot. (A)	A	f	3g	g	e	g	3e	3g	3g	3g	f	3f	3g
" " (B)	A	c	c	f	d	3g	3e	f	3f	3f	g	3g	3g
" " (C)	A	e	3f	3f	3f	3g	3e	f	-	-	3g	e	3g
				Pb	Ti	Mo	Ba	Sr					
				f	3g	-	f	-					
				f-	-	3g	3f	e					
				f-	3g	g	-	g					
A	American Potash ThO ₂ Mixture 115												
B	American Potash ThO ₂ Mixture 116												
C	American Potash ThO ₂ Mixture 112

Rockwell Kent III
Manager

Semi-quantitative code:

A = 10-100%, B = 1-10%, C = 0.1-1%, d = 0.01-0.1%, e = 0.001-0.01%, f = 0.0001-0.001%, g = 0.00001-0.0001%. A number before the letter multiplies the range. Thus 3B = 3-30%, 3e = 0.003-0.03%.

The following elements were sought. If they were not detected, they are not present or are below the sensitivity of the method used. Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Ge, Hg, In, Ir, K, Li, Mg, Mn, Mo, Nb, Ni, Os, P, Pb, Pd, Pt, Rb, Re, Rh, Ru, Sb, Si, Sn, Sr, Ta, Te, Th, Ti, U, V, W, Zn, Zr.

Figure 2.9 Results of an independent spectroscopic analysis of American Potash ThO₂

8020

(a)

(b)

(c)

(d)

Figure 2.10 American Potash ThO_2 Mixture 116 after grinding from the as-received "nugget" form to a relatively coarse powder form

8019

(a)

Mixture 115 ThO_2 particles. The particles all appear to be less than 1μ in size, as claimed by the manufacturer

(b)

Mixture 112 ThO_2 particles. Many particles are larger than the 4μ limit claimed by the manufacturer. (These particles were photographed while suspended in the liquid spray binder; hence the out-of-focus appearance).

Figure 2.11 American Potash ThO_2 particles; out-of-the-bottle

in fact appear to be the case. Mixture 112, however, is not made up exclusively of particles between 1 and 4μ . As Figure 2.11b shows, there are larger particles intermingled with the many small particles. Figure 2.12 presents three more photomicrographs of particles from Mixture 112. Because these particles have been sprayed onto molybdenum foils, they are more widely separated from one another and therefore are easier to view individually. Particles on the order of 25μ in size apparently are not uncommon in Mixture 112.

It was actually a stroke of luck that American Potash's Mixture 112 thoria did not contain particles exclusively between 1 and 4μ . Mixture 112 is a satisfactory "intermediate" thoria powder precisely because it does contain a significant number of $15\text{-}25\mu$ particles. That mixture, together with the "fine" thoria powder (Mixture 115) and the "coarse" thoria powder (the reworked Mixture 116), completed the required set of thoria powders needed to begin the foil spraying experiments.

DEPOSITED PARTICLES SIZE AS A FUNCTION OF ORIGINAL PARTICLE SIZE - Three separate spray mixtures were prepared - one using Mixture 115 thoria, another using Mixture 112 thoria, and the last using Mixture 116 thoria. Each spray mixture was rolled for 24 hours and then a series of molybdenum foils were sprayed with the different spray mixtures. In all cases, the spraying pressure was 10 psi. The goal was to determine the relationship between the size of the particles deposited on the foil surface and the size of the original particles used in the spray mixture. It was felt that either the rolling of the spray mixture or the spraying of the foils, or both, could break larger particles into smaller particles.

8018

(a)

(b)

(c)

Figure 2.12 Mo foils sprayed with American Potash ThO_2 Mixture 112; many particles larger than 4μ are clearly visible.

Figure 2.13 shows four photomicrographs of molybdenum foils sprayed with Mixture 115 thoria powder. It is immediately apparent that the deposited particles are the same size as the original particles. The most striking result from this spray test is the non-uniform coverage of the foil surface by the thoria particles. This is very clear in Figures 2.13a and 2.13c. The large solid dark spots are not individual particles, but rather piles of smaller particles caused by "spitting" by the spray gun. Figures 2.13b and 2.13d represent attempts to focus on these thoria piles.

The non-uniform coverage is a result of particles leaving the spray gun nozzle in clumps and having time to only partially disperse before striking the foil surface. In an effort to eliminate blotchy spray patterns more butyl alcohol was added to the spray mixture, effectively "thinning" it, and hopefully reducing the cohesion between particles. The result appears in Figure 2.14; thinning the spray mixture failed to eliminate the non-uniform particle coverage. The next idea, however, was more successful. Figure 2.15 presents two photomicrographs of a molybdenum foil sprayed with a normal, undiluted, spray mixture, but at a spray pressure of 50 psi rather than 10 psi. Figure 2.16 presents photomicrographs taken at approximately one-half the magnification of those in Figures 2.14 and 2.15. The foils sprayed at 10 psi were covered with particle blotches, but when the spray gun pressure was raised to 50 psi the particle coverage became far more uniform.

Figure 2.17 presents four photomicrographs of a molybdenum foil surface sprayed with Mixture 112 thoria. The particles deposited on the foil surface appear to be the same size as those found in the original thoria powder. In contrast to the small particles in Mixture 115,

8016

(a)

Focused on foil surface;
note the blotchy spray
pattern

(b)

Focused on particle
"pile" in center

(c)

Focused on foil surface;
individual particles are
less than 1μ in size

(d)

Focused on particle
"pile" in center

Figure 2.13 Mo foils sprayed with American Potash ThO_2 Mixture 115;
Roll time - 24 hours, Spray pressure - 10psi

8017

Figure 2.14 Mo foil sprayed with American Potash ThO_2 Mixture 115; a more dilute spray mixture was used. Roll time - 26 hour
Spray pressure - 10psi. The non-uniform particle coverage persists.

8015

(a)

The higher spray pressure has resulted in more uniform particle coverage

(b)

Individual particles, although very small, are visible. Note the scale.

