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Use of Silica Gel as a Getter for the Protection of Sol-gel Coated Optics: Concept Verification

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October 2, 2014

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This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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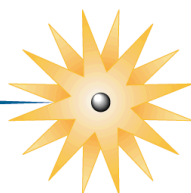
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Interdepartmental Letterhead

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December 21, 2000
NIF0075459/adf

To: File

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Subject: **Use of Silica Gel as a Getter for the Protection of Sol-gel Coated Optics: Concept Verification**

Background and Introduction

Because of their high damage thresholds, sol-gel coatings will be used as an antireflective (AR) coating on a substantial portion of the NIF optical train. Unfortunately, because of their high porosity such coatings can adsorb significant quantities of volatile or semi-volatile organic compounds. If a sufficient quantity of such compounds, which are often called airborne molecular contaminants (AMCs), are adsorbed by the coating the resulting change in the refractive index at the surface of the optic can be large enough to destroy the AR properties of the coating. Because the rate at which AMCs are transported varies inversely with pressure, this issue is particularly problematic in those portions of the beamline, such as the spatial filter, that are evacuated.

Recently[1] we estimated the loss of AR performance that would result if a sol-gel coated optic was placed in an environment containing stainless steel or aluminum surfaces that had been cleaned to A/10 (0.1 ug/cm^2) and A/3 (0.33 ug/cm^2). These values correspond to the NIF cleanliness goals for freshly cleaned surfaces (A/10) and for surfaces at the time of use (A/3). This preliminary analysis was based on a limited set of experimental measurements of the partitioning of a model AMC (dibutylphthalate) between the vapor and adsorbed phases on stainless steel, aluminum, and sol-gel coated quartz. This analysis showed that surfaces installed at A/10 probably pose little threat to AR coatings, while surfaces at a cleanliness level of A/3 may pose a significant threat to the AR properties of sol-gel coated optics.

Because of remaining uncertainties both with respect to the actual sensitivity of sol-gel coated optics to AMC contamination and uncertainties associated with respect the levels of cleanliness that can be achieved in practice, a supplementary method, using getters,

has been proposed[2] for protecting sol-gel coated optics from contamination by AMCs. The present memo describes experimental validation of this method of optical protection.

Experimental

The experiment is schematically represented in Fig. 1.   A set of three (two hardened, one unhardened) 5 cm diameter, 3ω AR sol-gel coated optics are placed in close proximity to a vapor source which consists of a pool of model AMC. The optical transmission of each optic is measured prior to exposure to the AMC, immediately following exposure to the AMC (in the absence of a getter bed), and immediately following several hours of exposure to both the AMC and a granular getter bed.
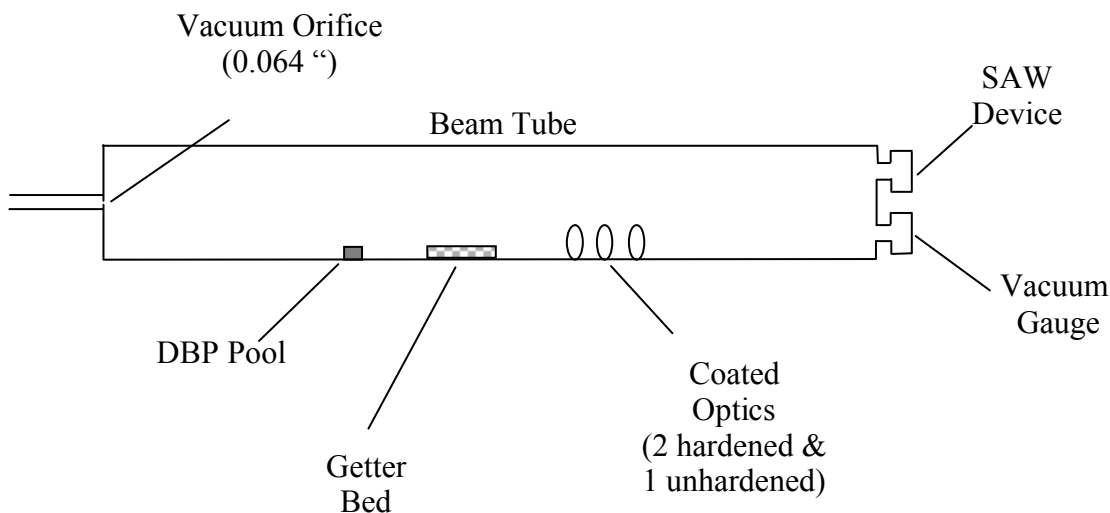

Figure 1: Schematic representation of getter demonstration. The initial phase of the experiment was run without the getter in place (see text).

