TRANSPARENT MATERIALS

Chairman	Mr. W. Conrardy
Speaker	Mr. R. Wittman
Panel Members	Capt G. H. Roberts Mr. K. H. Digges

TRANSPARENT MATERIALS

R. Wittman

Directorate of Materials and Processes, ASD

In any discussion of optical materials, the most important single parameter is wave length. A portion of the electro magnetic spectrum ranging from 0.150 to 20.0 microns is reviewed herein. The three definite areas included in this bracket are the ultraviolet, visible, and a portion of the infrared. In order to illustrate current capabilities and limitations, materials will be discussed under the type of application for which they are best suited.

Ultraviolet

Transmission of the ultraviolet wave lengths in windows and ophthalmic transparencies has been and is undesirable. Plastics used for this purpose have UV absorbers incorporated in the polymer structure to reduce transmission in the erythemal (sunburn) band. Military specifications for plastic window materials require less than 5 percent transmission between 290 and 330 millimicrons. Ophthalmic transparencies are more restrictive in this respect and specify when possible, less than 1 percent transmission between 200 and 320 millimicrons. The transmission of soda lime glass, which is the composition most used in aircraft, does not exceed 5 percent up to 325 millimicrons. Ultraviolet radiation has no noticeable detrimental effect on glass but over a period of time does tend to degrade acrylic plastics. This effect however is not of such a magnitude as to warrant its consideration in crew enclosure design. The transmission curves for glasses having higher use temperatures than soda-lime do move further out into the ultraviolet as is shown in figure 1. Each application of these special glasses will present an individual problem which can be aleviated through the use of compensating coatings and/or filters, depending on the severity of anticipated conditions.

Visible Spectrum

Windows for vision in Air Force vehicles are constructed primarily of soda-lime glass and acrylic plastics. The most widely used single material today is the thermoplastic polymethylmethacrylate.

The evaluation of methylmethacrylate formulations toward the presently used modified acrylic (MIL-P-8184) is well known. Also the introduction of laminated acrylic sheeting with a polyvinyl butyral interlayer to provide a non-shattering glazing material.

The subsequent development of a practical method to induce molecular orientation in the thermoplastic acrylics through a hot stretching technique has fostered more efficient design of windows. Material thicknesses have been reduced and attachment means simplified. The "toughness" or resistance to fracture imparted by this process is determined by a special testing method which measures the amount of work necessary to propagate a fracture. Results are reported in terms of a "K" value which is equal to $1.25\ (P/t)V_Z$ lbs/in.$^{3/2}$

Where:

t = thickness, in inches
B = width, in inches
P = ultimate load, in pounds
$Z = Y\,(2-Y^2-Y^4)^2$
$Y = X/B$ where X = width of fracture (in.) at point of instability.

The universal application of this development is shown by the fact that stretched acrylics have been incorporated into practically all operational USAF flight vehicles which require plastic windows. The polyester type thermosetting plastics cannot be oriented in this, or any other, fashion due to the high amount of cross-linking within the polymer. The operational use to date of this type of plastic has been quite restricted because of this limitation and the additional weight imposed by lamination.

In recent years, the Air Force has conducted several research programs aimed at the discovery and development of more thermally stable transparent plastics. Silicone materials have been studied extensively and formulations have been produced that have excellent thermal stability. But as yet it has not been possible to circumvent the inherent property of the silicone structure: that of resiliency and elasticity, consequently the strength properties of rigid sheet material even at room temperature are quite low. A more recent development sponsored by the Air Force has been the modification of a two-stage thermosetting acrylic plastic to produce a polymer that is structurally useable, from the standpoint of rigidity and strength, to temperatures of over 400°F. Attempts to achieve molecular orientation of this material while in the partially cured state were not too successful. It was possible to stretch the material mechanically and the optimum parameters were defined. However, the temperatures required to complete the curing of this material produced difficulty in maintaining the mechanical stretched stage. In the final product the orientation achieved was not of sufficient magnitude to warrant the use of this process. Figure 2 contains several properties of this developmental material.

Advancements have also been made by industry with the polyester family of materials and under other contracts within the Department of Defense on epoxy type materials which are quite transparent in the visible range of the electromagnetic spectrum. The capability of these modified materials insofar as strength retention at higher temperatures is concerned, is between 300 and 350°F.

The work on silicone polymers however has produced a material for use as an interlayer in laminates. The temperatures required to cure the interlayer are such that this material cannot be used effectively with plastics, and therefore has been utilized solely with glass. The need for an interlayer material having better temperature resistance than polyvinylbutyral and which would be compatible with plastics was recognized. Subsequently, such a material was developed and proven feasible by private industry. This "Cast-in-place" interlayer has made possible application of a composite concept which utilizes a non-structural outer ply of a more heat resistant notch sensitive plastic, the interlayer, and an inner structural ply of commercially available hot stretched acrylic. Aerodynamic generated temperatures beyond the normal capabilities of the structural member can be accomodated by a unit of this type. Research is continuing to define the best materials combination and to obtain criteria which will allow for the most effective design parameters with this composite concept.

Two standard interlayer materials are currently available for use in glass laminates. Polyvinylbutyral has been used for many years as a flexible sheet material to impart shatter resistance to both glass and plastics. It is quite easy to use in fabrication, and has been generally suitable in most respects although certain design considerations have had to be made to compensate for its elevated temperature stability (under 200°F) and low temperature resiliency. Previous mention was made of the development of a silicone type interlayer material which can be used at temperatures up to 300°F and still retains its elastic properties at very low temperatures. An improved silicone material having a temperature capability of approximately 425°F is under evaluation at the present time. Figure 3 gives a comparison of interlayer materials for the function of impact resistance versus temperature when laminated with glass and shows the obvious advantages of the silicones.

The glass composition used for aircraft windows today is that of soda-lime or commercial plate glass. This glass has satisfied materials requirements for visual capabilities, strength, temperature resistance, and in the laminated form, impact resistance. Other compositions having much higher temperature capabilities have been developed by the glass industry over the past few years. These glasses and their approximate capabilities temperaturewise are shown in figure 4. Actually at the time of development there was no need for these glasses, at least not for use in crew enclosures. However, specific requirements today have made the application of these glass types mandatory. It is definitely a big step to produce these glasses in sizes and of the quality necessary for aircraft windows. Two very important factors are: quality from a materials as well as an optical standpoint. It is relatively simple to obtain quality soda-lime glass for flight vehicles by the simple selection of the highest quality glass from a large volume of plate glass produced for industrial purposes. However, only a small portion of commercial production meets the rigid Air Force optical requirements. Rejected glass is diverted into the normal production cycle. The more heat-resistant glasses are not produced in as large a volume, therefore, the Air Force has sponsored a program over the past three years to develop a feasible manufacturing method to insure the production of a consistently acceptable heat resistant glass. This program has been carried out quite effectively and production methods have proved practical. The program has also evolved several improvements in glass finishing techniques, all of which are designed to increase the reliability of glass components. In addition, the Air Force has and is sponsoring a program at the National Bureau of Standards to obtain useable design criteria on these glasses at temperatures up through the capability of each particular glass involved.

Research and development efforts on ophthalmic transparencies have incorporated and put into practice much of the knowledge gained on window materials, such as hot-stretched acrylics, laminated polyester plastics, and conductive coatings for de-misting purposes. Research towards modifications of present plastics is being conducted to produce ophthalmic transparencies having higher transmission in the visible range and greater reflectivity in the infrared. Ideal condition and available materials are shown in figure 5. Other programs are directed toward self attenuating ophthalmic filter materials to provide eye protection from nuclear flashes. Phototropic compounds are being investigated for this application.