Figure 2.15 Mo foils sprayed with American Potash ThO_2 Mixture 115;
Roll time - 24 hours, Spray pressure - 50 psi

8014

(a)

10 psi spray pressure

(b)

10 psi spray pressure

(c)

50 psi spray pressure

(d)

50 psi spray pressure

Figure 2.16 Mo foils sprayed with American Potash ThO_2 Mixture 115;
a comparison of foils sprayed at 10 and 50 psi.
Roll time - 24 hours

8013

(a)

Focused on larger particles

(b)

Same as (a), except
focused on foil surface

(c)

Focused on larger particles

(d)

Same as (c), except
focused on foil surface

Figure 2.17 Mo foil sprayed with American Potash ThO_2 Mixture 112;
Roll time - 24 hours, Spray pressure - 10 psi

the larger particles of Mixture 112 were dispersed uniformly on the foil surface when the spraying pressure was 10 psi.

A unique situation arose during the preparation of the coarse thoria powder spray mixture. Because the larger particles were heavier they sank very quickly in the binder solution. Two or three seconds after the rolling of the spray mixture stopped the suspended particles had settled at the bottom of their container. It would have been very difficult, if not impossible, to spray such a solution of rapidly settling particles. Therefore, to improve the particle suspension, additional nitrocellulose was added to the binder, effectively increasing the binder's viscosity.

Figure 2.18 presents four photomicrographs of molybdenum foil sprayed with the more viscous binder. Figures 2.18a and 2.18b were taken before the foils were fired to remove the binder. The binder is visible as a faint "halo" around groups of particles. In Figures 2.18c and 2.18d, which were taken after the foil had been fired, the binder has disappeared. All the photomicrographs appearing in Figure 2.18 were taken at relatively low magnification (307X). The reader is therefore cautioned to compare individual particles with the scale superimposed on each photomicrograph before reaching any conclusions about particle size. Figure 2.19 presents four additional photomicrographs taken at a higher magnification. Once again, two were taken before firing and two after firing. The particles deposited on the foil surface are just as large as the particles in the original thoria powder.

In summary, with all three spray mixtures the deposited particles were the same size as the original particles. In the case of the very small

8028

(a)

Before firing. Note binder.

(b)

Before firing. Note binder.

(c)

After firing. Binder has disappeared.

(d)

After firing. Binder has disappeared.

Figure 2.18 Mo foil sprayed with American Potash ThO_2 Mixture 116; roll time 24 hours, spray pressure 10 psi, extra nitrocellulose in spray binder (307x).

8029

(a)

Focused on large
particles

(b)

Same as (a), except
focused on smaller
particles

(c)

Focused on larger
particles

(d)

Same as (c), except
focused on smaller
particles

Figure 2.19 Mo foil sprayed with American Potash ThO_2 Mixture 116;
roll time 24 hours, spray pressure 10 psi, extra nitrocellulose
in binder (1085x).

thoria particles, a spray pressure in excess of 10 psi was required to achieve uniform particle coverage on the foil surface. In the case of the very large thoria particles, a more viscous binder was required to properly spray the particles.

DEPOSITED PARTICLE SIZE AS A FUNCTION OF SPRAY

PRESSURE - Molybdenum foils were sprayed at 10, 30, and 50 psi pressure with a spray mixture containing Gallard-Schlesinger 325-mesh thoria particles. The spray mixture had been rolled 24 hours. These spraying tests were designed to determine whether the spraying pressure affects the size of the particles deposited on the foil surface. Figure 2.20a is a photomicrograph of a foil surface sprayed at 10 psi. The foil surface in Figure 2.20b was sprayed at 30 psi, while that in Figure 2.20c was sprayed at 50 psi. The foil sprayed at 10 psi exhibits the same non-uniform particle coverage observed when spraying American Potash's Mixture 115 thoria at 10 psi. The foil sprayed at 30 psi has a slightly heavier particle coating than the other two foils, but the particles comprising this coating are the same size as those on the other foils, and those in the original thoria powder. Figures 2.21, 2.22, and 2.23 present additional photomicrographs, taken at a higher magnification, of the foils sprayed at 10, 30, and 50 psi respectively.

In summary, the spraying pressure does not appear to effect the size of the particles deposited on the foil surface. Small particles, however, tend to form blotches on the foil surface when the spraying pressure is approximately 10 psi. Larger particles, and small particles sprayed at higher pressures, are distributed fairly uniformly.

8035

(a)

Spray pressure 10 psi. Note the non-uniform coverage.

(b)

Spray pressure 30 psi. A slightly heavier coat was applied.

(c)

Spray pressure 50 psi

Figure 2.20 Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO_2 ; roll time 24 hours

8030

(a)

(b)

Figure 2. 21 Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO_2 ; roll time 24 hours, spray pressure 10 psi, 1085x.

PAGE BLANK

8032

(a)

(b)

Figure 2.23 Mo foil sprayed with Gallard-Schlesinger 325-mesh ThO_2 ; roll time 24 hours, spray pressure 50 psi, 1085 x.

DEPOSITED PARTICLE SIZE AS A FUNCTION OF SPRAY MIXTURE ROLLING TIME - A spray mixture is prepared by mixing properly proportioned amounts of thoria powder and spray binder. The best spray coatings are obtained when the thoria powder is suspended uniformly in the spray binder. The thoria particles, however, have a natural tendency to settle at the bottom of their container unless the spray solution is constantly mixed. The rolling mill appearing in Figure 2.24 is used to constantly mix the spray solutions. This sub-task is designed to ascertain whether the rolling process breaks larger thoria particles into smaller thoria particles. If this were, in fact, the case the total rolling time would affect the size of the thoria particles deposited on foil surfaces.

A spray mixture was prepared using American Potash thoria, Mixture 112. After 5, 24, and 100 hours of rolling a portion of the total spray mixture was used to spray molybdenum foils. When the total rolling time reaches 500 hours a final set of foils will be sprayed.

Figure 2.25 presents photomicrographs of three foils. The spray mixture used to spray the first foil was rolled 5 hours. The spray mixture used to spray the second foil was rolled 24 hours and the spray mixture used to spray the third foil was rolled 100 hours. The particle density on the three foil surfaces varied slightly from foil to foil, but this variation has nothing to do with the rolling time. The important point is that the sizes of the particles on each foil surface are similar, which indicates that rolling the spray mixture does not break the particles in the spray mixture. Figures 2.26, 2.27, and 2.28 present additional photomicrographs, taken at a higher magnification, that further support the aforementioned conclusion.

