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Fast, easy-to-interpret microstructural and elemental analysis with scanning electron microscopy and energy dispersive X-ray spectroscopy

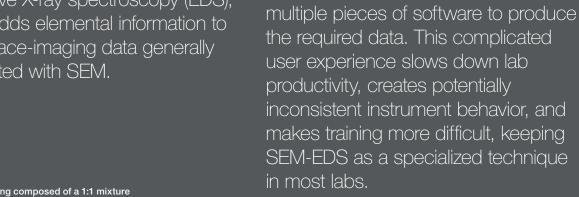


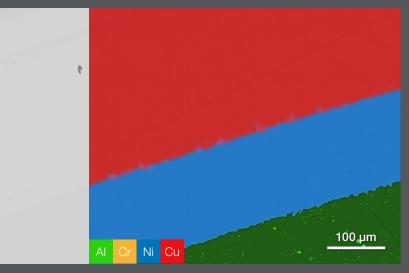
Combining EDS and SEM typically

involves complex workflows that use

Introduction

As the complexity of industrial processes increases so does the need for more rigorous, higher-quality analysis to ensure that products are meeting all standards of quality and reliability. Scanning electron microscopy (SEM) has become a common tool in modern research and development as well as failure analysis and troubleshooting laboratories, as it provides detailed (nanometerscale) information about the surface structure of samples. An SEM is often used in conjunction with energydispersive X-ray spectroscopy (EDS), which adds elemental information to the surface-imaging data generally associated with SEM.





SEM image of a chrome-plated aluminum sample, with and without the addition of EDS data. From the SEM image alone, it would be impossible to determine that there are distinct bands of nickel and copper on the sample surface.

In this document we will provide a brief introduction to SEM and EDS analysis and how their joint application supports in-depth material characterization for industrial applications. We will also show examples of how unique Thermo Scientific ChemiSEM Technology integrates these two techniques into a single, fast, easy-to-use package, accessible and valuable to novices and experts alike.

Cover: A coating composed of a 1:1 mixture of copper and silver. The clear differentiation between the two elements would not be possible without the addition of energy-dispersive X-ray spectroscopy data.

How does SEM work?

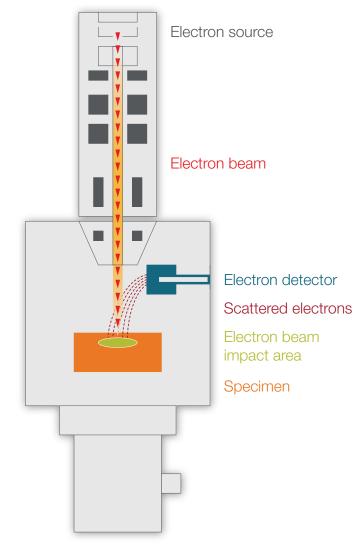
Scanning electron microscopy (SEM) uses a focused beam of electrons to provide information about a material's surface. This is done by raster scanning the sample beneath the electron beam, producing an image that can reveal topography, microstructure, etc. depending on the imaging signal that is used.

SEM has been widely employed as a critical sample-characterization technique across nearly every discipline or industrial application where it is important to know the structure and composition of a material.

Image formation in an SEM is most often the result of either secondary electron (SE) or backscattered electron (BSE) signal. SE images are formed by the inelastic interactions of the incident electron beam and the sample's atoms. Secondary electrons are generated very near the surface of the sample and are therefore utilized for basic imaging where topography and near-surface details are key. Backscattered electrons are formed when the incident electron beam interacts elastically with atoms in the sample; the BSEs are effectively reflected back out of the sample. This interaction results in a signal proportional to the atomic number of the material; high-atomic-number atoms produce bright areas, whereas low-atomic-number atoms result in relatively dark areas.

Taken together, SE and BSE images can reveal information on the microstructure and relative compositional characteristics of a sample. However, while SE and BSE are useful for illustrating what a sample looks like, neither can definitively tell you what the material is made of (i.e. the elemental content of the sample).

