VACUUM INSULATION USING PERLITE POWDER SEALED IN PLASTIC AND GLASS

by

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Abstract

Because of the environmentally-destructive nature of foam blown with CFC's, the development of alternative insulations is now being emphasized. One of the more promising alternative insulations is the evacuated enclosure. Development of such an insulation could possibly lead to an insulation with superior insulating properties than CFC foams at a comparable cost.

A type of evacuated-enclosure insulation which has received a great deal of emphasis is packed powder sealed in a vacuum and wrapped in a material which is impermeable to air. The powder provides support for the barrier material against the pressure difference while the barrier material is of sufficiently low gas permeability in order to maintain the vacuum for a suitably long period of time (approximately 20 years). The difficulties encountered with such a design is the need to support the vacuum for sufficiently long periods of time while keeping heat conduction through the barrier material low. Most industrial research has been focused on using foils or high barrier plastics to surround the evacuated powder. However, high barrier plastics which support a vacuum for a practical length of time are expensive to produce while the secondary heat losses of a foil-covered packet are significant. Glass is a viable alternative for a barrier material since it is very cheap to produce, has a very low gas permeability rate, and has a low thermal conductivity. Therefore, secondary heat losses can be kept in check if sufficiently thin layers are used.

Since wrapping a layer of glass around powder results in heating the powder to such a temperature that its insulating characteristics are damaged, an alternative technique was tried. The powder was first wrapped in a plastic envelope, vacuum-sealed, and then sealed once again in a preformed glass shell. Packets of approximately 3 1/2 X 3 1/2 X 1/2 inches were made. Results of thermal tests of the plastic packets alone showed K values of .086-.25 Btu/hr.ft.°F, compared with a .10-.12 K value for CFC blown foam. Tests of two plastic packets wrapped in a layer of glass gave a K-value of .18 Btu/hr.ft.°F. This is worse than the theoretically-predicted results; therefore, further research should be focused on improving the packet, such as baking the powder to remove moisture, creating a higher vacuum more efficiently, and achieving a closer

fit between the plastic and glass barriers.

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Title: Professor

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Chapter 1

Introduction

Because of the damage that foam blown with chloroflourocarbons does to the environment, alternative forms of insulation are now being sought. One of these alternatives is the evacuated enclosure. This type of insulation, once it is developed, may possibly have insulating characteristics which exceed those of CFC blown foam and do this at a comparable cost.

Research into insulation based on the evacuated enclosure began in the 1950's when Whirlpool patented the use of parallel walls filled with an evacuated material, connected by U-shaped metal strips, and hermetically sealed.[1][2] Following that, Union Carbide patented the use of an inner vessel surrounded by an outer shell, with the space between the shell and vessel evacuated and supported by brace members of low thermal conductivity.[3] In 1964, General Electric patented a hermetically sealed panel with filaments between its two walls for support and having an internal pressure of less than 100 microns of mercury.[4] Twenty years later, Young et al. patented an evacuated panel having walls made from a metal and plastic laminate and its edges sealed with a gas permeation barrier.[1]

The difficulties encountered with the evacuated enclosure is the need to support a vacuum for sufficiently long periods of time (15-20 years) while keeping the insulation's thermal conductivity sufficiently low. One design for evacuated-enclosure insulation which has been heavily studied is the use of powders which are sealed in a vacuum and surrounded by a material which has a low gas permeability (see Figure 1-1).
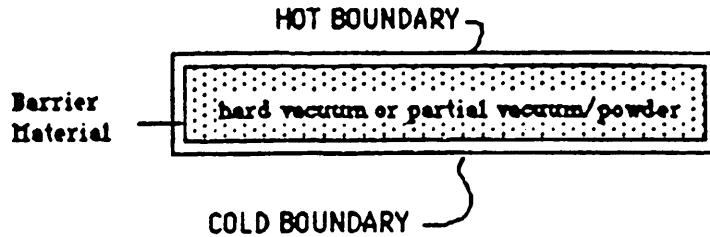
Figure 1-1: Schematic of a vacuum panel

The powder is rigid enough to support the pressure difference, allowing the barrier material to be flexible. The powder has a low thermal conductivity because of the high contact resistance of its particles. The powder also acts to attenuate heat transfer by radiation. The problem associated with this powder packet design is creating an encapsulating material which has both a very low gas permeability, a low thermal conductivity, and can be produced at a sufficiently low cost as to make it commercially viable.

The focus of the research discussed herein is the development of an insulating packet in which powder is evacuated and surrounded by both a layer of plastic and a layer of glass. Earlier attempts at creating an evacuated-insulation packet placed the powder in a vacuum oven between two sheets of glass. The glass and powder were then heated until the top glass sheet "slumped" over the powder and sealed itself to the bottom glass sheet. However, heating the powder destroyed its insulating ability. In order to avoid heating the powder to such a degree, the powder is evacuated, sealed in a layer of plastic, and then sealed in a preformed glass shell. Glass is used because it is cheap to produce, has a very low gas permeability, and has a low thermal conductivity. By using sufficiently thin layers of glass (.01 inches thick), secondary heat effects can be reduced sufficiently.

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Chapter 1 presents more information on the importance of developing evacuated insulations as an alternative to CFC blown foams. Chapter 2 takes a look at the physical principles involved using evacuated powders, the design criteria used in creating the packets, and the different materials that have been looked into for boundary materials. Chapter 3 gives a detailed analysis of the permeability of glass compared to high barrier films and presents a heat transfer model for a packet along with the predicted thermal conduction values for the packet. Chapter 4 goes through the equipment and procedure used to produce the prototype packets. Chapter 5 presents the results of testing the packets along with a discussion of possible improvements to be made in producing the packets. Finally, chapter 6 discusses the economic and manufacturing considerations that need to be looked at in order to produce glass-and-plastic-encapsulated powder packets commercially.

1.1 Need For Development of Evacuated Powder Insulation

Foam which contains pockets of chloroflourocarbon vapor is among the best insulation used today in appliances and for insulating buildings. However, the chloroflourocarbon vapors that give the foam such excellent insulating properties also escape into the atmosphere and cause damage to the earth's ozone layer. In an effort to curb the global production of chloroflourocarbons, the U.S. along with other developed nations have signed the Montreal Protocol, agreeing to sharply decrease CFC-11, CFC-12, CFC-113, and CFC-115 over the next ten years. Since approximately one sixth of the world's production of chloroflourocarbons is used in foam insualtors, development of an alternative insulation could make quite a difference to the health of our environment.

Foams produced with other blowing agents have resulted in foams with insulating characteristics below those of CFC-blown foam. Table 1 shows the effects other blowing agents have on the thermal conductivity of closed cell foams, as predicted by a theoretical model developed by Glicksman.[5] Foams blown with other gases

are not a viable replacement for CFC-blown foams in the short run because of their higher thermal conductivity and because these gases are not produced in large enough quantities today.

Effective evacuated powder insulation can be produced by using particle diameters of approximately 10-20 micrometers at air pressures below 100 Pascals. Evacuated powder insulation is a practical replacement to CFC-blown foams both because it is environmentally safe and because it can provide significant energy savings. These additional energy savings are increasingly important in light of the strict state and federal regulations regarding greater energy efficiency in appliances over the next few years. Table 2 shows some projections of the savings possible as a result of using improved insulators.[6]

CELL GAS	FOAM EFFECTIVE CONDUCTIVITY	
	W/m°C	(Btu/hrft°F)
CFC-11	.0180	(.0104)*
HCFC-123	.0200	(.0116)**
HCFC-141b	.0197	(.0114)**
Air (100%)	.0364	(.021)
Carbon Dioxide (100%)	.0266	(.0154)
Air/CFC-11 (50/50)(mass fraction)	.0317	(.0183)
Carbon dioxide/CFC-11 (33/66)	.0227	(.0131)
Carbon dioxide/CFC-11 (50/50)	0.241	(.0139)

* Base line, assumed
** Based on measured vapor thermal
conductivity
    (Shankin, I., Allied, 1988).

Source: Glicksman, *Methods to Enhance the Insulating Values of Closed-Cell Foam*, M.I.T., 1988.

Table 1.1: Thermal conductivity of closed cell polyurethanes and polyisocyanurate foams blown with various agents

Table 1.2: projected cost savings from using improved insulation in appliances

Appliance	Energy consumption (kWh/year)	Savings with improved insulation[a]					
		$k_a = 0.0144$ W/(m·K) $R = 10.0$[b]		$k_a = 0.0096$ W/(m·K) $R = 15.0$		$k_a = 0.0072$ W/(m·K) $R = 20.0$	
		(kWh)	($/year)	(kWh)	($/year)	(kWh)	($/year)
Refrigerator	1084	226	11.28[c]	448	22.40	551	27.54
Freezer (chest)	1143	229	11.43	451	22.55	545	27.25
Water heater							
Gas	8994	712	9.23[d]	838	10.87	899	11.67
Electric	5889	445	22.27	486	24.30	512	25.64
Oven							
Gas	826	50	0.64	59	0.77	64.5	0.84
Electric	360	50	2.50	59	2.95	64.5	3.22

[a]Savings at point of use compared with appliance having insulation R-value of 8.

[b]Thermal resistance per inch, ft²·h·°F/Btu.

[c]Electricity at $0.05/kWh.

[d]Gas at $3.80/10⁶ Btu.

Source: McElroy et al., *Development of Advanced Thermal Insulation for Appliances*, ORNL/TM-9121, ORNL, June, 1984.

Chapter 2

Evacuated Insulation

2.1   Properties of evacuated powders

Insulations which are porous can have their insulating properties improved by reducing the heat conduction which occurs between gaseous particles which are in the porous insulation. This can be done by reducing the transfer of energy from one gaseous molecule to the next by some barrier. Much fewer gas molecules are able to transfer energy if the pressure is reduced, reducing the number of gas molecules and insuring that the distance between the areas of solid contact in the insulation is smaller than the mean free path of the gaseous molecules. This can be accomplished in two ways. First, insulation made up of particles with extremely small diameters can be used so that at atmospheric pressures, the diameter of the particles used in the insulation are smaller than the mean free path of the gaseous molecules. A system based on this method has achieved an R-value of 20 $ft^2.hr.°F/Btu.in.$ at 80°F.[7] However, a problem with using very small particle sizes is that they tend to be expensive and hazardous to workers.

A second method of insuring that the mean free path of the gaseous molecules is greater than the distance between areas of solid contact in a material is to reduce the pressure of the gas and thereby increase the mean free path of its molecules. This allows the use of more abundant and cheaper materials in the insulation. Figure 2-1 shows the R values of several powders as a function of pressure as determined by

McElroy et al (see Appendix A for particle information).[6] Measurements were made of the powder alone with secondary heat transfer through a barrier. Notice that some powders have yielded R values as high as 34 $ft^2.hr.°F/Btu.in.$ at pressures of 10 Pa.

Several factors contribute to the R-value of an evacuated powder.[1] The ratio of mean free path of the gas to the average distance between areas of solid contact within the powder must be large. This is accomplished by the two ways discussed previously. Thermal conduction through the powder particles themselves can be reduced through a reduction in solid to solid contact. Also the transfer of heat through radiation can be reduced by using powders with larger diameters (10 microns) which give large extinction coefficients across a wide range of wavelengths. The perlite powder used in producing the prototype packets was of a 20 micrometer diameter since this particle size was found by a large Japanese Appliance manufacturer to be close to the optimal diameter for thermal resistivity.

2.2 Barrier Materials

The barrier materials which have been explored the most by industry are metallic foils and plastic laminates. While plastic laminates have been produced which keep secondary heat effects to a practical level, the permeation of air through the plastic laminates reduce the insulating ability of a packet over time by increasing its internal pressure. Metallic foils, on the other hand, have the advantage of a lower rate of permeability than plastic laminates but the disadvantage of high thermal conductivities. As a result, in order to reduce secondary heat effects, thinner foils with higher gas permeabilty rates must be used. Figure 2-2 shows how evacuated powder packets lose their insulating ability over time when plastic laminates and foils are used as the barrier materials.[8] The fumed silica packet is 20 X 40 in. and has a barrier made of plastic with aluminum foil attached. The perlite panel has an aluminized barrier and the precipitated silica panel is 18 X 18 in. with a layered polymer structure consisting of mylar and polyethylene.
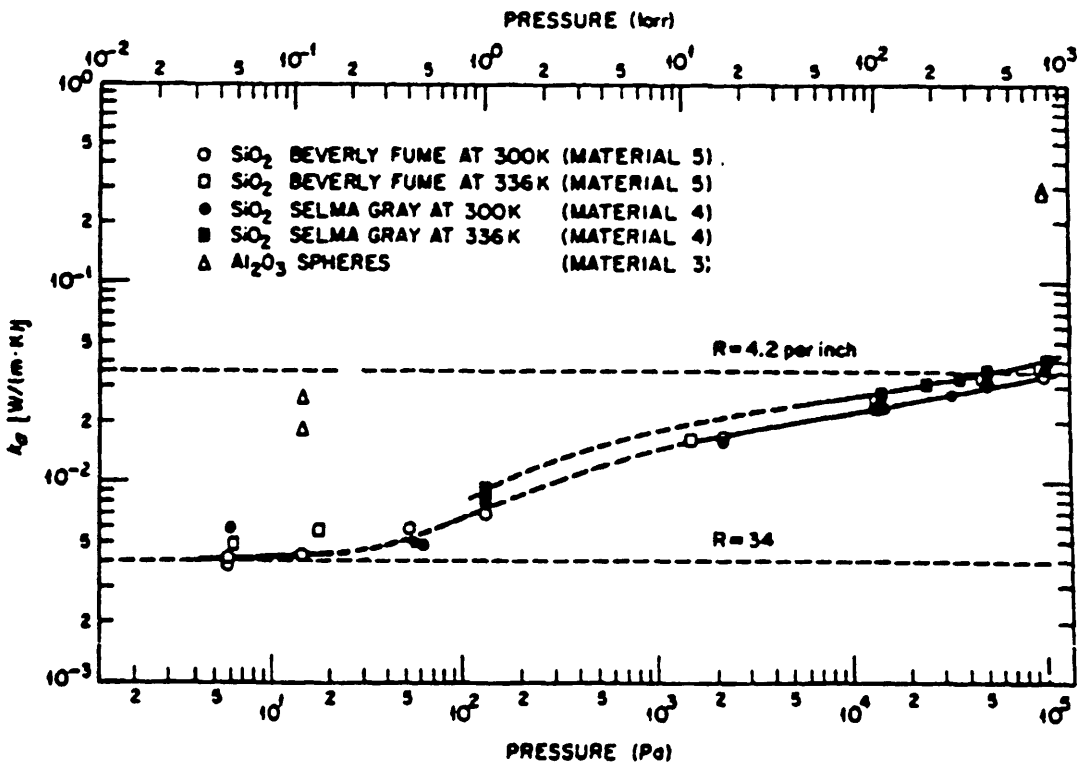
Figure 2-1: Measured thermal conductivities of five types of fine particles as a function of interstitial gas pressure.