The getter bed used during the latter portion of the experiment consisted of 55 grams of grade 03, mesh 8 silica gel (Aldrich, St. Louis Mo.). The stated surface area of this material is 660 m^2/gram.   The silica gel was arranged in a 9.5 cm diameter dish, which resulted in a bed depth of approximately 1.5 cm. This corresponds to a bed roughly 4-5 particle diameters deep.

Dibutylphthalate (DBP) was used as a model AMC for the present experiments. DBP and its less volatile homologue dioctalphthalate (DOP) are very common AMCs owing to their extensive use as plastiziers. DBP was also chosen because of its convenient transport properties. In moderate (millitorr) vacuum environments, DBP easily diffuses over distances typical of the present experiment in a few hours. At the same time, at ambient pressure the amount of DBP lost from the surface of the optic is minimal over the time required to measure its optical transmission. The DBP vapor source consisted of a small (2 cm diameter) pool of DBP, which was masked such that the exposed area of the pool was 0.54 cm^2

The experimental chamber (see Fig. 1) consisted of a 25.4 cm diameter x 122 cm long piece of stainless steel beam tube to which vacuum flanges (Conflat, Varian Vacuum Products, Lexinton MA) had been welded.   The chamber was evacuated, through a limiting orifice of 0.064", by means of a small (70 liter/min) turbomolecular pump, which in turn was backed by a dry mechanical pump (Triscroll, Varian Vacuum Products,

Lexington, MA). The pressure within the chamber is monitored using a convectron (Granville-Phillips, Boulder CO.) vacuum gauge.

The DBP vapor saturation within the chamber is monitored using a sol-gel coated surface acoustic wave (SAW) sensor. This sensor was prepared by coating a 1 cm^2 quartz crystal (TPL Albuquerque, NM) with a nominal 2ω sol-gel AR coating which was subsequently ammonia hardened and treated with hexamethyldisilasane (HMDS) vapor. The thickness and porosity of the coating as determined by ellipsometry was 214 nm and 60% respectively. Based on these values, a pore filling of unity would correspond to a surface loading of 10.7 ug/cm^2.

As the vapor phase immediately above the SAW becomes increasingly saturated with DBP, the mass loading on the SAW increases, in accordance to the applicable isotherm. The additional mass loading on the surface of the sensor, in turn, causes a reduction in the frequency of the SAW. The mass loading, x (ug/cm^2) can be related to the change (in Mhz) in the SAW frequency (f-f$_0$) using the expression:

$$x = .7759 \times 10^6 \, (f\text{-}f_0) \, / f_0^2$$

Both the output of the vacuum gauge and the frequency of the SAW were digitally recorded, at prescribed intervals, using a PC application written in Labview (National Instruments, Austin Texas).

Prior to, and immediately ($\approx$10 minutes) following DBP exposure (in the presence and absence of the getter), the optical transmission of each optic was measured over a wavelength of 200-900 nm using a Shimadzu model UV2100U recording spectrophotometer.

Results and Discussion

The temporal response of the SAW, showing the mass loading on the transducer, throughout the duration of the experiment is shown in Fig. 2. During the initial 91 hours of the experiment (the blue trace in Fig. 2) only the vapor source and the three optics were present in the chamber; the getter bed was not present. As shown in Fig. 2, the amount of DBP adsorbed on the SAW, and presumably each of the optics, gradually increases as the DBP diffuses from the pool throughout the chamber.

Initially (during the first $\approx$25 hours) the chamber was continually pumped through the 0.064" orifice. After a chamber reached a pressure of approximately 1 millitorr, the system was valved off and allowed to sit under a static vacuum. Periodically, between 25 and 91 hours, the isolation valve was opened to offset the effects of virtual and real leaks in the chamber. This resulted in the irregular rate of transport, which is evident in the SAW response shown in Fig. 2.

