PRELIMINARY DESIGN OF A HIGH TEMPERATURE SPACE MANUFACTURING FURNACE

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January 1970

Contract NAS8-21347

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#### PRELIMINARY DESIGN OF A HIGH TEMPERATURE SPACE MANUFACTURING FURNACE

#### January 1970

Contract NAS8-21347

#### Prepared for George C. Marshall Space Flight Center National Aeronautics and Space Administration

by

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#### FOREWORD

This report represents the results of work performed by the Thermal Environment Section of the Aeromechanics Department of Lockheed Missiles & Space Company, Huntsville Research & Engineering Center, for the National Aeronautics & Space Administration, Marshall Space Flight Center, Huntsville, Alabama. This task was conducted for the fulfillment of Modification No. 4 of Contract NAS8-21347, "High Temperature Furnace Design."

#### SUMMARY

A preliminary thermal design analysis has been performed for developing a high-temperature space manufacturing furnace that will be used for melting a glass specimen in the Orbital Workshop. The primary design objective was to develop a furnace which would melt the specimen under specified heat-up and cool-down rates. Secondary objectives were that thermal shock be avoided during heat-up and that temperature uniformity over the surface be assured during the entire process.

A nominal furnace with a 150-watt heat input was selected as a basic design about which parametric investigations were conducted. Parameters considered were insulation material, insulation material thickness, and spacing between components of the furnace. A thorough investigation was made to determine the best insulation material which would be applicable to the  $2600^{\circ}$ F,  $10^{-4}$  torr environment. Types of insulations considered were foams, powders, fibers, and systems composed of an insulation material with refractory radiation shields. A discussion is presented of the properties and limitations of these materials along with recommendations for their use on the high-temperature furnace. Transient thermal analyses were performed on the nominal furnace design considering each recommended insulation material, insulation material thickness and spacing between components of the furnace for evaluating heat-up and cool-down rates of the glass specimen.

Completion of the preliminary design indicated that the heat-up rates, temperature uniformity, and thermal shock requirements could be achieved through minor modifications to the nominal furnace design. Cooldown rates could not be met, however, using any of the insulation materials and natural cool-down of the furnace. As a result, the adaptation of quenching methods into the furnace system for cooling the specimen was recommended.

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Recommendations were also made for modifying the present furnace design to accommodate other space manufacturing experiments.

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## Section 1 INTRODUCTION

Current goals of the Apollo Applications Program are to investigate new concepts, through the utilization of an earth-orbital environment. Areas of interest range from remote sensing for helping farmers and fisherman with their crops and fishing methods to astronomical observations of our solar system and surrounding galaxies to yield a better understanding of the composition and origin of the universe.

One of the areas of investigation will be the manufacture of materials under the influence of a zero-gravity and possibly a vacuum environment. Goals of the space manufacturing effort are to produce new and greatly improved materials, to manufacture products more precisely and to process materials in a new and different way.

Materials under consideration for the space manufacturing effort range from metals to important pharmacenticals with great technological benefits anticipated from each type of experimentation. Dr. Emil W. Deeg of American Optical Company, indicates that a current need exists in the manufacture of glass for improved optical and semiconductive properties. This is particularly required in the manufacture of large lenses for cameras used in highlevel reconnaissance by the Air Force and in many other applications in which large quantities of glass are desired. Pictures produced using lenses which are not optically perfect cause distortion with respect to dimensionality and color contrast. Results of Dr. Deeg's investigations indicate that by solidifying glass in a zero-gravity, low-pressure environment, the resulting glass product will exhibit improved characteristics.

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An effort was begun, based on Dr. Deeg's investigations, to test a small specimen of glass under the zero-gravity/vacuum conditions of the S-IVB Workshop to verify his assumptions. Verification will require the use of a high-temperature furnace capable of melting, containing, and cooling a ball of glass under the condition of zero-gravity at a temperature of  $2600^{\circ}$ F and a pressure of  $10^{-4}$  torr over a specified time period.

Lockheed's role in this effort was to perform a preliminary design analysis of the proposed high-temperature furnace by selecting an appropriate insulation material and then performing transient thermal analyses to determine a best furnace design for the glass experiment. In performing the analysis, consideration was given to: (1) attaining uniform heating conditions on the glass specimen; (2) meeting specified heating and cooling time requirements and; (3) providing a means of venting or absorbing gases as a result of offgassing from the furnace insulation and the glass specimen.

## Section 2 INSULATION SELECTION

#### 2.1 APPROACH

To evaluate all possible insulations for the furnace application, an effort was made to find all pertinent information on existing materials. This effort was divided into two phases: (1) literature surveys, and (2) personal contacts with manufacturing company representatives.

Literature surveys involved utilization of AMC's Redstone Scientific Information Center retrieval information service as well as the classified documents and nonrestricted book-card catalogs. Results of this phase revealed that Authur D. Little, Inc., had performed, under an Air Force contract, many tests of insulation materials at high temperatures. Other testing had been performed under an Atomic Energy Commission contract by Linde Division of Union Carbide; however, the temperature range and the materials involved in the testing proved to be undesirable for the furnace application since their melting temperatures were below the operating temperature range of the furnace. Upon reviewing available literature, additional information was desired regarding the performance of certain materials and material combinations. To obtain the information, the companies which initially produced the source were contacted. Companies contacted included: Johns-Manville Corp., Authur D. Little, Inc., Linde Division of Union Carbide and Lockheed/Sunnyvale.

Information obtained from company contacts was helpful in that additional literature and the latest knowledge of the behavior of certain materials under test conditions were obtained. Behavior of the material and material combinations was primarily available from Arthur D. Little, Inc., and not from companies which manufacture the material. While the manufacturer

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seemed to know a great deal about the properties of his specific material, he was not familiar with the compatibility of his material in combination with other materials such as refractory metals for use as radiation shields.

Documents published by Arthur D. Little, Inc., (Refs. 1 - 3) and behavioral characteristics obtained from personal contacts provided enough information for an assessment of the state of the art of hightemperature insulators as of 1965-66, with an indication that the situation has not changed appreciably during the last four years. Recommendations as to possible action which should be taken as a result of this inadequacy are discussed later.

#### 2.2 MATERIALS

Furnace insulation requirements were such that the insulation material must maintain its thermal, chemical, physical and mechanical properties under the elevated temperature of  $2600^{\circ}$ F and pressure environment of the order of  $10^{-4}$  torr. Since further requirements were not specified, the initial investigation was directed toward finding all insulation materials that would at least meet these requirements and eliminate additional materials as further restrictions are specified.

Basically, there are four types of insulation materials: powders, fibers, foams and insulation systems composed of an insulation material and refractory radiation shields. General properties and expected behavior of these types of materials have been described as follows:

> • Powdered materials may be used in both high- and lowtemperature applications with little vacuum being required to eliminate the contribution to conduction by gaseous conduction. The sintering behavior of powders at 60 to 70% of their absolute melting point, however, may result in mechanical instability as well as an undesirable increase in thermal conductivity.

- Proper arrangement of fibrous materials can result in a low thermal conductivity which adds to its already desirable properties of low density, high porosity, and ability to absorb and scatter radiant energy, thus limiting radiation heat transfer.
- Foamed insulation materials are useful in the high-temperature regime in the form of ceramic and organic impregnated ceramic materials; however, most foamed materials have high thermal conductivities in the high-temperature region.
- By far the most attractive insulation material combination for low thermal conductivities in the high-temperature regime is the use of an insulation material having alternating layers of refractory metals. The refractory metals behave as radiation shields, thereby lowering the apparent thermal conductivity. This multilayered system is now in initial stages of development. Difficulties are being encountered in finding refractory metals that are compatible with currently existing low thermal conductivity insulation materials.

Therefore, materials considered in this study were mainly of the fibrous insulation type with emphasis on combining these as spacers with radiation shields and also on combining them with other insulation materials which are good performers in their temperature range. Materials (except those used in multi-component form) that would not be operable in the temperature range of  $2600^{\circ}$ F and also those which exhibited a relatively high thermal conductivity were not considered in the investigation.

