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GLOVEBOX WINDOW MATERIALS

A GLOVEBOX FIRE SAFETY APPLICATION

1969

Prepared by
FACTORY MUTUAL RESEARCH CORPORATION
for
UNITED STATES ATOMIC ENERGY COMMISSION

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Prepared by the Staff of
Factory Mutual Research Corporation

for

United States Atomic Energy Commission

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FOREWORD

Pursuant to a request from the Division of Operational Safety, Headquarters, U. S. Atomic Energy Commission (AEC), the AEC Chicago Operations Office (CH) contracted with the Factory Mutual Research Corporation (FMRC) to study glovebox fire safety problems relating to activities of the AEC and its contractors.

Most of the work under that contract was reported in "Glovebox Fire Safety, A Guide for Safe Practices in Design, Protection and Operation," TID-24236, issued in 1967. Other FMRC glovebox fire safety work in specialized areas is covered in separate reports such as the one given on the following pages.

Technical administration of the contract was handled largely by L. E. Oldendorf, Safety and Fire Protection Engineer, Safety & Technical Services Division, CH.

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Any comparison of the design or performance characteristics of manufacturers' products is not to be construed as an endorsement of one product over another. Stated preferences for a particular design or set of performance characteristics are intended solely to establish minimum acceptable specifications for a particular type of product when used in glovebox applications described herein. Any future procurement of products referred to herein by the AEC or its contractors, will be based upon normal competitive procedures. The procuring organization will in all instances specify the minimum specifications required.

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GLOVEBOX WINDOW MATERIALS

I ABSTRACT

The United States Atomic Energy Commission, through its Chicago Operations Office, requested the Factory Mutual Research Corporation (FMRC) to investigate fire protection and operating requirements for glovebox window materials. The FMRC conducted a literature search, visited AEC sites, and interviewed users of gloveboxes and others to identify the problems associated with window materials. Selection criteria were established and tests conducted on various glass and plastic materials, including most of those currently in use at AEC sites.

Of the 17 different window materials tested, none was found to be ideal. The glasses lacked impact resistance, were etched by fluorides, and were brittle, while the plastics were flammable in various degrees and were easily scratched. A ranking was established, nevertheless, for the more promising window materials:

Wired Glass
Laminated Glass
Polycarbonate

Investigation of glovebox window materials should be continued, particularly with regard to explosion resistance. Laminated semi-tempered glass, not yet commercially available, should be tested when it reaches the market. A survey by FMRC of 24 leading chemical and plastic manufacturers reveals that a development subcontract for a superior window material is possible.

II SCOPE

It is recognized that the potential for radioactive material release exists at all AEC sites at which hazardous materials, such as plutonium and polonium, are handled within gloveboxes. Loss of glovebox containment, if combined with failure of the air filtration system, could extend radioactive contamination spread concerns to areas external to the building. In addition to life and property hazards, such an occurrence may promote publicity adverse to AEC interests.

Loss of containment by fire, explosion, impact, or other cause is the principal concern in glovebox use. A fire can be tolerated providing it is extinguished before containment is lost. Similarly, an explosion which is contained or vented safely is not a potential disaster.

The window is not the only component of the glovebox whose failure could cause loss of containment. Gloves and bagports are also vulnerable to rupture. Windows are a weak element however, and are considerably greater in area than the combined cross sectional areas of all gloveports and bagports.

Containment can be lost due to failure of the window in several ways: through heat softening, fire, impact, or explosion. It was decided to evaluate these hazards, and to consider the resistance to them as safety requirements.

In addition to safety requirements, it was recognized that the window material should have other qualities not directly related to safety or containment. These properties would include resistance to abrasion, to chemical attack, and to radiation darkening, and ease of machining. They were considered to be operating requirements.

Both safety and operating requirements would be considered as criteria for the selection of window material. The safety requirements would receive more weight, in view of the potential disaster associated with loss of containment.

III BACKGROUND

FMRC, through its earlier work in Phase I of this contract, has investigated certain fire safety features of gloveboxes. It was recognized that additional work should be done in certain areas, including an investigation of window materials. Phase II, of which this report is a part, was then authorized.

Much literature on gloveboxes has been made available to FMRC by the AEC through its Division of Technical Information Extension. More than 450 technical papers relating to gloveboxes are either on file in the FM Library or are readily available. Of this number, 25 papers contained information useful in the preparation of this report. Such information included the types of window materials used at various AEC sites, properties of the materials, problems in their use, glovebox environmental data, and glovebox operating practices.

Product literature was obtained from 11 leading manufacturers of glass and plastics. Communication conducted by telephone and letter with their representatives yielded additional valuable technical information. Representatives from four manufacturers were interviewed in Norwood. Trade magazines of the plastic and chemical industry were reviewed regularly for new developments in transparent plastic.

Two AEC sources of accident information were reviewed: "Health and Safety Information Bulletin" and "Serious Accident Bulletin". Loss information involving glovebox windows covering a 10-year period was analyzed.

A number of AEC sites was visited. These were -

Argonne National Laboratory, Argonne, Illinois; Rocky Flats Plant, Golden, Colorado; Mound Laboratory, Miamisburg, Ohio; AEC Richland Operations Facilities, Richland, Washington; AEC Savannah River Operations Facilities, Aiken, South Carolina; Lawrence Radiation Laboratory, Berkeley and Livermore, California; and Brookhaven National Laboratory, Brookhaven, Long Island, New York.

