REPORT ON CHEMICAL ANALYSES OF PROVIDED SAMPLES

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SRI Project 3557
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Prepared for:

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A batch of four samples were received and chemical analysis was performed of the surface and near surface regions of the samples by the surface analysis by laser ionization (SALI) method. The samples included four one-inch diameter optics labeled windows # PR14 and PR17 and MgF2 mirrors 9-93 PPC exp. and control DMES 26-92. The analyses emphasized surface contamination or modification. In these studies, pulsed desorption by 355 nm laser light and single-photon ionization (SPI) above the sample by coherent 118 nm radiation (at ~5 x 105 W/cm2) were used, emphasizing organic analysis.

For the two windows with an apparent yellowish contaminant film, higher desorption laser power was needed to provide substantial signals, indicating a less volatile contamination than for the two mirrors. Window PR14 and the 9-93 mirror showed more hydrocarbon components than the other two samples. The mass spectra, which show considerable complexity, are discussed in terms of various potential chemical assignments.
A batch of four samples was received consisting of one inch diameter optics labeled windows # PR14 and PR17, and MgF2 mirrors 9-93 PPC exp. and control DMES 26-92. Chemical analyses of the surfaces were performed by the surface analysis by laser ionization (SALI) method. The analyses emphasize surface contamination. SALI uses nonselective photoionization of sputtered or desorbed atoms and molecules above but close (-1 mm) to the surface, followed by time-of-flight (TOF) mass spectrometry. In these studies, laser-induced desorption by 5 ns pulse-width 355 nm light (10-100 mJ/cm²) and single-photon ionization (SPI) by coherent 118 nm radiation (at \(-5 \times 10^5\) W/cm²) were used. SPI was chosen primarily for its ability to obtain molecular information, whereas multiphoton ionization (not used in the present studies) is intended primarily for elemental and small molecule information. The choice of laser desorption was made after initial studies with pulsed Ar⁺ sputtering; it was apparent that considerably more dynamic range and higher mass range were available with laser desorption; also charging of the optics was occurring with the pulsed Ar⁺ beam.

While SPI by 118 nm (10.5 eV) light is considered a generally "soft" (nonfragmenting) form of radiation, stimulated desorption can cause fragmentation and also produce internally hot molecules which photofragment relatively easily compared to lower temperature sources of molecules. Typically, the low mass regions of the mass spectra contain a good deal of molecular fragment information.

Some other comments on the mass spectra are appropriate. The signals from the microchannel plate detector were recorded in analog fashion by a 100 MHz transient digitizer; thus the voltage signals are given as "relative intensity" and not as ion counts. For the two windows with apparent yellowish contaminant films (PR14 and 17), higher desorption laser intensities were needed to provide substantial signals, indicating a less volatile contamination than for the two mirrors (or possibly poorer absorptivity of 355 nm light). For sample PR17, and a little bit for the control mirror (DMES 26-92), direct ion signals (intense and broad mass peaks on a different mass scale--different time zero) are seen in the low mass region.

Some of the mass assignments have been marked on the figures, especially for the major peaks. Considerable effort can be invoked for mass interpretation. Some possible brief general interpretations now follow. Window PR14 and the 9-93 MgF2 mirror show considerably more hydrocarbon fragments at low masses and thus likely contain much more hydrocarbon components than the other two samples.
For PR17 the m/z 105 and 207 peaks can be associated with Si and O (Si2O3H for 105 and Si3O3(CH3)5 for 207), although there are other interpretations such as C6H5-CO for m/z 105. Also it would be unusual to observe a strong 207 and not a strong 73 or 147 for a silicone. Polydimethylsilicone was run as a standard for comparison under these conditions; strong peaks at m/z 73, 147, 207, 221, and 281 were observed.

For the two MgF2 mirrors, there are numerous major mass peaks in common such as 43, 57, 97, 112, 149, 167, 185 and 221. Note that 221 can possibly be assigned to a silicone, but the other peaks are not commonly associated with silicones except m/z 129 in the 9-93 mirror which can be rationalized as a Si2C5H13 structure. Masses 97, 112, 149, 167, and 185 can be assigned to CxFyOz structures. As to the higher mass assignments for the control mirror, it is not clear (especially with no detailed knowledge of sample history) what structures are associated with these masses; however, these peaks will be very characteristic of whatever compound(s) is present.
PR14 WINDOW, 355 + 118 NM, AP1404
PR17 WINDOW, 355 + 118 NM, AP1410

RELATIVE INTENSITY

5000 4450 3900 3350

m/z

120 180 240 300
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RELATIVE INTENSITY

PR14 WINDOW, 355 + 118 NM, AP1404
RELATIVE INTENSITY

PRI7 WINDOW, 355 + 118 NM, AP1410
MgF2 MIRROR 9-93 PPPC exp., 365+118 NM, API1414
RELATIVE INTENSITY

MgF2 MIRROR 9-93 PPPC exp., 355+118 NM, AP1414
RELATIVE INTENSITY

MgF₂, MIRROR CONTROL, DMES 26-92, 355+118 NM, AP1417
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