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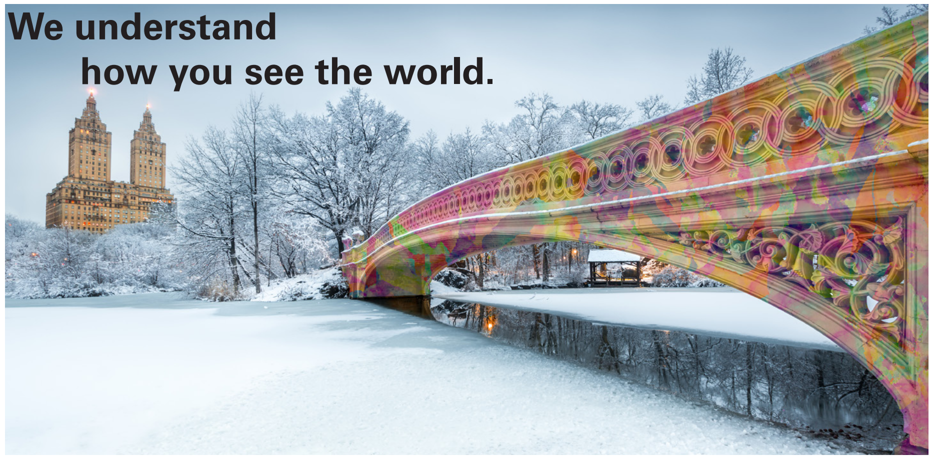

INSIDE

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NEWS

Enhancements from EDS to WDS

With the addition of wavelength dispersive spectroscopy (WDS) capabilities in the latest user-friendly EDAX APEX 3.0 software, users can seamlessly integrate the new functionalities and employ WDS when and where energy dispersive x-ray spectroscopy (EDS) reaches the limit. WDS significantly enhances the accuracy of results by resolving EDS peak overlaps, improving the minimum detection limit by 10x, and providing precise quantification.

EDS suffers insufficient energy resolution to differentiate energy lines close to each other. For instance, Si K, W M, and Ta M lines are separated by a mere 30 eV energy difference. If the three elements are present in the same region of interest, they manifest as an indistinguishable peak in the EDS spectrum (Figure 1 red outline). As depicted by the WDS spectrum (Figure 1 cyan color), EDAX Lambda WDS systems effectively resolve such ambiguities in analysis intrinsically with up to 15x better energy resolution than typical EDS systems.

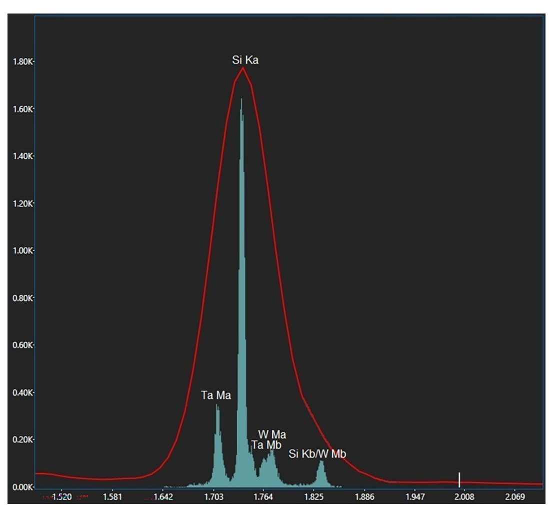

Figure 1. An overlay of EDS (red outline) and WDS (cyan color) spectra of a Si-W-Ta sample.

