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Quantifying Contamination in Mass Spectrometers Using SEM-EDX

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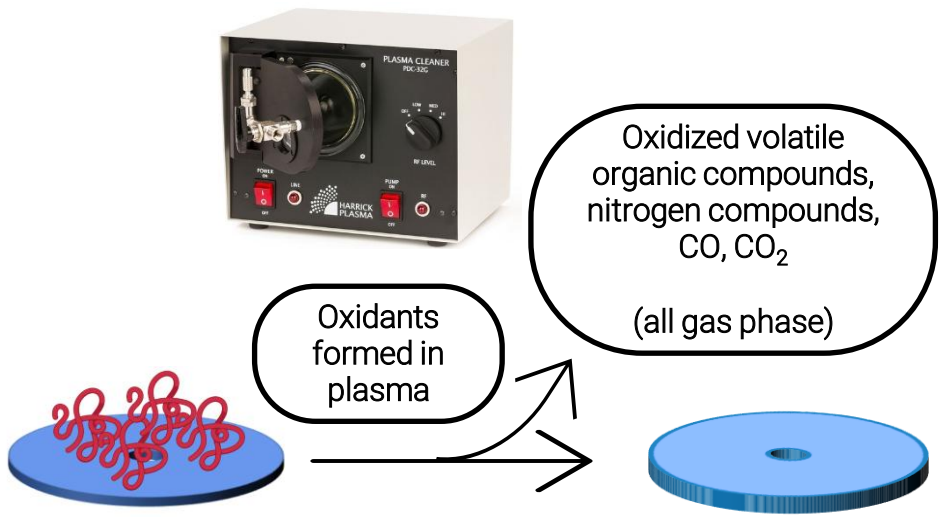
Introduction

Characterizing surface charging on a surface using the Duane-Hunt Limit for X-ray-dispersive spectroscopy

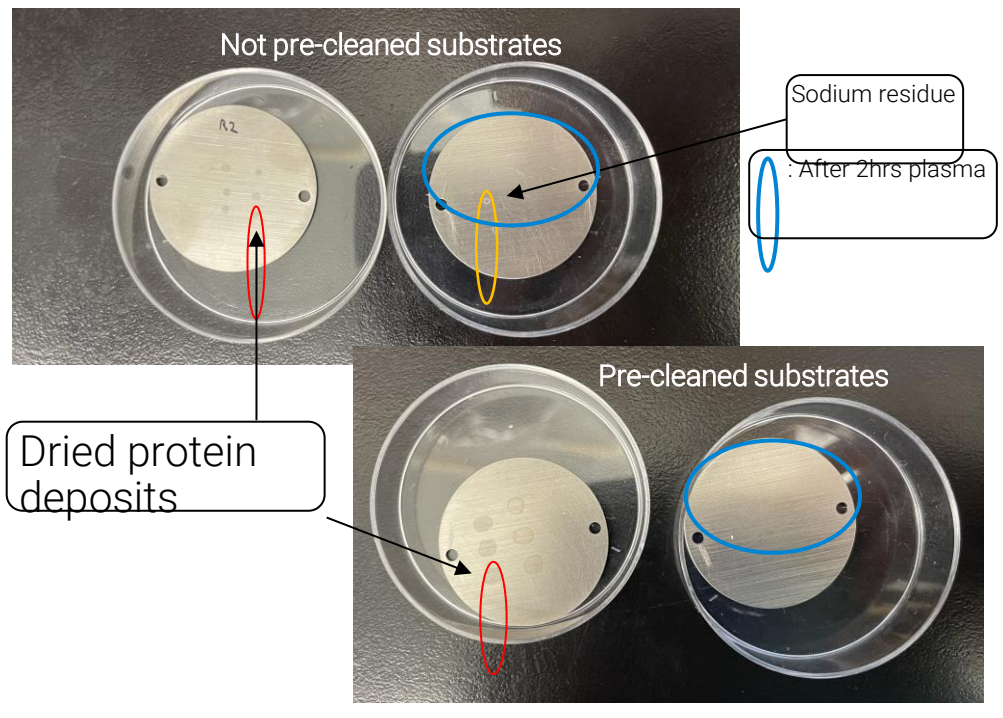
Background and motivation

- Contamination of surfaces by proteins and peptides causes performance degradation in mass spectrometers.[1]
- The Duane-Hunt limit (DHL) is the high-energy cutoff of the background (bremsstrahlung) X-ray emitted from the sample and can be measured in a scanning electron microscope with energy dispersive X-ray spectroscopy (SEM-EDX).[2]
- The DHL has been used to quantify the degree of charging on insulators.[3]
- Removing contamination by gas phase oxidants could enable in-situ cleaning of mass spectrometers. We investigate the effectiveness of gas-phase oxidants produced from air in a plasma cleaner.



