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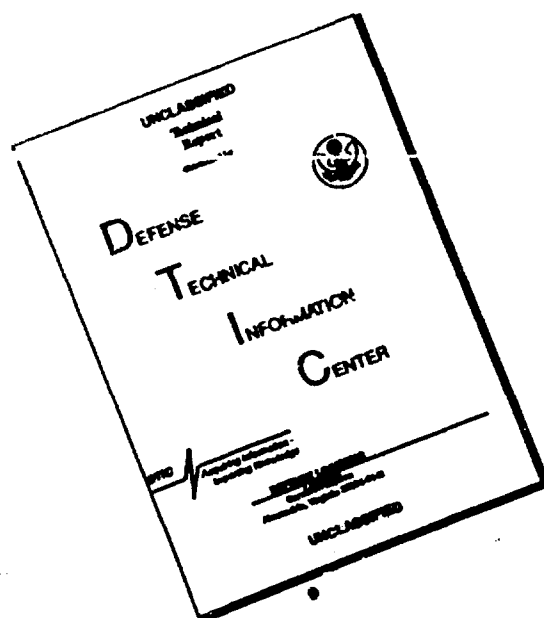
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Degradation of Vacuum-Exposed SiO₂ Laser Windows

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ABSTRACT

Observations of a damage phenomenon at the surface of fused silica and crystalline quartz windows are presented. Uncoated windows were mounted at Brewster's angle to facilitate the introduction of a vacuum chamber directly into the cavity of an Ar-ion laser (488 - 514 nm). The transmission of these windows, prior to evacuating the chamber to less than 1 Torr, approaches the theoretical value of $> 99.9\%$, remaining constant indefinitely. However in our normal usage, the chamber is evacuated ($P < 10^{-7}$ Torr), exposing the windows to high vacuum as well as UV borelight from the laser discharge. After several hours of operation, the intracavity power is observed to decrease monotonically (by approximately 15% per hour) accompanied by the development of a red fluorescence on the inside window surface where exposed to the visible laser radiation. Partial rejuvenation of the windows can be accomplished by reintroduction of gas into the vacuum chamber. Possible damage mechanisms will be presented.

1. INTRODUCTION

This paper presents observations of degradation in SiO₂ (fused silica and crystalline quartz) windows at the Atomic Oxygen Beam Facility (AOBF) at the University of Denver. This facility was constructed under a grant from the Air Force Office of Scientific Research to study the exposure of materials to oxygen ions and atoms in the energy range of 5-10 eV. The AOBF combines a versatile low energy ion source, an argon ion laser and quartz crystal microbalances to study a variety of processes involving the mass change of the materials exposed. Analyses of the modified surfaces are accomplished at the National Renewable Energy Laboratory (NREL).

2. EXPERIMENTAL DETAILS

The AOBF's primary task is to produce moderate fluences of oxygen atoms.¹ Atomic oxygen is produced, in vacuum, by photodetachment of electrons from O⁻ in a well characterized ion beam. This is accomplished by passing the O⁻ beam through the cavity of a Spectra-Physics 2040E Ar Ion laser (all lines 488-514 nm). Thus, the cavity of the laser is extended to include the vacuum chamber (Fig. 1). The vacuum chamber is sealed with fused silica (or crystalline quartz) windows mounted at Brewster's angle. By operating the laser with high reflection mirrors at both ends of the cavity, intracavity powers in excess of 3 kW have been obtained (power densities up to 170 kW/cm²).

In normal operation, the vacuum chamber is pumped with a liquid nitrogen-trapped, Varian VHS-6 diffusion pump using Santovac-5 diffusion pump oil. The foreline is ~ 4 m long, trapped and coiled and utilizes a dry nitrogen purge to prevent backstreaming of forepump fluid. Pressures in the 10^{-9} Torr range are routinely achieved. The methodology has been demonstrated to produce vacuum conditions with lower residual hydrocarbon partial pressures than do oil or grease-bearing turbo pumps.² XPS analyses of various samples (including the Brewster windows) show minimal hydrocarbon contamination (not inconsistent with exposure to air during sample transport).

3. DEGRADATION PHENOMENA

It is observed that the windows degrade under normal vacuum operating conditions. This degradation occurs over the period of hours, resulting in a decrease in the window's transmission. The degradation of the window's

performance is shown in Figure 2, for several samples of Corning 7940 Ultra-Violet Grade Synthetic Fused Silica (UVGSFS) with the laser operating with the standard 10% output coupler (low intracavity power).

