

Personal SEM[®] (PSEM) eXpress[™]:

The Game-Changer in Microscopy and Microanalysis



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INTRODUCTION

The advent of new manufacturing processes and close tolerance designs has increased emphasis on quality control and improved evaluation methods. Traditionally, quality departments have invested in various methods for inspections of products. These have ranged from optical microscopes to high-end scanning electron microscopes (SEM). Optical microscopes offer a low-cost, effective and traditionally robust method for inspections of materials. They require minimal training and do not require much maintenance. Unfortunately optical microscopes are limited in their magnification and resolving power capabilities: <1000 X for even the best microscopes. Consequently most companies outfit their laboratories with an electron microscope capable of reaching upwards of 50,000 X. These high-end microscopes offer excellent imaging and resolving power, while also allowing their operators to identify materials based on characteristic emissions from the material. This is accomplished through Energy Dispersive Spectroscopy (EDS), Wavelength Dispersive Spectroscopy (WDS) and X-ray Fluorescence Spectroscopy (XRF). As with most scientific equipment, the cost goes up exponentially as more functionality and power are added. What if these worlds – optical and electron microscopy – came together to produce a fully functional microanalysis tool that combined the high-end features of electron microscopes with the affordable cost of optical microscopes?

ASPEX has developed the Personal SEM[®] (or PSEM) eXpress to accomplish this feat. The PSEM eXpress[™] utilizes a proprietary integrated electron beam scanner, X-ray analyzer (EDS) and software to provide a seamless high-performance electron microscope that is small enough to fit on a bench-top (Figure 1&2). In addition to standard electron microscope functionality, the PSEM eXpress offers users the advantage of a large stage and fully automated analysis modes, which is unique to the family of products offered by ASPEX Corporation. The contents of this article will provide an outline of the full capabilities of the PSEM eXpress.



Figure 1. The PSEM eXpress, single standalone, bench-top electron microscope.

INDUSTRIAL PACKAGING and EASE-OF-USE

What would it take to analyze >30,000 particles/hour 24/7 using an SEM installed:

- At a flight line?
- In the melt-shop of a steel mill?
- At a mixing station in a pharmaceutical processing plant?
- On an automotive assembly line?
- On a battlefield or aircraft carrier?

...and don't forget the technicians required to operate it.

Ultimately, this requires a fundamental shift in the principal design decisions of standard scientific equipment often deployed at research-level laboratories. The primary function for most currently available SEM/EDX systems is to provide high-quality imaging and characterization in controlled laboratory settings. However, the environment experienced by routine inspection tools, like HPLCs and optical microscopes, and the objectives of their operators in quality-control labs are quite different. Instead of spending time capturing the best quality image, the operators now wish to have higher throughput and spend less time mining the data acquired. Using these parameters as guidance for design decisions, a next generation of electron microscope has been developed.

Specifically, ASPEX has designed the PSEM eXpress to have improved mobility, environmental shielding, simplified operation, and unmatched reliability in the field. The PSEM eXpress is equipped with a reduced-weight, low center of gravity, internal hardware structure, requires one single 110 or 240 V outlet, and draws less than 10 Amps during operation. In addition, the electron optics table has built-in vibration dampening to accommodate non-traditional imaging environments. Finally, the systems comes with a large chamber, allowing for multiple samples to be loaded into the instrument at the sample time, as well as one of the largest motorized stage travels available in the electron microscope community.

