4 cys

RECEIVED BY DTIC

1969
MASTER

ALO 3632-41
UC-33, Propulsion Systems
and Energy Conversion

AEC RESEARCH AND
DEVELOPMENT REPORT

UNION CARBIDE CORPORATION

LINDE DIVISION

QUARTERLY PROGRESS REPORT OF RESEARCH AND DEVELOPMENT
IN A THERMAL INSULATION STUDY

March - May, 1969

There is no objection from the Patent
Group of view to the publication or
dissemination of this document.
Patent Group (Brookhaven)
By.....
8/4 1969

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.

REPRODUCTION OF THIS DOCUMENT IS UNLIMITED

104

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.

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 with such contractor.

ALO 3632-41
UC-33, Propulsion Systems
and Energy Conversion

UNION CARBIDE CORPORATION
LINDE DIVISION

QUARTERLY PROGRESS REPORT
IN A THERMAL INSULATION STUDY

March - May, 1969

W. E. Grunert
H. Morihara

Contract No. AT(30-1)-3632
U. S. ATOMIC ENERGY COMMISSION

ALO 3632-41
UC-33, Propulsion Systems
and Energy Conversion

DISTRIBUTION

B. W. Colston, Director
Space and Special Programs Div.
Sandia Area Office
U.S. Atomic Energy Commission
Albuquerque Operations Office
Albuquerque, New Mexico 87115 (2 cys)

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

R. T. Carpenter, Chief
Isotope Power Branch
Division of Space Nuclear Systems
U.S. Atomic Energy Commission
Washington, D. C. 20545
Attn: P. A. O'Riordan (1 cy)

S. J. Seiken
Division of Reactor Development
and Technology
U.S. Atomic Energy Commission
Washington, D. C. 20545 (1 cy)

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 cy)

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

Isotopes, A Teledyne Company
Nuclear Systems Division
P. O. Box 4937
Middle River, Maryland 21220
Attn: Document Custodian (1 cy)

E. Ray
General Electric Company
Missile and Space Development
Valley Forge Space Technology Center
Post Office Box 8555
Philadelphia, Pennsylvania 19101 (2 cys)

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

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

Fred Hittman, General Manager
Hittman Associates, Inc.
4715 East Wabash Avenue
Baltimore, Maryland 21215 (1 cy)

Joseph Kenney, Program Manager
Compact Thermoelectric Converter
Westinghouse Electric Corporation
P. O. Box 10864
Pittsburgh, Pennsylvania 15213 (1 cy)

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

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

ALO 3632-41
UC-33, Propulsion Systems
and Energy Conversion

DISTRIBUTION (Cont'd.)

Mr. Albert Wallis
Aeropropulsion Laboratory
Wright-Patterson Air Force Base
Dayton, Ohio (1 cy)

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

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

Harold Garber
NUMEC
609 North Warren Avenue
Apollo, Pennsylvania 15613 (1 cy)

NASA/Marshall Space Flight Center
Huntsville, Alabama 35812
Attn: Mr. Richard Poorman (1 cy)

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

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

U.S. Atomic Energy Commission
Division of Technical Information
Extension
Post Office Box 62
Oak Ridge, Tennessee 37831
and 1 reproducible copy
plus Form 426 (3 cys)

NASA/Lewis Research Center
Cleveland, Ohio
Attn: Mr. H. Cameron (1 cy)

NASA/Lewis Research Center
Cleveland, Ohio
Attn: Mr. R. Davies (1 cy)

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	i
1.0 SUMMARY	
1.1 Thermal Conductivity Testing - Cylindrical Tests	1
1.2 Thermal Conductivity Testing - Flat Plate Tests	1
1.3 Insulation Interfaces - Penetration Effects	3
2.0 TECHNICAL REPORT	
2.1 Thermal Conductivity Testing - Cylindrical Tests	4
2.1.1 Nickel Opacified Quartz Multi-layer Insulation	7
2.1.2 1800°F Composite Multi-foil Insulation	10
2.1.3 Long-Term Performance Deterioration - Potential Causes and Proposed Investigation	13
2.2 Thermal Conductivity Testing - Flat Plate Tests	19
2.2.1 Aluminum-Glass Multi-layer Insulations - Retest	19
2.2.1.1 Aluminum Multi-foil Insulations	21
2.2.1.2 Glass Fibrous Multi-layer Insulations	25
2.2.1.3 Accurate Curve Fitting of Total Thermal Conductivity Data	34
2.2.1.4 Observations on Thermal Behavior of Aluminum - Glass Multi-layer Systems	39
2.2.2 Molybdenum-Zirconia Multi-foil Insulation - 2500°F Testing	43
2.2.3 Data Analysis Program	44
2.3 Penetration Effects Testing - Interim Status	46
3.0 ACTIVITIES DURING NEXT REPORTING PERIOD	52

ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
2.1.1	Thermal Conductivity Data - Cylindrical Testing	5
2.1.2	Long-term Performance - Nickel Opacified Quartz Insulation	8
2.1.3	Performance Deterioration - Nickel Opacified Quartz Insulation	9
2.1.4	Thermal Performance of 1800°F Composite Multi-foil Insulation System	11
2.1.5	Composite Multi-layer Insulation Performance	12
2.1.6	Long-term Performance - 1800°F Composite Insulation	14
2.1.7	Long-term Stability Evaluation - 1800°F Composite Insulation	17
2.2.1	Apparent Thermal Conductivity - Aluminum-106 Glass Multi-layer Insulation	22
2.2.2	Apparent Thermal Conductivity - Aluminum-Aluminum Opacified Multi-layer Insulation	23
2.2.3	Apparent Thermal Conductivity - 106 Glass Multi-layer Insulation	27
2.2.4	Apparent Thermal Conductivity - Aluminum Opacified Glass Multi-layer Insulation	28
2.2.5	Solid Conductivity Coefficients - Glass Fibrous Multi-layer Insulations	31
2.2.6	Effective Backscattering Cross-section - Glass Fibrous Multi-layer Insulations	32
2.2.7- 2.2.10	Empirical Fit of Thermal Conductivity	35-38
2.2.11	Effective Backscattering Cross-sections - Glass and Quartz Fibrous Multi-layer Insulations	40

ILLUSTRATIONS (Cont'd.)

<u>Figure</u>		<u>Page</u>
2.3.1	Penetration-Insulation Test Sample Configuration	48
2.3.2	Effective Thermal Conductivity - Penetration- Insulation Systems	49
2.3.3	Modified Sample Design - Penetration Effects Tests	51

TABLES

<u>Number</u>		<u>Page</u>
2.1.1	Current Cylindrical Tests - Insulation System Description	6
2.2.1	Aluminum Multi-layer Systems	20
2.2.2	Empirical Thermal Conductivity Parameters — Multi-foil Insulations	26
2.2.3	Empirical Thermal Conductivity Parameters — Fibrous Multi-layer Insulations	30

Linde Division personnel contributing to the AEC Contract AT(30-1)-3632 technical effort for the reporting period are: Messrs: W. E. Grunert, H. Morihara, F. Notaro, E. S. Kordyban, K. C. Kather, J. J. Parwulski, and P. N. Massing.

QUARTERLY PROGRESS REPORT OF RESEARCH AND DEVELOPMENT
IN A THERMAL INSULATION STUDY

Prepared By

W. E. Grunert
H. Morihara

ABSTRACT

Updated results of long-term stability tests on the 1800°F composite and nickel opacified quartz multi-layer insulation systems are presented. Performance deteriorations, ranging from 6% on the 1800°F composite (1000-hours operation) to 16% on the nickel opacified system (6000-hours operation), have been noted. Potential causes and subsequent investigation of this long-term deterioration are discussed.

The retest program on aluminum-glass multi-foil and glass fibrous multi-layer insulations is complete. Apparent thermal conductivity data (K_a) are presented at various temperatures and compressive loads. Thermal behavior of this class of multi-layer insulations is discussed. The 2500°F testing of the molybdenum-zirconia (Mo-ZrO_2) multi-foil insulation will be conducted in the next quarter.

The flat plate thermal conductivity test program is nearing completion. A final review, re-evaluation and summary of the K_a -data for the various multi-layer systems is planned.

