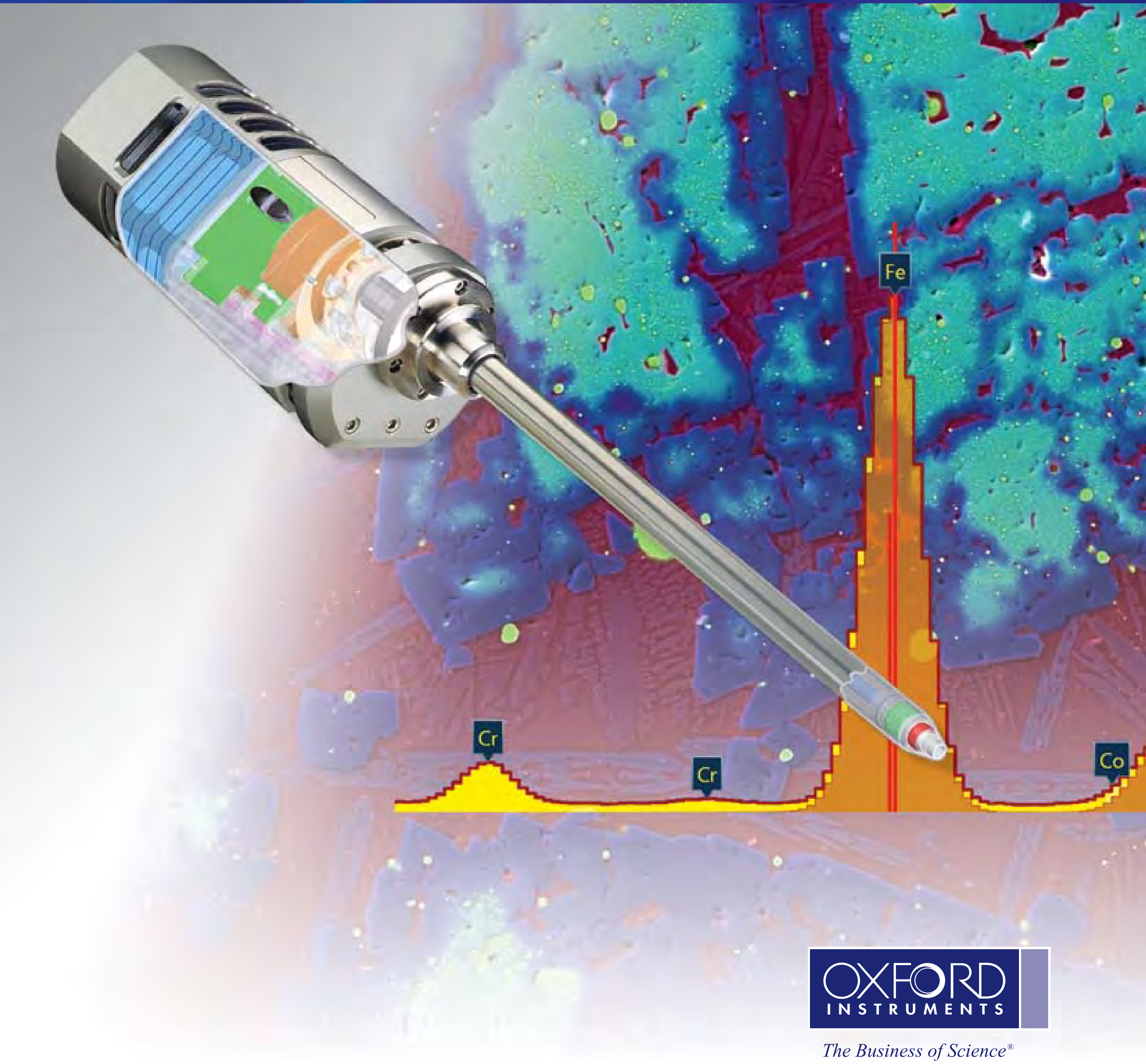
Silicon Drift Detectors Explained



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1.0 Introduction

EDS (Energy Dispersive Spectroscopy) is an established technique used to characterise the elemental composition of a sample under the beam of an electron microscope. The technique exploits the fact that an X-ray is generated when an orbiting electron is displaced by an electron of the microscope beam (Fig. 1). Analysis of the X-ray energy, a fundamental characteristic of an element, then leads to the identification of the element.

A material is characterised at a single point by a value showing the ratios of elements (so-called 'quant'), or, with an array of points over the sample by a so-called elemental map that shows the individual elemental concentrations as a series of colour-coded images.

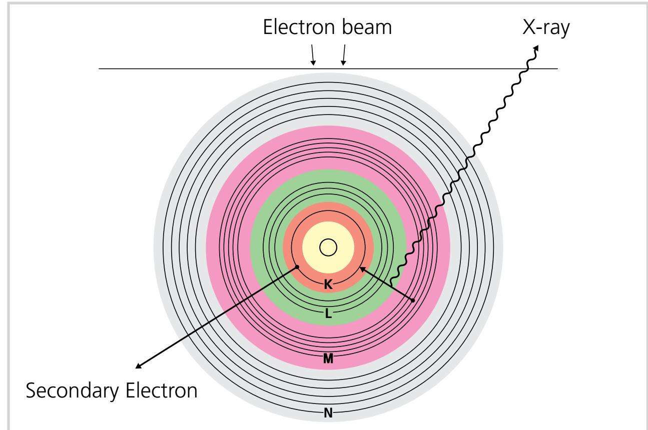


Fig. 1. Generation of X-rays by electron bombardment.

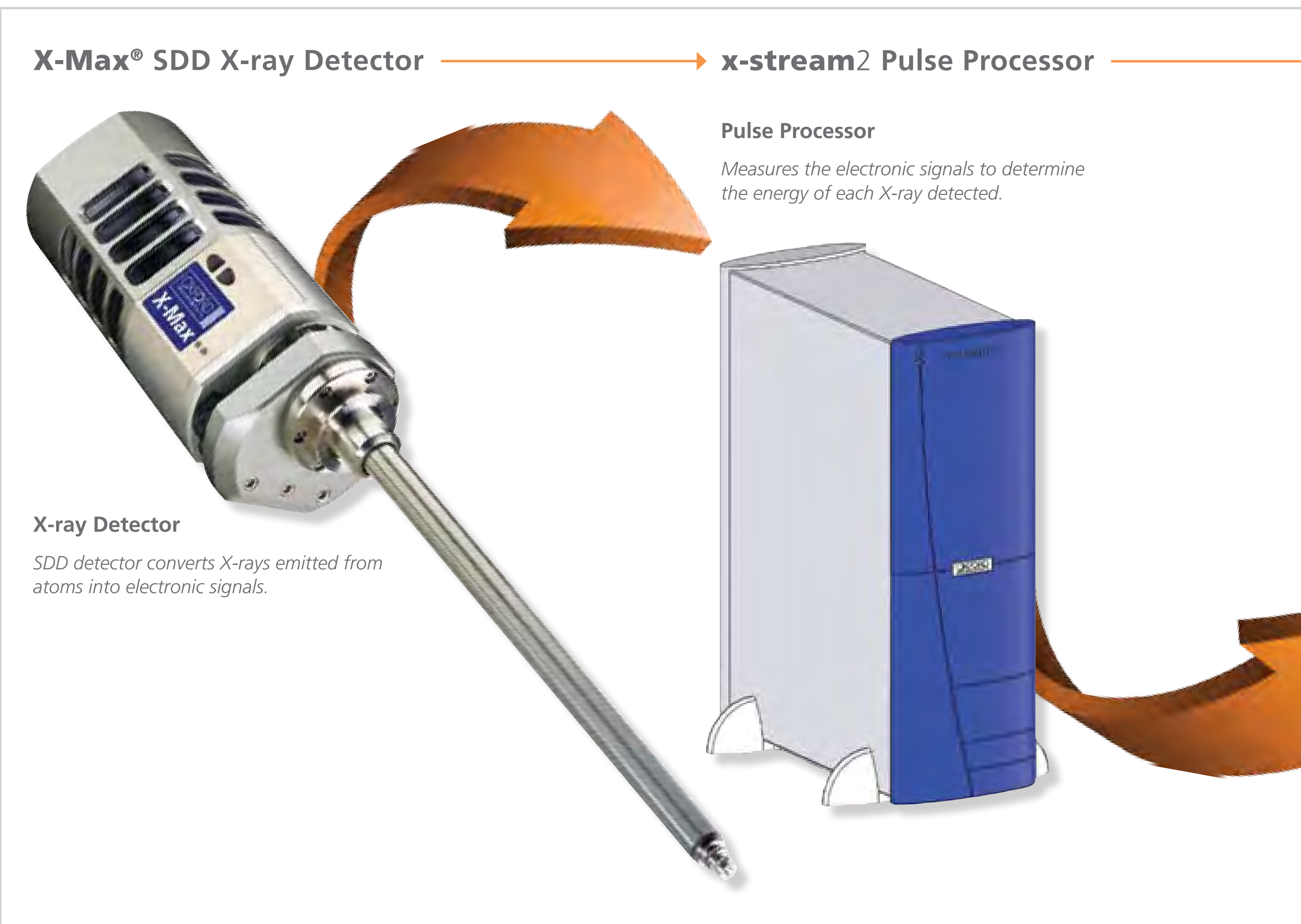


Fig. 2. An EDS system is comprised of three basic components that must be designed to work together to achieve optimum results.

The equipment needed to perform EDS is an X-ray detector operating inside the microscope, and suitable hardware and software outside the microscope.

In recent years, silicon drift energy dispersive X-ray detectors (SDD) have been growing in popularity. Usage of the more traditional Si(Li) type detectors has proportionately decreased so that the SDD is now the detector of choice for most EDS applications.