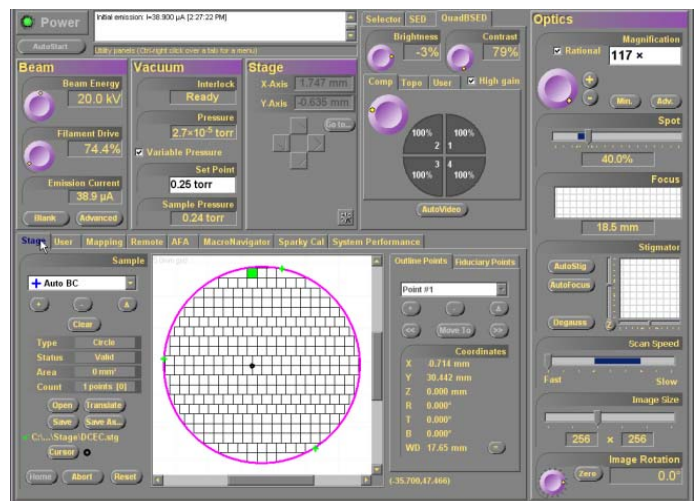


Figure 2. ASPEX Perception™ Console.

Table 1: Specifications for the PSEM eXpress.

System Features	System Components	PSEM eXpress
Imaging	Ultimate Resolution	25 nm
	Accelerating Voltage	20 - 2 kV
	Variable Pressure	0.15 & 0.3 Torr set points
	Solid State BSED	Standard
	Topographical Mode	Standard
Elemental Analysis	X-ray Analysis Capable	Integrated Liq. Ni-Free
Sample Handling	Specimen Stage Travel	80 x 100 XY mm
	Stage Movement	Motorized
	Maximum Specimen Size: (Diameter x Thickness)	150 mm (D) x 35 mm (T)
Software	Single Interface for Elemental Analysis	Standard
	Automated Analysis	Optional
	Advanced Automation	Optional
	Database Products	Optional
	Simple Quant Analysis	Optional
	X-ray Mapping	Optional
Hardware	Sound Dampening for Silent Operation	Standard
	Environmental Protection of Electron Optics	Standard

An important functionality for any scientific equipment is its user-friendliness. One might think that by reducing the size of an SEM to fit on a bench-top one would lose the traditional flexibility of imaging and control in the software. However, functionality and usability must be balanced to meet the needs of operators. ASPEX Corporation's Perception™ Software console (Figure 2) is known industry-wide as the most user-friendly. A full range of automated beam control is provided, including automated electronic beam aligners, one-click imaging, full stage control access and auto-focusing. All the functionality of an SEM/EDS instrument is maintained but presented in a very easy-to-use manner, making the PSEM eXpress ideal for companies interested in getting more out of their optical microscope. In addition, backlogs on SEM instruments can be transitioned to this platform, which offers very similar functionality and specifications to conventional SEM systems.

IMAGING

The PSEM eXpress offers a wide range of imaging functionality. Table 1 lists its full specifications. The PSEM eXpress comes equipped with a Backscatter Electron Detector (BSED) for all imaging needs. This detector gets its contrast from the average atomic number differences in the sample with an ultimate resolution of 25 nm. Typical SEM resolutions range from 3-7 nm for some of the best equipment on the market, so by comparison, the eXpress can support well over the majority of work necessary of an SEM. It also has the ability to operate in Topographical Mode as well, which can be used to identify surface-related defects and failure-analysis work. In addition, depending on the material being characterized, a variable accelerating voltage offers the operators an easy solution for characterizing and imaging sensitive samples. More often than not, higher KeV will not only damage some samples but also inhibit their ability to be imaged properly (Figure 3). In addition, organic and non-conductive samples will often produce charging in an SEM chamber. Charging occurs due to the build-up of

electrons from the source on the sample due to an inability to reach 'ground'. Consequently, images appear with bright streaks and shifting occurs in the image. To alleviate this scenario, the PSEM eXpress, like most standard state-of-art SEMs, has the ability to switch into a Variable Pressure (VP) mode (Figures 3 & 4). In VP-mode, the instrument automatically allows air in the sample chamber while maintaining a high vacuum in the electron source area of the instrument. The air molecules provide an avenue for the electrons on the surface of the sample to be dissipated rapidly and allows for normal imaging to be restored.