Gas phase oxidants can be produced in a plasma cleaner and remove contaminants from metal

Air plasma as a method to remove protein stains

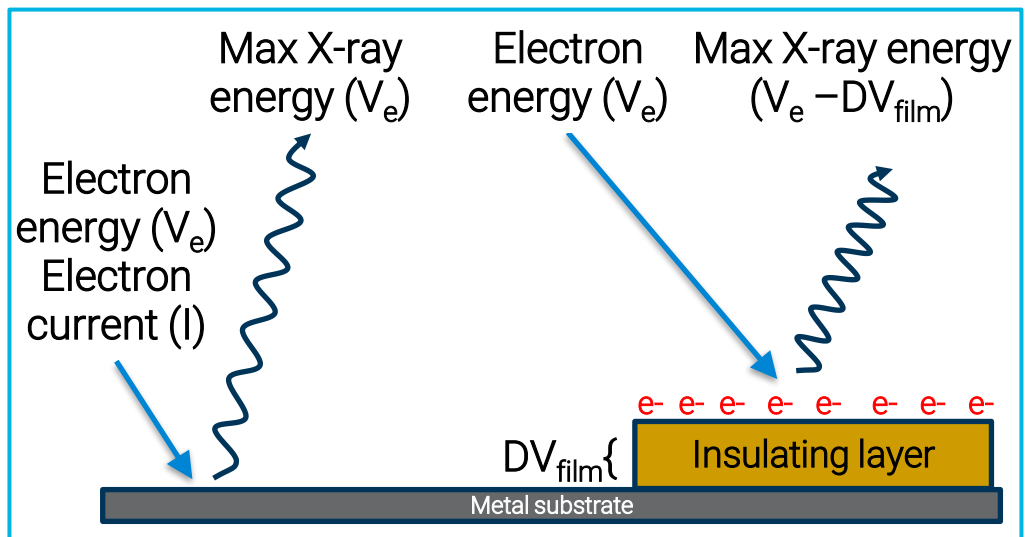


Visual comparison of protein deposits before and after cleaning. Labels are experimental details.