Figure 2-2: Thermal resistivity of evacuated powder-filled packets as a function of time.
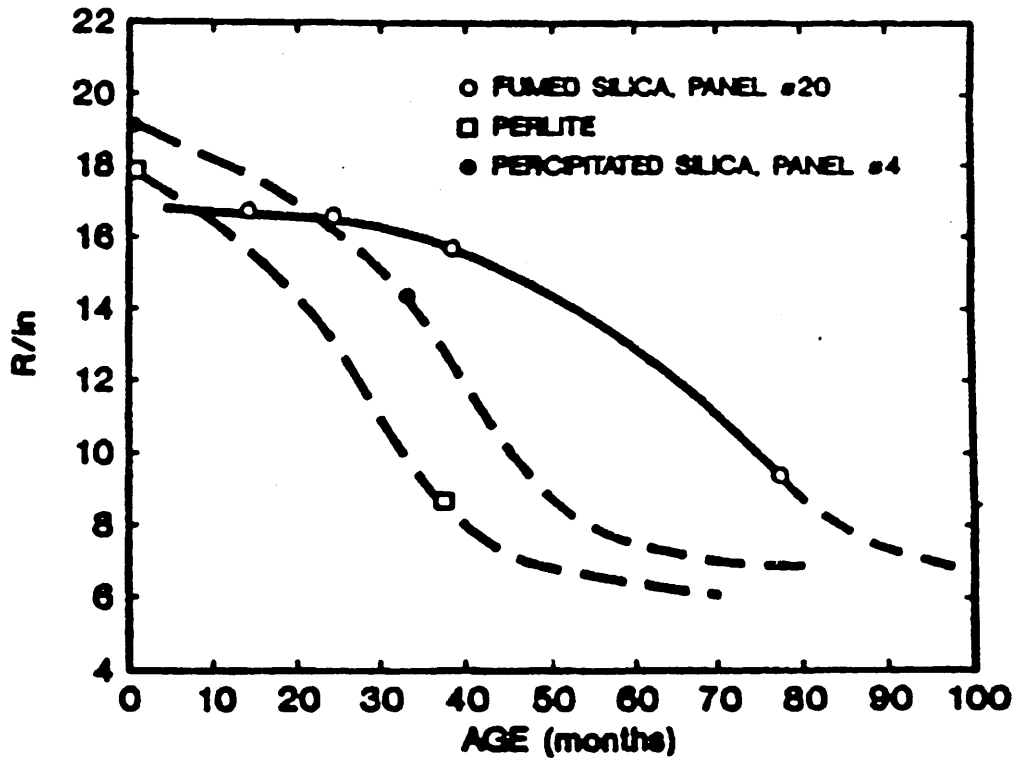
Source: McElroy, *Building Materials Research Program*, M.I.T. Energy Conference, March, 1990, as published by Tester, Wood and Ferrari, editors, *Energy and the Environment in the 21st Century*, M.I.T. Press, 1991.

2.3 Producing an Insulation Based on Perlite Powder Vacuum-packed in Plastic and Glass

The research discussed herein has focused on the production of a prototype insulation packet based on perlite powder vacuum-packed in a layer of plastic and then sealed inside a layer of glass. There are several reasons why this packet design was chosen.

(1) Sufficiently low pressures need to be maintained in the insulation packet for a period of time which is long enough to warrant replacing current insulation. Typically this length of time can be anywhere from 20 to 100 years. As stated earlier, plastic laminates and foils by themselves have a gas permeability rate which is too high to maintain adequate pressures for this length of time. However, because the gas permeability of glass is essentially zero, it can be used in thin layers in order to keep secondary heat effects at an acceptable level. A glass layer will be able to keep the internal pressure of a packet low over the required length of time.

(2) Glass has a thermal conductivity of approximately .5 Btu/hr.ft.°F. This is one hundred times less than the thermal conductivity of aluminum and about four times greater than that of polymers.

(3) Making the packet should not reduce the insulating properties of the powder. Previously, Precipitated silica was encased in glass by slumping a heated sheet of glass over a square of pressed powder.[6] Heating the powder to such a high temperature (1400°F) led to a fusing together of the powder particles, thereby destroying the powders insulating ability. By vacuum packing the powder in a layer of plastic and then sealing this packet in a pre-formed glass shell, heating of the powder to such a high temperature is avoided.

(4) The cost of producing the evacuated powder insulation should be comparable to the cost of insulation used today. Glass is both inexpensive and widely used compared to high-barrier plastic laminates. Since the plastic layer in which the powder is vacuum-packed in does not need to have a low permeability rate, less expensive plastics can be used. Also, perlite is already produced in large amounts and is relatively cheap to produce, giving perlite powder a large cost advantage over the use of silica

precipitate.

Chapter 3

Barrier Materials

3.1 Permeability Comparisons

In order to determine which material would sustain a suitable vacuum in a vacuum-packed powder packet, the permeabilties of several high barrier films and glass were compared. Table 3.1 shows the permeabilties for both oxygen and water vapor of some advanced, currently produced high barrier films.[1]

In addition to these high barrier films, the permeability of glass was also examined. At room temperature, the permeation of glass is too low to be measured using spectroscopy, therefore the permeation rate of silica glass at approximately $1000°C$ was used. (see Figure 3-1).[9] Since silica has the highest permeability of any glass, Figure 3-1 gives an upper bound for the permeation rate of all types of glass.

Interpolating the data in Figure 3-1 gives a permeation rate of 3×10^{-84} cm^3 gas/(100 in.^{2} 24hr atm. mil.) at $35°C$ (S.T.P.). Comparing this to the best high barrier film shown in Table 3.1, we can see that silica has a permeability rate which is many orders of magnitude less.

A sample 1 mil thick was used as a model by Burke to determine how the internal pressure would change due to oxygen permeation through all six sides of a packet.[1] Table 3.2 shows the time it would take for various size packets of glass and the best high barrier film to reach the pressure requirement of 100 Pa. In Table 3.2, a linear pressure gradient of 1 atm was assumed and pressure change calculated using

MATERIAL	O$_2$ TRANSMISSION (cm^3)/(.00in^2 24hr atm mil) at 23°C	WATER VAPOR TRANSMISSION (g)/(100in^2 24hr mil) at 90% RH, 37.8°C
BAREX 210® RESINS by BP Chemicals International	0.8	5.0
SARAN® HB by DOW	0.08	0.05
CAPRANH® by Allied-Signal	0.5	0.2
Vapor deposited quartz film on .5 mil polyester laminated with cast polypropylene by Toppan Printing Inc.	0.06	
Sandwich of .5 mil PET/ 400Å quartz/ .2 mil barrier adhesive/ 400Å quartz (vapor deposited)/ .5 mil PET by Toppan Printing Inc.	0.002	

Source: Burke, *Insulation Based On Silica Powder Vacuum-Packed In Thin Glass*,

M.I.T., MS Thesis, M.E. Department, 1990.

Table 3.1: Permeabilities of Some Currently Produced High Barrier Films

The graph shows a plot with:
- Y-axis: Log P, p in (cc/s.cm2 area.cm Hg) for a thickness of 1mm, ranging from -11.4 to -12.1
- X-axis: 1/T in (1/°C), ranging from 1.00e-3 to 1.20e-3
- Equation: $y = -8.8029 - 2931.2x \quad R^2 = 0.996$
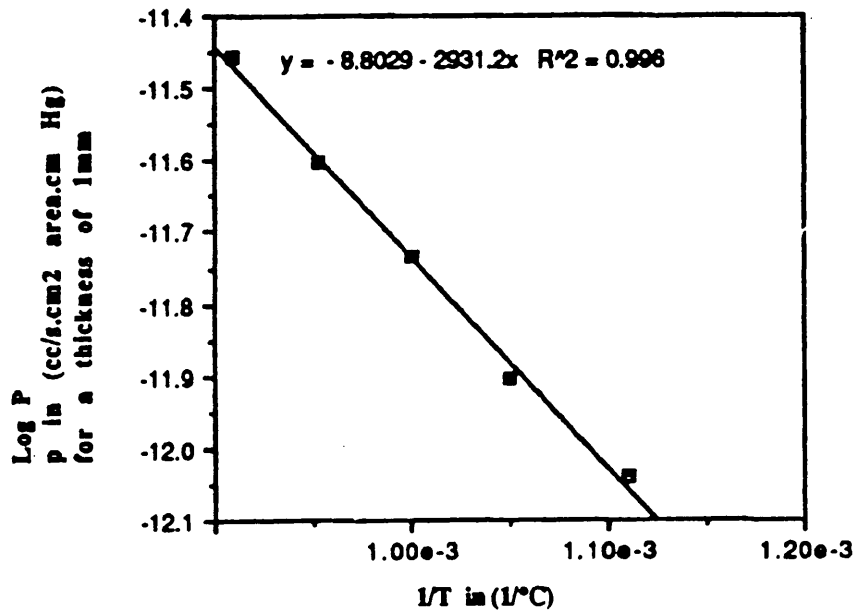
Source: Norton, *Permeation of Gaseous Oxygen through Vitreous Silica,* Nature (vol. 191), August 12, 1961, page 701.

Figure 3-1: Permeation of Gaseous Oxygen through Vitreous Silica Glass

(cm³)/(100in² 24hr atm mil)	6X6X1 in.	12X12X1 in.	24X24X1 in.	36X36X1 in.
.002 : Barrier Film by Toppan	.83	.96	1.03	1.06
3E-84 : Glass	5.5×10^{80}	6.3×10^{80}	6.8×10^{80}	7×10^{80}

Source: Burke, *Insulation Based On Silica Powder Vacuum-Packed In Thin Glass*, M.I.T., MS Thesis, M.E. Department, 1990.

Table 3.2: A comparison of the performance of 1 mil thick barriers for various panel sizes.

$$\triangle P/day = (6)P(p)(1/m)(RT/V) \qquad (3.1)$$

where P is the permeability in $cm^3/(100in^2 24hratm)$, p is the density in kg/cm^3, m is the mass per mole in kg/mol, R is the universal gas constant in $J/molK$, T is the temperature in K, and V is the volume in m^3.

As the table shows, the performance of the glass packet is much better than the performance of the high barrier film.

3.2 Thermal Conduction of Various Barrier Materials

In order to determine the theoretical heat transfer rates of various boundary materials, a two-dimensional heat transfer model developed by Glicksman and based on

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silica vacuum-paked in a various barrier materials is used.[10] The model begins by assuming that the packets width and thickness is much smaller than the packet's length. This is done so that thermal conduction in the third dimension can be assumed to be negligible. Also, the thermal conductance through the powder is assumed to be one dimensional.

For this model, two different boundary conditions for the theoretical packet are analyzed. First, the packet's hot boundary is assumed to be attached to a thick metal wall (see Figure 3-3).[1] In this case, it is assumed that there is no temperature gradient through the wall and packet barrier in the vertical direction. This makes the thickness and thermal conductivity of the hot surface greater than that of the cold surface. Just half the length of the packet needs to be considered since vertical symmetry is preserved. For the second boundary condition, the panel is exposed on both sides to air with convection (see Figure 3-2).[1]

The silica powder in the packets is assumed to have an R-value of 33 for a thickness of 1 inch. This value is based on the experiments done by McElroy et al..[6] To determine the convective constant, the equation

$$hp \simeq kp/w \qquad (3.2)$$

is used, where kp is the thermal conductivity of the material and w is the width of the powder (see Figure 3-2).

A finite difference analysis is done by by dividing the boundary walls into equal segments (see Figure 3-4[1]). An energy balance is then done using the equation

$$- C'_1 * T_{m-1} - C'_1 * T_{m+1} + (2C'_1 + C'_2 + C'_3) * T_m - C'_3 * T_{n-m+1} = T_{cold} * C'_2 \qquad (3.3)$$

where $C'_1 = k * t/dx$, $C'_2 = h_c * dx$, and $C'_3 = h_p * dx$. The k used in determining C'_1 is the thermal conductivity of the barrier material. A similar expression is written for the other boundary.

