EDAX FOCUS

The Apollo Silicon Drift Detector (SDD) Series: A Detector for Each Application

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Since the early days of X-ray microanalysis the Si(Li) detector was the only commercially available detector. All types of materials, from stainless steel to organic compounds were investigated with the same type of detector. Currently, the performance of the Si(Li) detector is at its technical limit and incremental improvements are achieved by incorporating the latest electronics advances. Even with changes in signal processing and electronics, the Si(Li) detector specification is approaching its maximum theoretical performance capability.

Today, time is money and the characterization of new materials e.g. nanotechnology, requires faster microanalysis to answer many questions during the development phase. A few years ago, silicon drift Detectors (SDD) were introduced and thought to have the potential for faster microanalysis over Si(Li) detectors. Today, the SDD is a viable alternative to the Si(Li) detector in many applications.

EDAX's Apollo series offers three detectors; Apollo 10, Apollo 40, and Apollo XV. These detectors deliver a resolution comparable or better to the best Si(Li) detectors. Each Apollo detector has a special range of application.

Apollo 10

The Apollo 10 is the legitimate successor to the Si(Li) detector. With its specified resolution of MnK_α 133 eV and C K_α from 65 eV, (Figure 1), this detector can be used for general X-ray microanalysis. The light element performance is comparable with the Si(Li) detector, as shown in the spectra of BN with Apollo 10 (red) and overlaid Si(Li) (black). In the spectra of $CaCO_3$ the resolution of C, O and Ca is marked. Quantification is possible from B K to Am L.

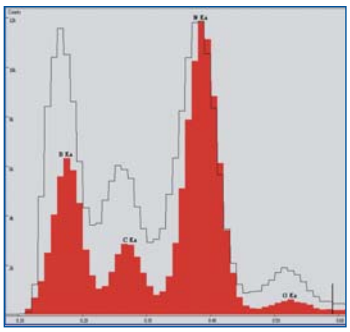

Figure 1: BN spectrum; Apollo 10 red; Si(Li) black line.

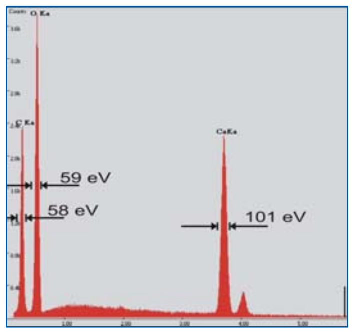

Figure 2: Apollo 10. $CaCO_3$ spectrum; energy resolution marked.

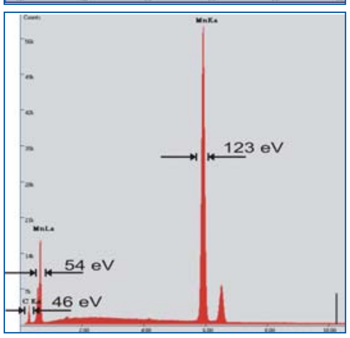

Figure 3: Apollo XV. energy resolution measured on Mn carbon coated.

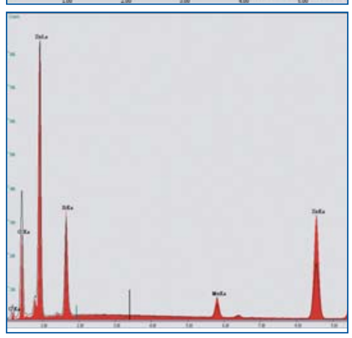

Figure 4: Willemite spectrum; Apollo XV red; Si(Li) black line.