Luckily, the interaction of electrons with the material also produces a range of additional signals that carry valuable information for other analytical techniques. For example, X-rays can be generated when the electron beam impacts an atom and displaces its electrons. This X-ray signal can provide quantitative details on the sample's elemental composition with techniques such as energy-dispersive X-ray spectrometry (EDS).

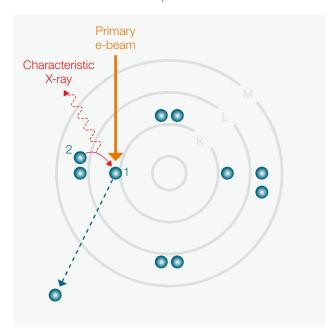


Schematic of a scanning electron microscope. The sample is raster scanned beneath the electron beam, generating an image of the sample's topography.

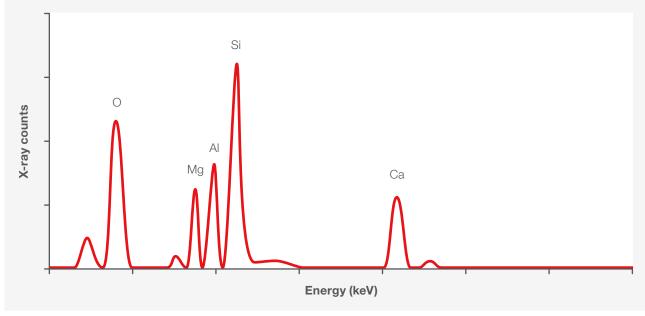
How does EDS work? 1/2

Introduction

The interaction of an electron beam with an atom can result in the loss of a core shell electron; a higher energy electron proceeds to fill this new, positively charged vacancy. The energy difference of this transition is dissipated, in part, by the emission of X-rays, whose energy spectra are unique to each element. By collecting these X-rays for each point in the SEM raster scan, you are able to obtain the spatial distribution of each element present.

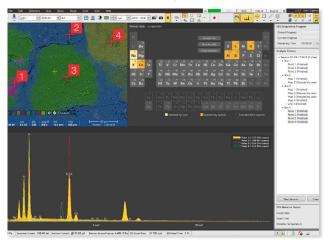


Schematic of the X-ray emission process that makes EDS analysis possible. The emitted X-ray carries crucial information about the identity of the atom.



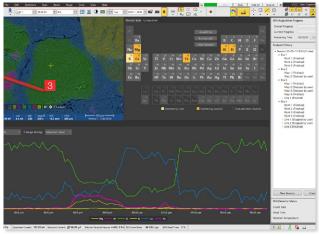
As the name implies, EDS is a spectral technique. The SEM generates X-rays across an energy spectrum up to the beam energy used (e.g. 15 kV). An EDS detector collects these X-rays all at once. The shape and height of the peaks in the spectrum are indicative, in part, to the relative concentration of the elements.

The three basic EDS techniques



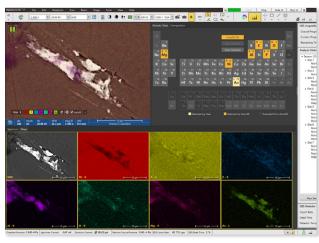
Point scan

Point scan mode (collecting an X-ray spectrum at a single location) is typically used to obtain the specific elemental content of a material in the SEM field of view.



Line scan

Line scan mode (collecting a series of X-ray spectra along a user-specified line) is deployed when compositional variability needs to be assessed.



Mapping

Mapping describes the collection of X-rays at a specified pixel spacing, creating a full, compositional picture of a sample. This mode is used to provide a more complete view of elemental variability within/between phases and features of interest.

While EDS is a quantitative technique it is limited by the concentration of elements that can be observed and by the overall precision of the reported concentrations. For most use cases, however, EDS can provide valuable compositional information much more quickly than other standard techniques. The ability to link compositional information to the microstructural details of a sample is where the use of EDS on a scanning electron microscope really stands apart from other analytical approaches.

