IITRI Project D6139
Final Report No. D6139

EVALUATION OF THE EFFECTS OF SOLAR RADIATION ON GLASS

for
National Aeronautics & Space Administration
George C. Marshall Space Flight Center
Alabama 35812

Contract No. NAS8-32521

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January 31, 1979
FOREWORD

The work described in this report, Final Report No. D6139, entitled "Evaluation of the Effects of Solar Radiation on Glasses," was performed under the sponsorship of the Marshall Space Flight Center. The work was conducted under Contract No. NAS8-32521, over the period May 6, 1977 to January 15, 1979, at IIT Research Institute. The Principal Investigator was Dr. R. Firestone and Mr. Y. Harada was the Program Manager. Space simulation tests were conducted at the Boeing Aerospace Company under the leadership of Mr. L.B. Fogdall.

We are pleased to acknowledge the valuable assistance and counsel of Mr. R.L. Nichols, NASA/MSFC Contracting Officer's Representative on this program. The fruitful discussions with Mr. Epaminondas Stassinopoulos and Dr. Alfred Eubanks of NASA/Goddard are also gratefully acknowledged.

Respectfully submitted,
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ABSTRACT

A program has been conducted for the investigation of the effects of solar radiation as encountered in a space environment, on glasses. Initial work was concerned with attempts to define the space environment. Secondly, a literature review was made on radiation damage mechanisms in glasses. Four optical materials were exposed to simulated solar and particulate radiation in a space environment. Sapphire and fused silica experienced little change in transmittance while optical crown glass and ultra low expansion glass darkened appreciably.
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EVALUATION OF THE EFFECTS OF SOLAR RADIATION ON GLASSES

1.0 INTRODUCTION

The objective of this research program was to evaluate the degradation of glass used on space structures due to electromagnetic and particulate radiation in a space environment. The data obtained can be used in choosing the optimum mechanically and optically stable glass materials for various long term space applications.

Glass components on a space structure must perform a wide variety of functions ranging from simple viewing ports for the visible region to lenses for UV and IR detection devices which transmit at wavelengths outside the visible region. Thus, degradation is a function of the intended use. For example, many IR transmitting materials may darken in the space environment to near opaqueness in the visible region, but their IR transmission remains virtually unaffected so that the functionality is maintained.

The space environment to be encountered by space vehicles is extremely complex, involving particulate as well as ultraviolet radiation. The penetrating radiation environment may result from a variety of sources of which the most important are probably cosmic radiation, trapped radiation, auroral radiation, and solar flare radiation. It is possible that such high energy protons and electrons will have a more significant effect on glasses than does ultraviolet, and that synergistic effects from the variety of radiations will occur.

In the sections which follow, an analysis of the space environment and its effects on optical materials is presented. The results of the research program to establish basic guidelines for choosing optimum materials are discussed. Recommendations are made for future work.
2.0 BACKGROUND

Four areas of importance were addressed in this program: materials, the space environment, the response of materials to the environment, and the selection of stable materials.

2.1 Optical Materials for Spacecrafts

The materials used aboard spacecraft for optical applications are in general those which transmit in the solar spectrum, nominally from 200 nm to 3000 nm. For the region 200-1000 nm silicon oxide based materials, notably fused silica, have found widest application. The selection of suitable optical materials, however, involves not only initial optical properties, but optical stability, mechanical properties and performance, and a host of other considerations, including contamination, contaminability, electrical properties, rf transparency, and, of course, cost.

From a materials point of view, there are three regions of optical interest: UV (<300 nm) visible (300-700 nm), and near IR (>700 nm). For UV applications, silica appears optimum, although some alkali halides might be considered; in any case, in UV applications, inorganic materials are almost mandatory. Visible region applications quite probably can utilize organic-based transparencies, except where UV transparency is also essential. IR applications, both traditionally and, quite logically, from materials initial properties and space stability considerations, have required inorganic materials. Organic materials inevitably exhibit either IR "fingerprint" spectra, poor mechanical properties, or other objectional properties. Simply stated, the primary criteria for selection of spacecraft optical materials are initial properties, environmental stability and mechanical/physical/environmental considerations. Only one region, the visible, was investigated on this program, and the materials were all inorganic oxides.

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2.2 **Space Environmental Stability**

Perhaps the most unpredictable factor in the use of optical materials in spacecraft applications is environmental stability. This, of course, is due to the tremendous variability in the space environment itself, and the difficulty in adequately simulating it. At synchronous and greater altitudes the charged particle environment varies so greatly in magnitude and is so sporadic that it is virtually impossible to estimate the long term performance of optical materials. Environmental stability thus depends on the materials involved, the environmental parameters and on time of exposure.

