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A STUDY OF MATERIALS FOR A BROADBAND MILLIMETER-WAVE QUASI-OPTICAL VACUUM WINDOW

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Introduction

Cryogenic millimeter-wave receivers require a low-loss vacuum window through which the incoming beam can pass on its way to the cold feed horn. Thick dielectric plates have been used as windows, but their loss can contribute significantly to the receiver noise temperature, and their useful bandwidth is limited even when matching grooves or dielectric matching layers are used. A simpler approach, which has been in use for many years at Berkeley [1] and Caltech [2], is to use a thin sheet of mylar (polyethylene terephthalate) as a vacuum window. Atmospheric pressure causes such a window to balloon alarmingly, but the great strength and flexibility of mylar are sufficient in many applications so long as no sharp object pricks the window (in which event the results could be hazardous). Radiation cooling of the mylar film by the cold innards of the dewar can cause condensation of water on the outside of the window, which can contribute significant electrical loss. To prevent this, the mylar can be dried by air from a small fan.

This report describes a window in which a thin plastic film vacuum barrier is supported by a thick slab of low density dielectric foam. The dielectric foam bears the full atmospheric pressure, and also acts as an infrared filter to reduce radiative cooling of the plastic film and radiative warming of the cold contents of the dewar. This scheme was inspired by the window for circular waveguide described of Bradley and Norrod [3, 4], which uses a plug of polystyrene foam inside the waveguide and a mylar film glued on the atmospheric side of the window to prevent moisture absorption by the foam over long periods of time.

The Vacuum Barrier

Table I lists the properties of some plastic films which might be suitable for use as a vacuum barrier in the present application. The high strength and low permeability to water vapor and atmospheric gases of the HR500/2S material make it appear the most attractive. This material, manufactured for food packaging by Hercules, Inc. [5], is a laminate of biaxially oriented polypropylene with 0.0001" layers of polyvinylidene chloride on both sides. It is available in 0.001" and 0.00075" thicknesses.

The water vapor and gas permeabilities of even the best of these films are not zero. However, experience at several observatories indicates that even mylar windows, with their high water vapor transmission rate, are satisfactory for extended periods of operation. This is presumably a result of the natural ability of cryogenic systems to maintain a good vacuum by cryopumping. The window leak rate may be significant, however, for large windows, or for multi-band receiver packages with many windows such as will be used on NRAO's proposed Millimeter Array. Just how much water and gas a receiver can cryopump before difficulties arise is not known. Accumulation of ice on cold quasi-optical components or inside feed horns may eventually degrade receiver performance, or an accumulation of frost may reduce the cryopumping capability of the system.

All the low loss plastic films we have tested have had substantial He leak rates [6]. This may have serious long-term consequences in applications where there is a high background level of He. It has been reported, for example, that boil-off from a liquid He cooled bolometer caused a nearby 15 K receiver to warm up [7]. Such problems should be easy to avoid with appropriate ventilation, or venting of boiled off He.

Fig. 1 shows the theoretical insertion loss and return loss of several thicknesses of film with ϵ_r = 3 (corresponding to mylar), as functions of frequency. Measurements of 1 mil and 2 mil mylar films at 230 and 260 GHz are also shown, and are seen to agree reasonably with the theoretical values. Fig. 2 shows the measured insertion loss of some plastic film windows at 230 and 260 GHz.

The Foam Support — Attenuation

Electrically, low density polystyrene foam is an almost ideal material for a millimeter-wave window. When the present work began in 1989, extruded polystyrene foam was available in several densities from Emerson & Cuming [8] as the Eccofoam PS series of materials, specified by relative dielectric constant, and also from Dow [9] as their Styrofoam Hiload series of materials, specified by compressive strength. These materials were foamed using freon gas. Because of its harmful effect on the atmosphere, freon was replaced around 1989 with more benign gases (Dow now uses a hydro-chloro-fluorocarbon called Isotron-142b). We have tested the newer materials from several manufacturers, and found them much too lossy for use in millimeter-wave windows (typically 1 dB/in at 100 GHz). Recently, Emerson & Cuming have stopped selling the Eccofoam PS materials.