NEWS

EDS

Ta M, Si K, and W M are not resolved

WDS

Ta M, Si K, and W M are resolved

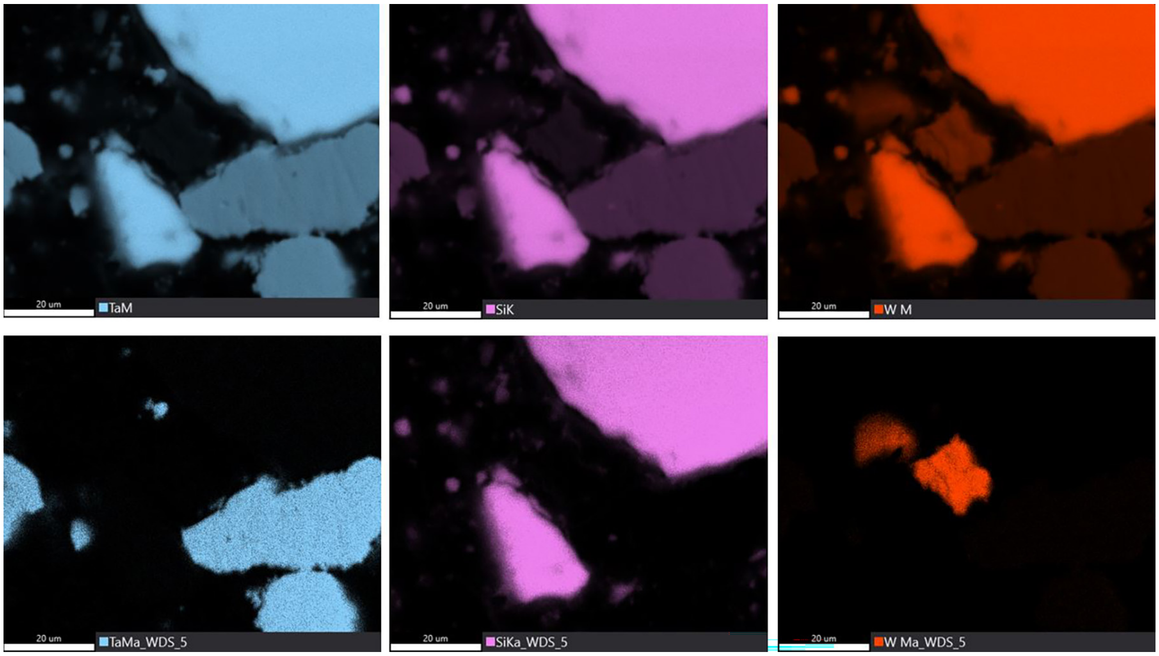

Figure 2. EDS (top) and WDS (bottom) maps of the Si-W-Ta sample. The WDS maps resolve the artifacts due to Ta M, Si K, and W M peak overlaps in the EDS maps.

In a Si-W-Ta sample, EDS struggles to differentiate these peaks, presenting them as identical distributions in the EDS maps (Figure 2 top). In contrast, WDS generates distinct visualizations of the individual distributions of these elements (Figure 2 bottom).

WDS reliably detects minor and trace elements with improved peak-to-background ratios (P/B), achieving detection limits up to 10x lower. For example, in a borosilicate glass containing 2 wt% of boron, the boron peak is hardly visible in the EDS spectrum. However, a clear boron peak is present in the WDS spectrum (Figure 3) owing to significantly lower detection limits. The Lambda WDS system, featuring a parallel beam design, also offers up to 8x higher P/B than Rowland's Circle WDS systems.

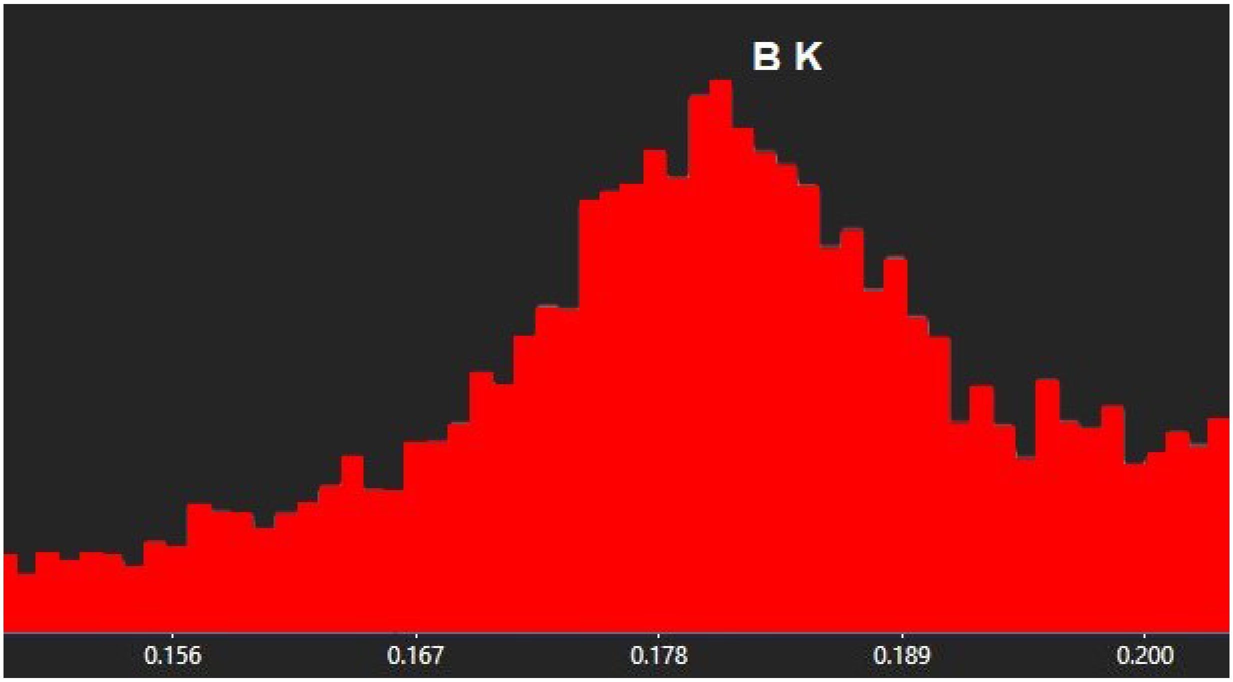

Figure 3. The boron peak in the WDS spectrum of a borosilicate glass containing 2 wt% boron.