The current status of the penetration effects test program is discussed. Testing difficulties have been traced to excessive interface resistance between the lead telluride penetrations and system thermal boundaries. Design changes to alleviate this problem are described. Additional testing with the modified samples is planned for the next quarter.

1.0 SUMMARY

1.1 Thermal Conductivity Testing - Cylindrical Tests

Long-term thermal performance stability tests continue on two multi-layer insulations --- the nickel opacified quartz multi-layer and 1800°F composite multi-foil systems. Results to date are summarized below:

<u>System</u>	<u>Elapsed Time Hrs.</u>	<u>T_H °F</u>	<u>T_C °F</u>	<u>K_a 10⁻³ Btu/hr-ft-°F</u>	<u>K_a Increase %</u>
Nickel Opacified	0	1485	577	7.6	-
Quartz	6000	1470	602	8.8	16
n = 65 ℓ /inch					
1800°F Composite	0	1790	430	1.95	-
n = 110 ℓ /inch	1000	1782	440	2.06	6

All systems are tested in a vacuum environment ($p < .5 \times 10^{-3}$ mm Hg).

The performance deterioration (as indicated by the percent increase in K_a) for the two multi-layer systems is felt to be due to some material degradation in the high temperature, vacuum exposure. A preliminary study of the 1800°F composite system, and particularly the molybdenum and Astroquartz materials, is being undertaken to define any metallurgical and chemical compatibility and/or stability problems which may relate to the observed deterioration. It is hoped that this study will provide additional insight into the nature and magnitude of the possible degradation mechanisms.

In addition to the study, close monitoring of the test insulation systems will be continued in order to determine any observable trends in insulation deterioration. Extension of the long-term test period to 10,000 hours duration on each system has been proposed.

1.2 Thermal Conductivity Testing - Flat Plate Tests

The flat plate thermal conductivity re-test program on aluminum and glass fiber multi-layer insulations has been completed. Pertinent K_a data at 800°F temperature and limiting compressive load conditions are tabulated on the following page.

System	T_h °F	T_c °F	Compressive Load psi	Layer Density lb/inch	K_a 10 ⁻³ Btu/hr-ft-°F
Al-106 Glass	800	100	.04	142	0.14
Multi-foil	800	100	15.0	345	1.00
Al-Al Opacified	800	100	.04	204	0.09
Glass Multi-foil	800	100	15.0	449	2.30
106 Glass Fibrous	800	100	.04	166	4.40
Multi-layer	800	100	15.0	444	3.00
Al Opacified Glass	800	100	.04	259	0.56
Fibrous Multi-Layer	800	100	15.0	542	2.00

All data were measured on multi-layer systems operating in a vacuum environment ($p < 0.5 \times 10^{-3}$ mm Hg). The new K_a -values on the above four multi-layer insulations supersede the original data,* which were rather limited and in error at the high compressive loads.

Based on the tabulated K_a -data, it is seen that:

(1) The aluminum flake opacified glass paper is considerably more effective in attenuating radiation heat than the plain 106 glass system, both operating at low load. At high load (15 psi), the effectiveness of the Al opacified system is reduced due to its higher solid conductance.

(2) Performance of the two multi-foil systems is comparable at low load. The indicated difference in K_a -values at .04 psi is due mainly to the layer density difference. At high (15 psi) load, the greater solid conductance contribution of the opacified spacer results in a higher K_a value for the Al-Al opacified glass insulation.

Thermal conductivity tests on the molybdenum-zirconia (Mo-ZrO₂) multi-foil insulation system will be conducted during the next quarter. Testing will be in vacuum, at hot-side temperatures ranging to 2500°F. The molybdenum foil (.0005" thick) and zirconia cloth (.005" thick) materials have been received. Necessary modifications to the flat plate test equipment have been made to permit 2500°F operation.

* Original K_a -data previously presented in Quarterly Progress Report ALO-3632-33, June-August, 1969.

A thermal conductivity data review and analysis program will be organized and implemented during the next quarter. The program will provide an overall summary of thermal performance data on the 14 multi-layer insulations evaluated in Phase II. Accuracy limits for the flat plate testing equipment and procedures, and effects of these limits on resulting K_a -data, will be discussed.

1.3 Penetration Effects Testing

Initial tests have been completed on a penetration-insulation system, operating in vacuum at temperatures to 900°F, with components defined as follows:

Insulation - Al-Al opacified glass multi-foil insulation, installed at $n = 200$ μ /inch.

Penetrations - Lead telluride (p or n type) elements; 0.1" diameter by ~ 0.2 " long, installed on 1" centers.

Measured effective thermal conductivity values for zero gap and filled gap penetration-insulation configurations were lower, by a factor of 5-10, than corresponding predicted values based on lead telluride material data. Measurement problems were traced to interface resistance between the lead telluride elements and thermal boundaries. Steps have been taken to minimize this resistance (i.e., increased contact pressure, indium contact sheets, etc.). Tests are being resumed.

2.0 TECHNICAL REPORT

2.1 Thermal Conductivity Testing - Cylindrical Tests

During this reporting period, long-term thermal performance tests have been in progress on two insulation systems -- nickel opacified quartz multi-layer and the 1800°F composite multi-foil systems. The standard cylindrical test techniques, with a spirally-wrapped multi-layer sample, are in use. These techniques have been previously described (Reference Quarterly Progress Report ALO 3632-33).

The objectives to be achieved through these tests are as follows:

- (1) Obtain basic thermal conductivity data.
- (2) Obtain long-term stability data through extended duration testing.
- (3) Obtain a preliminary assessment of the effect on thermal conductivity in vacuum due to exposure of the hot insulation system to air.

Objective 1 has been achieved for all systems tested within this program (Reference Figure 2.1.1). The necessary testing to reach the second objective is being carried out on the last two above-mentioned multi-layer insulations. Objective 3 has been directed mainly to the multi-foil systems, and is scheduled to follow the current long-term stability testing in vacuum.

Brief physical descriptions of the two insulations currently on test are presented in Table 2.1.1. The indicated performance parameters are obtained by an empirical fit of the thermal conductivity data of Figure 2.1.1 (K_a as a function of temperature (T) at the specified low-load layer density). The empirical thermal conductivity equation, used to treat all cylindrical test data, is a polynomial of the form:

$$K(T) = aT + bT^3 \quad (2.1)$$

Qualifications and limitations of this polynomial fit have been previously discussed (Quarterly Progress Report ALO 3632-33).

The nickel opacified quartz multi-layer and composite multi-foil insulation systems have been on continuous long-term test for periods of 6000 hours and 1000 hours, respectively. Performance deteriorations of the order of 6-16% have been experienced on these systems. The long-term performance histories are described and discussed in the ensuing sections.

FIGURE 2.1.1 THERMAL CONDUCTIVITY DATA - CYLINDRICAL TESTING

THERMAL CONDUCTIVITY, k_a , BTU/HR FT R° X 10⁻³ BETWEEN T_h AND AMBIENT

- 1 ALUMINUM FOIL - ALUMINUM OPACIFIED GLASS FIBER PAPER, 150 LAYERS/INCH
- 2 ALUMINUM FOIL - GLASS FIBER PAPER, 120 LAYERS/INCH
- 3 COPPER FOIL - QUARTZ FIBER PAPER, 83 LAYERS/INCH
- 4 COPPER FOIL - COPPER OPACIFIED QUARTZ FIBER PAPER, 70 LAYERS/INCH
- 5 NICKEL FOIL - QUARTZ FIBER PAPER, 96 LAYERS/INCH
- 6 NICKEL FOIL - ASTROQUARTZ CLOTH, 120 LAYERS/INCH
- 7 NICKEL FOIL - NICKEL OPACIFIED QUARTZ PAPER, 56 LAYERS/INCH
- 8 COMPOSITE SYSTEM, 96 LAYERS/INCH (Ni - Cu - Al)
- 9 ALUMINUM OPACIFIED GLASS FIBER PAPER, 74 LAYERS/INCH
- 10 COPPER OPACIFIED QUARTZ FIBER PAPER, 30 LAYERS/INCH
- 11 NICKEL OPACIFIED QUARTZ FIBER PAPER, 65 LAYERS/INCH
20. 12 COMPOSITE SYSTEM, 110 LAYERS/INCH (Mo - Ni - Cu - Al)