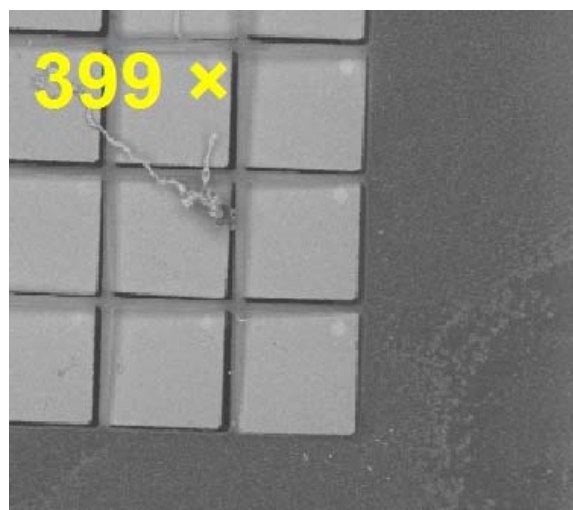


Figure 3. Organic film less than 1 um in thickness. Image acquired at 10 KeV and 0.3 Torr in the sample chamber. Noticeable unadhesion is observed around the perimeter of the square, raised features. This film was unobservable at higher KeV settings and High Vacuum settings.

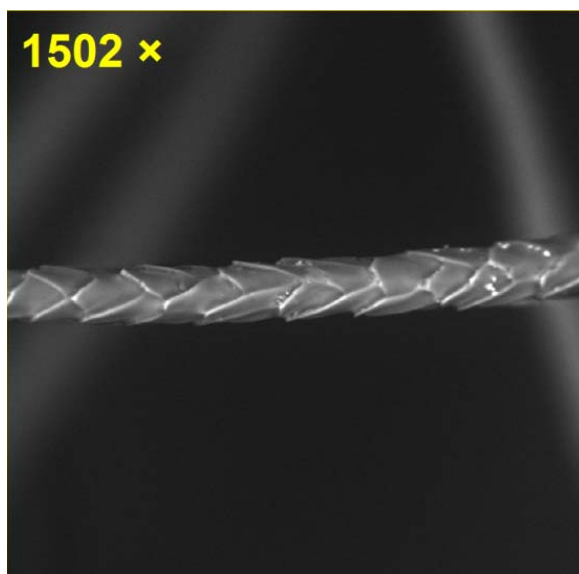
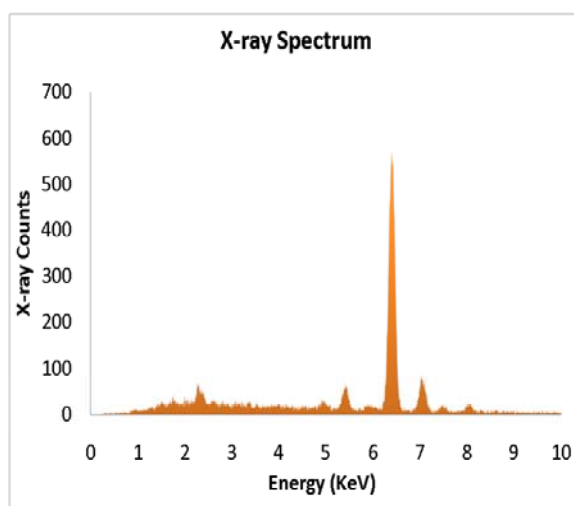
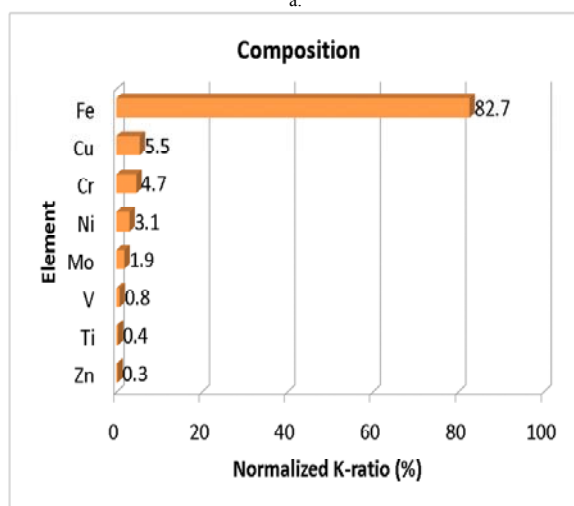


Figure 4. Cashmere fiber imaged using 0.3 Torr Variable Pressure and 15 KeV.



a.



b.