Apollo XV

The Apollo XV is considered to be a "premium" detector and is for advanced analytical applications. With energy resolutions that can reach 123 eV (typical) for MnK_α and 46 eV (typical) for C K_α (see Figure 3) and a peak to background (P/B) ratio of 15000:1, the Apollo XV will work well in the low energy region of the spectrum and at beam energies as low as 3 keV. (Cont'd on Pg. 2)

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The Apollo SDD Series: A Detector for Each Application (Cont'd. from Pg. 1)

The key advantages of the Apollo XV are the lower background, low noise level, and the excellent resolution in the low energy region (Figure 4). The energy resolution is shown in Figure 3. Apart from the good quantitative performance (from Be down to Am) the Apollo XV, in combination with EDAX's new electronics used in the Apex system, is the best option for mappings with more than 100,000 cps. With this performance good maps can be acquired in just two frames. The BSE image in Figure 5 is from a chromium steel sample and in Figure 6 one can see the RGB mixed Nb / Cr / Ni picture of this area.

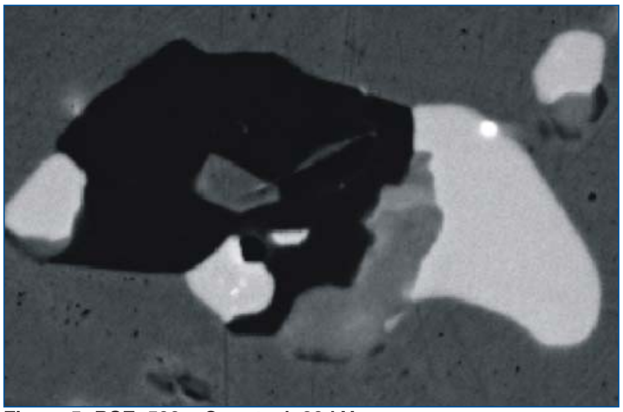

Figure 5: BSE, 500x, Cr - steel, 20 kV.

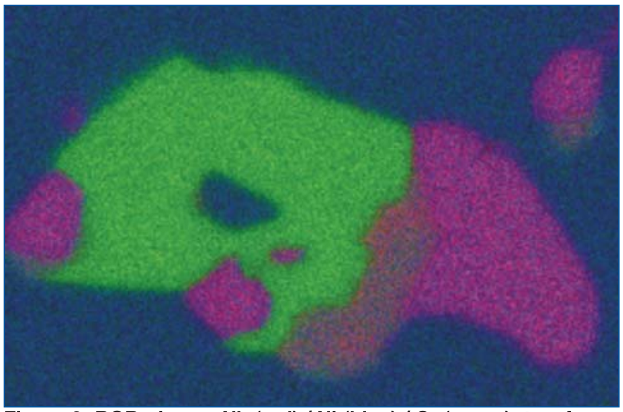

Figure 6: RGB - image Nb (red) / Ni (blue) / Cr (green): two frames.

Apollo 40

The advantage of the Apollo 40, as compared to the standard Si(Li) detector, is the 3x larger active chip area positioned in the same size end cap. These advantages produce a solid angle that will be 3x larger (red vs. green area). With the same size end cap, the SDD chip can be located as near as the Si(Li) detector to the sample, therefore the larger area Apollo 40 detector will have a significantly larger solid angle and higher count rates for the same beam current (see Figure 7).

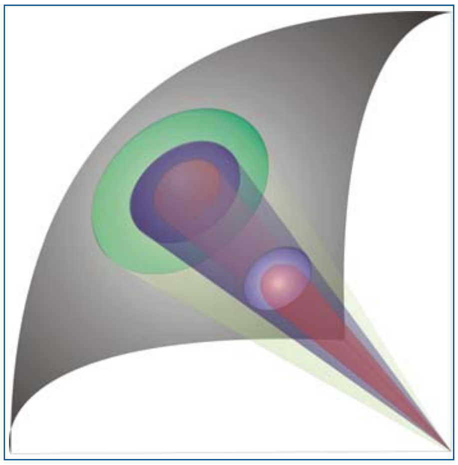

Figure 7: Relationship of active area solid angle: area red / green; distance purple.