$T_{cold} = 70^\circ$

TABLE 2.1.1

CURRENT CYLINDRICAL TESTS - INSULATION SYSTEM DESCRIPTION

<u>Insulation System</u>	<u>Layer Density (nominal)</u> lb/inch	<u>Total Number of Layers</u>	<u>Boundary Temperatures</u> °F	<u>Performance Parameters</u>		<u>Effective ϵ</u>
				<u>$a/2 \times 10^7$</u> Btu/hr-ft-°R ²	<u>$b/4 \times 10^{13}$</u> Btu/hr-ft-°R ⁴	
Nickel opacified multi-layer insulation No foils Ni opacified glass paper (.010" thick; 6.2 gms/ft ²)	65	35	1485 600	3.2	4.6	-
Composite multi-layer insulation Molybdenum - Quartz cloth Nickel - Quartz paper Copper - Quartz paper Aluminum - Al opacified paper	110	55 30 14 8 3	1800 500	- 0.7	1.13	0.17

2.1.1 Nickel Opacified Quartz Multi-layer Insulation

Apparent thermal conductivities of the nickel opacified quartz multi-layer insulation, measured at time zero and after 6000 hours elapsed operating time, are tabulated below:

<u>Elapsed Time</u> Hrs.	<u>T_{Hot}</u> °F	<u>T_{Cold}</u> °F	<u>Vacuum</u> 10 ⁻³ mm Hg	<u>Apparent Thermal Conductivity</u> 10 ⁻³ Btu/hr-ft-°F
0	1485	577	< 0.5	7.6
6000*	1470	602	< 0.5	8.8

* 8000-hour total test time is scheduled.

The data above indicate a performance deterioration of ~ 16% over the 6000 hour operating period. This decrease in insulation efficiency is further substantiated by the increasing cold-side temperature, implying a higher heat flux through the insulation system.

Performance history is described in more detail in Figure 2.1.2. Apparent thermal conductivity and hot-side temperature histories are plotted versus elapsed time for the entire 6000-hour period. It is evident from the data that the increase in K_a , although moderate, has thus far been approximately linear with time. Correspondingly, the hot-side temperature associated with the constant input heat flux has been decreasing linearly. Scatter in the measured data is due to the small ($\pm 1-2\%$) error associated with measurement and readability of heat flux and temperatures.

The abrupt jump in K_a value at the 6000 hour point resulted from an imposed increase in heat flux, in an attempt to re-establish the desired hot-side temperature of 1485°F. Up to 6000 hours testing, the hot-side temperature had decreased steadily for the constant heat input. At 6000 hours, the deviation from the specified 1485°F was 40-50°, and a heat flux adjustment was made to increase hot-side temperature. With this higher flux, the apparent thermal conductivity showed a corresponding sharp increase. In subsequent testing, the heat input will be adjusted at frequent intervals to maintain hot-side temperature near the desired 1485°F level.

Figure 2.1.3 gives percent performance deterioration versus elapsed time. The unbroken line represents deterioration as a function of time and decreasing temperature. It is estimated that this deterioration would correspond to a mean hot-side temperature of about 1460°F.

FIGURE 2.1.2 LONG-TERM PERFORMANCE - NICKEL OPACIFIED QUARTZ INSULATION

FIGURE 2.1.3

Because of the lower temperature, this plot is judged to be somewhat conservative. Based on the 6000-hour point, an estimated deterioration profile for a constant hot-side temperature of 1485°F is indicated by the broken line.

2.1.2 1800°F Composite Multi-foil Insulation

The 1800°F composite insulation system, described in Quarterly Progress Report ALO 3632-39, is currently on long-term test at ~ 1800°F. The installed sample is 0.5" thick with an average layer density of 110 lb/inch. Molybdenum - Astroquartz is used as a shield-separator system in the 1800°F - 1600°F range. Nickel-Refrasil quartz (1600 - 1400°F), copper-Refrasil quartz (1400-900°F), and aluminum - aluminum opacified glass (900-400°F) are the remaining insulation components.

Prior to starting the long-term stability testing, apparent thermal conductivity data were obtained at several hot-side temperatures from 400-1800°F. These data are presented in Figure 2.1.4. The measured K_a values range from $.17 \times 10^{-3}$ Btu/hr-ft-°F at 400°F to $\sim 1.8 \times 10^{-3}$ Btu/hr-ft-°F at 1800°F. Actual test data were corrected for a uniform cold-side temperature (T_c) of 100°F.

As shown in Figure 2.1.5, the apparent thermal conductivity of the composite insulation is consistent with performance data for the individual component systems. The K_a of the composite mainly reflects the thermal performance characteristics of the Mo - Astroquartz and Ni-Refrasil quartz systems, which comprise 50% and 30% of the total composite wrap, respectively. The Al-Al opacified glass component, accounting for less than 5% of the composite, has little effect on overall composite thermal performance. However, these lower temperature materials, while not having a large impact on thermal behavior, do represent cost and weight savings in the composite system.

The 1800°F composite insulation has been operating for approximately 1000 hours at hot and cold side temperatures of 1790°F and 440°F, respectively, and in a vacuum environment of pressure (p) $< 0.5 \times 10^{-3}$ mm Hg. The insulation vacuum space is maintained by dynamic pumping (oil diffusion pumping system).

The effective thermal conductivities of the composite system, at time zero and after 1000 hours continuous operation, are tabulated below:

<u>Elapsed Time</u> Hrs.	<u>T_{Hot}</u> °F	<u>T_{Cold}</u> °F	<u>Vacuum</u> 10 ⁻³ mm Hg	<u>K_a</u> 10 ⁻³ Btu/hr-ft-°F
0	1790	430	< 0.5	1.95
1000*	1782	440	< 0.5	2.06

* 4000-hour total test time is scheduled.

FIGURE 2.1.4

THERMAL PERFORMANCE OF 1800°F. COMPOSITE MULTI-FOIL INSULATION SYSTEM

FIGURE 2.1.5 COMPOSITE MULTI-LAYER INSULATION PERFORMANCE

Total increase in K_a over this 1000-hour period is approximately 6%.

Figure 2.1.6 shows apparent thermal conductivity as a function of operating time for the 1800°F composite system. Again, the measured data (unbroken line) present a somewhat conservative picture of performance deterioration, due to the decreasing hot-side temperature (1790°F to 1760°F). The dashed line estimates the K_a increase which might have been expected if the 1790°F had been maintained. In subsequent stability testing, the input heat flux will be re-adjusted frequently to maintain the specified near-1800°F hot face condition.

2.1.3 Long-term Performance Deterioration - Potential Causes and Proposed Investigation

The increases in measured apparent thermal conductivity for the nickel opacified quartz and 1800°F composite multi-layer insulations have been observed over operating time periods ranging from 1000-6000 hours. It is felt that these K_a increases are attributable to some high-temperature material degradation. The critical questions which must then be answered in rating these insulations for long-term service are:

(1) The nature of the performance deterioration; i.e., long-term, continuing degradation or short-term degradation with subsequent re-stabilization of the insulation system at a somewhat higher performance level.

(2) The magnitude of the performance deterioration; i.e., the total increase in base thermal conductivity which may be expected in continuous operation at maximum rated temperature for periods up to 5 years.

Answering these questions will require a two-fold effort. First, further, in-depth evaluation of the individual material components is required to define potential causes of instability. Secondly, continued close monitoring of the long-term performance of the systems now on test may possibly provide some direct evidence as to the short-term or continuing nature of the deterioration. The currently scheduled operating times, particularly the 4000 hours for the 1800°F composite, are not considered sufficient to experimentally rate these systems. Extension of these stability tests to 10,000 hours continuous operating time at temperature has been proposed.

The two insulation systems and potential causes of system deterioration are discussed in the following paragraphs.

1800°F Composite Multi-foil Insulation

Thermal performance history of the 1800°F composite insulation shows an approximate increase in measured thermal conductivity of

MODIFIED COMPOSITE MULTI-LAYER INSULATION - LONG TERM TEST

FIGURE 2.1.6

6% after 1000 hours of operation. Although moderate, the performance deterioration trend is clearly evident from the K_a versus time plot of Figure 2.1.6. Possible contributors to this K_a increase are:

(1) Increased solid conductance (K_s) through the Astroquartz woven spacer due to increased fiber-to-fiber and interface contact (material "softening") at high temperature and under mild loading.

