SEMs AND FORENSIC SCIENCE

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ABSTRACT: One of the latest developments in microscopy is the Environmental Scanning Electron Microscope (ESEM). The ESEM technology allows the microscope to operate in three modes: high vacuum typically at 10^{-4} mbar, low vacuum typically at 0.1 to 1.0 Tr and ESEM vacuum typically between 1 and 10 mbar. ESEM technology combines three microscopy modes in one instrument and therefore allows the widest range of samples to be investigated.

ESEM allows imaging and analysis of uncoated, otherwise vacuum unstable, specimens. The ESEM microscope offers the possibility to image the specimen surface in a gaseous environment (using water vapor). Imaging is done by employing backscatter detection, for atomic number contrast, or the dedicated GSED (Gaseous Secondary Electron Detector) for secondary electron imaging giving topographic information. Backscatter imaging (BSI) will show elemental contrast, therefore showing difference between materials. Both detectors are commonly used in forensic investigations and can be run simultaneously.

The SEM is very suitable for imaging non-conducting materials at high kV. For many materials such as plastics, polymers, glass, wood, paints, fibers, hairs, fingerprints, insects, etc., the ESEM allows these types of specimen to be viewed in the natural uncoated state. The ESEM can also be used as traditional high vacuum SEM without changing any mechanical set-up. The software automatically controls the transition from low vacuum to high vacuum mode of operation. The third mode of operation in the range 1 to 10 mbar is very useful for delicate samples such as pollen and fungi, wet samples, dirty and oily samples and strongly outgassing materials such as shoes.

KEY WORDS: ESEM; Specimen analysis.

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INTRODUCTION

Scanning electron microscopy has over the last few years become an indispensable tool for forensic investigation, allowing a focus on poorly visible or invisible microscopic traces of evidence by imaging, image comparison and X-ray micro-analysis. Since crime is a part of every day's world, the samples for a forensic lab come from a very wide range of micro-materials. Re-

cently, microscopic techniques have been improved to allow a far better match to the wide range of samples.

Key in the forensic investigation is the validity of the proof (accuracy), the non-destructiveness of the measurement method, and the speed of the analysis. Because of the nature of forensic samples, any changes are undesirable because of their value as evidence in the proceedings, as well as complexity and reproducibility of examination. Here, our forensic SEM is a good solution, since one of the key technologies for this instrument is ESEM: samples require no preparation, and can be examined in their natural state. Traditional techniques such as SE and BS imaging and X-ray analysis can also be used on all available modes of operation. Even non-conducting, out-gassing, dirty, oily or wet samples can be examined non-destructively: no coating, no cutting, no drying, no cleaning, no manipulation (see chapter "Instrument technology components").

One of the most attractive features is the wide variety of forensic applications for a single instrument. The forensic SEM can help detect forgeries, and identify the force or instrument which caused textile damage. It can also analyse pollen left on peoples' clothes, as well as paint chips at the scene of a crime, and even tell whether a car's headlights were on at the time of an accident. Special software can add major functions like highly-automated GSR (gunshot residue analysis). Here, the SEM can give detailed information of the explosive and primer batch to match residues found on the clothes and skin of people who have fired guns.

SEM has a much greater depth of field than optical microscopy, and can hence provide much clearer images of fibres, hairs, bullets, cartridge cases and other items that do not have a flat surface. Similarly, it is much more suitable than optical microscopes for studying shoeprints and similar clues.

DETECTING FORGERIES

Examination of handwritten documents can determine which of the two intersecting lines was drawn first, in order to detect forgeries.

The main problem with imaging un-coated paper surfaces is the charging effect. Paper is non-conductive and therefore in the past it was coated with a metal layer to eliminate charging at high kV. After coating, written lines can only be imaged if the imprint has changed the topography of the paper. Today, instead of using a high kV for imaging, an extreme low voltage (0.7 and 2.0 keV) is used and this allows imaging of paper in its natural state.

The image shows two ballpoint lines on a photocopied document. The left ballpoint line is clearly interrupted by the letters from the document, indicating that the ballpoint line was draw first. The second ballpoint line, on

the right was drawn afterwards, because the ink of the ballpoint line is covering the letters from the document. This can show if documents have been altered after the original has been made.