2.3 **Space Environment(s)**

The environment existing at synchronous altitudes (and at interplanetary distances from the earth) depends primarily upon distance from the sun and time. The electromagnetic flux is constant, and the fluxes of the particulate components of the solar flux vary strongly with solar activity. Over a 5-10 year period, however, the environmental parameters tend to approach an average value. Attempts at reproduction of the space environment at any synchronous or greater altitude represent merely that - attempts to duplicate the space environment at synchronous and at interplanetary distances. The primary problem is the temporal definition of the space environment; although the electromagnetic flux appears to be constant, the solar particulate flux is almost totally unpredictable. While in general, the character of the particulate flux is known, the spectral distribution (e.g., peak energy and peak width) of solar particulates varies with the solar substorm environment. In a substorm environment, the charged particle flux and the peak energy increase, as does the energy flux distribution. Even though the quantitative statistics may increase by several orders of magnitude, the average substorm remains unpredictable. Particle fluxes and energies with respect to time are highly statistical.
2.4 Optical and Mechanical Damage in Glasses

The choice of materials for visual applications on spacecraft is virtually unlimited. Silicate materials are frequently employed. The choice arises from many considerations, the most important of which is resistance to radiation damage, and darkening.

The response of most optical materials to ionizing radiation is such that great care must be exercised in their selection. Quite obviously, this choice must be based upon a knowledge of the spectral region in which high transmittance is required vs the spectral region(s) in which optical damage is induced. Though simply stated, the problem is indeed extremely complicated. Different types of radiation cause different types of effects at different penetration depths.

The mechanisms of degradation in transparent optical materials have been investigated extensively. Mechanistic approaches tend, however, to be sensitive to both materials and irradiation parameters and conditions. The effects of high energy electrons can be and often are greatly different from those of high energy protons, and the effects of the combination are frequently not a simple combination of the two. Some radiation effects are optically and/or thermally bleachable and others are annihilated upon exposure to oxygen. Accordingly, care must be taken to assure proper characterization of the materials and proper simulation of the space environment. Some materials display rate effects, possibly because they are dielectrics and quickly build up large electrical potentials upon exposure to charged particle radiation. The influence of this charge buildup is a multiple one: it discriminates against all radiations of like charge and equal or lesser kinetic energy; distorts the incident beam geometrically; and induces an electrical stress in the material which may predispose it to mechanical failure or to the formation of defects not otherwise possible or likely. Thus, the choice of operating parameters can be a critical one.
Measuring the spectral transmittance of a test material can also be a difficult matter. Most measurements have been made in a simple normal-normal transmission mode: the sample is illuminated at normal incidence and the undiffused (normal) transmitted radiation is detected. Normal-hemispherical measurements are very uncommon, yet in many applications, it is the hemispherical (diffuse) transmittance that is needed.

Theory regarding the transmittance vs angle of incidence and polarization has been well established. Unfortunately, however, the effects of radiation on the bidirectional properties of optical glasses have not been studied. These properties can become very important, especially in thin film filters and anti-reflection coatings.

The overall spectral region in which radiation damage is inflicted is relatively independent of the type of radiation. This is generally true in terms of spectrally integrated transmittance values. Differences, in terms of spectral effects, can be elicited between electrons and protons; sometimes the differences are subtle, requiring high wavelength resolution of the spectra.

In general, charge particle radiation effects are extremely difficult to analyze, especially in a multi-component irradiation environment. Nevertheless, a complete theoretical background is available. The classic work of Seitz (Ref. 4), for example, provides an extensive and detailed summary of radiation effects, degradation mechanisms and solid state analyses of the structure of color centers. Dexter's work (Ref. 5) established the quantitative relationships between induced defect concentrations and the resultant optical effect.

From careful analyses (Rejs. 6-8, e.g.) of the transmittance spectra of fused silica before and after charged particle irradiation, we can discern clearly three major defects created in this
material at 3 eV, 4.1 eV and 5.6 eV (Ref. 6). Similar analyses have been made for other glass systems (e.g., Refs. 9-12). Work on alkali halides is even more extensive (see, e.g., references in Ref. 4). Many of the techniques developed in the cited studies are highly relevant, although the results may not be.

For materials whose primary function is optical transparency transmittance measurements are essential. Such measurements must be made in-situ to avoid change on exposure to earth conditions.

Apart from the mechanisms that operate in glasses to degrade their transmittance properties, there are additional reasons for determining the effects of the radiation environment in space. Energetic charged particles (electrons and protons, principally) include fields in dielectric materials such as glasses. If field intensities exceed the local dielectric strength, material breakdown (additional absorption, cracking, loss of vacuum integrity, etc.) may result. NASA, IITRI, and Boeing have all conducted studies in this area.

2.5 Summary

The space environment is extremely complex and variable, and depends on the particular mission and time of the mission. The response of glass materials in terms of optical and mechanical behavior must be analyzed with the most accurate definition of the electromagnetic and particulate radiation to be encountered.