(2) Increased radiative heat transfer due to deterioration of the reflective properties of the molybdenum foil surface. This surface degradation (i.e., increase in effective emissivity) may result from thermal "etching" and/or recrystallization at the metal surface or, possibly, through some chemical interaction between the Astroquartz (SiO_2) and molybdenum (particularly at the grain boundaries).

Estimates as to the balance between conductive and radiative heat transfer for this system at 1800°F indicate 40% of total heat flux is transferred by solid conduction and 60% by radiation. Consequently, increases by either heat transfer mechanism would be significant in terms of the total measured performance.

The system performance investigation is being centered around the molybdenum-Astroquartz material combination; which is operating in the 1600-1800°F range. The other, intermediate-temperature material combinations are not felt to be influencing the system performance deterioration.* Also, the composite insulation was conservatively designed. Based on measured hot and cold boundary temperatures it does not appear that any of the intermediate-temperature components have exceeded their maximum rated service temperatures.

There has been no previous long-term stability testing on molybdenum-Astroquartz insulation materials at 1800°F. These two materials were selected over nickel and Refrasil quartz paper on the basis of 1800°F exposure and compatibility tests of 1 week duration (Reference Quarterly Progress Report ALO 3632-36, September-November, 1968). The molybdenum exhibited much better surface stability than nickel under the 1800°F and vacuum conditions, as indicated by reflectance tests and metallograph studies on both materials. While, on a comparative basis, these tests showed molybdenum to be superior to nickel, the one-week exposure obviously was not sufficient time to determine the stability and chemical compatibility of the molybdenum-Astroquartz system in long-term, 1800°F service.

In light of the observed deterioration (i.e., 6% increase in K_a after 1000 hours) in the 1800°F composite performance, the

* For example, a previous long-term (2000 hour) stability test on the nickel-Refrasil quartz system (used in the composite in the 1600-1400°F range) showed no performance deterioration in 1625°F operation.

composite materials are being evaluated at greater length in order to define potential causes of this deterioration. This initial study will qualitatively assess the metallurgical and chemical stability/compatibility characteristics of the molybdenum and Astroquartz (SiO_2) materials at 1800°F in vacuum. Potential sources of material degradation will be defined, and their effects on system thermal performance will be estimated.

Specifically, this initial study will provide information on the following:

- (1) Metallurgical or chemical interaction mechanisms which could contribute to this materials degradation and resultant deterioration in insulation performance.

- (2) Nature of these mechanisms (e.g., short-term or continuous effects); judgment as to the probability of their occurrence and the extent of their effect on the insulation materials.

- (3) Qualitative and/or quantitative support for these judgments (e.g., reference to literature data, etc).

- (4) Suggestions as to materials tests which would be within our testing capability, and would serve to further verify these judgments.

This preliminary study will be closely co-ordinated with the current performance stability testing, as indicated in Figure 2.1.7.

With regard to item 4, there may be some short-term, 1800°F stability tests which would provide valuable supporting data in the assessment of specific material degradation mechanisms. These tests, some of which would be within our testing capability as well as within the scope of our current contract, will be defined in the preliminary study. Obviously, any extensive experimental and analytical study of the various degradation mechanisms would be beyond the current financial and scheduling limits of the contract.

The materials degradation evaluations coupled with the 10,000 hour performance stability tests should result in an estimated performance rating for the composite insulation in long-term, 1800°F service. Factors to be considered in final rating of the system are:

- (1) Maximum performance deterioration which may be expected in 5-year service at 1800°F in vacuum. (It may be possible to design this level of deterioration into the system.)

FIGURE 2.1.7

LONG-TERM STABILITY EVALUATION - 1800°F

COMPOSITE INSULATION

(2) If the maximum deterioration is excessively high, alternatives which may be considered are:

(a) Other shield and/or spacer materials which may be more stable at 1800°F (e.g., columbium foils, woven zirconia spacer),

(b) Pre-treatment (i.e., pre-stabilization) of the existing molybdenum and Astroquartz materials before installation.

Nickel Opacified Multi-layer Insulation

Measured apparent thermal conductivity of the nickel opacified multi-layer insulation has increased by 16% over the 6000-hour continuous operating period. Performance history, as given in Figure 2.1.2, shows the K_a value to be steadily increasing throughout this time.

At the 1485°F temperature level, the most probable cause of performance deterioration is vaporization of the nickel flake opacifier. Based on a straight-forward free vaporization analysis for the nickel flake, it was estimated that the 8000-hour, 1485°F time-temperature condition would result in the loss of all nickel flake from the hottest insulation layer by the end of the 8000-hour period. However, it seems highly unlikely that degradation of one layer of material (less than 3% of insulation thickness) would result in a 16% increase in thermal conductivity. It would appear, then, that (1) the predicted extent of the nickel flake vaporization has been underestimated or, (2) another material degradation mechanism is influencing the performance. Underestimation of the vaporization is quite conceivable due to the very approximate nature of the calculations involved and the physical properties (e.g., nickel flake dimensions) used in the computations.

It is recommended that the nickel opacified insulation stability test also be extended to 10,000 hour duration. Post-test visual inspection and analysis of several material layers, exposed to various temperatures up to the maximum 1485°F, will be most significant in defining the material degradation mechanisms involved. Since no air exposure test is planned for this system, these post-test observations can be related directly to the 1485°F, vacuum exposure.

2.2 Thermal Conductivity Testing - Flat Plate Tests

The major objectives of the flat plate thermal conductivity test program are:

- (1) Obtain experimental thermal conductivity data under prescribed temperature and compressive load conditions, to a predictable degree of accuracy, utilizing flat plate testing techniques.
- (2) From these data, develop empirical expressions for the radiative and solid conduction contributions to thermal conductivity. This empirical relationship is then used to compute K-values for a given multi-layer system at any temperature boundaries within its prescribed service range, and at any layer density corresponding to 0-15 psi compressive loading.

The originally programmed work on aluminum, copper, and nickel multi-layer insulations has been completed and the data reported.* This report describes thermal conductivity evaluations in the following areas:

- (1) Re-testing of the aluminum multi-foil and aluminum opacified insulations, to provide sufficient performance data over their operating ranges.
- (2) Preparations for testing of a molybdenum-zirconia multi-foil insulation, at hot side temperatures up to 2500°F.

2.2.1 Aluminum - Glass Multi-layer Insulations -- Retest Program

Previous data for the aluminum multi-layer insulations were not sufficient to serve as a good basis for insulation system design over the entire operating range of these materials. The flat plate re-test program was designed to provide more extensive and more accurate data on the aluminum - glass systems, made possible through the use of revised and improved experimental techniques and procedures.

The multi-layer insulation material systems, test program and test objectives are described briefly in Table 2.2.1. Standard flat plate test apparatus, with combined sample-and-guard compressive loading, was used in all tests. This equipment and associated procedures have been described previously (Reference Quarterly Progress Report ALO 3632-33, pp. 44-47).

* Quarterly Progress Reports ALO 3632-33 and 3632-36.

TABLE 2.2.1

ALUMINUM MULTI-LAYER SYSTEMS

<u>Multi-layer Sample</u>	<u>Test Program</u>	<u>Objectives</u>
<u>Aluminum - 106 glass</u> .00025" Al foil (1.6 gms/ft ²) .003" 106 glass paper (1.7 gms/ft ²)	K _a measurement at 5 hot-side tempera- tures between 300- 900°F and 5 compres- sive loads between 0-15 psi.	Determine solid (K _s) and radiative (K _r) components of total thermal conductivity (K _a). Correlate data as a function of layer density (n) and temp- erature (T). Deter- mine effective emissi- vity of foil shields.
<u>Aluminum-Aluminum</u> <u>Opacified Glass</u> .00025" Al foil (1.6 gms/ft ²) .003" Al opacified glass (1.1 gms/ft ²)	As above.	As above.

<u>106 Glass Paper</u> No foil .003" 106 glass paper (1.7 gms/ft ²)	As above.	Determine K _s and K _r components of total K _a . Correlate data as a function of n and T. Determine effective backscat- tering cross-section (N) for fiber matrix as a function of n and at mean hot-side temperature (T _m).
<u>Aluminum Opacified</u> <u>Glass Paper</u> No foil .003" Al opacified glass (1.1 gms/ft ²)	As above.	As above.