NEWS

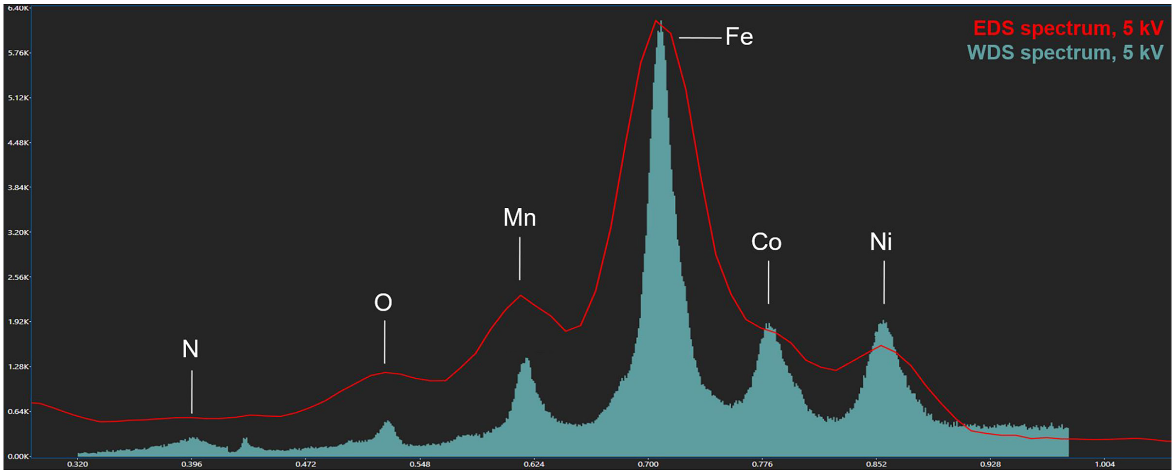

Figure 4. An overlay of EDS (red outline) and WDS (cyan color) spectra of an alloy sample at 5 kV.

The improved energy resolution and higher P/B make WDS an excellent tool for resolving the transition metal L lines. Alloy surface and inclusion analyses often need lower accelerating voltages to reduce the excitation volume. However, transition metal L line peaks have too many overlaps to be resolved by EDS, and P/B can be low (Figure 4 red outline) at 5 kV. On the other hand, WDS delivers data with superior peak separation and P/B to detect transition metal L line peaks that are difficult to observe by EDS alone (Figure 4 cyan color) at the same accelerating voltage.

The combined EDS/WDS quantitative analysis employs a robust analytical algorithm, ensuring reliable elemental quantification. Quantitative results from standardless EDS analysis of a Ni monocrystal reveal considerable discrepancies in Ta, W, and Re due to severe peak overlaps (Table 1). The combined EDS/WDS quantification enables Al, Ta, W, and Re to be quantified with WDS, while the other elements are quantified via EDS, resulting in concentrations consistent with the actual values.

Element	Wt. % (EDS)	Wt. % (EDS and WDS)	Discrepancy	Actual Wt. %
Al	6.30	6.62	5%	
Ta	9.61	7.48	28%	7.50
W	7.57	4.75	59%	4.75
Re	2.19	3.01	27%	3.00
Mo	0.79	0.74	7%	
Ti	1.11	1.15	3%	
Cr	6.21	6.04	3%	
Co	7.83	7.56	4%	
Ni	58.39	57.56	1%	

Table 1. Quantitative results of a Ni monocrystal.

In conclusion, improved energy resolution, sensitivity, precession, and P/B position WDS as an invaluable supplement to EDS detectors. The new APEX 3.0 is the ultimate materials characterization software, integrating EDS, WDS, and EBSD to deliver previously unattainable solutions.

TIP & TRICKS

Tips and tricks for OIM Matrix

OIM Matrix™ is a powerful software tool available as an optional module with the OIM Analysis™ platform that allows users to simulate electron backscatter diffraction (EBSD) patterns using dynamical diffraction models to produce more realistic pattern simulations compared to traditional kinematic diffraction simulations. These more realistic simulations can be easily and accurately compared to experimental patterns to improve indexing performance. With the release of OIM Analysis 9, spherical indexing is available in the OIM Matrix module, which significantly improves the time to results and reduces the barrier of entry to using this powerful technology. Tips and tricks for getting started and using spherical indexing are in this article.

What do I need to run OIM Matrix?

OIM Matrix is an optional module within OIM Analysis. For operation, you need a valid license for both OIM Analysis and OIM Matrix. To use OIM Matrix spherical indexing, you must be running OIM Analysis 9 and need an appropriate graphics processing unit (GPU) card installed on your PC. OIM Analysis and OIM Matrix can be used on the EDAX PC connected to the EBSD detector and the scanning electron microscope (SEM), plus on a separate PC using a remote software license.

What kind of GPU card do I need?

The GPU implementation for spherical indexing requires a modern Nvidia GPU card. For EDAX PCs on the SEM, the RTX A4000 card is recommended as a high-performance single-slot GPU. For remote analysis PCs, you can also use this card. Additionally, you can utilize cards within the RTX 40 series. All these cards have at least 6 GB of GPU memory, sufficient for spherical indexing analysis. The relative performance of different GPUs is available on the PassMark software website using the G3D rating as a benchmark.