3.0 RESULTS AND DISCUSSION

The objective of the program was to generate data in the following areas:

1. Identification and quantification of the space radiation environment on a space station mission.

2. Analysis of radiation damage mechanisms in different glasses, and determination of merit ranking for various glass systems.

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3. Laboratory studies involving glass systems with an expected range of poor to excellent resistance to radiation damage.

The research program was conducted in three phases which are as follows:

1. Review of open literature and discussions with cognizant government/industrial personnel to accomplish points 1 and 2 above. Recommendation of glass systems representing poor to excellent resistance to radiation, for MSFC concurrence for laboratory studies.

2. Space simulation studies of the aforementioned glass systems. Evaluation and analysis of induced changes in optical and mechanical properties.

3. Recommendations for additional study. Details of these studies are presented in the following sections.

3.1 Phase One: Background Studies

3.1.1 Identification of Space Environment

The highly complex and dynamic nature of a space environment demands in situ and current data for good definition. This type of information is best obtained from a source such as the Goddard Space Flight Center which continually monitors and analyzes such data. IITRI has established a dialogue with Mr. Epaminondas G. Stassinopoulos, Senior Acquisition Scientist, Radiation Environment.

Mr. Stassinopoulos pointed out that the space environment near the earth was extremely variable, both spatially and temporally. Photon radiation remains relatively constant at 1 Sun but particulate radiation, which is mainly protons and electrons, strongly depends on the orbit of the spacecraft and the solar activity during the time in orbit.
The space environment in which the glasses are to be used was further discussed with Mr. Ron Nichols and Mr. Jerry Wright, Astrophysics, and with Mr. Peter Priest, Space Programs, NASA/MSFC.

The conclusion from these discussions was that average conditions should be selected for the initial simulated solar radiation experiments. Later experiments could then explore the effect of variations from average.

The conditions which were selected for the space simulation testing carried out by Mr. Lawrence Fogdall, Boeing Aerospace Company, are listed below:

Table I
Space Simulation Testing Conditions

<table>
<thead>
<tr>
<th>Condition</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Continuum Ultraviolet Radiation (Xenon arc):</td>
<td>suns</td>
</tr>
<tr>
<td>Vacuum Ultraviolet Radiation (121.6 nm):</td>
<td>1 Sun</td>
</tr>
<tr>
<td>Electrons: 1 X 10^9 e/cm^2 - sec flux at 50 KeV</td>
<td></td>
</tr>
<tr>
<td>Protons: 1 X 10^9 P/cm^2 - sec flux at 30 KeV</td>
<td></td>
</tr>
<tr>
<td>Temperature: 20°C</td>
<td></td>
</tr>
<tr>
<td>Vacuum: greater than 5 x 10^-8 Torr</td>
<td></td>
</tr>
</tbody>
</table>

These conditions do not exactly simulate conditions in space since the higher energy electrons and protons are absent, but they should show any effects of solar radiation on glass and are within the equipment capabilities.

3.1.2 Literature Review. Radiation Damage in Glasses

The review of literature to analyze radiation damage mechanisms in glasses was initiated by a computer search of the technical literature performed in conjunction with the IITRI Computer Search Center. The search was primarily concerned with the period from 1964 to date since an annotated bibliography of
the literature from before 1964 was available. The search strategy was to make a broad first search with shallow indexing to include all possible documents, and then to sharpen it as familiarity with the literature was gained. The data bases searched are listed in Table II. The results are contained in Appendix A-Selective Bibliographs: The Effects of Radiation in Glass.

The types of radiation damage to glass systems can be optical and/or mechanical. Loss of transmission at different wavelengths can occur due to: color center formation; discoloration due to valency changes in the chemical constituents; and to opacification from devitrification. Mechanical damage can be in the form of brittle failure or breaking, pitting from particulate impingement causing loss of transmission or flaws which can act as crack initiators, or spalling due to electrical breakdown.

The damage depends upon the glass composition and thermal history, the types of particulate radiation and the wavelengths of electromagnetic radiation, the duration, energy spectrum and combinations of radiation, and the chemical and physical environment of the glass. The damage can be either superficial or bulk, depending primarily on the type of radiation. In fact, there are so many parameters that it is impossible to make valid generalizations and a priori predictions of glass performance.