A suitable substitute for extruded polystyrene appears to be *expanded* polystyrene, which has a visibly beaded structure and is commonly used for hot drink cups and drink coolers. Expanded polystyrene is fabricated from beads initially containing the styrene monomer, benzene, and pentane as a blowing agent. The beads are "popped" in a vessel with high pressure steam. The newly formed foam contains a considerable amount of water from the steam, but this diffuses away and the material stabilizes mechanically during a 90-day ageing period [10]. The beads come in several sizes. The samples we have tested from Radva Corporation [10] are made with ARCO Dylite size B beads;

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sizes X and T are smaller. A concern with this material was that the millimeter sized cell structure might cause scattering of millimeter-wavelength beams. However, measurements of Radva material (batches 3 and 4) indicate only about 1% scattered power from a 2"-thick sample at 260 GHz [11].

The insertion loss of a number of plastic foam materials is shown in Figs. 3 and 4. Fig. 3 shows the loss of expanded and (old) extruded polystyrene foams of density < 2.0 lb/ft^3. It is clear that the density is the critical parameter in determining the loss. In Fig. 4 the insertion loss is shown for higher density polystyrene samples, and for a number of other foam materials. The method of measurement is described in Appendix A.

We note, from Fig. 3, that the insertion loss of polystyrene foam appears to rise quite rapidly at frequencies approaching 300 GHz. It is not known whether this is the skirt of a molecular resonance or is due to some other phenomenon. Further measurements will be made at higher frequencies in the near future as appropriate equipment becomes available. For practical purposes, the increase in loss with frequency can be offset, at least partially, by using smaller diameter (and therefore thinner) windows at higher frequencies.

The Foam Support — Mechanical

The shear strength of expanded polystyrene is shown as a function of density in Fig. 5 [12]. The corresponding values for *extruded* polystyrene are close to these. For minimum millimeter-wave loss, the window material should be chosen to have the lowest density consistent with sufficient mechanical strength to support a pressure differential of one atmosphere with a reasonable safety margin.

A circular window of diameter D in. and thickness t in., supported at its perimeter, when subjected to a differential pressure of 1 atm, will experience a shear stress at its perimeter of 3.68 D/t lb/in^2. For the window described below, D = 3 in, t = 1.125 in, and the shear stress is 9.8 lb/in^2. Allowing a factor of two safety margin, the shear strength of the foam should be ≥ 20 lb/in^2. From Fig. 5, this requires expanded polystyrene with a density ≥ 1.2 lb/ft^3.

Destructive tests on windows with the above dimensions, made with expanded polystyrene of density ~1.3 lb/ft^3, have demonstrated a bursting pressure of ~3 atm.

Construction of a Broadband Vacuum Window

Fig. 6 shows the details of the broadband vacuum window. The polystyrene foam has a grid of small channels machined on the atmospheric pressure side, connected to two small through holes. This is to ensure that gas or water vapor which leaks through the plastic film will find its way rapidly to the inside of the dewar, whence it will be cryopumped by the refrigerator. Without the breather holes, molecules leaking through the film would diffuse through the foam. Because the vacuum side of the foam is

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radiatively cooled, molecules would freeze out when they reached a sufficiently cold region of the foam. Over a long period, this could cause an increase in the loss of a window, as was observed in the circular waveguide window of Norrod and Bradley [3] before they added a plastic barrier.

The polystyrene foam plug in Fig. 6 is glued into the aluminum tube. Prior to gluing, the aluminum surface is abraded with #80 sandpaper, and then cleaned with methanol followed by petroleum ether. The adhesive is applied to both the inside of the aluminum tube and the outside of the polystyrene plug, which is then pushed into the tube. This procedure prevents voids forming during assembly. Details of the adhesives, mix ratios, and curing schedules are given in Appendix B.

The plastic film is glued to its aluminum ring under tension. This is accomplished by first gluing the film to a larger ring, then placing the smaller ring, coated with adhesive, in the middle. Prior to gluing, the aluminum rings are cleaned in methanol and petroleum ether, and the film in methanol only. To provide tension, a 200 gm weight is placed on the smaller ring during the cure. Details of the adhesives, mix ratios, and curing schedules are given in Appendix B.

Conclusion

A number of such windows have been made at NRAO over the last few years, and are in use in the lab and on all the SIS receivers on the 12-m Kitt Peak telescope. Most of these have used low loss Eccofoam PS 1.04 extruded polystyrene, which is no longer available, and 0.001" or 0.00025" mylar vacuum barriers. Newer windows, using expanded polystyrene and 0.00075" HR500/2S vacuum barriers, are now under long-term evaluation, and are expected to be used for all future receivers.