Card	Benchmark	Slot requirement
RTX A4000	19354	1-slot
RTX 4060	19367	2-slot
RTX 4060 Ti	22512	2-slot
RTX 4070	26857	2-slot
RTX 4070 Ti	31680	Varies by manufacturer
RTX 4080	34704	3-slot
RTX 4090	38892	3-slot

Note that OIM Analysis and OIM Matrix can be run on laptop PCs using laptop versions of the RTX 40 series cards.

How do I set up my EBSD data acquisition in APEX™ to use spherical indexing?

Spherical indexing is an offline analysis of saved EBSD patterns. To perform this work, the EBSD patterns must be saved during data acquisition. This is done by activating the **Save Patterns** option in the **Scan Parameters** window, as shown in Figure 1. The UP2 format is recommended as it provides the highest bit-depth images, but the UP1 format, with its corresponding lower bit-depth, can be used if file size considerations are important. This option will create a *.up2/.up1 pattern file and save it in the same folder as the APEX project file. When performing analysis away from the EDAX PC, it is essential to copy both the APEX project file and the required patterns files to the remote analysis PC.

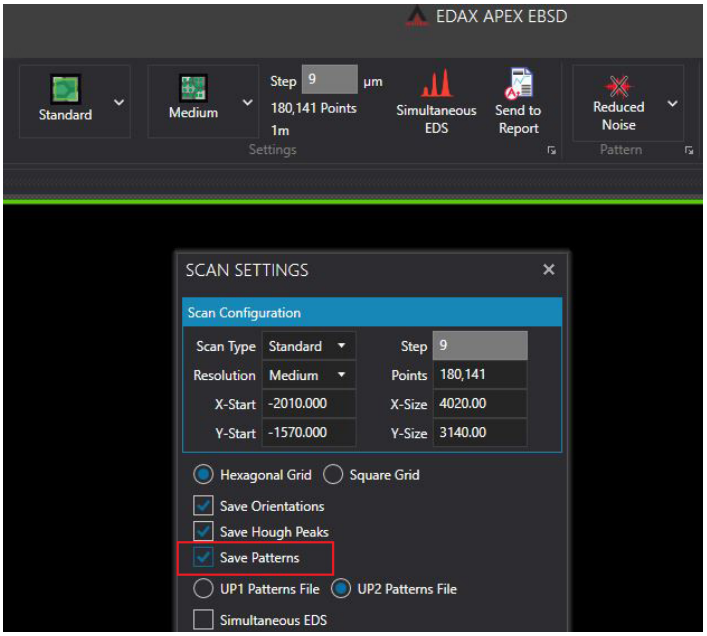

Figure 1. The Scan Parameters window.

Spherical indexing will work with all EDAX EBSD detectors. For Velocity™ and Clarity™ detectors, 4 x 4 binning is recommended for most applications. The EBSD pattern pixel resolution (120 x 120 pixels for Velocity and 129 x 129 pixels for Clarity) provides sufficient resolution for spherical indexing and orientation refinement performance. Examples of this are found on the Being more precise and Being more precise again EDAX blog posts.

How do I set up spherical indexing in OIM Analysis?

When you open the dataset you want to use for spherical indexing, ensure the associated pattern file is recognized within OIM Analysis. If the patterns are available, you will see a **Patterns** section in the **Sample Summary** window that details the pixel size, image bit depth, and file name and location. This association of the scan and pattern files should occur automatically if both files are in the same Windows directory. If they are not, you can still manually associate a pattern file with the scan by right-clicking on the dataset in the Project Tree and selecting the **Reindex Configuration\Associate Pattern** file option.

TIPS & TRICKS

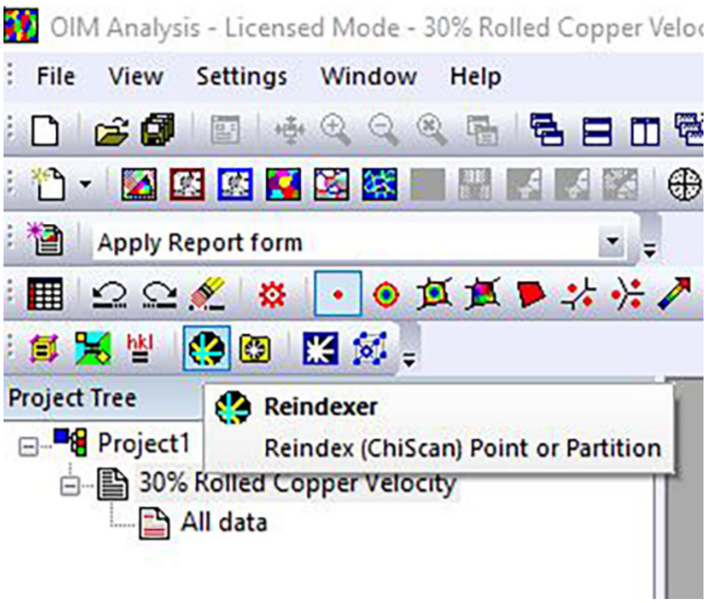

Figure 2. The Reindexer button in the Utilities toolbar.