Materials retained for investigation after the elimination process were:

- 1. Graphite fibers with tantalum shields and ADL-17 powder
- 2. Graphite fibers and tantalum shields
- 3. Combination of:
  - Dyna Flex
  - Flexible high temperature Min-K
  - Flexible standard Min-K
- 4. Dyna Flex
- 5. Dyna Quartz
- 6. Zirconia

- 7. Astroquartz
- 8. Sapphire wool
- 9. Dyna Quartz and tantalum radiation shields
- 10. Sapphire wool paper and stainless steel flakes
- 11. Refrasil (Type A-100, B-100, and Irish B-100)
- 12. Graphite fiber felt
- 13. Graphite cloth and pyrolytic graphite foam

Each material was individually analyzed with regard to its possible application to the zero-gravity furnace. Results of the analyses are presented in Appendix A and are summarized in Table 1. Of these materials several have been selected as being highly suitable for the furnace application.

#### 2.3 INSULATION SYSTEMS

#### 2.3.1 General Considerations

After each material and its possible use as an insulator on the furnace were considered, sample calculations were made for several types of insulation systems. Basically, they were divided into three categories: (1) solid; (2) multiple (composed of three materials, each a "best" insulator in its specific temperature range); and (3) multilayered (with radiation shields). Each type of insulation system was assumed to be enclosing a two-inch radius cylinder, four inches long, having a heat input of 150W, and existing in a vacuum environment with a mean temperature of 1300°F. Results of calculations are shown in Table 2. Results are presented in terms of the insulation thickness that would be necessary to meet the furnace requirements. Calculations considered only steady state conduction with radial heat transfer and did not include the effects of corners, ends, joints, and edges. These materials represent the best selected from the individual material analysis presented in Appendix A. Final selection of the insulation system was based on the selected materials' (1) thermal conductivity (2) chemical physical and mechanical stability and (3) overall suitability as an insulation system for this particular application. With respect to these three requirements the following observations have been made in Ref. 3 as a result of high-temperature tests conducted on these materials:

2.3.2 Thermal Conductivity

There is little difference in the apparent thermal conductivity among the silica-based materials (i.e., Astroquartz, Dyna Quartz, Dyna Flex). Conductivity of these materials generally increases with T<sup>3</sup>, which indicates the importance of radiation heat transfer.

Sapphire fiber materials in paper or mat form generally have a lower conductivity than Refrasil, but a higher conductivity than the silica-based fibers.

Fibrous insulations containing radiation shields exhibit conductivity values which are not greatly different from, but generally slightly lower than, simple single-component systems.

Graphite has a higher conductivity than either silica or zirconia insulations.

2.3.3 Chemical, Physical and Mechanical Stability

Sapphire fiber insulations generally show greater physical stability and chemical inertness at higher temperatures than the silica-based materials.

Dyna Quartz is stable up to temperatures near its melting point (2900°F).

Physical and mechanical stability of graphite fibers is superior to those of other materials at high temperatures; however, reaction occurs with the tantalum components at moderate and high temperatures.

Zirconia fibers exhibit considerable shrinkage at hot face temperatures near  $3000^{\circ}$ F, but are relatively inert chemically with the metallic components.

Tantalum components show less reaction with sapphire paper than with Dyna Quartz. The stainless steel radiation attenuator ("splat") is more reactive with sapphire paper than with the tantalum shielding.

2.3.4 Suitability for Insulation Systems

The single-component, silica-based fiber systems were most suitable for insulation applications because of their low cost, availability and ease of application.

Sapphire-whisker is limited by the excessive cost and the apparently higher conductivity values.

Graphite-fiber insulations are limited in usefulness by their high conductivity and reactions with non-graphite components.

Zirconia insulations have low conductivity, but are limited in high temperature (vacuum) applications by their stability.

Insulations composed of sheet-type radiation shields with insulating fiber spacers are:

- Limited by reactions between metal and the insulation
- Difficult to apply to real systems with curved surfaces or with small dimensions.

#### 2.4 RESULTS

Consideration of the insulation materials investigated in this study indicated that three insulation systems could be selected for use on the

furnace. These three were selected on the basis of low thermal conductivity, stability, ability to operate within the specified temperature range, and compatibility with the initial configuration of the furnace. They are:

- 1. Dyna Flex
- 2. Dyna Quartz
- 3. Zirconia

The combination of Dyna Flex, flexible high-temperature Min-K, and flexible standard Min-K had been considered as one of the better materials but has been eliminated. This decision was made because the material has very similar properties to the other three candidates and the difficulty in fabricating its complex design would not be worth any saving which might be realized.

Final selection of the specific insulation which should be used will depend on compatibility of the insulation with the relatively small dimensions of the furnace (Dyna Quartz is in a semi-rigid block form and may not be capable of meeting the dimensional requirements), availability of the material, and a thorough transient thermal analysis of the zero-gravity furnace.

## Section 3 FURNACE THERMAL ANALYSIS

#### 3.1 BASIC DESIGN OF FURNACE

In order to establish a best design for the high-temperature furnace, a logical procedure had to be determined by which each variable could be investigated. Therefore, a nominal furnace was selected based on information obtained from I. C. Yates of NASA/MSFC, Dr. Emil Deeg of American Optical Company, and simplified analyses involving basic physical and thermal constraints. This nominal case, shown in Fig. 1, consisted of a  $0.305 \text{ in}^3$  glass cylinder as the specimen, a 1.355 in. diameter platinum cylinder 1.355 in. long with a 0.03125 in. wall thickness acting as the crucible enclosing the glass, a 2.0 in. diameter ceramic cylinder 2.0 in. long with a wall thickness of 0.125 in. containing the heater element on the inside and enclosing the platinum cylinder, and 0.75 in. of Dyna Flex insulation surrounding the ceramic cylinder. The glass specimen is held inside the platinum crucible by six conical spikes 0.375 in. long with a 0.125 in. diameter base. Penetration of the spikes into the glass is assumed to be 0.0625 in.

Eight ceramic spikes are assumed to hold the platinum crucible in the center of the ceramic cylinder, thus providing uniform radiation to the platinum crucible. This nominal case was analyzed in detail and then used for conducting a parametric study of the furnace (see Table 3). The following parameters were varied individually:

- 1. Furnace components shape
- 2. Insulation material type
- 3. Insulation material thickness
- 4. Spacing between the crucible and the ceramic cylinder.

Parametric studies on the shapes of the furnace components involved beginning with the nominal furnace and modifying a specific component. Variations consisted of removing the ceramic cylinder from the nominal case and placing insulation around the platinum cylinder with the heater element attached to the outside surface of the platinum in such a manner that electrical shorting would not occur. Results obtained from performing a transient thermal analysis on these thermal models aided in determining the effect of employing a ceramic cylinder in the configuration of the furnace.

The insulation thickness of Dyna Flex was then varied from a minimum to a maximum dimension to determine the effect of insulation thickness on the temperature history of the glass specimen. The process was repeated for Dyna Quartz, and Union Carbide's ZYF-100 zirconia insulation. These insulation materials were selected as the three best candidates for the furnace application from the insulation investigation conducted in the initial phases of the preliminary furnace design. Therefore, by performing a transient thermal analysis on the above variations of the nominal furnace, the following information can be realized:

- The effect of insulation thickness on the temperature history of the glass
- The effect of different insulation materials (each with a different thermal capacity and thermal conductivity) on the temperature history of the glass.
- Effects of insulation and its thickness on the temperature gradient from surface to surface and from surface to center of the glass thus indicating the existence of a thermal shock on the glass.

Since the dimensions of the spikes holding the glass specimen have been specified, the distance between the platinum cylinder and glass cylinder were fixed. The dimensional relationships between the platinum cylinder and the ceramic cylinder have not been specified and were therefore studied to determine the

dimensional relationships that would provide the desired degree of temperature history and uniformity on the glass.