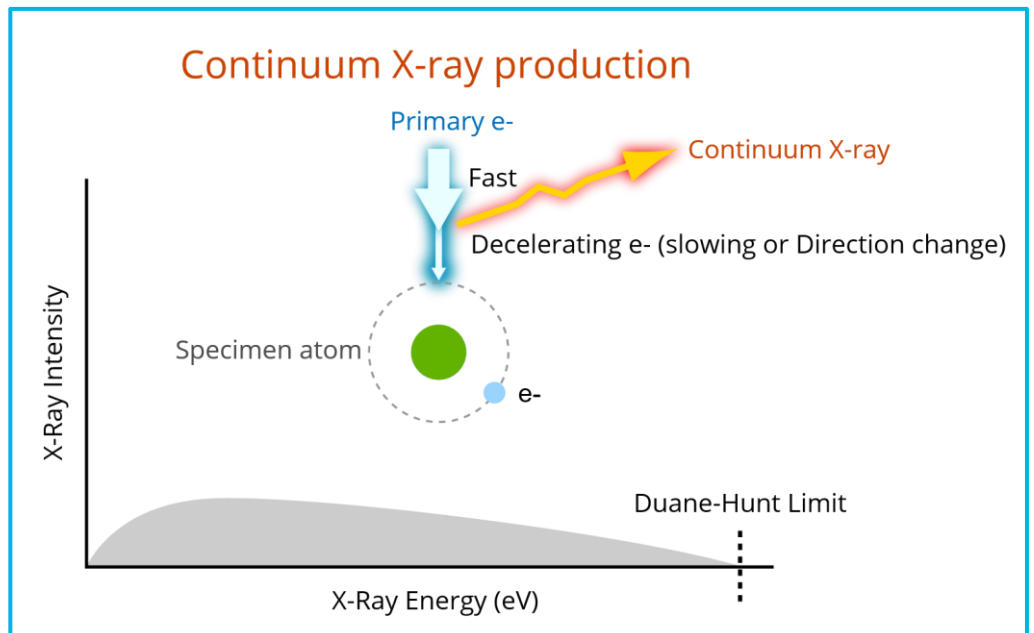
Experimental

Experimental Details

- A Hitachi SU3900 SEM with a 5kV accelerating beam was used to collect EDX spectra of selected points on each protein droplet.
- Characteristic X-rays from carbon were used to quantify protein content on the metal surface.
- Bovine Serum Albumin (Sigma) dissolved in Milli-Q water was deposited on stainless steel substrates. Substrate preparation was similar to that used for ion lenses in mass spectrometers.



Electron interactions on a conductive surface (left) and an insulating film (right) within a SEM. In the presence of an insulating layer, electrons are decelerated before interacting with the sample.

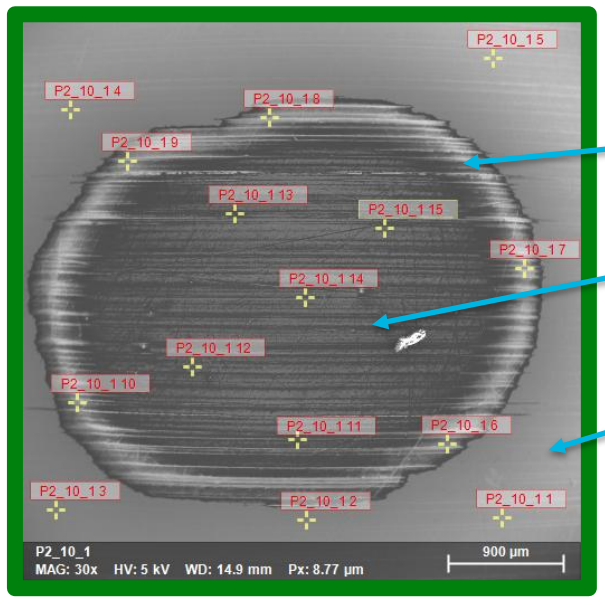


Continuum X-rays constitute the EDX background. The DHL is the maximum energy of the background X-rays. Surface charging on proteins electrostatically repels electrons, decreasing the DHL. Image from https://myscope.training/EDS_Bremsstrahlung_X_ray_generation

Surface charging by proteins can be measured with the Duane-Hunt limit from SEM-EDX