3.1.3 Establishment of Glasses for Space Simulation Studies

In order to establish a merit ranking of various glasses, a dialogue was conducted with Dr. Alfred G. Eubanks, Senior Scientist, Ceramics Materials, NASA Goddard Space Flight Center. Dr. Eubanks advises on the selection of optical materials for space missions of approximately one year duration such as the Nimbus spacecraft. The approach taken is to use radiation resistant materials, or to prevent degradation.
<table>
<thead>
<tr>
<th>NAME</th>
<th>TITLE</th>
<th>DATES</th>
<th>NUMBER OF CITATIONS</th>
<th>DESCRIPTION</th>
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<tbody>
<tr>
<td>CDA</td>
<td>Comprehensive Dissertation Abstract</td>
<td>1861-date</td>
<td>550,000</td>
<td>Virtually all American Doctoral Dissertations from all scientific and technical disciplines are covered</td>
</tr>
<tr>
<td>CHEMCON</td>
<td>Chemical Abstracts Condensates</td>
<td>1970-date</td>
<td>1,000,000</td>
<td>Proceedings, patents, reports, books and reviews of U.S. and foreign material in chemistry and chemical engineering</td>
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<tr>
<td>CLAIMS</td>
<td>IFI/Plenum</td>
<td>1975-date</td>
<td>4,500</td>
<td>American general, electrical and technical patents</td>
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<tr>
<td>COMПENDEX</td>
<td>Engineering Index</td>
<td>1970-date</td>
<td>400,000</td>
<td>Includes applied and theoretical papers proceedings, books and publications, (3500 journals)</td>
</tr>
<tr>
<td>INSPEC</td>
<td>Physics Abstract</td>
<td>1969-date</td>
<td>471,000</td>
<td>Worldwide coverage of all aspects of physics from over 2,000 journals</td>
</tr>
<tr>
<td>NASA</td>
<td>National Aeronautics and Space Administration</td>
<td>1962-date</td>
<td>600,000</td>
<td>Abstracts of published and unpublished reports of material found in &quot;International Aerospace Abstracts&quot; (IAA) and &quot;Scientific and Technical Aerospace Reports&quot; (STAR)</td>
</tr>
<tr>
<td>NTIS</td>
<td>National Technical Information Service</td>
<td>1964-date</td>
<td>500,000</td>
<td>The complete Government Reports Announcements from over 200 agencies of NASA, DDC, AEC, ERDA, HEW, DOT and others</td>
</tr>
<tr>
<td>SCI-SEARCH</td>
<td>Institute for Scientific Information</td>
<td>1974-date</td>
<td>800,000</td>
<td>Citations and bibliographic information gleaned from 2500 periodicals in the physical and life sciences</td>
</tr>
<tr>
<td>SSIE</td>
<td>Smithsonian Science Information Exchange</td>
<td>1974-date</td>
<td>120,000</td>
<td>Abstracts and projects descriptions of on-going research in all areas of research</td>
</tr>
</tbody>
</table>
Their standard radiation resistant optical materials are sapphire and Corning 7940 fused silica glass. These are used for the front elements of optical systems whenever possible. If other materials must be used, a 2-3mm thick fused silica glass window is used in front of the optical components. The window eliminates almost 90% of the particulate radiation and also the very short wavelength ultraviolet. If a window can not be used, then the equipment is designed to compensate for the degradation. For example, solar cell cover glass, Corning 0020, degrades approximately 5% in one year when exposed directly to solar radiation. Hence, for a one year mission, the cell is designed to have an initial output which is 5% greater than required.

The conclusion from this discussion was that sapphire or fused silica glass were the best materials available for resisting degradation by solar radiation in space.

A further review of the literature supported this conclusion and was used to develop the following ranking of resistance to radiation damage:

1. Excellent - sapphire
2. Good - fused silica
3. Fair - optical glass
4. Poor - ultra low expansion glass

These materials were chosen for exposure in a simulated space environment for the following reasons:

1. Sapphire is exceptionally strong and hard (9 on the Mohs scale) and is by all reports the best material for resisting radiation damage. While it is not a glass, it is available in single crystal form which is nearly as transparent as glass and readily available in sizes up to 12 inches diameter. Union Carbide Czochralski grown sapphire crystals were chosen since they are more uniform than Verneuil grown crystals and are produced in quantity for IC substrates.
2. Fused silica is a close second to sapphire in strength and hardness (7 on the Mohs scale) and in radiation resistance. It has been used on numerous space missions. It is available in sizes up to 10 feet in diameter. Corning 7940 synthetic fused silica was chosen because it is a well characterized, uniform material with very low solar absorptance.

3. Optical crown glass is used in many optical devices as the front element of the objective lens. It was observed to darken when exposed directly to the space environment in Spacelab experiments. Shott BK-7 was chosen since it is the most widely used glass of this type and readily available.

4. Ultra low expansion glass is a very interesting optical material which is almost immune to thermal shock failure. Window made of this material, either simple or composite, could be used for observation during the thermal heating caused by reentry without failure. Corning 7971 was chosen because it was readily available.

