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EDAX NEWS

Elite T EDS System for the TEM

With the recent launch of the Elite T EDS System, EDAX has added the Elite range of Silicon Drift Detectors (SDDs) to the Transmission Electron Microscope (TEM) portfolio. This allows TEM users to take advantage of the low noise, high speed, high stability electronics of the Elite pulse processor, coupled with excellent performance and resolution of the windowless SDD module optimized for TEM applications. The modules themselves have significant differences from the standard EDS modules to reduce beam shift and ensure ultra-high vacuum compatibility and in this article we will look at some of the performance metrics and design criteria specific to the TEM.

SEM vs. TEM

The TEM poses a very different set of challenges than the Scanning Electron Microscope (SEM) when it comes to the detection of X-rays generated in the sample. In the SEM, we usually deal with bulk samples where most of the incoming electrons deposit their energy in the sample and consequently generate X-rays. In the TEM, sample thickness is often below 100 nm and the acceleration voltages are much higher than in the SEM to achieve transmission through the sample. Because of this, only a small fraction of the incoming electrons will generate X-rays resulting in much lower signal levels than in the SEM. To compensate for the reduced signal levels, the solid angle must be optimized, i.e. the EDS detector is mounted much closer to the pole piece and sample than typically done in a SEM (Figure 1).

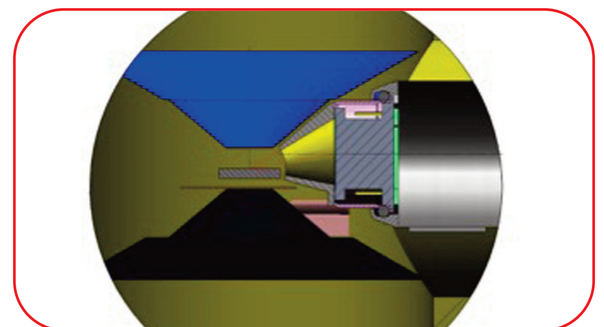

Figure 1. Illustration of the upper and lower pole piece in a TEM with the sample in the middle and the EDS detector on the right.

(Continued from Page 1)

Geometry and Solid Angle

The solid angle/field of view of the detector is one of the performance metrics often applied to an EDS system on a given TEM, but in reality, the number is a combination of both the geometry of the given microscope and the size and shape of the detector. Due to the limited space between the pole pieces inside the TEM, a change in the detectors' size rarely results in a proportional change in the solid angle. A larger detector will have to be moved further back to fit, which can in some cases result in a smaller solid angle although the active area increased. The most basic calculation of the solid angle in steradians is simply the detector area divided by the distance to the sample squared. However, this results in an inflated number since it neglects several finer aspects of the calculation, as described by Nestor Zaluzec of Argonne National Laboratory[1]. Accompanying the article is an interactive tool illustrating how changes in geometry, active area, and holder configuration influence the solid angle[2]. For each model of TEM, the Elite T systems go through a rigorous design process to ensure the best possible solid angle is achieved, as can be seen by the user in the mechanical drawings provided with every purchase.

Spectrum Quality: Fiori Number and Stray Signal

Although the solid angle is the starting point for the design process to ensure the maximum number of X-rays are captured, quantity without quality means nothing. To quantify the quality of the spectrum, two common measurements are the Fiori number and the stray signal or system peak ratio.

The Fiori number is often used as an indication of the quality of an EDS system on a TEM and is typically measured with a NiOx sample[3]. It is calculated by taking the scaled background subtracted counts in a 600 eV wide range around the Ni Kα peak and dividing them by the sum of the counts in two 300 eV background regions (Figure 2). An alternative method uses a Ge/SiNx standard for the measurements but with the same approach[4]. The Fiori number essentially gives a measure of the amount of stray electrons and X-rays coming down the column.