Air plasma cleaning removes carbon and surface potential

Before plasma cleaning

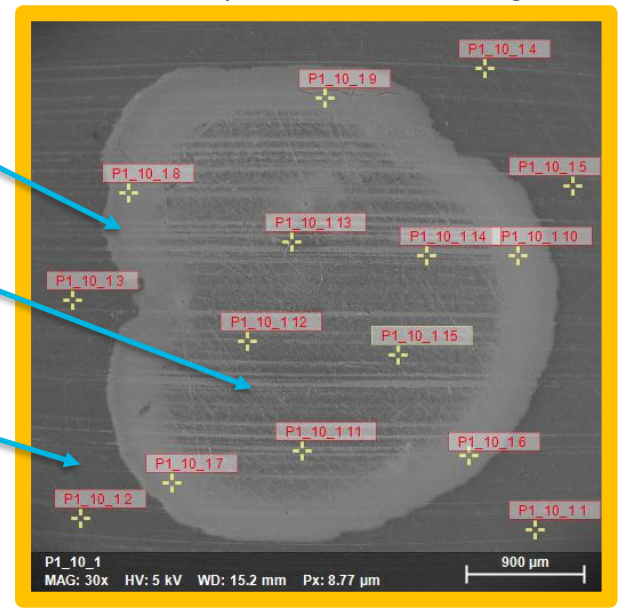


Thick deposit on edges

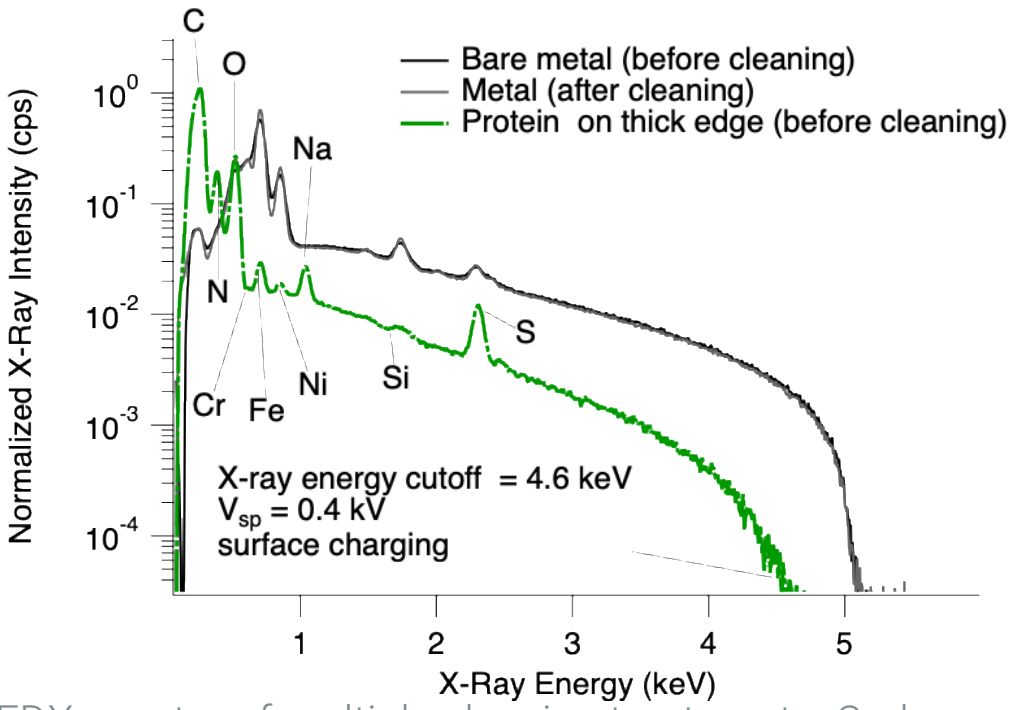