Figure 5. X-ray Spectrum from the EDS Detector on the PSEM eXpress (a), Elemental Composition of the sample (Stainless Steel) that was analyzed (b).

X-RAY ANALYSIS

One bi-product of electron beam interactions is the emission of X-rays from the sample. The energy of the X-ray corresponds directly to the identification of the elements present. By comparison to optical microscopy, EDS X-ray analysis is a concrete distinction between the technologies. While optical microscopes offer its user the ability to image materials, the characterization capabilities is often limited to organic materials using either fluorescence or inferred spectroscopy. The PSEM eXpress is equipped with an integrated EDS detector which offers the ability to characterize material using their X-ray emissions. Specifically, this allows the operator to identify the elements present in the material and quantify how much of each element makes up the material’s composition (Figure 5). In addition, these detectors are maintenance-free and offer a liquid nitrogen-free cooling device.

An additional functionality of the instrument is to perform routine, high-throughput X-ray analysis mapping. X-Ray mapping (Figure 6) is an imaging technique used to illustrate the distribution of elements in a sample. Each two-dimensional map represents a single element and the color or grayscale variations on the map represent differences in the concentration of the elements from point to point. Composite maps created by overlaying the images are commonly used for the characterization of region patterns in materials such as: glass, mineral,

and concrete. In addition, they can be used to study inclusions in steel samples and to monitor impurities in pharmaceutical tablets. By incorporating the use of silicon drift detectors (SDD) in the PSEM eXpress, the acquisition time of X-ray maps has been substantially decreased while maintaining the high resolution.

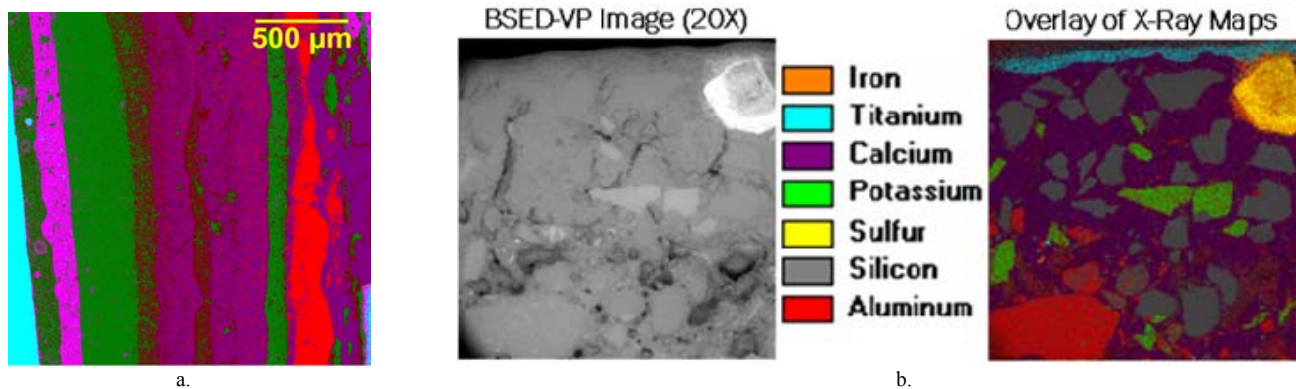


Figure 6. X-ray map of a Paint Chip (a) and Concrete cross-section (b). Each color represents a different element, allowing for easy distinctions between phases.

