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X-ray Mapping and Particle Searching with a Benchtop SEM

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The traditional SEM systems used for either x-ray mapping (XRM), gunshot residue (GSR) analysis and general particle searching (PSA) are usually expensive instruments that require large laboratory areas, involve extensive training of the users, and are to some extent difficult to operate. Over the last couple years, there has been a growing market for benchtop SEMs (also called desktop SEMs) mainly due to the requirements and demands of users. Benchtop SEM's are becoming more popular due to: (1) ease of operation; (2) minimal laboratory space requirements; (3) portable and easy to set up; and (4) affordability with less ongoing maintenance cost.

Initially benchtop SEM systems were designed to provide improved imaging capabilities over the conventional optical microscope. Over the last couple years, there have been some significant improvements in benchtop SEM design and capabilities with the addition of microanalysis systems (Figure 1). More recently, an automated gunshot residue system (GSR) system has been interfaced between the Phenom SEM (with automated stage) and a silicon drift detector (SDD) [1]. The system is also being extensively utilized for particle searching and x-ray mapping (XRM).

The Phenom XL Desktop SEM is placed on a standard lab desk. Anti-vibration facilities are not needed, and it only requires a standard power outlet. The benchtop instrument can be placed in practically any lab without additional requirements. The Phenom XL is equipped with a CeB6 source that enables extremely stable operation and has a typical operational life time of >1500 hours, which is ideal from a usability, serviceability and up-time perspective and aids in detection of smaller GSR particles due to the brighter source and higher resolution. It does not require any special facilities such as compressed air, chillers, etc., and has energy usage of maximum 300 Watts. Moreover, the instrument is easy to set up and transport, and can be relocated without difficulty (XL weight is 80 kg).

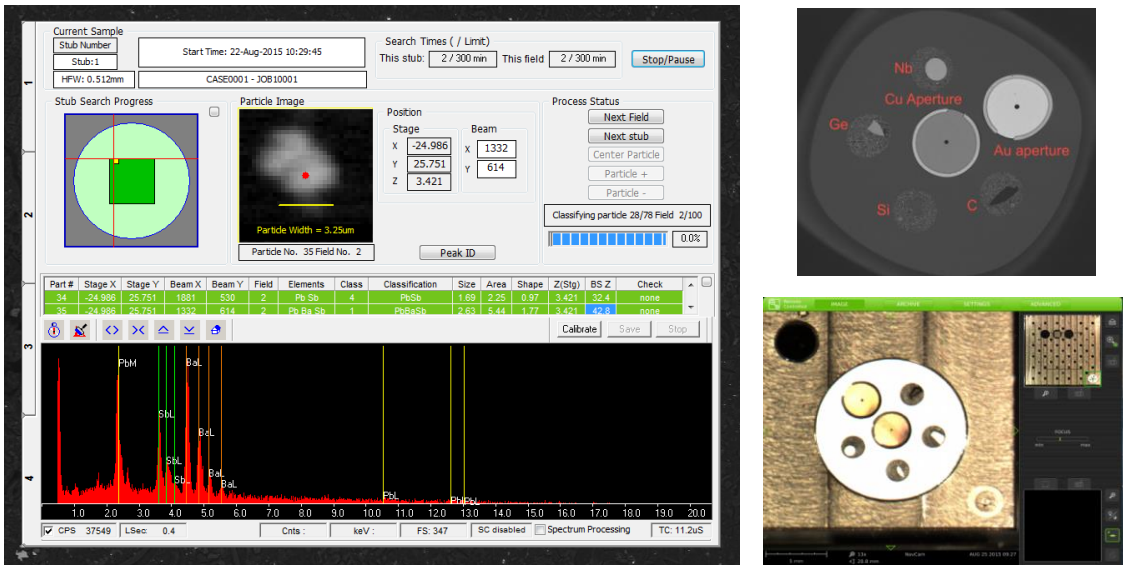
A Plano test sample (SPS-5P-2A) was tested with a hit rate of >98%. Particles as small as 0.5 µm were easily and reliably detected. The ASTM E1588-10E1 GSR standard was followed throughout the complete GSR analysis process, Figure 2a. To calibrate the system a standard is used. The standard contains Nb, Ge, Si ,C , Au aperture and a Cu aperture mounted in a 12.7mm pin stub (Figure 2b). This presentation will show GSR results collected as well as the x-ray mapping results of the GSR particles (Figure 3). The GSR system was not only used for finding traditional Pb, Ba and Sb particles, but also for the detection of elements in Pb-free primers (such as Ti and Zn). The automated benchtop SEM-GSR system was also used as a general particle searching package by simply changing the elemental classification system that exists in the GSR software.

References:

[1] R. Wuhrer and K. Mason, "Gunshot Residue Analysis and Particle Searching with a Benchtop SEM", ACMM24 Melbourne Austral Feb 2016.

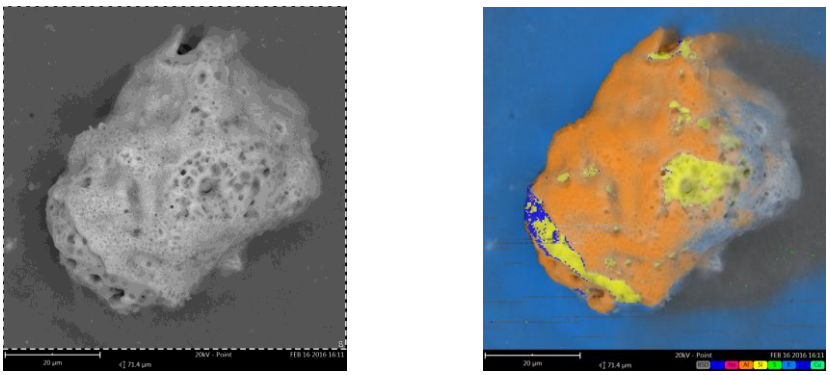
a. b.

Figure 1. a) Typical benchtop SEM system set-up (Phenom XL with monitor with the microscope control and monitor with four-step GSR interface and EDS/XRM software and b) the multi-sample holder that can handle large specimens.



a. b.

Figure 2. a) GSR Interface being utilized on the benchtop SEM. The GSR software is also used as a general particle searching package by simply changing the elemental classification system and b) Calibration standard used for GSR calibration and also calibration of SDD.



a. b.

Figure 3. a) BSE image of GSR particle (50um CBC Clean range particle) and b) x-ray map sowing O, Na, Al, Si, K, Ca and Cu key components in the CBC ammunition.

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