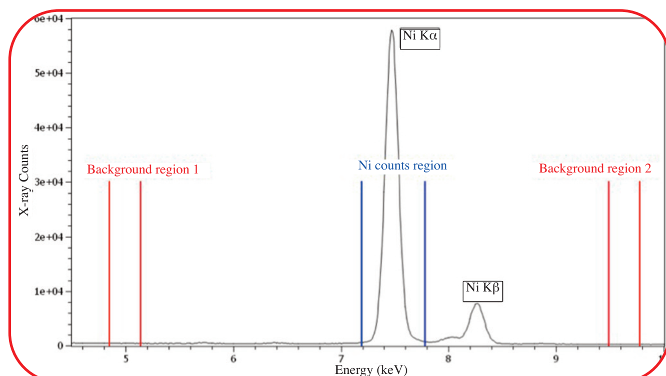

Figure 2. Illustration of the energy regions used in calculation of the Fiori number using a NiOx sample.

While the stray electrons and X-rays decrease the quality of the spectrum, it is important to realize that the EDS detector has little to no influence on this. It is primarily a function of the column design, acceleration voltage, and sample tilt.

System or stray peaks are typically seen as Fe and Co artefacts in the EDS spectrum that are present regardless of the composition of the sample. They originate from the pole piece and depending on magnitude and the sample being measured, they can severely limit the analysis capability. The system peaks can be limited by using a collimator in front of the EDS detector, essentially blocking the line of sight from the detector to the pole piece (Figure 3). At this point, a compromise will often have to be made between solid angle and system peaks. A detector with no collimator will have the largest possible solid angle since no signal is cut off, but it will also have very large system peaks. By closing down the collimator the system peaks can be reduced, but this can also result in a decrease of the counts from the sample. A common requirement is for the Fe/Co net counts to not exceed 1% of the Ni net counts using a NiOx standard, but much smaller numbers can be necessary depending on the application. This also means that the largest solid angle is not necessarily the best choice, a compromise must be made between quantity and quality and the specific configuration of collimator and module placement must be optimized for each TEM model.

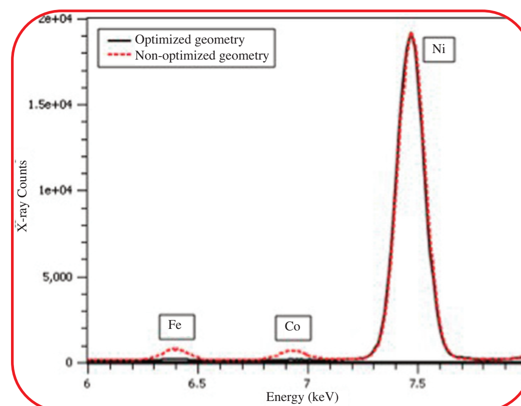

Figure 3. Fe and Co contribution from the pole piece with the same detector and two different collimator designs.

Summary

The design process for the Elite T EDS detector has focused on creating a TEM optimized system to ensure users see the highest quality spectra possible on their given microscope, balancing the compromises between counts and artefacts for each TEM column design. We are excited to bring the Elite platform to the TEM and are continuing development on the product range with more offerings to come in the near future.

1 N. J. Zaluzec, "Analytical Formulae for Calculation of X-Ray Detector Solid Angles in the Scanning and Scanning/Transmission Analytical Electron Microscope," *Microsc. Microanal.*, pp. 1318-1326, 2014.
2 "XEDS Tools Solid Angle Calculator," [Online]. Available: http://tpm.amc.anl.gov/NJZTools/XEDSSolidAngle.html.
3 R. C. S. Egerton, "Characterization of an analytical electron microscope with a NiO test speciment," *Ultramicroscopy*, vol. 55, pp. 43-54, 1994.
4 N. J. Zaluzec, J.-P. DesOrmeaux and J. Roussie, "A Ge/SiNx Standard for Evaluating the Performance of X-ray Detectors in the SEM, S/TEM and AEM," *Microsc. Microanal.*, pp. 322-323, 2016.