For the boundary condition of being mounted on a metal wall, the product of the
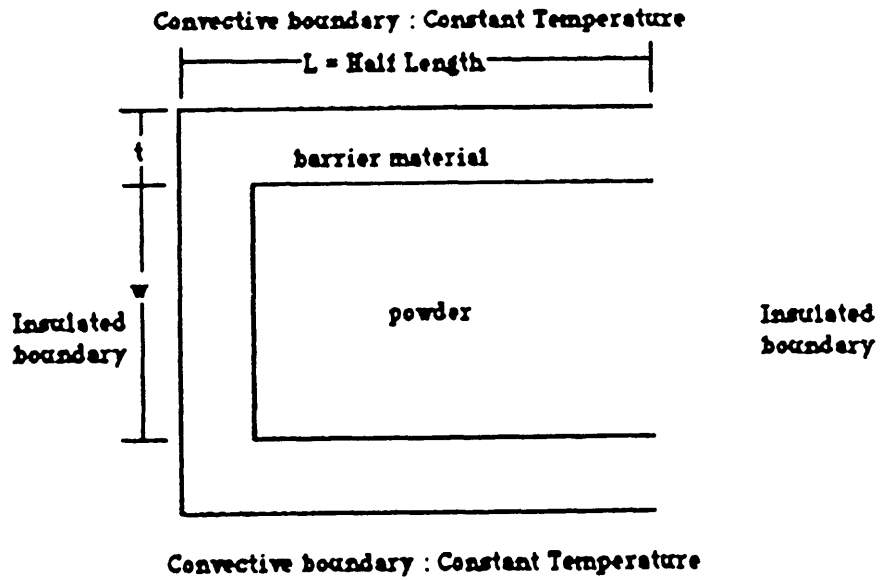
Figure 3-2: Heat transfer in a packet exposed to air on both sides
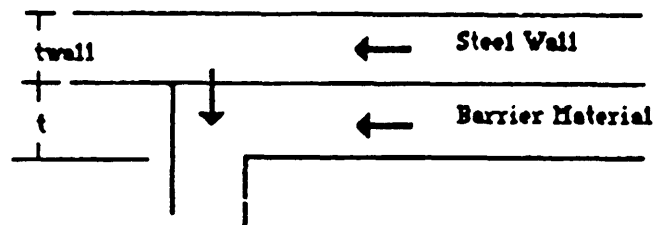
Figure 3-3: Heat transfer in a packet attached to a metal wall
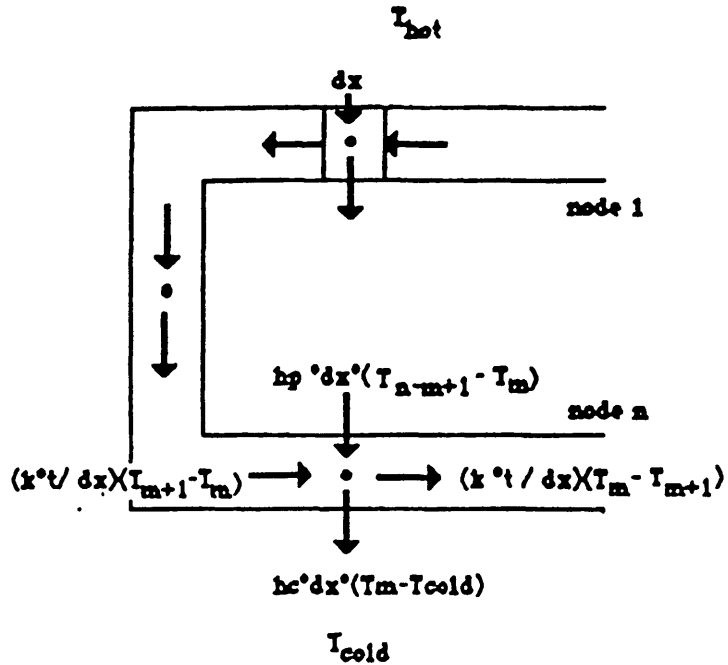
Figure 3-4: Energy balance for evacuated powder packet

thermal conductivity and the thickness is given by adding the products for the wall and the barrier. The distance between node points stayed constant at .125 inches; therefore, the number of nodes changed with the length of the panel. For a 4 inch half length and a width of one inch, 72 nodes were analyzed.

3.3 Theoretical Results of Heat Transfer Model

Both Glass and foil were analyzed using the heat transfer model. Figure 3-5 and Figure 3-6 show the results of the model.[1] Even though glass thicknesses greater than those of the aluminum were considered (.005 and .01 inches for the glass compared to .0003 and .001 inches for the aluminum), the glass packets still outperformed the aluminum packets. This allows smaller glass packets to be made without causing significant secondary effects.

.0003 inch foil thickness,

.0003 inch foil barrier with panel mounted on a .76 inch (ts=.76) thick steel plate,

.001 inch foil barrier,

.001 inch foil barrier mounted on a .76 inck thick steel plate
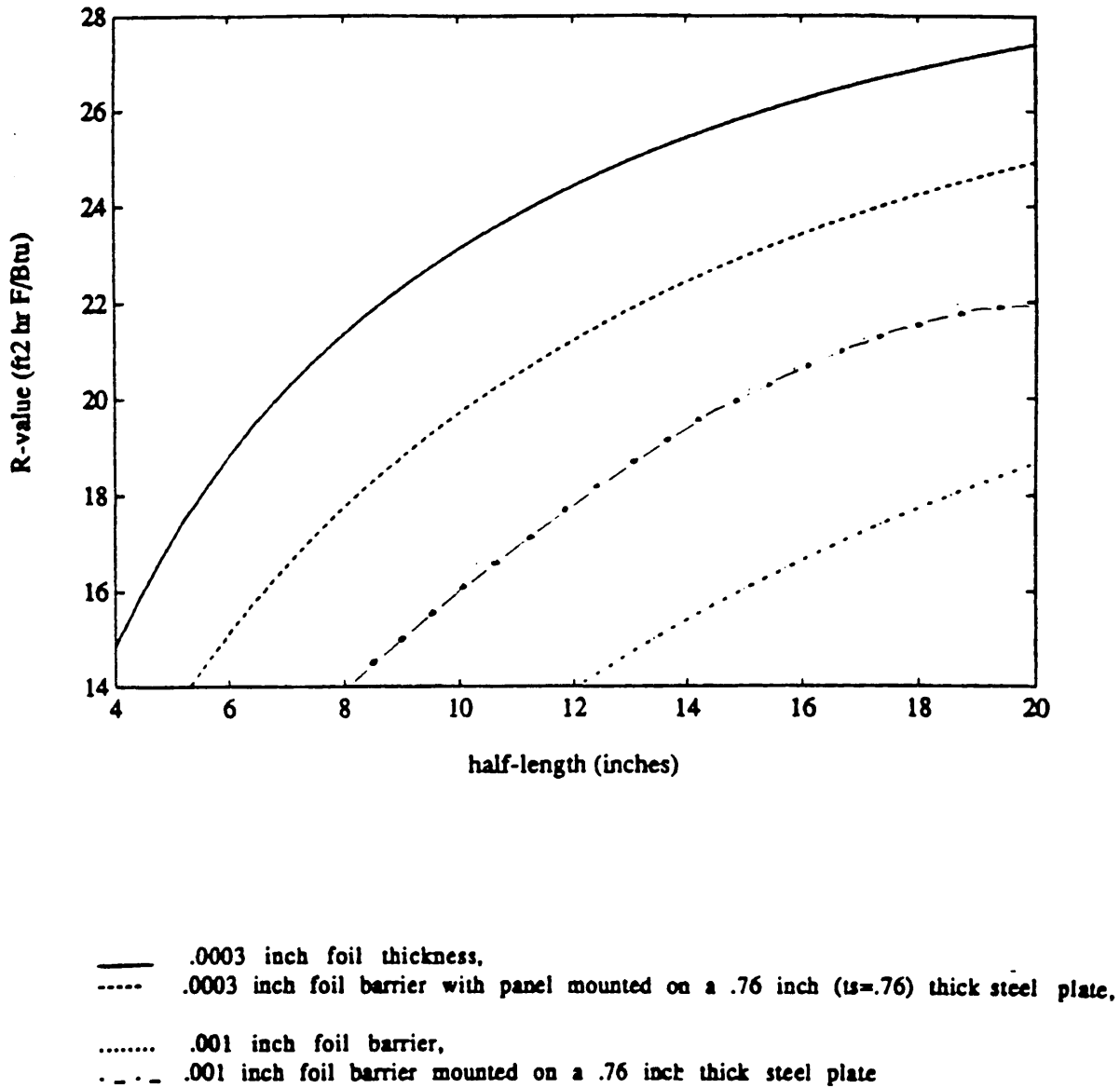
Figure 3-5: R-value vs. half length for 1-inch thick evacuated silica packets with aluminum foil barrier (k=115 Btu/hr.ft.°F).
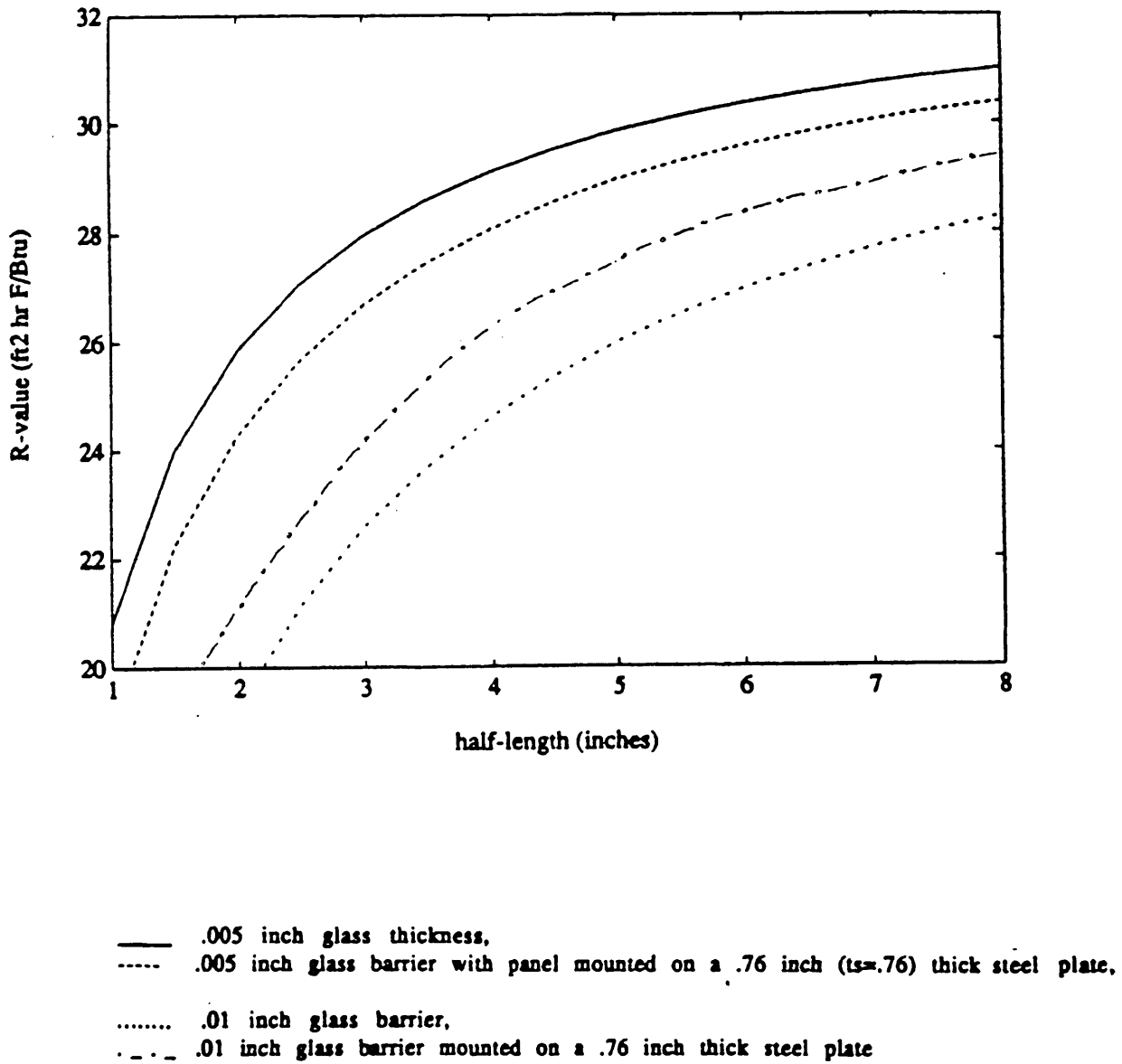
Figure 3-6: R-value vs. half length for 1 inch-thick evacuated silica packets with a glass barrier (k=.56 Btu/hr.ft.°F).

Chapter 4

Production of Prototype Powder Vacuum-Packed Packets

The aim of most of the research presented herein was the making of prototype packets of perlite powder vacuum-packed in a layer of plastic and a layer of thin glass (see Figure 4-1). This was done because (1) glass has a much lower permeability rate than other barrier materials and can therefore keep a vacuum for a commercially viable period of time, and (2) the powder needs to be sealed in a layer of plastic in order to avoid heating the powder and thereby destroying its insulating properties. This was accomplished by placing a powder packet already wrapped in plastic into a preformed glass shell and then sealing this glass shell with an adhesive with low permeability. Vacuum grease was also used in order to ensure that no air remained between the plastic and glass barriers.

