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EDAXinsight

October 2012 Volume 10 Issue 3



NEWS

Welcome to the first issue of EDAX Insight - the successor to EDAX FOCUS

EDAX has been a leading provider of innovative materials characterization systems for many years - in fact, 2012 marks our fiftieth year in business. As part of our celebration of this exciting anniversary, the team has been taking a close look at how we communicate with our partners and customers. Our goal is to ensure that what you see and hear from us accurately reflects the sound reputation we have built over the last fifty years and effectively introduces the key benefits of our new product portfolio.

At EDAX we are focused on providing the best materials analysis systems on the market, allowing you to push the limits of science, wherever you want to take them! Our new Smart Insight campaign is all about making sure that we develop and build our equipment with your materials challenges in mind, so that we can help you to perform analysis projects more quickly and easily. As one of our recent customers said, "EDAX makes me look good". That is our challenge.

We hope that you enjoy both the new **EDAX Insight**, which like the **EDAX FOCUS**, will offer you information about EDAX news, products and solutions, and the other tools of our new campaign. We look forward to powering your next insight!





NEWS

Introducing the Octane Silicon Drift Detector (SDD) Series



Octane Series Silicon Drift Detector

The Octane Series SDDs deliver high-quality EDS data at previously unachievable speeds. Until now, the potential speed advantages of SDD technology have been unrealized due to losses in data quality at high count rates.

With the Octane Series, users are no longer forced to choose between fast data collection and high-quality results. When compared with typical systems on the market, the new design provides stable energy resolution at high collection speeds. This resolution stability translates into higher-quality data at high input count rates. Finally microanalysis experts can benefit from both speed and data quality to maximize their materials insight.



Resolution stability drives increased materials understanding

The Octane Series includes four models - the Pro, Plus, Super, and Ultra - that are designed specifically to meet the demands of key microanalysis applications. Compared with typical systems on the market, the design provides stable energy resolution at high collection speeds, which translates into higher-quality data at high input count rates.



Octane Series SDD Models by Applications

Pairing the new SDD technology with EDAX's TEAM[™] EDS software allows users to take advantage of the Smart Features in TEAM[™] Analysis Systems to optimize their analysis time and get the best data possible from their sample.

The Importance of the Orientation Precision Performance of Electron Backscatter Diffraction

Electron Backscatter Diffraction (EBSD) has become the preferred technique for measuring crystallographic orientations and for quantifying microstructural features such as texture, grain size, and phase distribution in polycrystalline materials. EBSD provides rapid orientation measurements combined with high spatial resolution and excellent orientation precision for characterizing a wide range of materials. However, it is important to recognize that the performance and capabilities of EBSD as a technique are determined not only by the physics of EBSD pattern formation but also by the specific analytical approaches used to capture and analyze EBSD patterns. The TEAM™ Pegasus and TEAMTM EBSD Analysis Systems implement industry-leading EBSD pattern analysis Smart Features that provide enhanced capability to improve microstructural characterization capability.

One analytical approach incorporated in the TEAM[™] Pegasus and EBSD systems is orientation precision. Orientation precision is the spread in repeated measured orientations from a single reference orientation and defines the lower limit of detecting small changes in orientation. Typical estimates of EBSD orientation precision range from 0.5° to 2.0°. These values are often specified as dependent on both camera resolution and camera position. However, TEAM[™] Pegasus and TEAM[™] EBSD Analysis systems, through their innovative EBSD pattern analysis, are able to provide orientation precision values less than 0.1° even at standard EBSD mapping resolutions and camera positions. This unparalleled orientation precision performance allows a more accurate determination and visualization of deformed microstructures, as shown in Figures 1 and 2. In these figures, EBSD maps were collected from deformed ferritic steel with acquisition settings producing orientation precision values less than 0.1° and with settings producing precision values greater than 0.3° from the same area to compare the effects of orientation precision with standard Inverse Pole Figure (IPF) and Kernel Average Misorientation (KAM) maps shown for both values. With the less than 0.1° precision data, the subgrain structure within the grains is clearly defined indicating that dislocation lines have organized during the deformation process as expected. With the greater than 0.3° data, this detail is lost as the orientation precision value approaches the magnitude of the orientation changes within the grains. The resulting measurements produce a much noisier and undefined view of the microstructural changes occurring

during deformation compared to the high orientation precision data. It is important to note that the IPF orientation maps, due to the wide range of orientations they display, do not accurately represent the effects of orientation precision, and this type of map can hide poor orientation precision results.