Acknowledgements

Sources of Materials

Material	Manufacturer	Supplier
Plastic Films		
HR500/2S	Hercules, Inc. Wilmington, DE 19894	Manufacturer
Mylar	Du Pont Co. Wilmington, DE 19898	Elec. Insulation Suppl. Richmond, VA 23223
		Cadillac Chem. & Plastics Virginia Beach, VA 23455
Polypropylene	Unknown	Prof. P. L. Richards U of California Berkeley, CA 94720
Dielectric Foams		
Eccofoam PS	Emerson & Cuming Canton, MA 02021	Manufacturer
Dylite	Atlantic Richfield Co. Philadelphia, PA 19102	Radva Corporation Radford, VA 24143
Hiload	Dow Chemical Co. Midland, MI 48674	Samples from manufacturer
Fomular	U.C. Industries Parsippany, NJ 07054	Samples from manufacturer
Space Shuttle Tile	Rockwell International Kennedy Space Center FL 32815	Samples from manufacturer
Ethafoam	Dow Chemical Co. Midland, MI 48674	Read Plastics Rockville, MD 20852
Trymer	Hibco Plastics Yadkinville, NC 27055	Samples from manufacturer
Adhesives		
Eccobond	Emerson & Cuming Canton, MA 02021	Manufacturer
Scotch-Weld	3M Adhesives Atlanta, GA 30360	Waco Inc. Sandston, VA 23150

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[8] Emerson & Cuming, Canton, MA 02021.

[9] Dow Chemical Co., Midland, MI 48674.

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[11] J. W. Lamb, NRAO. Private communication, July 1992.

[12] ARCO Chemical Co., "Expanded Polystyrene Properties," Atlantic
     Richfield Co., Philadelphia, PA 19102, 1984.

[13] Du Pont Co., Polymer Products Dept, Industrial Films Div., Wilmington,
     DE 19898, *Ideas of Mylar Polyester Film*, Publication #E-41394
     (undated).

[14] *Handbook of Plastics and Elastomers*, C. A. Harper, Ed., McGraw-Hill,
     1975, pp. 1-101 to 1-102, 9-28. Note that the unit of permeability in
     Table 16 on p. 9-28 is gm/mil/(100 in^2)/24 hrs, and not gm/mil/in^2/24 hrs
     as stated.

[15] 3M Adhesives, Coatings and Sealers Division, St. Paul, MN 55144-1000.

TABLE I — PROPERTIES OF PLASTIC FILMS

	PTFE	Polyethylene	Mylar	Polypropylene (biax. orient.)	HR500/2S note 3
Relative dielectric const.	2.1	2.26	3.0	2.55	~2.6
Tensile strength (psi)	>1500	>1500	>20000	>12000	25000
Water vapor trans. (note 1)	0.3	1	1.8	0.4	0.3
Oxygen permeability (note 2)	-	500	6	120	<1
Nitrogen perm. (note 2)	-	315	1	-	-