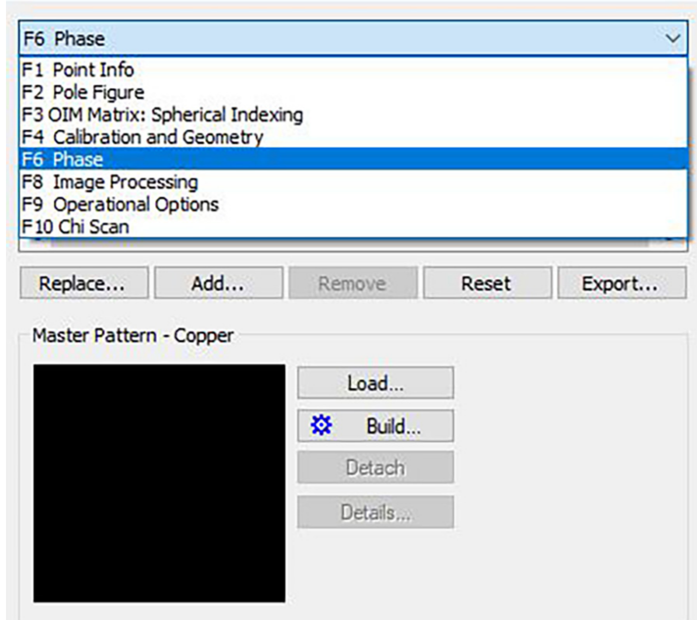

Figure 4. The Phase option pulldown list.

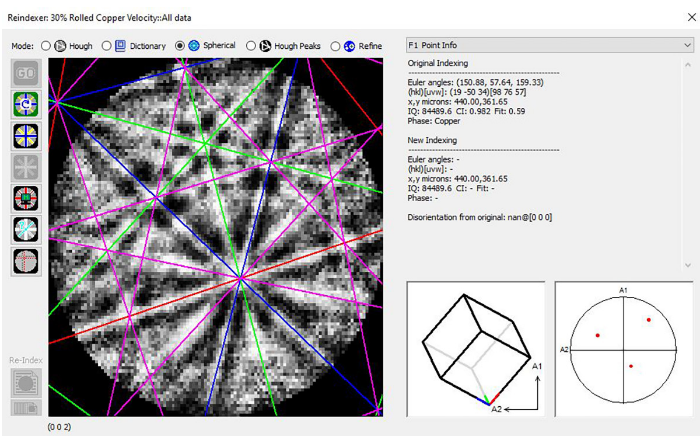

Figure 3. The Reindexer window.

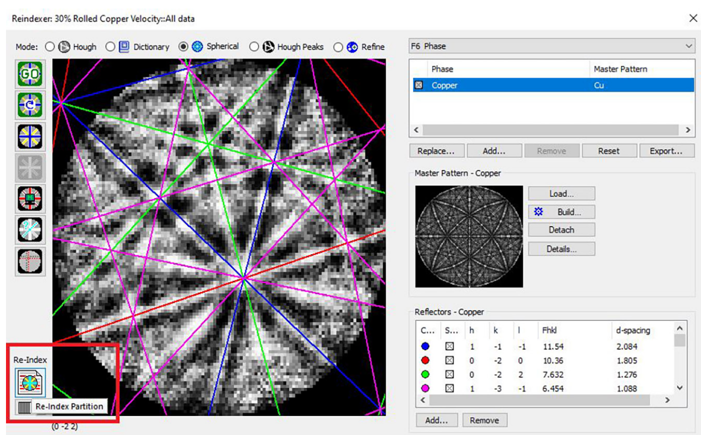

Figure 5. The Re-Index Partition button in the Reindexer window.

Select the **Reindexer** button on the Utilities toolbar, as shown in Figure 2. This will open the Reindexer window, as shown in Figure 3. Within the Reindexer, select **Spherical** as the indexing mode. Other reindexing modes available are dictionary indexing and Hough indexing that both require saved EBSD patterns; and Hough peaks indexing, which uses the Hough peak information saved during APEX EBSD mapping. The mode for orientation refinement is also available.

A simulated master pattern should be associated with each phase of interest. Select the **Phase** option by pressing F6 or selecting the Phase option from the pulldown list shown in Figure 4. Once selected, this will show the phase(s) used during initial indexing in APEX. Select the phase(s), then select **Load** to associate a master pattern file. OIM Analysis 9 contains a preloaded library of master patterns for common materials located in Program Files\OIM Analysis 9\Materials. If your material of interest is unavailable in this library, a new master pattern can be generated using the **Crystal Structure Builder** tool on the Utilities toolbar.

