doi:10.1017/S1431927616003032
Microsc. Microanal. 22 (Suppl 3), 2016

Standardless Quantification at Trace Elemental (PPM) Levels Using a Novel Attachment within an Electron Microscope and Microprobe

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Despite significant advances in quantitative compositional mapping using electron microscopy based techniques (e.g. SEM-EDS and EPMA), the vast majority of analyses are performed standardless [1], which can lead to systematic errors that limit the accuracy of the results [2]. Here we present a newly developed x-ray illumination beam attachment developed to enable complementary, parts-per-million sensitivity at microns-scale resolution. The attachment will provide 10 to 1000-fold improvement in detection limit, and can be used to supplement standard-less electron-induced fluorescence information.

The Sigray FAAST-SEM™ attachment relies on several key innovations, including: (1) a revolutionary, patent-pending new type of high flux x-ray source designed to be >10X brighter than the brightest rotating anode x-ray source currently available; and (2) an x-ray mirror lens with high spatial resolution, high focusing efficiency, and large working distances designed to accommodate the geometries of existing SEM chambers. Over existing commercial micro x-ray fluorescence (μXRF) attachments, the system provides 300-500X speed to provide equivalent trace elemental mapping within minutes rather than hours or days for conventional approaches. Moreover, the system resolution is designed to have resolution comparable to electron-based fluorescence spot sizes for correlative measurements.

A major design consideration for the illumination beam system was optimizing the quantification capabilities through careful selection of the x-ray focusing optic to be used. X-rays intrinsically provide high fidelity for compositional analysis, as photons do not encounter the same "bloom" effect as particle-based (e.g. electron-based) microanalysis and produce fluorescence from only the illuminated spot at the sample. However, current approaches to x-ray optics are primarily through use of polycapillary x-ray optics, which concentrate x-rays of various energies into illumination spots of differing sizes at the sample. This results in the fluorescent x-rays emerging from different regions within the sample that obscure the compositional information within a specific region. In the invention presented here, the x-ray mirror lens focuses all polychromatic x-rays onto a single spot, enabling precise determination of composition by eliminating interfering x-rays from adjacent regions.

In addition to the optic, the x-ray source used in the system utilizes a unique microstructured design that takes advantage of the outstanding thermal properties of diamond for rapid heat dissipation and to allow for brighter x-ray generation. Future directions for the illumination beam system will be discussed, including dual energy focusing to optimize detection sensitivity for a broad range of elements. Specifically, the little known potential and advantages of x-rays for the detection of light elements will be discussed.

Initial results of the system for trace elemental mapping and quantification in biological and geological samples at spatial resolutions on the microns-scale are discussed. Applications for use within a SEM-EDS and EPMA include: calibration of quantification, correlation for thin film analysis, and analysis of biological and low Z elements. [3]

References:

[1] DC Bell. *Energy Dispersive X-ray Analysis in the Electron Microscope* (2003).
[2] DE Newbury. *Scanning* (2013).
[3] Acknowledgments go to the NSF, Division of Industrial Innovation & Partnerships for funding the development of x-ray mirror lens (IIP-1448727) and to the NIH, National Institute of General Medicine Science for funding the development of the microstructured source target (GRANT11545218).

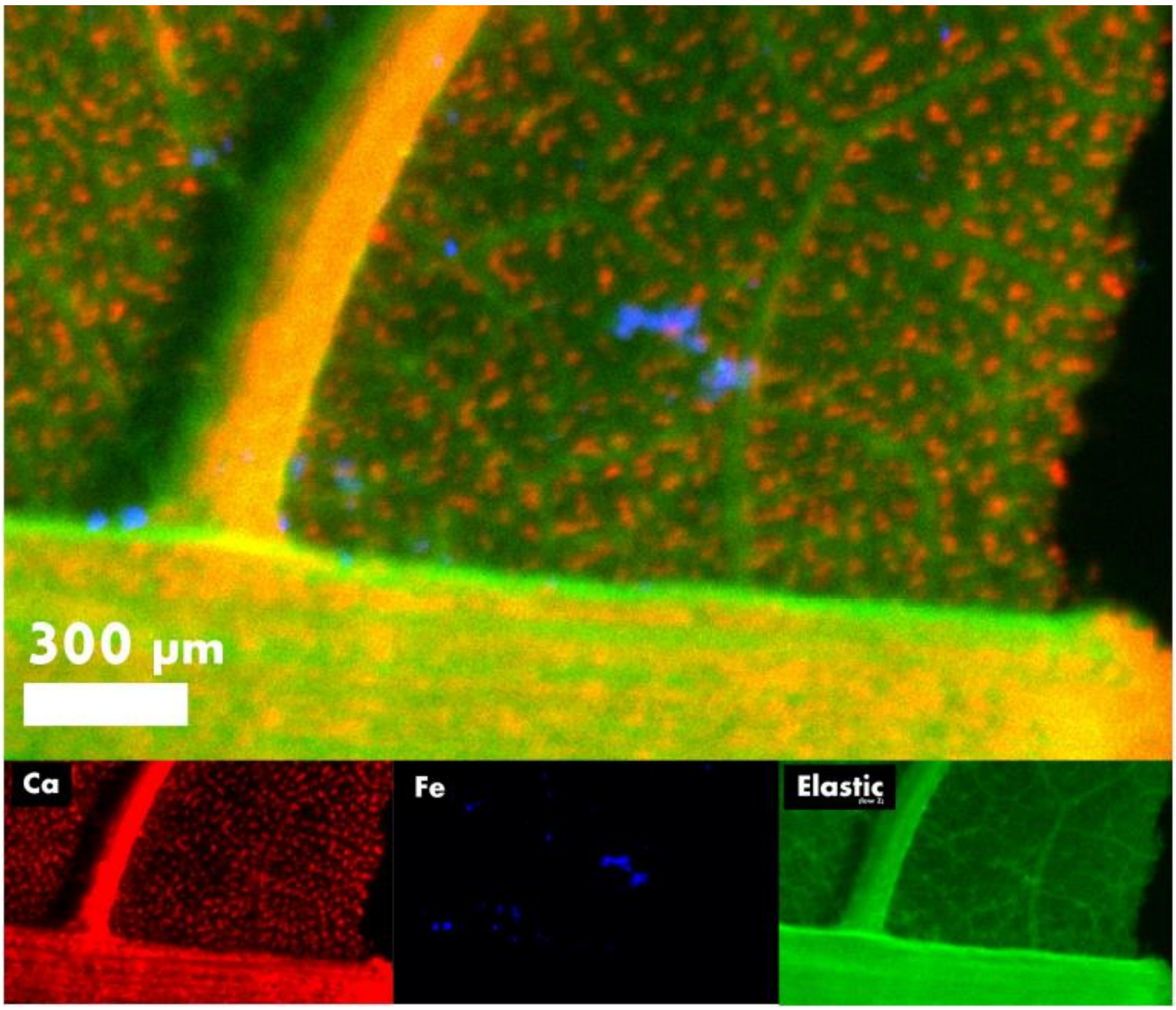

Figure 1. Distribution of three selected elements (Calcium, Fe, and low Z elements) in a leaf

https://doi.org/10.1017/S1431927616003032 Published online by Cambridge University Press