References: [5], [13], [14]
Note 1: gm/100 sq.in./mil/24 hrs at 90% RH.
Note 2: cc/100 sq.in./mil/24 hrs.
Note 3: Biaxially oriented polypropylene with 0.0001" layers of polyvinylidene chloride on both sides.
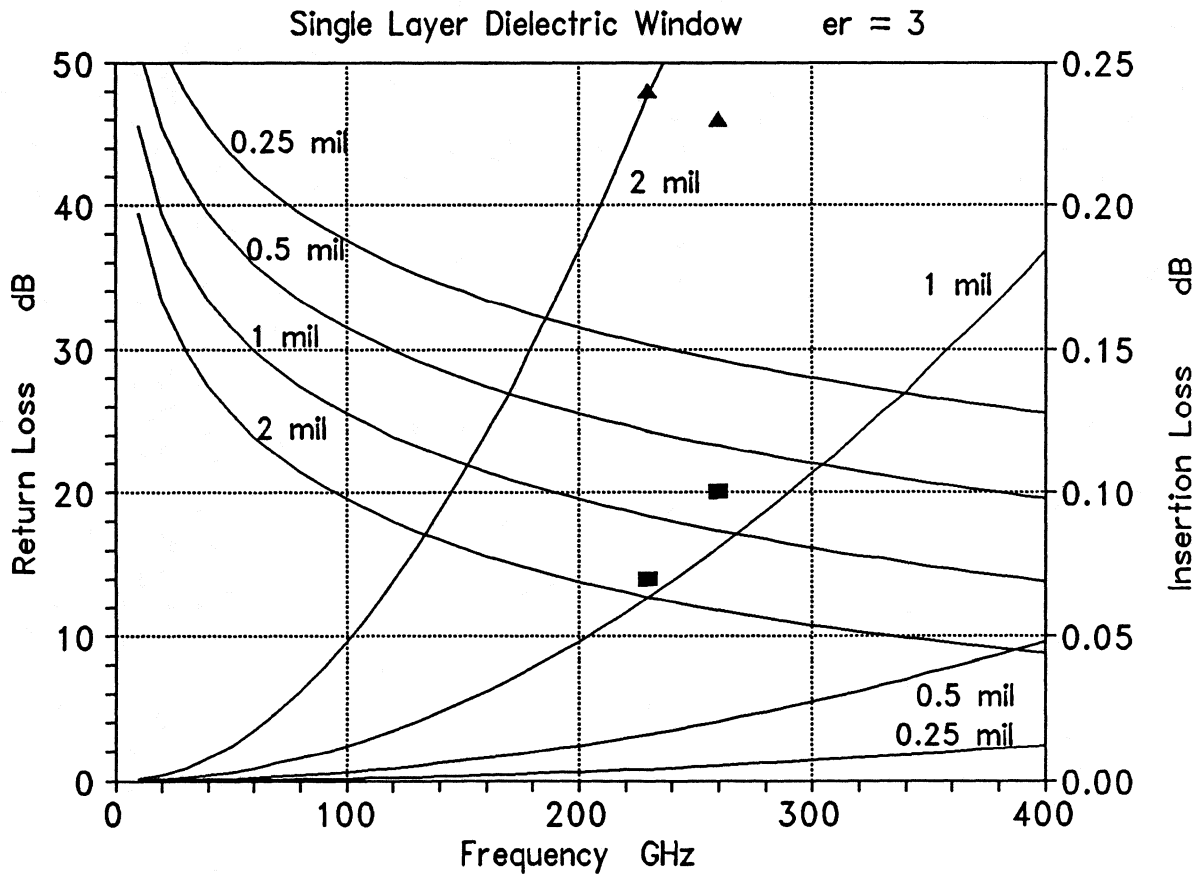
Fig. 1. Theoretical return loss and insertion loss vs. frequency for dielectric sheets with ϵ_r = 3 and thickness 0.00025", 0.005", 0.001", and 0.002". The points □ and ∆ are the measured insertion loss for 0.001" and 0.002" mylar.
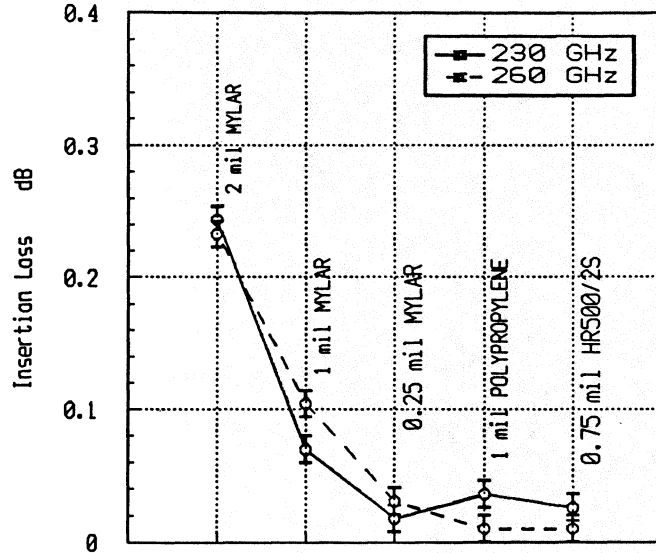
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Fig. 2. Measured loss of plastic films at 230 GHz (——) and 260 GHz (— — —).
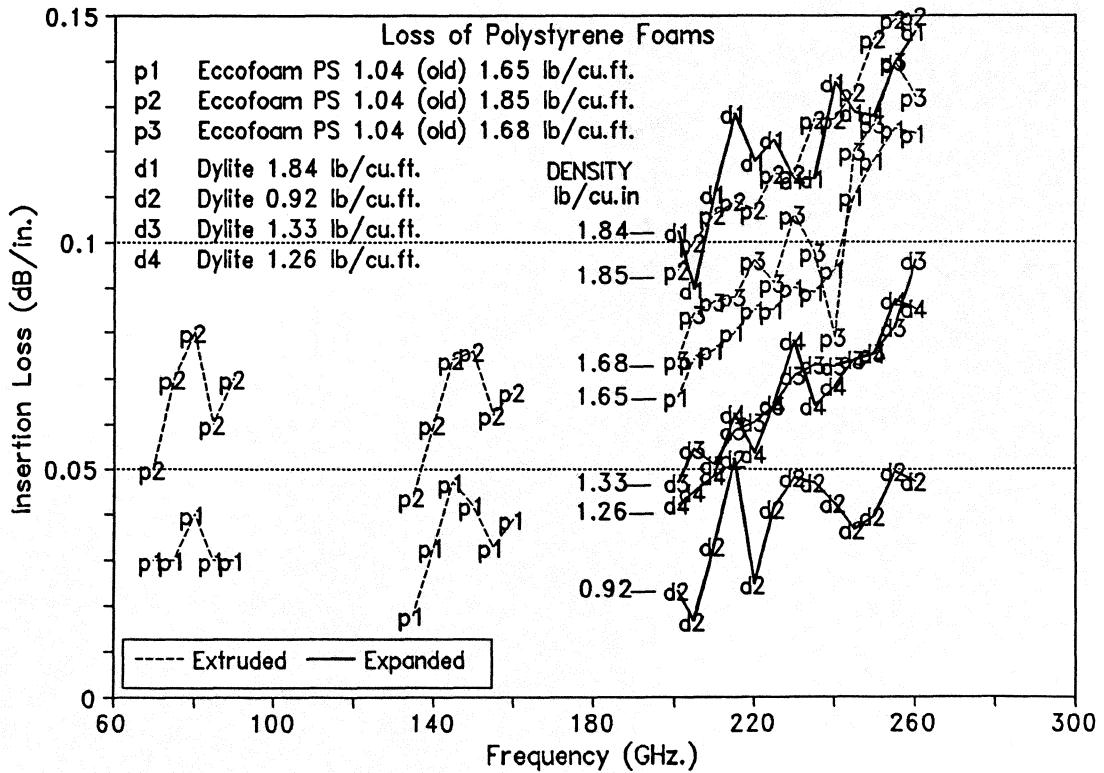