Once the master pattern is associated, you can start the reindexing process by pressing the **Re-Index Partition** button in the bottom left portion of the **Reindexer** window, as shown in Figure 5. Note that this reindexing is based on partitions. In this example, I am using the All Data partition (seen in the Project Tree in Figure 2), which allows all data points to be reprocessed using spherical indexing. This partition-based approach allows for the reindexing of defined subsets. For example, only low-confidence index points or a selected phase could be reindexed. This increases the efficiency of the reindexing process by only using it when required.

TIPS & TRICKS

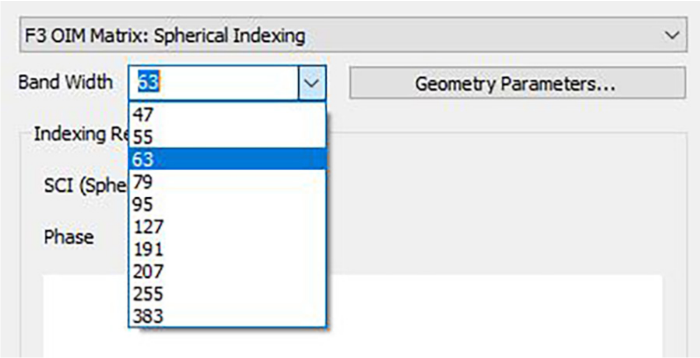

Figure 6. The band width is set in the Spherical Indexing section.

What band width setting should I use?

Spherical indexing single parameter users need to select, termed band width. Higher band width values provide finer pattern details from the simulated master pattern used in the pattern-matching indexing. Band width values of 63 or 95 can be used to start your analysis for many samples and applications. The band width value is set in the **Spherical Indexing** section (F3), as shown in Figure 6. When trying to differentiate similar patterns in challenging applications like pseudosymmetric patterns, higher band widths can be useful. Note that the indexing time is a function of the band width parameter.

Can I use NPAR™ with spherical indexing?

Yes, NPAR can be combined with spherical indexing to provide higher signal-to-noise EBSD patterns in the indexing algorithm for improved indexing success rates. Additionally, other image processing options are available in the **Image Processing** panel, as shown in Figure 7. A circular filtering mask can be used for the traditional phosphor-based EBSD detectors, while a mask designed for the Clarity direct detector is also available. NPAR and its NLPAR extension are both available to improve EBSD signal-to-noise through local pattern averaging. There are several background correction options that can be used to optimize the background processing for both single and multi-phase samples. There is also an Advanced image processing toolbox with a range of tools for tackling less frequent problems.

The **Adaptive Histogram Equalization** is a normalization function recommended for spherical indexing.

Figure 7. The Image Processing panel.

APPLICATION NOTE

Evaluation of the accuracy of standardless EDS analysis in APEX EDS software

An essential task of analysis using energy dispersive x-ray spectroscopy (EDS) is to determine the elemental composition of a sample quantitatively. In recognition of the importance of this task, great care is taken to develop robust analytical models to accurately and precisely determine a sample's composition. In this article, we report a summary of the evaluation of the quantitative performance of EDS standardless analysis in the EDAX APEX™ 3.0 EDS Standard and Advanced software programs.

An EDS spectrum is analyzed during quantitative analysis to determine the weight or atomic fraction of elements present in a sample. We have published several articles that explain the detailed process of this[1, 2, 3], but in summary, for standardless analysis, the x-rays characteristic of elements present in the sample are separated from each other and the Bremsstrahlung background. Then, an iterative algorithm is applied to correct for excitation efficiency (also a function of the primary electron energy), self-absorption of x-rays that are generated in the sample, possible enhancements of an x-ray line's intensity by secondary x-ray fluorescence, and detector efficiencies to convert from net x-ray intensities to elemental composition. The model used in the iterative algorithm (normalized standardless eZAF corrected analysis) has been developed continuously as there is a greater understanding of the physical processes of x-ray generation, emission, and detection[4].

Before release of a new APEX software version, the analytical performance of APEX EDS Standard and Advanced are validated by evaluation of spectra collected from more than sixty certified standards, including minerals, metal alloys, and binary compounds. We determine the total absolute error by summing the absolute difference between the measured and known composition for each element, i, present in the sample:

$$\text{Total absolute error (wt. \%)} = \sum_{i=1}^{i=n} |\text{Known (wt. \%)} - \text{measured (wt. \%)}|$$

Figure 1 reveals the significant improvements in the accuracy of compositional analysis by standardless eZAF correction in APEX software versions, emphasizing the step change in accuracy available with APEX EDS software version 3.0. Furthermore, APEX EDS Advanced software offers improved accuracy through the use of a computed physical model of Bremsstrahlung background rather than the mathematical filtering approach (SNIP) used in APEX EDS Standard software and other analysis software that is available commercially. Figure 2 shows the improvement for a selection of standards that were analyzed.