Understanding deformation is a key application of EBSD. Material fabrication and forming methods - such as solidification, forging, rolling and drawing - strengthening mechanisms - such as strain hardening - and failure modes - such as creep and fatigue all cause some level of deformation. Accurate characterization of this deformation through improved orientation precision allows better understanding of the deformation process. With this insight, optimization and improvements of the process may be realized.



Figure 1. Standard Inverse Pole Figure (IPF) and KAM orientation maps at >0.3°

Figure 2. IPF and Kernel Average Misorientation (KAM) maps at < 0.1°



Improved Trace Element Sensitivity with the New Apollo XRF ML-50 Silicon Drift Detector on the Orbis Micro-XRF Elemental Analyzer

Materials Challenge

The measurement of trace elements is important across a wide variety of materials characterization problems. When measuring small glass fragments collected from crime and accident scenes, forensics experts analyze trace strontium (Sr) and zirconium (Zr) typically unintentionally incorporated into the glass during manufacturing as one point of identification or comparison. Traces of lead (Pb) and cadmium (Cd) are measured in a wide variety of commercial materials for verification of adherence to environmental regulations such as the European Union's Restriction of Hazardous Substances (RoHS) directive. Trace chlorine (Cl) is depth profiled in concrete to quantify ion permeation from deicing agents. In these cases and many others, improvements in detector performance can lead to improvements in micro-XRF sensitivity and faster data collection, thereby providing users with faster, more accurate results.

Comparison with Existing Orbis Detector Solutions

Until now, the Orbis micro-XRF analyzer could be configured with two different X-ray detectors.

- 80 mm² Si{Li} detector
 - Liquid nitrogen cooled
 - Largest active area
 - High spectral resolution
 - Moderate throughput
- Apollo XRF-XL Silicon Drift Detector (SDD)
 - Electrically cooled
 - Moderate active area
 - High spectral resolution
 - High throughput

The Si{Li} detector technology is the most mature XRF detector technology having been developed and commercialized about 50 years ago. Over the last decade or so, XRF detector developments have moved towards electrically cooled SDDs providing comparatively better spectral resolution at significantly higher throughput rates.

The newly launched Apollo XRF-ML-50 detector is an SDD with improved active area yielding a larger solid angle of signal collection while maintaining high spectral resolution and throughput. This allows for improved overall sensitivity and measurement speed in many applications.

Orbis Micro-XRF Results

Improvements in speed and sensitivity of analysis can be achieved with detectors and associated signal processing electronics by improving spectral resolution, enhancing signal collection by size and positioning of the detector or increasing signal processing speed, also referred to as "throughput". The general goal in optimizing and improving these detector parameters is to avoid augmenting one at the sacrifice of the others. For example, it would do little good to increase the detector size while making significant sacrifices in detector resolution or throughput. Such a detector could collect more signal but would be unable to provide a high resolution spectrum or process the additional signal collected. This application note will explain the benefits of the new 50 mm² Apollo XRF-ML50 detector and describe analytical situations in which increased speed and sensitivity can be obtained.

Improvements in speed and sensitivity begin with the amount of sample X-ray signal collected by the detector, which is referred to as the "solid angle" by system designers. Detector solid angle is proportional to the signal collection area of the detector, known as active area, and inversely proportional to the square of the distance between sample and detector as shown in Equation (1).

Detector Solid Angle α (Active Area) / (Distance)² Eqn [1]

In general, augmenting active area leads to larger solid angles and increased X-ray signal collection as long as the distance between sample and detector can be optimized. In Table 1, a comparison of performance parameters is made between the three detectors which are available on the Orbis system.