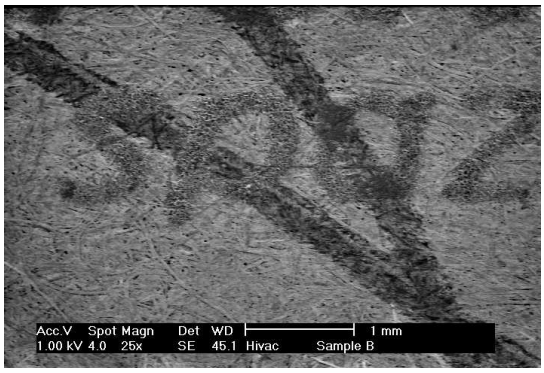

Fig. 1. Uncoated paper sample, showing different ink traces on a document.

So, the forensic SEM is ideal for investigating uncoated paper. In combination with the different imaging possibilities, such as SE and BSE signals, together with the signal mixing and image processing technique, the SEM allows the solution of intersection problems to trace and prove forgeries in documentation.

DISTINGUISHING THE CAUSE OF TEXTILE FIBER DAMAGE

Forensic investigations of fibers by use of SEM can be useful to determine the cause of textile damage like tears, cuts, fiber fractures, and bites. Earlier studies [1] were performed to reveal a method whether a SEM procedure could be a reliable tool in distinguishing between cuts and tears, and then to identify the force or instrument causing the damage, e.g. scissors or knives.

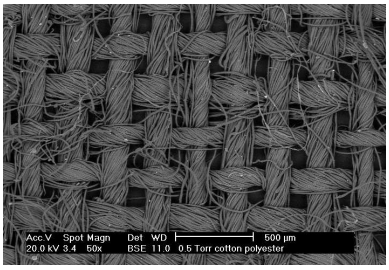

Fig. 2. BSE image, showing small particles.

Up to now imaging of fibers or yarn by use of scanning electron microscopy was only possible by coating the sample with a conductive layer. Since clothing, especially after a crime is committed, often is damaged or covered by dirt or blood, imaging can still be a problem even after coating, and this will result in blurry images (charging of the specimen).

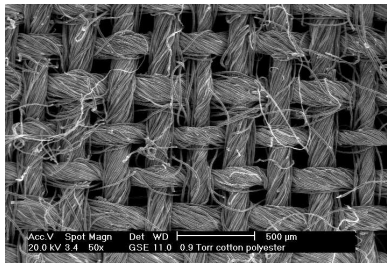

Fig. 3. GSE image, showing the fibre structure.

Using new SEM techniques that allow gas in the chamber to reduce charging, in combination with newly designed dedi-

cated detectors to work under low vacuum conditions, make it possible to image these fibres charge free, without any sample manipulation technique, like coating the sample. Therefore these samples can be imaged in their natural state.

Back-scatter imaging (BSI) will show elemental contrast, therefore showing difference between materials, like gunshot residue particles or debris on the sample (see upper image). This detector is normally available on most low vacuum instruments. The new imaging technique, gaseous secondary electron imaging (GSE) in a low vacuum pressure regime allows to show topographic information on uncoated samples (see lower image). These images will show much more surface detail than using the BSI technique, which in turn will help to distinguish the cause of textile fiber damaging. In other words, the SE image will give the information to reveal the details necessary to understand the cause of textile damage. This technique also allows to image large samples, therefore no individual fiber preparation is necessary.

INVESTIGATING PAINT CHIPS

During car accidents often small paint chips are left behind at the (crime) scene. These small paint chips can be investigated by use of SEM-EDX to determine the chemical composition of each individual paint layer in order to determine the color and manufacturer of the car.

Paint fragments also often are left behind as evidence in connection with burglaries where they may be scraped from doors and windows on entry or exit from the premises.

The use of the SEM in comparative mode, for examples by using 2 monitors or picture in picture features, allows investigation of multiple flakes. The comparative analysis shows whether the samples are identical or whether significant differences exist between the samples.

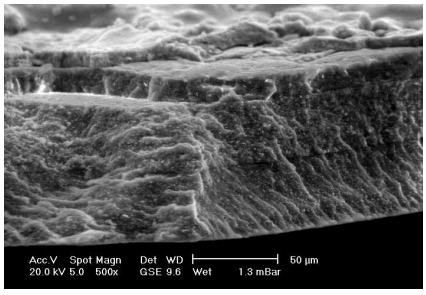

Fig. 4. GSE image, showing topographic information.