Many observations made at AEC sites confirmed what was documented in the glovebox literature. Additional observations follow:

1. Glovebox operating personnel were asked what their greatest fear was while working. The answers included puncturing of a glove, loss of vacuum by filter clogging, leaking of seals, fire, and explosion. All of these hazards involved loss of containment.
2. At two sites, it was mentioned that equipment left operating unattended in the glovebox, often over a weekend, was a problem.
3. At two sites, it was volunteered that windows seldom require additional gloveports or other modifications. Ease of machining was, therefore, not a primary consideration for their windows.
4. It is necessary to change 300 windows annually at one of the sites, due to loss of transparency.
5. Other stated problems included delivery by manufacturers of window material different from that originally specified, crazing of acrylics from alpha radiation, bubbling of safety glass inner plastic layer from gamma radiation, and deflection of plastic windows under the operator's weight.

Three manufacturers of window materials were visited. These were Rohm and Haas Company, Plastics Engineering Laboratory, Bristol, Pennsylvania (Plexiglas); General Electric Company, Chemical Materials Department, Pittsfield, Massachusetts (Lexan); and PPG Industries, Pittsburgh, Pa. (Glass products and plastic resins). Conferences were held with engineering and marketing specialists, during which product limitations and uses, potential products, and testing methods were discussed.

IV TEST PROGRAM

A test program was arranged to evaluate the following criteria for the selection of glovebox window material:

Safety Requirements

Heat Resistance
Fire Resistance
Impact Resistance
Explosion Resistance

Operating Requirements

Abrasion Resistance
Chemical Resistance
Radiation Darkening Resistance
Workability

ASTM data are available for some of the above properties. The test for flammability, D635, was considered to be on too small a scale to be appropriate, so a special test was designed. There are no readily available ASTM data on the abrasion resistance of the products evaluated. The Rockwell Hardness test, D785, was considered to be an adequate measure of it, although it is admitted that there is no direct relation between hardness and abrasion resistance. Data on the above requirements were gathered in the following manner:

<u>Property</u>	<u>Source of Data</u>
Heat Resistance	FMRC Tests
Fire Resistance	FMRC Tests
Impact Resistance	ASTM D256 - Available Data Accepted
Abrasion Resistance	ASTM D785 - Available Data Accepted
Chemical Resistance	ASTM D543 - Available Data Accepted

Data were not available for explosion resistance and workability. These properties were evaluated on the basis of engineering experience and judgment. Resistance to radiation darkening was not evaluated.

A Heat Resistance Tests

1. General

Heat resistance tests were run first in the test program as a simple and economical way of screening materials. It was expected that some of the materials heat tested would be eliminated on the basis of their performance, and this proved to be the case. Of 17 window materials tested, nine were eliminated from further testing for various reasons.

Two samples of each product were tested, unless otherwise noted.

2. Test Method

This series of tests was performed with a 12-in. by 18-in. radiant panel as a heat source, as shown in Figure 2. The radiant panel was fed a mixture of air and natural gas. A centrifugal blower forced the mixture through the vertically mounted panel where ignition took place. By adjusting the flow of air and gas, the panel was made to glow without visible flame, resulting in a primarily radiant heat source.

It was decided to expose samples 20 in. by 24 in. in size, so that the sample would extend beyond the radiant panel on all sides. This was done to avoid exposing an edge of the sample directly, and to provide enough area for a differential heating effect. By so doing, the condition of an actual glovebox window could be more closely approximated. Thickness measurements (Table 3) were made by micrometer at the mid-point of each edge and then averaged for each sample.

Samples to be tested were mounted in a sheet metal dolly constructed so that it could be rolled into position at the radiant panel. When in position, the sample was parallel to and 2 in. from the panel. Previous small scale experiments had established that a gap larger than 2 in. would prevent ignition of some plastic samples.

All plastic samples and some of the glass samples were fitted with a frame around the top and both sides, held in place with 1-inch C-clamps. This was necessary to prevent deflection of the sample away from the radiant panel when heated and also to simulate actual installation.

3. Characteristics of Radiant Panel

A temperature reading on a lower corner of the radiant panel was approximately 1500°F. A random temperature reading taken near the center of 1/2 of the radiant panel was 550°F higher. It is reasonable to assume that temperatures on the exposed sample also varied somewhat across the face.

Because of the deflection of most samples, it was not considered practical to monitor readings on the sample. In order to obtain some temperature information on the sample, readings were made on wired glass. A chromel-alumel thermocouple was arranged to touch the glass opposite the point on the radiant panel where temperatures were normally observed. The temperature did not exceed 500°F after 10 minutes of test time, as radiant energy passing through the transparent specimen prevents a build-up of higher temperatures.

4. Materials Tested

Window products currently in use at AEC sites, plus others whose properties appear promising, were subjected to the heat resistance test. They fell in two categories:

a. Glass Products

The answers to the Ad Hoc Committee questionnaire (edited by N. G. Garden - see Bibliography) revealed the following terms used to describe window glass in use: safety glass, laminated safety glass, safety plate glass, ordinary safety plate, double strength safety glass, laminated glass, and two-ply safety glass. All of the foregoing terms were assumed to mean laminated glass, from plate or sheet stock. Tempered glass, lead glass, and Pyrex glass sheet were also mentioned.

It was decided to test tempered glass, semi-tempered glass, laminated sheet glass, and laminated plate glass in two thicknesses.

b. Plastic Products

According to replies to the AEC questionnaire, the window plastics in use were Plexiglas, Plexiglas 5009, Lucite Non-UVA (methyl methacrylates); Homalite CR39, Homalite CR101 (allyl diglycol carbonates); and a laminate of Plexiglas and tempered glass. Several newer products are being used. These include Plexiglas SE3 and a laminate of Plexiglas and lead glass.

