Analysis of Gun Shot Residue (GSR) with Energy Dispersive Spectroscopy

Introduction

Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM/EDS) is an industry accepted technology for the recognition and analysis of characteristic Gun Shot Residue (GSR) primer particles due to its speed and ability to analyze individual particles. Systems, which cover a large amount of analysis area using integrated SEM/EDS controls, are designed for automated analysis of GSR particles with minimal user input. Hardware and software components are used to detect particles of interest and then further sort them into classes of interest that are characteristic of GSR. When a GSR system is designed to maximize the data collection efficiency, more sampling area can be covered in less time, which leads to increased productivity and a greater likelihood of finding even the smallest GSR particles in a sample.

Analytical Requirements

Two important aspects of a run are the image detection of each GSR particle, and the collection of spectral data from each particle from an entire set of stubs in the sample. It is of paramount importance to accurately and efficiently capture important parameters and details such as stage location, particle morphology, chemistry and classification matching. Many GSR samples have the potential of containing hundreds or even thousands of particles of interest, therefore even small decreases in collection time per particle, while still maintaining classification accuracy, will benefit the total throughput and productivity of a GSR analysis run. Furthermore, advanced follow-up analyses of classified GSR particles substantiate the match and increases the overall confidence in results.

EDS Silicon Drift Detector (SDD) Collection Parameters and Optimization

There are several factors that are important in the total EDS data collection process including:

1) Input count rate
2) Output count rate
3) Collection time or scan speed

Higher count rates and faster collection speeds will contribute to an improved speed of data collection. It is necessary to consider each aspect when describing the detector's collection speed for the analysis.

1) Input count rate (ICR) is simply the number of X-rays that hit the detector, as a value of counts per second (CPS). This factor is a function of the sample, SEM and detector geometry. (i.e. some low atomic number samples do not produce a high number of CPS, while metallic particles produce a high number of CPS). There are two main variables for the SEM operation, kV and beam current. The generally accepted practices for GSR analysis use a beam voltage of 20 – 25 kV, which is very well suited to the production of high CPS. The beam current of the SEM can be easily adjusted to achieve high ICR while still maintaining the required quality to detect and image even the smallest required submicron GSR particles. Finally, detector geometry considerations will impact the input count rate, but generally any size detector will readily achieve the highest needed count rates based on the sample and SEM conditions. The window material is also a factor, and while windowless detectors are not suitable for GSR due to contamination issues[1], advancements in thinner window materials allow for increased input count rate, while still assuring cleanliness and SDD protection.

2) Output count rate is a very important aspect in the EDS data collection process. It is a function of how efficiently the detector's electronics convert the raw input count rates into the useable X-ray counts in a spectrum. Only with a fast processing time, generally <1 μs, can high input count rates be converted with low dead time into usable counts for data characterization. It is also important that the detector maintains a high quality resolution at the faster processing time. Instruments having a resolution of approximately 130 eV for the Mn Kα peak will more clearly resolve close peaks, such as the Ca Kα and Sb Lα1 peaks.[1]

3) Spectral collection time becomes a secondary function when the SDD can be optimized for count rates. Collection times as fast as 1 second or better result in a quality GSR classification and analysis with a detector that offers high efficiency at the same time as quality resolution. For example, an SDD, which achieves over 100K CPS with lower than 30% dead time, while still maintaining a resolution of 130 eV or better may be used. This will ensure an analytical method where thousands of particles from multiple samples and stubs can be analyzed in fewer hours than the traditional overnight runs. Shorter run times give the user the opportunity to manually verify the results several times per day, rather than just once as with an overnight run.

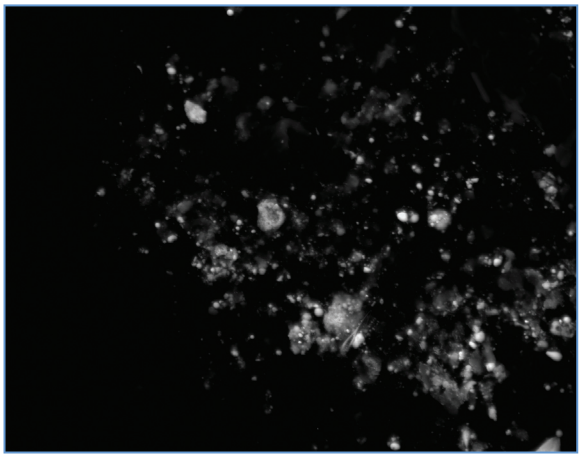

Figure 1 *shows a 500X magnification of a sample area of a GSR stub. At 2048 x 1600 pixel resolution spectra from particles as small as 0.25 µm can be collected.*