The availability of higher temperature glasses that have the necessary quality for more advanced Air Force applications, together with publishing of mechanical design criteria, is a large step forward. To utilize these glasses at temperatures where conventional direct attaching techniques are impossible, the Air Force has over the past four years sponsored research on attachment materials and techniques. This work has resulted in successful direct bonded attachments for soda-lime glass good for extended lengths of time at 500°F. In addition to the temperature capability of the attachments their use has made it increasingly possible to use windows as structural elements. Further developments have produced attachments compatible with lower expansion high temperature glasses and having a load carrying capability to 900°F. This program will continue to increase the operating temperature range and to provide design criteria from which to produce practical high temperature glazing assemblies. The most recent effort in the area of attachments is to produce a bonded attachment primarily for alumina windows which have a temperature capability of 2500°F for at least five minutes.

Within the area of visible light transmission some mention should be made of coatings which are used for de-fogging and de-icing purposes on windows and ophthalmic transparencies. Most aircraft today have glass incorporating these coatings primarily in front and side windows. The coatings are effective and tribute is certainly due to the suppliers for their research and rapid advancement of techniques in this area. The bulk of window replacements in today's flight vehicles, are due to the failure of this coating, rather than the glass itself. Close cooperation between the airframe manufacturers and window suppliers in the design stage is very critical. Concepts are being altered and revised to provide the necessary considerations generated by characteristics of coatings and their preservation under the severe aerodynamic and structural performance requirements of today's air vehicles.

As we make our initial explorations into space travel, the use of transparent materials in vehicle design is mandatory. Data on the capabilities of presently available transparent materials under all natural and predicted environments are not complete. The known basic materials properties make transparent glasses and plastics appear marginally satisfactory providing proper design considerations are used.

The exposure time to known natural and induced environments of transparent materials installations, with the exceptions of high vacuum, radiation, and micro meteroritic particle impact, will generally be a few minutes during initial powered flight, and again during a re-entry and landing phase. The areas of greatest concern are those of possible explosive decompression due to materials or structural failure, temperature effects, and loss of a clear vision area during flight.

To provide more reliable design criteria, additional research studies are being made at elevated temperatures on the spectral emissivity and heat transfer capabilities of coated glasses, combinations of transparent materials and coatings, and changes that occur when abraded surfaces are introduced. Data in this area is valuable not only from the material aspects but also the secondary effect on the internal conditioning system of the vehicle.

Controlled abrasion of glass does not materially reduce design strengths but the effects of random micrometeoric and charged particle impact on the erosion characteristics under high vacuum and extreme temperature conditions must be learned.

Areas that are generally related to individual vehicle design such as vibration effects and methods of mounting (seals), can and are being investigated on a laboratory scale, to define and provide materials requirements and effective design data under high vacuum, and re-entry temperatures. Initial structure designs will then be more tailored to the materials restrictions, if any, imposed by mounting requirements.

To obtain statistically reliable design data on known special glass compositions under high stresses at high temperatures over long periods of time, will continue to be necessary together with related basic properties data. The continuous upgrading of the state of the art in the production, processing controls, and fabrication techniques by industry, coupled with integration of these improvements into window design will further increase the efficiency and reliability of our present optically transparent materials.

Infrared

Transmission in the wave length band from 0.7 to 20 microns is by far the broadest of the groups within the scope of this review. The most important parameter for consideration is infrared transmission, but the practical and even experimental use of a material is dependent upon the perfection of its processing, to insure homogeneity, and optical uniformity.

One of the most important requirements right now is that of retention of transmission efficiency together with mechanical integrity at elevated temperatures. Desirable properties toward this end include low thermal expansion and light weight. Most materials either being used now or being considered for use in this area are of a brittle nature. Fracture and/or failure of an item is generally due to the presence of flaws within the material. Again the importance of consistent quality is evident.

A new or better material which appears to have the desired properties such as transmission, quality, and temperature stability, cannot be utilized unless it can be produced in the proper form. This is a recognized problem whose importance is as great as the research and discovery of new materials.

Window materials which have been used in airborne infrared systems include optical glasses, fused silica or quartz glass, calcium aluminate glasses, arsenic sulfide glass, silicon, Kel-F and silver chloride. The effective transmission limitations for these materials range from 2 microns out as far as 6 microns under ideal conditions.

The Air Force has sponsored studies on other materials including intermetallic semiconductor compounds, refractory oxide and sulphide glasses, antimonate glasses and sintered barium-fluoride. Industry has, both through company sponsored research and as contractors and sub-contractors through the Department of Defense carried out a considerable effort in this area. Comprehensive data and bibliographies on all infrared transmitting materials has been compiled and released by several organizations under contract to the Government. There are also in effect today many research studies designed to provide design criteria on infrared transmitting materials when subjected to hyper thermal and extra terrestial environments.

Advancements with and improvements of available infrared materials are continuing. One such development is the work by the Barr and Stroud Company, Glasgow, Scotland on calcium aluminate glass to remove the absorption band between 2.5 and 5 microns. Figure 6 shows a comparison of the Barr and Stroud modification and a typical calcium aluminate glass. Other notable developments by industry include Eastman Irtran I and II, Eastman Kodak Co., Rochester 4, N. Y. Irtran I appears good for shorter wave lengths (less than 5 microns) and at temperatures up to approximately 800-900°F. Irtran II has good transmission in the band of 8 to 13 microns but more knowledge of these materials and their limitations, especially in sections thick enough for use as external windows together with their mechanical behavior at elevated temperatures is needed. Figures 7 and 8 are the transmission curves on these materials at temperatures up to approximately 1470°F.

The necessity for producing infrared transmitting windows in useable shapes prompted an Air Force contract which has just recently been completed by The Texas Instrument

Company. The material selected for this program was silicon. The production of cast domes up to 8 and one-half inches in diameter was made feasible through this effort. Transmission of silicon out to 8 microns is good, however, as higher temperatures are imposed, the transmission is reduced and at approximately 900°F becomes essentially zero. The availability of large windows of silicon will make possible more widespread application of this material within its useable temperature and transmission ranges.

Currently, ASD has a contract concerned with the development of high energy pressing techniques to produce IR Domes of desirable shapes and sizes. Materials being investigated include barium and calcium fluorides, cadmium and magnesium oxides, and calcium oxide.

A considerable portion of the materials research and development in this area is conducted in support of specific equipment contracts for solution of particular problems. One fundamental materials study recently initiated directly by the Air Force however is concerned with Gallium Arsenide. This material appears to have good transmission characteristics from 2 to 20 microns and relatively good temperature resistance.

Research on materials for ophthalmic transparencies is directed toward the absorption of infrared wave lengths. Present requirements are for maximum transmittance of 1 percent between 800 and 2000 millimicrons. This requirement becomes obvious when it is considered that approximately 40 percent of the thermal energy from a nuclear flash is in the infrared region. At the same time infrared reflection is necessary, transmission in the visible range is not to be reduced.

Other infrared optical components have not normally been considered in the materials research and development programs. This would include such items as mirrors, selective filters and detector cell windows.

Re-Entry Air Vehicle Window Concept

The human eye's ability of retentivity of vision permits one to have good vision through a disc containing holes or slots when it is rotating at sufficiently fast rate. One can readily understand this after realizing it is the same effect as that of seeing through the rotating blades of a fan or aircraft engine propeller.

