

Measured limits to contamination of optical surfaces by elastomers in vacuum

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We have monitored the reflectivity of mirrors that were exposed to a fluoroelastomer (3M-Fluorel 2176) and a room-temperature vulcanizing silicone rubber (RTV-615) in vacuum. The 95% confidence limit on the decrease of mirror reflectivities was less than 0.35 ppm/week for Fluorel and <0.29 ppm/week for RTV-615.

Key words: Optical contamination, elastomers, vacuum outgassing, optical losses.

The contamination of optical surfaces in vacuum is an important issue in many critical optics applications. Elastomers are often desirable for inclusion in vacuum systems either as seals or for their mechanical properties. For example, gravitational-wave detectors¹ may use vibration isolation systems made with elastomers² in vacuum. Here we address the concern that the Fluorel and RTV-615 springs used in such isolation systems may contaminate mirrors in vacuum. Previous research in this area has focused on measuring outgassing and deposition kinetics, primarily for certifying materials as acceptable for satellite use.³ Predicting the change in optical properties from the amount of surface contaminant is difficult.

We have attempted to avoid these difficulties by measuring optical degradation directly, at room temperature, and at pressures and time scales relevant for laboratory optical experiments. We have tested sample elastomers by placing a specimen in a cleaned and baked vacuum chamber with a high-finesse Fabry-Perot cavity. Except for the specimen, only metal and glass were used inside the vacuum chamber. The chamber was then evacuated, and we measured the losses in the Fabry-Perot cavity periodically by monitoring the storage time for light injected into the cavity.⁴ Changes in the absorption and scattering of the mirrors were inferred from the measured changes in the storage time.

We used three test cavities, each in a separate vacuum chamber. One was used as a control with no specimen in the chamber, one was exposed to RTV-615 silicone rubber, and one was exposed to Fluorel. Over several months we noticed little change in the mirror losses, indicating that these materials may be used in vacuum systems with low-loss mirrors.

The optical cavities consisted of two 110-ppm (nominal) transmission mirrors. The input mirror was flat, whereas the other had a 50-cm radius of curvature; both mirrors were 2.5 cm in diameter. They were held 29.1 cm apart by an aluminum spacer, which was suspended with 150- μ m-diameter wires for vibration isolation. The entire spacer assembly was constructed of aluminum except for the steel wires, stainless-steel screws and balls on which the mirrors rested, and beryllium-copper springs. The spacer had a slot where a specimen could be placed close (≤ 5 cm) to the input mirror.

Each vacuum chamber was a 20-cm-diameter, 66-cm-long tube constructed of stainless steel with Kovar-sealed Pyrex windows at both ends. Before installing the mirrors, the beryllium-copper springs, and the specimen, we vacuum baked the vacuum chamber and all other components at 190 °C for 24 h. The beryllium-copper springs were unavailable at the time of the initial bake and instead were baked in air at 200 °C for 48 h. The vacuum chamber was evacuated through a turbomolecular pump (to reduce potential backstreaming of pump oil from the rotary vane roughing pump). An 8-L/s ion pump attached to the chamber was started when the chamber pressure dropped to less than 10^{-5} Torr. After the initial surge of ion-pump current, an all-metal valve was used to isolate the chamber from the roughing pump and the mechanical pumps were removed. In this way pressures of less than 10^{-8} Torr were achieved

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and maintained, except for the chamber containing Fluorel, which achieved only 2×10^{-7} Torr because of the high outgassing load from the specimen.

The specimens were prepared and handled carefully before being inserted into the test chambers. The fluoroelastomer specimen was a single piece of 3M-Fluorel 2176 (70 durometer) molded into a truncated cone, 5 cm high, 4 cm in diameter at the base, and 3 cm in diameter at the top. It was cured by the manufacturer for 25 min at 180 °C and was not postcured. (Postcuring is a process that is used to remove some volatile materials from Fluorel after it is cured. It is normally used with O-rings, for example.) After we received the part from the manufacturer, we baked it in vacuum for 46 h at 110 °C.

The RTV specimen consisted of six pieces of RTV-615 silicone rubber. Each was a cylinder 1 cm in diameter and 1 cm high. After casting, we soaked them in methylene chloride for 5 days with the solvent changed twice. The RTV pieces were then baked in vacuum for 90 h at 200 °C.

