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An In Between Study: The usage of cross-sectional SEM and Raman Spectroscopy to map the surface interfacing of a mastodon tusk and its lacquers

Katherine Plotzke
University of Northern Iowa

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An In Between Study: The usage of cross-sectional SEM and Raman Spectroscopy to map the surface interfacing of a mastodon tusk and its lacquers

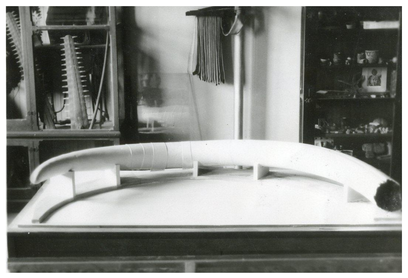
Katherine Plotzke; UNI Department of Chemistry & Biochemistry

ABSTRACT

- A microscopy study utilizing a DXR2 SmartRaman and Evex Mini-Scanning Electron Microscope (SEM) was done on the UNI Museum's Mammut americanum (American Mastodon) tusk. A map of the surface interfacing was generated and provided information about the elemental composition and layer interconnectedness. Additionally, this project will also aid in the future restoration work on the tusk funded by the Roy J. Carver Charitable Trust.

BACKGROUND

- In 1933, the University of Northern Iowa museum acquired a large Mammut americanum (American Mastodon) tusk from a sand pit near Hampton, Iowa[1].
- The tusk is dated between 120,000 and 200,000 years old and due to the usage of outdated methods of restoration is in a state of disrepair[1].
- Recently, the tusk became a topic of interest and funding to restore the tusk was granted through the Roy J. Carver Charitable Trust.



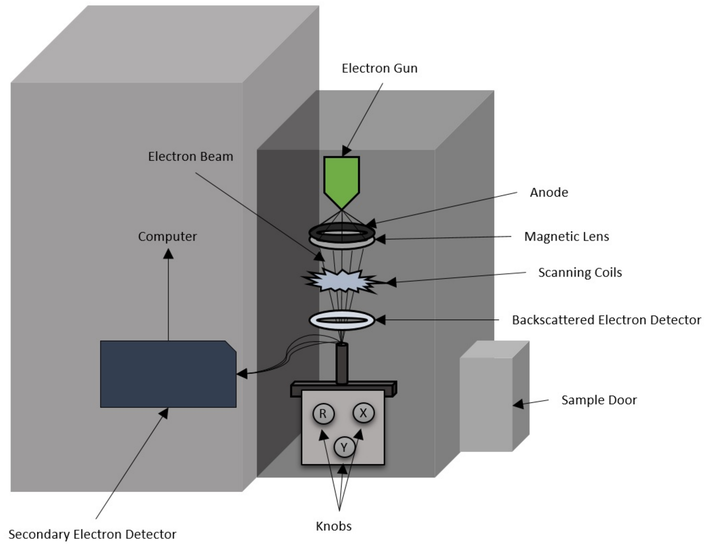
- Above is a picture of the tusk after the first round of restorations, including the reattachment of the tip of the tusk and banding of the tusk with sheet metal to hold it together.
- The tusk has had many different lacquers applied to it in the last century resulting in several layers. An example of these layers can be seen below.

- Differentiating between the different layers is difficult without the aid of instrumentation. Additionally, it is important to know how interconnected the layers are and why they are peeling in order to speed up the process of removing them in the future.

OBJECTIVES

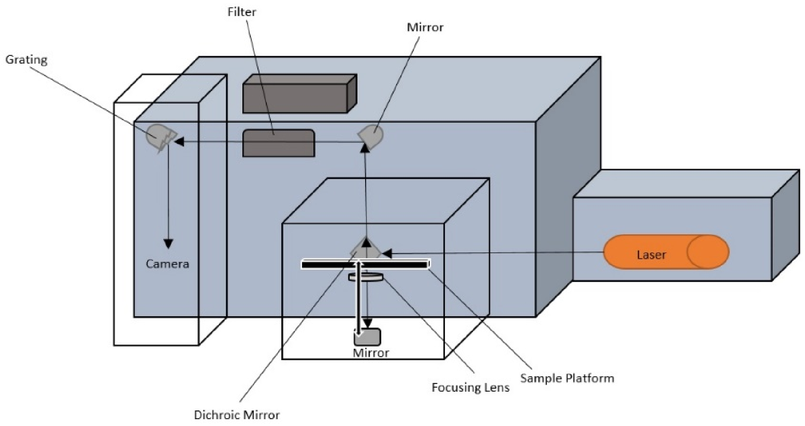
- Determine if the different layers of lacquer on the tusk are interconnected.
- Utilize SEM at a magnification of 300x or greater to visualize the layers.
- Demonstrate the compositional difference of the layers using SEM-EDX and Raman spectroscopy.
- Quantify the interfacing of the layers if any is found to have occurred.