The structural and performance characteristics of the 30 mm² and 50 mm² SDD detectors are very similar except for the active area while the throughput of the Si{Li} detector is limited.

| Detector | Active Area | Туре | Resolution [eV] | Throughput [kcps] |
|-----------------|----------------|--------|--------------------|----------------------|
| Apollo XRF-XL | 30 mm² | SDD | ~140 | 200 |
| Apollo XRF-ML50 | 50 mm² | SDD | ~140 | 200 |
| 80 mm² Si{Li} | 80 mm² | Si{Li} | <155 | 10 |

Table 1. Comparison of Orbis Detector Structural and Performance Parameters

(Continued on Page 5)

(Continued from Page 4)

The additional solid angle of the 50 mm² detector yields improvements in speed and sensitivity in analytical problems where the 30 mm² detector would be signal starved. When a detector is signal starved, the detector's processing electronics still have available capacity to process more signal. In these cases, having a detector with a greater solid angle allows for more X-ray signal to be passed to the processing electronics. As long as the larger SDD's spectral resolution and throughput are similar to that of the smaller SDD, which is the case here, improvements in speed and sensitivity can be achieved with the larger detector. Typical materials analysis problems which fall into this category include measurements of light element matrices such as glasses, plastics and aluminum alloys; measurements of trace elements where heavier filters are needed for best sensitivity such as trace Pb and Cd in solders and plastics and measuring thin residues, corrosion and coatings on light element matrices. Table 2 compares the limits of detection for several elements spanning the XRF spectral range.

| | | LOD (ppm) | | | |
|-----------------|-------------|-----------|---------|-------|---------|
| Detector | Std/Options | CI(K) | Pb(L) | Sr(K) | Cd(K) |
| Apollo XRF-XL | Standard | 17 | 3 | | 9 |
| Apollo XRF-ML50 | Option | 14 | 2 | 5.5 | 8 |
| 80 mm² Si{Li} | Option | 23 | 3 | 7.5 | 6 |
| Sample Matrix | | Glass | Plastic | Glass | Plastic |

Table 2. Limits of Detection [ppm] for Detectors Available on Orbis

For the data collected in Table 2, the SDD detectors were operating in the signal starved regime where the X-ray tube is run at full power for maximum excitation. Improvements in sensitivity of the 50 mm² over the 30 mm² SDD are approximately consistent with the increase in solid angle of the 50 mm² over the 30 mm² SDD.

In applications where improved sensitivity is not needed, the 50 mm² SDD can be used for increased productivity. The proportionality between sensitivity as described by the Limit of Detection [LOD] and measuring time is shown in Equation [2].

LOD
$$\alpha$$
 (Time)^{-0.5} Eqn [2]

Hence, the increased sensitivity provided by the 50 mm² SDD can be traded for faster measuring time. For example, using Eqn [2] and the improvements in measuring sensitivity shown in Table 2, the 50 mm² SDD can achieve the same measurement sensitivity obtained with the 30 mm² SDD with a nominal 40% reduction in measuring time.

The additional solid angle of the 50 mm² detector yields improved sensitivity and faster measuring times in materials analysis where the 30 mm² detector would be signal starved, but not in cases where the 30 mm² detector is saturated. Saturation refers to when the detector's rate of signal collection exceeds the throughput of the analyzer electronics. Hence, sensitivity and measurement speed are limited by throughput. In this analytical situation, the X-ray tube is generally running below maximum power limiting the input XRF signal to the analyzer throughput. Typical materials analysis problems which fall into this category include measurement of major elements in steels, heavier transition metal alloys and measuring thin residues, corrosion and coatings on substrates comprised of heavier transition metal alloys. Having equal throughputs, both the 30 mm² and 50 mm² SDDs provide similar speed and sensitivity for samples which can saturate the detectors; however, the 50 mm² SDD allows for a greater reduction in X-ray tube power, which in turn can improve tube life.