AUTOMATED FEATURE ANALYSIS:

Overcoming the Limitations of Conventional SEM Equipment

The need for more routine, advanced contamination control and auditing analyzers is becoming significantly important in most pharmaceutical, automotive, and industrial manufacturers. The PSEM eXpress analyzer utilizes a unique integrated algorithm with the electron beam scanner and X-ray analyzer to provide the size, shape, and elemental composition of particles in a sample. Once fully implemented, the new PSEM eXpress analyzer exhibits significant improvements in reliability, environment insensitivity, particle sensitivity, reproducibility, and throughput (>7,000 particles analyzed in an hour; size, shape, elemental composition).

The average atomic number contrast is defined using the BSED so that the analyzer is able to determine which part of the sample are particles and which part is background (Figure 7). The most unique feature of these analyzers is not how they define where the particles are, but how they determine the location, size, and shape of the features of interest. One way to automate these inspection processes is to use a frame-based analysis. In this mode of implementation, a conventional SEM is used very much like a camera. This method is very slow and inaccurate for automated feature detection. By comparison, ASPEX's PSEM eXpress dynamically scans the sample, which offers a far superior means to determine the location and speciation of particles (See Figure 8). Rather than capturing a high-resolution image of the field, the systems instead move the beam across the field in an array of fairly coarse steps. At each point, the brightness of the BSED, or SED, signal is noted. If the signal is bright enough to indicate that a particle is present at that position, then the software initiates a particle-sizing sequence. There are several algorithms that can be used for this purpose, but for simple particle shapes, the "rotating chord" algorithm (RCA) is both accurate and exceptionally fast. Once the "coarse" scanning (indicated by the dots) identifies a particle, the center is identified and Chords are drawn with the beam on the particle to define the particles Size and Shape. The major reason for an improvement in speed is because the instrument only spends time collecting detailed data where particles are known to be present, rather than spending time capturing and analyzing vast numbers of "empty" pixels. Since there is almost always much more empty space on the specimen than space occupied by particles, the result is a large speed advantage.

Overall Advantage

- No image processing required
- Dynamically scans the sample for particles
- Utilizes backscatter electron contrast mechanism as oppose to light microscopy
- Provides addition flexibility for particle shape analysis of "Complex" irregularly shaped features

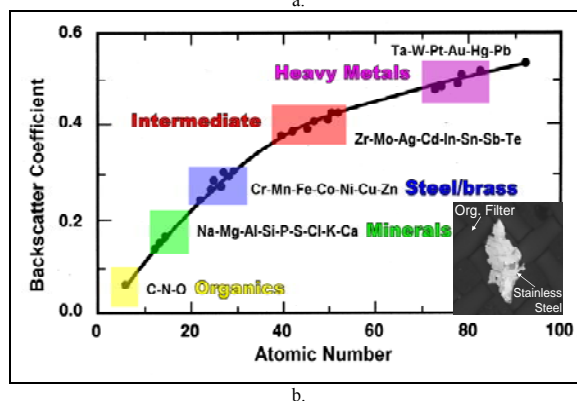
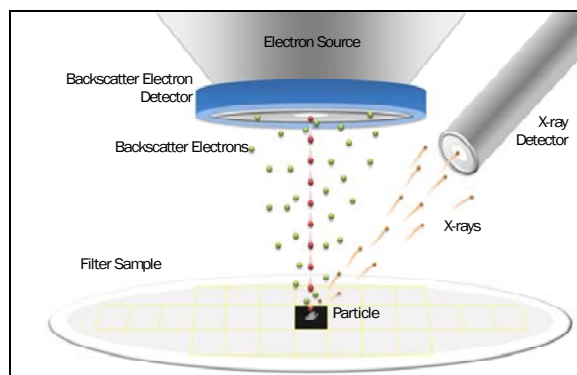


Figure 7. Schematic of how the instrument operates (a), Backscatter electron coefficient plot (b).