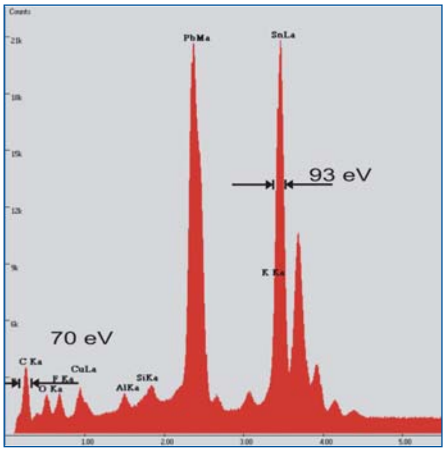

Figure 8: Apollo 40 energy resolution measured on lead solder.

For all applications with low count rates (e.g. cold field emitter SEMs, biological samples) the Apollo 40 is the best choice. The specified resolution for MnK_α is 136 eV and for C K_α 70 eV and is comparable with the Si(Li) detector (see Figure 8).

For the varied performance levels, the three Apollo's SDDs were optimized for different applications and cover a whole range of applications. All three detectors have a tremendous benefit in that they can handle very high count rates (up to 1,000,000) with very good energy resolutions.

Orientation Imaging Microscopy, Comboscan Explained

Orientation Imaging Microscopy, OIM™, is commonly conducted via beam scans. By deflecting the electron beam, the sample can be held in a single position and points can be mapped out in a precisely spaced grid. However, this is limited to the maximum area visible on the microscope. In order to map a larger region, the stage, and thereby the sample, can be moved underneath the pole piece (Figure 1).

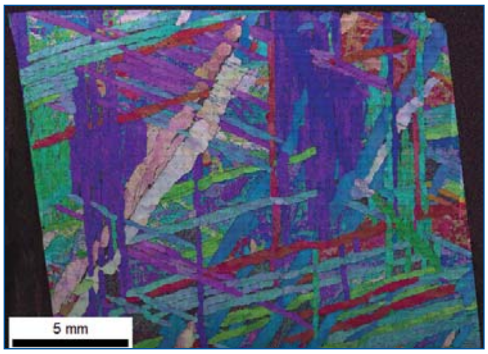

Figure 1: 2x3cm scan of Gibeon Meteorite. Note the IQ variation within each of the 400+ fields of view.

"Comboscan" refers to this combination of beam scan and stage movements. When Comboscan is selected in the OIM™ Data Collection software, the software runs a wizard to aid the user in selecting the top-left starting point, moving the stage, and collecting the bottom-right finishing point. The system will calculate the necessary stage movements to cover the area at the current magnification.

1. The quality of the pattern, as well as the precision of the beam placement, degrade as the beam is deflected over larger areas (see Figure 2) due to the variation in WD as the beam moves up and down a sample tilted at 70°. This effect is particularly noticeable when looking at the borders of scan fields in a Comboscan (Figure 3 – collected at 100x).

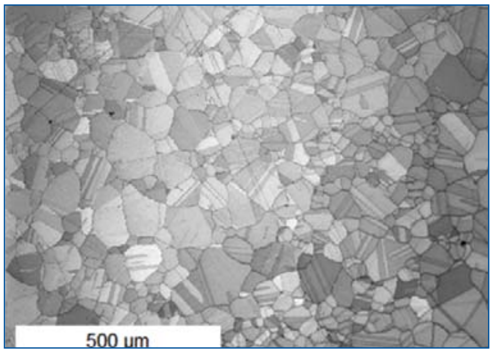

Figure 2: 1.5x1.5µm scan of nickel at 50x. As the beam deflects over larger areas, the WD is effectively lengthened, and the focus is no longer appropriate.