The data in Table 2 does show that the 80 mm² Si{Li} has better sensitivity for Cd measured at the 23.1 keV K α line. The Si{Li} detecting crystal is about 10 times thicker than the SDD detecting sensor allowing more than three times the absorption efficiency for the Cd(K α) X-ray signal. Hence, the better Si{Li} detection limit for Cd is the result of superior solid angle and absorption at high X-ray energies.

Recommended EDAX Solution

Orbis micro-XRF analyzers with a 50 mm² SDD detector are recommended for engineers and scientists who require the best overall detection limits or faster measurements from a non-destructive elemental analysis technique for measuring small samples or distributional analysis on larger samples. The Orbis with a 30 μ m polycapillary exciting optic can provide highest overall sensitivity on an extremely small measurement area which would be suitable for example in analyzing small glass and metal fragments or electrical contacts in electronics. In addition, the Orbis can be equipped with optional 1 mm and 2 mm collimators to provide measurements over larger sample areas where measurements with a finer spatial resolution are not necessary or where there is a need to properly average the effects of sample inhomogeneity which occurs, for example, in low gold alloys, traces in plastics and mixtures of particles of varying composition.



Worldwide Events

Please visit www.edax.com for a complete list of our tradeshows

2012-2013 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; Tilburg, NL; Wiesbaden, Germany; Japan, and China.

Please visit www.edax.com/support/training/index.aspx for additional information on our training classes.

EUROPE

| EDS Microanalysis | | | |
|--|----------------------|--|--|
| November 5-6, 2012 | Wiesbaden | | |
| November 8-9, 2012 November 20-22, 2012 | Tilburg Tilburg | | |
| EBSD | | | |
| November 7-9, 2012 November 12-14, 2012 | Wiesbaden Tilburg | | |
| EDS & EBSD (Pegasus |) | | |
| November 5-9, 2012 | Wiesbaden | | |
| Orbis: Course & Workshop Presented in English | | | |
| November 26-27, 2012 | Wiesbaden | | |

| EDS Microanalysis November 8-9, 2012 Osaka | |
|---|--|
| November 8-9, 2012 Osaka | |
| | |
| February 14-15, 2013 Tokyo | |
| April 11-12, 2013 Osaka | |
| June 13-14, 2013 Tokyo | |
| July 11-12, 2013 Osaka | |
| October 10-11, 2013 Tokyo | |
| November 14-15, 2013 Osaka | |
| EBSD | |
| December 2012 Tokyo | |
| February 2013 Tokyo | |

CHINA

Particle Analysis

December 4-6, 2012

NORTH AMERICA

| EDS Microanalysis | | | | |
|-----------------------|------------|--|--|--|
| TEAM™ EDS & Genesis | | | | |
| February 5-7, 2013 | Mahwah, NJ | | | |
| TEAM™ EDS | | | | |
| March 11-15, 2013 | Mahwah, NJ | | | |
| July 16-18, 2013 | Draper, UT | | | |
| September 17-19, 2013 | Draper, UT | | | |
| EBSD | | | | |
| August 20-22, 2013 | Mahwah, NJ | | | |
| November 12-14, 2013 | Mahwah, NJ | | | |
| EDS & EBSD (Pegasus) | | | | |
| May 13-17, 2013 | Mahwah, NJ | | | |
| EDS & WDS (Neptune) | | | | |
| November 13-15, 2012 | Mahwah, NJ | | | |
| Micro-XRF | | | | |
| April 9-11, 2013 | Mahwah, NJ | | | |
| October 1-3, 2013 | Mahwah, NJ | | | |

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EMPLOYEE SPOTLIGHT



Lisa Chan

Lisa H. Chan received her bachelor's degree in Chemical Engineering and Materials Science & Engineering from University of California Irvine and her Ph.D. in Materials Science & Engineering from Carnegie Mellon University. While at Carnegie Mellon she started her research on using EBSD to investigate the relationship between grain boundary orientations and intergranular corrosion cracking in aluminum alloys. Lisa then moved on to analyzing grain boundary distributions in three-dimensional microstructures of nickel-based superalloys with a thesis title of "Synthetic three-dimensional voxel-based microstructures that contain annealing twins".