4.1   Powders used in samples

Perlite (see Appendix B for information) of a 20 micrometer particle diameter was the primary powder used in the production of the prototype samples. There were five main reasons for this. First, perlite powder has the proper characteristics to be a good insulator in an evacuated environment. Second, perlite costs very little compared to other powders. This cost efficiency would be an advantage if these insulation packets

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GLASS BARRIER

POWDER IN VACUUM $\frac{1}{2}''$

VACUUM GREASE
TO FILL GAPS   PLASTIC BARRIER
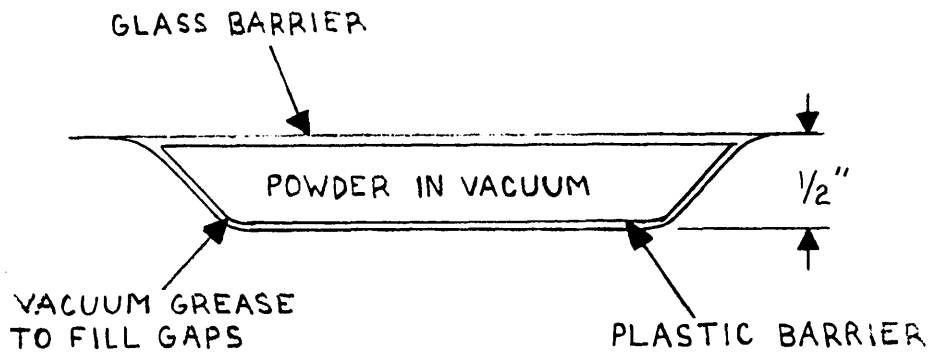
Figure 4-1: Schematic of assembled panel

are manufactured commercially. Third, a supply of perlite was readily available. Fourth, 20 micrometers is close to the optimal perlite particle diameter for lowest thermal conductivity. And finally, compared to silica precipitates, perlite was much easier to use in producing samples because it tends to stay packed down more when handling. Producing the packets required a certain amount of handling. The silica precipitate (see Appendix C for information) was difficult to keep in a packed block, spreading out onto the area of the plastic envelope that was to be sealed. As a result, the plastic was much easier to seal with the perlite than with the silica precipitate. This difficulty in sealing the silica precipitate packets also meant that trimming the edges of the vacuum sealed packet, which was necessary to fit the plastic packet into the glass envelope, tended to create a hole in the plastic packet and thereby destroy the vacuum.

4.2   Techniques for Encasing the Powder in Plastic

To make a sample insulation packet, the first step was to form the chosen plastic into a shape in which to pack the powder. The plastic used, a 5-layer high barrier film

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produced by Quantum Chemical Corporation, was chosen because (1) it's thermal conductivity was sufficiently low to be used as a barrier material, (2) it was relatively easy to form it into the desired shape, and (3) it was possible to seal the edges of the plastic together due to a special coating covering one side of the plastic. The plastic was formed using the steel mold on which the glass shell was formed (see Figures 4-2 and 4-3) This was to insure a close fit between the plastic packet and the glass shell. The sides of the mold were sloped in order to facilitate removing the glass shell. An alternate mold made of graphite was also used but was finally discarded because the plastic failed to shape properly on its surface and the graphite mold was extremely messy to handle.

In order to reduce the size of the plastic packet slightly in comparison to the glass shell, a thin sheet of copper was fitted to the shape of the glass mold and four holes were drilled into its bottom to correspond with the holes in the glass mold. This copper "filler" was then placed between the sheet of plastic and the steel mold when forming the plastic. After clamping an aluminum ring on top of the plastic sheet (see Figure 4-3), the plastic was heated quickly with a heat gun until the plastic visibly relaxed. Then a vacuum of approximately 10 inches of mercury was applied for about 2 seconds. The resulting pressure difference shaped the plastic into the desired shape. The plastic could be heated and a vacuum applied several times in order to ensure a close fit with the mold.

After forming the plastic shell, the powder was packed into it using a hydraulic press. The powder was packed under compressive stresses ranging from 25 psi to 2000 psi. At 25 psi, the perlite powder, which was the easiest powder to pack among those tested, proved difficult to handle. Perlite packed at 25 psi was loose enough as to make sealing the plastic edges together difficult.

After packing the powder into the plastic shell, another sheet of plastic was placed on top of the powder and the edges of the packet were sealed together using a levered-arm press (see Figure 4-4). The edges of the plastic packet were placed between the two hollow rectangular aluminum tubes, the top of which was heated via a heating coil wrapped around its inside. The best seal was produced by heating the coil with

FRAME

VACUUM
APPLIED

STEEL MOLD

2 ½" 3 ½"

¹⁄₁₆" DIAM.
HOLE

7" 7 ³⁄₄"

TOP VIEW

½" 3⁄8"

45° SLOPE

SIDE VIEW
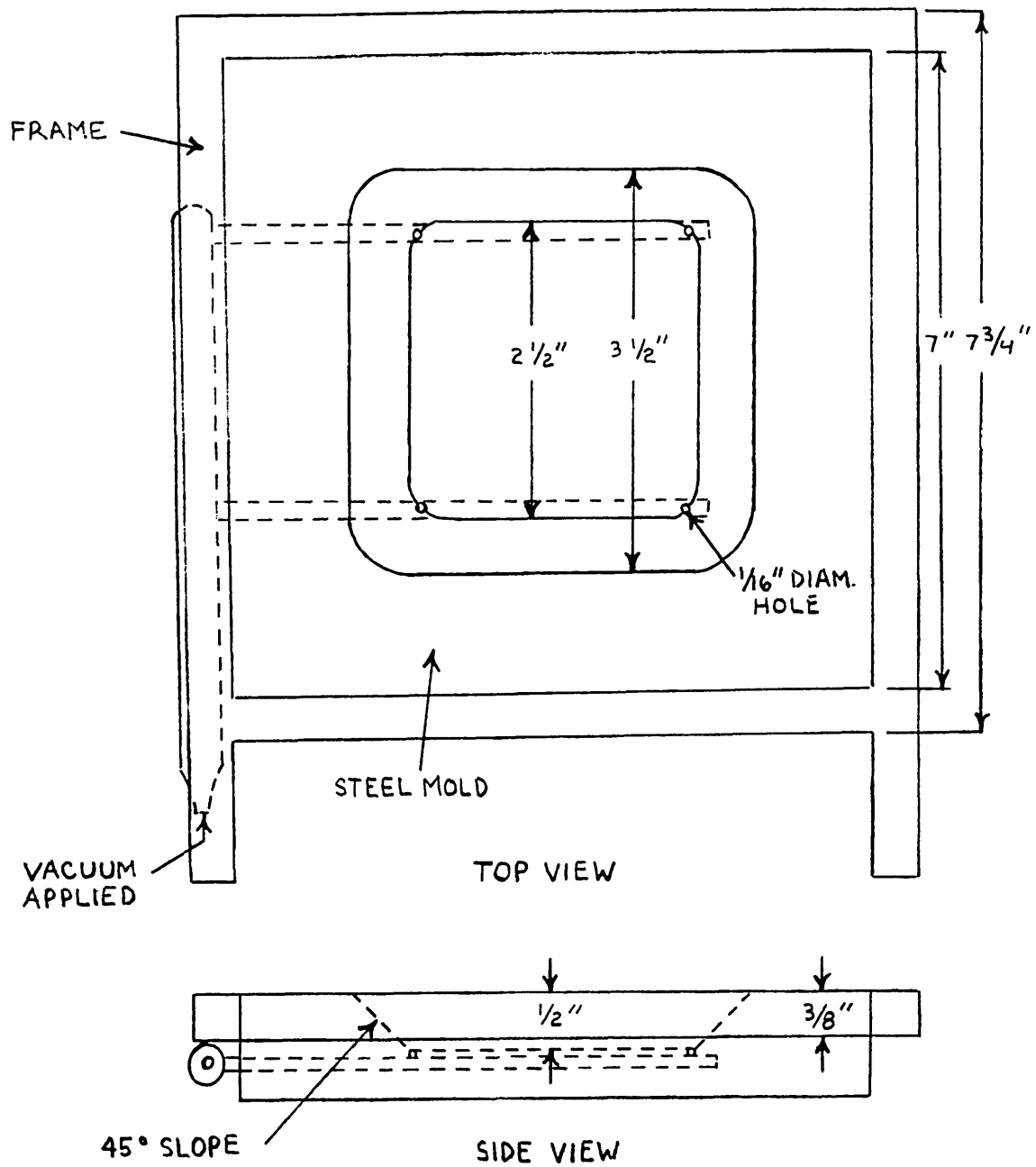
Figure 4-2: Steel mold used in shaping plastic and glass shells

ALUMINUM COVER FOR
FORMING PLASTIC

CLAMP

STEEL MOLD

STEEL COVER FOR
FORMING GLASS
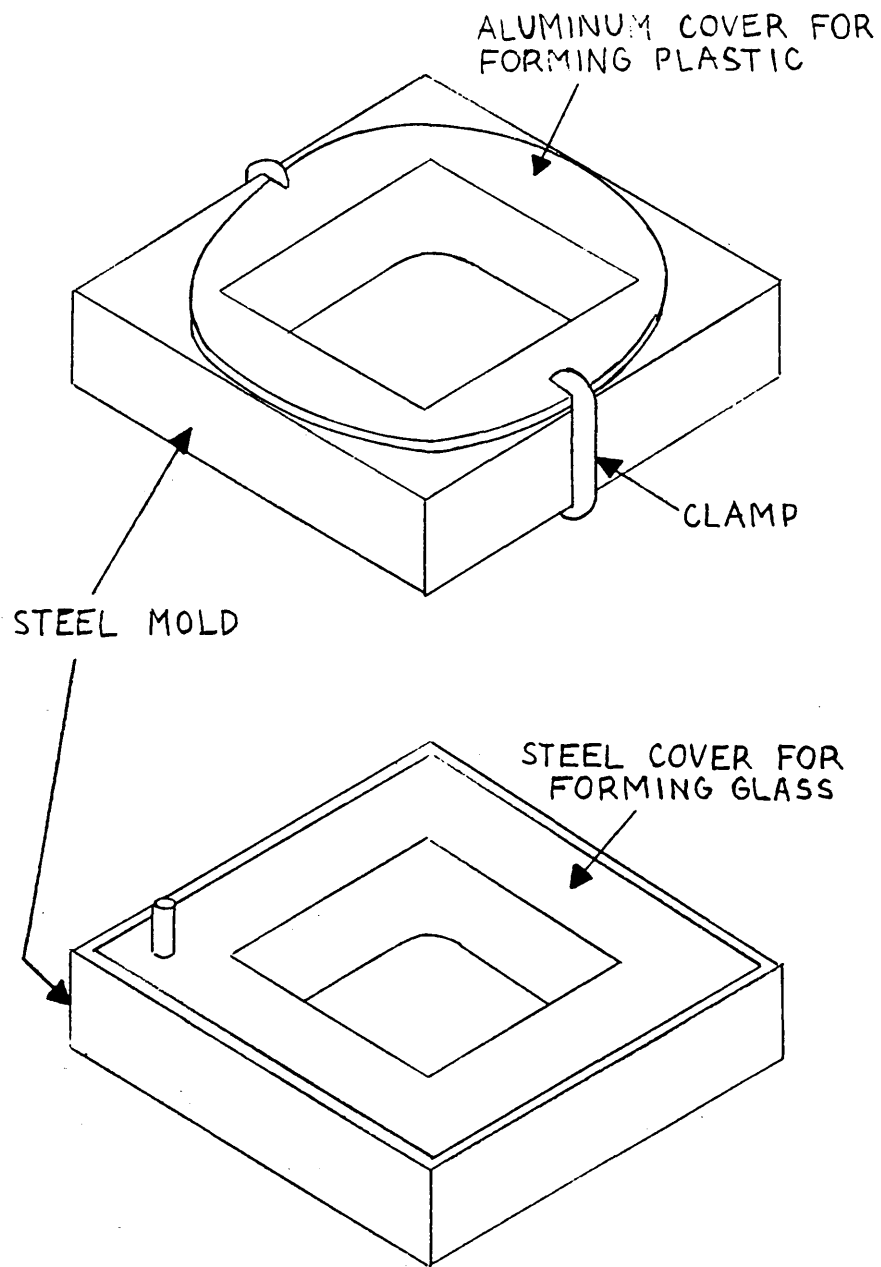
Figure 4-3: Forming the plastic and glass shells
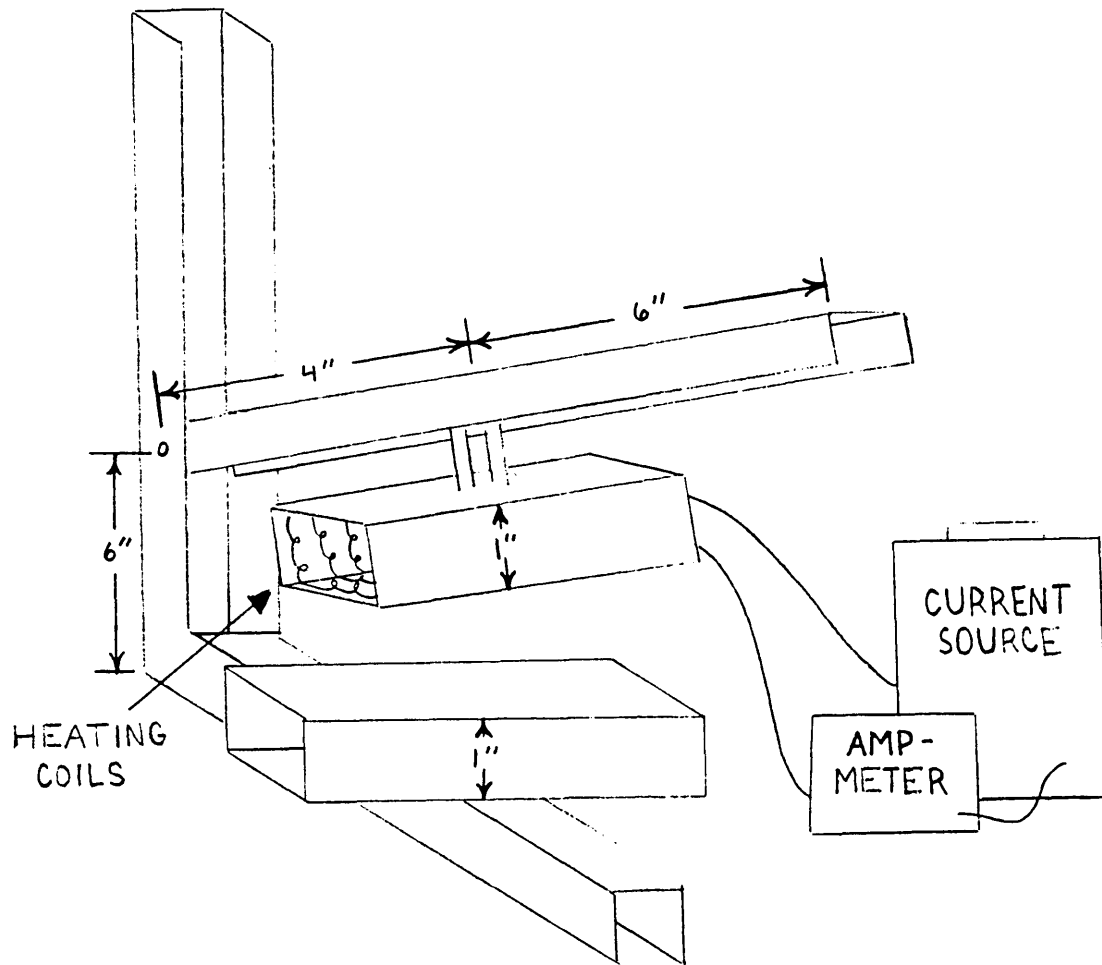
Figure 4-4: Press used to seal edges of plastic packet

