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Effects of Evactron® Plasma Cleaning on X-ray Detector Windows

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With the emphasis on higher spatial resolution imaging and microanalysis and low voltage operation in the scanning electron microscope, in situ cleaning of specimens and the SEM chamber must be considered a critical capability for a research grade instrument. The Evactron® Anti-Contaminator has been demonstrated to be effective in removing labile hydrocarbon contamination from both SEM chambers [1] and specimens. In order to address concerns over possible detrimental effects of Evactron plasma cleaning by neutral oxygen atoms (i.e., oxygen radicals), a previous study examined the long-term exposure of MOXTEK™ AP3.3Ultra Thin EDS windows [2]. Although the windows remained vacuum tight, light leaks in the form of pinholes in the Al light barrier coating developed during the plasma exposure. Light leakage may be a significant problem for a Si(Li) detector since the detector is light-sensitive. However, light and infrared radiation leakage may not be as serious for a microcalorimeter detector, as the detecting element is not inherently light sensitive and there are a number of additional thermal shields internal to the detector. The current study examined the windows from the previous study in order to judge the rate of pinhole formation and to understand the origin(s) of the pinholes.

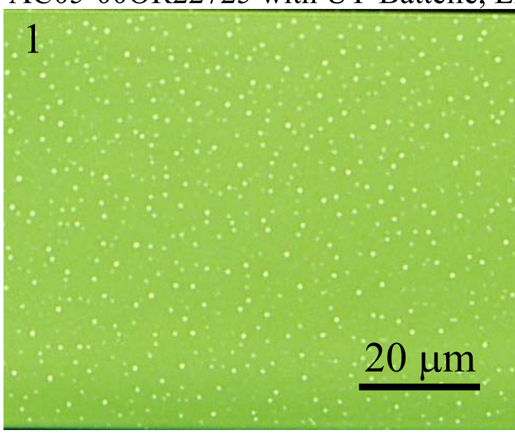
Figure 1 is a transmitted light image of a window exposed to the Evactron plasma for 30 hours. The light transmitted through the window is actually green, presumably the result of absorption by one of the window's layers. Unexposed windows do not exhibit the localized light leaks apparent in Fig. 1. Although there are a number of pinholes present, there was no significant increase in either their size or number density with additional exposures of 40, 30, 27 and 33 hours for a total exposure of 160 hours. It has been speculated that oxygen radical corrosion of the grain boundaries of the Al film is the origin of this degradation. Unfortunately, this window was damaged in shipment and microstructural examination was not possible. A second window was exposed for 160 hours in multiple exposures of about 20 hours each and a single, larger (~80 μm) pinhole formed. SEM examination of the pinhole revealed an impurity particle containing Si, Cl and K embedded in the window at the pinhole, indicating that this pinhole had resulted from particle impact. A third window was exposed to plasma cleaning for 60, 24, 62 and 14 hours. Figure 2 is a black-and-white transmitted light image of that window after a total of 160 hours exposure. Unfortunately, that window was contaminated with foreign particles between the plasma cleanings and the SEM examination. There is a difference in the number and size of the pinholes for these three windows (e.g., Figs. 1 and 2). However, it is not clear from the limited data whether those differences arise from differences in plasma cleaning or in the condition of the windows before exposure.

Imaging the pinholes in the SEM proved problematic: the thin (~30 nm) Al layer on the much thicker polymer film has a significant external oxide layer. Localized differences in the depth of oxidation do not provide significant secondary electron (SE) contrast. It was hoped that backscattered electron (BSE) imaging at low voltages (1-2 kV) would provide adequate contrast. However, as a result of the low probe currents (~0.3 nA) used to minimize damage to the window, the efficiency of the BSE detector at those low voltages and currents limited useable contrast. Although the contrast mechanism is not clear, it was found that secondary electron imaging at 2-3 kV and maximum contrast provided useable images (Figure 3). The pinhole was located by comparisons of both transmitted and reflected light images with the SEM images. The pinhole is significantly larger (~15 μm) than the grain size of the aluminum layer, which was estimated as ~30 nm from the granular structure of the Al surface at high magnifications.

