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Mapping Subsurface Composition with Attogram Sensitivity using Micro-XRF

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X-ray techniques have grown substantially in recent years. From developments in the instrumentation to advancements in the data processing techniques, X-ray technologies are at the cutting edge of characterization tooling. While rapid growth has been observed within X-ray imaging techniques, such as X-ray microscopy (XRM) [1], compositional analyzers such as X-ray fluorescence spectrometers (XRFs) have also been in a stage of rapid advancement [2]. Owing to the high penetrating power of X-ray radiation, XRF systems are able to probe deep (up to 100s of microns) below the surface of a material, affording researchers unique insight into the elemental composition of bulk materials [2]. This advantage drastically reduces the need for extensive specimen preparation, allowing many materials to be analyzed without any special preparation to the specimen's surface.

The XRF technique has, however, historically been somewhat restrictive: detection sensitivities are practically limited to the parts-per-million (ppm) regime, making these techniques most suitable for large-scale elemental analysis. Furthermore, the spatial resolution offered by the technique is often in the 100s of microns at best, which restricts the spatial specificity and introduces some uncertainty about which section of the specimen contributes the signal being detected. In spite of this, still much excitement remains about the XRF approach alongside conventional surface analysis techniques, as its unique non-destructive subsurface characterization abilities make it highly differentiated from other equipment that is often found in characterization laboratories. [2]

In our work, we have completely redesigned the micro-XRF concept, beginning with the X-ray source. Using a fine array of small micro-targets embedded in a diamond substrate, the heat dissipation has been significantly enhanced, enabling source brightnesses to be increased by nearly two orders of magnitude (>50x). These target arrays may be created with a variety of materials, allowing users to fine-tune the source spectrum to optimize the system for each new sample type. This source is paired with a novel twin paraboloidal X-ray focusing lens, allowing the array of microbeams to be condensed to a tiny spot, thus increasing spatial resolution while also increasing detection sensitivity. Combining the advancements in X-ray source and X-ray optic technologies, we have produced a micro-XRF system with spatial resolutions in the 1-10 um length scale and detection sensitivities in the parts-per-billion (ppb, or attogram) regime. This increases the general performance of laboratory XRF by several orders of magnitude and provides a precise characterization platform for advanced spectroscopic studies.

Here, we will present the architecture of this system and discuss its application to a variety of disciplines. The presentation will close by summarizing a study performed on different types of tempered glasses, where the XRF spectrometer was able to detect the differences in commercially-sourced mobile phone screen protectors. These results were achieved using the high spatial resolution of the spectrometer coupled with its trace element detection sensitivities, delivering a unique result that serves to illustrate the power of this technique.

References:

[1] A P Merkle and J Gelb, Microscopy Today **21** (2013), p. 10–15.
[2] T Arai in "Handbook of Practical X-Ray Fluorescence Analysis", ed. B Beckhoff, B Kanngießer, N Langhoff, R Wedell, and H Wolff, (Springer, Heidelberg) p. 1-26.

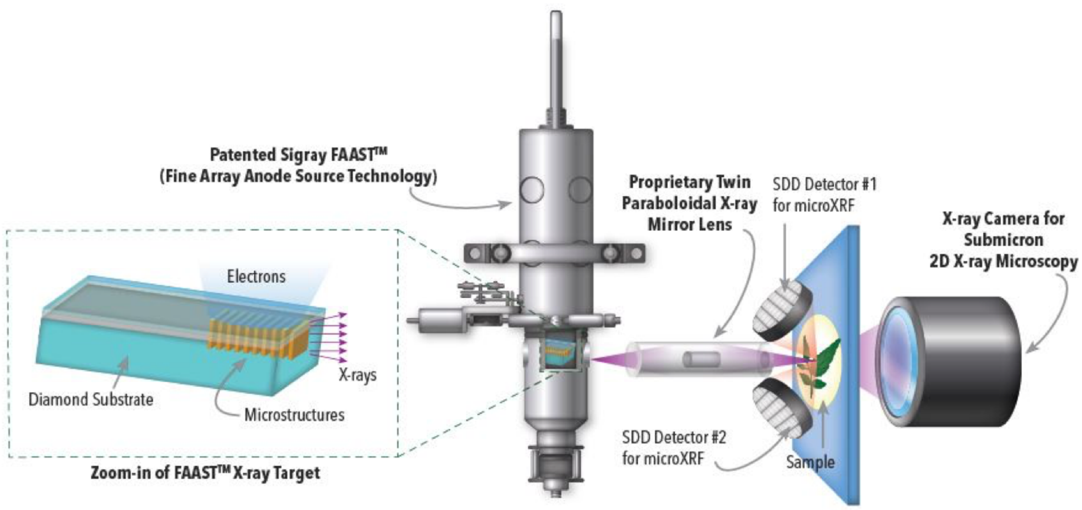

Figure 1. Schematic diagram of the Sigray Attomap micro-XRF spectrometer. A fine array of custom micro-beams is focused to a small probe source by the twin paraboloidal X-ray lens, providing high spatial resolution and ppb detection sensitivities for elements in the specimen being investigated.

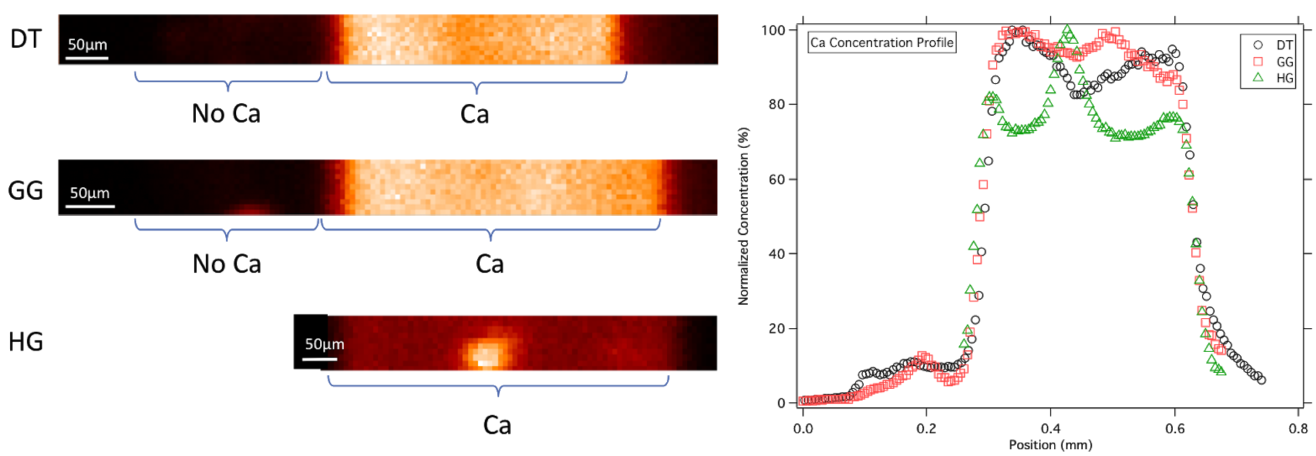

Figure 2. Example micro-XRF map of calcium (Ca) content in a commercial tempered glass. The high spatial resolution coupled with high detection sensitivity enables local hotspots of Ca buildup to be identified and properly classified.

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