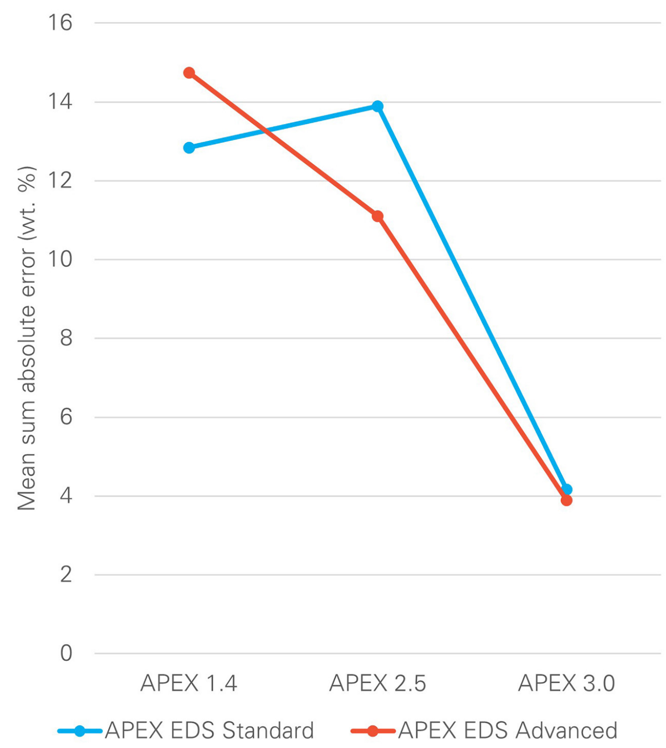

Figure 1. Improvement of the accuracy of compositional analysis of APEX EDS software evaluated by analysis of mineral, metal, and compound standards.

APEX EDS Standard software and other analysis software that is available commercially. Figure 2 shows the improvement for a selection of standards that were analyzed.

As seen from the figures, standardless analysis using eZAF correction has improved dramatically in recent years in APEX EDS Software. However, only you can determine if this level of accuracy is sufficient for the analysis challenges you face. If not, APEX EDS Advanced software offers the possibility of further improvements using standards customized coefficients (SCC) for eZAF standardless analysis—an empirical database that optimizes the iterative model for your installation—or full standards quantification (FSQ).

APPLICATION NOTE

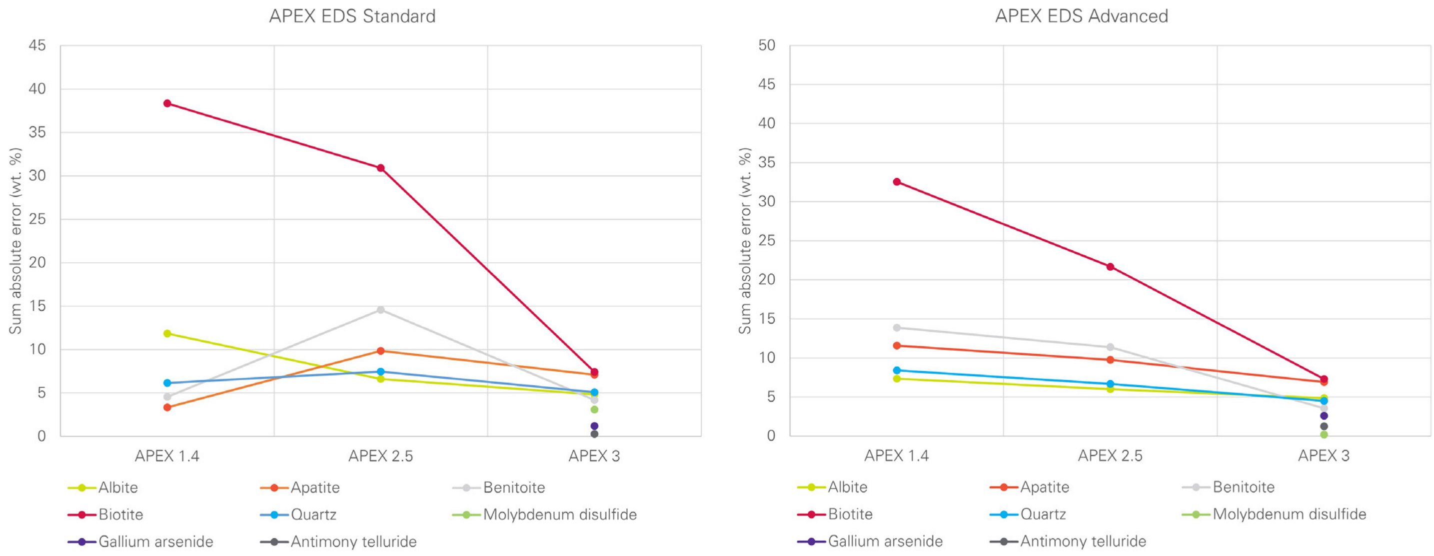

Figure 2. Error in the analysis of selected standards.