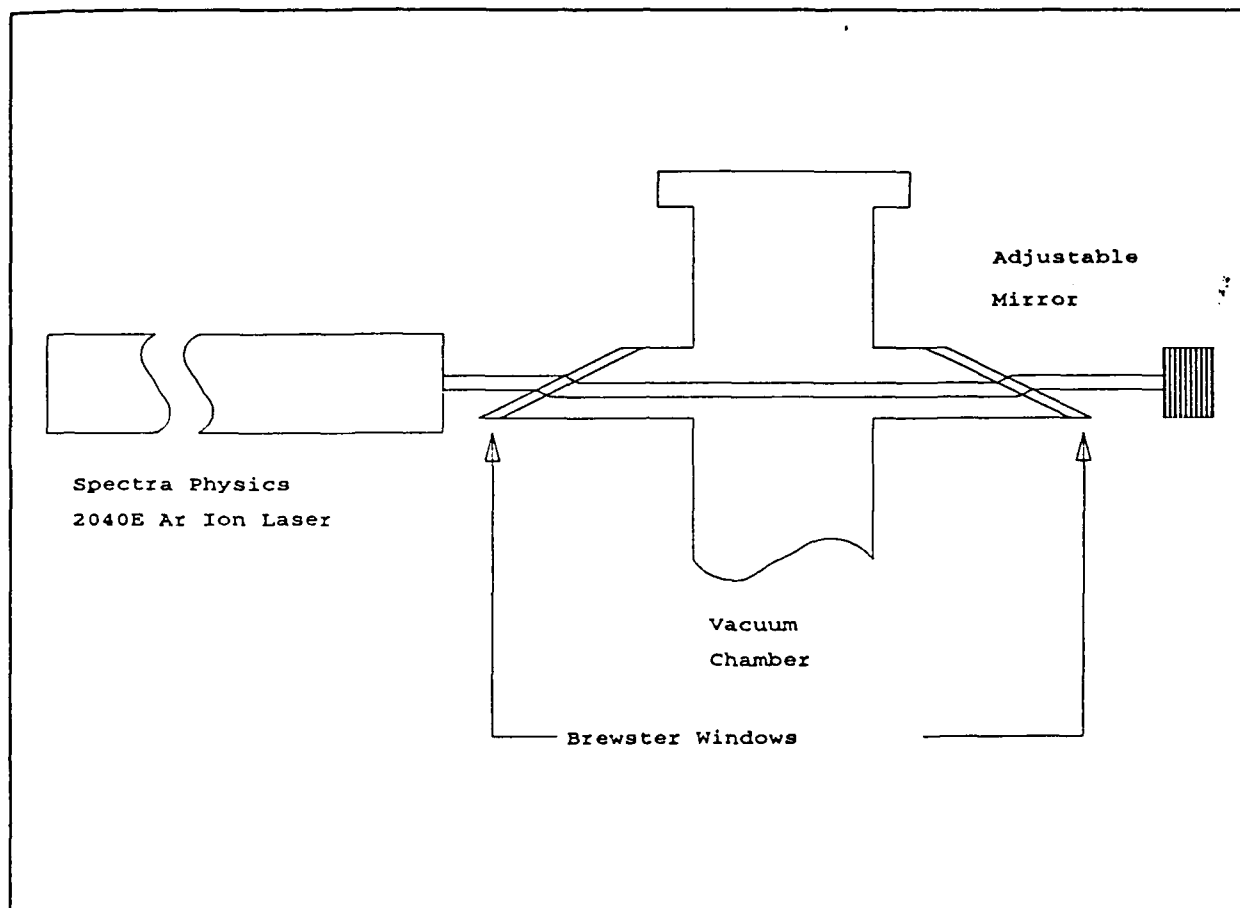


Figure 1. Schematic of the experimental apparatus showing the inclusion of the vacuum chamber within the cavity of the laser.

The degradation of fused silica windows has been observed in Dynasil 1103, Suprasil 1 and Infrasil, as well as the Corning 7940. Similar degradation has been observed in crystalline quartz windows, but the degradation proceeds more slowly (about one-third the rate) than for fused silica. Curves for a new window, a new window polished to remove subsurface damage and a new window exposed to vacuum for several hours prior to turning on the laser are presented.