Based upon this principle, a concept was developed by two ASD engineers: how one could have vision during re-entry heating periods and also maintain relatively modest temperatures on a transparent material located behind a rotating slotted disc. The latter feature is achieved because of the following facts. The rotating disc could be made of an insulating material with a high surface emissivity. To augment the heat inpedance of the disc, a cooling gas could be injected between the rotating disc and the window. At any instant only a small ever changing portion of the window is exposed directly to the heat coming through the slots in the disc. Figures 9 and 10 show views of the original model based upon this concept.

To demonstrate feasibility of the idea several questions required answering: Could one see adequately through a rotating slotted disc at high temperatures? Would the requirements for cooling be reasonable? To answer these questions light transmission, heating, and simulated re-entry tests were undertaken.

Light transmission values obtained from the rotating disc were equal to the percent of open area cut into the disc. The minimum light transmission requirement for landing a vehicle at dusk is approximately 12 percent, and for a moon lit night 16 percent. Hence, all disc configurations having more than this percent open area were more than adequate. Still photographs and motion pictures were taken under ambient and high temperature conditions. Excellent photos were obtained regardless of the number of slots in the disc and motion picture quality was controllable by adjustment of rotating speeds versus shutter speed. Results of tests in a high temperature, hypersonic gasdynamic facility are shown in figure 11.

The surface temperature of the rotating disc was above 2200°F while the maximum temperature on the outside glass surface was 780°F and the inside surface remained below 400°F. Air temperature was above 3100°F.

This concept has been shown to be practical. What is needed is further designing and testing to develop a window for specific hardward applications. Although the original application conceived for this device was human vision, several others are possible. Proper modifications may remove the need for the glass member and permit full transmission of the electromagnetic spectrum. This is only speculated but is a potential.

Summary

Transparent materials window requirements for the areas previously discussed are both specific and of a general nature.

a. Ultraviolet transmission is discouraged while simultaneously improving response in other portions of the electromagnetic spectrum.

b. Plastic, visible light transmitting, materials are needed which have a temperature capability to over 800°F for use as rigid members and for interlayer applications. Also for rigid, optical quality plastics which transmit neither above or below the visible spectrum.

c. More efficient processing of glassy materials to satisfy requirements such as increased reliability and more effective design.

d. Additional data on the behavior and properties of window materials when subjected to predicted space environments.

e. Development of infrared transmitting windows having high transmission through 15-20 microns and at temperatures up to 1500°F.

In this age of rapid scientific advancement, our continuing research studies will certainly produce materials which will surpass these generalized requirements. It is really a matter of first things first, by meeting each immediate challenge with the right answer in the shortest time.

Bibliography

1. Specification MIL-P-8184 Plastic Sheet, Acrylic Modified

2. Optical Requirements for Ophthalmic Transparencies - WADD Unpublished

3. Materials Handbook Corning Glass Work 1958

4. Batzdorff, A., Gouza, J. J., Hurst, D. A. - The Investigation of Multiaxially Stretched Acrylic Plastic WADC TR 54-619 Parts I and II 1957

5. Transparent Glazing Materials for Flight Vehicles - MIL-HDBK-Part II 1961

6. Helvey, G., Clark, H. A. Development of a Rigid Transparent Plastic Material Suitable for Aircraft Glazing at Elevated Temperature, WADC TR 56-352 Parts I and II 1958

7. Parker, E. E., St. Pierre, T. L., Stickley, W. E., George, E. A., Dzugan, M. R., Improved High Temperature Resistant Transparent Plastic Materials, WADD TR 60-72 May 1960

8. Development and Evaluation of High Temperature Resistant Composite Plastic Plates- Midwest Research Institute February 1961 (Bureau of Naval Weapons Contract No. as60-6099-c)

9. Evaluation of High-Temperature Cast-in-Place Transparent Plastic Laminates Suitable for Canopies on Supersonic Fighter Aircraft - Goodyear Aircraft Corporation - Bureau of Naval Weapons Contract No. a(s)59-6146-C Prog Rpts 1-6

10. Polmanteer, K. E., Campbell, F. J., Laur, T. L., Silicone Interlayer Material Program WADC TR 56-159 July 1956

11. Polmanteer, K. E., Campbell, F. J., Laur, T. L., Silicone Interlayer Material Program WADC TR 57-213 Parts I and II 1958

12. High Temperature Aircraft Windshield Development AF Contract 33(600)36852 Quarterly Progress Reports 1-10

13. Kerper, M. J., Diller, C. C., Eimer, E. H., Properties of Glasses at Elevated Temperatures, WADC TR 56-645, Parts II, III, IV and V.

14. A Summary of the Narmco, High Temperature Glazing Edge Attachment Effort - Telecomputing Corporation 1961

15. Wittman, R. E. Optically Transparent Materials for Space Flight Vehicles WADD TR 60-203 April 1960

16. Materials Research, Development and Application Technical Symposium WADC TR 58-655 July 1958

17. Evaluation of BS-37A Glass - WADD - WCLRWI Internal Report 40513 - Parts I and II 1959

Bibliography (Cont'd)

18. Cole, R. L., Mitchell, G., Hicks, J., Development of Silicon Infrared Optical Components AMC TR60-7-719 December 1960

19. Elevated Temperature Studies of Infrared Transparent Materials - Contract N123-(60530)-2167A, Quarterly Progress Reports No. 4, November 1960

20. Ballard, S. S., McCarthy, K. A., Wolfe, W. L., Optical Materials for Infrared Instrumentation - IRIA Report 2389 - 11-S, January 1959

21. Allinkov, S., Forbes, F., A New Concept for Re-Entry Vehicle Windows - Presented at the ARS Structures, Materials and Design Conference April 1961