Plexiglas G, Plexiglas 5009, Plexiglas SE3, Plexiglas XL-20A, Homalite 911 and Homalite 911XHT, polystyrene, cellulose acetate butyrate, Lexan (polycarbonate), and Flame Resistant Lexan (polycarbonate) were tested. The latter four are not known to be in use at any AEC site, but were selected on the basis of their published properties.

5. Test Results

Detailed test results are included in Table 1 and summarized below.

a. Glass products, all furnished by PPG Industries. All of the glass products tested, except semi-tempered and tempered glass, cracked within 35 seconds. With plate and lead glass, this crack shattered the glass, while the glasses reinforced with plastic or wire remained in place. The plastic-reinforced glasses eventually failed by separation, or delamination of the layers, followed by burning of the plastic (Figure 4). The wire-reinforced glasses continued to crack, but the wire held the glass in place throughout the 15-min. test (Figure 3).

Semi-tempering of the glass increases the time to cracking somewhat, to 45 and 52 seconds in the two FMRC tests. As with plate and lead glass, this cracking shattered the glass into fairly large pieces. Full tempering increases the time to cracking still further, but in an unpredictable manner. One piece failed at 300 seconds and the other at 95 seconds. In each test on fully tempered glass, hundreds of small cubical pieces were strewn about the laboratory.

b. Plastic products. The Plexiglas products were produced or furnished by Rohm & Haas, the Homalite products by G-L Industries, and the Lexan products by General Electric. The other plastics were obtained from local distributors.

All of the plastics deflected in various degrees when heated, and had to be fitted with a steel frame to preserve the 2-inch space to the radiant panel. Burning was usually preceded by surface activity, such as, bubbling, sizzling, or browning. With some of the fire retardant products, there was intermittent flashing and sparking prior to steady burning. Homalite, the only thermoset tested, exhibited the most deflection and, in several instances, broke apart after the test while cooling (Figure 6).

B Fire Resistance Tests

1. General

After the heat resistant tests, fire resistance tests were arranged for eight window materials selected for further evaluation. This test was considered to be more typical of what might happen in an actual glovebox fire.

As with the heat resistance tests, two samples of each product were tested unless otherwise noted.

2. Test Method

An enclosure was constructed to which a 35-in. x 35-in. window sample could be clamped, thus forming a sloping front face (Figure 9). Part of the back and part of the top were left open, to provide a draft and access to the inside. The frame itself was constructed of "metal lumber," and the bottom, sides, and upper part of the back, and part of the top of asbestos-cement board.

A chromel-alumel thermocouple was placed through a hole in the top board so that it was approximately 1/4-in. below the top and 1-1/4 in. horizontally from the top of the test sample. Part way through the test series, a second thermocouple was placed inside the enclosure, approximately 1-in. from the center of the sample. The readings for both thermocouples were continuously recorded.

The exposure consisted of fire in a 1-1/2 in. depth of alcohol in a 1-sq. ft. pan. The pan was placed on the floor of the enclosure. As the window samples sloped in at 18° from vertical, flame from the alcohol fire easily impinged on them.

Samples were first checked for thickness in the same manner as in the radiant panel tests (Table 3). Plastic samples were then clamped in position, using a welded frame of 1-in. by 1-in. by 1/8-in. angles against the unexposed face and three 2-in. C-clamps on each side, 12 in all. Glass samples were clamped in the same way, but a gasketing material was used at both faces of the glass to prevent cracking. The gasket was a pre-formed 3/8-in. round silicone bead of 50 durometer hardness. Before testing the plastic samples, their relative stiffness was noted by a deflection measurement (Figure 10). The enclosure was laid flat with the window sample horizontal, and a 62-lb. load of bricks placed in the center. The sag at the center was then measured. This measurement then led to the choice of 3/8-in. thickness for plastic window materials.

The fire resistance test observations were continued until failure, which was established to be a burn-through of the material, or burning on the outside of the material in the case of laminated glass, or pulling out of the frame, or shattering. The test was terminated by covering the pan and extinguishing any fire in the sample with water or carbon dioxide, if necessary.

3. Materials Tested

a. Glass Products

Only the laminated glasses were considered for fire resistance testing, although wired glass was tested as a control. Since the behavior of laminated sheet and plate glass in the same thickness had been very similar in the heat resistance test, no test was performed on regular 1/4-in. laminated plate glass.

Laminated sheet glass, 3/8-in. laminated plate glass with a 15-mil vinyl butyrate inner layer, and a special 1/4-in. laminated plate glass with a 30-mil vinyl butyrate inner layer were tested.

b. Plastic Products

Four plastics were tested, all of 3/8-in. thickness. These were: Plexiglas G, Plexiglas SE3, Lexan, and flame-resistant Lexan. Other plastics previously tested were either considered unsuitable or were unavailable in the 3/8-in. thickness with acceptable optical quality.

4. Test Results

Detailed test results are included in Table 2 and summarized below.

a. Glass Products

- (1) Laminated sheet glass, double strength (PPG Industries). Four samples were tested because of the variation in failure times of the first two tests - 185 and 925 seconds (Figures 28 and 30). It was thought that some error in test method existed, and two samples of wire glass were then exposed, using tighter controls on the alcohol temperature and depth. These tests having given consistent results, two more tests were conducted with the laminated sheet glass; this failed at 395 and 500 seconds. Failure in all but the first of these tests was by burn-through near the top.
- (2) Laminated plate glass (Libbey-Owens-Ford), nominal thickness 3/8 in. This product was tested because of its use by at least one AEC site. It is a laminate of two plates of equal thickness. Failure occurred by opening of a crack at 810 and 460 seconds in the two tests (Figure 29). In the first test, a piece of glass fell from the exposed face at 480 seconds, but the unexposed layer remained intact.
- (3) Laminated plate glass (PPG Industries), nominal thickness 1/4-in. This is the only glass tested with a 30-mil plastic inner layer. Failure in the two tests occurred at 205 and 696 seconds. In the second test, the fire in the plastic layer was intense at time of failure.

b. Plastic Products

- (1) Plexiglas G (Rohm & Haas), nominal thickness 3/8-in. Deflection was just under 1/4-in. under the 62-lb. load. After ignition, inward deflection was noticeable almost immediately, followed by evidence of burning, outward deflection, and failure at 475 and 468 seconds. Failure in both tests was by sagging from the top center of the frame, where the plastic had lost thickness and was no longer gripped by the frame (Figures 11, 13, 15, 17). In these tests, water was used as an extinguishing agent.