Transient analyses of the parametric investigations were achieved by developing a thermal model of the nominal furnace case and performing minor adjustments to accommodate each new parametric study. The nodal network for the furnace and glass specimen consisted of 270 nodes (shown in Table 4) which formed the conduction and radiation system (Fig. 2). The 150W heat source is assumed to be divided equally among the nodes (112 through 142) representing the heater element. Solutions to each model were obtained through two phases of endeavor. Phase I consisted of performing a radiation analysis in which all radiation view factors were computed (Ref. 7); while Phase II utilized the radiation view factors of Phase I together with the conduction thermal model to perform the transient thermal analysis (Ref. 8) of the entire configuration.

#### 3.2 RESULTS OF TRANSIENT THERMAL ANALYSIS

Transient thermal analyses performed on each of the previously defined parametric investigations revealed that results could be discussed in reference to four basic areas. These areas were selected because they are primary contributors for the successful design of the furnace to meet the requirements for melting the glass specimen. These are:

- 1. Temperature history of the glass center node,
- 2. Temperature uniformity of the glass surface,
- 3. Thermal shock of the glass caused by large surface-to-center node temperature gradients, and
- 4. Heat-up and cool-down rates of the glass center node.

Requirements for melting the glass specimen are that it be heated uniformly from an ambient temperature to its melting temperature and allowed to cool to a specified temperature. Specified thermal characteristics of the glass are that it has a melting temperature of  $2600^{\circ}$ F, a temperaturedependent thermal conductivity (Fig. 3), a thermal capacity of 0.16 Btu/lb- $^{\circ}$ F, a density of 156 lb/ft<sup>3</sup>, and an emissivity of 0.8. Of course, since the thermal conductivity is temperature-dependent, the thermal diffusivity will also be a variable. Properties of other materials used in the design of the furnace are contained in Table 5.

#### 3.2.1 Temperature Histories

Effects which insulation thicknesses have on the temperature history of the glass center node are shown in Fig. 4. These temperature histories were obtained by first performing a transient thermal analysis on the nominal case (Case 1, Mod 0 of Table 3) and then, keeping everything else constant, increasing Dyna Flex insulation thickness to 3.0 in. (Case 1, Mod 1) and 1.5 in. thickness (Case 1, Mode 2). Results were that as the insulation thickness is increased the time required for reaching the melting temperature is decreased while the time required for cooling is increased. Time required for cooling the center node to  $783^{\circ}$ F with 3.0 in. of Dyna Flex was 17,520 sec as compared to 11,880 sec for 1.5 in. of insulation. Therefore, for slow temperature increases and fast temperature decreases, insulation thickness should be small compared to 3.0 in. This will be discussed more fully under the section on heat-up and cool-down rates.

An insulation material's thermal properties could have a pronounced effect on the temperature history of the glass. Since the insulation analysis of Section 2 revealed that three insulation materials were suitable for performance on the furnace, transient analyses were performed using the nominal case with 1.5 in. of Dyna Flex and then considering Dyna Quartz and ZYF-100 insulation each with a thickness of 1.5 in. Investigation of Fig. 5 reveals that the selection of any one of the three insulation materials would provide approximately the same temperature history for the glass center node. Final selection of the insulation material for use on the furnace will not, therefore, depend upon the temperature history.

#### 3.2.2 Temperature Uniformity

Since the nominal case for the high-temperature furnace incorporates six platinum spikes to mount the specimen under gravitational and experimental conditions, some concern was registered as to the influence of the spikes on the thermal behavior of the glass. Without the spikes radiation would be the only mode of heat transfer from the heater element to the specimen; however, with the spikes conduction would be the primary mode of heat transfer and this would occur at only six locations on the glass.

Therefore, to investigate this phenomenon the transient thermal analysis of the nominal case was utilized. Referring to the nodal arrangement of Fig. 2 and the use of Table 4 one can see that nodes 6, 8, 16, 32, 17, 36, and 7 all lie in the same plane and are on the surface of the glass specimen. However, nodes 6, 32, and 7 are surface nodes which connect to spikes, the primary source of heat transfer. It was desired to show the temperature variation from one node to another at the same time and also to show the temperature variation for each node as a function of time. As a result, Fig. 6 was developed from which it can be seen that as the specimen is heated, the temperatures at the spiked nodes are greater than their neighbors, as would be expected. However as time increases and radiation becomes the dominant mode of heat transfer, small temperature gradients will exist between the nodes until the glass is melted and the heater element is turned off to allow cooling. Temperature variation past 1920 sec is not shown in Fig. 6 since it would be a repetitious increase in temperature and no nodal variation until the specimen melts. When cool-down of the glass melt begins the spikes behave as heat sinks, and temperature decreases in the same manner as it increased (i.e., the spike temperature decreases faster than the glass).

Maximum temperature gradient shown in Fig. 6 is 700<sup>°</sup>F between node 32 and nodes 8 and 36. Further observation of Fig. 6 reveals that spiked nodes 6 and 7 are a few degrees warmer than the furface nodes 8 and 36,

but there is a large difference between these nodes and node 32. This can be explained by referring to Figs. 1 and 2 and Table 4 to observe the flow of heat energy from the heater elements (nodes 112 to 143) to the glass specimen. The ceramic spike (node 89) is actually in a plane 45 deg from the platinum spike (node 32). This design was selected to minimize hot spots in the furnace system. As a result of this arrangement nodes 57, 88, 89 and 66 lie in the same 45-deg plane. Heat will flow by conduction from the ceramic spikes (node 89) to the junction node on the platinum cylinder (node 88). At this point heat will be transferred in two directions: (1) toward node 57 (the top of the platinum cylinder); and (2) toward node 66 (the side of the platinum cylinder). A temperature difference will exist between nodes 57 and 66 due to differences in the resistance between nodes 57 to 88 to 89, and 66 to 88 to 89 as well as capacitances of nodes 57 and 66. Resistances between 57-88-89 and 66-88-89 are 4.06 (10)<sup>6</sup> and 1.485 (10)<sup>6</sup> respectively while capacitances for nodes 57 and 66 are  $1.67 (10)^{-4}$  and  $3.55 (10)^{-4}$ , respectively. The temperature difference of nodes 57 and 66 is then carried to the top of the cylinder and side of the cylinder by way of 57-45-44-6-8 and 66-64-47-46-32-16 thus causing nodes 6 and 8 to be lower in temperature than nodes 16 and 32.

Current indications are that the temperature difference between the cylinder top and side as well as differences between spiked node 32 and nodes 16 and 17 exceed the requirements for temperature uniformity along the specimen. Solution to this occurrence is to decrease the thickness of the cylinder wall thus decreasing the capacitance of node 66 and increasing the resistance. The resulting effect is a decrease in the temperature of the node that connects the spike to the glass specimen and a slight increase in spiked nodes 6 and 7.

Because the glass specimen is three dimensional, a check on the temperature uniformity in a plane perpendicular to the previous plane gives a complete indication of the overall temperature uniformity of the glass.

It is recognized that an explanation of this flow of energy is difficult to present; however, of the presentation options available, the one used was deemed best.

Such a plane (see Fig. 2 and Table 4) will contain the circumferential surface nodes 16, 18, 20, 22, 24, 26, 28 and 30. By constructing a similar three-dimensional variations for these eight nodes (Fig. 7) as for the previous six, observations can be made as to the influence of the circumferential spikes. Nodes 16, 20, 24 and 28 represent nodes close to the spikes. As was the previous case, the nodes close to the spikes exhibit higher temperatures than the in-between nodes; however, temperature gradients in this plane are not as large because none of the nodes is a spiked node. Influence of the spikes is seen to have little effect of temperature uniformity in this plane. Indications are that from this temperature uniformity investigation; four hot spots will occur in the initial phases of heat-up with equilibrium of the surface temperature being rapidly approached.