8034

(a)

At rest; the spray solution has collected at the bottom of the bottle.

(b)

During rolling; note the mixing effect.

Figure 2. 24 Spray mixture rolling mill.

8043

(a)

Roll time 5 hours.

(b)

Roll time 24 hours.

(c)

Roll time 100 hours.

Figure 2.25 Mo foil sprayed with American Potash ThO_2 Mixture 112; spray pressure 10 psi, 307 x.

8033

(a)

Focused on large
particle

(b)

Same as (a); except
focused on smaller
particles

(c)

Focused on large
particles

(d)

Same as (c); except
focused on smaller
particles

Figure 2. 26 Mo foil sprayed with American Potash ThO_2 Mixture 112; spray pressure 10 psi, roll time 5 hours, 1085x.

8036

(a)

Focused on large
particles

(b)

Same as (a), except
focused on smaller
particles

(c)

Focused on larger
particles

(d)

Same as (c), except
focused on smaller
particles

Figure 2. 27 Mo foil sprayed with American Potash ThO_2 Mixture 112;
spray pressure 10 psi, roll time 24 hours, 1085x

8044

(a)

Before firing. Note binder.

(b)

Before firing. Note binder.

(c)

After firing. Binder has disappeared.

(d)

After firing. Binder has disappeared.

Figure 2. 28 Mo foil sprayed with American Potash ThO_2 Mixture 112; spray pressure 10 psi, roll time 100 hours, 1085x

In summary, the total elapsed rolling time of the spray mixture prior to the actual spraying process does not affect the size of the particles deposited on foil surfaces.

SUBTASK 2.1 CONCLUSIONS - The general feeling during the latter stages of Phase I was that the particles sprayed onto a foil surface were smaller than the particles in the original thoria powder. This implies that either the rolling process or the spraying process, or both, were breaking larger particles into smaller particles. The Phase II work just completed was designed to establish which process reduced the average particle size. Subtask 2.1, however, indicates that neither the rolling nor the spraying reduces the average particle size. Why, then, did the deposited particles appear smaller during Phase I? The answer lies with the Gallard-Schlesinger thoria that was used at that time. Its 325-mesh designation indicates that the powder contains particles up to 44μ (1.73 mils) in size. It was therefore reasonable to expect adjacent foil layers covered with oxide to be, on the average, about a mil and one-half apart. The observed average foil spacing however was less than one mil, indicating that the particles between foils were considerably smaller than 44μ . Whereas during Phase I the evidence indicated that the larger particles were being broken into smaller particles, the photomicrographs taken during Phase II have revealed that the vast majority of the particles in the 325-mesh mixture are very small before rolling and spraying. Therefore a more accurate knowledge of the particle sizes in the so-called 325-mesh powder has dispelled the theory that large particles were broken down into smaller particles.

Subtask 2.2

As originally conceived, Subtask 2.2 was to determine the overall thickness of an oxide coating as a function of the number of spray passes, the size of the original thoria particles, the spray gun pressure, and the spray mixture rolling time. Subtask 2.1, however has already established that the coating thickness will be independent of the spray pressure and the rolling time. In contrast, the coating thickness will be a function of the number of spray passes, provided that a reproducible "standard spray pass" is created. The reproducibility of spray coatings has, to date, been very subjective. Metal foils have been sprayed by passing them, by hand, in front of a spray gun. The coatings therefore are "light" or "heavy" depending on how fast the technician's hand moves.

To standardize the foil spraying process an automated foil spraying apparatus has been designed and fabricated. Three photographs of this apparatus appear in Figures 2.29, 2.30, and 2.31. Either one or two spray guns are mounted on posts in front of the spraying area. The apparatus will spray either a continuous roll of foil, as in Figure 2.30, or individual planar pieces as in Figures 2.29 and 2.31. Planar foil sections are placed on a motor driven conveyor belt which slowly lowers the foil at a reproducible speed into, and then out of, the oxide spray pattern. The conveyor belt is made from flexible screening so that the impinging air stream will pin the target foil against the conveyor belt. It was felt that a solid conveyor belt would reflect the air stream and blow the foil out of the target area.

Cylindrical insulation samples can be simultaneously sprayed and wrapped using the automated foil spraying apparatus. The unsprayed

8011

Figure 2.29. Automated Foil Spraying Apparatus.
Planar Foil Sample - Front View.

8012

Figure 2.30. Automated Foil Spraying Apparatus.
Cylindrical Insulation Sample.

8024

Figure 2.31. Automated Foil Spraying Apparatus.
Planar Foil Sample - Side View.

foil is placed on a feeder roll at the base of the apparatus. A constant tension device controls the drag as the foil is unrolled from the feeder roll. The foil is pulled upward through the impinging oxide spray pattern and is wound, once and for all, onto a properly-sized cylinder squeezed between two nylon cones at the top of the apparatus. The tension at which the sample is wrapped is controlled by the drag acting on the feeder roll. When spraying cylindrical insulation the conveyor belt may or may not be used. The foil roll is driven by an independent drive system which has its own speed control.

With the aid of the automated foil spraying apparatus it will be a relatively straight-forward job to measure oxide layer thicknesses as a function of the original particle sizes and the number of spray passes. The thickness of fifty layers of foil before and after spraying will be measured and the difference, divided by fifty, will be the thickness of one oxide layer.

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

Fifty feet of .0005 in. thick W(25)Re foil has been purchased from the H. Cross Company of Weehawken, New Jersey. The maximum available width of the foil (2 in.) was purchased. Wider pieces will be fabricated by spot-welding together smaller pieces.

The W(25)Re foil will be used in the fabrication of insulation corners when the source temperature is so high that only W-ThO₂ or W(25)Re - ThO₂ insulation can be used. W foil that has been exposed to very high temperatures becomes brittle when it is cooled

to room temperature. W(25)Re foil, however, remains relatively ductile at room temperature and therefore is ideal for high temperature insulation systems, and in particular corner configurations that will be thermally cycled. The W(25)Re foil will be used in Task 5 (Edge Heat Losses) and Task 7 (Life Tests). The fifty feet presently in stock represents approximately 25% of the total program needs.