TIPS AND TRICKS

Utilizing Standards to Improve Energy Dispersive Spectroscopy Quantification in TEAM™

Energy Dispersive Spectroscopy (EDS) quantification in TEAM™ utilizes a unique eZAF (atomic number (Z), absorption (A) and fluorescence (F)) algorithm to calculate the quantity of an element detected by the software application. Once a spectrum is captured, these measurements are available automatically by means of the "Quant" button in the Spectrum Toolbox, and can be displayed as weight percent, atomic percent, or oxide ratio. This measurement is known as a "standardless" quantification, as it does not employ any measured standards in the correction. The eZAF correction works well for both polished and rough samples alike, as well as samples tilted to 70° for EBSD work.

For those analyses requiring a higher degree of quantitative accuracy, standards can be incorporated into the quantification routine. Collection of standards for improved quantitation requires a set of known reference materials (purchased certified standards, samples independently quantified by another analysis technique, etc.), as well as a means of accurately measuring beam current, such as a picoammeter. If one is unable to measure beam current accurately and consistently, standards must be reevaluated every session to ensure consistent current between the standard and the unknown.

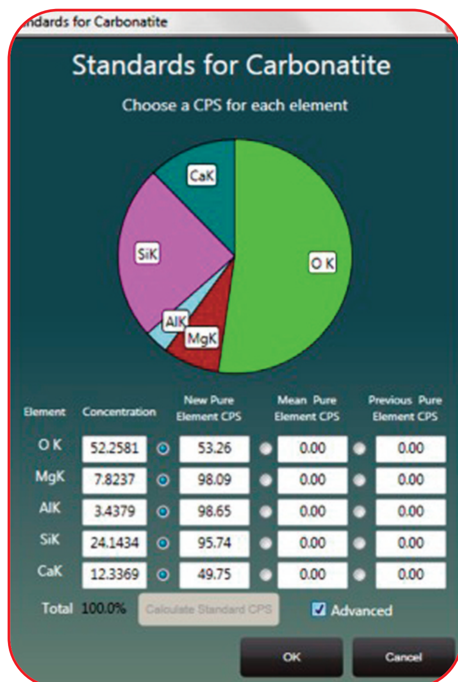

Figure 1. The Calculate Standard menu in TEAM™ .

After a spectrum for a known material is acquired, the standards can be incorporated into your quantification by selecting "Calculate Standard" under the Quantification section of the Spectrum Tools panel. Use this menu to input the concentrations of the known material and click "Calculate Standard CPS" when complete (Figure 1). One can then begin acquisition of unknowns while applying the newly measured standard by selecting "Calculate Standard" again, inputting the beam current, and selecting the desired standard from the saved options (Figure 2).

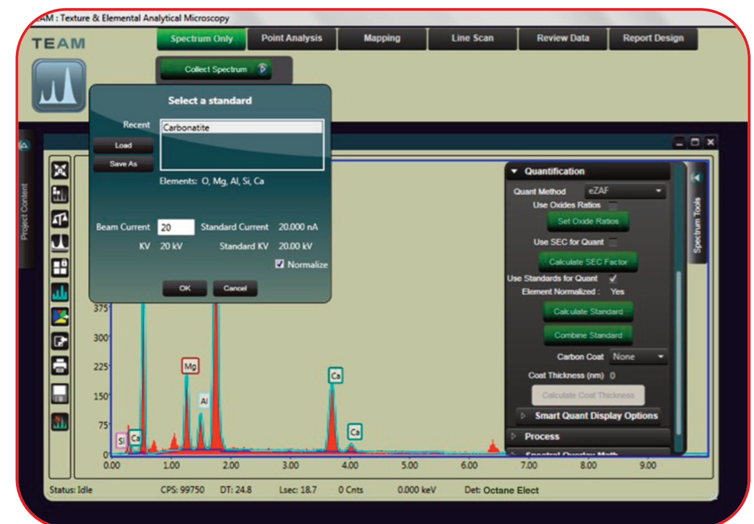

Figure 2. TEAM™ users can begin acquisition of unknowns while applying the newly measured standard. After clicking "Calculate Standard", the user is asked to input the beam current and select the desired standard from the saved options.

It is important to keep in mind that a critical part of the quantification process, be it standards-based or standardless, is to ensure a good background fit. TEAM™ offers both manual and automatic background corrections in the "Background" section of the Spectrum Toolbox (Figure 3).

