Solve Common Industrial Problems with Automated SEM Analyses

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Contaminant analyses are vital to industrial manufacturers because they help correct or identify the source of a known contamination problem, thereby improving the quality of a product.

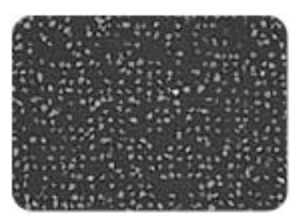
This is one of many SEMs specifically designed for the rapid characterization of particles. Source: McCrone Associates Inc.

The newest generation of scanning electron microscopes (SEM) utilizing energy dispersive X-ray spectrometers (EDS) include computer automation that allows for rapid elemental analyses of small particles or inclusions. Automated analysis yields chemical and morphological information on large numbers of particles or inclusions, which can be used to identify specific populations of materials present within a sample. Automated SEM analysis eliminates operator subjectivity inherent in time-intensive manual SEM or optical characterization techniques.

With automated SEM analysis, more particles can be analyzed per unit time, which allows for efficient and more complete sample characterization. This also increases the probability of finding and identifying rare materials in industrial samples. For example, specialized SEM-EDS systems can now analyze and classify between 500 to 1,500 particles or inclusions per hour, and current trends in detector technology promise to further improve this rate. For these reasons, automated SEM analysis is becoming a widely used, fundamental tool for solving industrial problems.

Automated SEM Analysis

Elemental analysis in an SEM, whether manual or automated, involves the interaction of a high-energy electron beam with the sample of interest. The primary electron beam voltage is typically between 5 and 30 kilovolts (kV); the setting is determined by the type of analysis (elemental vs. imaging) and the specific elements or features of interest.



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As the electron beam interacts with the specimen, several different processes occur that create characteristic signals used for imaging and elemental analysis. Of particular interest for automated SEM analysis are backscattered electrons and characteristic X-rays.

important to limit the amount of touching or overlapping particles, as this will often lead to an incorrect or inconclusive identification. Source: McCrone Associates Inc.

Backscattered electrons are high-energy electrons elastically scattered from within the specimen. Higher atomic number (Z) elements tend to backscatter more electrons, and therefore appear brighter—a higher gray level value—than lower Z materials in backscattered electron images (BEI). These contrast differences can be used during an automated elemental analysis to set thresholds that highlight the particles or compositions of interest while ignoring ancillary materials.

For example, when searching dispersions of industrial particulate for metal particles, minerals or organic particles may not be of interest. These particles are primarily composed of lower atomic number elements than metals, and therefore show up as darker particles on a BEI display. Gray-level thresholding can be utilized to ignore these darker particles and concentrate the analysis on particles containing higher atomic number elements.

Characteristic X-rays result from the relaxation of an atom after ionization by the electron beam. The electron beam ejects an electron from one of the inner orbital shells of an atom creating a vacancy. A higher energy electron subsequently replaces the ejected electron, resulting in the emission of a characteristic X-ray photon, which has an energy that corresponds exactly to the difference in the energy between the two orbital levels for the specific atom. As such, the X-ray energy is specific for the atom and therefore a spectrum of X-ray energies can be used to identify the elements present within a sample.

Sample Preparation

Sample preparation is of fundamental importance to achieve the best SEM analysis results. Techniques vary depending on the type of material being analyzed and the focus of the study.

Particulate samples are typically prepared as dispersions onto carbon planchets, carbon sticky tabs or other low atomic number substrates. Contaminant particles can be extracted from liquid samples by passing the sample through membrane filters, yielding a dispersion of particles that can be analyzed directly.

The primary requirements of sample preparation are that the sample must fit in the sample chamber, be vacuum compatible and must not become electrically charged during the analysis. Nonconductive samples may need to be coated with a thin layer of a conductive material such as gold, gold/palladium, platinum, or carbon to prevent the build-up of electric charge on the sample surface during analysis.

Many new SEM systems are equipped to operate in variable pressure or low vacuum mode, where the sample is at a higher pressure than the electron column, thereby reducing charge build-up. These types of instruments allow for a wider variety of samples to be analyzed, and may reduce the need to coat nonconductive samples.

Another important factor for a successful automated analysis is to limit the amount of touching or overlapping particles. Particles that touch or overlap often lead to an incorrect or inconclusive identification of the material during automated analyses.

Applications for Industrial Use

Automated elemental analyses have been used for applications in the pharmaceutical, food, metals, forensic, automotive, airline and environmental industries. The most common use of the technique is for quality control, specifically contaminant analysis.

Undesirable particles, even down to a single micrometer in size, can lead to defective products, product recalls, structural failure of materials and equipment, or costly lawsuits, often leading to extended laboratory or manufacturing delays. Contaminant analyses are thus vital to industrial manufacturers because they not only improve the quality of a product, but also help correct or identify the source of a known contamination problem, saving the company time and ultimately revenue.

In the automotive or airline industry it is known that particles smaller than 10 micrometers in size can result in engine or transmission failure. Many companies have begun to filter hydraulic fluids, transmission and engine oils, and fuels to analyze for metal wear particles, which can signal engine degradation. Knowledge of the composition and sizes of these materials can help manufacturers identify the source and severity of the problem.

The steel industry has used automated elemental techniques to analyze their products for non-metallic inclusions, several of which are known to lead to brittleness, cracking or failure of the steel. Characterization of the composition of these inclusions, as well as their size, can help steel companies improve their processing, ultimately leading to a higher quality product.

Automated SEM analysis provides a rapid, cost-effective means to characterize and classify large numbers of particles or inclusions. Automated analysis of particle populations is increasingly being used to monitor industrial products for foreign contaminants, search for characteristic particles in background materials and objectively classify samples for quality control studies. **Q**

Tech Tips

- With automated SEM analysis, more particles can be analyzed per unit time, which allows for efficient and more complete sample characterization.

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