Thin deposit on center

Metal spots outside

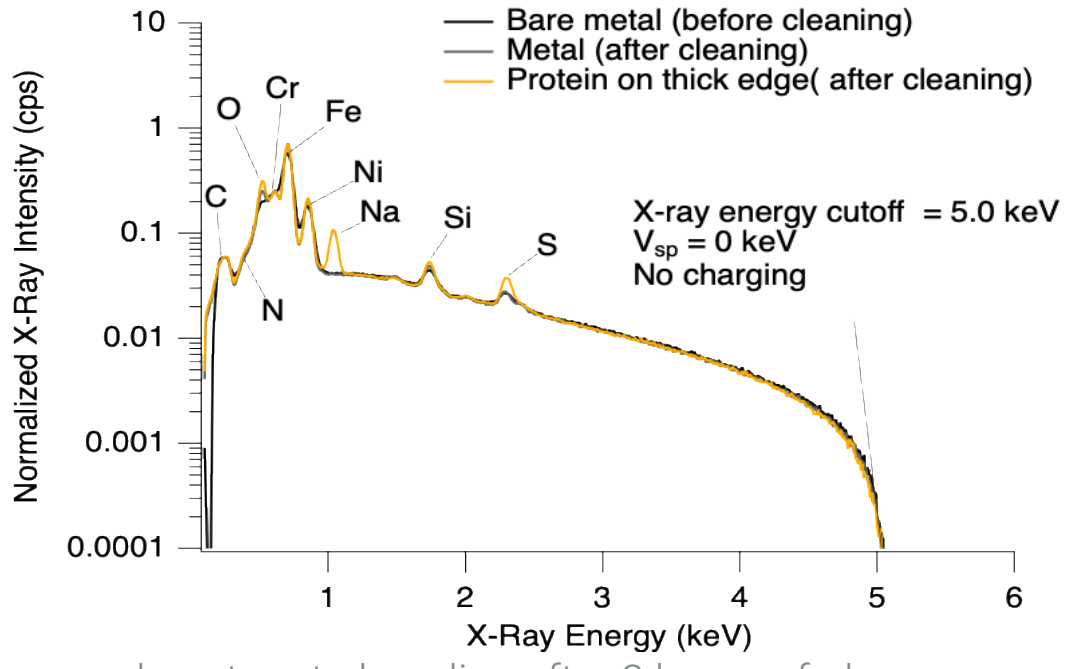
After plasma cleaning



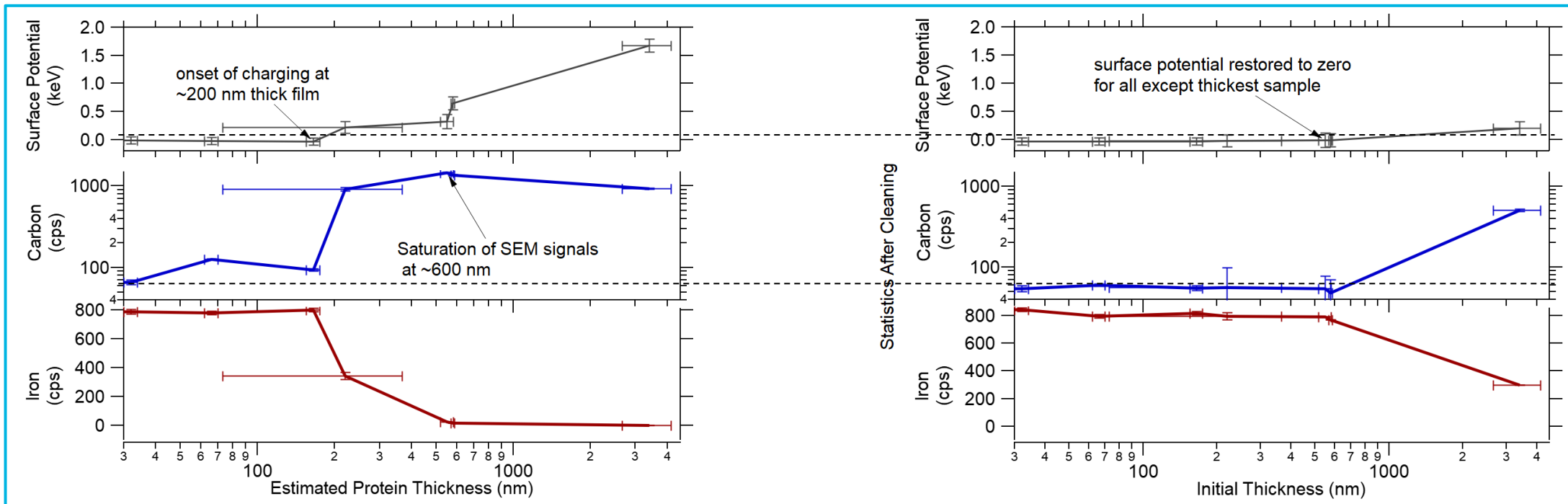
Contaminated protein layer shows charging



