Next Generation of Instruments Required - Not just X-Ray Imaging but Combined EDS, CL, GSR, XRM, XRD and Raman Systems

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The materials used in our society today are constantly changing. For example, in recent years, in the forensic world, heavy metal free (HMF) cartridge primers have been developed to reduce health risks to shooters (police and sporting shooters) from lead (Pb) and other heavy metals. Gunshot residues (GSR) from suspects and their clothing, or the environment, have traditionally been detected using SEM and EDS. However, with the advent of HMF residues, which consist of primer formulations of low average atomic number elements like NaAlSiK (glasses and feldspars) that are not so easily detectible by traditional SEM/EDS and GSR methods. Other techniques, combined with traditional SEM/EDS/GSR, are required for the analysis of these types of ammunitions. With ever increasing popularity of composite materials being utilized in the construction and engineering environment, not only is elemental information required, but also other spectroscopy techniques are used to determine the presence of polymeric materials, minerals and crystalline phases.

The laboratory tools of X-ray fluorescence (XRF) and X-ray diffraction (XRD) joined the instrumentation during the 20th century, as did the field tools of colorimetric spot tests and hand-held XRF devices. These tools work well for relatively common minerals and relatively large samples [1]. The examination of particles found in the environment, or at crime scenes, minerals, as well as other materials formed the basis of our search for better instrumentation to analyze these materials for various applications and disciplines.

Over the past 20 years it has become possible to analyze crushed mineral material, as well as GSR particles, using automated SEM and EDS systems. These processes are suitable for large-scale automatic analysis applications for both mineral liberation analysis (MLA) and GSR analysis [1]. However these techniques do not work well with minerals with low concentrations of light elements such as Li, Be and B, any water of hydration (.nH2O), or with the newly available HMF cartridge primers. Consequently, a new, rapid method of materials analysis, as well as mineral and inorganic particle classification, has been explored using a combination (hybrid system or toolbox) of welltechniques such as optical microscopy, SEM, EDS, Raman Cathodoluminescence (CL) and Fluorescence [1,2]. The SEM (including backscattered electrons) and optical systems allow the user to find particles of interest, the EDS to determine detectable elements, and the Raman to distinguish between polymorphs to ratify non-detectable elements. GSR of particles and/or Rapid X-Ray mapping (XRM) of materials and particles allows for mapping of chemical/elemental information, and hence chemical phase mapping (CPM) [3]. All of these detectors have been mounted on the SEM. Figure 1 shows an image of the combined SEM, EDS, GSR, XRM, Raman and CL system that have been built on a FEI Quanta SEM, as well as the incorporated optical microscopes. Figure 2 shows a schematic of the constructed hybrid system.

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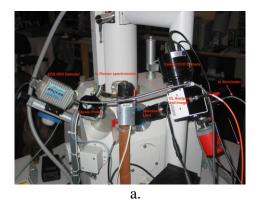
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Properties such as colour, density, light, fluorescence and crystal morphology are among the experienced mineralogist's tools and are required for many other disciplines (materials, science and engineering). For this reason, an optical microscope is an integral part of the system (not only to navigate, but to allow color, fluorescence and morphology to be seen). The talk will discuss the development and incorporation of new instrumentation on a traditional large scale SEM and Phenom desktop SEM that will benefit and assist analysis for many applications, and will cover the next generation of instrumentation required for those applications.

References

- [1] K. Mason, "A Forensic Mineralogy Toolbox the next generation of instrumentation for forensic applications", International Microscopy Congress IMC 2014, Prague.
- [2] R. Wuhrer and K. Mason, "Introduction to Gunshot residue Analysis and Recent Advances", Book of Tutorials and Abstracts, Electron Probe Microanalysis of Materials Today, EMAS, 2016, p241.
- [3] R. Wuhrer and K. Moran, "Quantitative X-ray mapping, scatter diagrams and the generation of correction maps to obtain more information about your material", IOP Conference Series: Materials Science and Engineering Volume 55, Issue 1, 2014.



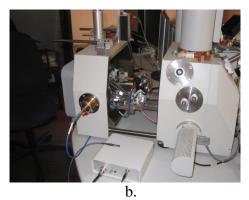


Figure 1. a) A FEI Quanta 600 SEM with EDS, GSR, XRM, CL, optical and Raman system incorporated and b) showing the optical fiber lead through, optical camera and CL on the stage.

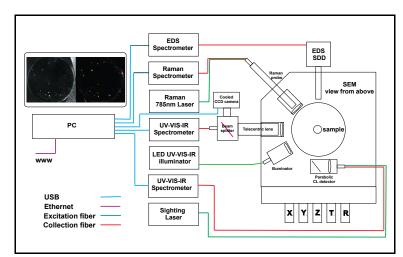


Figure 2. A schematic diagram of the hybrid system, "Next generation of instrumentation or a Forensic-Materials-Mineralogical Toolbox", developed by Eastern Analytical [2].