Fig. 3. Measured loss of samples of expanded (——) and extruded (— — —) polystyrene foam of
several densities. (p1-p3) = Eccofoam PS 1.04 batch 1-3; (d1-d4) = Dylite batch 1-4. It is clear
that the loss depends on density and not on whether the material is extruded or expanded.
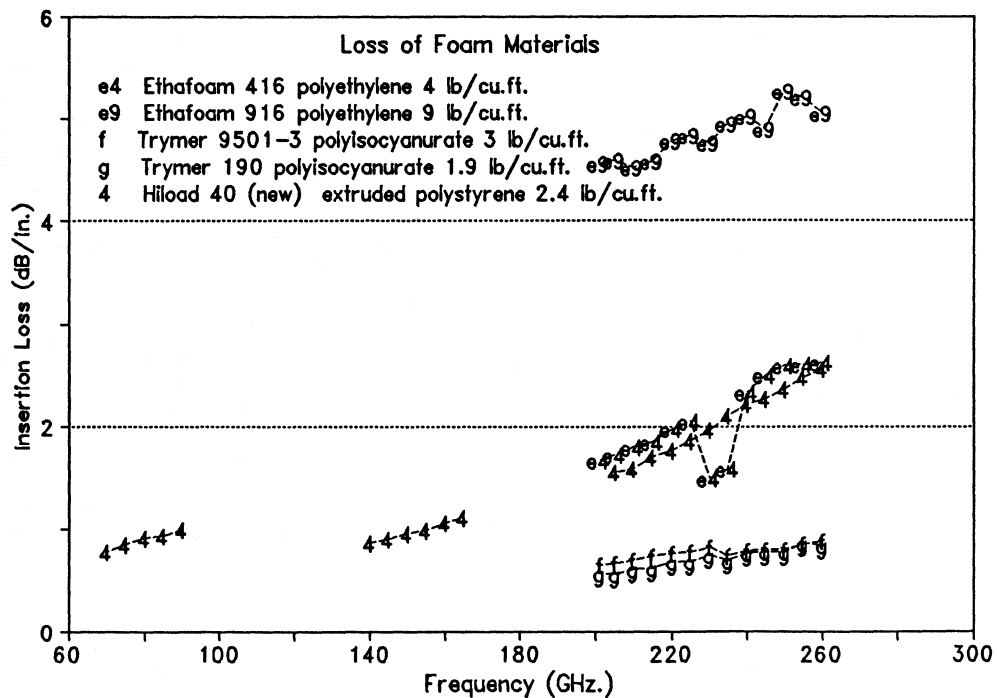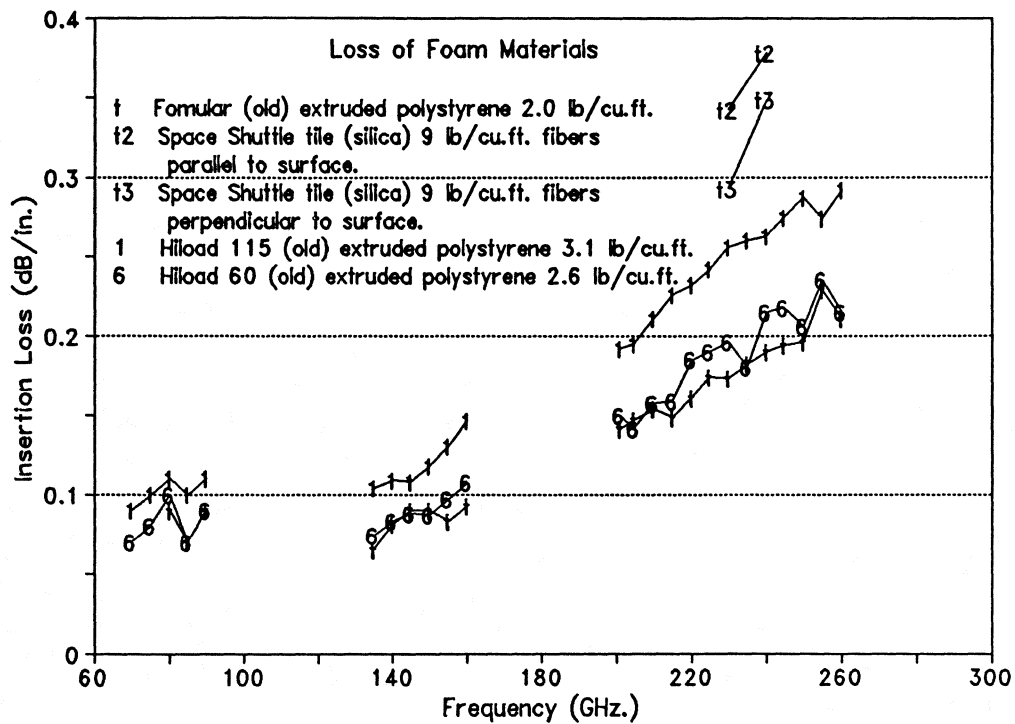
Fig. 4. Measured loss of higher density polystyrene foams and a number of other foam materials. Note the high loss of the (new) Dow Highload 40 extruded polystyrene (4) compared with the (old) higher density Highload 60 (6) and 115 (1).
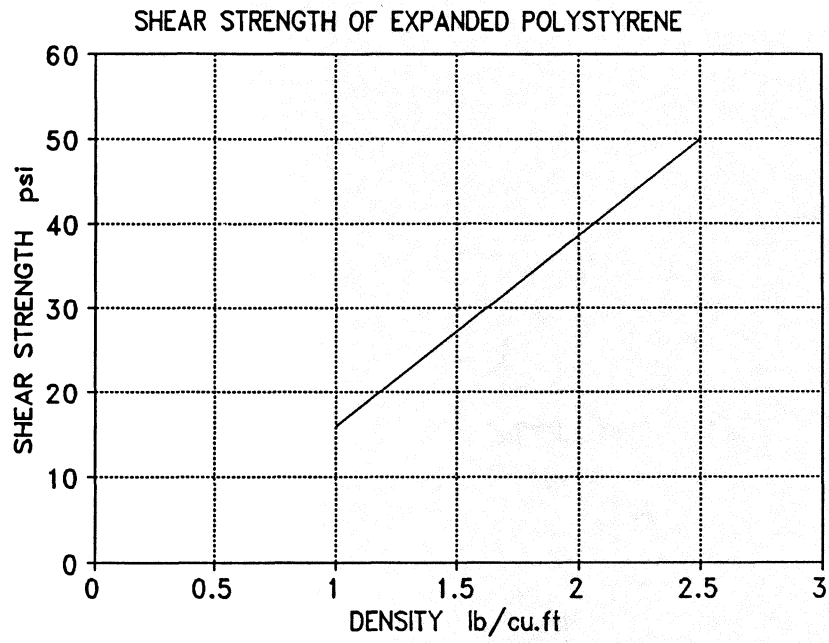
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SHEAR STRENGTH OF EXPANDED POLYSTYRENE