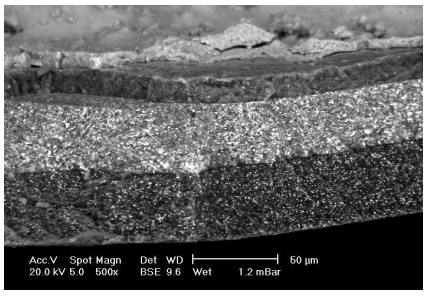

Fig. 5. BSE image, showing elemental composition of the layers.

Using the scanning electron microscope in a low pressure regime, again it is possible to look at paint chips, by using the GSE detector details with large depth of focus. In combination with BS imaging, atomic number contrast will clearly show the difference between the layers. Using EDX microanalysis under low vacuum conditions will show the chemical composition of the small layers (see EDX spectrum of the first layer). The accuracy of the EDX spectrum is optimized by way of the excellent ESEM geometry that reduces any residual scattering of electrons and hence offers the highest accuracy.

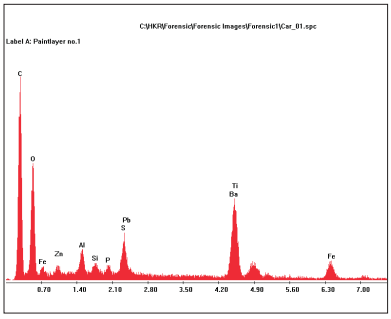

Fig. 6. EDX spectrum of one of the layers.

INVESTIGATING BULB FILAMENTS

The evaluation of vehicle lighting conditions at the time of an accident is an important task that is accomplished by examination of lamp and lamp-remains. Besides optical microscopy, scanning electron microscopy is a growing analytical technique due to its high resolution, large depth of focus and the EDX microanalysis option.

In traffic accidents at night it is important to establish whether headlamps and rear lights were on or off at the moment of the impact. Daytime accidents can bring up questions as to whether the direction indicators of a car were activated.

The effect of impacts on bulbs was studied [2] earlier. It will be dependent on the status of the filament (hot, cold) and whether or not the envelope of the filament was broken.

In case of a broken envelope at time of the impact, in combination with a hot filament, often the oxidation of the wire takes places. Also small spherical glass particles can be found on the filament. On hitting the hot filament glass

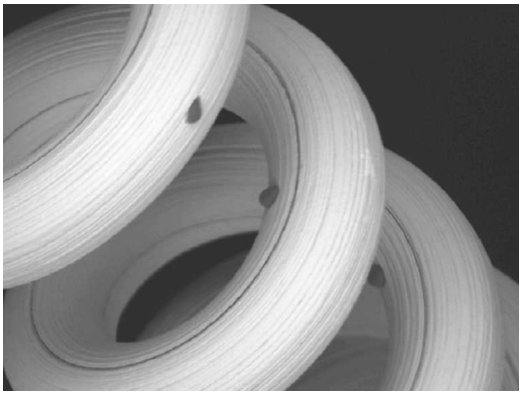

Fig. 7. BSE image of a filament showing small melted glass fragments.

fragments will melt and form spheres or droplets.

By carefully investigating the back-scatter image, one can see that the small particles are gray in comparison with the lighter tungsten wire. This indicates a lower mean atomic number of the particle.

Using the EDX microanalysis in spot mode will show the chemical composition of the small particles within seconds. Since the glass spheres on the wire are not electrically conductive, a metal coating is required on the sample. By using ESEM technology it is possible to investigate these types of samples without any sample preparation.

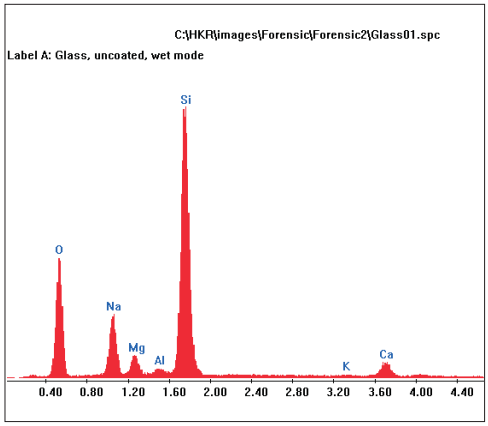

Fig. 8. EDX spectrum of the uncoated glass fragments.

INVESTIGATING CARTRIDGE CASES

In forensic science a direct comparison of items is often very helpful. These may be fingerprints, marks made by implements, fibers, hairs, bullets and cartridge cases.