These four materials have had or may have wide application in the space program. Their differences in composition and atomic structure will aid in analyzing radiation damage in optical materials.
3.2 Phase Two: Space Simulation Studies

3.2.1 Simulated Space Exposure Apparatus

The simulated space exposure for this program was performed in a facility at Boeing known as the CRETC (combined radiation effects test chamber). This facility was originally designed after completion of a number of years of testing thermal control coatings for government and corporate customers. It has a high quality of simulation of the space radiation environment as knowledge and the state-of-the-art will allow. Exposure capabilities include electron radiation and Lyman-α vacuum ultraviolet sources in addition to the usual proton accelerator and solar simulator.

The space simulation capabilities of the CRETC facility can be summarized as follows:

1. Continuum ultraviolet radiation (xenon arc discharge) at selectable intensities ranging from less than one solar constant to 20 solar constants (1 A.U.), simultaneously with:

2. Electrons with energies between approximately 10 eV and 200 keV and/or protons with energies from 0.5 to 85 keV (kilo electron volts). Electrons of greater than ~15 KeV are foil-scattered; protons are magnetically analyzed.

3. Vacuum ultraviolet radiation (VUV), primarily the Lyman-α wavelength of 1216 Å, from a contained discharge (no introduction of contamination into vacuum chamber). Intensity selectable up to and above one VUV sun at 1216 Å at 1 A.U.
4. Controlled temperatures for test and reference (standard) samples; temperatures range from -195°C (-320°F) to +180°C (+360°F). Temperature control is not interrupted for measurements in the chamber's integrating sphere.

5. Vacuum pumping (both rough and final) without resorting to organic and other contaminating fluids. The sequence used is (a) dry nitrogen gas aspiration, (b) cryo-sorption, (c) large-surface LN₂ cryogenic, and (d) ion pumping, to obtain a $5 \times 10^{-8}$ torr vacuum before testing begins.

6. Extensive automation, interlocks, and sequential shutdown procedures during unmanned night-time operations, to allow as high a reliability as possible during long-term, continuous testing.

7. High-precision spectral reflectance data system with in situ integrating sphere and double-beam spectrophotometer coupled to a data-logging module whose output is ready for computer processing.

8. Residual gas analysis from 1-100 amu with scan rates down to 0.1 second/scan and minimum partial pressure detectability of $2 \times 10^{-11}$ torr.

9. Electrical discharge event counter with relative magnitude indication.

The CRETC utilizes an integrating sphere reflectometer with detectors in situ. Only the measurement light sources, monochromator, and electronic and light chopping apparatus are external to the chamber. Sample reflectance measurements are made relative to the reflectance of the integrating sphere's magnesium oxide wall. Normalization to absolute reflectance (derived from

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National Bureau of Standards and other known reference measurements) is handled by computer since all original sample data is computer-processed routinely.

The thermophysical property of chief interest for this program, solar absorptance, is derived from solar reflectance, which is defined as

\[
\text{Solar reflectance, } R_s = \frac{\int I_s(\lambda)R(\lambda)d\lambda}{\int I_s(\lambda)d\lambda}
\]

where \( I_s(\lambda) \) is the solar irradiance as a function of wavelength \( \lambda \), and \( R(\lambda) \) is sample reflectance, generally a function of \( \lambda \). By measuring the reflectance of transmissive glasses with a suitable metal backing (aluminum), double sensitivity to radiation-induced changes is obtained. The measurement beam passing through the integrating sphere makes a double-pass through the sample, yielding a measurement proportional to change in transmission squared. This allowed a more exact determination of damage in the various optical quality glasses studied and compared during this program. In practice, change in transmission generally will be found to be half the measured change in reflectance.

Radiation dosimetry systems are integral to the CRETC and operating personnel have long-time experience in obtaining the pertinent irradiation and exposure parameters. Faraday cups and tabs are both employed for measuring on-coming particle radiation beams at the sample plane.

For ultraviolet radiation parameters, sun rates are determined from radiometer output levels taken with and without a UV-absorbing filter over the radiometer detector. Since the UV-absorbing filter also excludes ten percent of the incident radiation at wavelengths longer than the ultraviolet (five percent reflection
at each surface of the filter), a correction is made for radiometer readings taken with the filter over the radiometer sensor. For a total radiation reading $T$ and a UV-filtered reading $F$,

$$
T - \frac{10}{9} F
$$

Ultraviolet Sun rate $= \frac{(8.0)(0.091)}{1.37 (T - 1.11 F)}$,

where 8.0 is the radiometer sensitivity in millivolts per incident sun (=0.14 watts/cm²) and 0.91 represents the ultraviolet content of the sun (at air mass zero). The uniformity of ultraviolet radiation intensity across the sample array is determined by "mapping" with the radiometer held in a precise jig. Spatial uniformity of ultraviolet radiation can be maintained within plus or minus 10 percent across the sample array. The $F$ and $T$ values indicate that the ultraviolet content of the long-arc xenon sources is approximately 10 percent of their total input. Characteristic of all xenon arcs, the shape of this ultraviolet content is somewhat more steep than the sun at air mass zero (AMO).