In addition, 400 feet of .0010 in. W foil has been purchased from the Kulite Tungsten Company of Ridgefield, New Jersey. Half the W was 6 in. wide and the other half was 4 in. wide. The thickness of the foil will be reduced to .0005 in. by chemically etching the foil with molten NaOH. A decision will be made shortly about whether to do the etching at Thermo Electron or at GE-NMPO.

2.3 Task 4. Cylindrical Insulation

A new test fixture is currently being designed. This test fixture will measure the heat losses through cylindrical insulation samples that are subjected to non-uniform pressure loading. The non-uniform loading will result when a cylindrical heat source is supported on its side by the surrounding insulation. The weight of the heat source will therefore be carried by a relatively thin strip of the cylindrical insulation sample. The original Phase II program plan called for the design, fabrication and partial testing of this apparatus in FY1968. At the request of the Atomic Energy Commission, however, the fabrication and testing of this test device will be deferred until FY1969. Only the design will be finalized in FY1968.

2.4 Task 5. Edge Heat Losses

Edge heat loss activities during this quarter fell into three areas. The Phase I computer analysis is being modified to study a more sophisticated edge configuration. A concerted effort has been made to fabricate very thin metal foil cups which, when placed concentrically within one another and coated with oxide, could be used to fabricate insulation corner assemblies. And lastly, two corner configurations have been tested in the edge heat loss test assembly. Each area of activity will be discussed separately.

Subtask 5.1 Edge Loss Computer Study

The computer program developed during Phase I calculated the heat losses through overlap corner configurations. An analytical model of an overlap corner was formulated and an iterative calculation of the equilibrium temperature distribution in the corner was sought. Based on the equilibrium temperature distribution the heat losses through the corner were calculated. The Phase I final report describes the model in great detail.

After several weeks of experimentation in the machine shop it became apparent that the ability to draw this metal foil cup was being developed. Several appropriately sized cups, placed concentrically and sprayed with oxide, would constitute the ultimate edge configuration - a true concentric cup corner (Figure 5.1b). An analytical study of such a corner was therefore undertaken.

The Phase I study divided the overlap corner into three regions which are defined in Figure 5.2a. The concentric cup corner analysis will also divide the corner into three regions (Figure 5.2b),

8025

Figure 5.1 Alternate Edge Configurations

8026

(a)

OVERLAP CORNER REGIONS

(b)

CONCENTRIC CUP CORNER REGIONS

Figure 5.2 Corner Regions - Regions I and III are identical in both analyses

two of which are identical to those in the previous studies. Therefore, only Region II is entirely new.

Figure 5.3 presents a blowup of Region II in Figure 5.2b. The same type of nodal analysis that was used during the Phase I work is being continued in Phase II. Figure 5.3 shows the location and the names of the new nodes in Region II. The pages which follow present the detailed heat balances for all Region II nodes. The notation used throughout these pages is as follows:

- q_i = heat flux, watts
- T = nodal temperature, °K
- TT = temperature of node immediately above the node in question, °K
- TR = temperature of node immediately to the right of the node in question, °K
- TB = temperature of node immediately below the node in question, °K
- TL = temperature of node immediately to the left of the node in question, °K
- TEX = temperature of exterior environment opposing node in question, °K
- r = radius to the center of node in question, cm.
- $H(T)$ = heat transfer coefficient, watts/cm² °K
- δ_f = foil thickness, cm.
- δ_o = oxide gap thickness, cm.
- k_f = foil thermal conductivity, watts/cm °K
- $\epsilon(T)$ = foil emissivity

8027

Figure 5.3 Concentric Cup Corner Nodal Network

CCTL

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta''}$$

$$\text{where } \delta'' = \frac{\delta_f + \text{VDL}}{2}$$

$$q_2 = 2\pi r \delta_f H(\text{TR})(\text{TR} - T)$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - \text{TB}}{\delta_f}$$

$$q_4 = 2\pi r \delta_f \epsilon^* \sigma (T^4 - \text{TEX}^4)$$

$$\text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{\text{TEX}}} - 1}$$

$$\frac{\delta_f k_f}{\delta''} TT + \delta_f H(\text{TR}) \text{TR} + k_f \text{TB} - \delta_f \epsilon^* \sigma (T^4 - \text{TEX}^4) = \frac{\delta_f k_f}{\delta''} T + \delta_f H(\text{TR}) T + k_f T$$

$$T = \frac{\frac{\delta_f k_f}{\delta''} TT + \delta_f H(\text{TR}) \text{TR} + k_f \text{TB} - \delta_f \epsilon^* \sigma (T^4 - \text{TEX}^4)}{\frac{\delta_f k_f}{\delta''} + \delta_f H(\text{TR}) + k_f}$$

CCL1

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*} \quad \text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f H(TR)(TR - T)$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - TB}{\delta^*}$$

$$q_4 = 2\pi r \delta_f \epsilon^* (T^4 - \text{TEX}^4) \quad \text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{\text{TEX}}} - 1}$$

$$\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_f H(TR) TR + \delta_f \epsilon^* \sigma (\text{TEX}^4 - T^4) = 2 \frac{\delta_f k_f}{\delta^*} T + \delta_f H(TR) T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_f H(TR) TR + \delta_f \epsilon^* \sigma (\text{TEX}^4 - T^4)}{2 \frac{\delta_f k_f}{\delta^*} + \delta_f H(TR)}$$

CCL2

TEX

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_o H(TR)(TR - T)$$

$$q_3 = 2\pi r \delta_f \frac{T - TB}{\delta^*}$$

$$q_4 = 2\pi r \delta_o \epsilon^* \sigma (T^4 - \text{TEX}^4)$$

$$\text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{\text{TEX}}} - 1}$$

$$\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_o H(TR) TR + \delta_o \epsilon^* \sigma (\text{TEX}^4 - T^4) = 2 \frac{\delta_f k_f}{\delta^*} T + \delta_o H(TR) T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_o H(TR) TR + \delta_o \epsilon^* \sigma (\text{TEX}^4 - T^4)}{\frac{\delta_f k_f}{\delta^*} + \delta_o H(TR)}$$