approximately 3 amps of electricity for approximately 20 minutes before attempting
to seal the plastic edges. Pressure of about 20 ft.-lbs. was applied via the lever arm
for 30 seconds in order to insure a proper seal. If the plastic packet was held in the
press for only a few seconds longer than this, the plastic tended to become over-heated
and tear as it was pealed off the aluminum tubes.

Three of the packet edges were sealed completely while a hole about one inch in
width was left unsealed in the middle of the fourth side. The packet was then placed
in a glass jar in which a vacuum of about 32 inches of mercury was created using
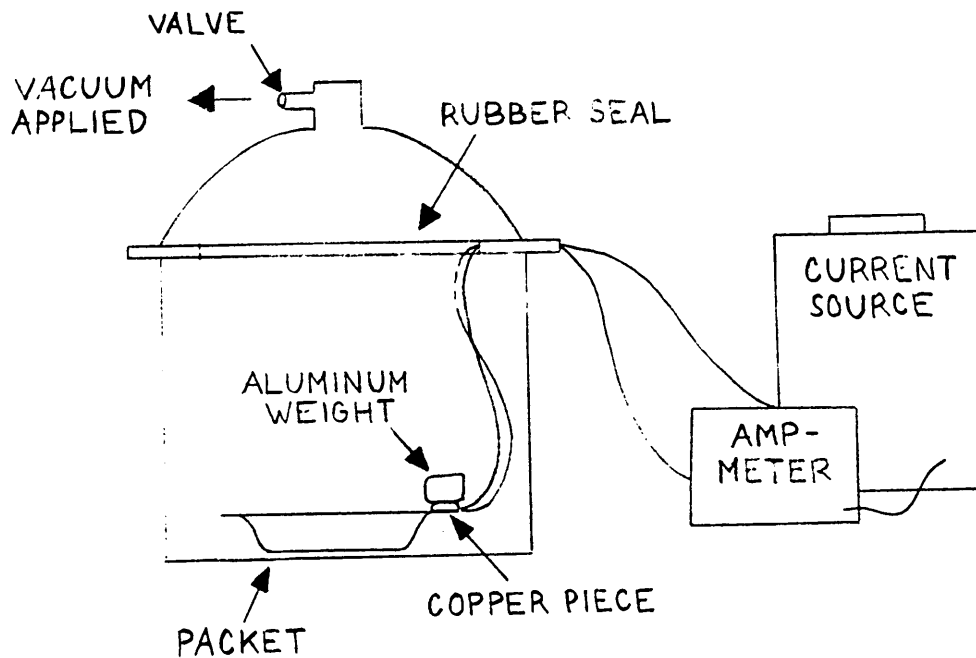
Figure 4-5: Glass jar used for vacuum-sealing powder packet

a vacuum pump (see Figure 4-5). This vacuum was held for 1 hour. A small, flat piece of copper placed over the hole in the packet was then heated via a heating coil with 2.5 amps of electricity for 2 1/2 minutes. A small piece of aluminum weighing approximately 5 ounces was placed on the copper piece to provide additional weight for sealing the hole. Using a weight heavier than this tended to keep air from being removed from the packet. After 2 1/2 minutes the electricity was switched off and the packet removed. As the pressure inside the glass jar returned to atmospheric, the pressure difference between the inside and outside of the packet caused the plastic to press itself tightly against the packed powder. A properly evacuated packet could visually be detected by the wrinkling of the plastic barrier as it was pressed against the powder. Also, an evacuated packet was much stiffer to the touch than an unevacuated packet.

4.3 Forming the Glass Shell

The glass shell was formed from 0211 glass made by Corning Glass using a drawing process. The glass is approximately .005 to .007 inches thick (see Appendix D for more info). Each sheet was approximately 7 1/2 inches square and .005 inches thick before being formed. The need to keep the secondary heat transfer through the glass from becoming too high necessitated the use of glass of this thickness.

To form a shell, a sheet of glass was placed on a steel mold (the same mold used in forming the plastic shell), covered and heated in an oven to approximately 1500°F. A vacuum of 15 inches of mercury was then applied for approximately 2 seconds in order to form the glass sheet to the steel mold. A steel cover on top of the glass hindered the formation of folds and wrinkles in the glass shell (see Figures 4-3 and 4-6). After forming the glass shell, the cover was immediately taken off and the oven was cooled to 1200°F over a period of about an hour. Cooling the oven much faster than this rate tended to produce further wrinkles in the glass shell. At 1200°F, the glass hardens enough to allow it to be lifted out of the steel mold by a square loop also made from steel. If the formed glass shell is not lifted out of the steel mold, the difference in the specific heat of the glass versus that of the steel, combined with a slight adhesion of the glass to the steel, causes the glass shell to crack as the oven cools. After being lifted from the steel mold, the glass shell is left in the oven and allowed to cool over a period of 6 to 7 hours in order to avoid too rapid a cooldown which tends to stress and crack the glass.

The steel mold, cover, and loop are all covered with Boron Nitride coating on areas that the glass sheet touches. Once dry, the Boron Nitride forms a powdery layer between the glass and the steel, keeping the glass sheet from sticking to the steel. More information on the Boron Nitride coating is available in Appendix E.
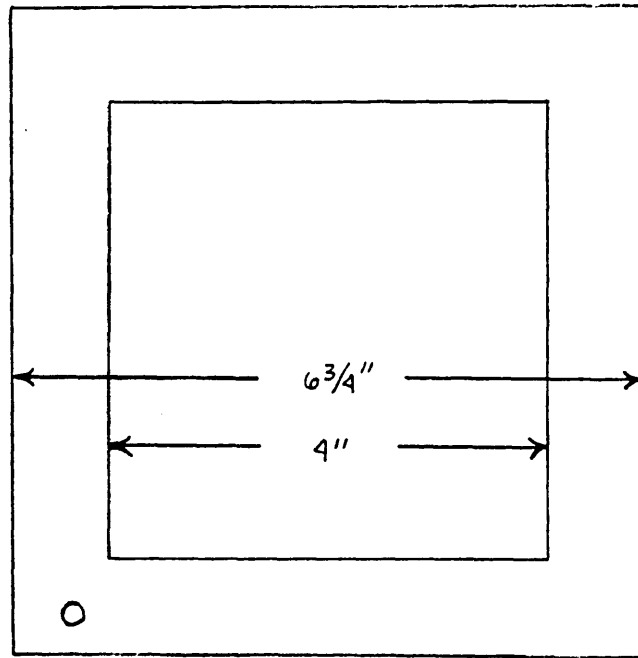
TOP VIEW
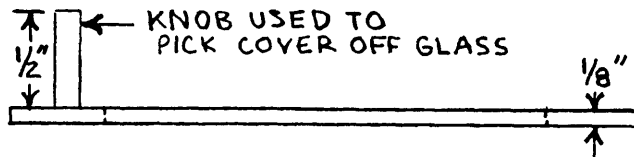
SIDE VIEW

Figure 4-6: Steel cover used in forming glass shell.

4.4 Combining the Plastic Packet and glass

The powder and plastic packet is placed in the glass shell and a flat sheet of glass is placed over it. In order to take up any gaps between the plastic powder packet and the glass shell in which air might be trapped, vacuum grease is used to coat the plastic packet. In the samples produced, this gap was approximately 1/8 in. on the bottom and sides of the plastic packet. The top sheet of glass is sealed onto the glass shell with Torr-Seal, an adhesive made by Varian Associates which is designed to seal glass to glass at a pressure level of 10^{-9} Torr.

Chapter 5

Testing of the Evacuated Powder Packets

Twelve packets measuring approximately 3 1/2 X 3 1/2 X 1/2 in. and using perlite and silica precipitate were produced and tested in which plastic was the only boundary material (see Table 5.1. This was done in order to determine which packets had been evacuated to a suitable internal pressure. Packets with only plastic as a boundary material were tested without being sandwiched between two layers of foam. A packet using silica precipitate was tested in order to determine the extent that this powder improved the insulating properties of a packet. Also, a packet was produced and tested in which the perlite powder was baked before being vacuum-sealed in order to remove moisture from the powder. This was done to see if moisture in the powder noticeably affected the k-value of the packet.

Two packets with a sufficient internal vacuum were then encased in glass and then tested again. The glass-encassed packets were sandwiched between layers of foam with a k-value of .29 Btu.in./ft^2.hr.°F in order to prevent accidental cracking of the glass as it was being tested (See Figure 5-1.

Thermal testing of the all packets was performed using an Anacon Model 88 thermal conduction machine to measure the k-value. The packets were placed between two plates. The top plate is kept at a constant temp of 100°F and the bottom plate is kept at a constant temperature of 50°F. The distance between the plates is adjustable
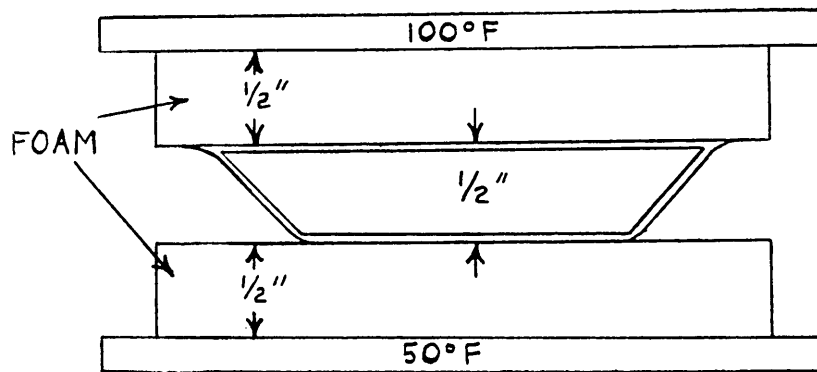
Figure 5-1: Testing the packets

to a maximum distance of 1.75 inches. K-value is determined by measuring the heat flux between the two plates.

5.1 Test Results

The results of the thermal tests done on the specimens are shown in Table 5.1. The best packet wrapped only in plastic achieved a k-value of .0086. Several other packets achieved k-values close to this, ranging from .092 to .12. Three of the packets did not have an adequate vacuum and therefore had k-values from .22-.25. Two packets achieved k-values better than those of CFC foams, which have k-values of .10-.12. Packets produced using silica precipitate packed at a pressure of 30 psi in high-barrier film have been produced in industry with a k-value of approximately .004.

The packets wrapped in plastic and glass achieved a much higher k-value of .18. This represents approximately a 50 percent decrease in the insulating ability of the packet wrapped in plastic alone and is much higher than the k-values for CFC-blown foams.