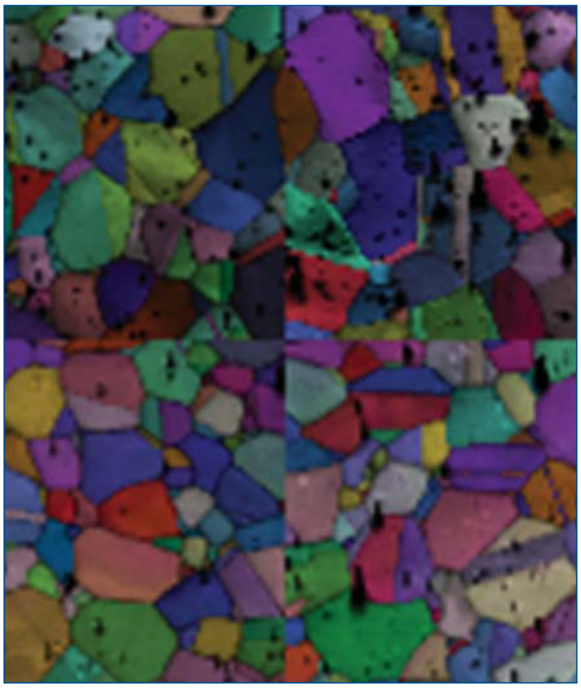

Figure 3: Because the beam is deflected down a tilted sample, the microscope's beam placement is affected, and the corners no longer meet. This example was collected at 50x.

In order to mitigate this effect, all Comboscans should be collected at no less than a 500x magnification, 1000x is recommended for grain size analysis. If there is a minimum magnification for Comboscan, then the user must be conscious of the desired size of the scan. If the scan can be collected with a single beam scan, the single field beam scan method should be used. A Comboscan is only used if the area cannot be mapped inside one field of view.

2. Comboscans will not collect an SEM image with the scan, and therefore it is recommended that a micrograph be collected independent of the scan.

3. Comboscans assume the surface to be planar and parallel to the stage movement. However, in the real world, this is not always the case. The microscope should be focused on the center of the area to be mapped, not necessarily the first or last field of view. This method allows any variations across the surface to be mitigated by using the center of the area.

The New Orbis Micro-XRF Analyzer Series

Building on more than 10 years of Micro-XRF experience, the Orbis spectrometer yields a system with excellent Micro-XRF capability while setting a new standard in analytical flexibility. The Orbis incorporates a unique motorized turret integrating video and X-ray optics allowing coaxial sample view and X-ray analysis. The turret can accommodate two additional collimators along with the X-ray optic for a total of three X-ray beam sizes to expand the Orbis analytical capabilities beyond traditional Micro-XRF analysis. Primary beam filters can be used with all spot sizes available on the turret to allow true XRF analytical capabilities in a micro-spot analysis. The working distance is increased to allow analysis over rougher sample topography without sacrificing signal intensity.

The Orbis is a table-top unit with powerful, easy-to-use analysis software. Orbis users can make elemental analyses on small samples such as particles, fragments and inclusions, or automated multi-point and elemental imaging analysis on larger samples with all the benefits and simplicity of an XRF analyzer. Benefits include:

- Non-destructive measurement
- Minimal sample preparation (e.g. no sample coating is necessary)
- Improved sensitivity for many elements in comparison to SEM/EDS
- Inclusion and coating thickness analysis with the penetrating power of X-rays
- Analysis of wet samples

Small Spot Analysis

Small spot XRF analysis is typically used for analysis of fragments, targeted analysis on larger components and high-resolution elemental imaging. The Orbis system can be equipped with a 30 μm ultra high intensity poly-capillary capable of providing fast, sensitive XRF analysis. As one example, glass fragments are typically analyzed by micro-XRF for criminal and industrial forensic applications. With the Orbis' poly-capillary and primary beam filter system, fast, non-destructive analysis can be done with improved sensitivity at a spot size of 30 to 60 μm (FWHM) depending on the elemental X-ray energy.