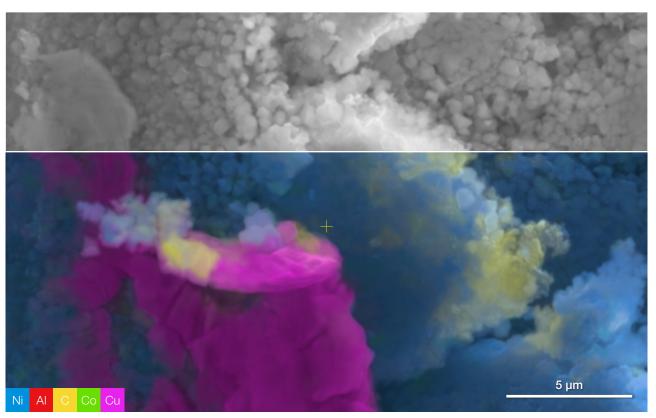
10 um

What are the benefits of combined SEM and EDS analysis?

Combining SEM and EDS analysis has proven to be highly valuable, granting both microstructural and elemental information. Previously indistinguishable materials with similar contrast in an SEM image (a common issue for SE and BSE imaging) can now be differentiated by their elemental identity. This in turn makes the analysis more informative and productive, accelerating research and development or failure analysis.

EDS goes beyond imaging to provide not just relative but quantitative elemental information. Still, SEM-EDS is not as efficient as it could be. A typical workflow involves manual movement of the SEM stage in search of specific features (generally in BSE mode). Users may then perform a quick EDS spot analysis of an interesting grain to see what components are present. Hunting around a sample in this way can be an iterative process and results in wasted time on the tool.

ChemiSEM Technology was developed to make compositional imaging as easy as traditional SEM analysis. Colorized, elemental maps are generated almost instantly, providing valuable feedback and improving the overall approachability of SEM-EDS.



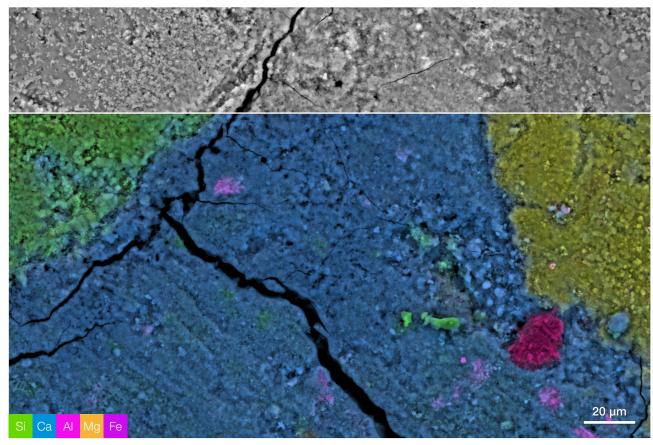
Cathode material imaged with SEM (top) and SEM-EDS (bottom). Without elemental information, the various components of the cathode are virtually indistinguishable in the SEM image. The addition of elemental analysis, however, shows clear differentiation between copper, carbon and nickel in the sample. This allows users to move across the sample with a clearer picture of compositional variability to locate areas of concern and move on to more advanced analysis.

What is ChemiSEM Technology? 1/3

The difficulty of combining SEM and EDS comes from the fact that, traditionally, the data for each method is obtained using separate technology. This means that you will need to work on two separate computers, using separate software, and then integrate the results.

Not only is this time consuming in and of itself, but it also means that standard operating procedure (SOP) development and training are unnecessarily complicated. These factors ultimately lead to more errors in data collection and increased dependence on specialized knowledge when using these tools.

Thermo Scientific ChemiSEM Technology was designed to address these problems by combining EDS and SEM analysis within a single software platform. ChemiSEM Technology is integrated within the main SEM user interface, making it easier to set up, easier to turn on and much faster at obtaining compositional information. Data from both techniques is processed live, allowing you to generate colorized images of your sample complete with microstructural and elemental information.