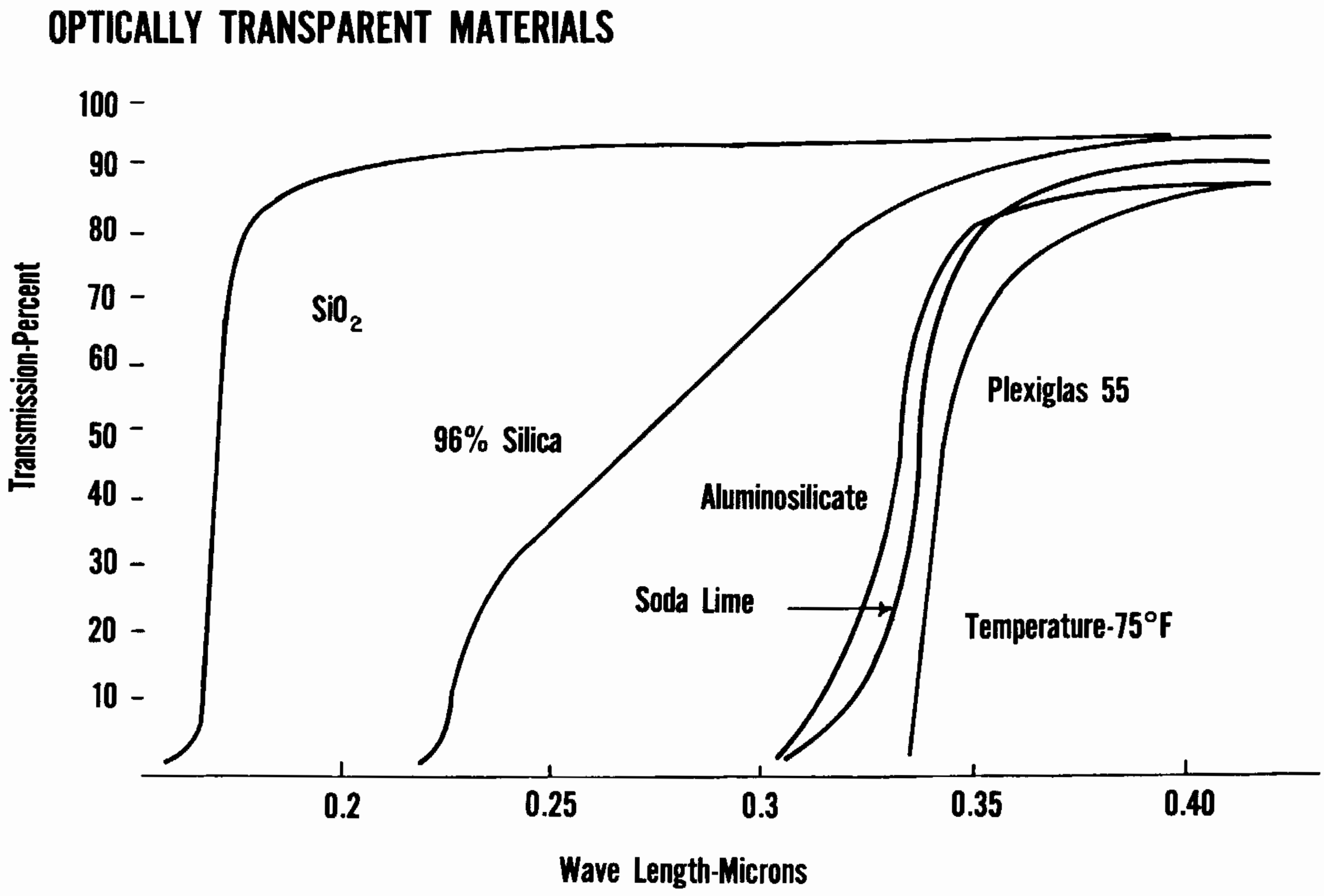

Figure 1. Ultraviolet Transmission

OPTICALLY TRANSPARENT PLASTICS

MODIFIED SELECTRON 400* (Formulation 796-55-4)

	TEST METHOD
Flexural Strength @ 77°F psi 13,600	ASTM D-790-58T
Flexural Modulus @ 77°F psi 521,000	ASTM-D790-58T.
Flexural Strength @ 480°F psi 3,500	ASTM-D790-58T
Flexual Modulus @ 480°F psi 188 ,000	ASTM-D790-58T
Tensile Strength @ 77°F psi 6000	ASTM-D-638-58T
Tensile Modulus @ 77°F psi 503,000	ASTM D 638
Heat Distortion Temperature °F 516	ASTM-D648
Luminous Transmittance % 88.6	ASTM-D1003-52 Proc B
Haze % 1.5	ASTM-D1003-52 Proc B

*Pittsburgh Plate Glass Co.