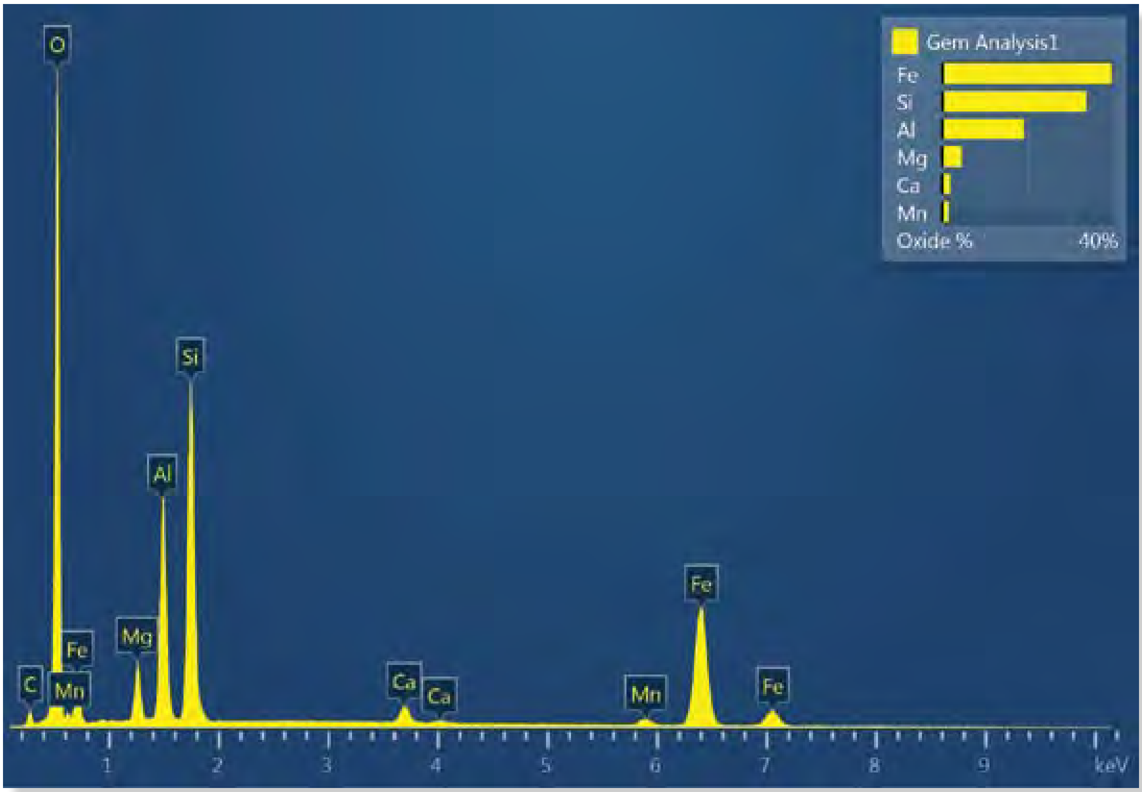
Recent advances in SDD design means that the latest generation of these detectors not only offer all the advantages of being liquid nitrogen-free and excellent energy resolution at high count rates, but now large active area detectors like X-Max also allow users to collect large amounts of data in shorter time periods, at lower excitation voltages, and under normal SEM imaging conditions.

This guide describes how SDD hardware detects and measures the X-rays and converts them into signals which can be used by EDS software to provide accurate and reliable analysis.

AZtecEnergy **Analyser Software**

Analyser

Displays and interprets X-ray data.



Section 2: EDS Detection Hardware

2.1 Components of an SDD detector

1. Collimator assembly

The collimator provides a limiting aperture through which X-rays must pass to reach the detector. This ensures that only X-rays from the area being excited by the electron beam are detected, and stray X-rays from other parts of the microscope chamber are not included in the analysis.

2. Electron trap

Electrons that penetrate the detector cause background artefacts and also overload the measurement chain. The electron trap is a permanent magnet assembly that strongly deflects any passing electrons. This assembly is only required on detectors with thin polymer windows, as thicker beryllium windows efficiently absorb electrons below 20 keV in energy.

3. Window

The window provides a barrier to maintain vacuum within the detector while being as transparent as possible to low energy X-rays. There are two main types of window materials. Beryllium (Be) is highly robust, but strongly absorbs low energy X-rays meaning that only elements from sodium (Na) can be detected. Polymer-based thin windows can be made much thinner than Be windows and therefore are transparent to much lower energy X-rays, many allowing detection of X-rays down to 100 eV. Although these window materials are far less robust, by placing them on a supporting grid they can withstand the pressure difference between the detector vacuum and a vented microscope chamber at atmospheric pressure. The greater transmission of the polymer-based windows means that they have largely replaced Be as the material used for detector windows. In instruments that operate under very high vacuum conditions, for example TEMs, it is possible to use windowless detectors which allow for even more sensitivity for light elements.

4. The sensor

The sensor is a semiconductor device that through the process of ionisation converts an X-ray of a particular energy into an electric charge of proportional size. Two main types of sensors are used for X-ray detection: traditional silicon crystals drifted with lithium, so-called 'Si(Li)', and newer faster Silicon Drift Detectors ('SDD') which have largely replaced them. SDD devices use a field gradient applied by ring electrodes on its back surface to collect the charge liberated by each X-ray detected, at the anode.

5. The FET

The field effect transistor (also known as the FET), is connected directly to the sensor. It is the first stage of the amplification process that measures the charge liberated in the crystal by an incident X-ray and converts it to a voltage output.

6. Detector cooling

SDD detectors operate at a few tens of degrees below zero and are cooled by Peltier (thermoelectric) devices bound to the SDD sensor. Heat from these devices is transferred to cooling fins in the body of the detector, by a cold finger or heat pipe, where it is dissipated.

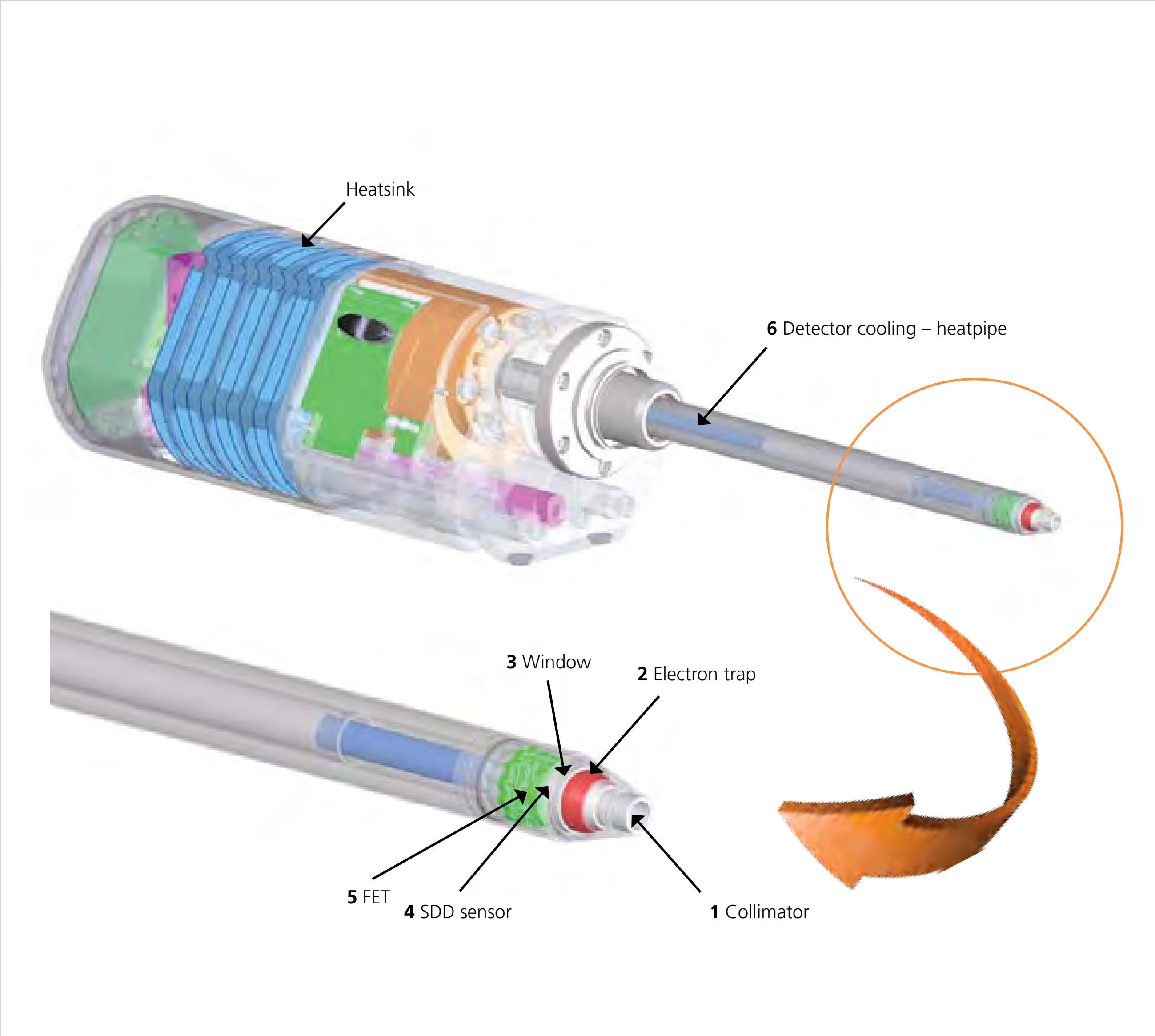


Fig. 3. Cutaway diagram showing construction of a large area SDD detector.

2.2 How the EDS detector works

The EDS detector converts the energy of each individual X-ray into a voltage signal of proportional size. This is achieved through a three stage process. Firstly the X-ray is converted into a charge by the ionization of atoms in the semiconductor crystal. Secondly this charge is converted into the voltage signal by the FET preamplifier. Finally the voltage signal is input into the pulse processor for measurement. The output from the preamplifier is a voltage 'ramp' where each X-ray appears as a voltage step on the ramp.

EDS detectors are designed to convert the X-ray energy into the voltage signal as accurately as possible. At the same time electronic noise must be minimised to allow detection of the lowest X-ray energies.

2.2.1 How the SDD sensor converts X-ray energy into charge

The silicon drift detector (SDD) sensor is fabricated from high purity silicon with a large area contact on the entrance side facing the incoming X-rays. On the opposite side there is a central, small anode contact, which is surrounded by a number of concentric drift electrodes (Fig. 4).