REPORTING DATA

One unique feature of Aspek Automation is our ability to provide seamless unattended reporting. Reporting using standard SEM/EDS technology and software can be time consuming and require considerable expertise in the technology (Figure 9). Once prepared, samples are manually or automatically scanned for contamination and a data array can be created and used for spreadsheet based reporting. Further review and historical referencing is required by an analyst to approved data integrity and process control. This limits the effective throughput of any instrument. Through the use of a Data Management Solution which was developed using database archiving and new software algorithms, a fully automated reporting system can be employed to reduce data analysis times from hours to minutes. In addition, the infrastructure requires minimal user interfacing once a sample is being analyzed. Ultimately, the PSEM eXpress offers its operators a cost effectively way to increase their through-put by automating most of their routine inspections.

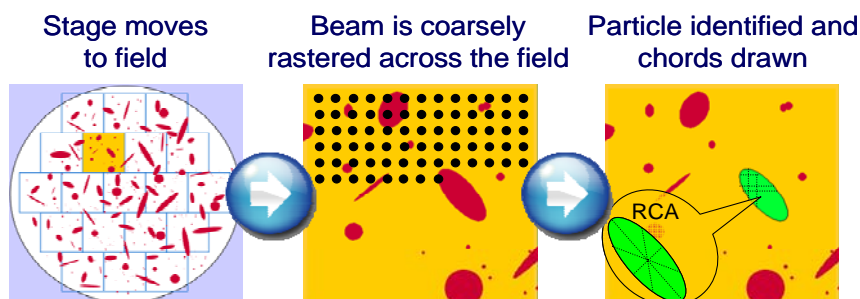


Figure 8. Fundamental overview of how automated feature analysis (AFA) works on the PSEM eXpress.

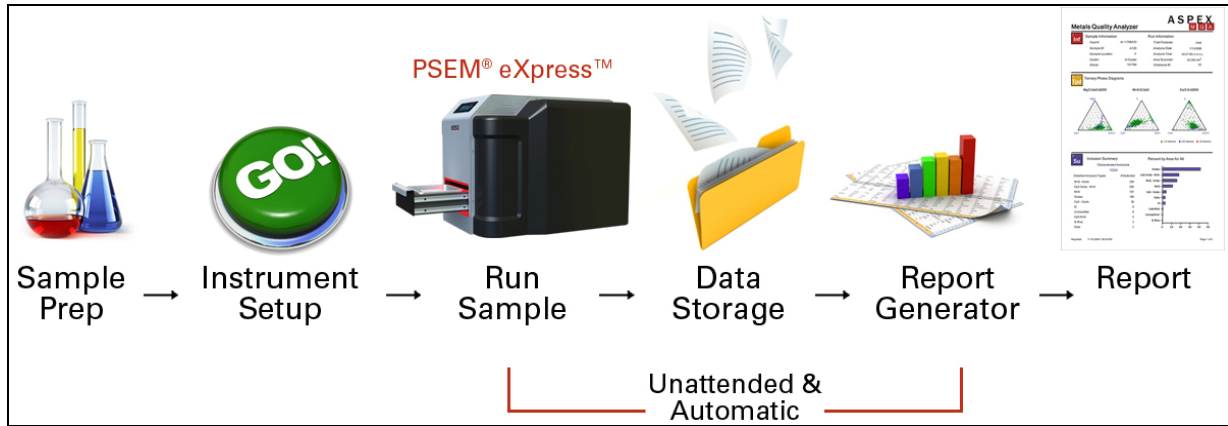


Figure 9. Fully Automated Reporting and Validation.