Low voltage EDS microanalysis of the pinhole and the surrounding film showed increased oxygen relative to aluminum in the pinhole. The relative difference varied from pinhole to pinhole. Spectra that show a significant increase are given in Figure 4. The O/Al intensity ratio for the pinhole was 1.31 at 3 kV, whereas the ratio for the film was 0.85. For comparison, the intensity ratio for an aluminum specimen with a native oxide was 0.254 at 3 kV, whereas the ratio for pure Al_2O_3 was 2.69. There is a significantly thicker oxide layer on the window relative to the Al specimen and the oxide is thicker in the pinhole [3]. Auger elemental mapping and depth profiling of a pinhole have been used to further characterize the three-dimensional elemental distribution.

References

[1] N. Sullivan et al, Microsc. Microanal. 8 (Suppl. 2) (2002) 720.
[2] R. Vane et.al., Microsc. Microanal. 10 (Suppl. 2) (2004) 966.
[3] Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering (EAK), U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.



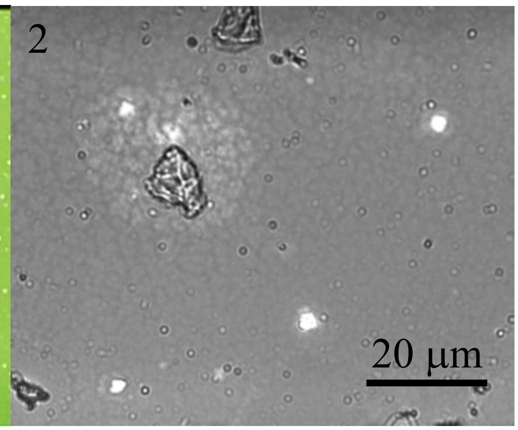
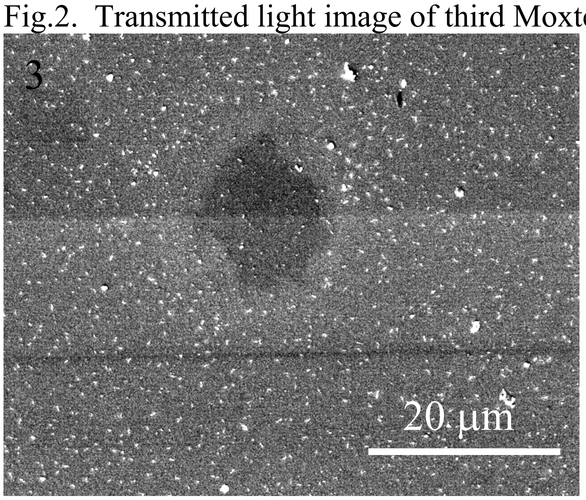


Fig.1. Transmitted light image of first Moxtek AP3.3 window exposed to plasma for 30 hours.
Fig.2. Transmitted light image of third Moxtek AP3.3 window exposed to plasma for 160 hours.



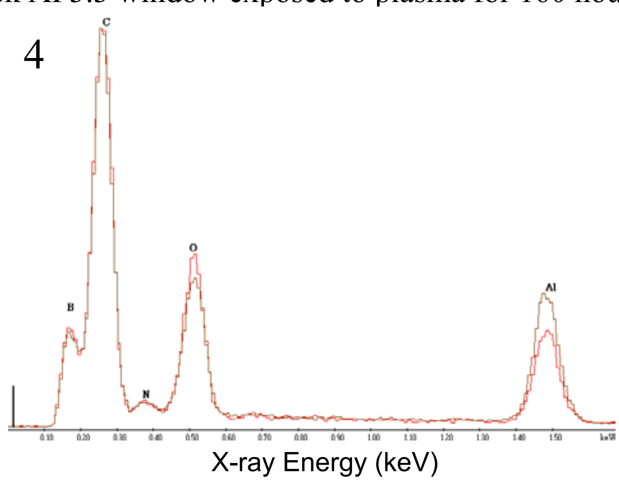


Fig.3. SE image of pinhole in third Moxtek AP3.3 window exposed to plasma for 160 hours.
Fig.4. Comparison of EDS spectra from pinhole (red) and adjacent film (brown) acquired at 3 kV in raster mode.

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