When a bias is applied to the SDD detector chip and the detector is exposed to X-rays, it converts each X-ray detected into an electron cloud with a charge that is proportional to the characteristic energy of that X-ray. These electrons are raised into the conduction band of the silicon semiconductor and leave behind holes that behave like free positive charges within the sensor. The electrons are then 'drifted' down a field gradient applied between the drift rings to be collected at the anode.

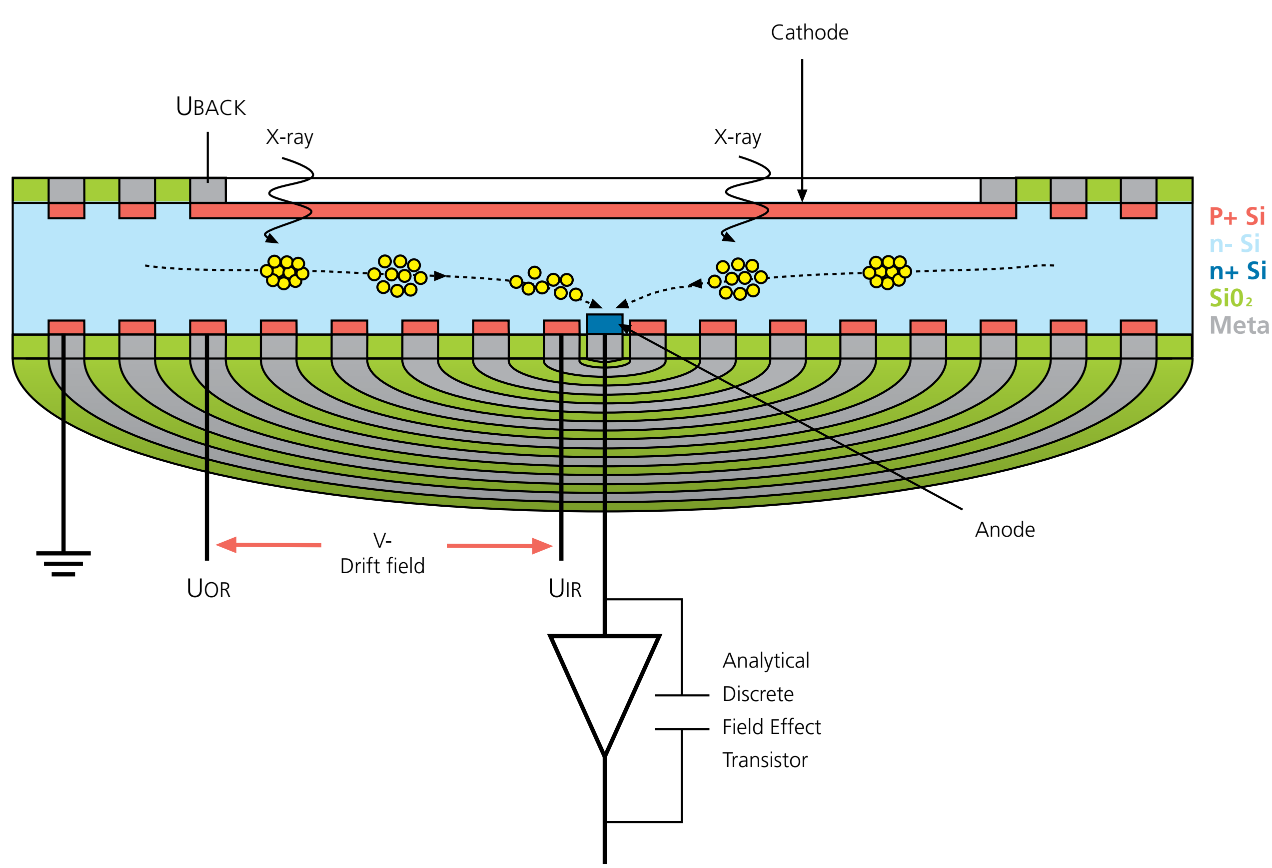


Fig. 4. Construction and Operation of an SDD detector.

2.2.2 The role of the FET

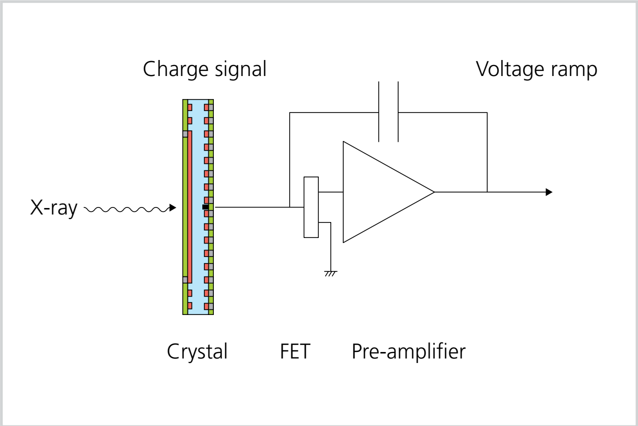


Fig. 5. Schematic of SDD connected to FET preamplifier and feedback capacitor.

The charge that accumulates at the anode is converted to a voltage signal by the FET preamplifier (Fig. 5). During operation, charge is built up on the feedback capacitor. There are two sources of this charge, current leakage from the sensor material and the X-ray induced charge from the photons that are absorbed in the detector. The output from the preamplifier caused by this charge build-up is a steadily increasing voltage 'ramp' due to leakage current, onto which is superimposed sharp steps due to the charge created by each X-ray event. The accumulating charge has to be periodically restored to prevent saturation of the preamplifier. Therefore at a pre-determined charge level the capacitor is discharged, a process called restoration, or 'reset'.

The output waveform exhibits fluctuations due to noise that limit how precisely each step can be measured. If the measurement is imprecise, this spreads the histogram of measurements for photons of the same energy. Thus, noise affects the width of X-ray peaks, particularly at low energies. Noise is influenced by the FET gain, the input capacitance and the leakage current.

Low noise is required to distinguish low energy X-rays such as beryllium Kα, silicon Ll and aluminium Ll from noise fluctuations (Fig. 6).

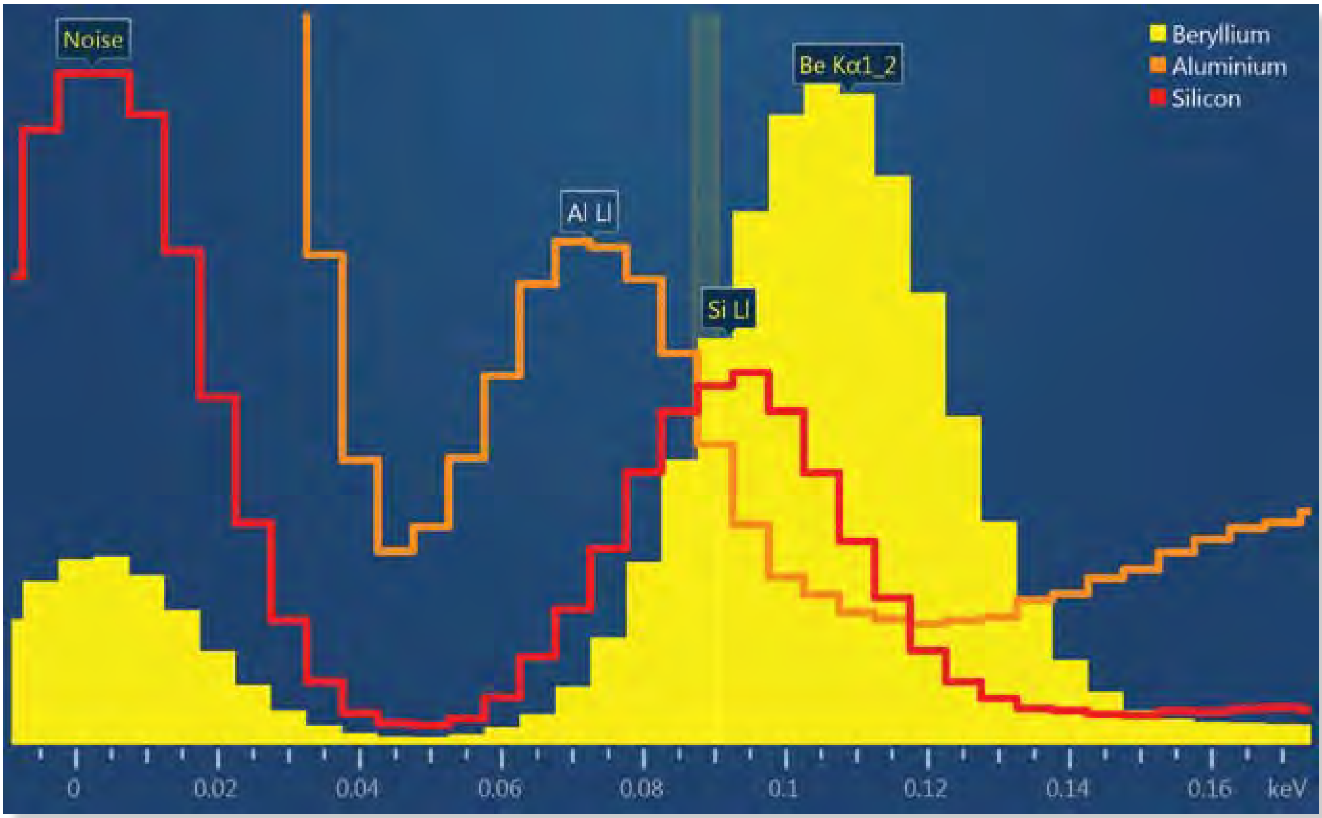


Fig. 6. Spectra from Al, Si and Be showing separation of K and L peaks from baseline noise.