Two additional samples of Plexiglas G were tested, using carbon dioxide as an extinguishing agent. Nominal thickness of the material was 3/8-in. Extinguishment was at 420 and 360 seconds, prior to failure. Observations were made on the flowing tendency after extinguishment. In both cases, the plastic failed, at 435 and 425 seconds, by sagging from the top (Figure 27).

- (2) Plexiglas SE3 (Rohm & Haas), nominal thickness 3/8-in. Deflection was 1/8+ in., or less than with Plexiglas G under load. After ignition, inward deflection was noticed, followed by sizzling and sparking, evidence of burning, outward deflection (second test only), and failure at 513 and 526 seconds. Failure in the first test was by burn through, and in the second test, by sagging from the top. Much black pungent smoke was developed. (Figure 26). After the first test, steel objects in the laboratory were found heavily corroded.
- (3) Lexan (General Electric), nominal thickness 3/8-in. Deflection was 1/4-in., about the same as with Plexiglas G. After ignition, bubbling of the surface, outward deflection, and evidence of burning were noted. Failure was at 510 and 482 seconds, occurring as a melt-through of the lower center, followed by flowing into the alcohol pan. Much black, particulate smoke was developed, especially in the second test (Figure 12, 14, 16, 18).

Because of a tendency of thermoplastics like Lexan to flow when subjected to heat, three additional tests were performed, in which the fire was extinguished at 450, 360, and 420 seconds. Observations were made on the flowing tendency after extinguishment. No tendency to flow was observed after the second test, in which the alcohol pan was covered at 360 seconds; the Lexan did not fail. In the first and third tests however, the Lexan continued to flow after extinguishment with failure occurring at 510 and 440 seconds, accompanied by heavy smoke (Figures 23-25).

- (4) Flame-Resistant Lexan (General Electric), nominal thickness 3/8-in. The performance of this plastic was similar to that of plain Lexan, except that the smoke generated was more particulate. In the second test, the alcohol pan was covered by 390 seconds, yet failure occurred at 405 seconds. In both cases, after extinguishment, the plastic flowed and dripped into the alcohol pan (Figures 19-22).

c. Heat Contribution

Figure 1 compares the performance of Plexiglas G, Plexiglas SE3, Lexan, and laminated glass in the fire resistance test. Each curve represents an average of two or more fire tests. The temperatures

shown were recorded by a thermocouple located just below the top of the test frame. The solid curve shows the performance of wired glass, which was considered a suitable reference in that it made no heat contribution.

From the curve for Plexiglas G, for example, it appears that the plastic absorbed heat from the alcohol fire for about 1-1/4 minutes, then started contributing heat. It contributed more heat than the other materials. With Plexiglas SE3, it appears that it started contributing heat at about 2 min. and with Lexan, at about 4 minutes. These values are confirmed by observation of ignition during the respective tests. The heat contribution from laminated glass may be attributed to the burning vinyl inner layer.

C Smoke Effect Test

1. General

Although not originally planned as part of the test program, smoke effect tests were added after viewing the fire resistance tests. Some of the plastics had generated large volumes of black particulate smoke, and it was felt that this smoke could clog a high-efficiency filter (Figure 33). The smoke effect test was designed to evaluate this tendency.

2. Test Method

An assembly was constructed that would permit smoke from a burning plastic to be blown through a high-efficiency filter, as shown in Figures 30 and 31. The test apparatus consisted of a 6-in. diameter galvanized sheet steel tube connecting the exhaust of an electrically driven blower to a filter. An elbow of galvanized sheet steel was arranged to capture the smoke from a burning piece of plastic, which was supported on a wire screen above a 3-in. diameter pan of alcohol (Figure 32). The elbow was connected to the intake of the blower. An orifice plate downstream of the blower permitted flow readings on a draft gauge.

The test was begun by taping a new filter in position at the downstream end of the tube. The blower was then started, and the air flow adjusted to 50 cfm by partially blocking the downstream surface of the filter. The alcohol was ignited under the plastic and the clock started at the same time. Flow readings were made every half minute, and continued for 10-12 minutes, until the alcohol was consumed. Some tests were continued for an additional 10-12 minutes to determine whether the air flow would return to a level close to the original 50 cfm.

3. Materials Tested

Only three plastics were tested in this manner: Plexiglas G, Plexiglas SE3, and Lexan. Other plastics had been eliminated from consideration during the heat resistance tests, and there was no need to test glass products. The samples were 3-in. square and 3/8-in. thick.

4. Test Results

- a. Plexiglas G. The airflow dropped from 50 cfm to 29 cfm at 10 minutes in the first test, and to 31.5 cfm at 9.5 minutes in the second test. In the second test, the blower was allowed to run, and the airflow returned to 47 cfm at 18 minutes.
- b. Plexiglas SE3. The airflow dropped from 50 cfm to 35.5 cfm at 10.5 minutes, and recovered to 47.5 cfm at 20.5 minutes. In the second test, airflow dropped to 36 cfm at 10 minutes and recovered to 47 cfm at 18 minutes.
- c. Lexan. The airflow dropped from 50 cfm to 39 cfm at 11.5 minutes. In the second test, the airflow dropped to 37 cfm at 10.5 minutes. No recovery test was performed.