3.2.2 Specimen Preparation

Specimens were not available from a single source and in most cases were obtained directly from the manufacturer. The sapphire specimens were obtained from the Crystal Products Division of Union Carbide Corporation as standard optical windows 25.4 mm diameter and 2 mm thick. The optical glass specimens were also standard windows, 25.4 mm diameter and 3mm thick obtained from Melles Griot Inc., Irvine, California. The fused silica and ultra low expansion glasses were obtained from the Corning Glass Works as rough cut discs. They were ground and polished to 2.5.4mm diameter and 3mm thick by the Karl Lambrecht Corporation, Chicago, Illinois.

All the specimens were made of commercially produced materials. Standard industrial pitch polishing procedures were used to produce specimens which were flat within 1/4 wavelength of...
light per 25mm, parallel within 30 seconds of arc, and surface quality of 80-50 scratch and dig.

Samples were cleaned and approximately one half of the surface of each sample was coated with aluminum and then with silica by vacuum evaporation. The coating provided a back surface mirror for transmission optical measures by double reflection.

The samples of the selected glasses were furnished to Boeing Airospace Corporation. Brief descriptions of the glasses, the number of specimens supplied, and the number of each type put into the exposure array, are indicated below.

Table III
WINDOW MATERIALS FOR SPACE SIMULATION STUDIES

<table>
<thead>
<tr>
<th>GLASS TYPE</th>
<th>NO. SUPPLIED</th>
<th>NO. TESTED</th>
</tr>
</thead>
<tbody>
<tr>
<td>7971 (low expansion glass)</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>BK-7 (crown glass)</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>7940 (fused silica)</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Sapphire (alpha-alumina)</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

The test array configuration was three horizontal rows having two samples each. Each sample is approximately one inch in diameter, so a 2-inch by 3-inch (approximate dimensions) array was irradiated. The sample array, as placed in the Boeing CRETC II chamber, is shown in Figure 1. The reflective half of each sample was mounted closest to the center of the array. The CRETC II's spectrophotometer's measuring beam (approximately 1/4-inch square) measured the centers of the reflective halves during each periodic measurement cycle.
Figure 1  Specimens in radiation chamber before exposure. (0.5X)
The best six samples were selected by Roeing from the ten supplied for testing. The six actually irradiated were specimen numbers 1 and 2 of type 7971; 2 and 3 of type BK-7; specimen number 1 of type 7940; and sapphire specimen number 1. These full designators and abbreviations used on data charts and graphs during the program are indicated below. The two BK-7 samples were

<table>
<thead>
<tr>
<th>DESIGNATION OF TESTED SAMPLES</th>
<th>CHART ABBREVIATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>7971, Specimen #1</td>
<td>71-1</td>
</tr>
<tr>
<td>7971, Specimen #2</td>
<td>71-2</td>
</tr>
<tr>
<td>BK-7, Specimen #3</td>
<td>K-3</td>
</tr>
<tr>
<td>BK-7, Specimen #2</td>
<td>K-2</td>
</tr>
<tr>
<td>7940, Specimen #1</td>
<td>4-1</td>
</tr>
<tr>
<td>Sapphire, Specimen #1</td>
<td>S-1</td>
</tr>
</tbody>
</table>

placed in the top row of the exposure array, the two 7971 samples in the middle row, and the sapphire sample and the 7940 sample in the bottom row.

3.2.3 Exposure Conditions

Irradiation parameters were as follows:

1. Solar UV—one sun intensity, assuming Earth orbit and air mass zero conditions; 0.2- to 0.4-micrometer spectral content from xenon arc continuum; infrared $\lambda > 1.4$ micrometer suppressed by water column around xenon arc; radiation introduced into CRETC II chamber through fused silica window.

2. Vacuum UV—one sun intensity at hydrogen Lyman-$\alpha$ wavelength (1216 Å); radiation introduced into chamber through the VUV source's window.

3. Electrons—intensity (flux) $1 \times 10^9$ e/cm$^2$-sec, at 50-KeV energy.

4. Protons—intensity $1 \times 10^9$ p/cm$^2$-sec, at 30-KeV energy; introduced into chamber after magnetic bending to separate mass-one ($H^+$) from $H_2^+$ and other species.
The protons were incident upon the sample array at a normal angle. The other forms of radiation were incident at angles approximately, 30° from the normal. Their intensities were increased by a factor equal to the secant of 30°.

Uniformity of radiation over the exposure array was ±10% for solar UV, ±20% for vacuum UV, ±15% for protons, and ±5% for electrons. Constancy with time was ±10% for solar UV, +0 to -10% for VUV, ±20% for protons, and ±20% for electrons.