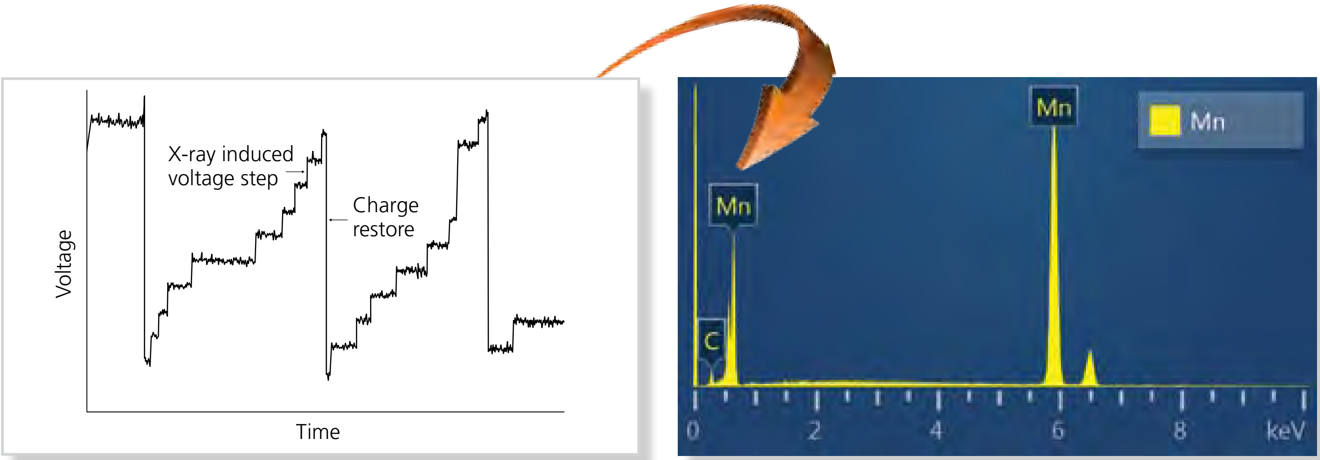


Fig. 7. Digital pulse shaping signal from the processor converted to an ED spectrum.

2.3 The pulse processor

The charge liberated by an individual X-ray photon appears at the output of the preamplifier as a voltage step on a linearly increasing voltage ramp (Fig. 7).

The fundamental job of the pulse processor is to accurately measure the energy of the incoming X-ray, and give it a digital count in the corresponding channel in the computer. It must optimise the removal of noise present on the original X-ray signal and it needs to recognise quickly and accurately a wide range of energies of X-ray events from below 100 eV up to 40 keV. It also needs to differentiate between events arriving in the detector very close together in time to prevent pulse pile-up effects.

2.3.1 Digital pulse processing

With digital pulse shaping the signal from the preamplifier is digitised at the input of the pulse processor, and shaping and noise reduction are achieved by digital computation.

The preamplifier output is sampled continuously by an analogue to digital converter (ADC) and X-ray pulse heights are typically measured by subtracting the average of one set of values, measured before an X-ray event, from that for another set, measured after the event. The resultant value of the step measurement is then sent directly to the computer multi-channel analyser.

The noise on the voltage ramp from the detector is effectively filtered out by averaging the signal. The time over which the waveform is averaged is sometimes referred to as the process time but different timing definitions may apply depending on the processing scheme used.

Section 3: EDS Hardware Performance

3.1 Resolution and detector noise

Resolution is the single most accepted measurement of detector quality and provides a useful indication of a detector's performance. Resolution is quoted as the width of the peak at half its maximum height (FWHM). The lower the number the better the resolution a detector has and the better it will be at resolving peaks due to closely spaced X-ray lines.

The resolution achieved by a detector is dependent on the sources of noise from the sensor and how they are processed by the counting chain. Unwanted noise has three main sources:

I. **Voltage noise** is affected by the FET gain and the sum of the capacitances of the components in the sensor/FET circuit. One major component of capacitance is the sensor anode; since SDDs have much smaller anodes than the older Si(Li) detectors, they have a lower capacitance and much less voltage noise. Voltage noise is reduced by signal averaging using long process times in the pulse processor. With SDDs much shorter process times can be used to reduce this noise to an equivalent level. As a result, SDD sensors can maintain good resolution at much higher count rates than was previously possible with Si(Li)s. Therefore SDD hardware offers the potential for high count rate analysis in a very short time and has revolutionised high speed mapping, for example.

II. **Leakage current** which results from the bias voltage applied to the sensor, gives rise to the slope on the voltage ramp (Fig. 8). Leakage current is a source of 'shot noise' and affects resolution at long process times where the base-line signal changes over the time taken to integrate the ramp. For a given design of detector, leakage is proportional to the area and thickness of the sensor and increases with increased temperature. To achieve best resolution, Si(Li) detectors require long process times to reduce voltage noise and therefore require extensive cooling using liquid nitrogen to minimise leakage current noise. In contrast, SDD sensors can achieve the same voltage noise at shorter process times and can therefore tolerate higher leakage current while still maintaining the same resolution. As a result, SDDs work very well at the higher temperatures that can be maintained by more convenient Peltier cooling technology.

III. **1/F Noise.** The 1/F contribution to resolution is due to the properties of the detector (e.g. contacts and dielectric materials) and is largely independent of the process time.

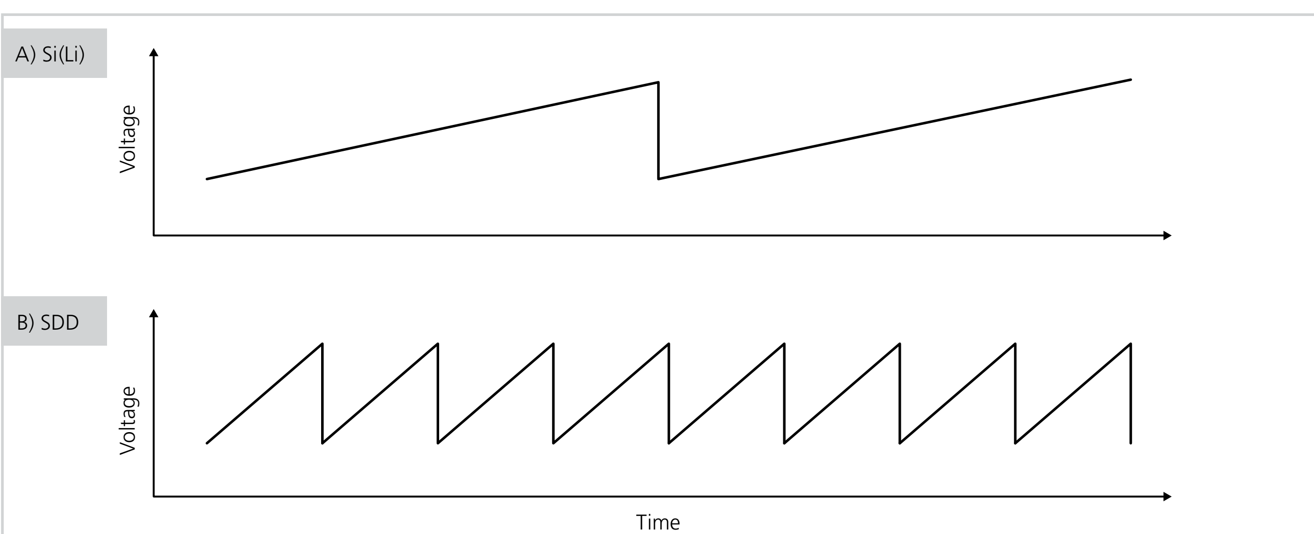


Fig. 8. Leakage current (gradient on the voltage ramp) is larger for SDDs but since process time (T_p) is shorter, leakage current is of lesser importance than with Si(Li) detectors.

3.2 How is resolution specified?

Resolution is traditionally measured using the X-rays of the manganese Kα line (MnKα). This is convenient for manufacturers of EDS detectors, because they can use a MnKα emitting ^{55}Fe radioactive source to monitor individual sensor and detector performance without the need for an SEM. MnKα resolution can also be easily measured in an electron microscope by placing a piece of pure manganese under the electron beam.

3.2.1 The importance of low energy resolution

The identification and quantification of closely spaced X-ray peaks becomes easier and more accurate as the peak energy increases and the separation between them increases. X-ray lines are closer together at low energy and this is apparent in Fig. 9, where the energy of the major line for K, L and M series is plotted for all elements. At the MnKα energy (5.9 keV) most commonly used to specify EDS resolution, peaks are well separated. However, it is clear that much more serious overlaps occur below 3 keV and the resolution performance at low energies is crucial if a good performance for all elements is to be expected.

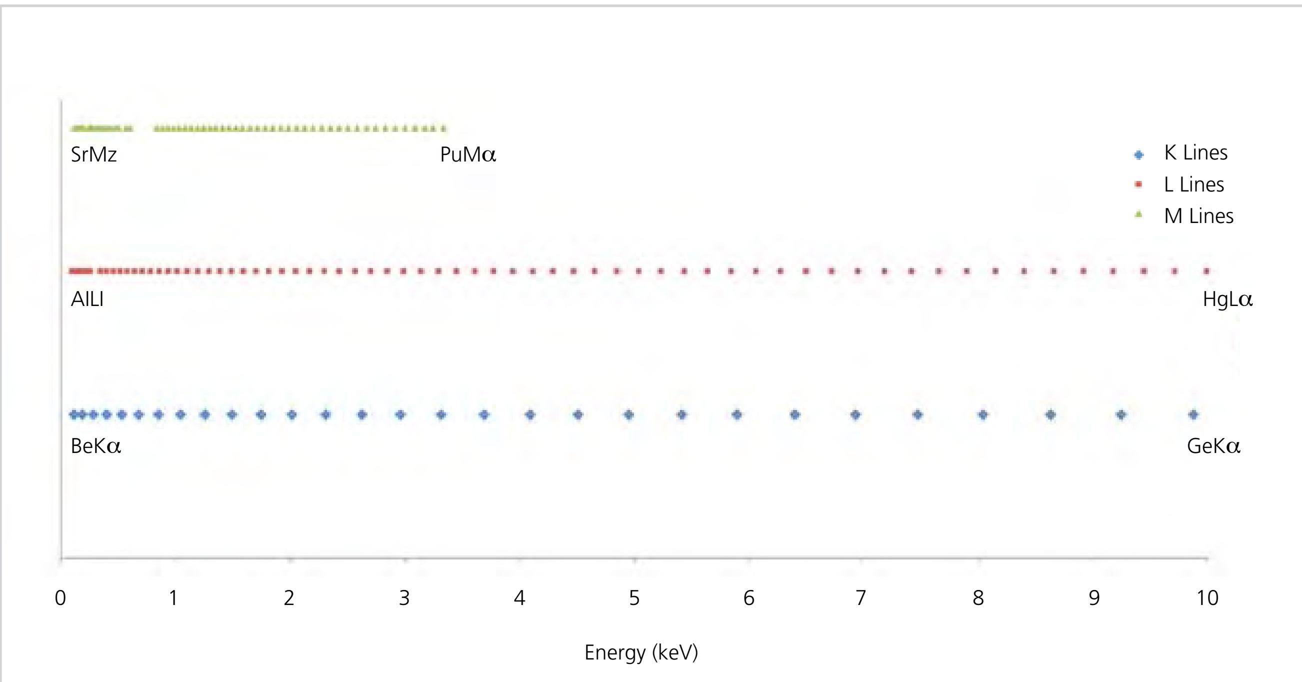


Fig. 9. Energy of the major lines of K, L and M series for all elements.