After 91 hours of exposure to the DBP vapor, the chamber was brought to ambient pressure, each of the optics was removed and the transmission of each optic was recorded. Each optic, together with its adsorbed DBP, was then again placed in the chamber along with the DBP pool and the getter bed.
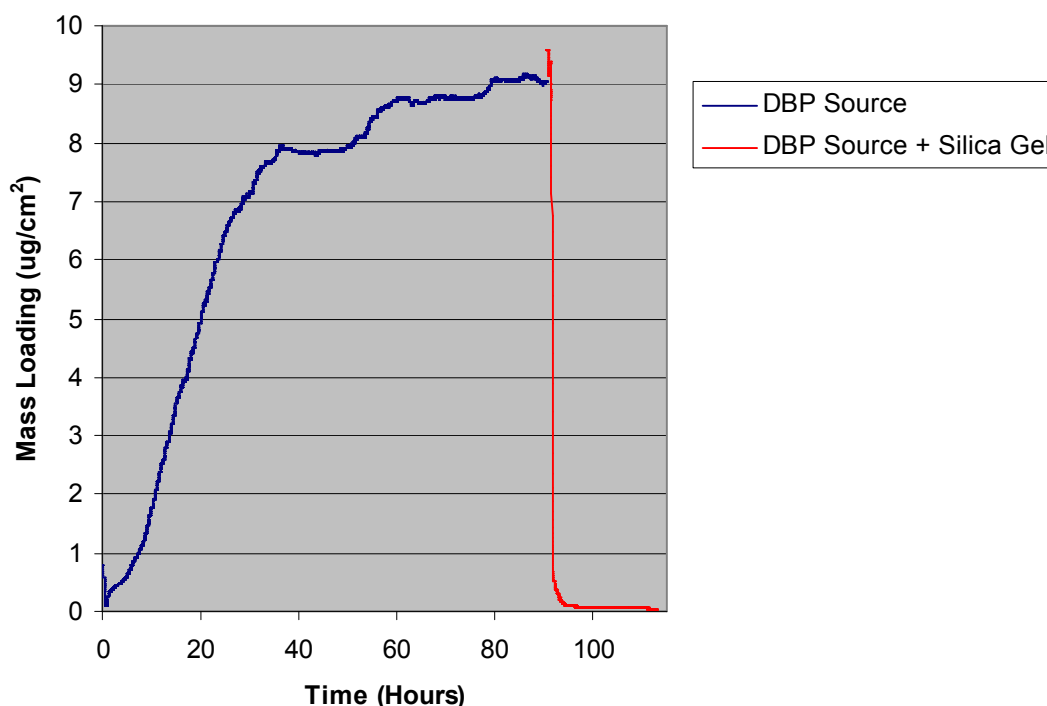
Figure 2: SAW response during the duration of the experiment. The frequency change (f-f$_0$) of the SAW has been scaled to reflect the mass loading on the transducer by use of equation 1.

The response of the SAW following evacuation of the chamber is shown as the red trace in Fig. 2. The reader will note that the mass loading on the SAW immediately after the optics are reintroduced into the chamber is somewhat higher than the mass loading immediately prior to when the chamber was vented. This is due to the adsorption of a few monolayers of water and/or nitrogen onto the surface of the SAW. More importantly, the effectiveness of the getter at reducing the fractional vapor saturation of the DBP in the chamber is immediately evident by noting the precipitous drop in the quantity of DBP adsorbed on the SAW transducer (red trace) shown in Fig. 2. After 22 hours of exposing the optics to DBP, in the presence of the getter bead, the chamber was vented and the optical transmission of each optic was again recorded.

A comparison of the optical transmission of a typical ammonia hardened optic prior to and following exposure to DBP, both in the absence and the presence of the getter bed, is shown in Fig. 3. Similar results were also observed for the unhardened sol-gel coated optic. With respect to the time scale shown in Fig. 2, these spectra were taken at 0, 91, and 122 hours respectively.