CCCV2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1$$

$$\text{HEAT OUT} = q_2 + q_3$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{T - TB}{\delta^*}$$

$$q_3 = 2\pi r \delta_o \epsilon^* \sigma (T^4 - \text{TEX}^4)$$

$$\text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{\text{TEX}}} - 1}$$

$$\frac{\delta_f k_f}{\delta^*} (TT + TB) - \delta_o \epsilon^* \sigma (T^4 - \text{TEX}^4) = 2 \frac{\delta_f k_f}{\delta^*} T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TT + TB) - \delta_o \epsilon^* \sigma (T^4 - \text{TEX}^4)}{2 \frac{\delta_f k_f}{\delta^*}}$$

CCC

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$q_3 = 2\pi r \delta_f \epsilon^* \sigma (T^4 - \text{TEX}^4)$$

$$q_4 = 2\pi r \delta_f \epsilon^* \sigma (T^4 - \text{TEX}^4)$$

$$\text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{\text{TEX}}} - 1}$$

$$\frac{k_f}{\delta^*} (TT + TR) - 2\epsilon^* \sigma (T^4 - \text{TEX}^4) = \frac{2k_f}{\delta^*} T$$

$$T = \frac{1}{2} (TT + TR) - \frac{\epsilon^* \sigma \delta^*}{k_f} (T^4 - \text{TEX}^4)$$

CCCH2

HEAT IN = HEAT OUT

HEAT IN = q_1

HEAT OUT = $q_2 + q_3$

$$q_1 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$q_3 = 2\pi r \delta_o \epsilon^* \sigma (T^4 - TEX^4)$$

$$\text{where } \epsilon^* = \frac{1}{\frac{1}{\epsilon_T} + \frac{1}{\epsilon_{TEX}} - 1}$$

$$\frac{\delta_f k_f}{\delta^*} (TR + TL) - \delta_o \epsilon^* \sigma (T^4 - TEX^4) = 2 \frac{\delta_f k_f}{\delta^*} T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TR + TL) - \delta_o \epsilon^* \sigma (T^4 - TEX^4)}{2 \frac{\delta_f k_f}{\delta^*}}$$

CBOT2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f H(TT)(TT - T)$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$q_3 = 2\pi r \delta_f H(T)(T - \text{TEX})$$

$$q_4 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$\delta_f H(TT)T + 2 \frac{\delta_f k_f}{\delta^*} + \delta_f H(T)T = \delta_f H(TT)TT + \delta_f H(T)\text{TEX} + \frac{\delta_f k_f}{\delta^*} (TR + TL)$$

$$T = \frac{\delta_f (H(TT)TT + H(T)\text{TEX}) + \frac{\delta_f k_f}{\delta^*} (TR + TL)}{\delta_f (H(TT) + H(T)) + 2 \frac{\delta_f k_f}{\delta^*}}$$

CBOT1

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_o H(TT)(TT - T) \quad \text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$q_3 = 2\pi r \delta_o H(T)(T - \text{TEX})$$

$$q_4 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$\delta_o H(TT)T + 2 \frac{\delta_f k_f}{\delta^*} + \delta_o H(T)T = \delta_o H(TT)TT + \delta_o H(T)\text{TEX} + \frac{\delta_f k_f}{\delta^*} (TR + TL)$$

$$T = \frac{\delta_o (H(TT)TT + H(T)\text{TEX}) + \frac{\delta_f k_f}{\delta^*} (TR + TL)}{\delta_o (H(TT) + H(T)) + 2 \frac{\delta_f k_f}{\delta^*}}$$

CBOTR

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f H(TT)(TT - T)$$

$$\text{where } \delta' = \frac{\delta_f + \text{HDL}}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta'}$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_4 = 2\pi r \delta_f H(T)(T - \text{TEX})$$

$$\begin{aligned} \delta_f H(TT)T + \frac{\delta_f k_f}{\delta'} T + \frac{\delta_f k_f}{\delta^*} T + \delta_f H(T)T &= \delta_f H(TT)TT + \frac{\delta_f k_f}{\delta'} TR \\ &+ \frac{\delta_f k_f}{\delta^*} TL + \delta_f H(T)TEX \end{aligned}$$

$$T = \frac{\delta_f (H(TT)TT + H(T)TEX) + \delta_f k_f \left(\frac{TR}{\delta'} + \frac{TL}{\delta^*} \right)}{\delta_f (H(TT) + H(T)) + \delta_f k_f (1/\delta' + 1/\delta^*)}$$

CCIT

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta''}$$

$$\text{where } \delta'' = \frac{\delta_f + \text{VDL}}{2}$$

$$q_2 = 2\pi r \delta_f H(\text{TR})(\text{TR} - T)$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - \text{TB}}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_4 = 2\pi r \delta_f H(T)(T - \text{TL})$$

$$\frac{\delta_f k_f}{\delta''} TT + \delta_f H(\text{TR})\text{TR} + \frac{\delta_f k_f}{\delta^*} \text{TB} + \delta_f H(T)\text{TL} = \frac{\delta_f k_f}{\delta''} T + \delta_f H(\text{TR})T$$

$$+ \frac{\delta_f k_f}{\delta^*} T + \delta_f H(T)T$$

$$T = \frac{\frac{\delta_f k_f}{\delta''} TT + \delta_f H(\text{TR})\text{TR} + \frac{\delta_f k_f}{\delta^*} \text{TB} + \delta_f H(T)\text{TL}}{\delta_f k_f \left(\frac{1}{\delta''} + \frac{1}{\delta^*} \right) + \delta_f (H(\text{TR}) + H(T))}$$

CCVI2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_o H(TR)(TR - T)$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - TB}{\delta^*}$$

$$q_4 = 2\pi r \delta_o H(T)(T - TL)$$

$$\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_o H(TR) TR + \delta_o H(T) TL = 2 \frac{\delta_f k_f}{\delta^*} T + \delta_o H(TR) T + \delta_o H(T) T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_o H(TR) TR + \delta_o H(T) TL}{2 \frac{\delta_f k_f}{\delta^*} + \delta_o (H(TR) + H(T))}$$