The decrease in transmission of the windows is accompanied by an increase in light scattering from the vacuum exposed window surface and the onset and intensification of red fluorescence on the interior surface. Spectra of the fluorescence indicate a broad peak at 6200Å-7000Å.

3.1 Dependence on Vacuum

The degradation in the window's performance (Figure 2) as a function of time under vacuum ($P < 1$ Torr) is suggestive of two processes. In the first approximately two hours, there seems to be a "cleaning up" process resulting in high laser powers. This is followed by the degradation of the window resulting in the fall off of power as a function of time. The window that was exposed to vacuum for several hours before turning on the laser only

shows the fall off in power. This seems to indicate the windows are "cleaning up" due to the vacuum conditions above.

This degradation only occurs when the chamber is evacuated to less than 1 Torr. In fact, the laser power is observed to return to $\sim 80\%$ of its initial power when the vacuum chamber is backfilled to > 1 Torr with a variety of gases, including Ar and He. This rejuvenation does not, therefore, appear to be of a chemical origin.

3.2 Dependence on Ultraviolet Exposure

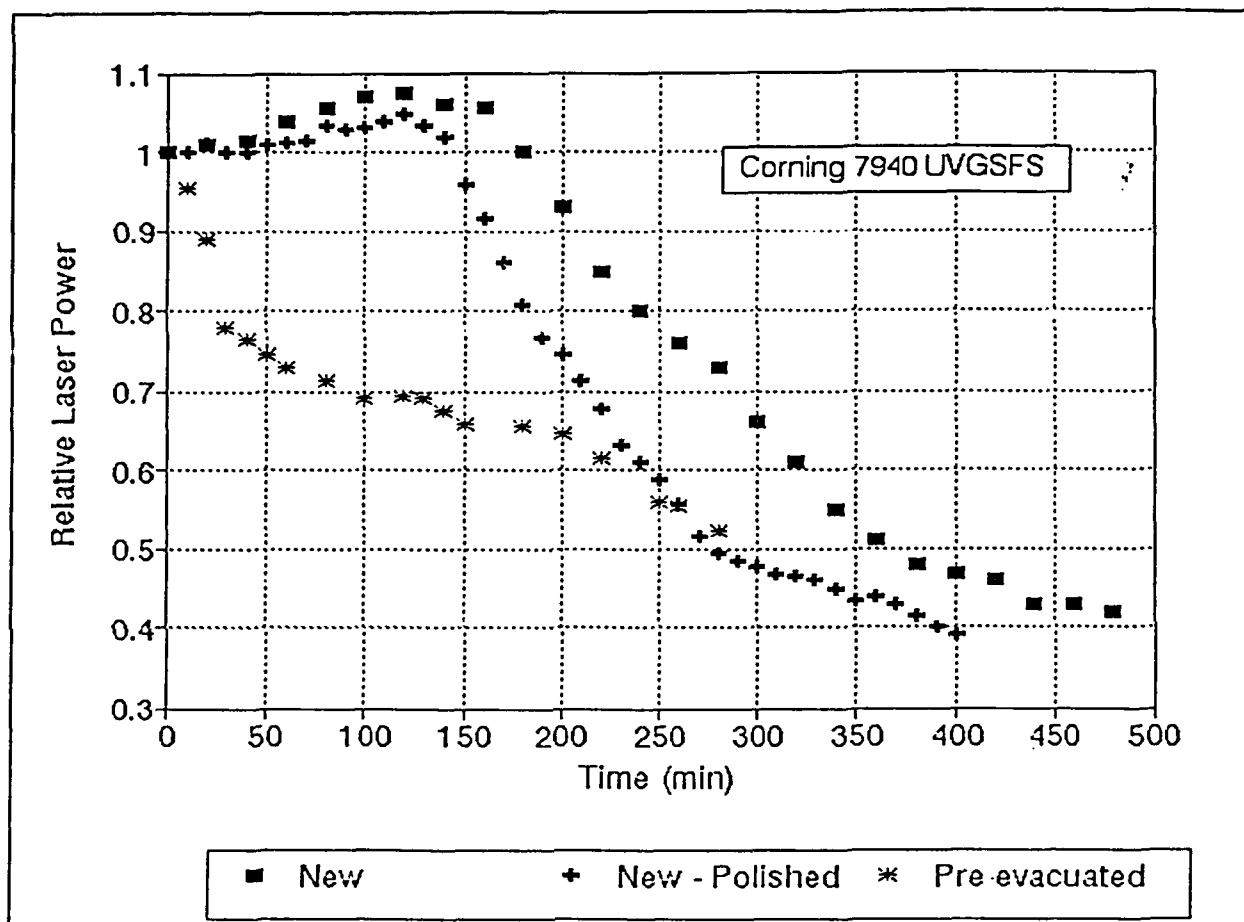


Figure 2. Relative laser power as a function of window exposure time to vacuum for three windows.