Several factors may account for the k-factors below those predicted by the theoretical model. The internal pressure of several of the plastic packets was higher than desired. This may have been caused by (1) not achieving a low enough internal pressure before sealing (a better gauge is needed to measure the vacuum accurately), (2) by air entering through a small leak in the plastic boundary, or (3) the giving off of moisture by the powder after the plastic boundary was sealed. Also the glass packets were made using vacuum grease with a k-value of .21 W/m-°K to fill in the gaps between the plastic and glass boundaries. A layer of vacuum grease approximately 1/8 in. thick was used on the bottom and sides of the packets, allowing additional heat flow of up to .5 J/s during testing of the packets.

POWDER USED	COMPRESSIVE STRESS ON POWDER (PSI)	K-VALUE $(Btu.in./ft^2.hr.°F)$	
		PLASTIC BARRIER*	GLASS AND PLASTIC***
PERLITE	80	.25	—
PERLITE**	150	.12	—
"	150	.24	—
"	25	.092	—
"	40	.114	.18
"	400	—	—
"	700	.22	—
"	1000	.115	.18
"	1300	.086	—
SIPERNAT 50S PRECIPITATED SILICA	1250	.11	—

* TESTED WITHOUT FOAM
** POWDER WAS BAKED AT 220°F FOR THREE HRS
*** TESTED WITH FOAM

Table 5.1: k-values of evacuated powder packets

5.2   Improvements to be made in producing glass and plastic packets

There are several ways in which to improve the method of making evacuated powder packets, some of which will improve their insulating ability. The most important improvement is the creation of a sealed plastic packet which has a shape closer to that of the glass shell. This will greatly reduce the amount of vacuum grease needed to fill in gaps between the glass and plastic. Thermal conductivity will then only be significant for the glass, plastic and powder.

A better vacuum should be produced in the packets. This can be done by improving the way the air is taken from the packet. The easiest way of doing this is by increasing the strength of the vacuum that the pump delivers to the glass jar in which the packets are sealed, i.e. get a stronger pump.

The effect of moisture within the powder was not noticeable in this limited test of the insulation. However, powders used in industry are routinely baked before being placed in insulating packets because moisture in the powder can have a negative effect on the k-values of the packets. Further research should focus on achieving better results by baking all the powder that is used to make packets.

Several different pressures were used to pack the powder into the plastic shell before the packet was sealed. While the limited testing done here did not find conclusive evidence that any one range of pressures was ideal, further research should aim at determining whether such a range exists. To this end, "tea bags" should be used to encase the powder. Tea bags are porous bags used in industry which permits gas to permeate but prevent the loss of powder. By wrapping the powder in a tea bag before sealing it in a layer of more impermeable plastic, the powder can be contained more easily while sealing and thus cut down on the amount of handling the researcher has to do. Because handling the powder packs before they are sealed tends to loosen the powder, tea bags will allow a more accurate determination of the pressure at which a powder is packed.

Finally the making of the glass shell should be inproved in order to eliminate

wrinkles in the glass. Eliminating the wrinkles will not only make for a better fit between the glass shell and the plastic packet, but also make sealing the glass shell easier. Also, the glass shells as they are made now need to have the edges trimmed in order to have a closer fit with the plastic packets. By creating a mold with smaller edges, the need to trim the edges of the glass shells can be eliminated.

Chapter 6

Commercial considerations involved in producing glass and plastic-covered evacuated powder packets

6.1 Cost and Demand Considerations

CFC-blown foam costs about ten cents a board foot (1 ft. X 1 ft. X 1 in.) to produce commercially. Most industrial research has focused on the use of evacuated powders surrounded only by a high barrier laminate or foil. As stated earlier, foils allow secondary heat effects in the insulation to become too high while high barrier laminates are expensive to make and may not be able to manntain the vacuum for the lifetime of the insulation. As a result, finding an alternative to CFC-blown foam which performs as well at around the same cost is difficult. Perlite powder evacuated and surrounded by plastic and glass is a workable alternative. Perlite powder and glass are both produced in sufficient quantities to make their use commercially viable. In addition, because the plastic barrier used to surround the powder initially only needs to hold a vacuum for a few days (rather than 20 years), its cost is substantially

reduced. As a result, the production of this type of insulation is comparable to that of CFC foams. In addition, this research has shown that perlite evacuated and sealed in plastic and glass has insulating properties slightly better than CFC-blown foams.

The desire to stop the damage that chloroflourocarbons are causing to the environment along with stringent government guidelines for energy efficiency that are due to go into effect soon mean that the demand for an improved and less damaging insulation is high.

6.2   Manufacturing considerations

There are several possible ways that evacuated powder packets surrounded by plastic and glass can be produced in large numbers. All of them will involve forming the glass shells seperately from the evacuated powder packets and then combining the two. Production of this type of insulation will probably involve producing a matrix of small packets, a sheet 3 ft. X 3 ft. made up of packets 3 in. X 3 in. for example. This allows the insulation to remain functional should one of the packets be punctured. Also the insulation should be packed in a material, foam for example, which will cushion the glass-covered insulation packets from shock. This will prevent the glass from cracking and allowing air to enter the packet.

Producing the glass shells in the matrix can be accomplished by heating a sheet of glass over a mold which will form a matrix of connected shells. The glass can be formed much as it was in producing the prototype, by applying a vacuum to the mold in order to suck the heated glass into the mold (see Figure 6-1). A stamp from above the mold could also be used to form the glass. Because the glass is so thin, it may be wise to create each of the glass shells seperately in order to avoid the occurence of a single crack which compromises more than on packet. The individual packets can be separated by a thin layer of foam.

The powder packets themselves can be made using the "tea bags" mentioned earlier and which are now used to create prototype packets in industry. Once a powder square has been baked to remove moisture and pressed to the proper pressure, a tea
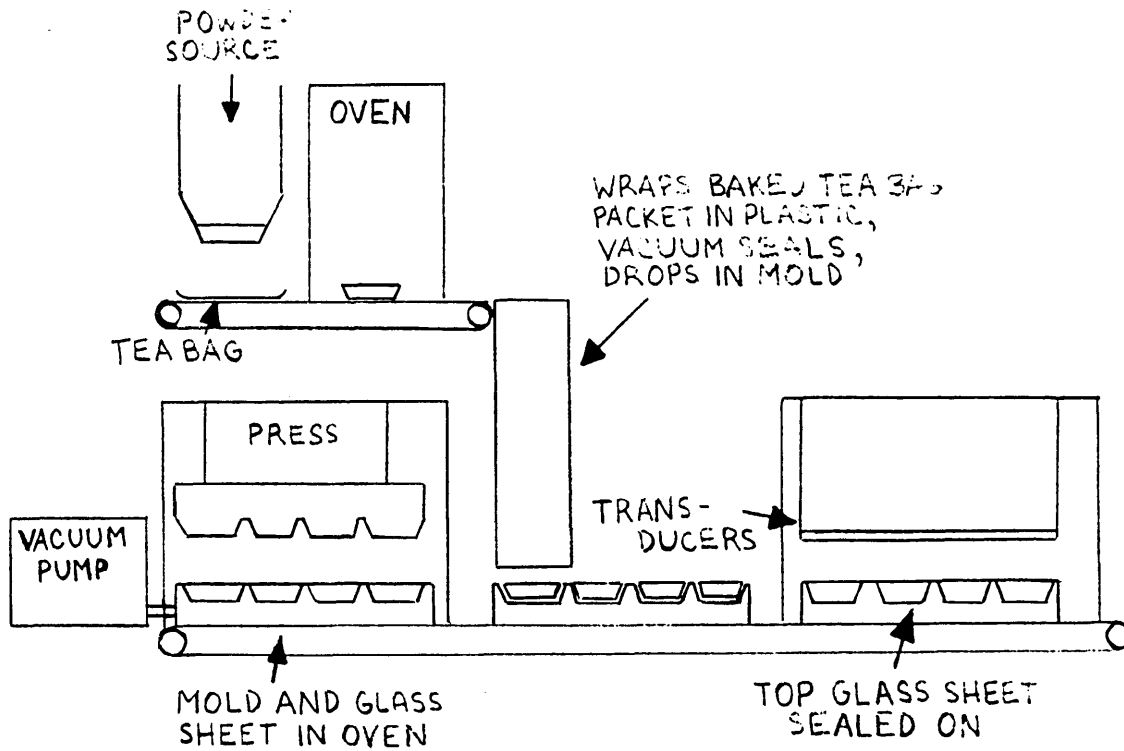
Figure 6-1: Manufacturing glass shells

bag can be wrapped around the powder (see Figure 6-1). At this point, these tea bag packets can be placed in the glass shells. From here the packets and glass shells will move to an enclosure in which a vacuum is established and a sheet (or sheets if the shells are not connected) of glass is sealed onto the top of the matrix. In order to avoid heating the powder, this sealing process can be accomplished using either thermocouples to heat the glass locally or, if thermocouples prove impractical due to the creation of large stresses in the glass, a low temperature adhesive can be used to seal the glass.

References

1. Burke, M. S., *Insulation Based On Silica Powder Vacuum-Packed In Thin Glass*, Massachusetts Institute of Technology, MS Thesis, M.E. Department, 1990.

2. Brooks et al., US patent 2,989,156, 1956.

3. Beckman, US patent 3,094,071, 1960.

4. Strong et al., US patent 3,179,549, 1964

5. Glicksman, L. R., *Methods to Enhance the Insulating Values of Closed-Cell Foam*, Massachusetts Institute of Technology, 1988.

6. McElroy et al., *Development of Advanced Thermal Insulation for Appliances*, ORNL/TM-9121, Oak Ridge National Laboratory, June, 1984.

7. Pelloux-Gervais et al., *Insulating Material with Low Thermal Conductivity Formed of a Compacted Granular Structure*, US patent 4,159,359, 1977.

8. McElroy, D. L., *Building Materials Research Program*, MIT Energy Conference, March, 1990.

9. Norton, *Permeation of Gaseous Oxygen through Vitreous Silica*, Nature (vol. 191), August 12, 1961, pg. 701.

10. Glicksman, L. R., *Two-Dimensional Heat Transfer Effects on Vacuum and Reflective Insulations*, XXI ICHMT Symposium, 1989.

Appendix A

Information on materials tested by Oak Ridge National Laboratory as presented in
Figure 2-1

Information on materials tested by Oak Ridge National Laboratory for thermal conductivity
as a function of pressure as presented in figure 2

1) Beverly Fume Silica
   density tested: 0.1809 g/cm3

   SOURCE: Amorphous   Silica
               Interlake, Inc.
               Globe Metallurgical Division
               Beverly, OH

2) Selma Grey Silica

   density tested: 0.361 g/cm3

   SOURCE: Amorphous Silica
               Alabama Metallurgical Corporation (ALAMET)
               P. O. Box 348
               Selma, AL   36701-0348

3) Aluminum oxide microspheres

   density: 2.265 g/cm3

   particle size: 500 microns

   SOURCE: General Atomic Company
               San Diego, CA

Appendix B

Product information provided by manufacturer on perlite powder used in experimentation

Material Safety Data Sheet

May be used to comply with
OSHA's Hazard Communication Standard,
29 CFR 1910.1200. Standard must be
consulted for specific requirements.

U.S. Department of Labor

Occupational Safety and Health Administration
(Non-Mandatory Form)
Form Approved
OMB No. 1218-0072

IDENTITY (As Used on Label and List) HARBORLITE	Note: Blank spaces are not permitted. If any item is not applicable, or no information is available, the space must be marked to indicate that.

Section I

Manufacturer's Name HARBORLITE CORPORATION	Emergency Telephone Number (616)649-1352 – (619)745-5900
Address (Number, Street, City, State, and ZIP Code) 1950 EAST "W" AVE. P.O. BOX 100	Telephone Number for Information (616)649-1352
VICKSBURG, MI 49097-0100	Date Prepared 6/19/92
	Signature of Preparer (optional)

Section II — Hazardous Ingredients/Identity Information

Hazardous Components (Specific Chemical Identity; Common Name(s))	OSHA PEL	ACGIH TLV	Other Limits Recommended	% (optional)

NO DETECTABLE HAZARDOUS COMPONENTS. ALL HARBORLITE PRODUCTS ARE

CLASSIFIED AS AMORPHOUS ALUMINA SILICATE. TYPICAL CHEMICAL ANALYSIS:

SiO_2 77.40%; Al_2O_3 12.79%; Fe_2O_3 0.45%; MgO 0.17%; CaO 0.56%; Na_2O 3.12%;

K_2O 5.41%; TiO_2 .049%; SrO .002%; ZrO_2 .002%; MnO .002%

DUST FROM HARBORLITE PERLITE PRODUCTS HAS BEEN ASSIGNED:

ACGIH TLV RATING OF $10mg/M^3$.

OSHA PEL RATING OF $15mg/M^3$ TOTAL DUST, OR $5mg/M^3$ RESPIRABLE DUST.

NO DETECTABLE CRYSTALLINE SILICA, USING XRAY DIFFRACTION AND

MICROSCOPIC ANALYSIS.

C.A.S. NO. 93763-70-3

Section III — Physical/Chemical Characteristics

Boiling Point	SOLID	Specific Gravity (H_2O = 1) TRUE – ALL PRODUCTS 2.28 BULK Sp.GR.-EXPANDED PRODUCTS .08-.20
Vapor Pressure (mm Hg.)	SOLID	Melting Point 2400°F
Vapor Density (AIR = 1)	SOLID	Evaporation Rate (Butyl Acetate = 1) NIL

Solubility in Water
NON SOLUBLE IN WATER AND MOST ACIDS

Appearance and Odor
UNEXPANDED PRODUCTS:GREY SANDY OR POWDERY/NO ODOR;EXPANDED PRODUCTS-WHITE/
NO ODOR.