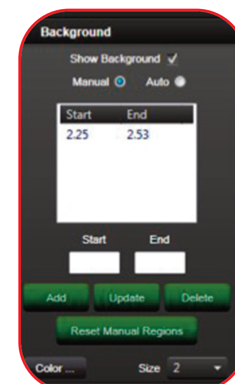

Figure 3. Background menu located in the Spectrum Toolbox in TEAM™ .

EDAX Smart Insight

APPLICATION NOTE

How to Get the Most Out of Orbis Micro-XRF Measurements with Multiple Acquisition Conditions

Micro X-ray Fluorescence (Micro-XRF) is a versatile, non-destructive elemental analysis technique providing qualitative and quantitative elemental composition analysis, as well as thickness and composition measurements on coated samples. For benchtop micro-XRF systems, the elemental measurement range typically runs from Na to Bk. Within this range, measurements can be further optimized by using specific acquisition conditions for certain ranges of the elemental spectrum. For example, in this application note, primary beam filters were used while maintaining constant tube excitation conditions. Primary beam filters are typically pure, thin metal foils used to condition the primary excitation beam. The benefits of filtering are:

- Improved Peak-to-Background (P:B) ratios by reducing background scatter
- Elimination of overlapping tube characteristic lines and Bragg diffraction peaks
- Elimination of sum peaks by suppressing the intensity of the parent peaks

Filtering typically optimizes the acquisition conditions over a limited range of the spectrum. So, the overall spectral analysis may benefit from acquiring the spectrum using more than one filtering condition. Of course, the time for each measurement condition can be varied as well depending on the LOD requirements for the elements of interest to accommodate the overall, allotted measuring time.

Examples:

One example of this type of analysis was described in an Insight Application Note: *Discriminating Glass Fragments Using Micro-XRF Spectrometry with Poly-Capillary Optics* distributed in 2015 (EDAX Insight Vol. 13 No. 4) describing the forensic comparison of glass fragments. In this analysis, elemental ratios are used to determine if fragments from unknown glass sources are consistent with glass fragments from a known source (e.g. the window at a crime scene). Specific filtering conditions were used to remove characteristic tube line overlaps (i.e. to observe Cl K and S K without the overlap of Rh L scatter) and to eliminate sum peaks generated by Si K and Ca K lines. A filter was also employed to improve P:B for trace Pb, which was used to distinguish bottle glass. In this example, the goal was not to do a full quantitative analysis but rather compare and distinguish fragments using characteristic elemental ratios.

Full quantitative analysis of glass is also important as glass composition often affects the performance parameters of specialty glasses, such as high strength glasses. In this case, three acquisition conditions were used to measure the light elements from Na to Si; S to Ca; and Ti to Zr. SRM 1831, which is a soda lime glass, was used as a standard to calibrate all three acquisition conditions. A calibration standard of some type is required for each acquisition condition. This can be a high purity element, oxide or other compound “pseudo” standard(s) or it can be a compositional type standard(s). Elements found in the unknown, which are not found in the calibration setup standards, will be quantified based on interpolation or extrapolation from the calibrated elements. Calibration results for SRM 1831 are shown in the Table 1.

Filter	Component	Measured	Reported	Relative Error
0	Na_2O	13.32	13.32	-0.02%
0	MgO	3.51	3.51	-0.02%
0	Al_2O_3	1.21	1.21	-0.02%
0	SiO_2	73.06	73.08	-0.02%
1	SO_3	0.250	0.250	-0.02%
1	K_2O	0.330	0.330	-0.02%
1	CaO	8.20	8.20	-0.02%
2	TiO_2	0.019	0.019	-0.02%
2	Fe_2O_3	0.087	0.087	-0.09%
2	SrO	0.010	0.011	-0.38%
2	ZrO_2	0.0037	0.0059	-36.93%
2	MnO	0.0014	0.0019	-29.11%

Table 1. Results for SRM 1831 Soda Lime Glass calibration standard.