When small features <1 µm in size are being analysed, the beam voltage needs to be reduced to avoid generation of X-rays from surrounding material. However, at low kV only low energy lines are available for analysis. Spectra collected from a nickel alloy at 20 kV and 5 kV (Fig. 10) illustrate the importance of resolution at low energy. When working at 20 kV, the separation of widely spaced K lines of Cr, Fe and Ni, will not be affected much by a few eV variation in resolution. When working at 5 kV however, where identification relies on L lines which are very close in energy, a detector with better resolution will allow the L lines of Cr, Fe and Ni to be separated enough for confident identification.

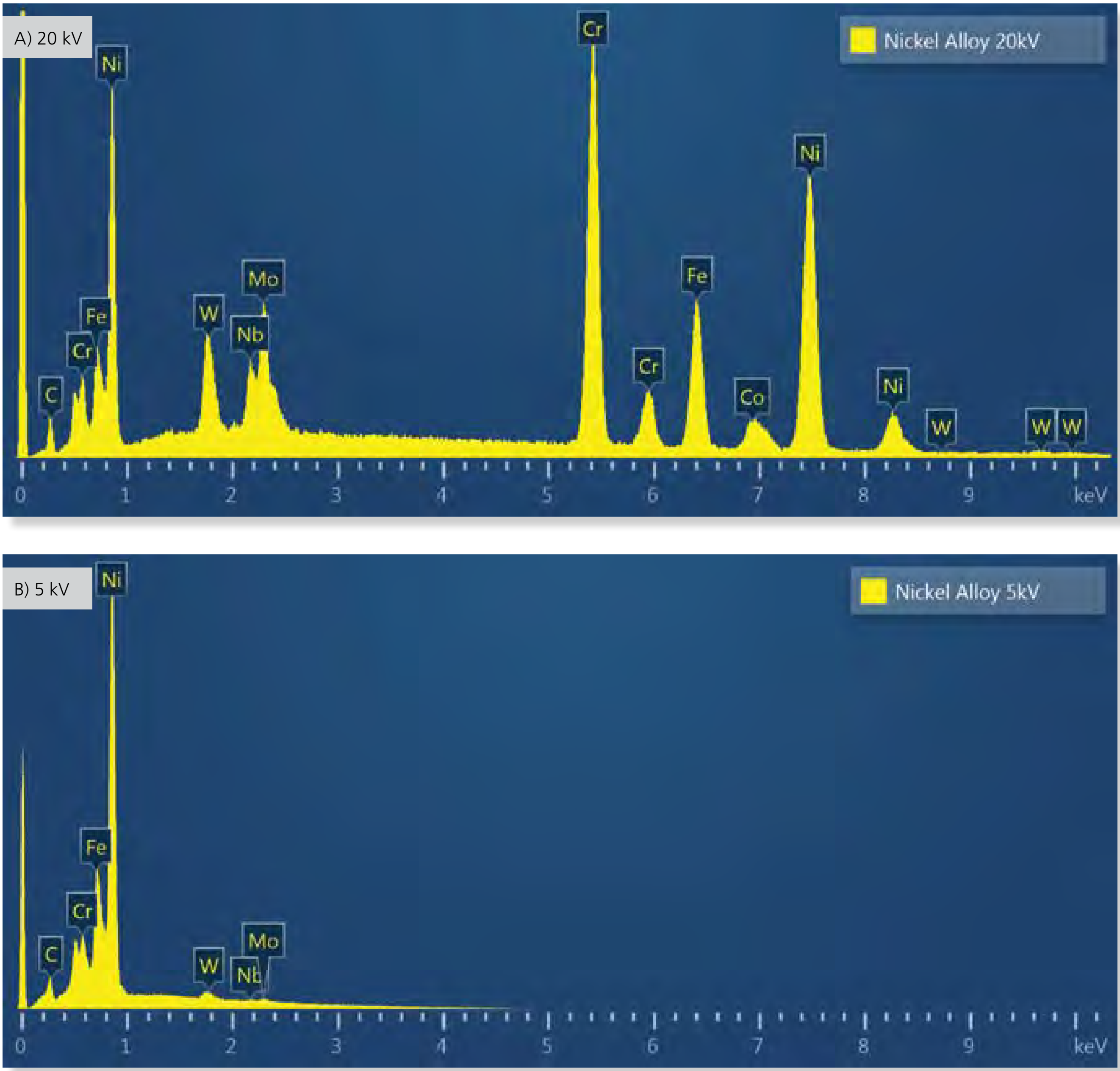


Fig. 10. Nickel alloy spectra collected at A) 20 kV and B) 5 kV.

3.2.2 Fluorine resolution FKα FWHM

The width of the peaks in a spectrum will vary depending on the energy of the X-ray line. In Fig. 11 this variation with energy has been calculated to show how the resolution should change with X-ray detectors made from silicon (SDD) and germanium (HpGe) with different Mn resolution specifications. The curves are calculated from the equation

$$FWHM^2 = k.E + FWHMnoise^2$$

where k is a constant for the detector material, and E is the energy.

These curves demonstrate that as the energy decreases, the resolution of the X-ray peaks improves. The variation is also clearly different for SDD and HpGe detectors. At low energy the electronic noise contribution (FWHMnoise) has a greater effect on resolution. Mn FWHM resolution is not a good measure to characterise the noise of a detector and predict the resolution at low energy. Therefore to characterise the resolution of a detector at low energy, the resolution is also quoted for another line, typically fluorine Kα. As can be seen from the diagram, modern SDD detectors can have significantly better lower energy resolution than HpGe detectors even though SDD Mn resolution is worse.

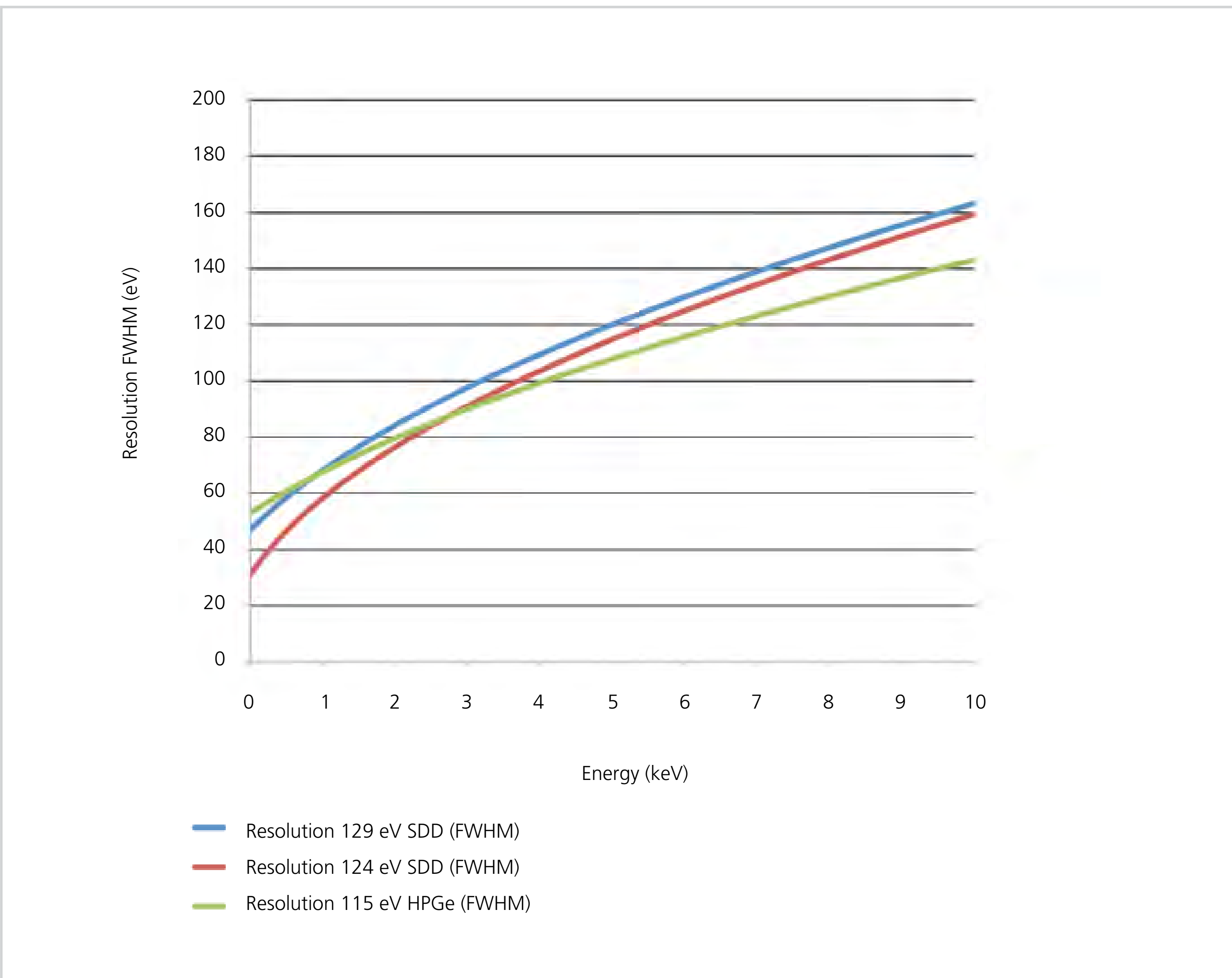


Fig. 11. Curves showing how resolution changes with energy for different SDD and HpGe detectors. The calculation ignores the effects of incomplete charge collection that can make some detectors give worse performance than these curves predict, particularly at low energies.