V DISCUSSION OF TEST RESULTS

On the basis of heat resistance, fire resistance, and smoke effect tests where applicable, certain statements can be made about window materials.

A Glass Products

1. Plate Glass. This was tested only because it appeared to be in use at AEC sites. Because of its tendency to fail quickly under heat, and to break into sharp shards, this product cannot be generally recommended for glovebox use. Its use should be continued only with an inert box atmosphere or with automatic box fire detection and extinguishing devices, and preferably with automatic sprinklers in the laboratory.

2. Laminated Sheet and Laminated Plate Glass. These products can be made to burn, but loss experience at AEC sites indicates that burning of laminated glass is unlikely. When laminated glass does burn, there is a wide variation in failure time.

There seems to be no safety advantage in laminated plate glass over laminated sheet glass, nor is there any advantage, according to FMRC tests, in adopting a 30-mil inner layer over the customary 15-mil inner layer.

3. Semi-Tempered and Tempered Glass. These products exhibit good resistance to heat. However, when failure occurs, they completely evacuate the frame and, in the case of tempered glass, create flying missiles. Tempered glass is inherently subject to spontaneous failure and should not be used in gloveboxes. Semi-tempered glass should be used only subject to the restrictions stated for plate glass.

4. Wired Glass. This is by far the most satisfactory of the glass products in terms of heat and fire resistance. Its use will probably be objected to by operators on the grounds of visibility. There should be no objection to its use in a top window for lighting purposes only, or in other applications where continuous viewing is not necessary.

5. Lead Glass. This product has similar properties to plate glass of the same thickness. It should be used as a demountable overlay on the exterior, over a safer window, or it should be subject to the same limitations as plate glass.

B Plastic Products

1. Plexiglas G. This widely used product can be readily ignited and can burn vigorously. If extinguishment is by water, it retains its dimensional stability, but if extinguishment is by carbon dioxide or any other agent without substantial cooling properties, it continues to soften and sag.

2. Plexiglas 5009. This is an improvement over Plexiglas G from a heat resistance standpoint, but it appears to have been superseded by Plexiglas SE3 and XL-20A.

3. Plexiglas SE3. By these tests this product is superior to both Plexiglas G and 5009. The smoke generated was intense, unpleasant, particulate, and appeared corrosive. The effect of this smoke on a high-efficiency filter was less detrimental than the smoke from Plexiglas G, however. This was probably so because the relatively large particles did not penetrate as deeply into the filter.

4. Plexiglas XL-20A. In the heat resistance test, this product appeared much better than Plexiglas G, but not quite as good as Plexiglas SE3. It generates very little visible smoke. Because it is not yet commercially available in 3/8-in. thickness, fire resistance tests were not performed.

5. Homalite 911 and Homalite 911XHT. These materials would be quite satisfactory, except that they completely evacuate the frame upon failure. In the case of 911XHT, as with tempered glass, the fragments become missiles which would be a decontamination problem. The use of these window materials should be continued only subject to the restrictions mentioned for plate glass.

6. Cellulose Acetate Butyrate. While this product performed reasonably well, further testing was discontinued when it was learned that its optical qualities were not good in the 3/8-in. thickness. Such a thickness was deemed necessary in an actual installation due to flexural requirements.

7. Polystyrene. The pieces delivered were optically poor due to waves in the surface and the surface was easily scratched. Its performance under heat exposure was only fair.

8. Lexan. All three of the tests--heat, fire, and smoke--indicate that this is the best plastic for glovebox window use, although not by a wide margin. Its resistance to burning is very good and its heat contribution quite low. The time to failure in the fire resistance test was not quite as long as with Plexiglas SE3, but it was felt that its low heat contribution and its low rate of smoke production offset this.

9. Flame Resistant Lexan. There appears to be no particular advantage to the use of this product over ordinary Lexan.

VI DISCUSSION OF OTHER (UNTESTED) PROPERTIES

A Impact Resistance and Hardness

ASTM tests D256 (impact) and D785 (hardness) apply to plastic materials and not to glass. A precise comparison between the various plastic and glass products is not possible on the basis of these tests alone. An indication of

relative impact resistance of laminated and tempered glass has been cited by Malecha (R. F. Malecha--see Bibliography), and between Plexiglas G and laminated glass by Dow Chemical Co. (Plexiglas (Lucite) --A Potential Fire Hazard--see Bibliography). From these and other sources, an approximate comparison of impact and hardness has been made and appears in chart form in VI-F below.

B Chemical Resistance

Glovebox windows may be exposed to a wide variety of chemicals in the form of acids, alkalis, and organic solvents in various concentrations. Glass windows are attacked by hydrofluoric acids. Plastic windows are attacked, and in some cases dissolved, by a number of different chemicals. The ASTM test involves total immersion for seven days, a condition not likely to be imposed on a glovebox window. Plastic windows of Plexiglas G, Plexiglas SE3, and Homalite are successfully being used. From a study of product literature, it appears that Lexan would be about as satisfactory, from the chemical resistance standpoint, as the plastics currently in use.

Before selecting a window material, the manufacturer's product data should be studied to assure compatibility with the expected glovebox environment. Care should be taken in cleaning and decontamination that harmful agents are not used.