In Figure 1, analysis of glass using the 30 μm poly-capillary and primary beam filters for comparative purposes and compositional analysis is shown. Analysis of Cl down to an LOD ~ 25 ppm was achieved using a filter to remove the overlapping Rh(L) tube scatter line.

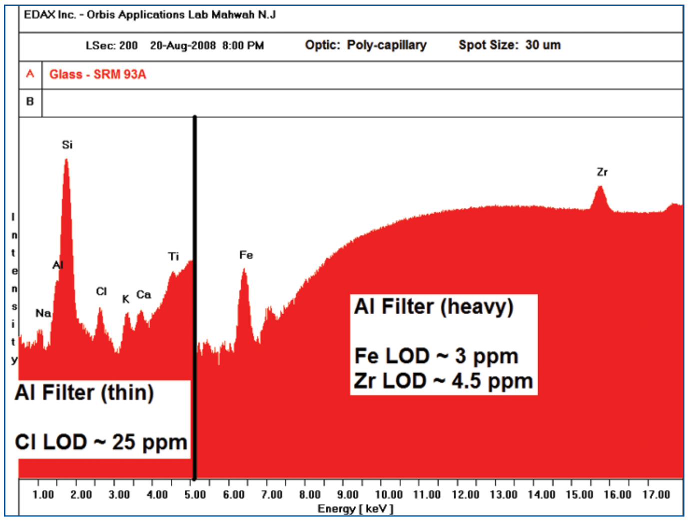

Figure 1: Orbis XRF spectrum (logarithmic intensity scale) of SRM 93A glass using two primary beam filters.

In another example (see Figures 2a and 2b), the Orbis poly-capillary is used to elementally image a portion of a fading black and white photo using the Ag(L) signal to digitize the image. This was done as part of an initial effort to evaluate the suitability of micro-XRF imaging for the archival preservation of Ag-based photos.

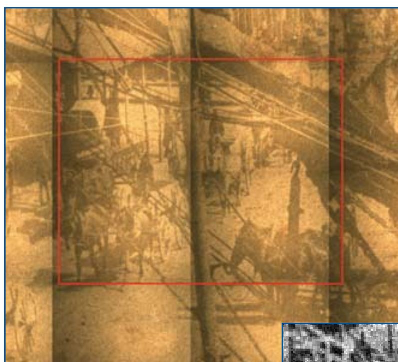

Figure 2a: 10x magnification Orbis video of faded black and white photo. The image is of a horse-drawn wagon on a city street.

Figure 2b: Ag(L) element image of photo using 30 μm poly-capillary at 250 DPI pixel density.

The New Orbis Micro-XRF Analyzer Series (Cont'd. from Pg. 4)

Large Spot Analysis

In a variety of analytical scenarios, XRF analysis using a larger analytical beam diameter is more advantageous than probing the sample with a small beam analysis. Analyzing inhomogeneous materials, for example, with a small probe beam yields information on the level of inhomogeneity of the material while analyzing with a large beam can give a compositional analysis more consistent with the average composition. A larger analytical beam is also useful for screening and searching large areas of a sample for specific elements. Good examples of this come from RoHS analysis and screening.

Plastic components used in electronic products are typically screened for the RoHS hazardous elements and compounds (Cd, Pb, Hg, Cr+6, certain Brominated fire retardants) using XRF. Plastics can be relatively inhomogeneous materials with respect to additive components. Average compositional analysis of trace elements in plastic is simplified by analyzing the sample with a large beam (see Figure 3).