Figure 2. Selected Properties of Modified Selection 400

INTERLAYER MATERIALS

IMPACT RESISTANCE vs TEMPERATURE*

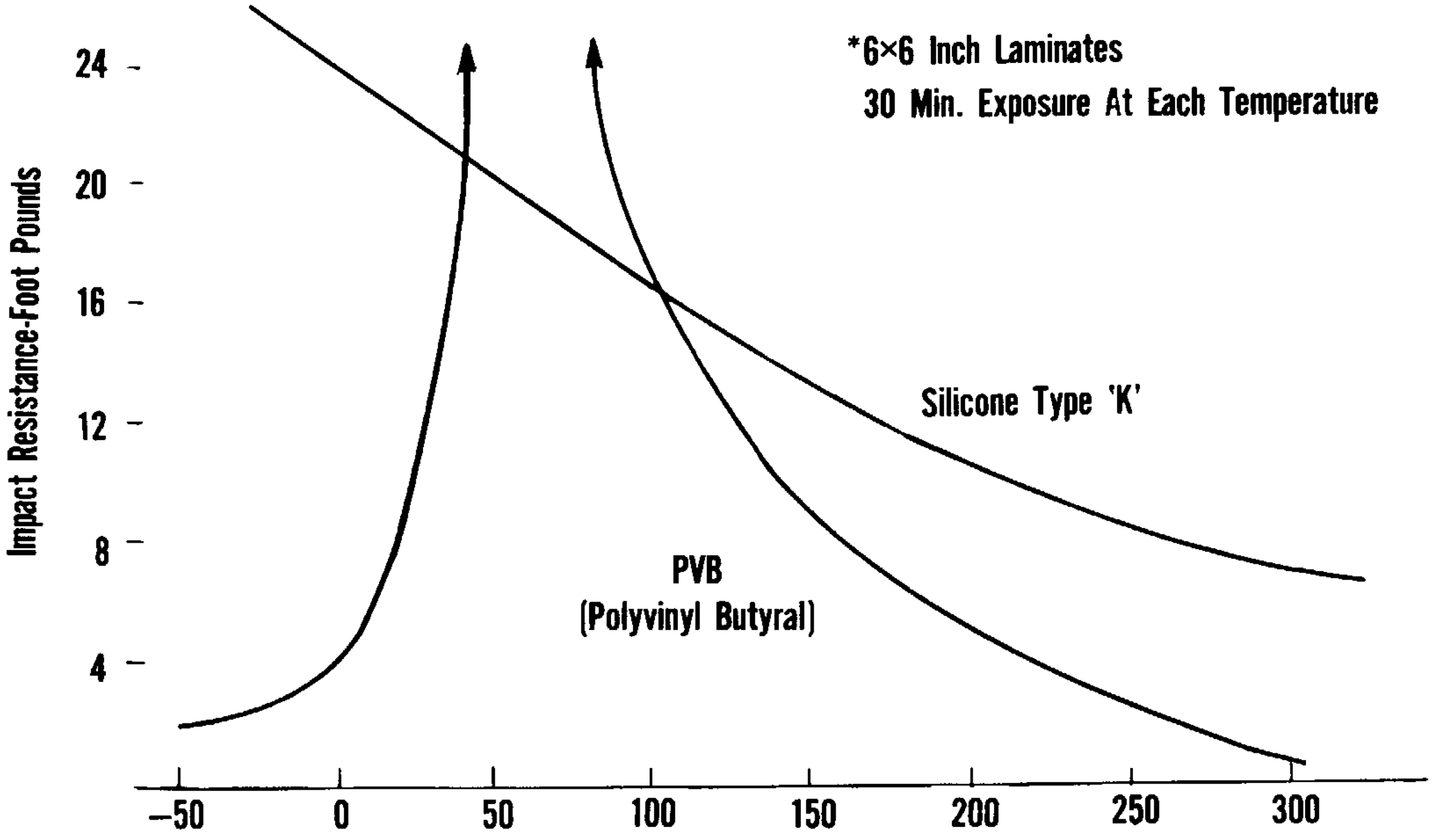

Figure 3. Relative Impact Resistance of the Interlayer Materials

OPTICALLY TRANSPARENT GLASSES