Many of these items do not posses a flat surface, therefore examination under an optical microscope is often difficult. Because the SEM has a great depth of field it can produce images covering a wide range of magnifications and depths on many types of surfaces, including rough and curved surfaces.

Fig. 9. SEM image of a cartridge case, revealing more depth of focus.

When comparing for example two cartridge cases simultaneously in the SEM a reference image is first obtained from one of the cartridge cases and is stored digitally. Then the second cartridge case can be scanned. By use of rotating the second sample one can carefully position the shell and the recorded image can be overlaid with the reference image, allowing the two samples to be accurately compared. The possibility to image the samples at

extreme low magnification, up to 5×, will turn the SEM into a versatile imaging tool.

Since the SEM is equipped with fixed chamber geometry, all detectors are always on the same position, resulting in series of images from samples with the same illumination. Especially with bullets and cartridge cases this will result in excellent images showing tool marks from firing pins, extractors or ejectors, in case of pistols and submachine guns (SMG).

Since bullets and cartridge cases contain a large amount of firearms residue, such as primer decombustion particles, also known as GSR, precaution has to be taken not to contaminate the instrument when dedicated GSR analysis is required in the future. Ultra sonic cleaning could be a solution.

INVESTIGATING GUNSHOT RESIDUE PARTICLES

In forensic sciences, gunshot residue analysis is an important issue. The analysis of gunshot residue can be performed through dedicated GSR software ensuring reliable, fast and fully unattended analysis, even at an overnight period of time. The software also identifies other indicative particles like the particles, which originates from the bullet, containing a large amount of lead (Pb). Other particles of interest are titanium (Ti) and zinc (Zn) as found in lead free ammunition. Also other more environmental related particles can be characterized by the system to provide additional information about the suspect and crime scene.

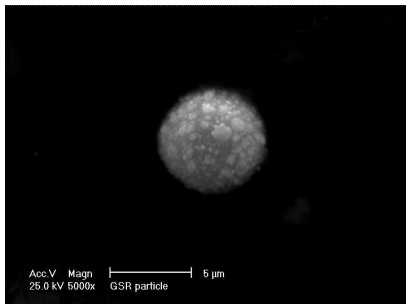

Fig. 10. GSR particle.

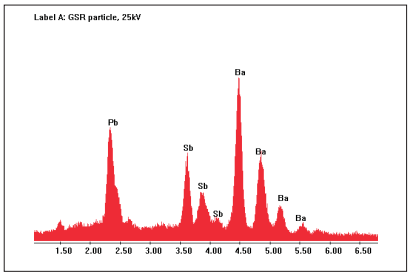

Fig. 11. EDX spectrum of a typical 3 component GSR particle.

The particles are taken from the suspect normally by using a tape lift method. By this way particles are transferred from the area of interest onto a small sample for further examination in the SEM. The small stub area that needs to be examined is split into multiple fields and each field is scanned one by one. The back-scatter electron signal is used to detect the particles. Typically GSR appears as bright particles within the image and by applying a threshold to the signal, the particles are distinguished from the back

ground and their actual position is recorded. Then the electron beam to perform an X-ray analysis will revisit the particle. The emitted X-ray spectrum from each individual particle is compared to a classification scheme. This scheme consists out of maximum 256 individual user definable classes. The GSR software employs the latest advances in X-ray spectrum processing techniques by combining region of interest (ROI), spectrum stripping routines and rule-based (intelligent) analysis methods. These methods provide the flexibility needed to correctly interpret the spectra from inhomogeneous, often irregular and small particles. All results are stored and a dedicated report of all detected particles is produced after each sample.

Each detected particle can be revisited very easily. The accurate motorized stage will drive to the field of interest and the selected particle will be centered and automatically magnified. Positioning the beam on the particle to manually confirm the presence of specific elements is now very easy. Fast report generation is a logic capability of the system.

INVESTIGATING GYM-SHOE DEBRIS

One of the aspects of identification of a crime scene can be related to finding small particles on clothing of a suspect. In particular shoes can be of interest because the foot print they may leave behind in the soft earth around the scene. From this foot print, the type of shoe can be identified and the characteristic wear is specific for the persons way of walking.

In addition small particles present at the scene may stick to the shoe and hence identification of these particles may add to the proof that the suspect has been at the scene of the crime. The main difficulty with analysis of small particles is that the shoe is out-gassing enormously and hence the SEM must be able to handle this.