(Continued from Page 4)

Filter	Component	SRM 620 Soda Lime (Measured)	Rel. Error	SRM 93A Borosilicate (Measured)	Rel. Error	FGS 2 Float Glass (Measured)	Rel. Error
0	Na_2O	14.06	-2.3%	4.27	7.2%	14.61	7.9%
0	MgO	2.63	-28.8%	0.080	1501.5%	4.12	6.0%
0	Al_2O_3	1.64	-8.7%	2.03	-11.0%	1.01	-27.5%
0	SiO_2	73.69	2.2%	80.82	0.0%	70.17	-2.2%
1	SO_3	0.187	-33.1%	0.00	NR	0.02	NR
1	Cl			0.12	101.4%		
1	K_2O	0.414	1.0%	0.03	131.6%	0.63	14.3%
1	CaO	7.21	1.4%	0.02	84.7%	8.81	6.2%
2	TiO_2	0.015	-17.8%	0.01	-14.4%	0.056	3.0%
2	BaO					0.036	61.2%
2	MnO					0.03	7.4%
2	Fe_2O_3	0.043	-1.0%	0.03	-2.6%	0.43	16.2%
2	Rb_2O					0.0040	3.5%
2	SrO	0.031	NR	0.001	NR	0.030	-0.5%
2	ZrO_2	0.0180	NR	0.028	-32.8%	0.025	-17.8%
2	As_2O_3	0.0626	11.8%				
ND	B_2O_3			12.56			

Table 2. Results for three standard glasses using the SRM 1831 calibration. "NR" indicates there is no reported value for this element in the certification. Total measuring time was five minutes.

From the results in Table 1, we can see that the method is working as we are measuring the glass standard against itself. Also, ZrO_2 and MnO, highlighted in green, were not entered into the calibration. So, we can see what type of accuracy to expect for oxide traces when we are interpolating or extrapolating the calibrations under more ideal circumstances.

In Table 2, measurement results are shown for other NIST glass standards. SRM 620 is another soda lime glass. FGS 2 is a float glass which is simply soda lime glass formulated for flat glass applications, i.e. windows. Soda lime glass also has a somewhat different formulation for bottle glass. Finally, SRM 93A is a borosilicate glass with a significantly different composition to the soda lime glass. Results for elements highlighted in green are interpolated/extrapolated from the elements in the SRM 1831 calibration. Errors for the soda lime and float glass are consistent with what is observed for the MnO and ZrO_2 shown in Table 1. Note that the error level of some elements may be related more to line fitting, particularly of overlapping peaks, than the calibration itself. This is probably true of the BaO result in FGS 2. Overall, the errors in the borosilicate glass seem to be a bit higher than those for the other glasses. This may be related to a difference in glass matrix or simply that many of the observed elements in SRM 93A are at lower trace levels. The MgO is below the limit of detection and the error here is related to background and peak fitting noise.

Conclusion:

Primary beam filters can be used to remove various spectral interferences to get a more reliable value for the affected peak intensities. In the examples discussed here, the Rh L scatter peak could be filtered to better analyze S and Cl; sum peaks which could interfere with K, Mn and other potential traces found in these glasses were suppressed by filtering; and the P:B for trace Pb was improved by filtering which allowed for distinguishing bottle glass fragments. Full spectral analysis and quantification using multiple acquisition conditions is done in a stand-alone SW routine, which can be ordered from EDAX.

EVENTS AND TRAINING

2018 Worldwide Events

October 4-5
Appalachian Regional Microscopy Society (AReMS) Knoxville, TN
October 14-18
Materials Science & Technology (MS&T) 2018 Columbus, OH
October 23-27
Northeastern Association of Forensic Scientists Bolton Landing, NY
October 28-November 1
International Symposium for Testing & Failure Phoenix, AZ
November 12-14
Münster EDX Course Münster, Germany
November 25-30
Materials Research Society (MRS) Fall Meeting Boston, MA

Please visit http://www.edax.com/news-events/conferences-tradeshows for a complete list of our conferences and tradeshows.

2018-19 Worldwide Training

To help our present and potential customers obtain the most from their equipment and to increase their expertise in EDS microanalysis, WDS microanalysis, EBSD/OIM™ , and Micro-XRF systems, we organize a number of Operator Courses at the EDAX facilities in North America, Europe, Japan, and China.