#### 3.2.3 Specimen Melting

Melting of a glass specimen required not only surface temperature uniformity but also the avoidance of high-temperature gradients from the surface-to-center nodes in order to prevent thermal shock across the specimen. Maximum temperature gradients across the glass specimen will occur from a surface node connected to a platinum spike to the center node of the specimen. Figure 8 was generated using data from the nominal case to investigate this phenomenon. Behavior of the temperatures at the two locations is observed to be similar to the surface nodes; however, greater temperature gradients are present during the initial 1500 sec of heat-up. A maximum temperature difference of  $645^{\circ}$ F occurs after 720 sec of heatup time with the gradient decreasing to zero close to the melting temperature of glass. This heat up rate exceeds the maximum allowable for avoiding thermal shock and must be reduced. Avoidance of thermal shock can possibly be achieved by decreasing the platinum cylinder side wall thickness as discussed previously in relation to temperature uniformity.

Previous discussions of the thermal response of the glass specimen have considered temperature histories, temperature uniformity, and thermal shock with each aspect indirectly considering the heat-up and cool-down rates of the glass specimen. Cool-down rates are of secondary importance in this effort since this phase can be accomplished by methods other than allowing the specimen to cool naturally without outside influence. However, heat-up must be accomplished using a combination of three variables: (1) separation distance between the platinum cylinder and ceramic cylinder in the nominal case; (2) insulation materials and insulation thickness applied to the ceramic cylinder; and (3) heat input (maximum available is assumed to be be 150 W). Of these three variables the heat input was assumed to be a constant 150 W in all of the transient analyses; however, this could be varied from 0 to 150 W if required to obtain the best high-temperature design.

Separation distance of the ceramic and platinum cylinders for the nominal case was varied from 0.3225 in. to 0.0 in. (insulation wrapped around platinum cylinder). Figure 9 shows that the effect of increasing the separation distance with the same insulation material is to decrease the heat-up rate. Increasing the insulation thickness (Fig. 10) for a specific separation distance increases the heat-up rate. Thus, to obtain a low heat-up rate such as  $20^{\circ}$ F/min, a tradeoff must be made between the required insulation and the separation distance. Discussion of these results as applied to the preliminary design of the high-temperature furnace are presented in Section 4.

## Section 4 PRELIMINARY FURNACE DESIGN

#### 4.1 BEST FURNACE DESIGN

By considering the thermal behavior of the glass specimen as influenced by the parametric investigations, a preliminary high-temperature furnace design can be recommended. This design, although satisfactory from a thermal standpoint, should be further investigated to ensure its structural integrity.

Requirements for the furnace design are that it heat the specimen uniformly from ambient  $(70^{\circ}F)$  conditions to melting  $(2600^{\circ}F)$  at a rate of  $18^{\circ}F/min$ , maintain the melting temperature for 10 min, and then cool to  $2373^{\circ}F$  at a rate of  $18^{\circ}F/min$ . From  $2373^{\circ}F$  the specimen must be essentially quenched at a rate of  $109^{\circ}F/min$  to  $1023^{\circ}F$ . Cooling from  $1023^{\circ}F$ must proceed at a rate of  $10.8^{\circ}F/min$  to  $213^{\circ}F$ . At  $213^{\circ}F$  the specimen is in a form that requires no further cooling, thus completing the experiment.

Observations of the temperature histories of Figs. 4 and 5 reveal that none of the insulation materials or thicknesses considered in this analysis are capable of achieving the severe cool-down requirements which must be imposed on the glass specimen. In order to obtain such cooling rates, methods of quenching (which are beyond the scope of this study) must be investigated. Sufficient information is available, however, for determining a thermal design of the furnace to meet the heat-up requirements.

Preliminary design of the furnace in this investigation will assume that the nominal high-temperature furnace configuration of Fig. 1 is acceptable. Specific parameters which affect the thermal performance of the furnace are

the insulation material, insulation material thickness, and separation distance between the ceramic and platinum cylinders.

Investigation of the temperature histories (Fig. 4) of the insulation materials and the heat-up rate of Fig. 10 indicate that Dyna Flex and Dyna Quartz have almost the same thermal performance and will meet the furnace requirements easier than ZYF-100. But, because Dyna Quartz is in semi-rigid block form and is difficult to fabricate, Dyna Flex is recommended as the insulation material for the furnace.

The influence of the thickness of Dyna Flex on the heat-up rate is shown in Fig. 10. Glass center node heat-up rate for the nominal furnace is shown to be  $48^{\circ}$ F/min as compared to the required  $18^{\circ}$ F/min. Projection of the Dyna Flex curve indicates that the required heat-up rate could be achieved with very little or no insulation at all. While this may be possible it is not feasible for the furnace design since the furnace will be housed in a vacuum chamber in the Orbital Workshop. Maximum vacuum chamber wall temperature has been defined as  $140^{\circ}$ F, therefore, a desirable backside temperature of the furnace is one in the neighborhood of  $140^{\circ}$ F. Relationships of insulation thickness and its influence on the backside temperature of the furnace are shown in Fig. 11 for insulation thickness of 0.75, 1.5 and 3.0 in. In each instance the backside of the furnace is radiating to a constant  $70^{\circ}$ F vacuum chamber environment.

Figure 12 shows the effect of insulation thickness on maximum backside temperature of the insulation material. From this figure it can be seen that an insulation thickness of 2.35 in. will provide a maximum backside temperature of  $140^{\circ}$ F for the nominal furnace configuration.

One method for providing the necessary heat-up rate and preventing unnecessary heating of the Workshop is to increase the separation distance between the platinum and ceramic cylinders. For an insulation

thickness of 0.75 in. a separation distance of 0.7 in. would provide the necesary heat-up rate of  $18^{\circ}$ F/min; however, this would not provide the low backside temperatures required for existence in the vacuum chamber. In order to apply the necessary 2.35 in. of insulation to the furnace and still provide the heat-up rate of  $18^{\circ}$ F/min, a separation distance approaching the dimensional limitations of the furnace would be necessary. As a result this approach is not recommended.

A second method toward elimination of the high heat rate, which has not been investigated here, is to: (1) keep the nominal furnace design; (2) apply 2.35 in. of Dyna Flex instead of 0.75 in.; and (3) incorporate a variable power input. A low power input would be used in the initial stages of heatup when conduction is the primary mode of heat transfer, and more power applied as radiation becomes dominant and the melting temperature of the glass is approached. This method of heating the specimen is highly suitable and is recommended for the preliminary design of the furnace.

#### 4.2 SUPPORT INSTRUMENTATION

Actual operation of the high-temperature furnace will require hardware items such as devices for measuring temperatures of glass, heater elements and vent systems. For each of these, methods must be established for incorporating these into the furnace system in a manner which will be as close as possible to the ideal operation of the furnace.

Two of the accepted methods for obtaining temperature measurements are the use of thermocouples and optical or electrical pyrometers. The use of thermocouples in such a furnace as this would be excellent under normal gravity conditions since little concern is given to the existence of a gravity environment. For the space manufacturing effort however, the assurance of a zero-gravity or near zero-gravity condition is of vital importance. The incorporation of a thermocouple into the glass specimen could cause distortion of the glass due to its resistance to any free floating attitude the specimen might tend to assume and could also provide a hot spot since lead-in

wire would be exposed to the radiation and conduction heat transfer nodes of the furnace system.

A method of temperature measurement which would not affect the performance of the glass specimen and still provide accurate temperature measurements of the specimen is that of utilizing an optical pyrometer. Quartz view ports built into the platinum, ceramic, and insulation would allow the measurements to be taken from outside the vacuum chamber through proper positioning of the furnace. At present, measurement by optical pyrometry is highly advisable.

Methods considered for heating the furnace consist of tungsten wire and possibly a tungsten mesh to provide a more uniform heat source. Investigations revealed that tungsten mesh would be highly suitable but requires more power than is available. Therefore, a tungsten wire installed on the ceramic cylinder to provide uniform heating to the specimen is required.

As the temperature within the furnace is increased and the specimen begins to melt, venting is sometimes required to allow gases from the glass to escape the furnace. Consultation with Dr. Deeg revealed that the glass specimen for the space manufacturing effort will require no venting. Therefore, the only venting, if any, would be for allowing air to escape the space between the cylinders and the insulation material.