Particle Analyzed/Found 308/308

Class	Class name	Analyzed	Total	Part/sq.mm	Area Fraction (%)
1 3	Comp	94	94	1835.94	0.9888
3 2	Comp Sb Pb	8	8	156.25	0.033
4 2	Comp Ba Pb	153	153	2988.28	2.3
6 1	Comp Ba Pb	33	33	644.53	0.307
7 1	Comp Pb	15	15	292.97	0.062
14	Fe	2	2	39.06	0.253
	Unclassified	3	3	58.59	0.01

Figure 2 *shows the analyzed results of the 308 particles found in this single field of view. At one second collection time, this data was collected in about 5 minutes.*

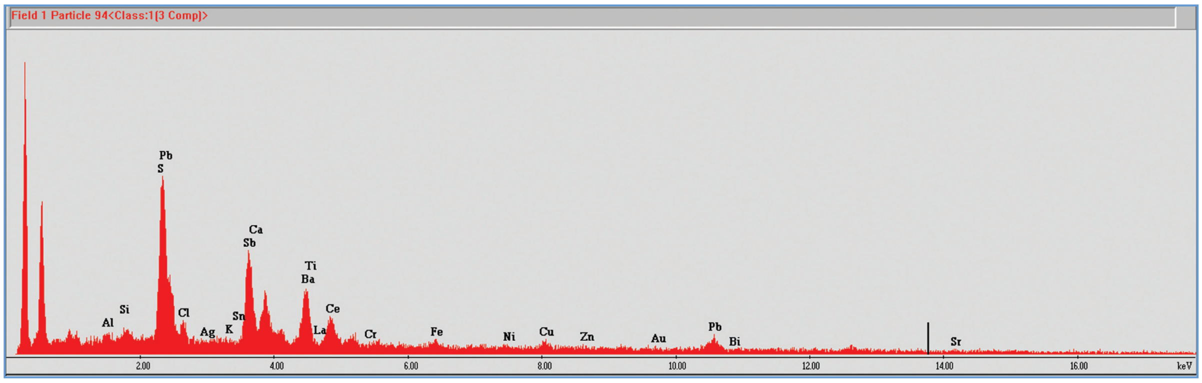

Figure 3 *shows a spectrum collected in 1 second. Octane Elite yielded high input count rate (117 KCPS) and low dead time (26%) with a high output count rate of 86 KCPS with a 130 eV quality resolution, so there is high confidence in the statistical accuracy of the classified 3 component GSR particle.*

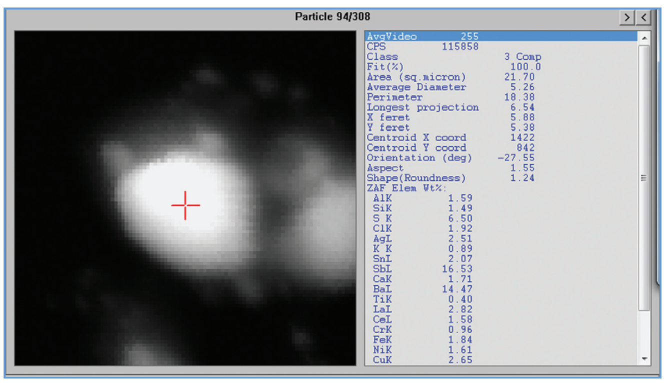

Figure 4 *shows a zoomed in area of the 3 component particle that corresponds with the spectrum in Figure 3.*

EDAX Octane Elite Series

The EDAX Octane Elite SDD Series has several notable design characteristics which make it well suited to the GSR application. The most remarkable advantage is the revolutionary Silicon Nitride window material which is fabricated to be nearly 10 times thinner than traditional polymer windows, while still keeping the detector integrity. This will yield higher input count rates to the detector. Furthermore, Octane Elite electronics are among the fastest on the market with processing times below 0.2 µs, which ensures the reliable and efficient conversion of input count rates into output count rates. Also delivering resolution stability, the Octane Elite electronics will ensure the detector resolution quality to separate close peaks and provide accurate GSR classification solutions. In conclusion, the latest design technologies of the Octane Elite detectors take GSR analysis to a new level.

[1] *Guide for Primer Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray, Spectrometry 11/29/11.*