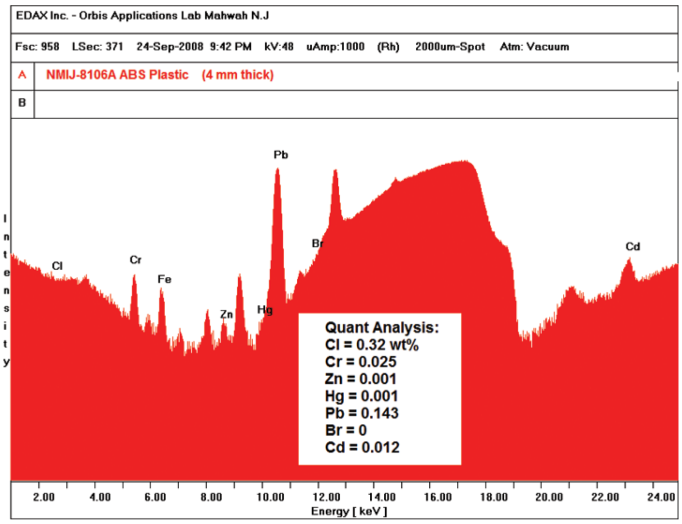

Figure 3: FP 1-standard quantitative analysis of trace elements in a 4 mm thick ABS plastic manufactured as a RoHS standard using a 2 mm X-ray beam diameter.

Using a larger X-ray beam facilitates the speed at which larger multi-component samples can be screened for restricted materials. A typical example is the screening of printed circuit boards for Pb-based solders (see Figure 4). The printed circuit board can be elementally imaged using a larger beam size with lower pixel resolution. Potential problem areas identified by the elemental images can then be analyzed in more detail with a smaller beam size if necessary.

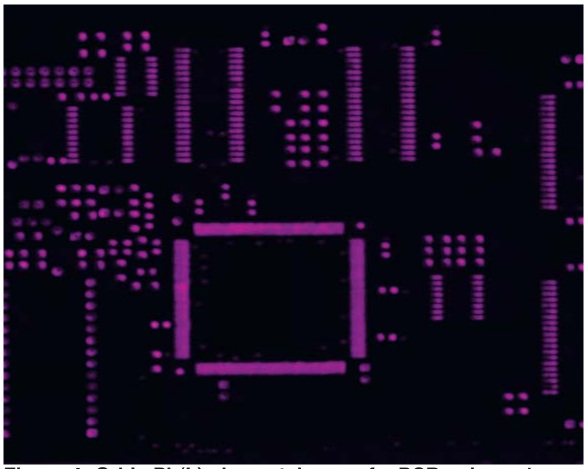

Figure 4: Orbis Pb(L) elemental map of a PCB using a 1 μm collimator. The map indicates the use of Pb-based solder to mount the components on the board.

Conclusion:

The Orbis micro-XRF spectrometer was developed with the mindset of providing a state-of-the-art micro-XRF analyzer with excellent analytical flexibility. The goals of the development were to provide the user with:

- Access to both ultra-high intensity X-ray optics and larger beam collimators
- Primary beam filters at all spot sizes, small and large
- Improved elemental sensitivity

With this analytical flexibility, the Orbis system is capable of providing solutions to a wide variety of problems suited to XRF analysis.

TRAINING AND EVENTS

World-Wide Events

April 20-23	MAS Spring 2009 (Microanalysis of Particles II)	Westmont, IL
May 1-2	NESM (New England Society for Microscopy)	Woods Hole, MA
May 4-7	Scanning	Monterey, CA
May 10-14	EMAS 2009	Gdansk, Poland
May 26-28	Annual meeting of Japanese Society of Microscopy	Sendai, Miyagi, Japan
May 28-29	(SEMS) The Southeastern Microscopy Society	Athens, GA
May 31 - June 12	Lehigh School	Bethlehem, PA
June 8-10	Scandem	Reykjavik, Iceland
June 17-19	MSC/SMC (36th annual Meeting of the Microscopical Society of Canada)	Winnipeg, MB, Canada
July 27-30	Denver X-ray Conference	Colorado Springs, CO
July 27-30	M&M (Microscopy & Microanalysis)	Richmond, VA
August 25-29	THERMEC 2009 (International Conference on Processing & Manufacturing of Advanced Materials)	Berlin, Germany
August 30 - September 4	Microscopy Conference (MC) 2009	Graz, Austria
September 2-4	JAIMA Show	Makuhari, Japan
September 8-11	Electron Microscopy of Analysis Group (EMAG) 2009	Sheffield, UK
September 14-18	The 20th International Congress on X-ray Optics and Microanalysis (ICXOM20)	Karlsruhe, Germany
October 25 - 29	MS&T (The Materials Sci. & Tech. Expo.) Formerly called ASM	Pittsburgh, PA
December 1-3	MRS (Materials Research Society)	Boston, MA