CCVI1

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f H(TR)(TR - T)$$

$$q_3 = 2\pi r \delta_f k_f \frac{T - TB}{\delta^*}$$

$$q_4 = 2\pi r \delta_f H(T)(T - TL)$$

$$\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_f H(TR) TR + \delta_f H(T) TL = 2 \frac{\delta_f k_f}{\delta^*} T + \delta_f H(TR) T + \delta_f H(T) T$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TT + TB) + \delta_f H(TR) TR + \delta_f H(T) TL}{2 \frac{\delta_f k_f}{\delta^*} + \delta_f (H(TR) + H(T))}$$

CCICV2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1$$

$$\text{HEAT OUT} = q_2 + q_3$$

$$q_1 = 2\pi r \delta_f k_f \frac{T - T_L}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_o k_o \frac{T - T_B}{\delta^*}$$

$$q_3 = 2\pi r \delta_o H(T) (T - T_L)$$

$$2 \frac{\delta_f k_f}{\delta^*} T + \delta_o H(T) T = \frac{\delta_f k_f}{\delta^*} (T - T_B) + \delta_o H(T) T_L$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (T - T_B) + \delta_o H(T) T_L}{2 \frac{\delta_f k_f}{\delta^*} + \delta_o H(T)}$$

CCIC

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$q_3 = 2\pi r \delta_f H(T) (T - TL)$$

$$q_4 = 2\pi r \delta_f H(T) (T - TB)$$

$$2 \frac{k_f}{\delta^*} T + 2 H(T) T = \frac{k_f}{\delta^*} (TT + TR) + H(T) (TL + TB)$$

$$T = \frac{\frac{k_f}{\delta^*} (TT + TR) + H(T) (TL + TB)}{2 \frac{k_f}{\delta^*} + 2 H(T)}$$

CCICH2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1$$

$$\text{HEAT OUT} = q_2 + q_3$$

$$q_1 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$q_3 = 2\pi r \delta_o H(T) (T - TB)$$

$$2 \frac{\delta_f k_f}{\delta^*} T + \delta_o H(T) T = \frac{\delta_f k_f}{\delta^*} (TR + TL) + \delta_o H(T) TB$$

$$T = \frac{\frac{\delta_f k_f}{\delta^*} (TR + TL) + \delta_o H(T) TB}{2 \frac{\delta_f k_f}{\delta^*} + \delta_o H(T)}$$

C2

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f H(TT)(TT - T)$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_3 = 2\pi r \delta_f H(T)(T - TB)$$

$$q_4 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$\delta_f H(TT)T + \delta_f H(T)T + 2 \frac{\delta_f k_f}{\delta^*} = \delta_f (H(TT)TT + H(T)TB) + \frac{\delta_f k_f}{\delta^*} (TR + TL)$$

$$T = \frac{\delta_f (H(TT)TT + H(T)TB) + \frac{\delta_f k_f}{\delta^*} (TR + TL)}{\delta_f (H(TT) + H(T)) + 2 \frac{\delta_f k_f}{\delta^*}}$$

C1

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_o H(TT) (TT - T)$$

$$\text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta^*}$$

$$q_3 = 2\pi r \delta_o H(T) (T - TB)$$

$$q_4 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*}$$

$$\delta_o H(TT) T + \delta_o H(T) T + 2 \frac{\delta_f k_f}{\delta^*} = \delta_o (H(TT) TT + H(T) TB) + \frac{\delta_f k_f}{\delta^*} (TR + TL)$$

$$T = \frac{\delta_o (H(TT) TT + H(T) TB) + \frac{\delta_f k_f}{\delta^*} (TR + TL)}{\delta_o (H(TT) + H(T)) + 2 \frac{\delta_f k_f}{\delta^*}}$$

CR

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f H(TT) (TT - T) \quad \text{where } \delta' = \frac{\delta_f + \text{HDL}}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta'}$$

$$q_3 = 2\pi r \delta_f H(T) (T - TB)$$

$$q_4 = 2\pi r \delta_f k_f \frac{T - TL}{\delta^*} \quad \text{where } \delta^* = \frac{\delta_f + \delta_o}{2}$$

$$\delta_f H(TT) T + \delta_f H(T) T + \frac{\delta_f k_f}{\delta'} T + \frac{\delta_f k_f}{\delta^*} T = \delta_f H(TT) TT + \delta_f H(T) TB + \frac{\delta_f k_f}{\delta'} TR + \frac{\delta_f k_f}{\delta^*} TL$$

$$T = \frac{\delta_f (H(TT) TT + H(T) TB) + \delta_f k_f \left(\frac{TR}{\delta'} + \frac{TL}{\delta^*} \right)}{\delta_f (H(TT) + H(T)) + \delta_f k_f \left(\frac{1}{\delta'} + \frac{1}{\delta^*} \right)}$$

CCTR

HEAT IN = HEAT OUT

$$\text{HEAT IN} = q_1 + q_2$$

$$\text{HEAT OUT} = q_3 + q_4$$

$$q_1 = 2\pi r \delta_f k_f \frac{TT - T}{\delta''}$$

$$\text{where } \delta' = \frac{\delta_f + \text{VDL}}{2}$$

$$q_2 = 2\pi r \delta_f k_f \frac{TR - T}{\delta'}$$

$$q_3 = 2\pi r \delta_f H(T) (T - TB)$$

$$q_4 = 2\pi r \delta_f H(T) (T - TL) \quad \text{where } \delta' = \frac{\delta_f + \text{HDL}}{2}$$

$$\frac{\delta_f k_f}{\delta''} TT + \frac{\delta_f k_f}{\delta'} TR + \delta_f H(T) (TB + TL) = \frac{\delta_f k_f}{\delta''} T + \frac{\delta_f k_f}{\delta'} T + 2\delta_f H(T)$$

$$T = \frac{\frac{\delta_f k_f}{\delta''} TT + \frac{\delta_f k_f}{\delta'} TR + \delta_f H(T) (TB + TC)}{\frac{\delta_f k_f}{\delta''} + \frac{\delta_f k_f}{\delta'} + 2\delta_f H(T)}$$

The executive program which iteratively invokes each local subroutine in the correct sequence has been modified to analyze a concentric cup corner. During the next quarter the debugging of the revamped edge loss computer analysis will begin.