The reflective coating of the test samples was held at a temperature of 20°C throughout the test. All six samples were mounted on a copper block through which water at 20°C was circulated once and returned to city drains.

The specimens were in a contamination-free hard vacuum throughout the irradiation and measurement periods of the test. Average levels were 5 x 10⁻⁸ torr during exposure periods and 2 x 10⁻⁸ torr during spectral reflectance measurement periods. Vacuum was obtained with a sequence of (1) dry nitrogen aspiration, (2) cryosorption, (3) cryogenic surface pumping, and (4) ion pumping (no mercuric or organic pumping fluids was involved in any step).

The hemispherical spectral reflectance value of each sample was measured using precise double-beam, ratio-recording equipment indicated in Figure 2. Measurements were made before exposure, after 50 hours, after 154 hours, after 313 hours, and finally after 503 hours exposure, without breaking vacuum. At these times the following proton and electron fluence values were reached:

<table>
<thead>
<tr>
<th>EXPOSURE TIME</th>
<th>PROTON FLUENCE</th>
<th>ELECTRON FLUENCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>50</td>
<td>1.8 x 10¹⁴ p/cm²</td>
<td>1.8 x 10¹⁴ e/cm²</td>
</tr>
<tr>
<td>154</td>
<td>5.5 x 10¹⁴</td>
<td>5.5 x 10¹⁴</td>
</tr>
<tr>
<td>313</td>
<td>1.1 x 10¹⁵</td>
<td>1.1 x 10¹⁵</td>
</tr>
<tr>
<td>503</td>
<td>1.8 x 10¹⁵</td>
<td>1.8 x 10¹⁵</td>
</tr>
</tbody>
</table>

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Each reflectance measurement scan resulted in an 11 x 17 inch chart of continuous wavelength data (not point-by-point data). To provide very fine wavelength resolution, separate charts were made for three wavelength regions: 250-360 nanometers, 360-710 nanometers, and 710-2500 nanometers. Simultaneously with each scan, computer cards were punched and subsequently processed to provide graphs of absolute reflectance data as a function of wavelength.

3.2.4 Effect of Simulated Space Exposure

Examination of the specimens immediately after 503 hours of exposure showed that the optical glass and ultra low expansion glass specimens were appreciably darkened. All specimens were intact after exposure and did not suffer any gross mechanical damage. Since the specimens were in good thermal and electrical contact with the water cooled copper support, heating and charge buildup during exposure was probably very small.

The graphs of the absolute reflectance data for each specimen are shown as Figures 3 through 8. In each of these figures the uppermost data curve was obtained before exposure began. Small reflectance changes were measured at each subsequent measurement point, with the 503-hour data being represented by the lowest curve.
Figure 3. Effect of real-time solar UV, vacuum UV, protons, and electrons on the reflectance of BK-7 crown glass in situ.
Figure 4: Effect of real-time solar UV, vacuum UV, protons, and electrons on the reflectance of BK-7 crown glass in situ.

Hemispherical Spectral Absorption

Hemispherical Spectral Reflectance
FIGURE 5. EFFECT OF SOLAR UV, VACUUM UV, PROTONS, AND ELECTRONS ON THE REFLECTANCE OF 7971 LOW EXPANSION GLASS IN SITU

HEMISPHERICAL SPECTRAL ABSORPTANCE

HEMISPHERICAL SPECTRAL REFLECTANCE
FIGURE 6. EFFECT OF SOLAR UV, VACUUM UV, PROTONS, AND ELECTRONS ON THE REFLECTANCE OF 7971 LOW EXPANSION GLASS IN SITU
FIGURE 7. EFFECT OF REAL-TIME SOLAR UV, VACUUM UV, PROTONS, AND ELECTRONS ON THE REFLECTANCE OF 7940 FUSED SILICA IN SITU
FIGURE 8. EFFECT OF REAL-TIME SOLAR UV, VACUUM UV, PROTONS, AND ELECTRONS ON THE REFLECTANCE OF SAPPHIRE IN SITU.

HEMISPHERICAL SPECTRAL ABSORPTANCE

HEMISPHERICAL SPECTRAL REFLECTANCE

WELL

HMISPHERICL SPECTRAL

REFLECTANCE

23
The computer program also calculated solar absorptance coefficient values corresponding to each measurement. These are tabulated below.