C Explosion Resistance

No window material is truly explosion resistant; all are expected to fail under anything above a very minor overpressure. Failure may occur by fracture of the window or by flexure sufficient to allow slipping from the frame. While loss of containment is a distinct possibility in an explosion, three other associated hazards can be avoided by a proper choice of window material: (1) the personnel hazard from flying sharp shards, (2) the decontamination problem from scattered small window fragments, and (3) possible breakage of other glovebox windows by impact from large flying window fragments.

An excellent test report compares the explosion resistance of laminated glass, wired glass, tempered glass, and acrylic plastic (W. J. Taylor and R. O. Clark--see Bibliography). No comparative data, including data on other glasses or plastics, are known to be available. Engineering judgment has been applied to develop the ratings in the chart.

D Workability

Workability of plastics did not prove to be a problem for FMRC personnel. Homalite, Lexan, and Plexiglas were successfully sawed, using the manufacturers' recommended tools and methods. The criterion of workability, therefore, proved to be of less significance than expected.

E Radiation Darkening Resistance

Resistance to radiation darkening was not evaluated.

F Graphical Presentation of Criteria

Evaluation of glovebox window materials can be made in terms of six criteria: three safety requirements and three operating requirements. In the absence of more precise values, the more general terms "excellent," "good," etc. are used for comparison. The various window materials that were not eliminated on the basis of the discussion under "Test Results" are listed.

Glovebox Window Selection Criteria

<u>Material</u>	<u>Fire</u>	<u>Impact</u>	<u>Explosion</u>	<u>Abrasion</u>	<u>Chemical</u>	<u>Workability</u>
Plate Glass	Poor	Poor	Poor	Exc.	Good	Poor
Laminated Glass	Good	Good	Fair	"	"	"
Semi-Tempered Glass	Fair	Fair	"	"	"	"
Wired Glass	Exc.	Good	"	"	"	"
Lead Glass	Poor	Poor	Poor	Good	Fair	"
Plexiglas G	Fair	Good	Good	Poor	"	Exc.
Plexiglas SE3	"	"	"	"	"	"
Homalite	"	Fair	"	Fair	"	Good
Lexan	Good	Good	"	Poor	"	"

VII CONCLUSION

There is no universal glovebox material. For some applications, glass is preferred, but for others it cannot be used. Glass and plastic products may be ranked in the following order on the basis of safety and operating requirements:

Glass Products

Wired Glass
Laminated Glass
Plate and Lead Glass

Plastic Products

Polycarbonate (such as Lexan)
FR Methyl Methacrylate (such as Plexiglas SE3)
Methyl Methacrylate (such as Plexiglas G)

If the glovebox atmosphere is inert, semi-tempered glass and allyl diglycol carbonate can be added to the above lists, in this order:

Glass Products

Wired Glass
Laminated Glass
Semi-Tempered Glass
Plate and Lead Glass

Plastics Products

Polycarbonate
Allyl diglycol carbonate (such as Homalite 911 and 911 XHT)
FR Methyl Methacrylate
Methyl Methacrylate

The choice between glass and plastic window materials may be dictated by considerations of chemical attack, radiation effect, workability, economics, or some other factor. If the choice is not so dictated, glass and plastic materials can be grouped together as follows:

Wired Glass
Laminated Glass
Polycarbonate
FR Methyl Methacrylate
Methyl Methacrylate
Plate and Lead Glass

For gloveboxes with an inert atmosphere, semi-tempered glass and allyl diglycol carbonate can be added to the list:

- Wired Glass
- Laminated Glass
- Polycarbonate
- Allyl diglycol carbonate
- FR Methyl Methacrylate
- Methyl Methacrylate
- Semi-Tempered Glass
- Plate and Lead Glass

Where polycarbonate and methyl methacrylate glovebox windows are used, and where considerations of criticality permit, provision should be made for water cooling of the gloveboxes in event of fire. This can be done by automatic sprinklers or portable water type hand extinguishers. FMRC test experience has shown that the cooling effect of water on a burning thermoplastic can retard or prevent sagging and failure, thus preserving glovebox containment.

A lamination of semi-tempered glass offers promise for future investigations, when it becomes available in the desired thickness. This would have approximately double the heat and impact resistance of ordinary laminated glass, without the undesirable breakage characteristics of unlaminated semi-tempered glass.

Twenty-four leading chemical and plastic resin manufacturers were asked if they would participate in a sub-contract to develop a superior glovebox window material. Of the 12 who responded, two indicated interest in connection with products already under development. These were the Rohm and Haas Company, who produce Kydex, an alloy of PVC and acrylic, and Union Carbide Corporation, who produce polysulfone. Both products appear satisfactory for glovebox window use, but neither is sufficiently transparent as yet in the necessary 3/8-in. thickness.

RBB/ez

Attach. Tables 1-3

Figures 1-33

Bibliography

TABLE 1

Summary of Heat Resistance Tests

RP Test	Material	Ignition Time-Secs.	Remarks
24	1/4-in. Polished plate glass	None	Shattered at 25 seconds
25	"	"	" " 35 "
26	1/4-in. Dualite (laminated sheet glass)	125	Cracked at 31 seconds Separating at 78 "
27	"	107	Smoking at 105 " Cracked at 32 seconds Separating at 57 " Smoking at 72 "
28	1/4-in. Duplate (laminated plate glass)	108	Cracked at 30 seconds Separating at 85 " Smoking at 105 "
29	"	135	Cracked at 26 seconds Separating at 90 " Smoking at 105 "
12	1/4-in. Heat-Strengthened Glass (semi-tempered)	None	Shattered at 45 seconds
14	"	"	Shattered at 52 seconds
13	1/4-in. Herculite (tempered glass)	"	Disintegrated at 300 seconds with force, in plane of glass
16	"	"	Disintegrated at 95 seconds, as in RP13
10	3/8-in. Duplate (laminated plate glass - 1/4-in. layer exposed)	"	Cracked at 17 seconds
11	"	195	Cracked at 30 seconds Separating at 83 "
38	Same with 1/8-in. layer exposed	162	Cracked at 18 seconds Separating at 82 "
40	1/4-in. Lead Glass	None	Shattered at 26 seconds
41	"	"	" " 26 seconds
15	1/4-in. Wired Glass	None	Cracked at 24 seconds
23	"	"	" " 27 "
1	3/8-in. Plexiglas G (methyl methacrylate)	23	Moderate deflection " "
3	"	21	" "
8	"	26	Slight " "
17	"	26	" "

TABLE 1, Cont.