Subtask 5.2 Corner Configuration Fabrication and Testing

FOIL CUP FABRICATION - In the past a concentric cup insulation corner was always considerably unattainable because of the difficulty associated with fabricating a large number of foil cups that differ only slightly in size from one another. However, because such a configuration represents the ultimate corner, a concerted effort was initiated to deep draw thin foil cups. Circular foil blanks were placed in a punch and die combination and, with varying degrees of success and failure, foil cups were drawn.

At the outset copper foil was used because of its ductility. If copper foil cups could not be drawn, then certainly neither could nickel or molybdenum or tungsten cups. As one might expect, the thinner foil blanks are more difficult to draw into cups. A less obvious result, however, is that it is more difficult to draw a small diameter cup than a large diameter cup.

After a great deal of experimentation it became obvious that the key factor in deep drawing thin foil cups is to achieve just the right amount of slipping as the center of the foil blank is pushed into the die by the punch. A typical punch and die is diagrammed in Figure 5.4. Approximately the outermost quarter inch of the circular foil blank is squeezed between a spring-loaded ring and the shoulders of the die. If this band is squeezed so tightly that the foil remains absolutely stationary

Figure 5.4 A typical cup-drawing punch and die combination

during the draw, brittle fracture will probably result. Too small a region of the foil blank yields and the sidewall thickness of the resulting cup approaches zero, or at least is full of cracks. If, on the other hand, the outer perimeter of the circular foil blank is held too loosely, excessive foil material is pulled into the cup sidewalls and the walls appear crinkled. The proper balance of slipping and holding is more difficult to achieve with the less ductile and thinner foil materials.

Another trick which eventually led to an acceptable drawn foil cup is to sandwich the original foil material between thicker blanks of a more ductile material. Specifically, the best molybdenum cups were obtained when 1 mil molybdenum foil was placed between two 15 mil copper blanks. Strangely, 10 mil copper was too thin and, when a smaller diameter cup was sought, no thickness of copper was satisfactory and nickel had to be substituted. Cup drawing definitely appears to be an art rather than a science.

Figures 5.5 through 5.15 present photographs of various successes and failures during the cup drawing experiments. The early attempts were very discouraging but eventually thin copper and nickel cups, first at 5 mils and then at 1 mil, were successfully drawn. The next target, and the first that would be useful in a 1700° C insulation system, was a 1 mil molybdenum cup. Initially, 5 mil molybdenum cups were successfully drawn and shortly thereafter 3 mil cups were a reality. The 1 mil molybdenum cup was considerably more difficult to draw. A great deal of trouble was encountered optimizing the slippage of the foil blank between the spring-loaded ring and the shoulders of the die. It wasn't until the 15 mil copper sheet was placed on either side of the 1 mil molybdenum that a perfect cup was obtained (Figure 5.12).

Material: Copper

Thickness: .001"

Comments: First attempt to draw a foil cup. No attempt was made to keep outer foil circumference from slipping into the side wall area.

(a)

1 mil Cu

Material: Nickel

Thickness: .001"

Comments: No attempt was made to hold the outer circumference of the foil blank. Note the numerous crinkles in the side walls.

(b)

1 mil Ni

Figure 5.5 Deep drawn cup attempts

(a)
5 mil Ni

(b)
5 mil Mo

Figure 5.6 Deep drawn cup attempts

Material: Molybdenum
Thickness: .003"
Comments: Outer circumference of blanks squeezed by spring loaded ring. Load not sufficient. Resulting slipping produced wrinkles.

(a)
3 mil Mo

Material: Molybdenum
Thickness: .003"
Comments: First draw with new holding technique. A ring which squeezes the foil blanks was bolted to the die. Substantially better holding, less wrinkles. Shallow draw.

(b)
3 mil Mo

Figure 5.7 Deep drawn cup attempts

(a)
3 mil Mo

(b)
3 mil Mo

Figure 5.8 Deep drawn cup attempts

(a)

1 mil Mo

(b)

1 mil Mo

Figure 5.9 Deep drawn cup attempts

Material: Molybdenum
Thickness: .001"
Comments: Allowed a limited amount of slipping. Annealed blank at 1300°C in vacuum. This appeared to help. The draw, however, was shallow.

(a)
1 mil Mo

Material: Molybdenum
Thickness: .001"
Comments: A better combination of circumferential slipping and holding. No annealing. Draw still shallow.

(b)
1 mil Mo

Figure 5.10 Deep drawn cup attempts

(a)
1 mil Mo

(b)
1 mil Mo

Figure 5.11 Deep drawn cup attempts

(a)

A perfect 1 mil Mo cup

(b)

The adjacent Cu blank after drawing

Figure 5.12 Deep drawn cup attempts

(a)
 $\frac{1}{2}$ mil Mo

(b)
 $\frac{1}{2}$ mil Mo

Figure 5.13 Deep drawn cup attempts

(a)
 $\frac{1}{2}$ mil Mo

(b)
 $\frac{1}{2}$ mil Mo

Figure 5.14 Deep drawn cup attempts

Material: Molybdenum
Thickness: .0005"
Comments: Brittle fracture.
Success will apparently depend
on optimization of holding and
slipping.

(a)

$\frac{1}{2}$ mil Mo

Material: Molybdenum
Thickness: .0005"
Comments: Seven 1/2 mil
layers backed up by a .003"
layer. First attempt to draw slit
foils. Voids between slits
easily reduced by proper blank
shape. Stress-relieving will
minimize the springiness.

(b)

$\frac{1}{2}$ mil Mo

Figure 5.15 Deep drawn cup attempts

The next goal was a 1/2 mil molybdenum cup. The slippage optimization proved exceptionally difficult and a satisfactory cup was obtained only by chemically etching a 1 mil cup. The etching procedure produced perfect molybdenum cups with wall thicknesses as small as 1/5 of a mil (.0002 in).

Another interesting configuration is that appearing in Figure 5.15. Several blanks with slits cut radially inward were simultaneously drawn into a cup shape. The slits vastly facilitated the drawing procedure.