***Please see our website, www.edax.com for a complete list of our tradeshows

World-Wide 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, we organize a number of Operator Courses at the EDAX facilities in North America; Tilburg, NL; Wiesbaden, Germany; and Japan.

Europe

Tilburg = (T) (in English)
Wiesbaden = (W) (in German unless stated otherwise):

Microanalysis Courses: 3-4 Day
- ◆ June 22-25 (T)
- ◆ September 28 - October 1 (T)
- ◆ November 17-26 (W)
- ◆ November 23-26 (T)

2 Day
- ◆ June 18-19 (T)
- ◆ September 17-18 (T)
- ◆ November 12-13 (T)

LEX Course: 3 Day
- ◆ March 31- April 2 (T)
- ◆ October 13-15 (T)

TEX Course: 2 Day
- ◆ June 23-24 (W)

Pegasus Courses:
- ◆ May 11-15 (W)
- ◆ June 15-19 (T)
- ◆ September 14-18 (T)
- ◆ October 19-23 (W)

OIM™(EBSD) Courses:
- ◆ June 15-17 (T)
- ◆ September 14-16 (T)

Japan

Microanalysis Courses:

◆ April 8-10	Osaka
◆ June 3-5	Tokyo
◆ July 1-3	Osaka
◆ October 7-9	Tokyo
◆ November 11-13	Osaka

For more information on our training classes, please visit our website at: **www.edax.com/service/user.cfm**

North America

Microanalysis Courses:

◆ April 27 - May 1	Mahwah, NJ
◆ June 22-26	Mahwah, NJ
◆ July 14-16	Draper, UT
◆ August 10-14	Mahwah, NJ
◆ September 14-18	Mahwah, NJ
◆ October 19-23	Mahwah, NJ
◆ December 7-11	Mahwah, NJ

Particle Course:

◆ May 12-14	Mahwah, NJ
◆ November 10-12	Mahwah, NJ

EBSD OIM™ Academy Course:

◆ April 28-30	Mahwah, NJ
◆ September 1-3	Mahwah, NJ
◆ October 13-15	Draper, UT

Micro-XRF Course:
- ◆ April 14-16 (Eagle/Orbis)
- ◆ October 6-8 (Orbis only)

EMPLOYEE SPOTLIGHT

Dennis Puglia joined EDAX in April of 2000 and performs the role of Field Service Engineer from Boston, Massachusetts. He is assigned to support the Northeast Region of the Americas including Canada.

Dennis began his career in the US Army learning about electronics in their Missile Guidance Systems schools. Upon his completion of duties with the Army he knew his passion was helping customers with technical issues. Dennis landed a job with Dictaphone and was promoted to Assistant Service Manager for support of their electronic products.

In the mid 1980's, Dennis found a new interest working in the X-ray field. This is where Dennis gained over 15 years of EDS customer support experience working with Kevex. His unique experience during this stage of his career has proven to be an asset in support of our customers.

During his free time, Dennis enjoys traveling with his wife Nancy as well as cruising the Caribbean Islands on vacation.

Christiane Lettmann is the Sales & Office Assistant for the EDAX office in Wiesbaden, Germany. Christiane was born and raised near Heilbronn, Germany. After receiving her university-entrance-diploma she made a special apprenticeship as a department manager in a big German store where she worked until she has started a family.