## Section 5 CONCLUSION AND RECOMMENDATIONS

#### 5.1 CONCLUSION

Thermal analyses of the furnace have resulted in the selection of a furnace configuration based on the nominal furnace of Fig. 1. The preliminary furnace design that has resulted consists of the basic dimension, components and configuration of the nominal furnace (Fig. 1) except the Dyna Flex insulation material thickness has been increased from 0.75 in. to 2.35 in. and the constant power input of 150 W has been changed to a variable power input of from 0 to 150 W. Additional analyses consisting of methods for obtaining rapid cooling must be performed, however, before the system will meet all of the thermal requirements for the glass-melting experiments.

#### 5.2 RECOMMENDATIONS

Current design of the high-temperature space manufacturing furnace is for the glass-melting experiment only. As a result, furnaces to accommodate additional Orbital Workshop experiments must be developed causing increased expenditures for development and unnecessary storage and weight penalities for orbiting the Workshop. A reduction for these undesirable increases can be achieved by directing future furnace modifications toward developing a versatile tool which can be utilized on several experiments.

Planned experiments that will require similar furnace designs to the glass-melting experiment are composite casting and foam-metal experiments. Minor modifications to the present preliminary furnace design would have to be performed to accomplish the desired heat-up rate. These modifications would consist primarily of changing the temperature limit of the furnace from  $2600^{\circ}$ F to  $3000^{\circ}$ F and changing the platinum crucible to contain new experiments.

Since quenching methods must be developed to meet the glass cooldown requirements, the incorporation of quenching techniques as required for other experiments should be considered also. Evaluation of methods for quenching a specific specimen should be based on the effect of location of the quenching fluid relative to the specimen, means of injecting the fluid, and loading induced on the specimen due to fluid contact.

Future analyses should also consider the effect of incorporating an atmosphere into the furnace since some experiments require such an environment. Additional factors to be considered are methods by which the specimen is to be enclosed within the furnace, critical furnace materials which might influence the behavior of the furnace or specimen as atmospheres are required for the experiments, types of controls necessary for performing these experiments, and the availability of these controls as flight-qualified instruments.

Incorporation of these recommendations into the furnace design could easily be accomplished at this stage of the high-temperature furnace design. These changes would result in a versatile tool for the space manufacturing effort for experimentation purposes. As new and additional experiments are defined, further modifications could be made to accommodate their required environments.

#### REFERENCES

- Glaser, Peter E. et al., "Investigations of Materials for Vacuum Insulation up to 4000°F," ASD TDR-62-88, Wright-Patterson Air Force Base, Ohio, January 1962.
- 2. Wechsler, Alfred E., and Peter E. Glaser, "Investigation of the Thermal Properties of High-Temperature Insulation Materials," ASD-TDR-63-574, Wright-Patterson Air Force Base, Ohio, July 1963.
- 3. Wechsler, Alfred E. et al., "Investigation and Development of High-Temperature Insulation Systems," AFML-TR-65-138, Wright-Patterson Air Force Base, Ohio, June 1965.
- 4. Pike, J. N. and I. R. Todd, "Fibrous Zirconia for High-Temperature Heat Shielding and Thermal Insulation," Report No. UCRI-507, Union Carbide Research Institute, Tarrytown, N.Y., August 1968.
- 5. Ryan, J. M. et al., "Lightweight Thermal Protection System Development, Vols. I and II," Technical Report AFML-TR-65-26, Wright-Patterson Air Force Base, Ohio, January 1965.
- DeWitt, W. D. et al., "Final Report of Research and Development In a Thermal Insulation Study - Phase I," AEC R&D Report ALO 3632-20, Oak Ridge, Tenn., 14 November 1967.
- Lovin, J. K. and A. W. Lubkowitz, "User's Manual for 'RAVFAC,' A Radiation View Factor Digital Computer Program," LMSC/HREC D148620, Lockheed Missiles & Space Company, Huntsville, Ala., November 1969.
- 8. Bible, A. E. et al., "Thermal Analyzer Control System for IBM 709-7090-7094 Computer Engineering Utilization Manual," Report 3-56-65-8, Lockheed Missiles & Space Company, Sunnyvale, Calif., September 1965.
- 9. Rohsenow, Warren M. and Harry Choi, <u>Heat, Mass and Momentum Trans</u>fer, Prentice-Hall, Inc., Englewood Cliffs, N.J., 1961.
- 10. Wiebelt, J. A., <u>Engineering Radiation Heat Transfer</u>, Holt, Rinehart and Winston, Inc., New York, 1966.
- 11. Goldsmith, Alexander et al., <u>Handbook of Thermophysical Properties of</u> Solid Materials, Vol. 1-5, Macmillian, New York, 1961.

4 1

Appendix A

## MATERIALS ANALYSES

#### CASE I: GRAPHITE FIBERS, TANTALUM SHIELDS AND ADL-17 POWDER

Tests that were reported in Ref. A.1 showed that the combination of graphite fibers, tantalum shields and ADL-17 powder would be suitable for the furnace application from a standpoint of low thermal conductivity. From personal contacts about the material combination, however, it was found that this material must be fabricated independently (i.e., the graphite, tantalum, and powder must be purchased and the materials attached to the furnace in the desired proportions by the agency desiring the specific material). Another point that was brought out was that tantalum is not compatible with graphite at high temperatures.

Calculations for the application of this material on a 2 in. radius furnace indicate that 0.689 in. of insulation would be required, which would result in a weight of insulation ( $\rho = 8.8 \text{ lb/ft}^3$ ) of 0.206 lb.

The conclusion is reached, therefore, that although the graphite-tantalum shield-ADL-17 powder is quite good from a thermal performance standpoint, its chemical and physical characteristics as well as degree of difficulty for fabrication make this material combination undesirable.

#### CASE II: GRAPHITE FIBERS AND TANTALUM SHIELDS

Insulation properties of a graphite fibers and tantalum shield insulation system are about the same as those of the Case I system (thickness = 0.986 in. and weight = 0.332 lb). Incompatibility of tantalum and graphite as well as difficulty in fabrication make the system undesirable.

#### CASE III: COMBINATION OF DYNA FLEX, FLEXIBLE HIGH TEMPERATURE MIN-K, AND FLEXIBLE STANDARD MIN-K

The combination of Dyna Flex, flexible high-temperature Min-K, and flexible standard Min-K was chosen because the materials are three excellent insulators within their temperature range. By using materials which had outstanding performance within their temperature range, an optimum material would be expected to result, which would have a low density and require only a small volume to perform the required task. It was shown that for a 2-in. radius cylinder an insulation thickness of 0.725 in. would be required.

An insulation system of this combination is definitely in contention for use on the furnace. The only limiting factor is the difficulty in assembling the three materials.

#### CASE IV: DYNA FLEX

Dyna Flex, a refractory fiber felt formed from alumina-silica-chromia fibers, has a melting point well over  $3000^{\circ}$ F and the ability to retain a soft fibrous structure at  $2700^{\circ}$ F. Shrinkage rates are very low compared to other similar insulators, such as refractory fibers and silica fibers. Dyna Flex was found to shrink 3.7% over a 4-hr soak time at  $2700^{\circ}$ F while the other fibers showed considerable shrinkage. Thermal conductivity of Dyna Flex at a mean temperature of  $1300^{\circ}$ F and  $10^{-4}$  torr is approximately 0.03 Btu/ft-hr- $^{\circ}$ F for a 24 lb/ft<sup>3</sup> density specimen. Calculations for the 2-in. radius furnace at  $2500^{\circ}$ F using Dyna Flex alone as the insulator show that 0.70 in. (thickness = 0.70) of insulation is required, resulting in a weight of 0.288 lb of insulation. Dyna Flex is a low-cost material:  $$1.20/ft^2$  for a  $\rho = 6$  lb/ft<sup>3</sup> and 1/2-in. thick x 42 in wide x 48 in or 96 in. long.