RP Test	Material	Ignition Time-Secs.	Remarks
21	1/2-in. Plexiglas G	45	Slight deflection
22	"	30	" "
4	1/2-in. Plexiglas 5009 (FR methyl methacrylate)	75	Moderate deflection. Inter- mittent flashing before ignition.
5	"	72	Same as RP4
18	3/8-in. Plexiglas SE3 (FR methyl methacrylate)	75	Slight deflection. Smoking, sizzling, and intermittent flashing before ignition.
19	"	74	Same as RP18
44	3/8-in. Plexiglas XL-20A (methyl methacrylate)	70	Slight deflection. Bubbling, sizzling, and flashing before ignition.
45	"	57	Same as RP44
6	3/8-in. Homalite 911 (allyl diglycol carbonate)	84	Large deflection. Particles thrown from surface. Sample broke after test.
9	"	32	Moderate deflection. Particles thrown from surface. Cracked at 33 seconds.
31	"	70	Moderate deflection. Particles thrown from surface. Piece fell out at 220 seconds, after test.
7	3/8-in. Homalite 911XHT (FR allyl diglycol carbonate)	None	Extreme deflection. Spark ing. Shattered at 42 seconds, after test.
20	"	78	Moderate deflection. Spark- ing. Shattered at 108 seconds, after test.
30	"	75	Moderate deflection. Spark- ing. Shattered at 105 seconds, after test.
32	1/4-in. Cellulose acetate butyrate	31	Smoking at 20 seconds. Burned rapidly and flowed.
33	"	40	Smoking at 23 seconds. Burned rapidly and flowed.
35	3/8-in. Polystyrene	42	Bubbling. Fire spread rapidly over surface.
36	"	34	Same as RP35. Dark smoke.

TABLE 1, Cont.

RP Test	Material	Ignition Time-Secs.	Remarks
34	1/4-in. Lexan (polycarbonate)	115	Bubbling. Slight deflection. Smoking at 95 seconds. Flowing after test.
39	"	72	Bubbling. Slight deflection. Browning at 60 seconds. Flowing after test.
42	1/4-in. F.R. Lexan	90	Bubbling. Moderate deflection. Browning at 82 seconds. No flowing.
43	"	95	Same as RP42, except browning at 80 seconds.

NOTE: All tests were conducted in numerical sequence. Plastic test specimens beginning with RP8 were fitted with a restraining steel frame to prevent excessive deflection.

TABLE 2

Summary of Fire Resistance Tests

DF Test	Material	Failure Time-Secs.	Remarks
6	1/4-in. Duolite (laminated sheet glass)	183	Cracked at 35 seconds Separating at 70 " Smoking at 135 "
7	"	925	Failure--burn through Cracked at 60 seconds Smoking at 105 " Browning at 220 "
10	"	395	Failure--fire on outside Cracked at 80 seconds Separating at 140 " Smoking at 265 "
11	"	500	Failure--burn through Cracked at 105 seconds Smoking at 225 " Separating at 290 " Failure--burn through
8	1/4-in. Wired Glass	None	Cracked at 35 seconds Continued cracking and dark- ening until test ended at 890 seconds.
9	"	"	Cracked at 33 seconds Continued cracking and dark- ening until test ended at 1070 seconds.
14	3/8-in. Laminated Safety (plate) Glass-laminated from two equal layers	810	Cracked at 54 seconds Separating at 130 " Smoking at 245 "
15	"	460	Failure--hole Cracked at 90 seconds Separating at 180 " Smoking at 330 " Failure--cracked through
21	1/4-in. Laminated Safety (plate) Glass-with 30-mil innerlayer	205	Cracked at 50 seconds Separating at 150 " Smoking at 165 "
22	"	696	Failure--burn through Cracked at 82 seconds Separating at 140 " Smoking at 195 " Failure--burn through

TABLE 2, Cont.

DF Test	Material	Failure Time-Secs.	Remarks
1	3/8-in. Plexiglas G (methyl methacrylate)	475	Deflection inward at 30 seconds. Burning at 130 " Deflection outward at 150 " Flames 4 ft. above box at 360 seconds. Failure--sagging from top.
2	"	475	Deflection inward at 80 seconds. " out at 170 " Flames 3 ft. above box at 240 seconds. Failure--sagging from top.
3	"	468	Burning at 105 seconds. Deflection outward at 180 seconds. Failure--sagging from top.
26	"	462	Similar to DF 1, 2, 3. Alcohol pan covered at 420 seconds. Plastic continued to sag. No water used.
27	"	435	Similar to DF 1, 2, 3. Fire extinguished by CO ₂ at 420 seconds. Plastic continued to sag.
28	"	425	Similar to DF 1, 2, 3. Fire extinguished by CO ₂ at 360 seconds. Plastic continued to sag.
4	3/8-in. Plexiglas SE3 (FR methyl methacrylate)	513	Deflection inward at 45 seconds. Loud sizzling, black smoke. Failure--burn through.
5	"	524	Deflection inward at 30 seconds. " outward at 165 " Sizzling, but less smoke than DF4. Failure--sagging from top.
12	3/8-in. Lexan (polycarbonate)	510	Deflection inward at 25 seconds. Bubbling at 40 " Deflection outward at 205 " Sizzling, particulate smoke. Failure--melt through.
13	"	482	Bubbling at 40 seconds. Deflection outward at 320 seconds. Sizzling, much particulate smoke. Failure--melt through.
18	"	510	Similar to DF 12,13. Alcohol pan covered at 450 seconds. Failure--melt through.