Section IV — Fire and Explosion Hazard Data

Flash Point (Method Used) NONE	Flammable Limits NON FLAMMABLE	LEL NONE	UEL NONE

Extinguishing Media
NOT APPLICABLE

Special Fire Fighting Procedures
NOT APPLICABLE

Unusual Fire and Explosion Hazards
NONE

Section V — Reactivity Data

Stability	Unstable		Conditions to Avoid	
	Stable	X	NONE	

Incompatibility (Materials to Avoid)
NONE

Hazardous Decomposition or Byproducts
NONE

Hazardous Polymerization	May Occur		Conditions to Avoid .	
	Will Not Occur	X	NONE	

Section VI — Health Hazard Data

Route(s) of Entry:	Inhalation? YES	Skin? NO	Ingestion? NO

Health Hazards (Acute and Chronic)
NONE   ACGIH DOCUMENTATION STATES "...NO DATA...FOUND...TO INDICATE THAT EXPOSURE TO PERLITE DUST...HAS RESULTED IN ADVERSE PHYSIOLOGICAL EFFECTS."

Carcinogenicity: NONE	NTP? N/A	IARC Monographs? N/A	OSHA Regulated? NO

Signs and Symptoms of Exposure
NONE

Medical Conditions Generally Aggravated by Exposure PERSONS SENSITIVE TO INERT DUST MAY EXPERIENCE COUGHING WHEN EXPOSED TO HEAVY CONCENTRATIONS OF AIRBORNE MATERIAL.

Emergency and First Aid Procedures IF DUST PARTICLES LODGE IN THE EYES, USE STANDARD EYE WASH SOLUTIONS OR WATER, AND ALLOW EYES TO CLEAR.

Section VII — Precautions for Safe Handling and Use

Steps to Be Taken in Case Material Is Released or Spilled
SWEEP OR VACUUM AND DISPOSE OF AS FOR ANY INERT, NON-CARCINOGENIC SOLID WASTE.

Waste Disposal Method
LANDFILL, SOIL ADDITIVE, OR IF NOT CONTAMINATED WITH UNSUITABLE FOREIGN MATTER MAY BE INCORPORATED IN ANIMAL FEED FORMULATIONS AS INERT FILLER,

Precautions to Be Taken in Handling and Storing   OR MAY BE USED IN OTHER INERT FILLER APPLICATI
AVOID EMPTYING BAGS IN WINDY AREAS TO MINIMIZE DUSTING.

Other Precautions
NONE

Section VIII — Control Measures

Respiratory Protection (Specify Type)   MSA DUSTFO
RESPIRATORY PROTECTION SUITABLE FOR INERT, NON-CARCINOGENIC DUSTS:77 or EQUIVALE

Ventilation	Local Exhaust APPLY NEGATIVE PRESSURE AIR AT DUMP STATIONS TO ENTRAIN AIRBORNE DUST.	Special
	Mechanical (General) -EASILY TRANSPORTED PNEUMATICALLY	Other NONE

Protective Gloves NOT REQUIRED	Eye Protection NOT REQUIRED

Other Protective Clothing or Equipment
NOT REQUIRED

Work/Hygienic Practices   NORMAL GOOD HOUSEKEEPING IS ADEQUATE.

Appendix C

Product information provided by manufacturer on Sipernat 50S Precipitated Silica

used in experimentation

NORTH AMERICA SILICA COMPANY
11 Executive Mall
Post Office Box 840
Valley Forge, PA 19482
(215) 293-7400

Material Safety Data Sheet

I.   PRODUCT INFORMATION

Trade Name: Sipernat® 50S Precipitated Silica

Chemical Name: Synthetic amorphous silicon dioxide hydrate

CAS Registry No.:   112926-00-8

II.   SUMMARY OF HAZARDS

May cause eye and skin irritation. Causes irritation to lungs and respiratory tract. If inhaled, immediately remove to fresh air. In case of eye contact, immediately flush eyes with plenty of water for at least 15 minutes, call a physician. Flush skin with water. See Section V for additional information on health hazards.

III.   HAZARDOUS INGREDIENTS

A new CAS registry number has been assigned to Amorphous Precipitated Silica which will differentiate it from crystalline forms of silica. The new CAS number is as shown, 112926-00-8, changed from 7631-86-9.

Name	CAS No.	%	TLV®
Amorphous silica	112926-00-8	88%	10mg/m^3

Not listed by OSHA, NTP, or IARC as a carcinogen.

IV.   CHEMICAL AND PHYSICAL PROPERTIES

APPEARANCE:	Dry, white powder	SPECIFIC GRAVITY:	N/A
SOL. IN WATER:	Negligable	VAPOR PRESSURE:	N/A
EVAPORATION RATE:	N/A	BOILING POINT:	N/A
VAPOR DENSITY:	N/A	pH:	N/A

V. HEALTH HAZARD DATA & FIRST AID PROCEDURES

Amorphous silica, unlike crystalline silica, is considered biologically benign.

EYE CONTACT:	May cause eye irritation.
SKIN CONTACT:	May cause skin irritation.
INHALATION:	Causes irritation to lungs and respiratory tract.
CHRONIC HAZARDS:	No known chronic hazards.
SIGN AND SYMPTOMS OF EXPOSURE:	Sneezing and dryness mucous membranes (inhalation). May cause dry, chapped skin (skin exposure). May cause redness and tearing (eye exposure).
MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:	Skin diseases (dermatitis), asthma and lung diseases.

VI. EXPOSURE CONTROL MEASURES

GLOVES:	Tightly woven cotton, plastic, or rubber.
EYE PROTECTION:	Chemical goggles are recommended.
RESPIRATORY PROTECTION:	Use NIOSH approved dust respirator where dust occurs.
ENGINEERING CONTROL:	Use with adequate ventilation.
OTHER PROTECTIVE EQUIPMENT:	Safety shower and eyewash fountain should be within direct access.
PERSONAL HYGIENE:	Avoid breathing dust. Wash thoroughly after handling.

VII. REACTIVITY DATA

STABILITY:	Stable
CONDITIONS TO AVOID:	N/A
INCOMPATIBILITY:	N/A
HAZARDOUS DECOMPOSITION PRODUCTS:	None

VIII. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT: Noncombustible

FLAMMABLE LIMITS: N/A

EXTINGUISHING METHOD: N/A

FIRE FIGHTING
   INSTRUCTIONS: N/A

UNUSUAL FIRE AND
   EXPLOSION HAZARDS: None

IX. ENVIRONMENTAL & DISPOSAL INFORMATION

ENVIRONMENTAL HAZARDS: No known adverse effects. Not a listed toxic chemical under SARA Title III, §302, §304, or §313.

SPILLAGE: Sweep, scoop, or vacuum discharged material.

WASTE DISPOSAL METHOD: Landfill according to local, state, and federal regulations. Not a hazardous waste under RCRA regulations.

X. SUBSTANCES FOR WHICH STANDARDS HAVE BEEN SET

COMPONENT: Amorphous Silica/Percent: 88% as SiO_2

OSHA Exposure Limit: 20mppcf (5mg/m^3) SiO_2 Respirable dust. 8 hour time weighted average.

TLV®:   10mg/m^3 Total dust; 5mg/m^3 respirable.

EXPOSURE ANALYSIS METHODS: NIOSH Manual of Analytical Methods, 3rd ed., Method 7501, (1984).

XI. SOURCE OF INFORMATION

John G. Blumberg, Product Safety Coordinator, 10/19/89

Appendix D

Product information provided by manufacturer on Micro-sheet glass used in prototype production

Materials Business
Corning Glass Works
Corning, New York 14831

Product Information

Product Benefits

Micro-sheet is drawn by a unique process which imparts a practically flawless, fire-polished surface which is suitable for many applications without the grinding and polishing usually associated with other glasses. In addition, Micro-sheet offers all the permanence, protection and transmittance of glass.

Perhaps the most unusual feature of Micro-sheet is its exceptional thinness and the uniformity to which it is held. The table under "Available Product Forms" lists these thicknesses.

Glass 0211, altho... n containing alkali, has a soda content approximately one half of that found in soda lime glasses. This glass when subjected to a soda extraction test yields values that are nominally 25% of those measured for soda lime glasses.

Chemical Durability

Micro-sheet (Glass Code 0211) has been given chemical durability tests in which it was compared to Code 7740, a chemically resistant borosilicate glass, and to Code 0080, a common soda lime glass.

	Weight Loss, mg/cm^2		
Glass Code	5% NaOH 6 Hrs. at 95°C	N/50 Na$_2$CO$_3$ 6 Hrs. at 95°C	5% HCl 24 Hrs. at 95°C
0211	2.5	0.10	0.03
0080	0.5	0.13	0.02
7059	3.7	0.30	5.50
7740	1.1	0.12	0.005

After one year's stora...o under normal conditions, Micro-sheet shows no evidence of surface weathering. In a controlled atmosphere (9...% Relative Humidity, 110°F), Micro-sheet stacked without paper separation showed no tendency to stick together, and the surface showed only slight hazing.

Optical Qualities

Micro-sheet has good transmittance of the near ultraviolet, excellent transmittance of the visible, and very good transmittance in the near infrared regions.

Micro-sheet is flat. It has a minimum of bow or wave to cause distortion or reflection. The glass is "water white" and, therefore, is not color selective.

Typical Applications

A few applications for Micro-sheet are suggested below, but the bulk of the applications for it will be limited only by your imagination.

Cover Glass
Photoconductors
Thin & Thick Film Substrates
Touch Panels

Protective Covers
Magnetic Film Memory Devices
Flat Panel Display Substrates

Available Product Forms

Micro-sheet is available in full size as drawn sheets with ends as cut at the draw. In addition, smaller sizes cut from a full sheet can be supplied. The standard sizes and thicknesses are as follows:

Thickness (in.)								
Min.	0.0020	0.0033	0.0051	0.0063	0.0075	0.0098	0.0130	0.0180
Max.	0.0033	0.0051	0.0063	0.0075	0.0098	0.0138	0.0190	0.0240
Reference Number	00	0	1	1½	2	3	3½	4
Full Sheet Size								
Width (In.)	15¼	15½	16	16	16	16½	16½	16½
Length (In.)	14-16	14-16	14-16	14-16	14-16	14-16	14-16	14-16
Minimum Quality Area (Sq. In.)	156	156	169	169	169	169	169	169
Width (In.)	12	12	13	13	13	13	13	13
Length (In.)	13	13	13	13	13	13	13	13
Product Code Number	580097	580107	580108	580109	580120	580121	583736	580212

Description

Glass Type — potash, soda, zinc borosilicate
Color — water-white clear
Forms available — drawn sheet, powder
Principal uses — microscope cover glass, thin film substrate

Properties

Mechanical	Metric	English
Density	2.53 g/cm^3	157.9 lb/ft^3
Young's Modulus	7.59 x 10^3 kg/mm^2	10.8 x 10^6 psi
Poisson's Ratio	0 22	
Shear Modulus	3.09 x 10^3 kg/mm^2	4.4 x 10^6 psi
Knoop Hardness (KHN$_{100}$)	458	

Viscosity

	Metric	English
Working Pt. (10^4 poises)	1008°C	1846°F
Softening Pt. (10$^{7.6}$ poises)	720°C	1328°F
Annealing Pt. (10^{13} poises)	550°C	1022°F
Strain Pt. (10^{14} poises)	508°C	946°F

Thermal

	Metric	English
Coefficient of Expansion (0-300°C)	73.8 x 10^{-7}/°C	41.0 x 10^{-7}/°F
(25°C to Set Point 513°C)	84.0 x 10^{-7}/°C	46.7 x 10^{-7}/°F
Thermal Conductivity, 0°C	.0023 $\frac{cal\ cm}{sec\ cm^2\ °C}$	.54 $\frac{Btu\ ft}{h\ ft^2\ °F}$

Optical

	Metric
Refractive Index (589.3nm)	1.523
Transmission @ 420 nm	92%*
2200 nm	92%*

*Through a sample thickness 1 0 mm

Electrical

|	
Log$_{10}$ Volume Resistivity @ 250°C	8 3 ohm-cm
@ 350°C	6.7 ohm-cm
Dielectric Constant @ 20°C; 1 MHz	6.7
Loss Tangent @ 20°C; 1 MHz	.46%

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Chemical

Weathering   *   1
Acid Durability   ** 2

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*   Weathering is defined as corrosion by atmospheric-borne gases and vapors such as water and carbon dioxide Glasses rated 1 will almost never show weathering effects. those rated 2 will occasionally be troublesome, particularly if weathering products cannot be removed; those glasses rated 3 require more careful consideration

** The Acid Durability column classifies glasses according to their behavior in 5% hydrochloric acid at 95°C (203°F) for 24 hours

Classification	Thickness Loss (in.)
1	$< \quad 10^{-6}$
2	$10^{-6} - 10^{-5}$
3	$10^{-5} - 10^{-4}$
4	$> \quad 10^{-4}$