A diagram of the experimental apparatus is shown in Fig. 1. An argon-ion laser was used as the light source for the mirror loss measurements. Feedback to a Pockel's cell inside the laser cavity locked the laser frequency to the test cavity modes for times of the order of 0.1 ms. The feedback loop derived its error signal from the light reflected from the cavity with the rf reflection-locking technique.⁵ When the light transmitted through the cavity reached a preset trigger level, an acousto-optic modulator was used to shut off the light incident on the cavity. At the same time a storage scope began recording the output of a photodiode monitoring the light that leaks out of the cavity. Typically 32 such decay transients were averaged together. The data were then fit to an exponen-

tial of the form

$$V = A + B \exp(-t/\tau_e). \quad (1)$$

The resulting τ_e is the storage time given by

$$\tau_e \approx \frac{2l}{cL_{\text{cav}}}, \quad (2)$$

where l is the length of the cavity, c is the speed of light, and L_{cav} is the (small) cavity round-trip loss including absorption and scattering as well as transmission. The light trigger level was set so that only the storage time of the TEM₀₀ mode was measured. Ten such measurements (each representing the average of 32 decay transients) were made and averaged to provide an estimate of the measurement uncertainty.

After the experiments described here were performed, we developed a better method of cleaning Fluorel that significantly decreased its outgassing. After being cured, it was postcured by being baked in air at a temperature that is ramped from 80 to 230 °C over a period of 46 h. The slow ramp was necessary to avoid cracking the Fluorel piece. It was then baked at 230 °C for 14 h. After cooling, the Fluorel piece was vacuum baked for 10 days at 200 °C.

A plot of total cavity loss versus time for the control cavity and the two materials tested so far is shown in Fig. 2. The large differences in the initial losses arose from spatial nonuniformity of the mirrors. We repeated the measurements often for the first 48 h to look for rapid degradation. In all three cases the losses measured immediately before pumping down were ~10 ppm higher than losses measured after 1 h of pumping. This decrease is consistent with the expected decrease in Rayleigh scattering from the air in the chamber.

The results of linear fits to the data are shown in Table 1. We do not necessarily expect a contamination effect to be linear, but a linear fit is a convenient way of quantifying trends in the data for comparisons

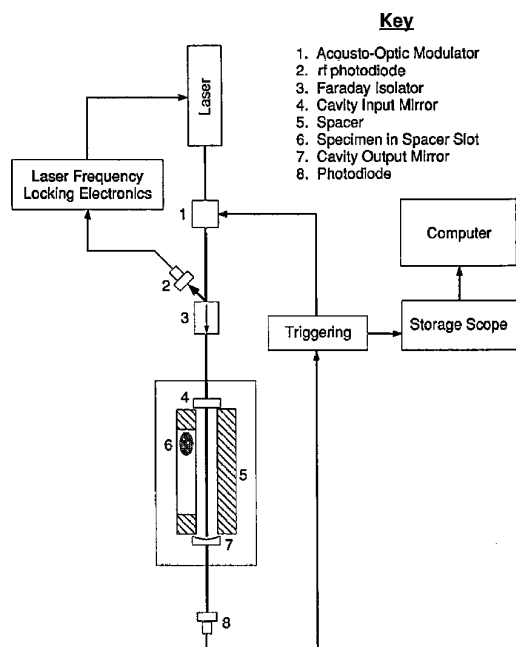


Fig. 1. Schematic diagram of the experimental apparatus.

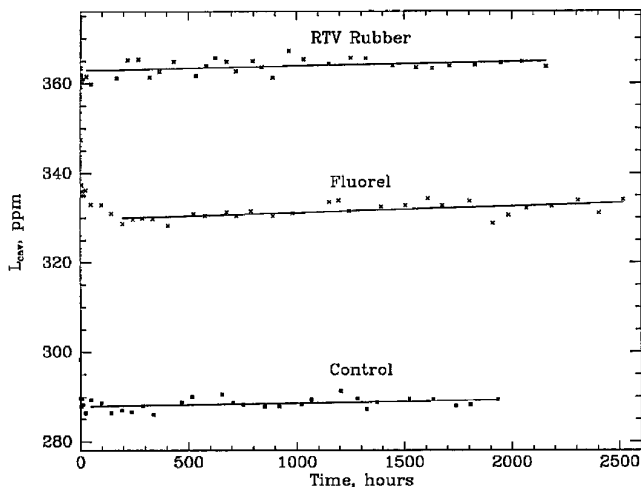


Fig. 2. Total cavity loss versus time for three test chambers with straight-line fits. The lengths of the lines indicate the regions over which the fits were taken.