The degradation of the windows only occurs when the UV borelight of the laser ($\lambda < 3400\text{\AA}$) is incident on the windows. Corning 0211 glass was used as a UV filter in the cavity of the laser to block the SiO_2 windows from UV borelight ($\lambda < 3400\text{\AA}$), no degradation of the SiO_2 windows occurred under these conditions. This indicates that UV radiation with $\lambda < 3400\text{\AA}$ plays a role in the degradation of the windows. UVGSFS windows will transmit radiation down to wavelengths on the order of $1700\text{\AA}$. Infrared grade fused silica transmits down to $\sim 2000\text{\AA}$. This damage is observed in Infrasil, thus indicating that the UV light involved is $> 2000\text{\AA}$.

3.3 Dependence on Visible Laser Light Exposure

The damage to the windows only progresses as in Figure 2 if the visible lasing radiation is present. If the cavity is spoiled for a period of time (UV still incident on the windows and vacuum on) and lasing is then resumed, the loss curve picks up where it left off. Once the degradation occurs, it is observed that the fluorescence is associated

with illumination by the visible laser radiation. The fluorescence occurs with the UV borelight blocked by the Corning 0211 glass, but does not occur if only the UV borelight is incident on the window.

4. DISCUSSION OF POSSIBLE CAUSES

The degradation in the transmission of the SiO₂ windows has been a concern in our laboratory. Several possible causes for this degradation have been explored. Although no cause has been positively identified, some have been identified as not responsible for the phenomenon.

4.1 Stress Induced Birefringence

Stress induced birefringence might initially appear to be a likely candidate as the cause. Stress in the window could occur from either the pressure differential across the window or from compaction of the window surface. However, the pressure differential across the window is not significantly different at 1 Torr than at 10⁻⁷ Torr. It does not then seem likely that the pressure differential across the windows is responsible. The damage occurs over an extended period of time, which is not inconsistent with compaction. Damage due to compaction is not likely to rejuvenate. Stress induced damage does not appear to be the cause of this degradation phenomenon.

4.2 Subsurface Damage

The interaction of lasing radiation with subsurface damage cannot be neglected as a possible contributor to the phenomena discussed. Corning 7940 UVGSFS windows were polished to partially remove subsurface damage at Lawrence Livermore National Laboratory. The polished windows were then used in the laser cavity. The performance of these windows (Figure 2) is not significantly different from other windows. Although there has been a single test of this type of polished windows, subsurface damage does not appear to be responsible for the decrease in transmission of the windows.

4.3 OH Content

A wide variety of fused silica, fused quartz and crystalline quartz windows were tested. No correlation of the rate of degradation versus OH content has been observed.

4.4 Contamination

Contamination was and still remains a possible cause of the degradation. A number of tests were made to better clarify contamination issues. X-ray photoelectron spectroscopy (XPS) was used to analyze exposed and damaged windows. The XPS analysis of the windows did not show unusual hydrocarbon contamination. Nor were any other contaminants found. It was also determined that windows purposely contaminated degraded very rapidly and did not rejuvenate. It is not clear why there would be a pressure dependence involved with the degradation due to contamination. The level of contamination in our vacuum chamber should not be significantly different at 1 Torr than at lower pressures.

Perhaps the strongest evidence against contamination is the difference between the degradation of fused silica and quartz. Repeatedly, the degradation of quartz occurred on a time scale at least 3 times that for fused silica.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

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2. T.J. Gay, J.A. Brand, M.C. Fritts, J.E. Furst, M.A. Khakoo, M.T. Sieger, and W.M.K.P. Wijayaratna, in the Proceedings of the Seventeenth International Conference on the Physics of Electronic and Atomic Collisions, Edited by I.E. McCarthy, W.R. MacGillivray and M.C. Standage (Griffith University, Brisbane, Australia, 1991), p. 634.

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