2.2.1.1 Aluminum Multi-Foil Insulations

Flat Plate test data for the two aluminum multi-foil insulations are presented in Figures 2.2.1 and 2.2.2. Apparent thermal conductivities (K_a) are given as a function of layer density (corresponding to a compressive load range from 0-15 psi) at several hot-side temperature levels from 300°F to 900°F. Cold-side temperature in all cases was 80-100°F. The insulations were tested in a vacuum environment ($p \leq 1 \times 10^{-3}$ mm Hg).

Inspection of the apparent thermal conductivity plots indicates that the thermal behavior of these two aluminum multi-foil systems is typical of the behavior of multiple-layer systems under compressive load. The K_a values increase rapidly under the initial low compressive load (e.g., K_a for the Al-106 glass system at $T_h = 700^\circ\text{F}$ increases from $.15 \times 10^{-3}$ to $.45 \times 10^{-3}$ Btu/hr-ft-°F for a load increase from .04 psi to 1.0 psi). The rate-of-rise of K_a with load is much less severe in the 1.0 to 15.0 psi range (e.g., K_a for the Al-106 glass system at 700°F increases from $.45 \times 10^{-3}$ to $.95 \times 10^{-3}$ Btu/hr-ft-°F in the 1-15 psi range).

The initial, rapid increase in K_a for these systems is due to a significant increase in solid conductance as a result of breakdown in interface resistance between adjacent foil and spacer layers. At "zero load", contact between foil and spacer is minimal implying a low solid conductance. Under even mild loading, the contact increases sharply, resulting in a rapid rise in total thermal conductivity.

In the load range 1.0 - 15.0 psi, this interface effect no longer predominates. The solid conduction (K_s) increase is governed mainly by the increase in inter-fiber contact within the fibrous matrix, the physical characteristics of this matrix (e.g., woven cloth or fibrous paper), and the thermal conductivity of the fiber material itself. Within this range the layer density (n) increases significantly with load, thereby reducing the radiative contribution (K_r). This compensating or offsetting behavior of the K_s and K_r contributions results in a lower rate of increase of total conductivity (K_a).

These observations on thermal behavior of multi-foil systems under compressive load have been discussed previously (Reference Quarterly Progress Reports ALO 3632-33 and ALO 3632-36).

In comparing the total thermal conductivities of the Al-106 glass and Al-Al opacified glass insulation systems at corresponding load and temperature conditions, it is observed that:

(1) Total K_a of the Al-Al opacified glass insulation is 30-50% lower than K_a values for the Al-106 glass system, at all temperatures in the 0 - .5 psi range.

FIGURE 2.2.2

APPARENT THERMAL CONDUCTIVITY - -
ALUMINUM - ALUMINUM OPACIFIED MULTI-LAYER INSULATION

(2) In the 1 - 15 psi range the converse is true, with K_a values for the Al-Al opacified glass insulation running 50 - 100% higher than corresponding values for the Al-106 glass system.

This behavior is explained by examining the solid conduction (K_s) and radiative (K_r) contributions to total conductivity for the two insulations. Near zero load where K_s is very small, the 40% higher layer density* of the Al-Al opacified insulation results in a lower K_r contribution for this insulation in comparison with the Al-106 glass. This layer density difference accounts for essentially all of the difference in total K_a value. The use of the aluminum opacified separator in the multi-foil system provides little improvement in radiation attenuation, since the effect of flake opacification is so small in comparison to the densely packed, reflective foil shields.

At higher compressive loads where solid conductance is substantial, the higher K_s contribution of the aluminum flake opacified spacer overshadows the better radiation attenuation of the Al-Al opacified glass insulation. The plain glass spacer, on the other hand, has an inherently lower K_s factor resulting in a better overall performance for the Al-106 glass system.

Apparent thermal conductivity data are correlated by an empirical fit to a modified Wang equation of the form:

$$K(T, n) = a T^{1/2} + b T^3 \quad (2.2.1)$$

where a and b are functions of layer density (n). In integrated form, the mean thermal conductivity (K_a) of a multi-foil system between boundary temperatures T_h and T_c and operating in a vacuum environment is expressed as:

$$K_a \int_{T_c}^{T_h} = \left(\frac{2}{3}a\right) \frac{T_h^{3/2} - T_c^{3/2}}{T_h - T_c} + \frac{b}{4} \cdot \frac{T_h^4 - T_c^4}{T_h - T_c} \quad (2.2.2)$$

where T_h, T_c - boundary temperatures ($^{\circ}R$)
 $\frac{2}{3}a$ - solid conductance coefficient, a function of layer density (n)
 $\frac{b}{4} \approx \frac{\sigma \epsilon}{24n}$ - radiative coefficient, a function of layer density (n)
 n - layer density (ℓ/inch)

* Layer density of Al-Al opacified glass multi-layer is 204 ℓ/inch at .04 psi load, compared to 142 ℓ/inch for Al-106 glass insulation at the same .04 psi load.

This empirical correlation has been discussed in detail in previous reports (Reference ALO 3632-33, 3632-36, 3632-40).

Using experimental data at several temperatures and loads, the solid conduction ($2a/3$) and radiative ($\frac{b}{4}$) coefficients of Equation 2.2.2 were determined by means of the empirical - graphical techniques. These coefficients, and the temperature and layer density ranges for which they are valid, are given in Table 2.2.2.

The higher solid conductance of the Al-Al opacified glass system under compressive load is reflected in the $2/3a$ coefficient, which is approximately three times higher than that of Al-106 glass insulation at 15 psi. The effective emissivity of the radiation shields increases slightly over the load range as indicated. The "effective" values are somewhat higher than literature emissivity data for aluminum. This is felt to be due to the high degree of contact between the reflective surfaces and the adjacent separator materials, particularly at the higher compressive load. Other investigators have also noted this effect.*

In general, K_a values calculated by the empirically-developed, modified Wang expression (Equation 2.2.2) agree with measured data to within 10 - 20%. Deviations in the order of 20 - 40% occur, in some instances, at the zero load, lower temperature condition where random interface effects (influencing the solid conductance) may result in poorer agreement between the empirical fit and the measured data.

2.2.1.2 Fibrous Multi-layer Insulations

Experimental thermal conductivity data on non-opacified 106 glass and aluminum flake opacified 106 glass multi-layer insulation systems are presented in Figures 2.2.3 and 2.2.4. The K_a data are given at hot-side temperatures ranging from 300 - 800°F, and at compressive loads from 0 - 15 psi. Cold side temperature was 80 - 100°F in all cases.

It must be noted that the thermal conductivity curves of Figures 2.2.3 and 2.2.4 are applicable only for glass fibrous systems with thickness large enough to render boundary effects insignificant. These minimum thicknesses can range from .1" (for $\epsilon_b = 1.0$) to ~ 1." (for $\epsilon_b = .05$). In all test cases, the conditions of black boundaries ($\epsilon_b = 1.0$) and sample thickness > 0.1 " were met. Under these conditions, thermal performance of the fibrous systems is independent of thickness and is expressed as an effective thermal conductivity. For other boundary and

* Caren, R. P., "Radiation Transfer From a Metal to a Finely Divided Particulate Medium", Transactions of the ASME, February, 1969, pp. 154-156.

TABLE 2.2.2

EMPIRICAL THERMAL CONDUCTIVITY PARAMETERS

MULTI-FOIL INSULATIONS

<u>MULTI-FOIL SYSTEM</u>	$\frac{\frac{2}{3}a}{10^{-4} \text{ Btu/hr-ft-}^\circ\text{R}^{3/2}}$	$\frac{\frac{1}{4}b}{10^{-12} \text{ Btu/hr-ft-}^\circ\text{R}^4}$	ϵ_{eff}
<u>ALUMINUM-106 GLASS</u>	[.54 log n - 1.19]	72.1 $\frac{\epsilon}{n}$	.12 - .15
$T_h = 300 - 900^\circ\text{F}$ $n = 142 - 345 \text{ g/inch}$			
<u>ALUMINUM-ALUMINUM</u> <u>OPACIFIED 106 GLASS</u>	[1.67 log n - 3.97]	72.1 $\frac{\epsilon}{n}$	.08 - .10
$T_h = 300 - 900^\circ\text{F}$ $n = 200 - 450 \text{ g/inch}$			

T_h = hot-side temperature; T_c = cold-side temperature ($^\circ\text{R}$)

n = layer density; the given range corresponds to 0-15 psi compressive load.

$$K_a = \frac{2}{3} a \cdot \frac{T_h^{3/2} - T_c^{3/2}}{T_h - T_c} + \frac{1}{4} b \cdot \frac{T_h^4 - T_c^4}{T_h - T_c}$$

FIGURE 2.2.3

APPARENT THERMAL CONDUCTIVITY -
106 GLASS MULTI-LAYER INSULATION

FIGURE 2.2.4

APPARENT THERMAL CONDUCTIVITY -
ALUMINUM OPACIFIED GLASS MULTI-LAYER INSULATION

thickness parameters, the radiative coefficients of Table 2.2.3 may be used in computing the corresponding thermal conductivity. This characteristic of the fibrous multi-layer systems has been discussed previously (Reference Quarterly Progress Report ALO 3632-33).