EUROPE

APEX™	
November 21-22	Weiterstadt#
EDS Microanalysis	
TEAM™ EDS	
November 12-14	Weiterstadt#
EBSD OIM Academy	
November 14-16	Weiterstadt#
TEAM™ Neptune (EDS & WDS)	
June 18-22	Weiterstadt#
TEAM™ Pegasus (EDS & EBSD)	
November 12-16	Weiterstadt#

*Presented in English
#Presented in German

JAPAN

EDS Microanalysis	
TEAM™ EDS	
October 18 November 15	Tokyo Osaka
OIM School	
October 19-20 November 1-2 January 25-26	Tokyo Tokyo Osaka

CHINA

EDS Microanalysis	
December 4-6	Shanghai
TEAM™ EBSD OIM Academy	
December 11-13	Shanghai

NORTH AMERICA

APEX™	
October 10-11	Mahwah, NJ
EDS Microanalysis	
December 11-12 February 25-26	Mahwah, NJ Draper, UT
EBSD OIM Academy	
October 23-25 February 27- March 1	Draper, UT Draper, UT
TEAM™ Pegasus (EDS & EBSD)	
February 25- March 1	Draper, UT

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EMPLOYEE SPOTLIGHT

(left to right): Frank and Daniela Eggert at Oktoberfest.

Frank Eggert

Frank joined EDAX in April 2006, after serving as a consultant since 2002. A Senior Scientist, he works from his home-office in Berlin, Germany most of the time. Frank is responsible for the analytical algorithm base in Energy Dispersive Spectroscopy (EDS) and Wavelength Dispersive Spectrometry (WDS) software. His work provides the fundamentals for EDS spectra evaluation and X-ray software qualitative and quantitative abilities for analysis of element compositions.

Prior to EDAX, Frank worked at Röntgenanalytik GmbH, a company with close relations to EDAX (e.g. the Eagle product), from 2001-2006. As a Senior Scientist on X-ray Fluorescence (XRF), he concentrated on soft- and hardware development. Frank was one of the founders of RÖNTEC company from 1991 to 2001. He started as a product manager and was eventually named a development director. Frank pioneered the silicon drift detector (SDD) application and its introduction into the scanning electron microscope (SEM) market. He contributed the first known SDD applications in SEM before the turn of the century, with very early focus on the core advantages of high count-rate processing for X-ray images in the SEM.

Frank studied physics at Dresden Technical University in Germany, and earned the degree of a 'Diplom-Physiker' in 1983. After University, he spent several years at the Academy of Science in Berlin working on EDS detector technology and physics. His thesis to get a PhD degree was completed but was never officially submitted because of the confusion around the fall of the Berlin Wall. Also, his focus changed and Frank faced new challenges associated with founding a new company that was entering into the EDS market.

Frank and his wife, Daniela, have been married since 1982. They have two children, a son (32) and a daughter (29), who both have their own families or partners. Frank and Daniela have one granddaughter. In his spare time, Frank likes spending time with his family, who all live in Berlin. As a hobby, he enjoys Standard and Latin dance with his wife. Additionally, Frank likes traveling to nice places and riding around with a tandem bicycle in the nearby countryside.

(left to right): Tianyi Li and Harris Jiang.

Harris Jiang

Harris joined EDAX in July 2018 as the Regional Sales Manager based in Shanghai, China. It is his second stint with the company. Previously, he was a Sales Engineer in North China out of the Beijing office from 2016-17. Harris' responsibilities include, the sales of microanalysis and other related products and services in the Eastern part of China. He also develops and maintains strong working relationships with customers and electron microscope manufacturers in the area.

After leaving EDAX in 2017, Harris was a Sales Account Manager for the MSD-Semi group at Thermo Fisher Scientific in Shanghai. Prior, he served as a Sales Engineer of electron microscope and light microscopy products at Carl Zeiss from 2013-16.