The next goal was to simultaneously draw more than one 1 mil molybdenum cup. This surprisingly, proved to be a very difficult task and no more than two cups could be simultaneously drawn in a given die. Unfortunately, this is a very crippling result because a prohibitive number of punch and die combinations would presently be required to fabricate a reasonable multi-layer insulation corner. At present the only type of cup that can be fabricated at a reasonable cost is a slit cup corner.

No attempt has yet been made to deep draw tungsten or tungsten - 25% rhenium cups. In view of the brittle nature of tungsten, and the difficulties encountered drawing molybdenum, the probability of success appears relatively low.

EDGE HEAT LOSS TESTS - The edge heat loss test assembly was used to test the performance of two Ta-ThO₂ insulation corner assemblies. A 40-layer slit cup corner was compared with a 40-layer overlap corner of the type tested throughout Phase I. Figure 5.16 presents schematic representations of the corners tested. Figure 5.17a presents

8037

Figure 5.16 Alternate Corner Configurations

Figure 5.17 Edge Heat Loss Data

the overlap corner test results. Three separate test runs were made on three separate days; the curve in Figure 5.17a represents the average of the three tests and indicates the current level of reproducibility in edge loss testing. Two separate runs define the slit cup corner test results in Figure 5.17b. A comparison of the two curves indicates that the heat flux through the overlap corner is approximately 1.4 watts less at a given source temperature than that through the slit cup corner. On a watts per cm. of edge basis the overlap corner was approximately 15% better. This result is contrary to the pre-test expectations and may indicate that there are a few tricks associated with fabricating a slit cup corner. The slit cups are quite springy and were therefore packed rather tightly together in the final corner assembly. It is entirely possible that, after spraying, the cups were packed too tightly and, as a result, the conduction losses were unnecessarily high.

Motivated by the unexpected results reported in the previous paragraph, the edge heat loss test apparatus was scrutinized closely in an effort to pinpoint any extraneous heat paths to the collector and the calorimeter. Figure 5.18 presents a drawing of the essential features of the test fixture with the forty layer overlap corner installed within it. Above the heater block, each planar 20-layer insulation subassembly rests on a tantalum foil platform 5 mils thick. Each 20-layer cylindrical insulation subassembly is capped by an outer 3 mil foil cylinder, and the entire corner assembly, sides and bottom included, is capped by an additional 3 mil cup. In retrospect it appears that heat was conducted radially outward along the 5 mil foils to the large 3 mil cup, and then down the 3 mil cup to the calorimeter. Both the 5 mil foils and the 3 mil cup were removed. In addition the 3 mil cylinder

8041

Figure 5.18 Overlap corner Insulation Test Configuration

which capped the outer 20-layer cylindrical insulation subassembly was removed and replaced with a 1 mil cylinder. A final refinement involved dusting the collector top with a light coating of yttria. This prevents the insulation corner from resting directly on the collector top. In the planar test assembly the coldest foil radiates across a small gap to the collector, and since the planar data is used to reduce the edge loss data, the gap caused by the intervening yttria recreates the planar configuration more exactly.

These efforts were justly rewarded. Figure 5.19 plots the total heat flowing through the calorimeter as a function of the source temperature for the same 40-layer Ta-ThO₂ overlap corner configuration. The results represent a striking improvement. Using 40-layer Ta-ThO₂ cylindrical and planar data obtained in Phase I, that portion of the heat flow through the calorimeter, q_1 , directly attributable to the corner can be calculated. At a source temperature of 1185°C, q_1 is 0.0 watts; at a source temperature of 1695°C, q_1 is 2.35 watts (or .29 watts/cm of edge). The first result indicates that the corner contributed nothing to the calorimeter heat flux. This, of course, is highly unlikely, but it is safe to conclude that the overlap corner configuration is better than originally thought. Future Phase II work will search for similar improvements in the planar and cylindrical test configurations. When new planar and cylindrical data is obtained the data in Figure 5.18 can be reanalyzed. For the present, the unreduced heat loss data can be used to compare competing corner configurations.

The slit cup corner configuration also will be tested using the improved experimental configuration. Particular attention will be paid to the corner assembly procedure.

8038

40 LAYER OVERLAP CORNER
Ta-ThO₂ INSULATION

Figure 5.19 Edge Heat Loss Test Data -
Improved Experimental
Configuration

2.5 Task 6. Lower Temperature Applications

The first step in Task 6 is to test the compatibility of the prospective lower temperature insulation material combinations. These tests will be analogous to those in Phase I using the higher temperature materials. The bonding characteristics of each insulation material combination will be evaluated at the following temperatures:

<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

Samples of each insulation will be examined after 100, 500, and 1000 hours at the individual test temperatures.

As the preceding table shows, material compatibility tests will be conducted at six temperatures. The two test fixtures used throughout Phase I will be used again for the 1000 and 1200°C tests. The tests at the remaining four temperatures will be conducted in four new constant temperature ovens currently being fabricated. Figure 6.1 presents a drawing of one of the ovens. The insulation packets that will be placed in the new ovens will be the same size as those in the older fixtures. In addition, the packets will be held in position by identical nut and bolt arrangements (see Figure 6.1).

2.6 Task 7. Life Tests

No life test work was scheduled for the first quarter of Phase II.

8042

Figure 6.1 Material Compatibility Constant Temperature Oven

III. PLANNED SECOND QUARTER WORK

Task 2. Spray Coating

Using the automated foil spraying apparatus, the oxide layer thickness will be determined as a function of the original particle size and the number of spray passes. Various combinations of particle size and oxide coating thickness will then be tested in the planar heat loss test apparatus, with and without an applied load.

Task 3. W and W(25)Re Foils

Additional foil will be purchased, and a decision will be reached about whether to etch the 1 mil tungsten down to 1/2 mil in-house or at GE-NMPO.

Task 4. Cylindrical Insulation

The design of the non-uniform loading cylindrical heat loss test apparatus will be finalized. The fabrication and testing of this apparatus, however, will be deferred until FY1969.

Task 5. Edge Heat Losses

The computer analysis of a concentric cup corner will continue. In addition, the slit cup corner tests will be rerun and a new tapered corner will be fabricated and tested.

Task 6. Lower Temperature Applications

The material compatibility tests will be initiated, completed, and analyzed.

Task 7. Life Tests

No work is planned.