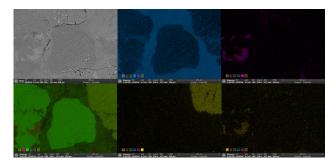


The ChemiSEM difference: A typical cement sample, shown with just BSE (top) and BSE+ChemiSEM (bottom) imaging. The top image, obtained at low magnification (500x), reveals some textural differences, but the atomic-number contrast of the material does not lead to easy identification. After pressing a single button, ChemiSEM Technology turns on a compositional view of the material, enabling easy location of areas of interest and providing an initial view of the phases present. You can now move across the sample with a clearer picture of compositional variability.

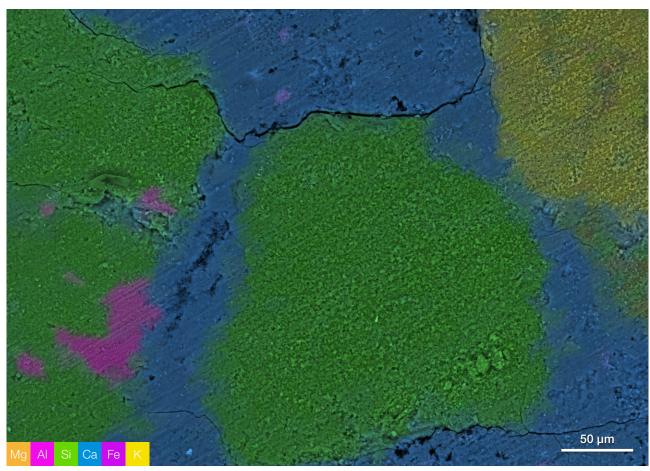
What is ChemiSEM Technology? 2/3

EDS data is visualized with a single button press. Once on, ChemiSEM Technology remains on, providing instant compositional data as you move around the sample, blending standard electron imaging with elemental information.

As a result, you can instantly see the context of compositional data through the backdrop of high-resolution microstructure. The integration of these signals makes your data interpretation much easier for many quick inspection tasks. Direct integration of EDS control within the main microscope user interface also means that it is easier to train new users, and limits characterization mistakes when following SOPs.

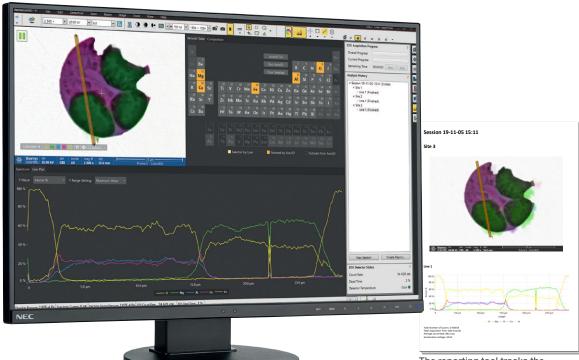


During X-ray acquisition, Traditional EDS splits X-ray information from the key microstructural features a user is attempting to quantify. In addition, it takes much longer for a quality signal to build up, meaning that the EDS data often appears pixelated. For elements with lower concentrations, this results in a pixelated, difficult to interpret image, requiring you to wait for post-processing data integration or to depend on inference and guessing for quick inspection.



ChemiSEM Technology directly integrates X-ray imaging with the main SEM imagery (SE or BSE). ChemiSEM Technology also includes unique rendering capability that produces an accurate, high-fidelity compositional map faster than traditional EDS. This means you can move from discovery to reporting faster than with traditional EDS. Of course, you can still choose to visualize individual elements for targeted analysis.