Harris earned his undergraduate degree from Zhejian University in Hangzhou City, China. In 2014, he received a master's degree in Material Science and Technology from Zhejiang University in Hangzhou.

Harris and his wife, Tianyi Li were high school classmates. They got married in 2016 after Tianyi Li earned her master's degree from the University of California, San Diego. In his spare time, Harris enjoys watching movies with his wife.

CUSTOMER NEWS

AK Steel, Research and Innovation Center Middletown, OH

AK Steel is a leading producer of flat-rolled carbon and stainless and electrical steel products, mainly used in the automotive, infrastructure and manufacturing, and electrical power generation and distribution markets. The company's Research and Innovation Center in Middletown, OH focuses on improving current processes and products, as well as developing new processes and products to meet its customers' current and future needs. The center also supports projects at the company's production plants and handles customer support issues.

The Research and Innovation Center at AK Steel faces several materials challenges. The scientists at the center must fully understand the microstructure of next generation, advanced high strength steels, used in the automotive industry. They use Electron Backscatter Diffraction (EBSD) and X-ray Diffraction (XRD) as complementary techniques. The scientists measure phases with XRD to get a bulk measurement and perform EBSD analysis to identify size, morphology, and distribution. The need to understand the microstructural development of these products is essential to designing better alloys and processing techniques.

The center also faces the challenge of texture development in steels. Texture of electrical steels is a factor in determining the final magnetic properties of the product. Ensuring that the correct texture and grain sizes are developed after each step of production is essential to delivering good products to the company's customers. EBSD results are very important in confirming the right texture after each step of processing. The scientists use XRD to determine bulk texture and then look at the orientation grain-by-grain via EBSD.

Failure analysis and defect analysis are also conducted at the Research and Innovation Center. The scientists need to identify the root cause of customer complaints or equipment failures to minimize errors and the associated costs. Energy Dispersive Spectroscopy (EDS) analysis is utilized to determine root causes of defects and failures in the company's products and equipment.

"The key factor in purchasing our EDAX TEAM™ Pegasus Analysis System (which includes an Octane Elite Silicon Drift Detector and Hikari Super EBSD Camera) was the OIM Analysis™ software for EBSD data," stated Metallurgical Engineer, Ana Araujo. "The software is very powerful and allows us to do some very specific analysis, as well as batch processing. The hardware is also very powerful and was the best available in the market at the time we were getting the new system. Since we are in the steel industry, that is the only material we typically look at. Sometimes we get a few challenges that involve looking at zinc or aluminized coatings in steels or heavily deformed steels. I believe having both EDS and EBSD analysis available is essential to being successful in the development of any new grade of steel, especially the ones with more complex and finer microstructures."

Figure 2. AK Steel Research and Innovation Center Metallurgical Engineer, Ana Araujo.

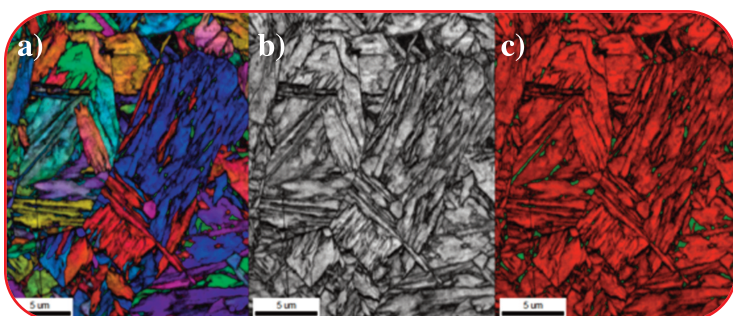

Figure 1. A representative scan of new materials that are being developed as part of the new generation of advanced high strength steels. a) IPF, b) Image Quality, and c) Phase maps where red is BCC-Fe and green is FCC-Fe.

EDAX Inc.
91 McKee Drive
Mahwah, NJ 07430
Phone (201) 529-4880
E-mail:
info.edax@ametek.com
www.edax.com

Art and Layout
Jonathan McMenamin

Contributing Writers
Jonathan McMenamin
Jens Rafaelsen
Bruce Scruggs
Debbie Siples

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