TABLE 2, Cont.

DF Test	Material	Failure Time-Secs.	Remarks
19	3/8-in. Lexan (polycarbonate)	None	Similar to DF 12,13. Alcohol pan covered at 360 seconds. No flowing or melting.
20	"	440	Similar to DF 12,13. Alcohol pan covered at 420 seconds. Plastic flowed. Failure--melt through.
24	3/8-in. F.R. Lexan (polycarbonate)	432	Deflection inward at 45 seconds. Bubbling at 50 " Dripping at 215 " Deflection outward at 228 " Particulate black smoke. Failure--melt through.
25	"	405	Deflection inward at 40 seconds. " outward at 265 " Dripping at 290 " Black smoke. Alcohol pan covered at 390 seconds. Plastic flowed. Failure--melt through.

TABLE 3

THICKNESS OF MATERIALS TESTED

<u>Material</u> <u>Heat Resistance Tests</u>	<u>Nominal Thickness, in.</u>	<u>Actual Average Thickness</u> <u>Per Sample, in.</u>
Plate Glass	0.250	0.229,0.232
Laminated Sheet Glass	0.250	0.226,0.225
" Plate "	0.250	0.256,0.260
" " "	0.375	0.374,0.374,0.376
Semi-Tempered "	0.250	0.245,0.243
Tempered "	0.250	0.242,0.245
Wired "	0.250	0.276,0.279
Lead "	0.250	0.284,0.286
Plexiglas G	0.375	0.340,0.362
" 5009	0.500	0.468,0.461
" SE3	0.375	0.361,0.345
" XL-20-A	0.375	0.376,0.374
Homalite 911	0.375	0.391,0.376
" 911XHT	0.375	0.382,0.373
Cellulose Acetate Butyrate	0.250	0.232,0.237
Polystyrene	0.375	0.384,0.398
Lexan	0.250	0.251,0.246
F.R. Lexan	0.250	0.262,0.260
<u>Fire Resistance Tests</u>		
Laminated Sheet Glass	0.250	0.231,0.229,0.223,0.223
" Plate "	0.375	0.418,0.418
" " "	0.250	0.268,0.259
Plexiglas G	0.375	0.387,0.385,0.358,0.347
Plexiglas SE3	0.375	0.353,0.351
Lexan	0.375	0.372,0.404,0.398,0.403, 0.369
F.R. Lexan	0.375	0.350

Figure 2.
General arrangements,
Heat Resistance Test.

Figure 3.
Wired glass, after
Heat Resistance Test.

Figure 4.
Laminated Plate Glass,
after Heat Resistance
Test.

Figure 5.
Plexiglas SE 3, after
Heat Resistance Test.

Figure 6.
Homalite 911, after
Heat Resistance Test.

Figure 7.
Lexan, after Heat Resistance
Test.

Figure 8.
F. R. Lexan, after Heat
Resistance Test.

Figure 9.
Test frame, Fire Resistance
Test.

Figure 10.
Deflection measurements,
plastic clamped in test
frame.

Comparative Fire Resistance Tests - Plexiglas G and Lexan

Figure 11.
Plexiglas G, 2 min.

Figure 12.
Lexan, 2 min.

Figure 13.
Plexiglas G, 4 min.

Figure 14.
Lexan, 4 min.

Comparative Fire Resistance Tests - Plexiglas G and Lexan

Figure 15.
Plexiglas G, 6 min.

Figure 16.
Lexan, 6 min.

Figure 17.
Plexiglas G, 7 min. 55 sec.

Figure 18.
Lexan, 8 min.

Fire Resistance Test - FR Lexan

Figure 19.
2 min.

Figure 20.
4 min.

Figure 21.
6 min.

Figure 22.
After failure at 7 min.
12 sec.

Figure 23.
Lexan, after Fire Resistance
Test, in which alcohol pan
was covered at 6 min. (no
failure)

Figure 24.
Lexan, after Fire Resistance
Test, in which alcohol pan
was covered at 7 min. Failure
was at 7 min. 20 sec.

Figure 25.
Lexan, after Fire Resistance
Test, in which alcohol pan
was covered at 7 min. 30 sec.
Failure was at 8 min. 30 sec.

Figure 26.
Plexiglas SE 3, after
Fire Resistance Test,
extinguishment by water
at 8 min. 23 sec. (failure).

Figure 27.
Plexiglas G, after Fire
Resistance Test, extin-
guishment by CO₂ at 6 min.
Failure was at 7 min. 5 sec.

Figure 28.
Laminated Sheet Glass at
3 min. 5 sec. (failure).

Figure 29.

Laminated Plate Glass at
13 min. 5 sec. Failure was
at 13 min. 30 sec.

Figure 30.

Laminated Sheet Glass,
after Fire Resistance
Test. Failure was at
15 min. 25 sec.

Figure 31.
Smoke Effect Test assembly,
general view.

Figure 32.
Smoke Effect Test assembly,
upstream end view.

Figure 33.
Filters after exposure
to Smoke Effect Test.

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