By comparing the blue and red spectra in Fig. 3, one observes the deleterious effects that AMC adsorption can have on the transmission of a sol-gel AR coated optic. Similarly, one can also observe that introduction of the silica-gel bed (green spectra) provides a means of effectively removing the adsorbed DBP from the sol-gel and restoring the AR properties of the coating.
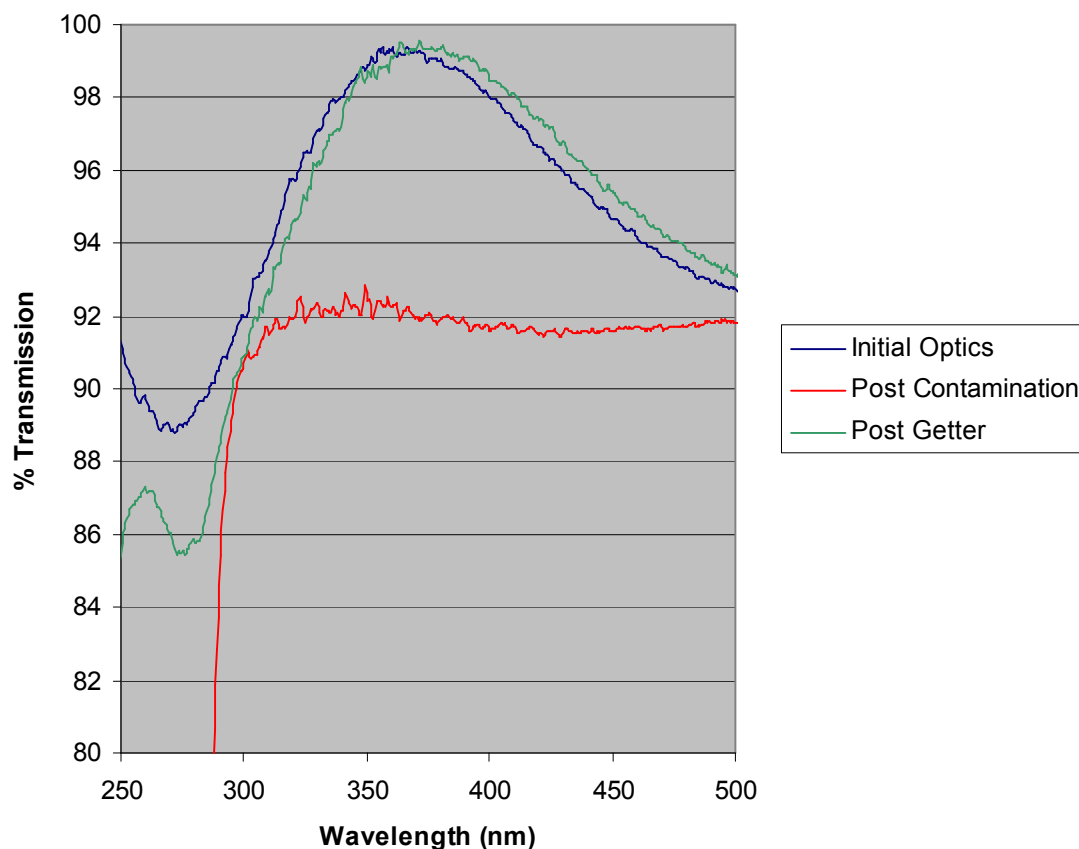
Figure 3: Optical transmission spectra of ammonia hardened sol-gel coated optic prior to, and following DBP exposure both in the presence and absence of silica gel getter bed.

Conclusions and Future Work

The present data demonstrate that getters, such as silica gel, can be effectively used to prevent the loss of AR properties that result when AMCs adsorb onto sol-gel coated optics. Similarly, these data also demonstrate the ease with which one can monitor the vapor saturation using a SAW sensor.

At equilibrium a silia-gel getter bed can adsorb 15-20% of its weight in AMCs. In practice, the capacity or size of the bed that will be required is a function of the rate at which AMCs are transported to and adsorbed by the getter itself. Measurements of the rate at which typical AMCs are adsorbed by particles of silica-gel and activated carbon have been reported elsewhere[3]. Similarly, the performance of NIF sized beds, based upon experimental rates of single particle adsorption, have been modeled[4].

The largest potential impact that the use of getters might have is in the area of vacuum compatibility. In particular, a variety of handling or pretreatment strategies need to be experimentally evaluated that will minimize any potential burden on the vacuum system used to evacuate the spatial filters.

[1] John Ertel, Dan Behne, Keith Kishiyama, Phil Miller, and Chuck Thorsness, *Dibutlylphthalate Isotherm for the Vacuum Test Chamber Walls* NIF0065255, May 21, 2001

[2] C.B. Throsness *NVR Contamination and the NIF Filter Beam Tubes –1st Order Analysis of the Effectiveness of an Active Getter Surface* NIF0064572 May 7, 2001

[3] P.E. Miller amd C.B. *Thorsness AMC Adsortion onto Candidate Getter Bed Materials –Preliminary Data and Analysis* NIF0072277 October 11, 2001.

[4] C.B Thorsness and P.E. Miller *Update of NVR/Getter Bed Calculations for the NIF Spatial Filter* NIF0072986 October 31, 2001

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