In 2000 Christiane joined Röntgenanalytik Messtechnik GmbH as an office assistant and in October 2003 she joined the EDAX team, responsible for Germany, Austria, and Switzerland. Christiane´s duties and responsibilities have grown to sales support, service scheduling, order processing, and numerous other duties.

Christiane and her husband will be celebrating their 25th anniversary this year. They have two adult children, Jonas (23) and Alisa (20).

Christiane enjoys visiting the palace and famous gardens in Germany or Great Britain. She also enjoys gardening and vacationing on the Baltic Sea as well as spending time with her family.

Netherlands Gemmological Laboratory, Leiden, the Netherlands

The Netherlands Gemmological Laboratory (NGL) is owned by Naturalis, the National Museum of Natural History, in Leiden, the Netherlands. It offers objective and scientifically sound identification and grading services to jewelers, auction houses, dealers, and consumers. Services are available for diamonds, colored gemstones and pearls.

Diamonds are graded according to international standards based on carat, color, clarity and the quality of the cut. The assessed value of a particular stone is related to the grade of the stone. Diamonds and other gems, like rubies, sapphires and emeralds, are frequently treated in order to enhance their appearance. They can also be synthesized by modern manufacturing processes or substituted by lower cost materials of similar appearance (e.g. cubic zirconia for diamond). The provenance of a particular gemstone, whether it is natural, cosmetically treated, synthetic, or an imitation, will significantly impact its value.

In the past, the use of relatively simple optical instruments, such as a refractometer, polariscope, and microscope were generally sufficient to distinguish between the various categories of gemstones. However, with the development of synthesis techniques and cosmetic treatments, a modern laboratory also needs more sophisticated measuring technologies, e.g. Ultraviolet-Visible (UV-VIS) and Fourier Transform Infrared (FTIR) spectrometers to measure absorption/transmission characteristics, as well as energy dispersive X-ray Fluorescence (EDXRF) for non-destructive elemental analysis. Given the potential value of the gemstone submitted for analysis, non-destructive measuring technologies are critical.

As an example of NGL's capabilities, the NGL is often asked to distinguish between natural and synthetic sapphire which is sometimes difficult by traditional methods. Elemental analysis by micro-EDXRF often provides convincing evidence, pointing to the sapphire's origin. Some synthetic sapphires contain traces of molybdenum in much higher concentrations than natural sapphires. These synthetic sapphires were grown in a high-concentration molybdenum flux. In another type of synthetic sapphire, chromium and nickel are the source of the color, instead of iron and titanium, which are most often the source of the color in natural blue sapphire. In addition to differentiating natural and synthetic stones, micro-EDXRF can yield supporting evidence for the provenance (country of origin) of gemstones, as well as for cosmetic treatments (e.g. diffusion of foreign chemical elements, coatings, glass filling) and can assist in distinguishing freshwater from saltwater pearls.

For years, the NGL used the first generation EDAX Eagle micro-EDXRF analyzer with great satisfaction, due to its user-friendly interface, high speed measurements (i.e. non-destructive; no sample preparation), accuracy of semi-quantitative results, trace element measuring capability, and the possibility to quickly scan small inclusions that reach the surface of the gem material. (These inclusions may reveal the nature and/or provenance of the gem.) NGL has also had very good experience with EDAX service support. When it came time to replace the old system, NGL did not think long before deciding to purchase the next generation Orbis micro-EDXRF analyzer from EDAX. Although NGL has only worked with the system since January, it is clear that the Orbis design has surpassed the previous generations of EDAX micro-EDXRF systems. It is remarkably versatile, with a built-in multiple X-ray optic turret and no compromise in analytical speed. Improved software is much in line with previous versions, which together with a dramatically improved video camera system makes the Orbis more user-friendly with regard to NGL's applications and surpasses NGL's expectations.

Article courtesy of Dr. Hanco Zwaan

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EDAX advanced microanalysis solutions
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