3.2.3 Incomplete charge collection

If all the electron-hole pairs generated by an X-ray are not drifted to the electrical contacts, the charge signal measured by the FET will be lower than expected, and the energy measured will be lower than the energy of the incident X-ray. This phenomenon is known as incomplete charge collection (ICC), and results in counts appearing in the spectrum at lower energies than the energy of the X-ray that they represent, typically as a tail on the low energy side of the peak. All detectors suffer from incomplete charge collection to some extent. Low energy X-rays have a very shallow depth of penetration and charge collection is usually poor near the front contact. The peak for a low energy X-ray will therefore be broader and have a mean energy lower than expected, due to varying levels of charge collection as each X-ray is measured (Fig. 12). Thus, incomplete charge collection results in detectors with resolutions measured at low energy that are worse than those predicted by theory. In extreme cases it can be the dominant factor controlling resolution at these very low energies (Fig. 12).

3.2.4 Carbon resolution CKα FWHM

It is very important to know what the resolution specification of a detector is at low energy. A detector with good energy resolution at low energy will have good resolution throughout the X-ray spectrum. However, the reverse is not true. A good manganese resolution specification does not guarantee good low energy performance. This is because a measurement of resolution at low energy is affected by both noise and incomplete charge collection. The resolution of the CKα peak measured using pure carbon is very useful due to its sensitivity to incomplete charge collection. A low value guarantee here really does mean excellent detector resolution for all energies.

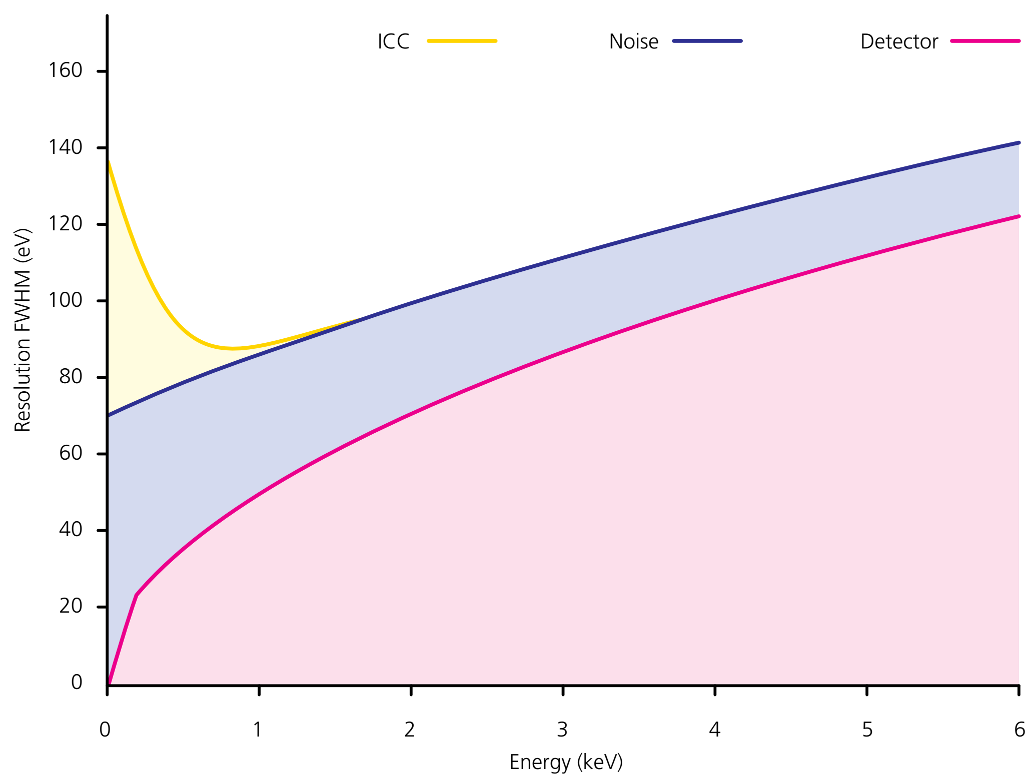


Fig. 12. The variation in detector resolution with energy is controlled by the contribution of three factors: a constant dispersion based on the crystal material used in a detector, the level of electronic noise, and low energy peak broadening due to incomplete charge collection. The curves here are based on a detector with a resolution of 140 eV at manganese that has severe incomplete charge collection.

3.2.5 Detector specifications based on tests using a ^{55}Fe radioactive source

Testing the performance of an EDS detector using a radioactive source is convenient for a detector manufacturer because it can be done without having to mount a detector on an electron microscope column. However, measurements with a ^{55}Fe source don't necessarily guarantee the performance when the detector is mounted on a column and collecting X-rays emitted during electron bombardment.

One useful feature of the ^{55}Fe source (Fig. 13) is the absence of the continuum, or Bremsstrahlung, X-rays that would be generated in an electron microscope. However, a background contribution is still visible in an ^{55}Fe spectrum and this is due to incomplete charge collection. Events appearing at 1 keV correspond to MnKα photons where 83% of the charge has not been collected, whereas events appearing at say 4 keV have lost 32% of the charge. One measure of this ICC is the 'peak: background' ratio comparing the height of the MnKα peak to the average background between 0.9-1.1 keV (Fig. 13). Although the original IEEE standard suggested measurement at several energies, manufacturers have most commonly used 1 keV. As shown in Fig. 13, 1 keV is typically the lowest part of the ICC background and a low value at this energy (high peak to background ratio) does not guarantee that charge collection will be good for the smaller charge losses that contribute to the tail or plateau on the low energy side of a peak. ^{55}Fe peak to background therefore only gives a general indication of charge collection efficiency, and does not ensure good peak shapes or give any indication of ICC for low energy X-rays. Incomplete charge collection is most important where X-rays are strongly absorbed and only penetrate a short distance into the crystal, for example just above the absorption edge of the elements making up the crystal (1.84 keV for the silicon in SDD detectors), and for very low energy X-rays (less than 0.5 keV). The CKα resolution measurement on the microscope represents a more useful and sensitive measurement of incomplete charge collection than peak:background ratios measured with an ^{55}Fe source. However, the CKα resolution measurement is costly to perform as part of the production process and some manufacturers only guarantee ^{55}Fe peak:background in order to save costly SEM time during detector manufacture. If CKα resolution performance on the microscope is not guaranteed, there is a risk that peak resolution at low energies could be poor, even when ^{55}Fe peak:background and resolution measures are good.

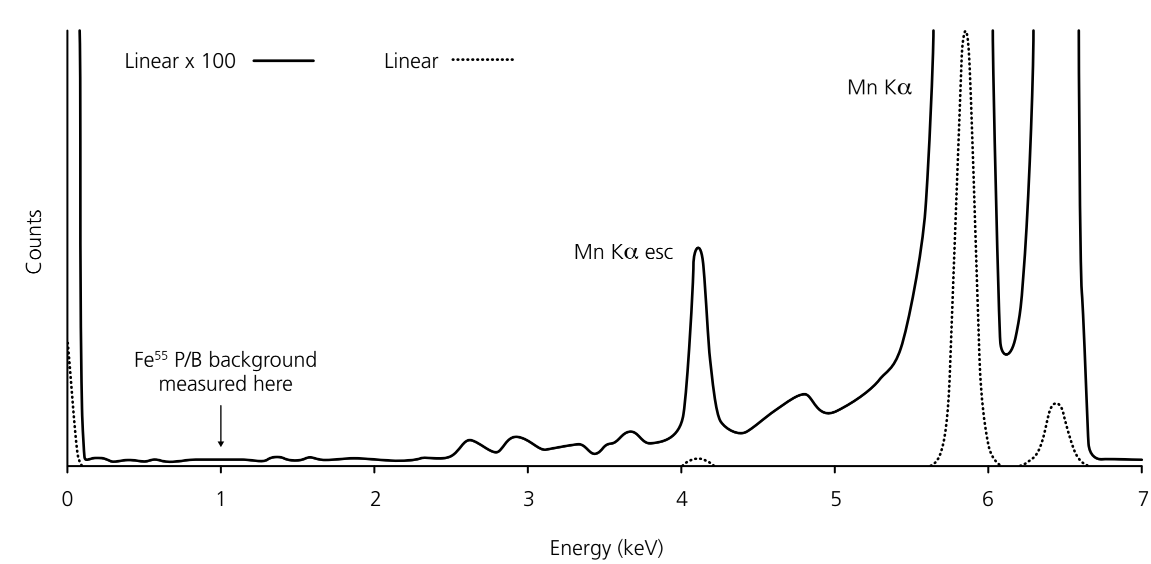


Fig. 13. A schematic of a spectrum collected from an ^{55}Fe radioactive source showing how peak:background ratio is measured on the bench.

3.3 Using resolution to assess EDS hardware performance

As we have seen, resolution must be considered at a number of energies not simply at MnKα. The international standard for instrumental specification of energy dispersive X-ray detectors for microbeam analysis, ISO 15632, recognises this key point. To comply with this standard, EDS detectors that can measure X-rays of less than 1 keV in energy (i.e. all modern EDS detectors that use thin polymer windows), resolution must be guaranteed by the manufacturer at CKα and FKα as well as at MnKα. This is particularly relevant for the increasing number of EDS systems being used to investigate nano-materials and other nano-scale features using low accelerating voltages, and relying on the measurement of low energy peaks in the EDS spectrum.