References

1. Ways to improve the EDS quantitative results accuracy: Efficiency and eZAF SCC database. EDAX Insight Vol. 19, Issue 3. September 2021.
2. Ways to improve the EDS quantitative results accuracy: eZAF MACC database and correction avoidance. EDAX Insight Vol. 19, Issue 4. December 2021.
3. Ways to improve EDS quantitative results accuracy: FSQ and SmartStandards. EDAX Insight Vol. 19, Issue 4. December 2021.
4. F. Eggert. Abilities towards improved accuracy in EPMA. *Microscopy and Microanalysis (M&M) 2021.*

EMPLOYEE SPOTLIGHT/EVENTS AND TRAINING

Roy Stanley

Roy joined Gatan/EDAX as a Content Creator in June 2023. He is based out of the AMETEK Instruments office in Bangalore, India. Roy's responsibilities include generating social media and email content, graphic creation, and managing the translation process for all Gatan and EDAX collateral.

Before joining Gatan/EDAX, Roy was a Research Scholar at Madurai Kamaraj University while pursuing his Ph.D. in journalism and mass communication with a focus on analyzing newspaper reporting during extreme climate events to assess its gender sensitivity and responsiveness. He later worked at BYJU's Learning as a Content Developer and exited as a Content Lead. Roy was part of a team that developed some of the largest and most ambitious education technology products.

Roy graduated from St. Joseph's College in Tiruchirappalli, India, in 2012 with a bachelor's degree in computer science. He received master's degrees in E-media communications and communication studies from Madurai Kamaraj University in 2014 and 2016, respectively.

Roy and his wife, Shiny Leo, reside in Bengaluru, India. Roy is fascinated with history and exploring diverse culinary delights. He likes cycling around town and indulging in photography. In his downtime, Roy enjoys playing video games.

2024 Worldwide Events

Microscopy Society of Ireland (MSI) 2024
January 17 – 19 — Limerick, Ireland

SEMICON Korea
January 31 – February 2 — Seoul, South Korea

Electron Microscopy UK & Ireland (EM-UKI)
February 8 – 9 — York, United Kingdom

The Minerals, Metals & Materials Society (TMS)
March 3 – 7 — Orlando, FL

2024 Worldwide Training

Europe

APEX EDS
January 29 – 31 — Unterschleissheim#
March 11 – 13 — Unterschleissheim*

APEX EBSD
January 31 – February 2 — Unterschleissheim#
March 13 – 15 — Unterschleissheim*

APEX Pegasus (EDS & EBSD)
January 29 – February 2 — Unterschleissheim#
March 11 – 15 — Unterschleissheim*

#Presented in English
*Presented in German

Japan

EDS Microanalysis (APEX™ EDS)
February 16 — Virtual (Standard)
April 12 — Virtual (Advanced)

China

EDS Microanalysis
March 12 – 13 — Shanghai (ACES)

EBSD OIM Academy
March 13 – 15 — Shanghai (ACES)

Self-Paced

EDS Microanalysis
On-Demand — em-academy.info

EBSD and OIM Basics
On-Demand — em-academy.info

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Training courses – https://www.edax.com/support/training-schools

COMPANY NEWS

EDAX and Gatan merge to offer the ultimate suite of microscopy tools

PLEASANTON, CA – Gatan, Inc., a business of AMETEK Inc. as well as a global leader focused on enhancing and extending the operation and productivity of electron microscopes, has merged with EDAX, LLC, a leader in x-ray microanalysis and electron diffraction instrumentation. The combined organization will retain the Gatan name, offering the ultimate suite of tools for transmission electron (TEM) and scanning electron (SEM) microscopes.

With broad product and organization overlap between Gatan and EDAX, transitioning to Gatan as the prominent tradename for all products will allow the organization to capitalize on its synergies fully. EDAX, also a business of AMETEK Inc., will still provide customers high-quality, reliable products and responsive customer care and support they expect. Joining Gatan will provide EDAX with more resources and allow it to leverage Gatan's strong analytical capabilities and high-level technical expertise.

"This merger will allow Gatan to continue to push the boundaries of science on both the TEM and SEM," said Narayan Vishwanathan, Vice President and Business Unit Manager of AMETEK Electron Microscopy Technologies. "Gatan and EDAX have already demonstrated synergies in their product portfolios, including the Cipher System, the first and only system that quantitatively reveals the distribution of lithium in the SEM, and the EDAX EDS Powered by Gatan, the most intuitive and easy-to-use analytical tool for STEM (scanning) applications. We look forward to further integrating our teams, knowledge, and skills and are excited about what the future holds."

About Gatan

Gatan is the world's leading manufacturer of instrumentation and software used to enhance and extend the operation and performance of electron microscopes. Gatan's products, which are fully compatible with all brands of electron microscopes, cover the entire range of the analytical process from specimen preparation and manipulation to imaging and analysis. Its customer base spans the complete spectrum of end-users of analytical instrumentation typically found in industrial, governmental, and academic laboratories.

The applications addressed by these scientists and researchers include new materials research, semiconductors, electronics, geosciences, biological science, and biotechnology. The Gatan brand name is recognized and respected throughout the worldwide scientific community and has been synonymous with high-quality products and the industry's leading technology. Gatan is a business of AMETEK, Inc., a leading global provider of industrial technology solutions serving a diverse set of attractive niche markets with annual sales over $6.0 billion.

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