A lower thermal conductivity can possibly be obtained through the application of Dyna Flex with radiation shields. A tantalum radiation shield would definitely not be suitable, since tantalum reacts with silicon, aluminum, nickel, chromium and iron. However, the use of another refractory material such as molybdenum columbium (niobium) could very well be in order. Before going to such a system the benefits to be gained should be weighed against the extra cost of fabrication, time spent in assuring that the metal will not react with the insulation, and the possibility that the metal might not have proper contact with the insulation and therefore result in higher conductivities.

#### CASE V: DYNA QUARTZ

From documents available on conducted tests of Dyna Quartz, this material has been found to perform quite well at elevated temperatures at high vacuum environment, making it another excellent material to be considered as a possible furnace insulator.

Dyna Quartz is composed of 99% pure silica fibers formed into heatstabilized, semi-rigid blocks or tiles. It displays exceptional stability at soaking temperatures of  $2750^{\circ}$ F and up to  $3000^{\circ}$ F for transient exposure. Shrinkage is also very low: less than 1% for a 24-hr soak at  $2750^{\circ}$ F. Thermal conductivity of Dyna Quartz at a mean temperature of  $1300^{\circ}$ F and  $10^{-4}$ torr is approximately 0.0229 Btu/ft-hr- $^{\circ}$ F for a 24 lb/ft<sup>3</sup> density material. Calculations for Dyna Quartz in the required environment and for a 2.0 in. radius cylinder indicate that 0.508 in. (thickness=0.508) if insulation is required, resulting in a 0.40 lb weight of the insulation system. Cost of Dyna Quartz is \$35.40 for a 10 lb/ft<sup>3</sup> density block 12 in. x 14 in. x 1 in. Cost would be higher for a 24 lb/ft<sup>3</sup> density block.

Probably the only disadvantage to using Dyna Quartz in the furnace application is its semi-rigid form. Since a small diameter (4 in.) cylinder is being used, considerable difficulity would be experienced in applying the

insulation around the cylinder; however, since the insulation required is  $\approx$  0.5 in. making the diameter of the cylinder  $\approx$  5 in., the cylinder could be fitted between two 3-in. blocks with a 2-in. radius cylinder cut out of the blocks. If the length of the cylinder was decreased from 4 in. to 3 in., a cylinder 3 in. long and 2 in. radius could be cut from a block 3 in. x 14 in. x 12 in. A third way of using this insulation (and probably the best) would be to attach two blocks together to form a 6-in. or 5-in. thick block, then cut a cylindrical hole 4.5 in. deep and with a 2 in. radius into the block. This would allow for a 0.5-in. end insulation on one end and a 0.5-in. plug on the other. It might also be possible to get Johns-Manville<sup>\*</sup> to produce a thicker block than 3 in.

#### CASE VI: ZIRCONIA

Information on zirconia was available from Refs. A.2 and A.3. Ref. A.3 had tested the material zirconia A, C, E and zircon and purchased it from Hitco<sup>\*\*</sup> while the second source was from data sent from Union Carbide Corporation of New York on their ZIRCAR types ZYF-100 and 200.

#### H. I. Thompson

During the testing period, the H. I. Thompson product displayed a relatively low apparent thermal conductivity (about the same as Dyna Quartz). Zirconia fibers darkened and experienced considerable shrinkage, but the fibers were relatively inert chemically with metallic components. Zirconia insulation have low conductivity, but are limited in high temperature (vacuumapplications by their stability.

\*\* formerly H. I. Thompson Fiber Glass Co., Gardena, Calif.

<sup>&</sup>lt;sup>\*</sup>Johns-Manville Corp., New York

#### Union Carbide

From Ref. A.3 it was found that zirconia ZYF-100 has very good thermal conductivity (about the same as Dyna Quartz  $\approx 0.0333$  Btu/ft-hr-<sup>o</sup>F) in a vacuum at a mean temperature of  $1300^{\circ}$ F. Zirconia is stabilized in the tetragonal form by the addition of yttrium oxide (yttria). Since these materials are stabilized in the tetragonal form over the entire temperature range, mechanical failure (loss of fibrous form) due to phase transformation or thermal shock is minimized. On exposure to high temperature, zirconia cloth and felt materials exhibit excellent dimensional stability in the radial directions since shrinkage normally associated with high temperature softening and sintering results only in the reduction of the thickness of the material.

In the absence of oxygen, zirconia does not react appreciably with refractory metals; however, minor reaction is reported with tantalum. At temperatures up to  $4030^{\circ}$ F ZYW-30 cloth shows no reaction with either tungsten or molybdenum, but it does show a minor amount of reaction with tantalum.

It is possible that the H. I. Thompson zirconia does not incorporate the yttria in its material as does the Union Carbide zirconia; therefore, further investigation should be made into the obvious discrepancy between the two reports.

Zirconia seems to have approximately the same thermal properties as Dyna Quartz. However, it is definitely known that shrinkage of Dyna Quartz is small (< 1%).

Application of zirconia with radiation shields of molybedenum (based on tests of H. I. Thompson that state that zirconia is relatively inert with

metallic components) might very well be an excellent system. According to a Union Carbide Project Plan for January 1969 to October 1969 (Ref. A.4), this type of system will be tested in the temperature range of 2500°F.

#### CASE VII: ASTROQUARTZ

Manufactured in New York, Astroquartz is a 99.99% quartz-fiber material available in mats and fabric form. Although it has a low density  $(3 \text{ lb/ft}^3)$  and good melt temperature (T =  $3000^{\circ}$ F), its high thermal conductivity values (0.108 and 0.175 Btu/ft-hr- $^{\circ}$ F at 1200 and 1600 $^{\circ}$ F, respectively) make it an undesirable insulator.

The possibility of using Astroquartz and radiation shields should not be ruled out except for the case of usage with tantalum radiation shields. Again the factor of reaction is the limiting case for such an application and should be considered carefully.

Calculations for Astroquartz on a 2 in. radius cylinder indicate that thickness = 4.88 in. and weight = 0.432 lb.

#### CASE VIII: SAPPHIRE WOOL

Contact with George Cunnington of Lockheed/Sunnyvale indicates that Sapphire wool has a low density and relatively high thermal conductivity. Very little additional information was available.

Calculations for a 2 in. radius cylinder indicate that thickness = 5.14 in. and weight = 0.1645 lb.

<sup>&</sup>lt;sup>\*</sup>by J. P. Stevens & Co., Inc.

## CASE IX: DYNA QUARTZ AND TANTALUM RADIATION SHIELDS

Alfred Wechsler of Arthur D. Little, Inc., (Ref. A.2) under an Air Force contract, experimented with Dyna Quartz and tantalum foils as radiation shields. The report states that data from the test were difficult to interpret due to the following occurrences:

- 1. Reflectance of the radiation barrier degraded during the test and the specimen tended to warp and twist as heat was applied. Specimen warping both distorted the heat flux pattern and decreased the significance and accuracy of the thickness measurements. Distortion was particularly troublesome with this type of sample.
- 2. Temperature range under consideration was higher than the probable range for furnace application and therefore little information was available for comparison with other systems.

The main item to note here is that Dyna Quartz tended to warp when it was combined with the radiation shield.

CASE X: SAPPHIRE WOOL PAPER WITH STAINLESS STEEL FLAKES

The sapphire wool specimen of Ref. A.2 consisted of two layers of their sapphire paper similar to that used in previous tests and a separating layer of stainless steel flakes, or "splat."

Sapphire wool and "splat" was shown to be very stable. It was also noted that sapphire wool did not react with tantalum. A "splat" radiation barrier could easily be incorporated into insulating batting without greatly increasing its density (it can also be made from materials such as refractory or inert metals like platinum).

Thermal conductivities range from  $\approx 0.025$  to 0.0416 Btu/ft-hr-<sup>o</sup>F for a mean temperature range of 1300<sup>o</sup> to 2500<sup>o</sup>F.

#### CASE XI: REFRASIL (TYPE A-100, B-100, IRISH B-100)

The apparent measured conductivity values of the three Refrasil materials (Ref. A.2) were considerably above the three determined for other silica-based materials possibly due to the lower contact resistance during the test.