Predicting Imminent Failures

In order to better ascertain the quality of products and predicting imminent failures, the reliability of the measurement and several benchmarking measures must be determined. Typically these are also used for trending purposes to determine when the process is ‘in control.’ The repeatability of any measurement system is critical to its success in industrial applications. To this end, the PSEM eXpress was evaluated for its ability to characterize the total area of particles for a routine inspection of contamination in an engine manufacturer (Figure 10). The overall percent relative standard deviation (%RSD) of all engine total areas of particles was 3.5 %RSD. Given the instrument’s routine implementation in quality control environments, this affords a solid foundation to determine and monitor changes in a process. In order to understand these changes, size distribution and ternary diagram charts can often be used (Figures 11 & 12). Size distribution shifts can be used

to indicate additional fatigue on a product in use. Based on the result shown in Figure 11, the operator can easily determine the difference between the baseline sample and two characteristic failure samples. In Figure 12, distributions are characterized using elemental ternary diagrams. The plots act a descriptive picture for the operator. Each dot is representative of a single particle that was detected. Based on the location in the ternary system, the populations of different stainless steel being generated as wear debris in these engines can be differentiated. In addition, the size distributions of the particles are illustrated using the color of the dots. Finally, this information can be stored and accessed routinely to get trending information on the historical performance of your process (Figure 13). Overall red flags & warning signals can also be implemented based on the historical data. Upon analysis, engineers can then conduct the necessary investigations into the process to improve future products and obtain the source of the problem.

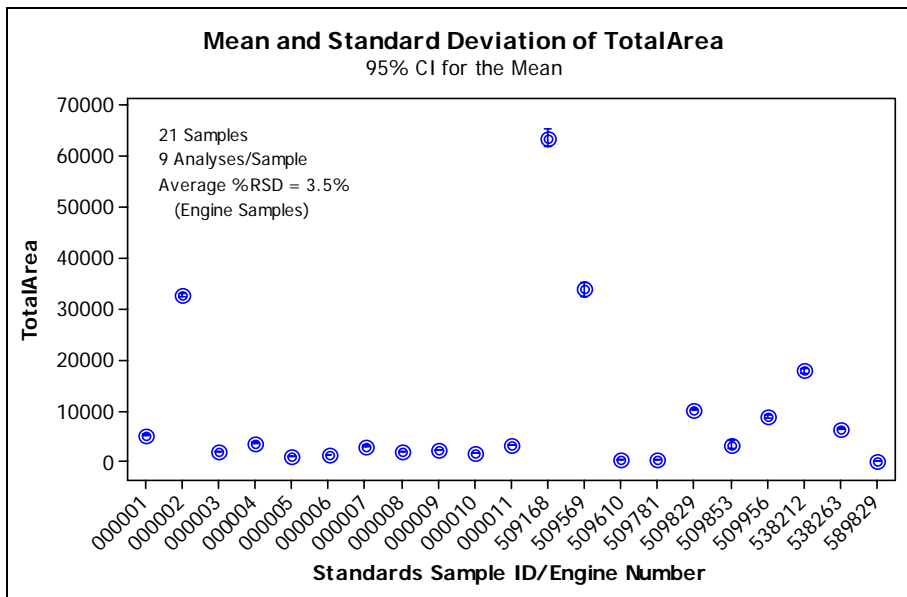


Figure 10. Repeatability for Total Area of Particles.

CONCLUSIONS

The ASPEX PSEM® eXpress package was developed to alleviate the need for highly trained SEM experts and data analysis personnel. In addition, it can be deployed where optical microscopy fails to provide enough functionality and magnification to characterize materials. Overall productivity can improve due to increased throughput and knowledge of the samples. Utilizing new, advanced quality control algorithms, engineers can turn to the PSEM eXpress to easily track

foreign particle material and how it affects their processes. To accomplish these tasks, the PSEM eXpress uses database architectures to take large amounts of information and perform automated data analysis to generate an easily interpreted output. In addition, particles types, quantities and failure rates were established for trending purposes and process improvement initiatives. By comparison, PSEM eXpress offers superior usability, reproducibility and performance...a true game-changer.