SCANNING ELECTRON MICROSCOPE



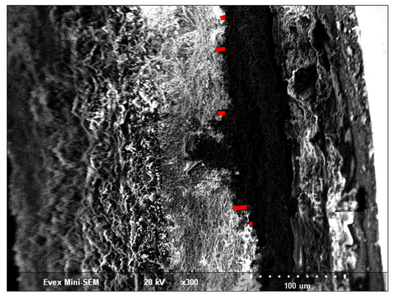
- The Evex Mini-SEM is most often used to determine the morphology or texture, chemical composition, crystalline structures, and orientation of materials that make up a solid sample[2].
- This is made possible by focusing a high-energy beam of electrons at the solid surface of the sample, in this case a cross section of all the layers. The cross section was obtained from the tusk, reduced to the size of a fingernail, attached to a stub mount, and then placed inside the SEM for analysis
- The SEM has magnifications between 20x to 30,000x, this instrument proved highly useful by generating high resolution, 2-dimensional images of the surface of the mastodon tusk surface interfaces[2].
- An Energy-Dispersive Spectrum (EDS) generated the elemental map of the sample. The EDS presented as a plot of x-ray counts vs. energy, where each peak represents a different element[3].

RAMAN SPECTROMETER



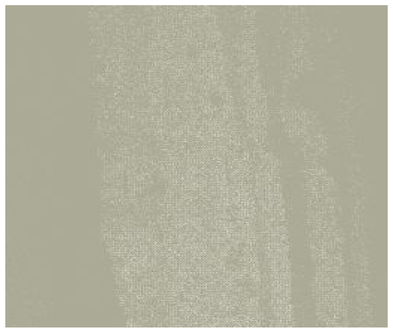
- Raman focuses on the chemical analysis of the different lacquer surfaces being examined by the SEM.
- Raman spectroscopy is the act of focusing a mono-wavelength laser at the sample surface and recording the scattering of light that occurs afterwards[4].
- This effect is due to the change in the vibrational, rotational, or electronic energies of the different molecules and was very essential in the determination of the chemical makeup[4].
- Raman spectroscopy was vital in determining the differences between the layers of the tusk.

RESULTS



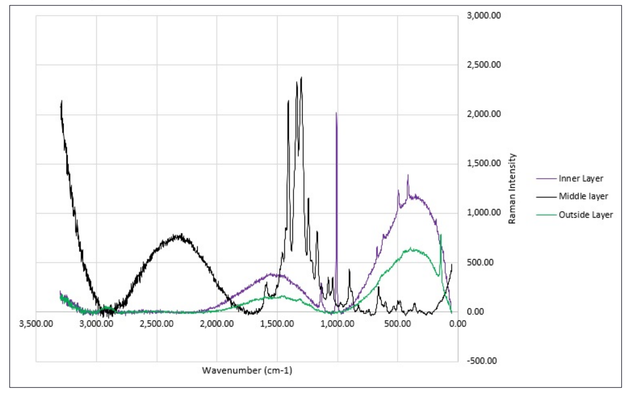
300x Magnification SEM scan of all the layers

- SEM picture taken of all the layers of the tusk at 300x magnification.
- The layers of the tusk were shown to be 16.8 µm overlapping, on average(see red markings on picture).



700x Magnification EDX scan displaying the differences between organic (brown) and inorganic layers (white)

- Image generated by EDX using an SEM image.
- Allows for the different layers to be determined. The tusk is displayed in red, the organic layers can be seen in brown, and the inorganic layers can be seen in white.
- Further complicates the interfacing of the layers.



Raman spectra of the first three layers of lacquer of the tusk

- Raman spectra displayed, differentiates between three layers. The outer three layers of lacquer can be seen here and each spectrum was taken separately with the laser focusing solely on the layer pictured.
- The inner layer is the only spectrum with a Phosphate peak at 1003 cm^{-1}, the middle layer has a band for Sulphur at 1470 cm^{-1}, and the outside layer has a small peak for Titanium near 100 cm^{-1}.

CONCLUSIONS

- The average interfacing between two layers is 16.8 µm. This indicates that the layers are highly interconnected with each other.
- The EDX demonstrated that all the layers are comprised of alternating compositions, further complicating the surface interfacing.
- Raman spectroscopy indicated that the layers even though they are comprised of some of the same materials, are vastly different from each other in terms of complete chemical make up.

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