Table V
SOLAR ABSORPTANCE CHANGES IN SPACE SIMULATION STUDIES

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>PRE-IRRAD IN VACUUM</th>
<th>AFTER EXPOSURE FOR 50 HR.</th>
<th>AFTER EXPOSURE FOR 154 HR.</th>
<th>AFTER EXPOSURE FOR 313 HR.</th>
<th>INCREASE AFTER 503 HR.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sapphire</td>
<td>0.109</td>
<td>0.110</td>
<td>0.115</td>
<td>0.118</td>
<td>0.009</td>
</tr>
<tr>
<td>Crown #3</td>
<td>0.094</td>
<td>0.106</td>
<td>0.109</td>
<td>0.114</td>
<td>0.118</td>
</tr>
<tr>
<td>Crown #2</td>
<td>0.098</td>
<td>0.109</td>
<td>0.111</td>
<td>0.117</td>
<td>0.119</td>
</tr>
<tr>
<td>Low exp. #1</td>
<td>0.085</td>
<td>0.092</td>
<td>0.094</td>
<td>0.101</td>
<td>0.108</td>
</tr>
<tr>
<td>Low exp. #2</td>
<td>0.083</td>
<td>0.088</td>
<td>0.090</td>
<td>0.097</td>
<td>0.108</td>
</tr>
<tr>
<td>Fused Silica</td>
<td>0.078</td>
<td>0.080</td>
<td>0.081</td>
<td>0.088</td>
<td>0.087</td>
</tr>
</tbody>
</table>

The change in the absorptance of the sapphire and fused silica specimens was very small and close to the limit of accuracy of the measurements.

A plot of these $\alpha_s$ values against exposure time (Figure 9) shows that sapphire and fused silica have no particular initial increase in $\alpha_s$, but rather have a somewhat regular $\alpha_s$ increase rate. On the other hand, the crown glass samples and the low expansion glass samples underwent larger initial increases in $\alpha_s$ that tapered to the slower rates of increase more typical of fused silica and sapphire. (Scatter in $\alpha_s$ data values, such as fused silica at 313 hours, up to about 0.005 can be attributed to the reflectance measuring apparatus).
Figure 9. Solar Absorptance vs Exposure to Simulated Space Environment
3.2.5 Discussion

Examination of the reflectance curves before and after various exposure times does not reveal the appearance of any new absorption regions. Rather for all specimens there is an increase in absorption with time, particularly at the fundamental absorption edge in the near UV. The damage threshold, if any must be less than 50 hours for all specimens. The absorptions at 1.2 and 2.2 micrometers of the glasses relate to the silica network and is most pronounced in fused silica. Sapphire being alumina does not, of course, show these. It is surprising that the modifiers present in the optical glass and ultra-low expansion glass do not result in new absorptance regions after exposure although they do suppress the silica regions in optical glass.

The change in solar absorptance values with time which was tabulated in the previous section and shown in Figure 9 indicate that the a priori ranking of the optical materials was correct. Sapphire is the most stable material followed by fused silica, crown optical glass and ultra-low expansion glass. For some applications fused silica may be preferred because of its lower initial absorption since even after 503 hours of exposure it was still less than sapphire. The optical glass and ultra low expansion glass on the other hand, while reasonably stable for 503 hours, would have limited usefulness for very long space missions.

3.3 Phase Three: Recommendations for Future Work

The recommendations for future work can be divided into two parts: further analysis of the specimens exposed in this program and additional exposures in simulated space environments.

The principal method used to characterize the specimen changes induced by simulated solar exposure was optical transmittance by a reflected beam technique. Although this is a true
measurement of the change in transparency of the materials, it
did not add in indentification of the mechanisms causing the
changes since the change was fairly uniform.

Thermoluminescence is a technique which can be used to
identify changes caused by irradiation. In this method, the
specimen is slowly heated and the light given off (luminescence)
is measured. The mechanism causing the changes may be inferred
from the brightness and color of the light as a function of

Windows for space missions are required to not only have
transparency but also mechanical integrity after exposure.
There were no gross changes in the integrity of the specimens
but there may have been subtle changes which could cause
failure on a long mission. The effect of exposure on the
mechanical properties of the specimens can be determined by
measuring the microhardness as a function of depth from the
exposed surface.

Additional exposures should be carried out under conditions
in which each parameter: light and particulate beams; are varied.
This would allow the effect of each parameter to be determined,
and also allow the presence of synergetic interactions to be
detected.

Longer exposures also should be made since 503 hours is
only about three weeks, an insignificant part of the time
required for some long space missions which may last for years.

Particular emphasis should be placed on studying the effects
on optical glass materials which are suitable for front elements
of optical instruments or which are available in large sizes.
REFERENCES


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REFERENCES (Cont'd)

APPENDIX

SELECTIVE BIBLIOGRAPHY
ON
THE EFFECT OF RADIATION ON GLASS


Naohiro Soga and Megumi Tashiro, (Univ. Kyoto, Japan), "Role of Cerium Ion in Pretreating the Coloration of Glass Induced by γ Rays," Yogyo Kyokai Shi 70, pp. 143-7, (1962).


