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XEDS Performance of Atmospheric Membrane Holders in the AEM

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The use of SiNx windows has been well established as a technology [1] which can be readily employed to observe microstructural evolution during *in situ* studies in the TEM/STEM as an alternative to experiments using a differentially pumped environmental TEMs. In previous studies [2-3] using SiNx membrane windows in liquid cells it was shown that the penumbra of the holder was the principle limitation in the use of X-ray Energy Dispersive Spectroscopy (XEDS). This was principally due to the cover/lid design, which can partially or completely block x-ray signal detection. The modifications which were established for that work have recently been extended to gaseous holder configurations. In this work FEI Tecnai F20, and CM200F TEMs equipped with windowless SDD systems from EDAX (Apollo) were used to evaluate the performance of an implementation of this technology on a Protochips Atmospheric Holder both for simple spectroscopy as well as mapping at nominally 1 ATM.

Although the penumbra of the holder has been minimized as in the liquid cell design, next in importance is the penumbra created by Si frame, which illustrated in Figure 1. Shown schematically is the 300 µm tall Si frame which overshadows the SiNx window area. The penumbra angle can be simply calculated by noting the beam position D with respect to the Si side wall of height H i.e. $\theta = \arctan(H/D)$. To measure the relative effect of the Si side wall penumbra for a given location, we investigated a 50 nm thick SiNx window which was covered by a uniformly thick Au film the combination of which was supported on 300 µm Si frame sandwich (Figure 2a) with a 5 µm air gap. By tilting the holder to the optimum angle unshadowed angle dictated by the holder penumbra [4], we then measured the AuL Intensity as a function of position across the width (Y) and length (X) of the SiNx film. In Figures 2b and 2c shows the results while traversing the parallel (Y) and perpendicular (X) directions relative to the tilt axis as indicated. In 2b, one can see the relatively uniform intensity profile across the Y axis of the chip, indicating minimal shadowing, while traversing the X axis one, not surprisingly, sees the top half of the window has little to no shadowing while the bottom half gets progressively worse as one approached the Si sidewall. Of course with dual XEDS systems located on either side of the tilt axis the penumbra effect on the each side can be minimized by simple tilting of the holder toward the oppositely oriented detector. In figure 3a and 3b we show the effects of multiple scattering in the 5 µm thick air gap obtained by traversing across an edge into fortuitous holes in the Au metal film with the film on first the top then the bottom window. The gas path introduces a significant scattering profile and decreases the spatial resolution, due to an electron skirt effect. On the top window, the Au signal drops to near zero, but the skirt at the bottom window introduces a significant background level. Indicating that for optimum XEDS and/or STEM the material of interest should be on the top window, while for EELS and/or TEM the materials should be on the bottom window. Finally in figure 4, we confirm that high resolution ($\leq$ 10 nm) hyperspectral data sets can be obtained at 1 ATM with a 5 µm air gap when the region of interest is located on the appropriate top window.

References:

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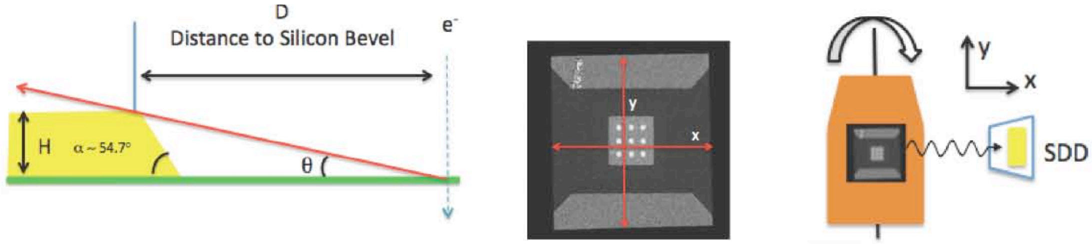

Fig. 1a) Penumbra of Silicon Side wall of eChip b) Window of eChip c) Chip /Detector Geometry