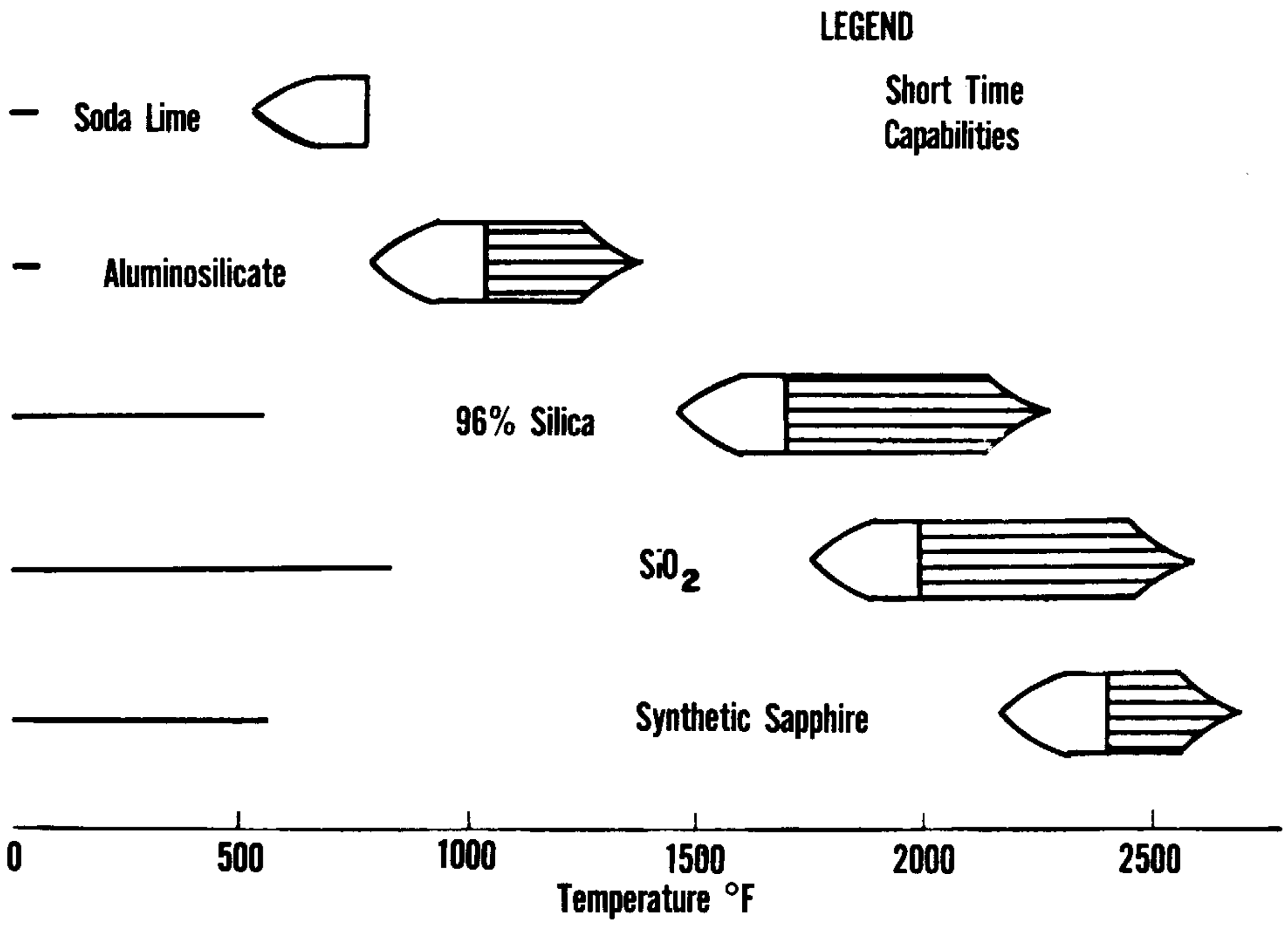

Figure 4. Optically Transparent Glasses

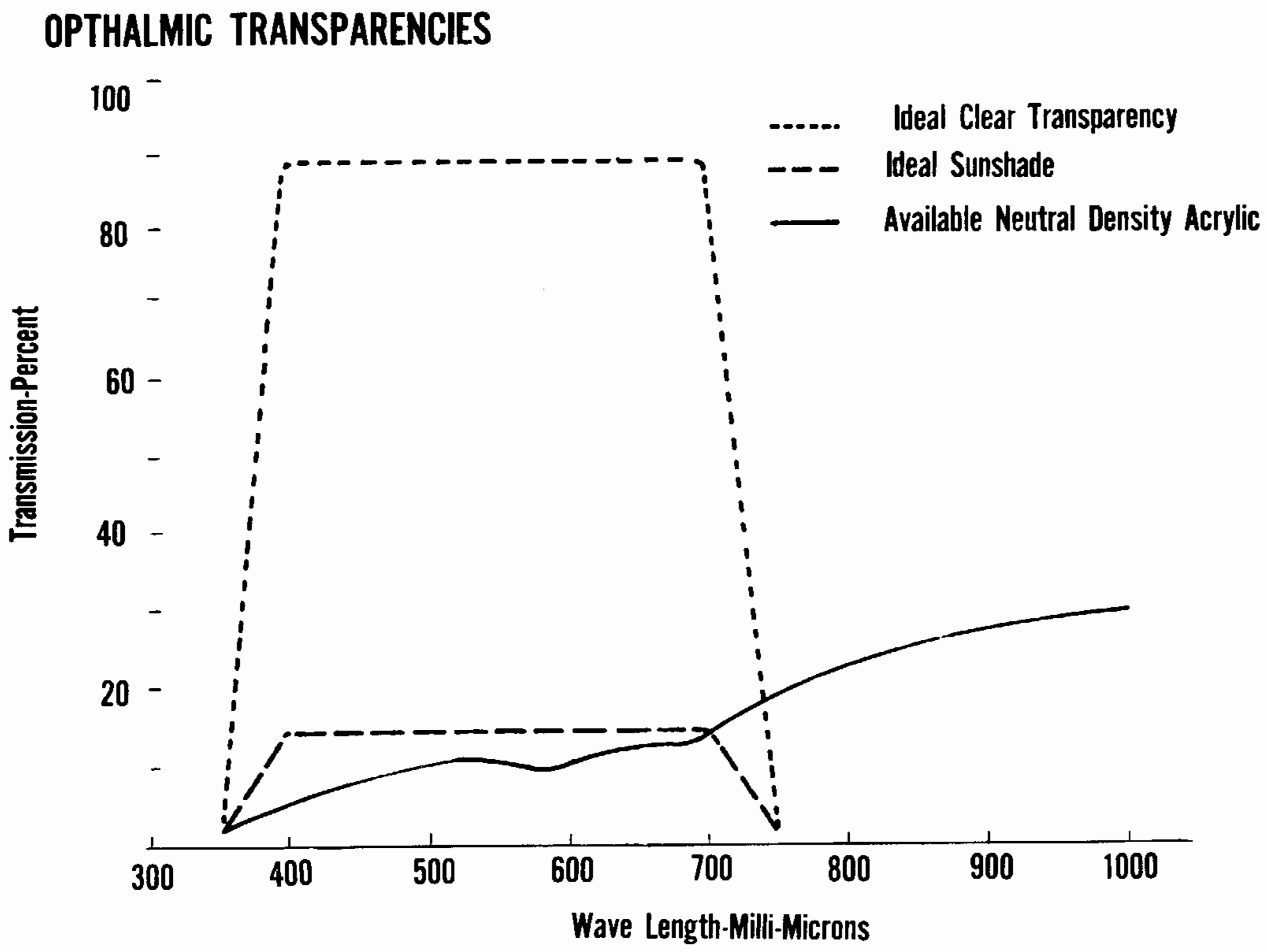

Figure 5. Ophthalmic Transparencies

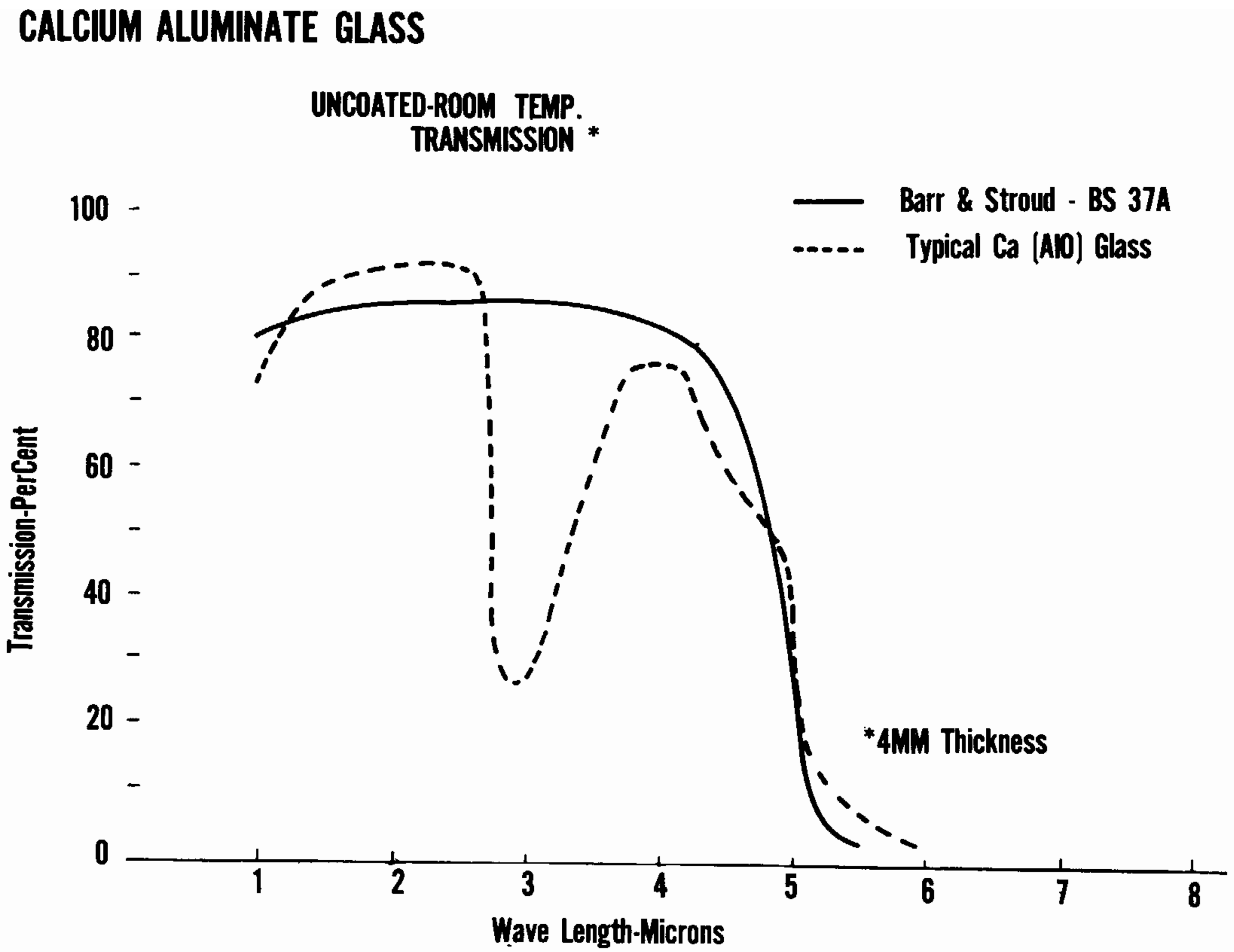

Figure 6. Transmission of Calcium Aluminate Glass