As with the aluminum multi-foil systems, thermal performance data for the fibrous multi-layers are correlated using the semi-empirical expression for K_a as given in Equation 2.2.2.

$$K_a \int_{T_c}^{T_h} = \frac{2}{3} a \left(\frac{T_h^{3/2} - T_c^{3/2}}{T_h - T_c} \right) + \frac{b}{4} \cdot \frac{T_h^4 - T_c^4}{T_h - T_c} \quad (2.2.3)$$

Again, the solid conduction term $(2a/3)$ is a function only of layer density, n . Figure 2.2.5 represents the relationships between $(2a/3)$ and n for plain and opacified 106 glass. As in evaluation of previous multi-layer insulation systems, the $(2a/3)$ coefficient is approximated by a simple log function. Unfortunately, it is evident that for both systems the simple log function is somewhat inaccurate in relating the data. The inaccuracy is most prevalent at the low loads (layer densities) where interface effects are most significant.

Based on the Larkin-Churchill expression for a fibrous system in which backscattering is the dominant mode of radiation attenuation, the radiation coefficient is expressed as follows:

$$\frac{b}{4} = \frac{\sigma \lambda}{12n} \cdot \frac{1}{\left[\frac{1}{\epsilon_h} + \frac{1}{\epsilon_c} - 1 + \frac{\lambda}{n} \cdot N \right]} \quad (2.2.4)$$

where λ = total layers in sample
 n = layer density (ℓ/inch)
 N = effective backscattering cross-section, (inch^{-1})

For black boundaries and thickness $> 0.1''$, the expression may be simplified to the following:

$$\frac{b}{4} \approx \frac{\sigma}{12N} \quad (2.2.5)$$

The effective backscattering cross-section (N) is determined from experimental performance data, and related to layer density (n) in

TABLE 2.2.3

EMPIRICAL THERMAL CONDUCTIVITY PARAMETERS
FIBROUS MULTI-LAYER INSULATIONS

<u>FIBROUS SYSTEM</u>	$\frac{2}{3^a}$ $10^{-4} \text{ Btu/hr-ft-}^\circ\text{R}^{3/2}$	$\frac{1}{4^b}$ $10^{-12} \text{ Btu/hr-ft-}^\circ\text{F}^4$	$\frac{N_{\text{eff}}}{\text{in}^{-1}}$
<u>106 GLASS</u> $T_h = 300 - 900^\circ\text{F}$ $n = 200 - 450 \text{ lb/inch}$	$[.15 \log n - .33]$	$144 \frac{\lambda}{n} \left[\frac{1}{E + \frac{\lambda}{n} (N)} \right]$	$(.32 n + 68)$
<u>ALUMINUM OPACIFIED</u> <u>106 GLASS</u> $T_h = 300 - 900^\circ\text{F}$ $n = 275 - 550 \text{ lb/inch}$	$[1.31 \log n - 3.21]$	$144 \frac{\lambda}{n} \left[\frac{1}{E + \frac{\lambda}{n} (N)} \right]$	$(.15 n + 1000)$

T_h = hot-side temperature ($^\circ\text{R}$); T_c = cold-side temperature ($^\circ\text{R}$)
 n = layer density; the given range corresponds to 0-15 psi compressive load.

$$K_a = \frac{2}{3^a} \cdot \frac{T_h^{3/2} - T_c^{3/2}}{T_h - T_c} + \frac{1}{4^b} \cdot \frac{T_h^4 - T_c^4}{T_h - T_c}$$

$$\frac{\lambda}{n} = \Delta X; \quad E = \frac{1}{\epsilon_h} + \frac{1}{\epsilon_c} - 1$$

FIGURE 2.2.6 EFFECTIVE BACKSCATTERING CROSS-SECTION
FOR GLASS FIBROUS MULTI-LAYER INSULATIONS

Figure 2.2.6. The variation in N can be closely approximated by a linear function, within the layer density (load) range under consideration. The empirically determined N -values are denoted as effective or mean values for use over the entire 300-900°F. temperature range. The mean hot-side temperature is indicated for each curve.

Solid conduction ($\frac{2}{3}a$) and radiative ($\frac{b}{4}$) coefficients of Equation 2.2.3 are given in Table 2.2.3, along with temperature and compressive load ranges for which they apply. The effective backscattering cross-section (N), expressed as a function of layer density (n), is also included. This is the key parameter in comparing radiation attenuating capability of the various fibrous multi-layer insulations. It will be further discussed in Section 2.2.1.4.

The effects of solid conduction and radiation heat transfer mechanisms on system performance are illustrated clearly by the unique thermal characteristics of non-opacified and aluminum opacified glass multi-layer systems. The nature of the thermal performance for each of the fibrous multi-layer systems is discussed below:

106 Glass (non-opacified) -- Heat transmission is primarily by radiation, as indicated by the relatively small effective backscattering cross-section (N) of Figure 2.2.6. The increasing N -value with increased layer density is reflected directly in the total apparent thermal conductivity curves of Figure 2.2.3. The improved radiation attenuation at higher density, and the dominance of the radiation mechanism in total heat transfer result in a total K_a which decreases with increasing layer density.

Al Opacified 106 Glass -- Total apparent thermal conductivity increases with increased load (layer density), as illustrated in the curves of Figure 2.2.4. Radiant heat transfer by scattering is small as implied by the N -value plot of Figure 2.2.6. The contradictory behavior of the total K_a and the radiative component K_r (i.e., K_r decreasing with load while K_a increases with load) would indicate that solid conduction K_s is the dominant mode of heat transfer in this system. The significance of the aluminum flake opacifier in increasing solid conduction is also illustrated in current data on the Al-Al opacified glass multi-foil system.

It must be noted that the observations are somewhat qualitative in nature but, nonetheless, are quite useful in gaining a further understanding of the heat transfer mechanisms which affect thermal performance of these multi-layer systems. A more comprehensive study would require rigorous analysis of the individual heat transfer mechanisms; this effort is beyond the scope of the current program.

2.2.1.3 Accurate Curve Fitting of Experimental Data

Evaluation of the solid conduction coefficient and the back-scattering cross-section per unit volume is useful to the extent that it enables us to obtain the relative size of each mode of heat transfer. However, this approximate expression may not always give us the most accurate fit of the total thermal conductivity for the system. In cases where the total apparent thermal conductivity is of prime importance, it may be convenient to define one equation which is valid for all combinations of the pertinent parameters -- T_h , T_c and n . T_c can be fixed without losing generality (see Equation 2.2.6). Then, there are only two parameters T_h and n relating to K_a , and curve fitting with octagonal polynomials can be accomplished quite easily.

This mathematical fit of the K_a data has been obtained for the two aluminum multi-foil insulations as well as the two glass fibrous multi-layer systems. Data based on this fit are compared with experimental K_a values in Figures 2.2.7 to 2.2.10.

Generally, the values generated by the empirical expressions agreed with measured data to within 5-10%. This represents an improvement over agreement obtained with the modified Wang expression (i.e., 10-20% in most instances).

Apparent thermal conductivity may also be predicted when cold-side temperatures are other than 100°F (560°R), by using the following expression in conjunction with the empirical relationships given on Figures 2.2.7 to 2.2.10.