Fig. 5. Shear strength of expanded polystyrene as a function of density [12]. Extruded polystyrene is similar.
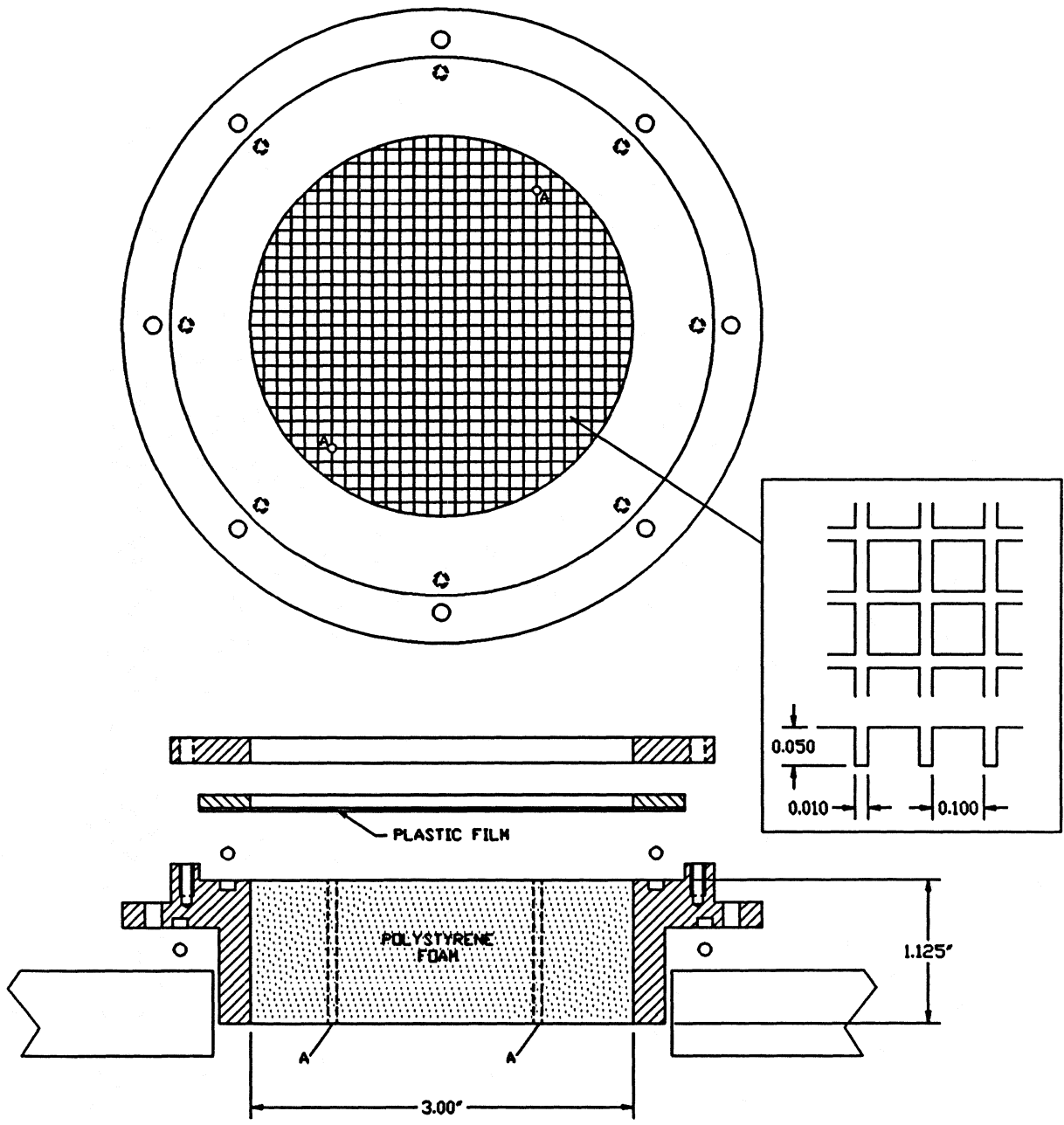
Fig. 6. Construction of the broadband vacuum window. The plastic film vacuum barrier is supported by the polystyrene foam plug. The small through holes "A" (1/16" dia.), connected to the grid of grooves in the upper surface of the foam, ensure that any gas molecules permeating the vacuum barrier are pumped away and do not condense at a cold region inside the foam.