IRTRAN AB-1

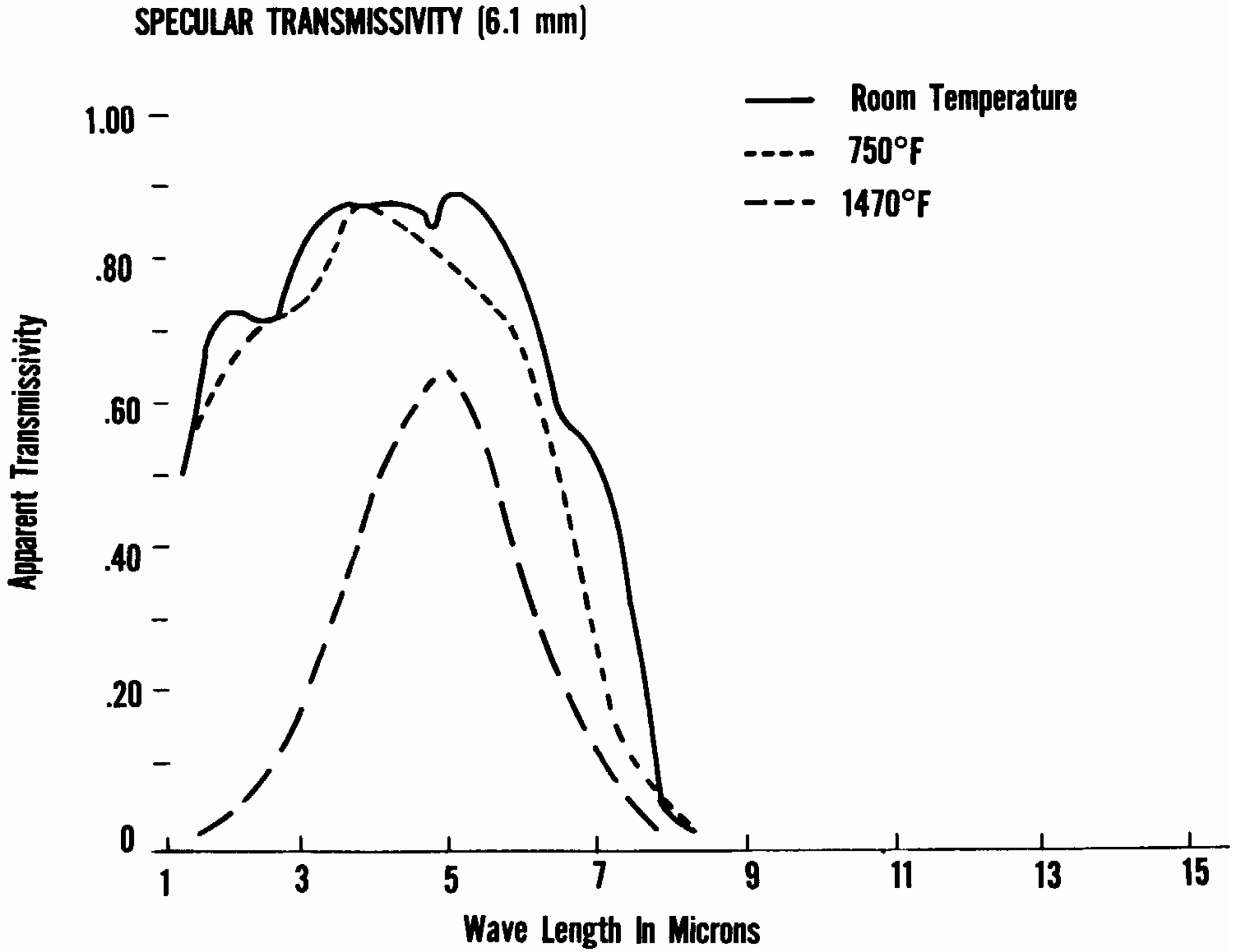

Figure 7. Infrared Transmission Versus Temperature

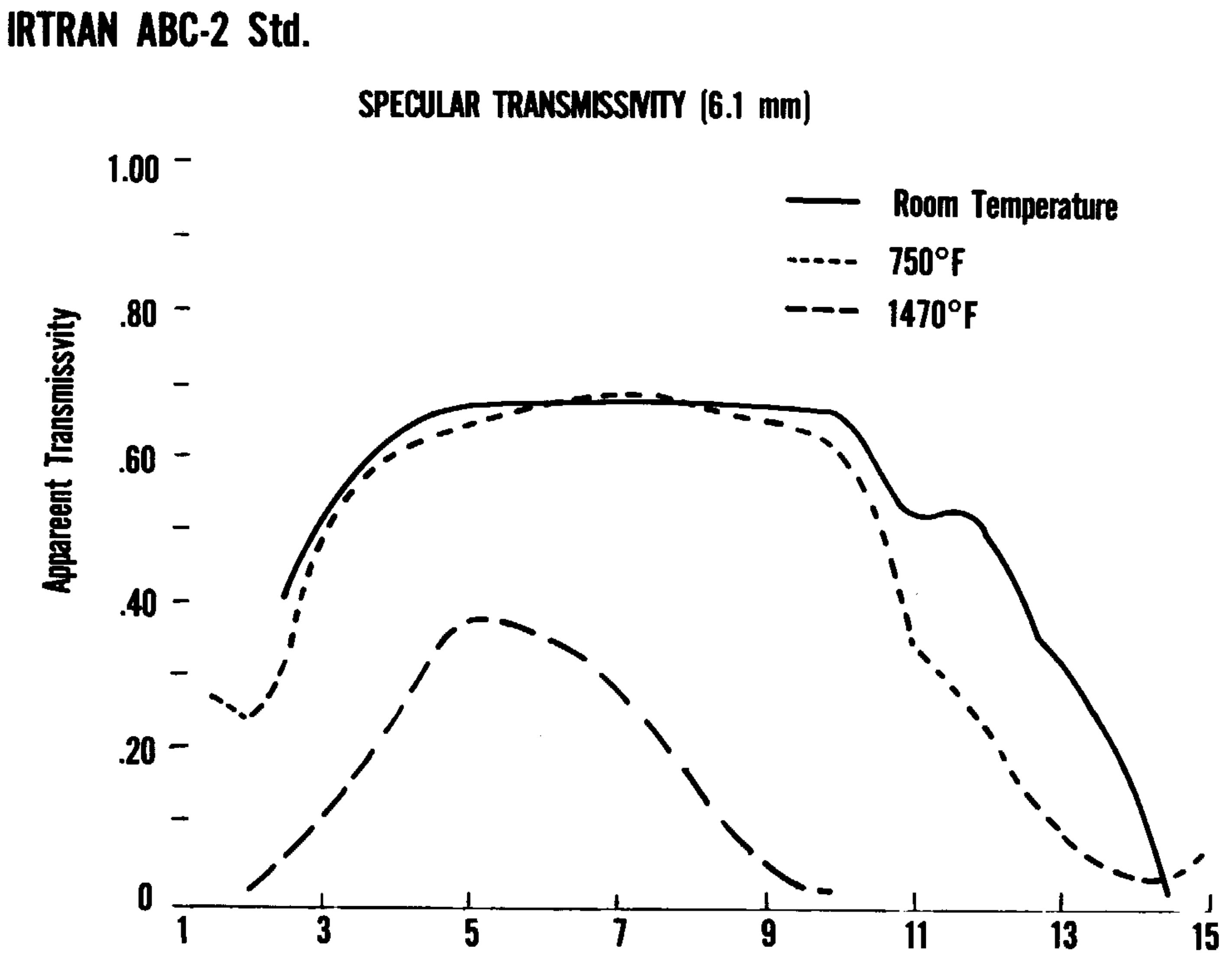

Figure 8. Infrared Transmission Versus Temperature

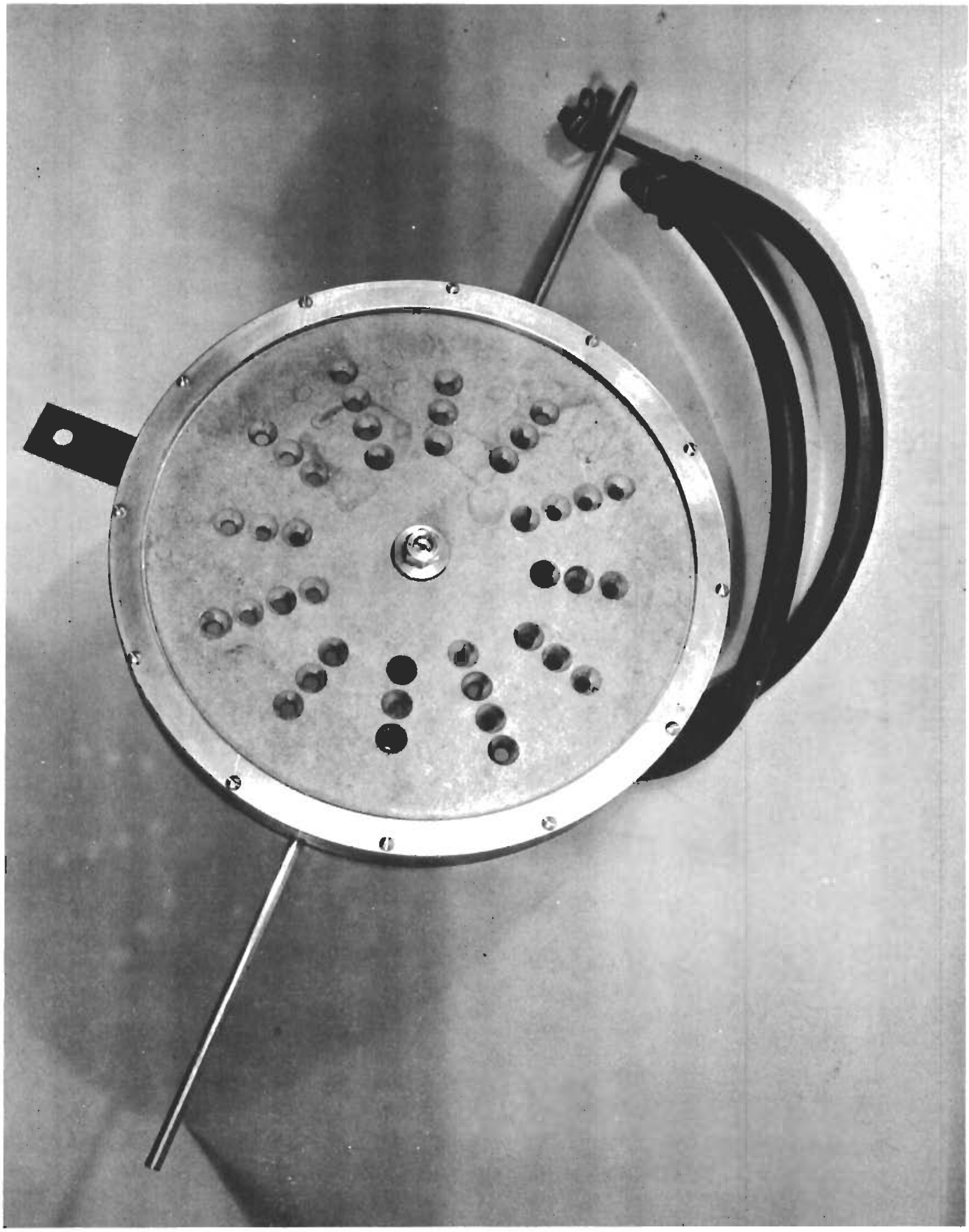