Charging removed after cleaning

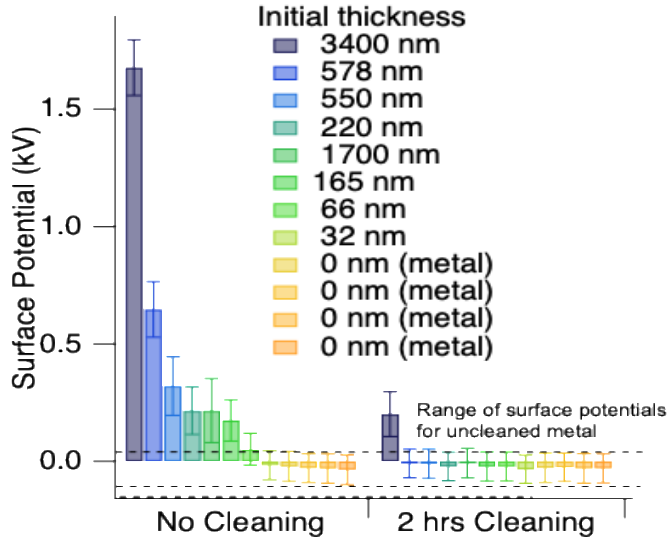


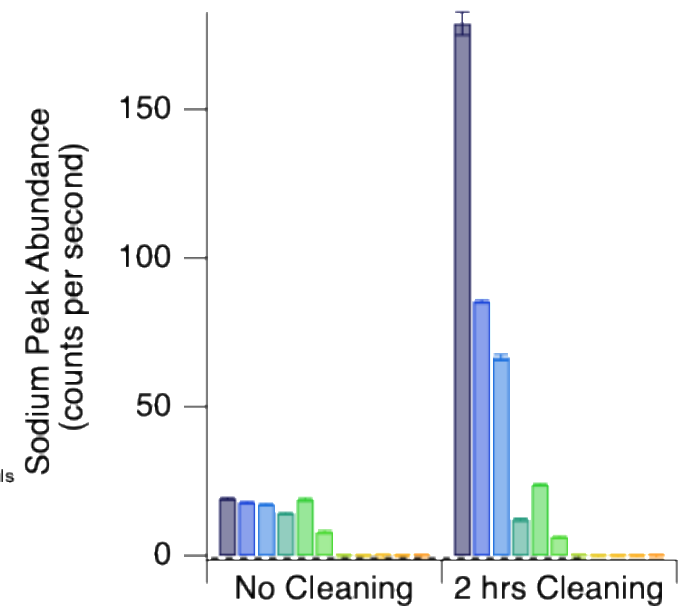
EDX spectra of multiple cleaning treatments. Carbon and nitrogen peaks return to baseline after 2 hours of plasma cleaning, metal substrate peaks are restored, and the DHL returns to 5 keV after plasma cleaning, indicating removal of the surface potential.

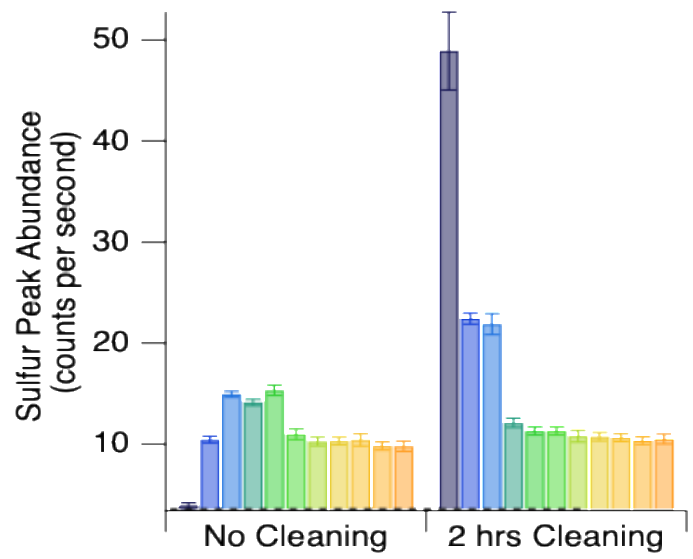


Comparison of surface potential and carbon abundance before and after two hours in the plasma cleaner. Thickness estimated from concentration and diameter of protein drop. The uncertainty in thickness comes from uncertainty in droplet relative thickness due to the coffee ring effect. Surface potential, carbon counts, and iron counts returned to the baseline for all spots except for the thickest, indicating successful removal of the insulating layer.