APPENDIX A: Millimeter-Wave Loss Measurement

Measurements of insertion loss were made on samples of various films and foams using the method described below. It was found that the skin on some of the foam samples caused a substantial reflection loss. Samples were therefore deglazed by machining both sides where necessary.

Transmission loss measurements were made as shown in Fig. A1. The receiver output power was measured with the following loads in front of the receiver: (i) the liquid nitrogen cold load (output power P_1), (ii) the sample under test in front of the cold load (output power P_2), and (iii) the room temperature load (output power P_3). The two Y-factors $Y_1 = P_3/P_1$ and $Y_2 = P_2/P_1$ were calculated. If the sample is at room temperature, its loss is then

$$L = \frac{1 - 1/Y_1}{1 - 1/Y_2} , \tag{A1}$$

which is independent of the hot and cold load temperatures. During the measurements, the samples and the hot and cold loads were slightly inclined to the axis of the receiver beam to avoid multiple reflections. Note that the loss given by eqn. (A1) includes the small reflection loss from the samples.
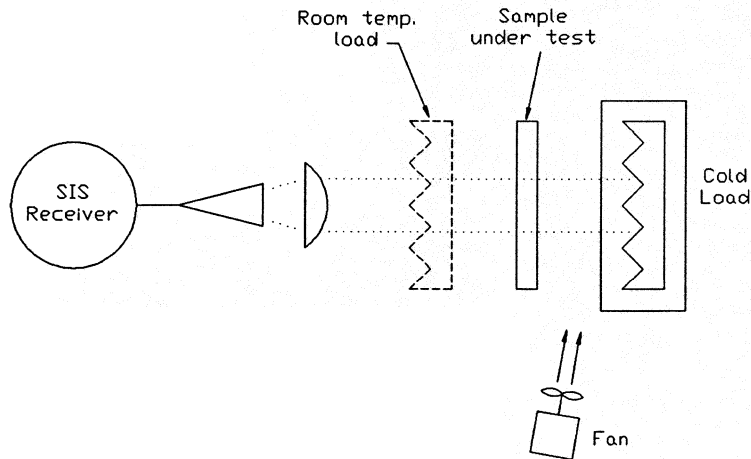

Fig. A1. Measurement of insertion loss of foam and film samples.

APPENDIX B: Adhesives Used in Constructing the Vacuum Window

Polystyrene to Aluminum

Eccobond 45 (Clear) epoxy adhesive [8].

Mix ratio: 1 part resin to 2 parts catalyst #15 (by weight).

Cure: Room temperature for 12 hours.

HR500/2S to Aluminum

Scotch-Weld Epoxy Adhesive DP-190 [15]

Mix ratio: 1:1 (by volume).

Cure: Room temperature for 12 hours.

Alternative:

Scotch-Weld Epoxy Adhesive 2216 [15]. (This is very similar to DP-190 (above) but has a different mix ratio.)

Mix ratio: 2(B):3(A) (by volume).

Cure: Room temperature for 12 hours.

Mylar to Aluminum

Considerable effort was required to find a suitable adhesive for mylar. Eventually, Eccobond 45 (Clear) was found to give a bond strong in tension and peel provided a special two-stage curing schedule was used.

Eccobond 45 (Clear) epoxy adhesive [8].

Mix ratio: 1 part resin to 2 parts catalyst #15 (by weight).

Cure: 15 minutes at 220°F (104°C) followed by 10 minutes at 350°F (177°C). The second stage is considerably hotter than normally recommended for Eccobond 45; however is was found necessary to achieve a strong bond to mylar.