Table 1. Results of Optical Tests

Specimen	Rate of Increase of Losses (ppm/week)	
	Best Fit	95% Confidence Limit
Control	0.13 ± 0.07	<0.24
Fluorel	0.25 ± 0.06	<0.35
RTV	0.16 ± 0.08	<0.29

between the three cavities. Since fluctuations in the data typically exhibited time scales of several days, only one point was retained for the fit from the initial 48-h period in the cases of the control cavity and RTV test cavity. The Fluorel test cavity showed a decrease in losses for the first 200 h. There are several mechanisms that could explain such an effect. For example, the mirror coating could have absorbed water or a contaminant film could have formed on the mirror before evacuation of the chamber, which then desorbed in vacuum. Whatever the explanation, it was decided that the most conservative estimate of mirror degradation caused by exposure to Fluorel would be obtained by starting the linear fit after the initial downward trend at 200 h.

In general our short-term measurement uncertainty for τ_e was much smaller than the observed day-to-day fluctuations in this parameter. We suspect that the larger fluctuations resulted from movement of the spot positions on the mirrors. A small temperature change in the aluminum spacer could cause it to bend slightly in addition to changing its length. Since the waist (radius) was 0.2 mm, a small spot motion would significantly change the total losses if the mirror losses were not uniform. In fact mirrors tested from this coating batch typically showed factor-of-2 variations in losses with position across the surface of the mirror. Losses measured successively in a cavity on the TEM₀₀ and TEM₀₁ modes were observed to differ by 25%, indicating that the length scale for these loss variations was comparable with the spot size. We expect that, with the use of better mirrors in the future, our measurements will become more accurate.

For comparison with the optical tests, the outgassing properties of identically prepared specimens were characterized by a residual gas analyzer (RGA). The measurements were carried out in the same chamber in which the specimens were baked so that the specimens were not exposed to air between their baking and our taking the spectra. We calibrated the outgassing rates by opening a calibrated N₂ leak into the test chamber. (The calibrated leak itself was checked against another calibrated leak.) Table 2 shows the outgassing rates at various mass numbers. These outgassing rates indicate the successful removal of volatile hydrocarbon residues by the cleaning and baking procedure. Note, however, that total outgassing rates on elastomers prepared in this way and then exposed to air before installation in a

Table 2. Results of Residual Gas Analyses

Mass	Effective Outgassing Rates (Torr L/s cm ²)		
	Fluorel (No Postcure)	Fluorel (Postcured)	RTV-615
18	1.8×10^{-9}	8.8×10^{-14}	$<7.5 \times 10^{-14}$
28	3×10^{-10}	1.9×10^{-12}	$<6.9 \times 10^{-13}$
44	8×10^{-12}	1.6×10^{-12}	$<9 \times 10^{-14}$
All others	$<6.8 \times 10^{-11}$	$<7.3 \times 10^{-14}$	$<3.8 \times 10^{-15}$
RGA electronic noise level	2.3×10^{-15}	1.4×10^{-16}	1.9×10^{-16}

vacuum system are expected to be higher than the values in Table 2. During exposure to air, gases are reabsorbed into the elastomers. The pressures in test chambers that contained elastomers were observed to decrease more slowly than in the control chamber because of this subsequent outgassing. This effect was far more prominent in the Fluorel sample than in the RTV sample, which we attribute to the lower permeation rate in the Fluorel specimen and the larger size of that specimen.

Within measurement errors the cavities exposed to Fluorel or RTV did not show a significantly higher rate of mirror loss increase than the control cavity. These materials should be acceptable for use in many vacuum applications with low-loss mirrors at room temperature. Although the best linear fit for the control cavity shows a slight loss increase, we feel that it is premature to speculate on its cause given our measurement accuracy. In ongoing experiments we are attempting to improve the limits set for Fluorel and RTV and test other materials.

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