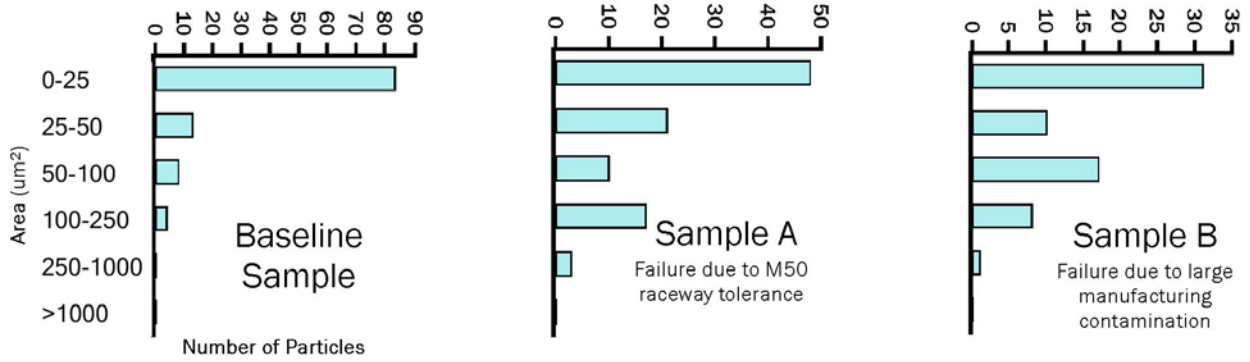


Figure 11. Size Distributions Plots comparing changes in a process.

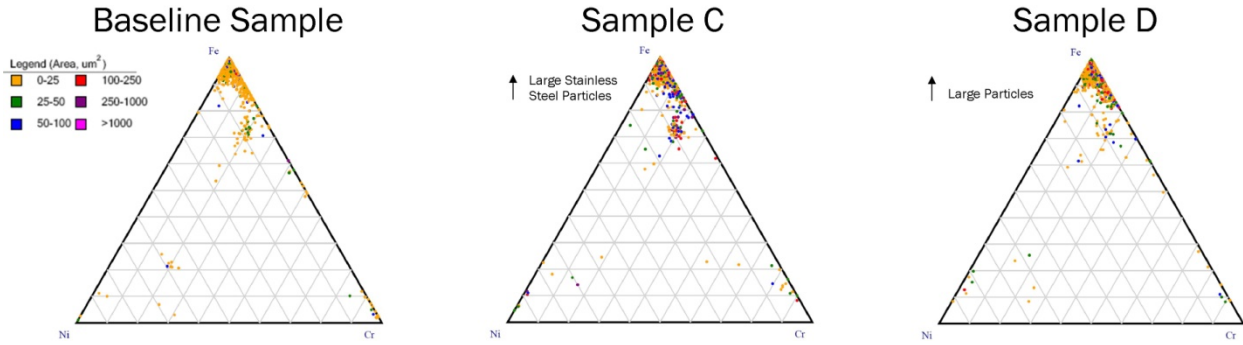


Figure 12. Ternary diagrams depicting stainless steel particles from three different engines.

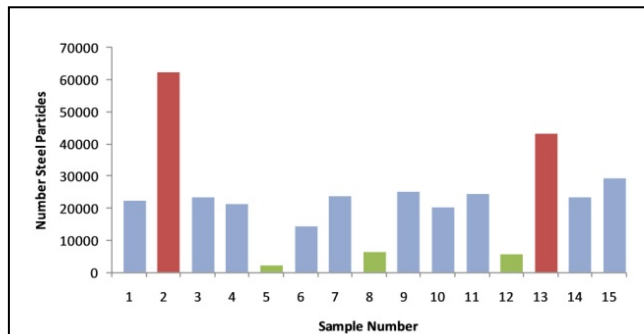


Figure 13. Trending characterization of process where the number of steel particles plotted versus sample number (time). Red = out-of-control process, Blue = in-control process, Green = out-of-control process but not detrimentally.