Figure 9. Front View of Original Re-Entry Window

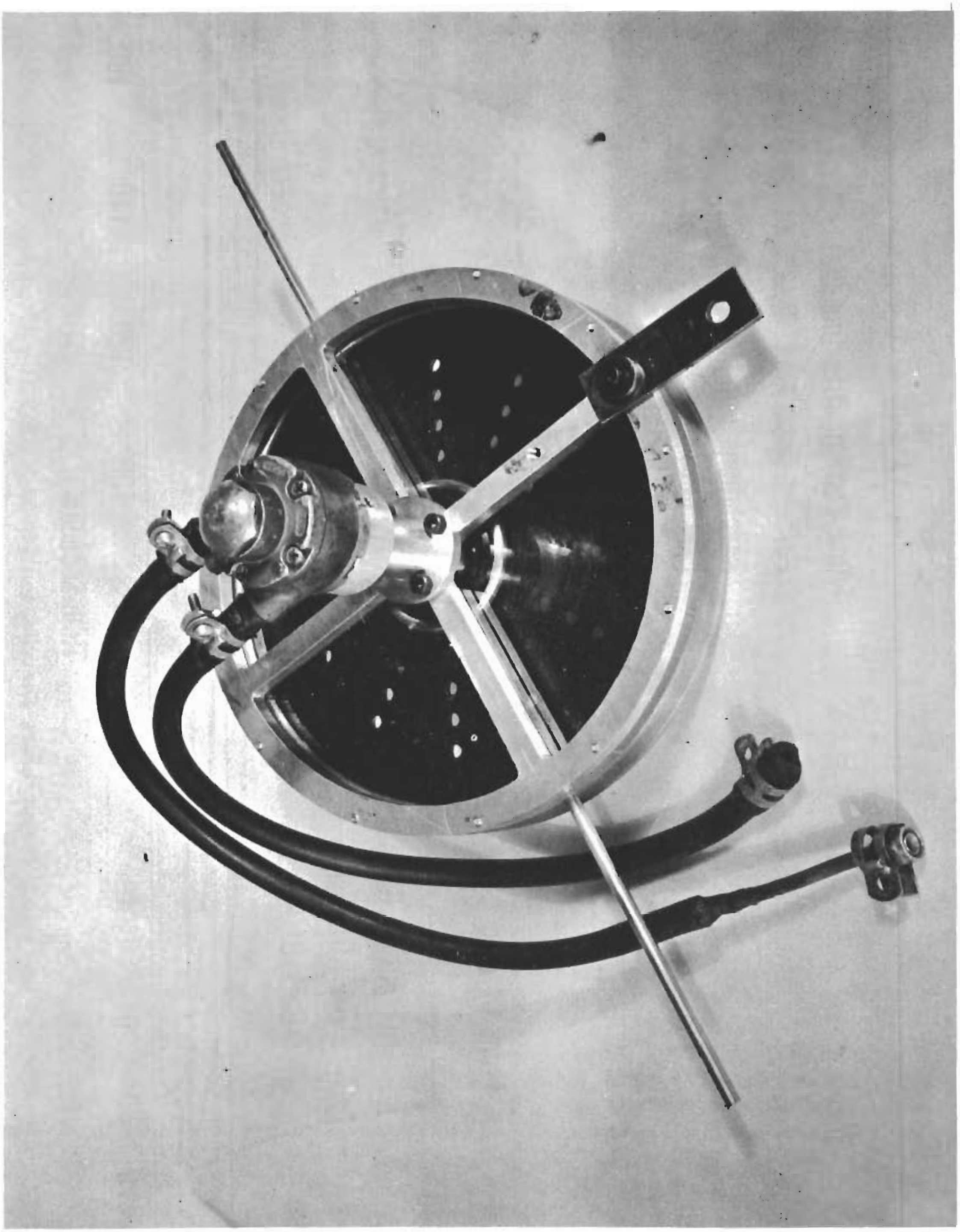

Figure 10. Rear View of Original Re-Entry Window

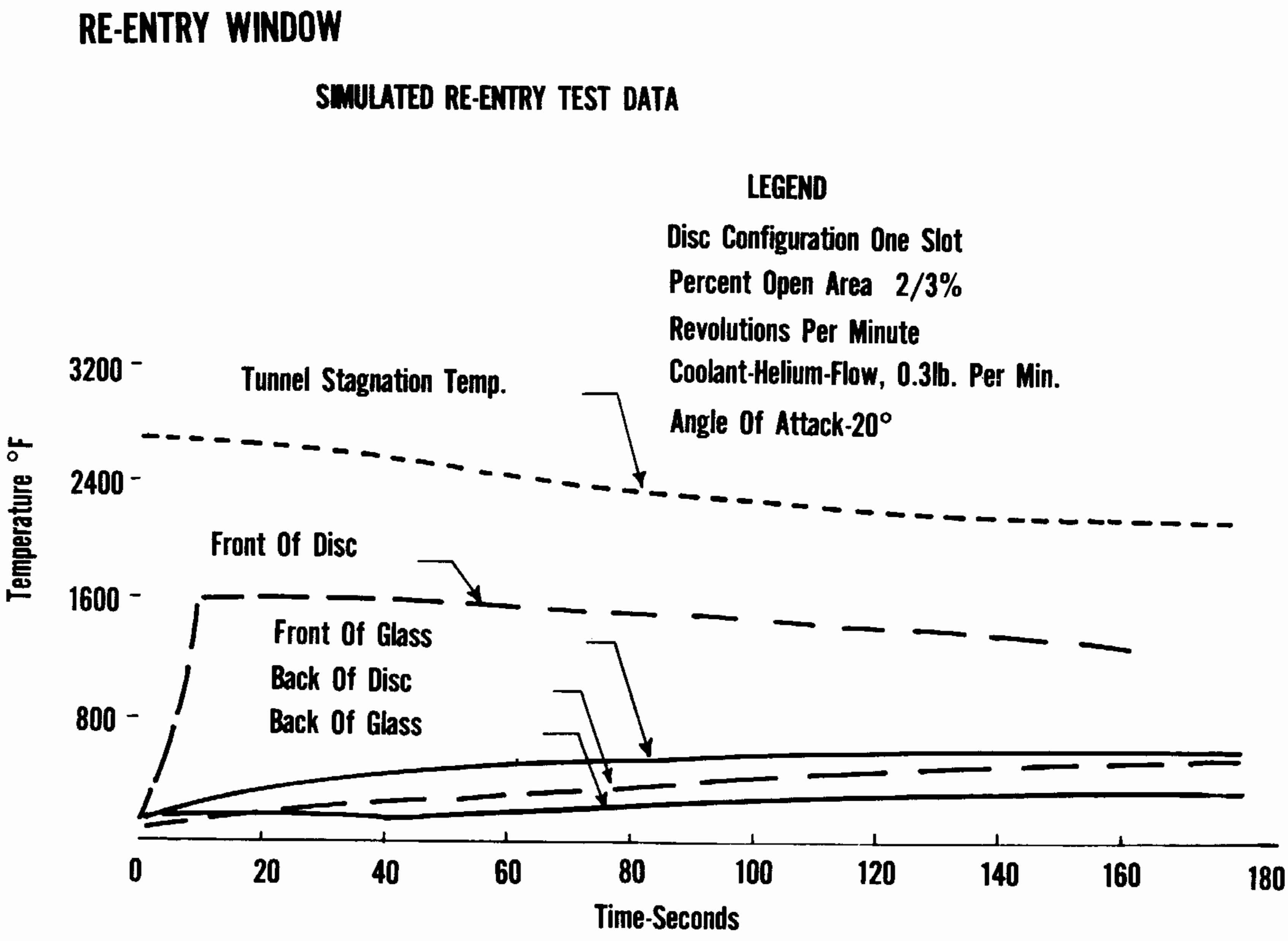

Figure 11. Temperature Distribution Through Re-Entry Window