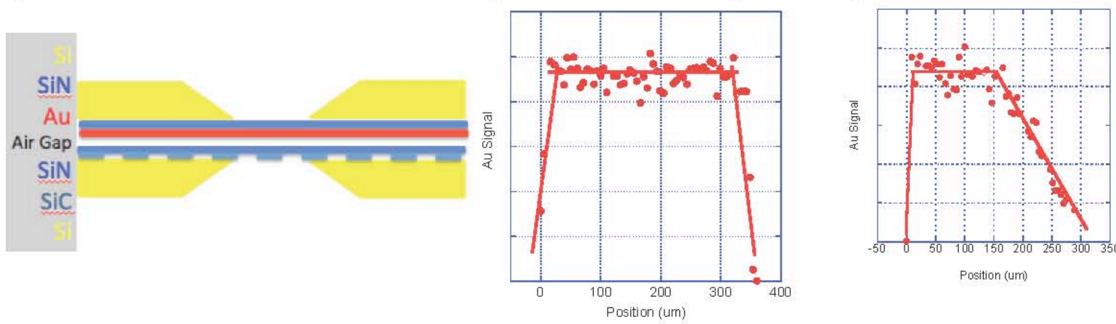

Fig. 2a): Gas Cell Geometry b): Au L Profile along Y axis c): Au L Profile along X axis

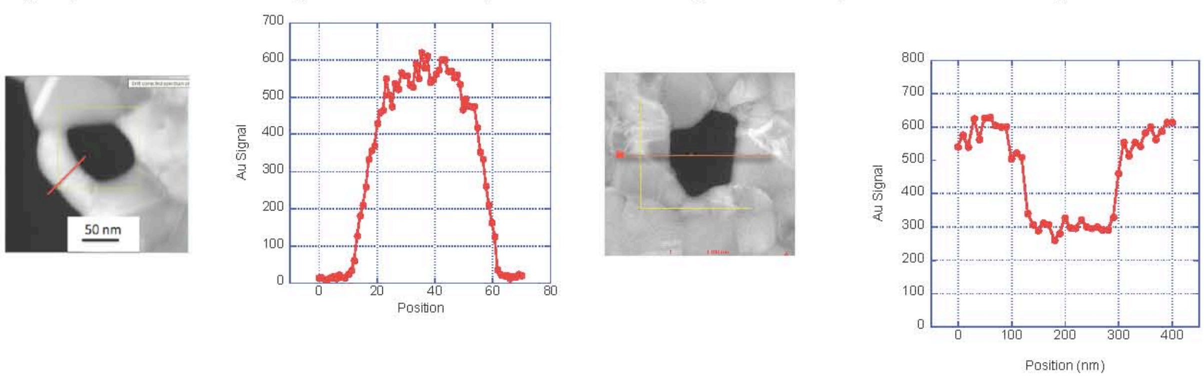

Fig. 3a) Au Bridge on top window, b): AuL Profile across bridge to hole in 3a, c): Hole in Au Film at bottom window, d): AuL Profile across hole in Fig. 3c

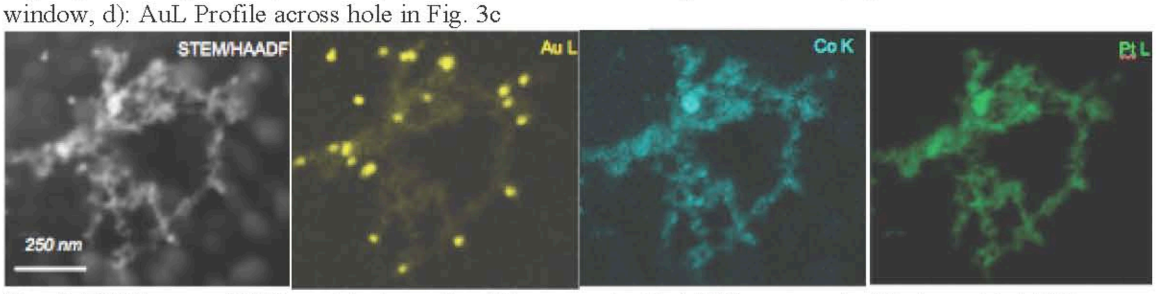

Fig. 4) XEDS map of Pt/Co catalysts on Graphite support with 10-30nm nanoAu Markers at 1 ATM