What is ChemiSEM Technology? 3/3



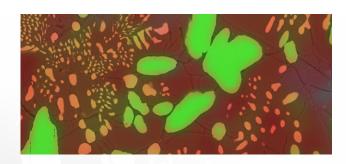
ChemiSEM Technology is unique because it keeps the compositionalmicrostructural relationships at the center of your analysis and enables you to move from basic compositional imaging to point and line scan quantification in the same environment. Here, a user is investigating a multi-phase steel inclusion for compositional variability. The reporting tool tracks the locations and data associated with X-ray map, line scan and point analyses. A single click generates a report that preserves all location information as well as spectral and quantified compositional tables.

Key Benefits

- Easy data acquisition through direct integration of SEM and EDS in the user interface
- Treat EDS like any other detector on the microscope with single-click engagement
- Simplify training and SOP development
- Make inspection and interpretation easier by preserving the context of your data
- Ideal for quick analysis; a single-click report logs all your point and line scans as well as imagery

troduction How does SEM work? How does EDS work? Benefits of combining What is ChemiSEM? How can it help you? Conclusion

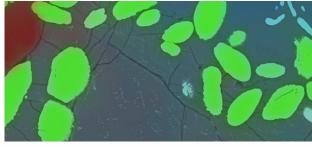
How can ChemiSEM Technology help you?



If you're an industrial lab manager...

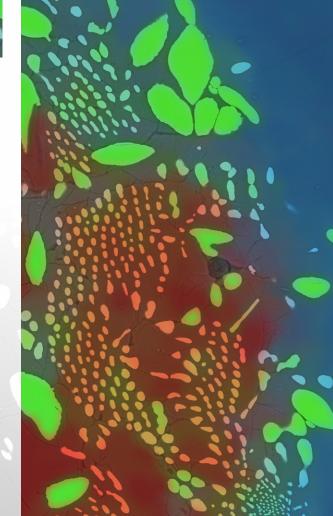
ChemiSEM Technology provides a quick, easy way to screen samples for target features and defects, to identify locations for further analysis, and to generate initial compositional reports. In many instances, ChemiSEM data can replace basic EDS use cases and the associated report generation. We know that the types of samples you're investigated can change throughout the day, and that your priorities can shift quickly, which is why ChemiSEM Technology is also flexible and can quickly change imaging conditions to suit your imminent needs.

For failure analysis and troubleshooting labs, context is essential, as it informs the relevance of your data. The imagery produced with ChemiSEM Technology is instantly useful, granting spatial and compositional information. ChemiSEM Technology offers a superior user experience that helps streamline your observations, combining increased throughput with improved data quality, allowing you to easily visualize and interpret your observations.



If you're an employee in an industrial lab...

You're often utilizing multiple instruments throughout your shift, or you're overseeing technicians performing basic data collection on several tools. In either case, a flexible, easy-to-use system is required. ChemiSEM Technology is straightforward to learn and provides actionable data quickly, even for users new to EDS collection. All EDS and imaging controls are integrated into the same user interface, allowing the work at hand to be your focus. Reporting is automated and includes location and data tracking. A single click generates a report and allows you to move on to the next sample quickly without having to transition between applications.



Ca Mg Zr

100 µm



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Conclusion

Combined EDS and SEM analysis is increasingly becoming a standard tool in failure analysis as well as research and development settings, providing both microstructural and compositional insight for materials.

As the need for this valuable data increases, so does the burden on the laboratories running this analysis. New technology in these fields shouldn't just focus on generating higher quality results, it should also consider the logistics of acquiring and processing the resulting data. Speed, reliability and ease of use are critical for new tools in a successful industrial lab setting.

That is why ChemiSEM Technology provides a combination of high-quality data, speed and system integration, giving you increased throughput for quick inspection tasks along with quantitative data for more advanced work. ChemiSEM Technology offers an accurate picture of spatial elemental data within seconds. The overall combination of improved user experience and time to data enables you to own an approachable solution that incorporates advanced technology within an easy to use platform. Regardless of the use case, ChemiSEM Technology makes compositional microscopy routine, even for novice users.



Watch the webinar

Request a demo

Find out more at thermofisher.com/chemisem