Results and Discussion

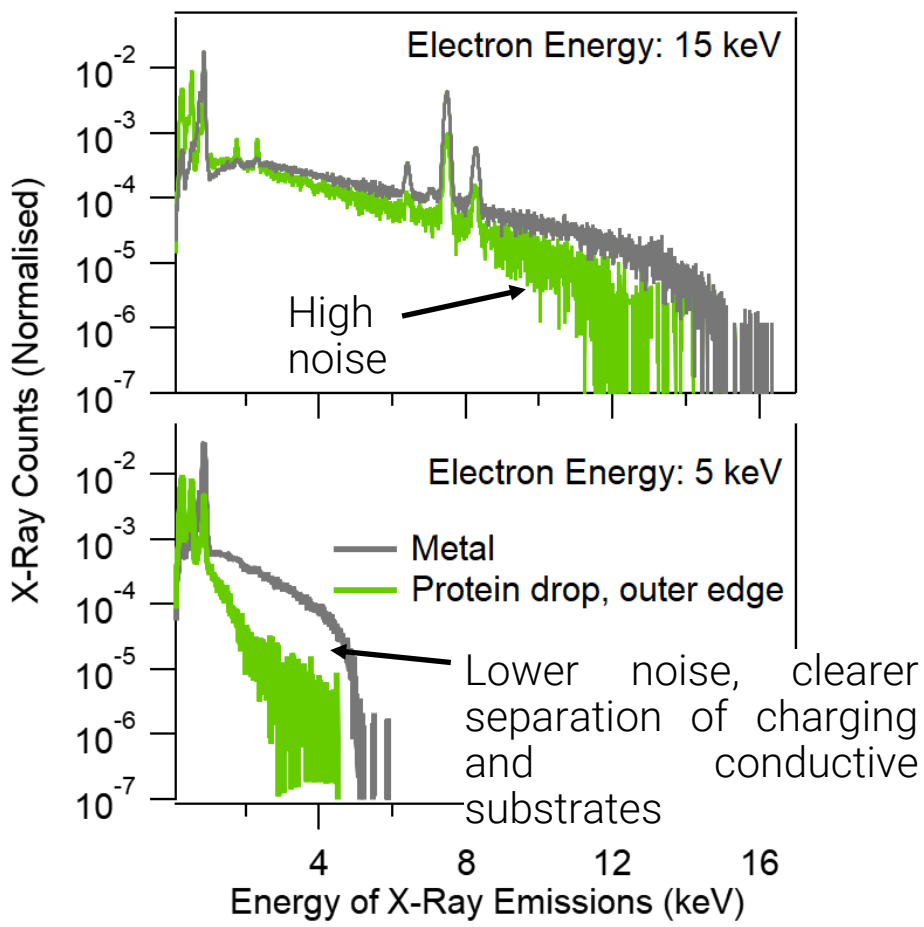


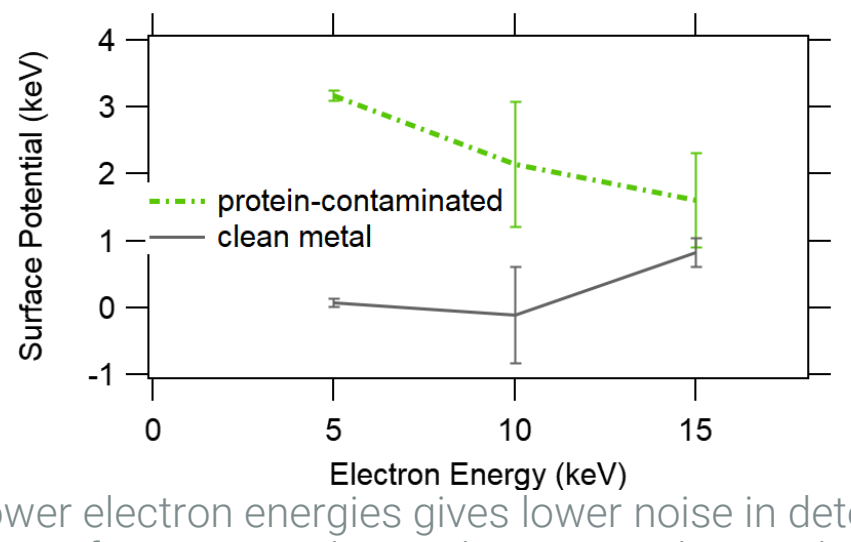




After plasma cleaning, surface potential is restored to bare metal levels. The concentration of sodium and sulfur peaks, increases, indicating that those elements remain on the surface after the organic material is removed. The sulfur most likely originated from amino acids in the protein. Future work may need to investigate if the recalcitrant portion causes charging at higher concentrations. Results are shown for stainless steel, but gold substrates shows similar trends.

Methodology: lower electron energy improves signal to noise of surface potential measurement





Lower electron energies gives lower noise in determining the surface potential. Note larger error bars at higher electron energies.

Conclusions

Quantifying surface charging

- SEM-EDX is a quantitative method of simultaneously characterizing surface charging and elemental composition of a protein on a metal surface.
- >200 nm of protein material will cause charging that can be detected by the Duane-hunt limit.

Surface cleaning

- Air plasma can effectively remove organic material and restore the surface potential and carbon abundance to clean surface levels. Recalcitrant sodium and sulfur remain.

References

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