With the almost complete switch from Si(Li) to SDD technology, excellent resolution performance is now possible at much higher count rates than ever before. Therefore, the ISO 15632 standard also requires that the count rate at which the resolution specification can be achieved must also be stated. For example for SDD, best resolutions can now be achieved at count rates of tens of thousands of counts per second, rather than the 1-2 kcps that was typically offered for best resolution performance with Si(Li). In some situations (low kV, thin or dose sensitive specimens, small area detectors) it may not be possible to collect X-rays at high rate so it makes sense to configure the SDD system to suit the count rate being generated. Consequently, more than one specification may be offered by manufacturers, each one corresponding to a different count rate.

However, resolution does not tell the whole story about the capability of an EDS system to produce accurate results and only gives an indication of how sharp the peaks will appear in the visual spectrum. It is the area of individual peaks, corrected for background and overlap from neighbouring peaks, that is need to determine concentration values. Even with modern automated algorithms for spectrum processing, it is critically important for the software to have accurate parameters for peak resolutions, positions, shapes and relative heights if accurate quantitative analysis is to be achieved [1].

3.4 How resolution changes with count rate and the effect of process time

The longer the process time (T_p), the lower the voltage noise. Provided the leakage current noise is still low, the resolution of the peak displayed in the spectrum is improved (see Fig. 14), and it becomes easier to separate or resolve, from another peak that is close in energy. However, there is a trade-off between the process time that is used, and the speed at which data can be measured. The longer the process time, the more time is spent measuring each X-ray, and the fewer events that can be measured. The longest process time used by a processor gives the best resolution possible while the shortest process time gives the maximum throughput into the spectrum, but there will be some loss of resolution.

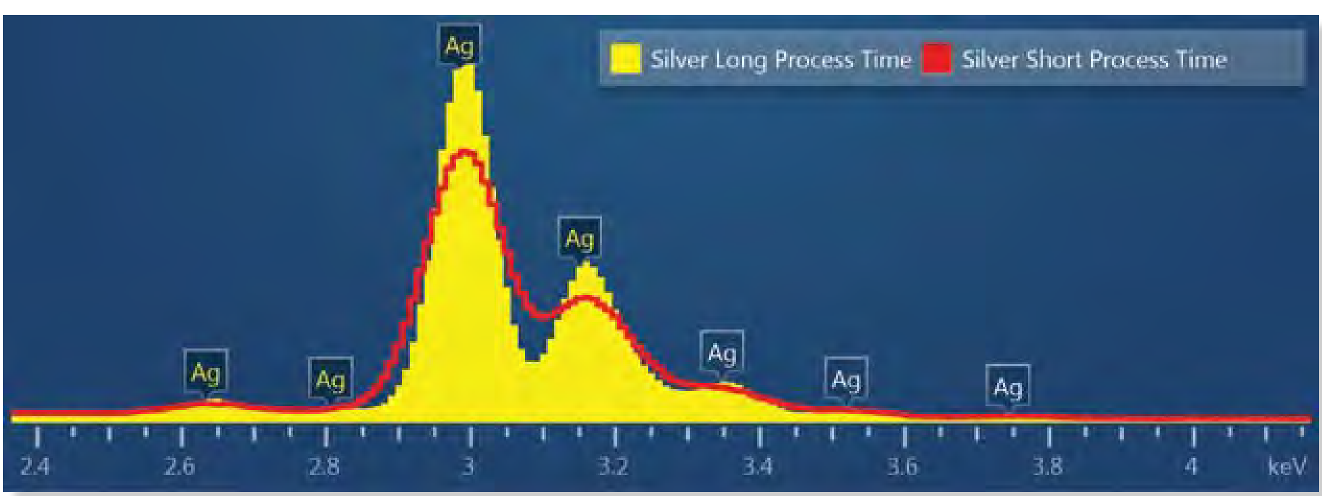


Fig. 14. Effects of changing process times. Resolution is improved by using longer process times whereas shorter process times allow faster throughput with a lower peak resolution.

Productivity depends on the rate of counts measured, called the acquisition rate, rather than the input rate (into the detector). As the input rate increases so does the acquisition rate, but an increasing number of events are rejected because they arrive in a shorter time period than T_p (Fig. 15b). If input rates increase sufficiently, the proportion rejected will exceed the increase in measured events and the acquisition rate will start to decrease with further increases in input rate.

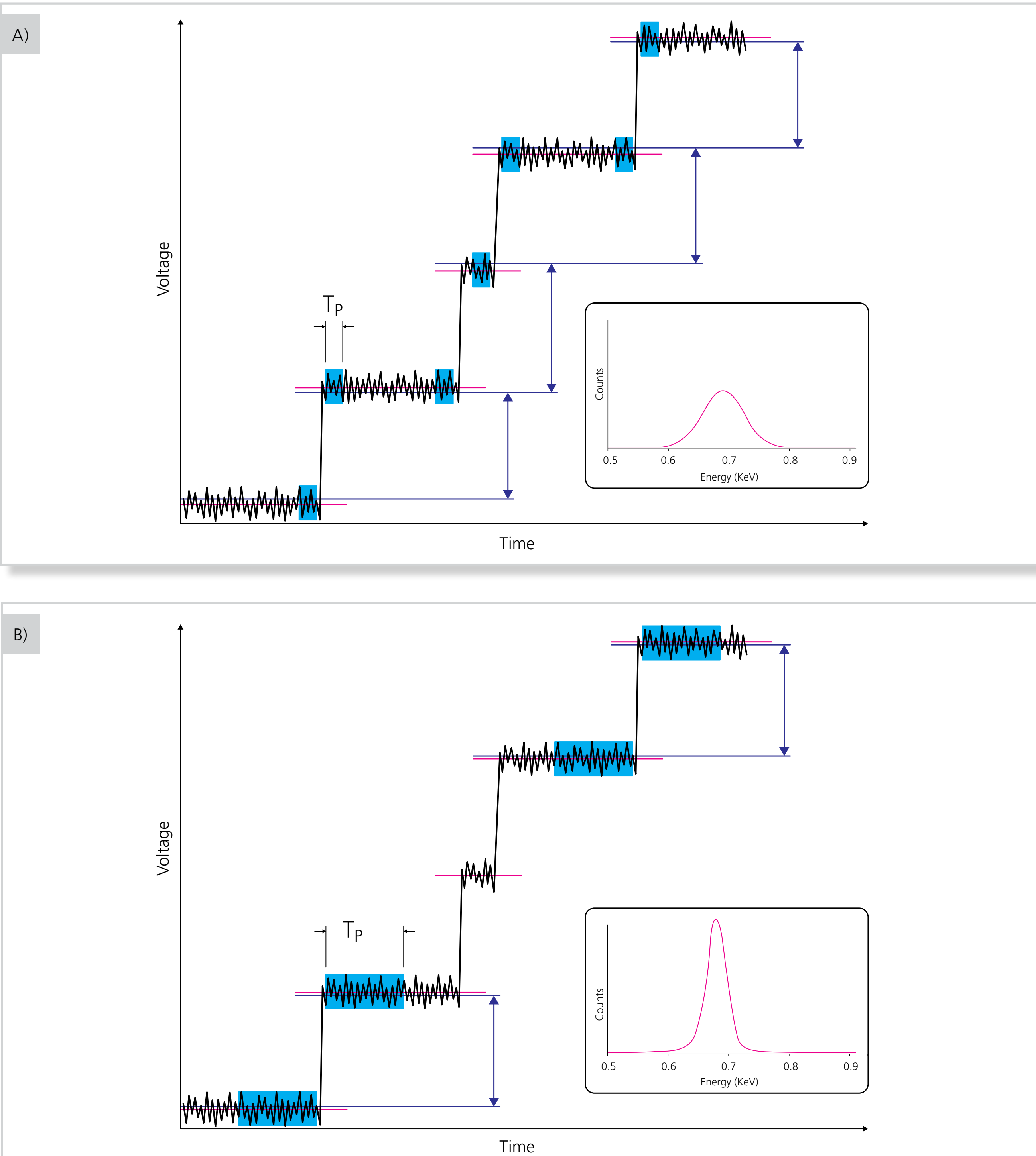


Fig. 15. Measurement of steps on a voltage ramp by averaging differing numbers of measurements of the signal. A) Short T_p permits all steps to be measured, but the variation of each measured step is large, so the X-ray energy is not measured accurately and peaks show poor resolution. B) Long T_p means that some steps arrive too close together to be measured. However, noise averaging is better and therefore peaks show better resolution.

Therefore for each process time there is a maximum acquisition rate (Fig. 16) which corresponds to the maximum speed possible for a chosen resolution. The maximum acquisition rate for each process time is characteristic of the detector pulse processor used. By determining, for each processor setting, the maximum acquisition rate and the resolution at this rate, the productivity and performance of a detector/ processor measurement chain can be evaluated.

This trade off of resolution vs process time is true of both SDD and Si(Li) detectors, however due to the much lower levels of voltage noise characteristic of SDD design, process times are much shorter for equivalent resolution performance. This means best resolutions can be achieved at 10s of thousands of counts per second (input rate) and maximum throughputs are measured in 100s of thousands of counts per second.