Fig. 12. CCD camera overview image of a gym-shoe.

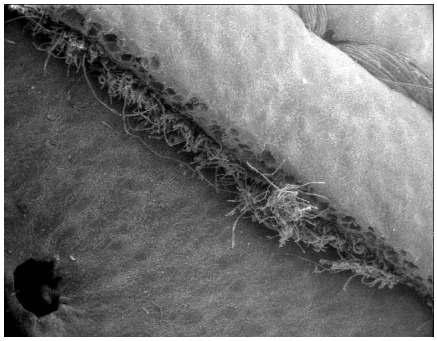

Fig. 13. Details of shoe construction materials.

Instead of tape lift techniques, a non-destructive technique can be used, leaving the proof material in its original state. Of course, also here, precaution has to be taken not to contaminate the microscope.

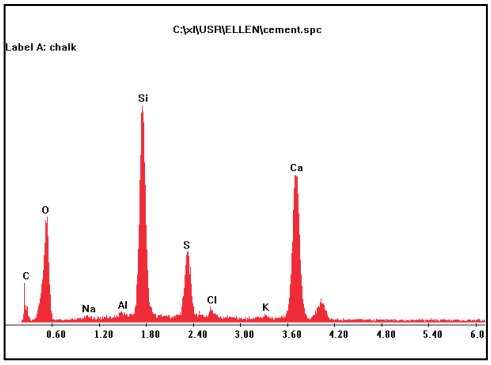

Fig. 14. EDX spectrum of some small particles on the shoe.

In the ESEM this kind of out-gassing specimens is not a problem, since it can operate at a relative high pressure. Also the imaging at this high pressure shows very good detail of the construction material of the shoe. A part of the shoe may show some debris particles and, even though the material is completely uncoated, these particles as shown in the top left corner can be identified using EDX. In this case chalk components Ca-S-Si-O are clearly present.

FRESH POLLEN ON CLOTHES

When a person moves through a bush or a natural landscape, he may touch flowers, a tree, or small plants on the ground. In general the flower will "drop" some pollen that get stuck to the clothing and hence can serve as a micro-trace of a persons presence. Depending of the type of pollen and the weather condition, these small traces can be used in an investigation. Imaging of pollen can be very delicate and some of them may dry out. In those cases coating of these kind of specimens is out of the question and good SE images are necessary to determine its species and the stadium of development.

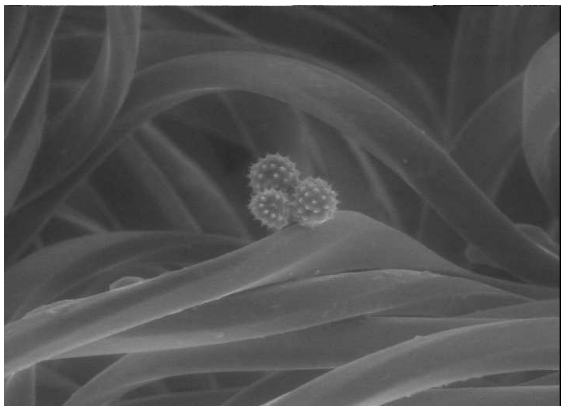

Fig. 15. Fresh pollen on fabric (GSE image).

Not only is the pollen non-conductive, but in general the clothing itself is non conducting and will show great depth variations. Depending on the type of pollen (size) and the "matrix" structure of the

clothing the pollen may be well on top or as shown in the image lying in the actual tissue. A good depth of focus and a good quality secondary image can reveal the fine structure of the pollen.

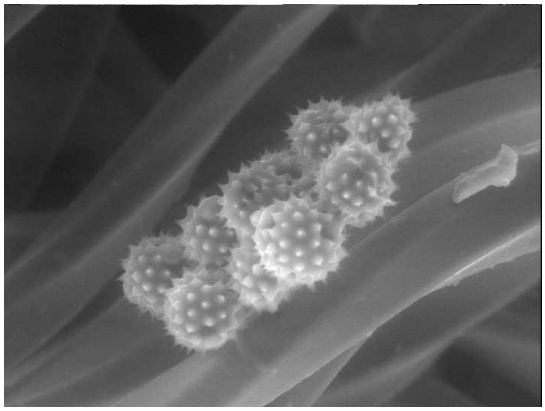

Fig. 16. Fresh pollen on fabric (GSE image).