In July 2010, Lisa joined EDAX as Applications Engineer responsible for supporting EBSD and EDS customers. She is also responsible for performing demos, hosting training classes, and testing and developing new software functions. Lisa has attended conferences and held workshops in many places including Argentina, Australia, and Singapore, as all over the United States.

Lisa was born in Hong Kong and grew up in Vancouver, Canada. She currently resides in Nyack, New York with her husband Nicholas. Lisa enjoys visiting New York City for the good restaurants, Broadway shows, and the Christmas markets.



KK Loke

KK Loke joined the EDAX team in February 2011. He is the EDAX Sales Manager for 'Rest of Asia' (ROA) and is located in the Singapore office. KK's responsibilities are managing sales, applications and service for India, Australia, New Zealand, Taiwan and South East Asia.

According to KK, "EDAX has given me the opportunity to work with different people with different cultures as I have to manage the distribution channels in these countries. I graduated with a Diploma from Singapore Polytechnics in Electrical & Electronics Communication."

KK's previous job was as a factory manager with Vonroll Shanghai, a Swiss company, which manufactures insulation materials and copper wire. KK gained his electron microscopy experience working for FEI in Singapore. It was during this time that he had his first meeting with EDAX. He appreciated the high quality & reliability of the EDAX EDS system and this is the main reason he joined EDAX when he was given the opportunity to do so.

KK has been married for 17 years and he and his wife have two sons. His favorite sports are swimming & golf. During his spare time, he goes on regular outings with his family. If you visit Singapore, KK would be happy to hear from you and promises that he will do his best to give you a tour.



CUSTOMER NEWS

School of Earth and Climate Sciences, University of Maine, Orono, ME



EDAX Team[™] Pegasus Analysis System mounted on a TESCAN Vega II XMU SEM and being used by Ph.D. student Deborah Shulman

The School of Earth and Climate Sciences at the University of Maine is located in Orono. It is a four year research university offering B.A., B.S., M.S., and Ph.D. degrees. The school owns an EDAX TEAM[™] Pegasus integrated EDS-EBSD system, which was installed in June 2009. Users of the TEAM[™] Pegasus system include a mix of faculty members, research scientists, graduate students, and undergraduates, and the staff also performs demonstrations for classes and visiting groups.

The primary use of the TEAM[™] Pegasus Analysis System at the University of Maine is for mineralogical studies. These include calculating rotations of deformed minerals, mapping microstructures to calculate bulk mechanical properties, determining modes of weathered material, and characterizing crystallographic orientations within fault rocks. Analyses performed on the system since its installation have resulted in many scientific papers.¹ Data from the system has been included in presentations at Geological Society of America, American Geophysical Union and Gordon Conferences, and in six separate theses.

The School of Earth and Climate Sciences team particularly appreciates the fact that the TEAM[™] Pegasus system gives them the ability to map large areas quickly. They also like having the ability to modify and create their own structure files. "We definitely would recommend EDAX (and have). One of the positive interactions we had was working with the software developers to incorporate an EDS-only phase mapping system into the EBSD software. We benefited from good support early in our use of the instrumentation".

¹Frieman, B., Gerbi, C., and Johnson, S.E., in review, The effect of microstructural and rheological heterogeneity on porphyroblast kinematics and bulk strength in porphyroblastic schists: Tectonophysics.

Price, N.A., Johnson, S.E., Gerbi, C., and West, D.P., Jr., 2012, Identifying deformed pseudotachylyte and its influence on the strength and evolution of a crustal shear zone at the base of the seismogenic zone, Tectonophysics, v. 518-521, p.63-83, doi:10.1016/j.tecto.2011.11.011.

Naus-Thijssen, F.M., Goupee, A., Johnson, S.E., Vel, S., and Gerbi, C., 2011, The influence of crenulation cleavage development on the bulk elastic properties and seismic wave velocities of phyllosilicate-rich rocks, Earth and Planetary Science Letters, v. 311, p. 212-224

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