

Risk Assessing the Use of Variable Pressure SEM for the Examination of Cultural Heritage Artefacts

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Variable pressure SEM (VPSEM) combined with microanalysis to provide detailed compositional information has become the method of choice for high resolution imaging and analysis. However, the SEM chamber is not a benign environment for delicate and often unique artefacts [1][2][3]. Understanding the risks associated with the examination of samples using VPSEM should be a vital part of every research project involving cultural heritage materials. However, there is a lack of information about what actually happens within the chamber.

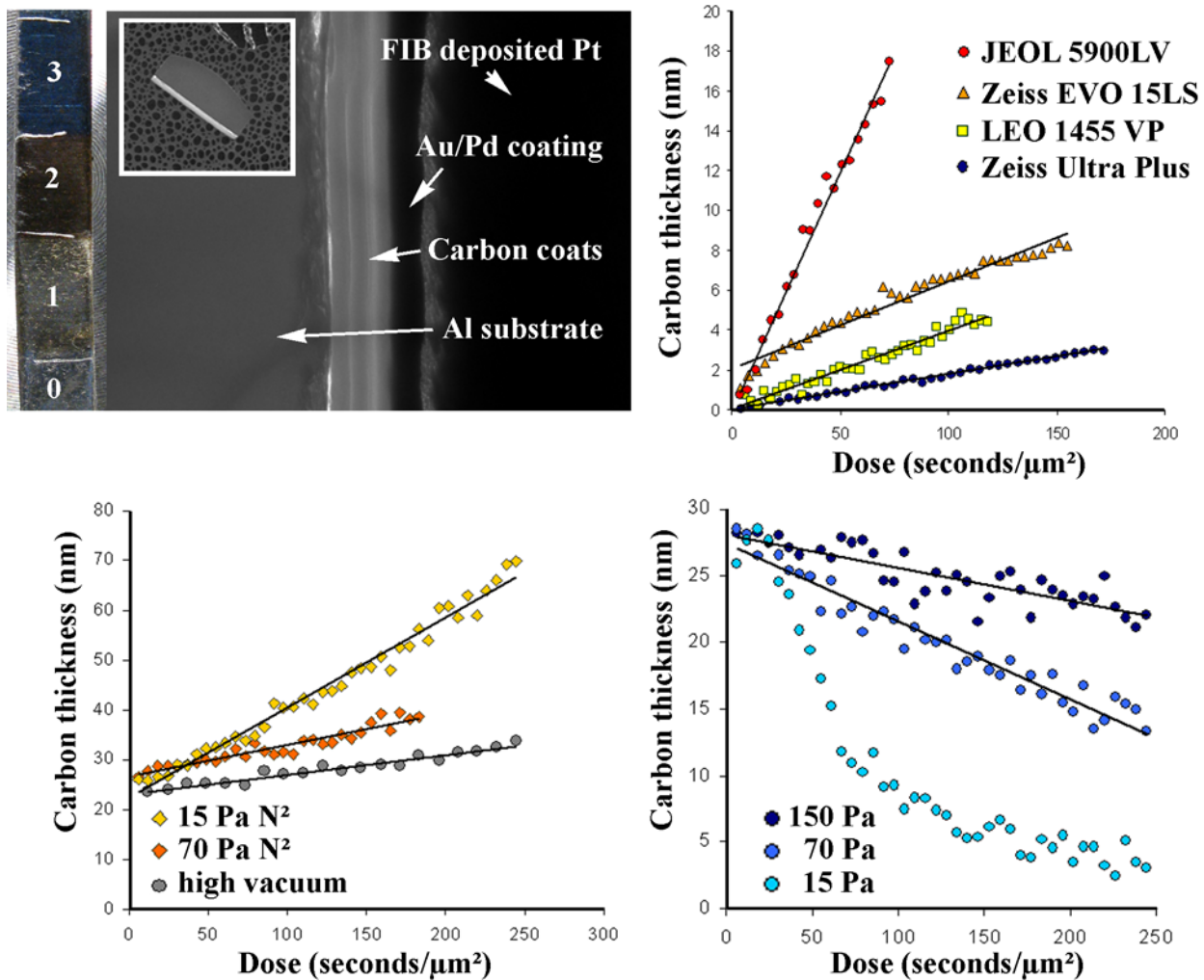
Our study investigated methods for quantitative assessment of contamination within the SEM under a wide range of operating conditions and using different chamber gases. We propose a simple and repeatable technique to assess an individual SEM to determine how likely it is to contaminate or damage a sample (Fig. 1). The technique requires access to an EDX system and measures the conditions in the microscope directly. As such, it represents an improvement on the rather qualitative or instrumentally demanding procedures which have previously been proposed [4]. It could be adopted as part of a routine quality control monitoring process of the instrument, by researchers visiting another institution or amongst collaborators to ensure the proper handling of loaned samples.

Within the VPSEM, samples are exposed to rapid decompression and recompression. The interactions between the imaging gas (usually nitrogen, air or water vapour), the beam and the sample are complex [5] and can lead to the deposition of contaminants (derived from the vacuum system, the instrument itself or from the sample) (Figs 2 and 3) [6] and the formation of corrosive products (e.g. hydroxyl and oxygen radicals)(Fig. 4) [7]. The beam also contains sufficient energy to damage the sample directly [1][2][3], especially when it is maintained in one place for an extended period of time (for example, during spot analyses). The combination of these effects can lead to the sample being materially altered during examination within the microscope or the acquisition of inaccurate or misleading data [8]. We suggest that a proper understanding of the conditions involved can lead to an informed risk assessment of the procedure which can only improve the quality of care for the collections which many of us curate or study.

References

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Figures

Fig. 1. An aluminium test strip (left) was coated with three layers of carbon to give progressively thicker coated regions (labelled 0-3). Coating thicknesses were measured from a FIB section (inset) by TEM.

Fig. 2. The rate of carbon deposition was measured under high vacuum at 5kV, using calibration plots derived from the standard, for a range of SEMs.

Fig. 3. Using nitrogen as the imaging gas, the deposition rates can be compared at high vacuum and under VP conditions (15 and 70Pa) on a coated section of the foil. This showed that carbon was deposited onto the foil more rapidly at 15Pa than at 70Pa.

Fig. 4. Using water vapour as the imaging gas, carbon is eroded from the coated region of the foil. The erosion rate is greatest at low chamber pressures.