Let $K_a (T_h, T_c)$ be the desired thermal conductivity, where

T_h = hot-side temperature, °R

T_c = cold-side temperature, °R (not equal to 560°R)

Then,

$$K_a (T_h, T_c) = \frac{K_a (T_h, 560)(T_h - 560) - K_a (T_c, 560)(T_c - 560)}{(T_h - T_c)} \quad (2.2.6)$$

7-15-69 *Am*

Fig. 2.2.8 Experimental data vs. best empirical expression for the thermal performance of Al foil-Al opacified glass paper multi-layer insulation system

7-15-69 *km*

5-13-69/m

Fig.2.2.10 - Experimental data vs. best empirical expression for the thermal performance of al-opacified glass paper multi-layer insulation system

2.2.1.4' Observations on Thermal Behavior of Aluminum - Glass Multi-layer Systems

Thermal performance of the aluminum - glass multi-foil systems and for the plain and aluminum opacified glass fibrous multi-layer systems can be regarded as typical of these types of multiple-layer insulations. Thermal conductivity relationships to temperature and to compressive load (layer density) are characteristically similar to previously tested systems (i.e., nickel-quartz and copper-quartz multi-layers).

The new data on these insulations cover a much wider range of compressive loads and temperatures than the originally reported data (Reference Quarterly Progress Report ALO 3632-33, June-August, 1968). Also, the new K_a values at the higher compressive loads (e.g., 1-15 psi) directly contradict the original data and confirm the suspicion that the initial measurements at these higher loads were in error.* For future reference, the K_a -data on aluminum - glass insulations presented in this report supersede the original values given in Quarterly Progress Report ALO 3632-33.

The key thermophysical parameters (i.e., effective emissivity (ϵ) for the multi-foil systems and effective backscattering cross-section (N) for the plain fibrous insulations) were qualitatively in agreement with literature values. The effective emissivities for the tested multi-foil insulations were, in most cases, about a factor of two higher than corresponding literature data.** This increase is attributed mainly to the high degree of contact between the reflective foil surface and the fibrous spacer materials in the densely-packed multiple foil systems. As indicated in Table 2.2.1, this increase in $\epsilon_{\text{effective}}$ is more pronounced at the higher loads (i.e., layer densities). Other investigators have attempted to define this effect quantitatively from electromagnetic theory. (Reference given in Section 2.2.1.1.) Some effort will be made to apply results of these studies to our evaluation of effective emissivities of multi-foil systems.

Effective backscattering parameters for plain and opacified fibrous multi-layer insulations are presented, as a function of density and for indicated mean temperatures, in Figure 2.2.11. This parameter indicates the radiation attenuation capability of the fibrous materials. For comparative purposes, data for Refrasil quartz, plain and nickel flake opacified, are presented along with the recent values for aluminum opacified and plain 106 glass fibrous systems.

* The original experimental technique provided for sample compression only (i.e., the guard uncompressed at all times) which created significant thermal mismatch between sample and guard at higher loads. It is felt that this mismatch is minimized in the current technique where guard and sample are compressed simultaneously.

** Literature emissivity data are reported for surfaces radiating to a vacuum or inert gas environment. In multi-foil systems, the shields radiate to a fibrous media (i.e., adjacent spacer) which is in intimate contact with the foils.

FIGURE 2.2.11

EFFECTIVE BACKSCATTERING CROSS-SECTION
GLASS AND QUARTZ FIBROUS
MULTILAYER INSULATIONS

Specific observations on the backscattering characteristics of these fibrous systems are as follows:

(1) Magnitudes of the N-values for plain 106 glass ($.6\mu$ nominal fiber diameter) and Refrasil quartz ($1.0 - 1.5\mu$ nominal fiber diameter) multi-layer are essentially the same (100 in^{-1} to 200 in^{-1} over the density range).

(2) All N-density plots show an increase in backscattering cross-section corresponding to increasing density.

(3) Both aluminum and nickel flake opacification improve radiation attenuation (i.e., increase effective N) of the fibrous materials, with the aluminum flake being much more efficient in this capacity. For example, at low density, aluminum flake opacification increases effective N by a factor of 7. Nickel flake opacification, on the other hand, results in a factor of 2 increase in effective N.

Observations 1 and 2 are in accord with findings reported by other investigators (e.g., Larkin and Churchill). Observation 3 is of particular significance and indicates that an important criteria in metal flake opacification is the volumetric quantity of flake which is retained within the fibrous matrix. On a weight basis, nickel opacified quartz contains 40% nickel flake while aluminum opacified glass has 30% aluminum flake. However, on a volumetric basis, the aluminum opacified glass contains a quantity of dispersed flake (per unit volume of bulk fibrous matrix) which is ~ 2.5 times greater than in the nickel opacified system, due to density difference between aluminum and nickel. This greater volume of dispersed flake (i.e., greater flake surface area) makes the aluminum opacification much more effective.

In conclusion, the thermal performance characteristics of the aluminum - glass multi-layer insulations can be summarized as follows:

(a) Aluminum - Glass Multi-foil Systems

In closely-packed, multiple foil systems at low loads ($< 0.5 \text{ psi}$), performance improvement gained in using an aluminum opacified glass spacer instead of a plain 106 glass spacer is insignificant.* At loads from $.5$ to 15 psi , the higher solid conductance of the Al opacified spacer results in a higher total K_a for this system. For example, K_a of the Al-Al opacified glass system is a factor of 2 higher than K_a of the Al-106 glass system at 15 psi .

* The lower K_a values for the Al-Al opacified glass multi-foil system in this low load range were due to the higher shield density achievable through using the thinner, Al opacified spacer. They were not due to the Al opacification itself.

To summarize, in multi-foil systems where radiation is already minimized by the foil shields, an opacified spacer does not give significant performance improvement and, furthermore, can be detrimental at high loads due to its higher solid conductance.

(b) Non-Foil Glass Fibrous Multi-layer Systems

The aluminum flake is an efficient opacifying agent for use in glass fibrous papers. At the 800°F operating level, apparent thermal conductivity (K_a) is reduced by a factor of 8 (at near zero load) and by a factor of 2 (at 15 psi load) by using the opacifier. The diminished total effectiveness at higher loads is due to a corresponding increase in solid conductance in the aluminum opacified paper over the plain 106 glass.

2.2.2 Molybdenum - Zirconia Multi-foil Insulation - 2500°F Testing

Apparent thermal conductivity measurements on the Mo-ZrO₂ multi-foil insulation system, operating at hot-side temperatures to 2500°F, will be conducted during the next quarter.

The basic flat plate test configuration will be retained in these tests. Modifications to permit the 2500°F operation have been completed and the hardware has been installed. These design modifications (e.g., water cooled sink plates, alumina shielded heater, etc.) were described and discussed in Quarterly Progress Report ALO 3632-39, February, 1969.

The insulation for 2500°F testing is a multi-layer system of molybdenum foil and zirconia cloth. The molybdenum foil is .0005" thick and, in this thickness, weighs 12 gms/ft². Its emissivity ranges from .03 - .18 for temperatures ranging from 500°F to 2500°F. The zirconia cloth is nominally .005" thick with a specific weight of ~ 6 gms/ft². The open weave cloth is produced by Union Carbide Research Institute, by means of a "replica" technique.

The molybdenum and zirconia materials have been received. Physical dimensions and weights for these materials, along with load-deflection characteristics of the Mo-ZrO₂ multi-layer system, will be checked prior to thermal testing.

The apparent thermal conductivity of the molybdenum-zirconia cloth multi-foil system will be measured at several hot-side temperatures within the 500-2500°F range and at layer densities corresponding to compressive loads in the 0 - 15 psi range. Emphasis will be placed on the K_a-data at the low, near-zero loadings where the insulation would normally be used. The K_a data will be correlated to layer density and temperature. A suitable expression for apparent thermal conductivity, of the form:

$$K_a \left[\begin{array}{c} T_h \\ \\ T_c \end{array} \right] = \left(\frac{2}{3}a \right) \cdot \frac{T_h^{3/2} - T_c^{3/2}}{T_h - T_c} + \frac{b}{4} \cdot \left(\frac{T_h^4 - T_c^4}{T_h - T_c} \right), \quad (2.2.7)$$

will be determined for subsequent use in regenerating K_a-values at any temperature and layer density conditions within the 500 - 2500°F and 0 - 15 psi ranges.