#### CASE XII: GRAPHITE FIBER FELT

Thermal conductivity of graphite fiber felt, as shown in the test of Ref. A.2, increases from 0.025 to 0.075 Btu/ft-hr- $^{\circ}F$  over a mean temperature range of 650 $^{\circ}$  to 1400 $^{\circ}F$  and  $\approx$  0.082 Btu/ft-hr- $^{\circ}F$  at a hot-wall temperature of 2500 $^{\circ}F$ .

Testing revealed a slight shrinkage and discoloration but no apparent change in the graphite fibers. Use of this material with radiation shields of tantalum is not recommended since tantalum becomes severely embrittled due to carbide formation. Other refractories might be possible such as molybdenum or columbium (niobium); however, no experimental data are currently available.

#### CASE XIII: GRAPHITE CLOTH AND PYROLYTIC GRAPHITE FOAM

Tests of these two materials in Ref. A.2 yielded inaccuracies due to:

- 1. Considerable contact resistance in the case of graphite cloth inaccurate temperature readings and thereby low thermal conductivities.
- 2. Apparent reactions and difficulty in sample preparation.

In both cases, as in the case of graphite felt, reactions with tantalum indicated that the use of this material as a radiation shield would not be wise.

#### REFERENCES

- A.1 Glaser, Peter E. et al., "Investigations of Materials for Vacuum Insulation up to 4000°F," ASD TDR-62-88, Wright-Patterson Air Force Base, Ohio, January 1962.
- A.2 Wechsler, Alfred E. et al., "Investigation and Development of High-Temperature Insulation Systems," AFML-TR-65-138, Wright-Patterson Air Force Base, Ohio, June 1965.
- A.3 Pike, J. N. and I. R. Todd, "Fibrous Zirconia for High-Temperature Heat Shielding and Thermal Insulation," Report No. UCRI-507, Union Carbide Research Institute, Tarrytown, N.Y., August 1968.
- A.4 DeWitt, W. D. et al., "Final Report of Research and Development In a Thermal Insulation Study - Phase I," AEC R&D Report ALO 3632-20, Oak Ridge, Tenn., 14 November 1967.

4.1

Appendix B

## TABLES

Material	Max. Applicable Temperature <sup>(0</sup> F)	The rmal Conductivity (Btu/ft-hr- <sup>0</sup> F) Vacuum Environment at Tmean = 1300 <sup>0</sup> F	Density (lb/ft <sup>3</sup> )	Composition	Available Form	Source	Shrinkage	Chemical Stability	Potential for Combination with Radiation Shields	Dimensional Stability	Ove rall Suitability
Graphite libers with tantalum shields and ADL-17 powder	2700	0.0125 at 2000 <sup>0</sup> F 0.033 at 2700 <sup>0</sup> F	8.8	See individual materials	Separate forms of tantalum, ADL-17 & graphite fiber mat	Must be fabricated	Undetermined	Tantalum reaction with graphite	N. <b>A</b> .	Undete rmined	Incompatible
Graphite fibers and Lantalum shield	3500	0.02 at 2000 <sup>0</sup> F 0.04 at 3000 <sup>0</sup> F	9.5	See individual materials	Separate forms of tantalum shields & graphite fiber mat	Must be fabricated	Undetermined	Tantalum reaction with graphite	N. A.	Undetermined	Incompatible
Combinations of: Dyna Flex Flexible High-Temp, Min-K Flexible Standard Min-K	2700	Undetermined	≈ 20.0	See individual materials	Separate forms of Dyna Flex & Min-K	Must be fabricated	Undete rmine d	Undetermined	Undete rmined	Undetermined	Excellent
Dyna Flex	2700	0.0304	3.0+24.0	Alumina-silica- chromia fibers	Flexible mat	Purchased from Johns-Manville	3.5% at 2600 <sup>0</sup> F 6.0% at 2700 <sup>0</sup> F	Tantalum reaction	Excellent except with tantalum	Low shrinkage	Excellent
Dyna Quartz	2750	0.0229	6.2-12.0	99% Si O <sub>2</sub>	Semi-rigid block	Purchased from Johns-Manville	Less than 1% after 24 hr soak at 2750 <sup>0</sup> F	Tantalum reaction	Difficulty in fabricating specimen	Excellent	Excellent
Zi rconia	3300	0.0333	14.0	Zr02, Y <sub>2</sub> 03	Felt	Purchased from Union Carbide	2.0% radial shrinkage after 16 hrs at 2800 <sup>0</sup> F	with 5102 Stabilized by yttria	No reaction with W or Mo; minor with Ta	Excel. stability (Union Carbide)	Excellent
Ast roque rtz	Undete rmined	0.125	3.0	99.99% Si O <sub>2</sub>	Mat	J. P. Stephens & Co., Inc.	Undete rmined	Undete rmined	Not with Ta; other refrac- tories might be good	Undete rmined	The rmak conductivity too high
Sapphire Wool	3700	0.129	1.0	99.5% A1 <sub>2</sub> O <sub>3</sub>	Mat	Thermo Kinetic Fibers, Inc.	Undetermined	Undete rmined	Excellent	<b>Undete rmined</b>	Thermal conductivity too high
Dyna Quarte and tantalum radiation shields	3300	≈ 0.03	Undeter- mined	See individual materials	Separate forms of tantalum & Dyna Quartz	Must be fabricated by Lockheed	Undetermined	Undete rmined	Tends to warp	Undetermined	Fabrication & com- patibility difficulties
Sapphire Wool paper and stainless steel flakes	3360	æ 0.025	Undeter - mined	See individual materials	Separate forms of Sapphire Wool & Stainless Steel flakes	Flakes: Nuclear Metals, Inc. Sapphire Wool: Ther- mo Kinetic Fibers, Inc.	Undete rmined .	Undete rmined	Small evidence of reaction with stainless steel	Very good	Good
Refraeil (Type A-100, B-100, and Irish B-100)	2000 -+ 2500	0.03 - 0.066	≈ 3.0	99% SiO <sub>2</sub>	Felt	H. I. Thompson	Limited shrinkage at temps. up to 2800 <sup>0</sup> F	Undetermined	Undetermined	<b>Undete r</b> mined	Fair to good
Graphite fiber felt	3200 <sup>0</sup> F	0.083	5.0	Pure graphite	Felt	National Carbon Co.	Slight shrinkage	Discoloration	Carbon formation causes Ta		The second s
Graphite cloth and pyro- lytic graphite foam	3200 +	0.04	4.0	Pure graphite	F. oa F	High Temp. Materials, Inc.	Undetermined	Stable at temps. up to 3000 <sup>0</sup> F	to become embrittled Undetermined	Undete rmined Undete rmined	Instrmal conductivity too high Difficult to prepare for small applications
Cost: Dyna Flex \$1.2 Dyna Quartz \$27 \$35. Zirconia \$50.	20/ft <sup>2</sup> for 6 lb/ft <sup>3</sup> -20 for 12 in. x l -40 for 12 in. x l	, ½ in.thick 4 in. x l in. block, 6.2 4 in. x l in. block, 10	1b/ft <sup>3</sup> 1b/ft <sup>3</sup>							Table I	

Table I INSULATION MATERIAL SUMMARY

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Zirconia

#### Table 2

## INSULATION SYSTEMS CALCULATION RESULTS

#### SOLID INSULATION SYSTEMS

Material	Insulation Thickness	
Dyna Flex	0.700	
Dyna Quartz	0.508	
Zirconia	1.86	
Astroquartz	4.88	
Sapphire wool	5.14	

COMPOSITE INSULATION SYSTEM COMPOSED OF THREE MATERIALS

Material Number	Temp. Range ( <sup>0</sup> F)	Component Thickness
l. Dyna Flex 2. Flexible high	<b>2500</b> → 1500	0.535
temperature Min-K 3. Flexible standard	1500 -> 500	0.1555
Min-K	500 <b>→</b> 70	0.035
Total Comp	osite Thickness	0.725 in.