Values are listed with four degrees of accuracy Those that are underscored (e g. $\underline{2}$) result from recent determination and are reliable Values not underscored are estimates offered with confidence When two values are listed with one underscored, this indicates the range within which the true value lies the underscored value is the more probable one (e.g 2-$\underline{3}$). A question mark indicates considerable uncertainty
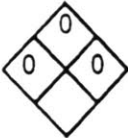
VISCOSITY
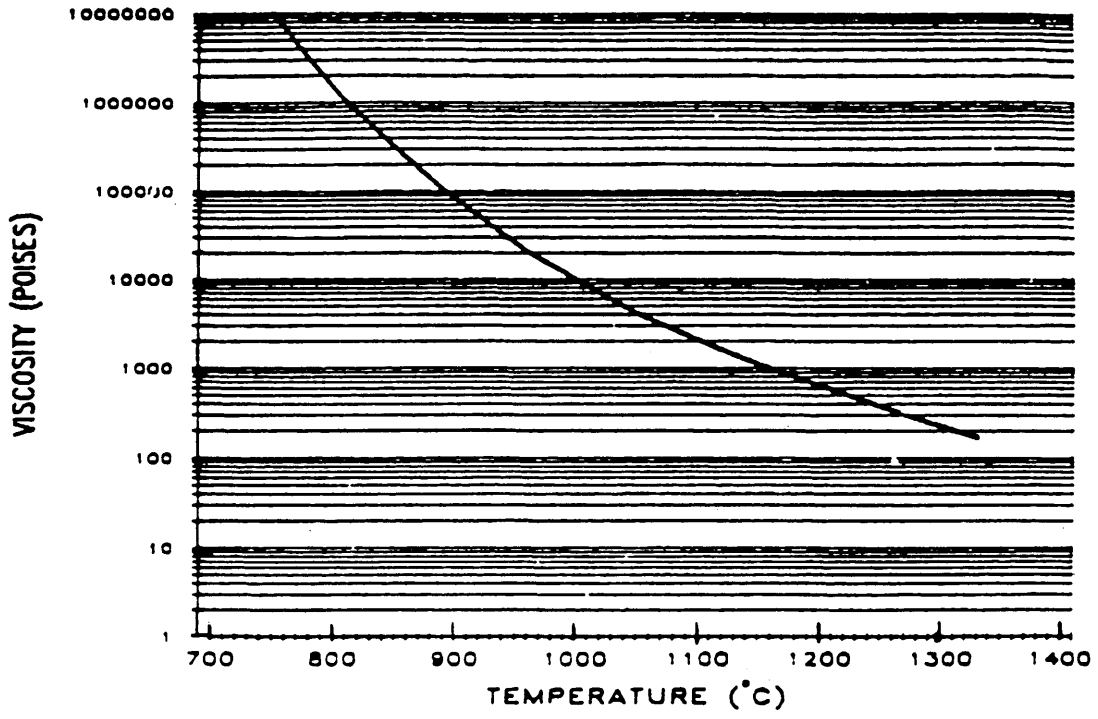
THERMAL EXPANSION
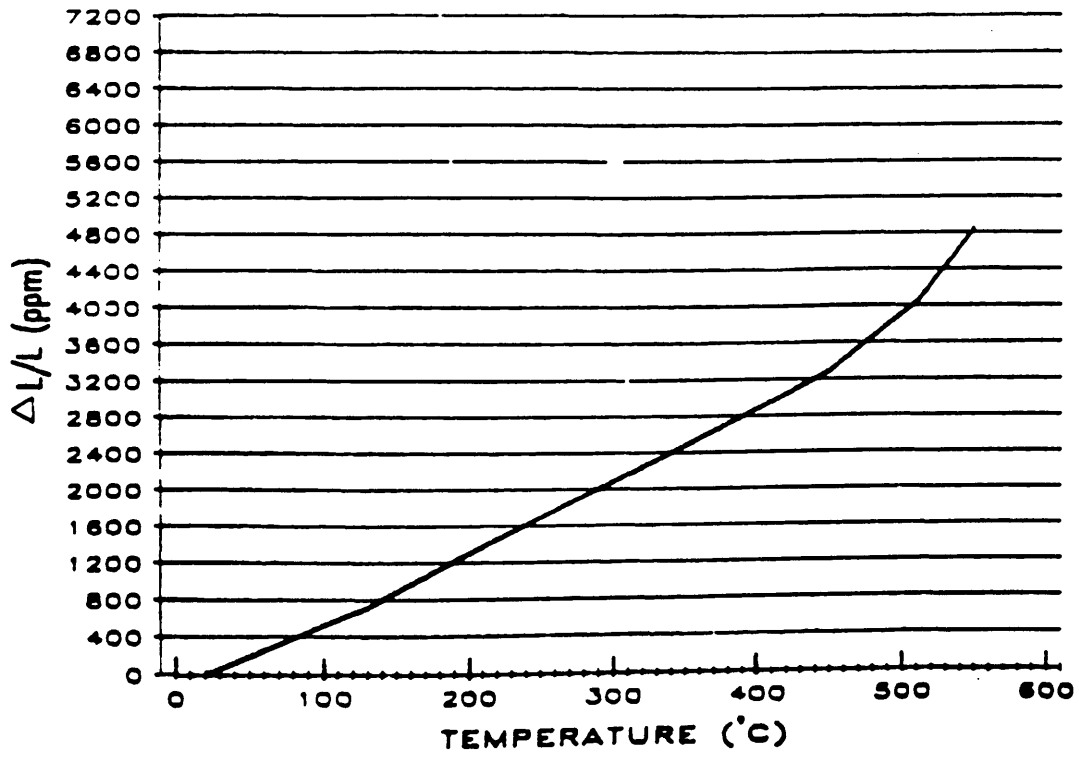
Appendix E

Product information provided by manufacturer on Boron Nitride coating used in experimentation

24-HOUR EMERGENCY ASSISTANCE	GENERAL ASSISTANCE	NFPA FIRE HAZARD SYMBOL*
BP America (In Ohio): 800-362-8059 (Outside Ohio): 800-321-8642 CHEMTREC Assist: 800-424-9300	716-278-2000	Flammability 4-Extreme 3-High 2-Moderate Health Reactivity Special Hazards 1-Slight 0-Insignificant *-See Text
MSDS Number > **407**		

MANUFACTURER/SUPPLIER: The Carborundum Company - Electronic Ceramics Division
ADDRESS: P.O. Box 337, Niagara Falls, NY 14302

PRODUCT IDENTIFICATION

TRADE NAME:

COMBAT ® BORON NITRIDE COATING: TYPE S

CAS NUMBER:	MIXTURE
SYNONYM(S):	ND
CHEMICAL FAMILY:	NITRIDES/ALUMINA
MOLECULAR FORMULA:	NA
MOLECULAR WEIGHT:	ND
PRODUCT CODE:	NA

HIERARCHY: NA

PRODUCT HAZARD SUMMARY

HEALTH   MAY BE IRRITATING TO SKIN AND EYES
                DUSTS FROM DRIED MATERIAL MAY CAUSE RESPIRATORY TRACT IRRITATION

REACTIVITY   **STABLE**

PRODUCT HEALTH HAZARD INFORMATION

INGESTION:
May cause gastrointestinal disturbances. Symptoms may include irritation, nausea, vomiting and diarrhea.

SKIN:
SLIGHTLY TO MODERATELY IRRITATING. Repeated or prolonged skin contact may cause reddening, itching and inflammation.

EYE:
SLIGHTLY TO MODERATELY IRRITATING. Abrasive action may cause damage to the outer surface

of the eye.

INHALATION:
Dusts from dried product may cause respiratory tract irritation.

SPECIAL TOXIC EFFECTS:
Powder from dried product considered a "nuisance" dust. Excessive airborne concentrations of nuisance dusts may seriously reduce visibility, cause unpleasant deposits in the eyes, ears and nasal passages and cause irritation to skin or mucous membranes by chemical or mechanical action.

FIRST AID

INGESTION:
Do not induce vomiting. Get medical attentio if irritation persists.

SKIN CONTACT:
Wash area of contact thoroughly with soap and water. Get medical attention if irritation persists.

EYE CONTACT:
Flush immediately with large amounts of water for at least 15 minutes. Eyelids should be held away from the eyeball to ensure thorough rinsing. Get medical attention if irritation persists.

INHALATION:
Remove affected person from source of exposure. Get medical attention if irritation persists.

PERSONAL PROTECTION INFORMATION

EYE PROTECTION:
Avoid eye contact with this material. Wear safety glasses. Do not wear contact lenses when working with this substance.

SKIN PROTECTION:
Avoid skin contact. When working with this substance, wear appropriate chemical protective gloves. Depending upon conditions of use, additional protection may be necessary such as face shield, apron, armcovers, etc.

RESPIRATORY PROTECTION:
If exposure limits are exceeded or if irritation is experienced, NIOSH approved respiratory protection should be worn. NIOSH approved respirator for particulates with a TLV of greater than 0.05 mg/M3 is generally acceptable except supplied air respirators are required for high airborne dust concentrations. Ventilation and other forms of engineering controls are often the preferred means for controlling chemical exposures. Respiratory protection may be needed for non-routine or emergency situations.

PHYSICAL PROPERTIES

BOILING POINT:	107.000 C (225 F)
SPECIFIC GRAVITY:	1.800- 2.100
MELTING POINT:	NA
% VOLATILE:	60.000- 70.000

ND = No Data
NA = Not Applicable

VAPOR PRESSURE: NA
EVAPORATION RATE (WATER=1):   NA
VAPOR DENSITY (AIR=1):   NA
VISCOSITY: NA
% SOLUBILITY IN WATER: 100.000
OCTANOL/WATER PARTITION COEFFICIENT: ND
POUR POINT: NA
pH: 5.000- 6.000
APPEARANCE/ODOR: ODORLESS WHITE PASTE.

FIRE AND EXPLOSION DATA

FLASH POINT: NA
AUTOIGNITION TEMPERATURE: NA
FLAMMABILITY LIMITS IN AIR (% BY VOL.) LOWER: NA
FLAMMABILITY LIMITS IN AIR (% BY VOL.) UPPER:

BASIC FIREFIGHTING PROCEDURES:
  This material does not give a flash point by conventional test methods. Use extinguishing
  agent suitable for type of surrounding fire.

UNUSUAL FIRE AND EXPLOSION HAZARDS:
  NA

REACTIVITY DATA

STABILITY/INCOMPATIBILTY:
  Avoid contact with strong oxidizers.

HAZARDOUS REACTIONS/DECOMPOSITION PRODUCTS:
  Contact with strong oxidizing agents may produce flammable and toxic ammonia gas.

ENVIRONMENTAL INFORMATION

SPILL OR RELEASE TO THE ENVIRONMENT:
  No special procedures are required for clean-up of spills or leaks of this material.
  Avoid methods that result in water pollution. Caution should be exercised regarding
  personnel safety and exposure to the spilled material, as set forth elsewhere in this data
  sheet.

WASTE DISPOSAL:
  This substance, when discarded or disposed of, is not specifically listed as a hazardous
  waste in Federal regulations; however it could be hazardous if it is considered toxic,
  corrosive, ignitable, or reactive according to Federal definitions (40 CFR 261).
  Additionally, it could be designated as hazardous according to state regulations. This
  substance could also become a hazardous waste if it is mixed with or comes in contact with
  a hazardous waste. If such contact or mixing may have occurred, check 40 CFR 261 to
  determine whether it is a hazardous waste. If it is a hazardous waste, regulations at 40
  CFR 262, 263, and 264 apply.

  The transportation, storage, treatment, and disposal of this waste material must be
  conducted in compliance with all applicable Federal, state, and local regulations.

ND = No Data   407/Page 3 of 5
NA = Not Applicable

SARA TITLE III INFORMATION:

Listed below are the hazard categories for the Superfund Amendments and Reauthorization Act (SARA) Section 311/312 (40 CFR 370):

Immediate Hazard: -_ Delayed Hazard: -_ Fire Hazard: -_ Pressure Hazard: -_ Reactivity Hazard: -_

ADDITIONAL ENVIRONMENTAL REGULATORY INFORMATION:

There may be specific regulations at the local, regional or state level that pertain to this material.

SPECIAL PRECAUTIONS/SUPPLEMENTAL INFORMATION

HANDLING/STORAGE:

Avoid freezing of product during storage.

TRANSPORTATION REQUIREMENTS

D.O.T. PROPER SHIPPING NAME (49 CFR 172.101):   NA
D.O.T. HAZARD CLASS (49 CFR 172.101):   NA
UN/NA CODE (49 CFR 172.101):   NA
BILL OF LADING DESCRIPTION (49 CFR 172.202):   PRODUCT NAME
D.O.T. LABELS REQUIRED (49 CFR 172.101):   NA
D.O.T. PLACARDS REQUIRED (49 CFR 172.504):   NA

INGREDIENTS/HEALTH HAZARD INFORMATION

COMPONENT	CAS NO.	%	EXPOSURE LIMITS - REF.
Boron nitride	10043-11-5	20-30	10 mg/M3 (total) TLV (ACGIH), as as nuisance dust 5 mg/M3 (resp.); 15 mg/M3 (total) PEL (OSHA)
alpha-Alumina	1344-28-1	5-10	10 mg/M3 (total) TLV (ACGIH), as Al 5 mg/M3 (resp.); 10 mg/M3 (total) PEL (OSHA), as Al
Water	7732-18-5	60-70	None established
Remaining components not determined hazardous and/or hazardous components present at less than 1.0% (0.1% for carcinogens).	NA	Trace	NA

ND = No Data
NA = Not Applicable