2.2.3 Data Analysis Program

The flat plate thermal conductivity testing program is near completion. Extensive data have been obtained on 12 of the 14 multi-layer insulation systems scheduled for evaluation under the contract. During the next quarter, data review procedures will be organized and implemented to interpret and analyze all thermal conductivity data for these systems. The principal objectives of this program are:

(1) Establish and verify accuracy of the measured thermal conductivity data.

(2) Re-evaluate the flat plate test equipment and procedures.

In the absence of experimental proof tests or multi-layer insulation standards which could serve as direct verification of data accuracy, a comprehensive data review program will be a valuable tool in estimating limits of accuracy for these thermal conductivity data. Extensive K_a -data (cylindrical and flat plate testing) are now available on a variety of multi-layer insulation systems. The wide range of these data should permit a thorough and meaningful analysis. In addition to the data evaluation, the overall study will also include re-assessment of the flat plate equipment and testing methods. Any design or test procedure changes, which may have potential for upgrading the overall flat plate testing approach, will be defined and discussed.

Pertinent steps in the data review and analysis program are outlined below:

(1) Re-assessment of estimated error bands associated with both cylindrical and flat plate test results.

(2) Comparison of cylindrical and flat plate test data in all cases where good correspondence exists in the test conditions (e.g., temperatures, layer density, etc.). Determination of degree of agreement and consistency of agreement between K_a -data obtained by the two methods.

(3) Comparison of measured K_a -values with predicted thermal conductivity data, the latter based on thermophysical properties (e.g., foil emissivities) of the component materials taken from the literature.

(4) Review of the flat plate test method, and discussion of potential means of improving or upgrading the equipment and techniques.

The main value of the study will be as a comprehensive summary of thermal performance data on the high temperature multi-layer insulation systems. For all data, accuracy limitations and range of applicability will be described. In addition, it is hoped that this overall study will provide further insight into the thermal characteristics of multiple layer systems, and the role played by shield and spacer material properties in establishing the overall system performance.

2.3 Penetration Effects Testing - Interim Status

The experimental program is designed to measure the effects on thermal performance of an array of penetrating elements, uniformly spaced over a multi-foil insulation section. Principal objectives of the program are to:

1. Provide verification of the analytical techniques used to assess thermal effects of penetrations.
2. Assess the effect on overall performance of various methods of insulating around penetrating elements --- e.g., open gap, zero gap, and opacified paper filled gap.

The test sample is comprised of an aluminum-aluminum opacified glass multi-foil insulation system, containing an array of 0.1" diameter lead telluride* elements (flush penetrations) located on 1" centers. The test system geometry and boundary conditions selected for this study are as follows:

Boundary Temperatures	900°F. (Hot Side) 100-200°F. (Cold Side)
Penetrations	
(1) Material	Lead Telluride (p or n type)
(2) Size	0.1" Dia., .2" length
(3) Spacing	1.0"
(Adiabatic Radius	(0.5")
(4) Gap Encircling	0.075"
Penetration	
Insulation	
(1) Materials	.00025" Al foil .003" Al opacified glass paper
(2) Thickness	~ 0.165"
(3) Layer Density	~ 200 g/in.

The lead telluride penetration material described above was supplied by Gulf General Atomics. The material was readily available only in the ~ .2" long elements.

The experimental basis, description of sample configurations, test procedures and apparatus were discussed in detail in Quarterly Progress Report ALO 3632-39, December, 1968 - February, 1969.

* This material is being used in certain types of thermoelectric generators.

System Thermal Performance Testing

The guarded flat plate test configuration, used in all penetration-insulation system tests, is shown schematically in Figure 2.3.1. The 6" x 6" aluminum-aluminum opacified glass multi-layer insulation sample contains an array of 36 lead telluride elements, mounted flush with the insulation and spaced on 1" centers. The edge guard sections are constructed of the same multi-layer insulation materials but do not contain penetrations. Heat flux through the penetration-insulation system is measured over a 2" x 2" central area by a flux meter located at the cold boundary. The peripheral penetration-insulation sections provide a "self-guarding" feature within the sample. Hopefully, this guarding effect is sufficient to achieve approximately one-dimensional heat flow within the 2" x 2" metered section.

A reference insulation system --- aluminum-aluminum opacified glass multi-foil at a layer density (n) of 200 g/inch (~ 0.165 " sample thickness) and without penetrations --- was first tested. The purpose was to provide base heat flux data on the multi-foil system to be used in all subsequent penetration-insulation system studies, and to provide a simple, comparative calibration on the penetration effects test apparatus. Testing was in vacuum ($p < .5 \times 10^{-3}$ mm Hg) at hot-side temperatures ranging from 300-900°F. Cold-side temperatures were maintained in the 100-150°F range by water cooling.

Effective thermal conductivity data on the reference aluminum-aluminum opacified glass multi-foil insulation system are presented in Figure 2.3.2. Corresponding values for an identical multi-foil system, tested on the regular flat plate apparatus, are also plotted for comparison. Agreement between the two sets of test data is generally within 10% over the temperature range.

Effective thermal conductivities of the same insulation system containing the lead telluride elements (on 1" centers) were estimated from 3M Company K-data for the lead telluride material. These predicted K_a -values, determined from simple, one-dimensional calculations, are also plotted in Figure 2.3.2. Comparing K_a -data for the insulation alone with corresponding penetration-insulation system values, it is noted that heat transfer through the penetration material comprises > 95% of the total flux through the penetration-insulation system.

Initial tests on the penetration-insulation system configurations (zero-gap and filled gap) yielded effective K-values which were approximately a factor of 10 lower than predicted. The problem was traced to severe interface resistance between the penetrations and the system

Figure 2.3.1

Schematic of Penetration - Insulation Test Samples

boundary surfaces. The simple, bearing contact established between penetrations and boundary plates was not adequate to overcome this resistance.

Specific factors contributing to this interface problem were:

(1) The rather weak bearing contact produced by the static weight of the sink plate.

(2) Non-uniformity of contact between the penetrations and the boundary surfaces due to slight deviations in penetration length.

(3) Oxide coating on the end areas of the penetrations, which added more thermal resistance.

(4) Chemical interaction at the hot-side between the lead telluride material and the copper shield plate on the heater, which created further interface resistance at the hot boundary.

It was determined by means of comparative tests that simple design changes in the sample configuration would produce marked reduction in interface resistance. These and other changes which are being incorporated into future penetration-insulation test systems, are illustrated schematically in Figure 2.3.3 and discussed briefly below:

(1) Bearing load at the interface between the penetrating elements and the boundary surfaces has been increased substantially. A spring-loaded system is now used to compensate for any dimensional changes as the sample is taken up in temperature.

(2) A soft, .010" thick indium sheet is used as an intermediate contact layer between the cold boundary (flux meter) and the penetrations. This relatively thick, ductile sheet will tend to "absorb" slight deviations in penetration height and promote uniform thermal contact across the entire penetration array.

(3) Ends of penetrations are abraded to mechanically remove the oxide and, then, indium coated to provide a high-conductivity contact surface.

(4) Gold foil tabs (~ .002" thick) are bonded to the hot end of the penetrating elements to eliminate reaction between the lead telluride and heater shield plate.

These design modifications, all directed toward minimizing the interface resistance problem, will be incorporated on all penetration-insulation system samples. Re-test of the various configurations will be conducted during the next quarter, and performance will be compared to analytical prediction. Test results will be reported in Quarterly Progress Report ALO 3632-43, June-August, 1969.

FIGURE 2.3.3

MODIFIED SAMPLE DESIGN - PENETRATION EFFECTS TESTING

3.0

ACTIVITIES DURING THE NEXT REPORTING PERIOD

- Continue long-term testing of the nickel opacified multi-layer insulation system.

- Continue long-term testing of the 1800°F composite multi-layer insulation. Complete preliminary study on potential causes of long-term performance deterioration currently being experienced.

- Flat plate thermal conductivity tests on the molybdenum-zirconia multi-foil system at hot-side temperatures up to 2500°F.

- Complete penetration effects testing (to 900°F) on the Al-Al opacified glass multi-foil insulation system with lead telluride penetrations.

- Begin thermal conductivity data re-evaluation and summary, covering K_a -data on all multi-layer insulations tested in the Phase II program.