#### MULTIPLE INSULATION SYSTEMS WITH RADIATION SHIELDS

System	Insulation Thickness
Graphite fibers, tantalum shields, and ADL-17 powder	0.689
Graphite fibers, tantalum shields	0.968

Case/Mod	Insulation Material	Insulation Thickness (in.)	Separation Distance* (in.)	Geometry of Platinum Crucible
1/0	Dyna Flex	0.75	0.3225	Cylinder
1/1	Dyna Flex	3.0	0.3225	Cylinder
1/2	Dyna Flex	1.5	0.3225	Cylinder
1/3	Dyna Quartz	0.75	0.3225	Cylinder
1/4	Dyna Quartz	3.0	0.3225	Cylinder
1/5	Dyna Quartz	1.5	0.3225	Cylinder
1/6	ZYF-100	0.75	0.3225	Cylinder
1/7	ZYF-100	3.0	0.3225	Cylinder
1/8	ZYF-100	1.5	0.3225	Cylinder
2/	Dyna Flex	0.75	0.0	Cylinder

# Table 3 PARAMETRIC INVESTIGATIONS

\* between ceramic cylinder and crucible

Table 4

## NODAL EXPLANATION OF NOMINAL HIGH-TEMPERATURE FURNACE

$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Config.	Node No.	Represents Node Numbers	Connects to Radiation Resistor	Connects to Conduction Resistor	Dummy Node
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		1	1		x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	2		x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		3	3		x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Glass	224	224 + 231		x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Cylinder	232	232 + 239		x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		6	6		x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		7	7		x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		8	8 <del>&gt;</del> 15	x	x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		16, 17	16 + 31	x	x	x
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		32	<u>32</u> → 35		x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		36	36 + 43	x	x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		44	44	x	x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		46	46, 48, 50, 52	x	x	
Spikes $45$ $45$ $45$ $x$ $x$ $47$ $47, 49, 51, 53$ $x$ $x$ $x$ $55$ $55$ $x$ $x$ $x$ $56$ $56 + 63$ $x$ $x$ $x$ $64, 65$ $64 + 79$ $x$ $x$ $80$ $80 + 87$ $x$ $x$ $Cylinder$ $88, 91$ $88, 91; 94, 97; 100, 103; 106, 109$ $x$ $x$ Ceramic $89, 92$ $89, 92; 95, 98; 101, 104; 107, 110$ $x$ $x$ Spikes $90, 93$ $90, 93; 96, 99; 102, 105; 108, 111$ $x$ $x$ Heater $112$ $112 + 119$ $x$ $x$ $x$ $120, 121$ $120 + 135$ $x$ $x$ $x$ $136$ $136 + 143$ $x$ $x$ $x$	Platinum	54	54	x	x	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Spikes	45	45		x	x
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		47	47, 49, 51, 53		x	×
Platinum Cylinder $56$ $64, 65$ $80$ $80$ $80, 91$ $56 + 63$ $64 + 79$ $80$ $80 + 87$ $88, 91$ xCeramic Spikes $89, 92$ $90, 93$ $88, 91; 94, 97; 100, 103; 106, 109$ $90, 93; 96, 99; 102, 105; 108, 111$ xxHeater Element $112$ $136$ $112 + 119$ $136 + 143$ xxx	l <u></u>	55	55		x	x
Platinum Cylinder $64, 65$ $80$ $64 + 79$ $80$ $80 + 87$ xCylinder $80$ $88, 91$ $80 + 87$ $88, 91; 94, 97; 100, 103; 106, 109$ xCeramic Spikes $89, 92$ $90, 93$ $89, 92; 95, 98; 101, 104; 107, 110$ $90, 93; 96, 99; 102, 105; 108, 111$ xxHeater Element $112$ $136$ $112 + 119$ $136 + 143$ xx		56	56 + 63	x	,	
Cylinder80 $80 + 87$ x88, 9188, 91;94, 97;100, 103;106, 109xxCeramic89, 9289, 92;95, 98;101, 104;107, 110xxSpikes90, 9390, 93;96, 99;102, 105;108, 111xxHeater112112 + 119xxLement120, 121120 + 135xx136136 + 143136 + 143xx	Platinum	64,65	64 + 79	x		
88, 91 $88, 91; 94, 97; 100, 103; 106, 109$ x       x         Ceramic $89, 92$ $89, 92; 95, 98; 101, 104; 107, 110$ x       x         Spikes $90, 93$ $90, 93; 96, 99; 102, 105; 108, 111$ x       x       x         Heater $112$ $112 + 119$ x       x       x         Heater $120, 121$ $120 + 135$ x       x       x         Heater $136$ $136 + 143$ x       x       x	Cylinder	80	$80 \div 87$	x		
Ceramic $89, 92$ $89, 92; 95, 98; 101, 104; 107, 110$ x         x         x           Spikes $90, 93$ $90, 93; 96, 99; 102, 105; 108, 111$ x         x         x           Heater $112$ $112 + 119$ x         x         x           Heater $120, 121$ $120 + 135$ x         x         x $136$ $136 + 143$ $241 - 248$ $x = 120$ $x = 120$ $x = 120$		88,91	88, 91;94, 97;100, 103;106, 109		X	x
Spikes         90, 93         90, 93; 96, 99; 102, 105; 108, 111         x         x         x           Heater         112         112 + 119         x         x         x         x           Element         136         136 + 143         x         x         x         x	Ceramic	89,92	89, 92; 95, 98; 101, 104; 107, 110	x	x	
Heater $112$ $112 + 119$ $x$ $x$ $x$ $x$ Element $120, 121$ $120 + 135$ $x$ $x$ $x$ $x$ $x$ $136$ $136 + 143$ $241 - 248$ $241 - 248$ $x$ $x$ $x$ $x$	Spikes	90,93	90, 93; 96, 99; 102, 105; 108, 111		X	X
Element         120, 121         120 + 135 $x$ $x$ $x$ $x$ 136         136 + 143         241         248 $x$	Heater	112	112 + 119	x	x	x
	Element	120, 121	120 + 155	x	x	x
		130	130 + 143			
Ceramic $240,250$ $240,264$ x	Ceramic	240 250	241 + 240	1 1 1	x	
Cylinder 265 265 272	Cylinder	249,250			X	
		144			×	<u>}</u>
$152$ $152 \times 150$ $x$ $y$		152			A V	
$160 161$ $160 \pm 175$ x		160 161	$160 \rightarrow 175$			
Insulation $176 177$ $176 - 191$	Insulation	176 177	176 - 191		x	v
192 192 199 x	AIN WELLINII	192	192 - 199		x	A
200 200 + 207 × ×		200	200 + 207		x	x
208,209 208 + 215 x x		208.209	208 - 215	1	x	x
216.217 216 + 223 x x		216.217	216 + 223		x	x
240 240 Outer Space Node	<u></u>	240	240	Οι	iter Space No	de
Temperature = $70^{\circ}$ F				Te	emperature =	70 <sup>0</sup> F

. . . . .

Material	Density (1b/ft <sup>3</sup> )	Specific Heat (Btu/lb- <sup>0</sup> R)	Emissivity	Thermal Conductivity (Btu/ft-sec- <sup>0</sup> F)
Platinum	1340.0	0.037	0.18	1.038 (10) <sup>-2</sup>
Ceramic	230.0	0.28	0.50	8.25 (10) <sup>-4</sup>
Dyna Flex	24.0	0.27	0.85	Temp. Dependent
Dyna Quartz	12.0	0.29	0.85	Temp. Dependent
ZYF-100	14.0	0.20	0.85	Temp. Dependent

# Table 5PROPERTIES OF MATERIALS USED IN FURNACE DESIGN

Appendix C FIGURES



Fig. 1 - Initial High-Temperature Furnace Configuration

C-1





C-2



Fig. 3 - Thermal Conductivity of Glass Specimen as a Function of Temperature







C-4



Fig. 5 - Effect of Insulation Material on Temperature History of Glass Center Node

**C-**5











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Fig. 9 - Separation Distance of Platinum Cylinder and Ceramic Cylinder with Heater Element

C-9



Fig. 10 - Effect of Insulation Material on Heat-Up Rate of Glass Center Node

#### C-10



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