The daffodil pollen as shown in higher magnification images are quite strong and could stand even a high vacuum condition for a short period of time. However, using ESEM technology all types of delicate pollen can easily be examined without damage, drying out or charging up.

INSTRUMENT TECHNOLOGY COMPONENT: ESEM

One of the latest developments in microscopy is the Environmental Scanning Electron Microscope (ESEM). The ESEM technology allows the microscope to operate in three modes: high vacuum typically at 10^{-4} mbar, low vacuum typically at 0.1 to 1.3 mbar (0.1 –1.0 Tr) and ESEM vacuum typically between 1–13 mbar (1–10 Tr). ESEM technology combines three microscopy modes in one instrument and therefore allows the widest range of samples to be investigated.

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vacuum mode of operation. The third mode of operation in the range 1 to 13 mBar is very useful for delicate samples such as pollen and fungi, wet samples, dirty and oily samples and strongly outgassing materials such as shoes.

INSTRUMENT TECHNOLOGY COMPONENT: SW CONTROL

As a result of computer control through the MS-Windows operating environment, the system is very easy to operate. The data are compatible with very common word and data processing programs. Also network printing and image transfer is a natural extension of the system. The microscope is standardly equipped with an image archiving system, called XL-Docu. The archiving software can be very useful in relation to comparing images made from bullets and shells. All data collected during image acquisition is saved automatically along with the image. One can set up own databases and images can be stored under specific classes, also images made by a light microscope. For documentation purposes, XL-Docu supports all kinds of document types and many overlay and annotation functions.

INSTRUMENT TECHNOLOGY COMPONENT: STAGE AND SPECIMEN HANDLING

The SEM can be equipped with a eucentric goniometer stage, 4 axis motorised (x, y, r, z) with full manual override and an xy-movement of 50 × 50 mm. Integrated readout on-screen of all movements for this high precision motorized stage is standard. The z-movement is also fully motorized, allowing each sample to be moved carefully to the field of interest. The z-movement is 45 mm (25 mm motorized). For ease of use a comp-eucentric rotation is included in the software.

In case of the need for investigating large samples the SEM can be equipped with a 5-axis motorized (*x, y, r, z, t*) stage with large movement of 100 × 100 mm in *x* and *y* direction. The z movement of this stage is about 65 mm (fully motorized), therefore especially suitable for comparison measurement like bullets and cartridges.

After specimen change the microscope will prompt for an image in focus. In this way the actual free working distance will be coupled to the z-movement, resulting in a security for sample handling. Therefore practically no damage can be done to the specimen in case of specimen movements. For investigating bullets and cartridge cases a special ballistics comparison stage can be installed. This ballistic stage allows investigation of two cartridges at

the same time by means of rotation. For investigation of GSR samples, special holders are available to support 16 small, half-inch aluminum-stubs, for automatic unattended overnight runs. The high precision motor stage allows easy revisiting and auto centering of individual particles. The stage shows a very low wear, meaning that this high accuracy will remain in tact for several years, without loss of specification.

INSTRUMENT TECHNOLOGY COMPONENT: MICRO-ANALYSIS

X-ray microanalysis performed with the SEM also has entered a new time frame. The EDX software is fully embedded with single keyboard and mouse operation, which in turn will result for the user in an unsurpassed ease of use and fully automatic exchange of error free data. The microscope can be directly controlled from within the EDX software, without calling sub-windows or sub-programs, that may clutter up the viewing screen.

Stability of the system is high and does not require regular calibration. The EDX detectors used on the SEM are very sensitive to low energy X-rays as from carbon, oxygen or nitrogen. The thin window material can stand regular, routine venting of the microscope chamber as well as pressures present in the ESEM. All detectors are safety locked for the presence of liquid nitrogen.

Microanalysis nowadays is very simple. Fast and reliable automatic identification of all elements present in the sample. For further confirmation of trace elements, the manual peak ID can be used, which in turn shows with clear colored markers the position of the expected X-ray element line. Quantification of the acquired data is easy. The software works fully standardless, therefore no difficult procedures need to be followed. Thanks to the design of the system, it is possible to do high accuracy EDX analysis in the low vacuum mode of the system. The unique vacuum design includes very much attention to minimizing the beam gas path length and this ensures a minimum beam skirt and hence gives the best EDX results on small particles, resulting in accurate chemical composition of the sample under all circumstances.

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