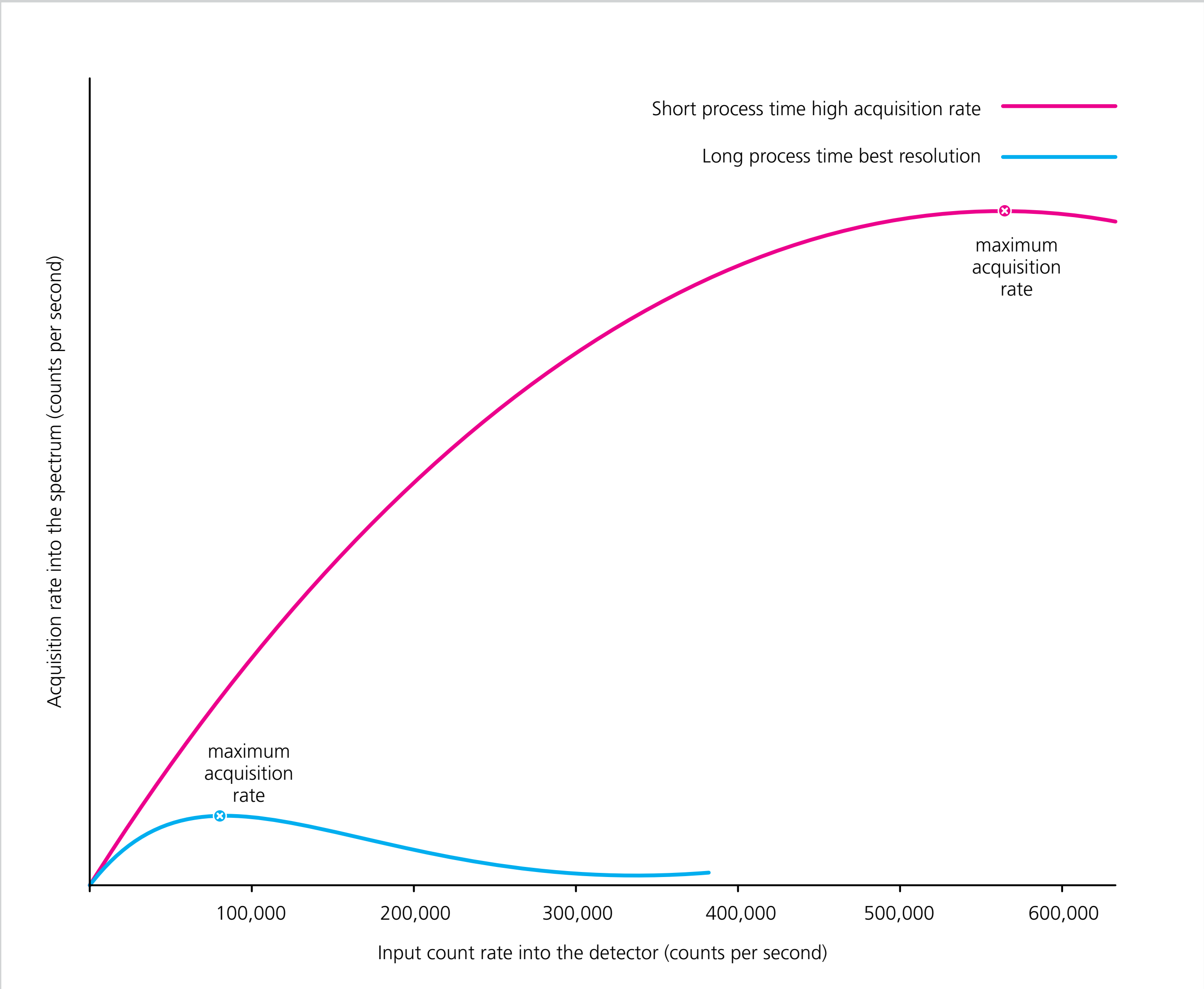


Fig. 16. Curves showing the variation of acquisition rate with input rate for two fixed process times. The longer process time gives good resolution but limited maximum acquisition rate, the short process time allows much higher acquisition rates but resolution is worse.

Section 4: Different Types of SDD Detector

Current commercially available SDD detectors are based around two different design philosophies, one based on an integrated FET, the other on a discrete external FET. The performance of the SDDs provided by these two technologies differs in a number of ways, with each offering advantages and disadvantages whose importance depends on customer need.

4.1 SDD sensors with integrated FET

By integrating the FET into the sensor design as part of the anode assembly, parasitic bond pad capacitances are avoided so that the capacitance of the anode-FET combination can be minimised. The gain and noise is also affected by the silicon material used for the FET. The high resistivity material used for detectors is very different from the lower resistivity material that has typically been used to optimise gain and noise for discrete FETs. Therefore, there are some tradeoffs to consider when building a FET using the same silicon as the detector. Nevertheless, in a good design of integrated FET, the reduction in capacitance due to avoiding conventional bond pads can be sufficient to reduce the overall voltage noise. With an SDD of this type, lower voltage noise means that good resolution can be achieved at the minimum possible process time, and therefore is maintained at the highest possible count rate.

However, to make use of this performance design challenges of the integrated FET must be overcome. Firstly, because the FET is part of the sensor it is susceptible to irradiation by incident X-rays and the electrostatic fields surrounding the FET result in performance losses at low energies. To overcome this issue the shape of the sensor can be re-designed to place the anode and FET at the margins protected by a collimator. The drift rings are designed in a tear-drop shape so the electrons drift towards the anode. The teardrop shape means that maximum distance that the charge cloud must travel is roughly three times further than in equivalent radial sensors, causing longer maximum signal rise times at the anode (Fig. 17). This typically limits the practical area of these sensors to about 10 mm^2, as ballistic deficit effects (see section 5.1) will either reduce resolution or speed, and will therefore negate the design advantages of the integrated FET architecture.

The use of an integrated FET also leads to more complex electronics design. Early integrated FET designs used continuous restore methodology, however, this is unsatisfactory for EDS because as the count rate increases the resolution degrades, therefore losing the speed advantage of the design. To mimic the advantages of the traditional external FET, pulse restore was developed and a charge sensitive amplifier configuration implemented using a parasitic capacitance as feedback. This produces a ramp similar to that seen with an external FET, but this is not a truly linear ramp because the effective feedback capacitance changes with voltage. This arrangement is not ideal but the non-linearity can be corrected by complex amplifier designs. Any uncorrected change in the base-line will lead to side effects such as changes in resolution or peak position with time or count rate. When using such an SDD optimised for high count rate performance it is important to check peak position and resolution stability at different count rates, and over time, particularly after the sensor is switched on or cooled down.

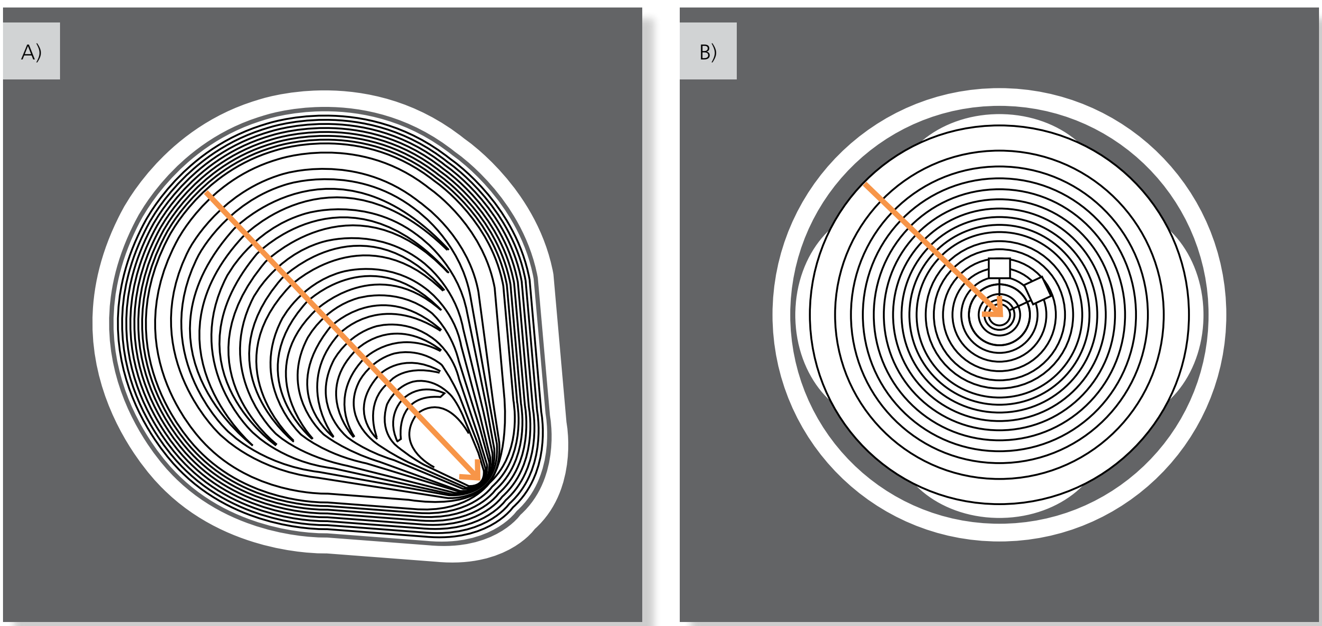


Fig. 17. Most common SDD shapes. A) Tear-drop with integrated FET. The FET is at the periphery of the design meaning greater charge cloud travel distance than required for a radial design B) where an external FET is bonded to a central anode. (Images courtesy of Ketek GmbH).

4.2 SDD sensors with external FETs

SDDs can also be designed with an external FET which uses a dedicated feedback capacitor and a well-proven method of pulsed charge restoration. This makes design of the sensor and the measurement chain straightforward, making it possible to guarantee stability and provide more accurate X-ray measurement. It also means FETs can be designed and manufactured separately and materials chosen to maximise their performance. The trade-off for this approach is that with current technology the bonding between anode and FET introduces higher capacitance than with integrated designs. Therefore, the speeds at which best resolution can be achieved are lower, although still much faster than can be achieved with Si(Li).

With large area sensors that use a central readout anode to reduce rise time effects, the external FET design allows use of a small central anode and avoids the problems due to having a FET integrated with this anode. Thus, very good low energy performance can be achieved even with large area sensors.

4.3 Large area SDD detectors

SDD detectors clearly possess an important advantage because they maintain best resolution performance at much higher count rates than for Si(Li). Higher productivity is achieved by collecting data faster with no loss of analytical performance. Spectra collection can take seconds instead of minutes and X-ray map acquisition minutes instead of hours.

However, of course many more X-rays must be generated and collected for the productivity advantages of SDD technology to be realised. For 10-30 mm^2 SDD detectors with the same size as traditional Si(Li) detectors this means using significantly higher beam currents in the electron microscope (Fig. 18). While this is possible for some micro-applications on stable samples, it is undesirable, difficult or impossible for beam sensitive, unstable, easily contaminated samples, or for nano-analysis at low kV where much fewer counts are generated.

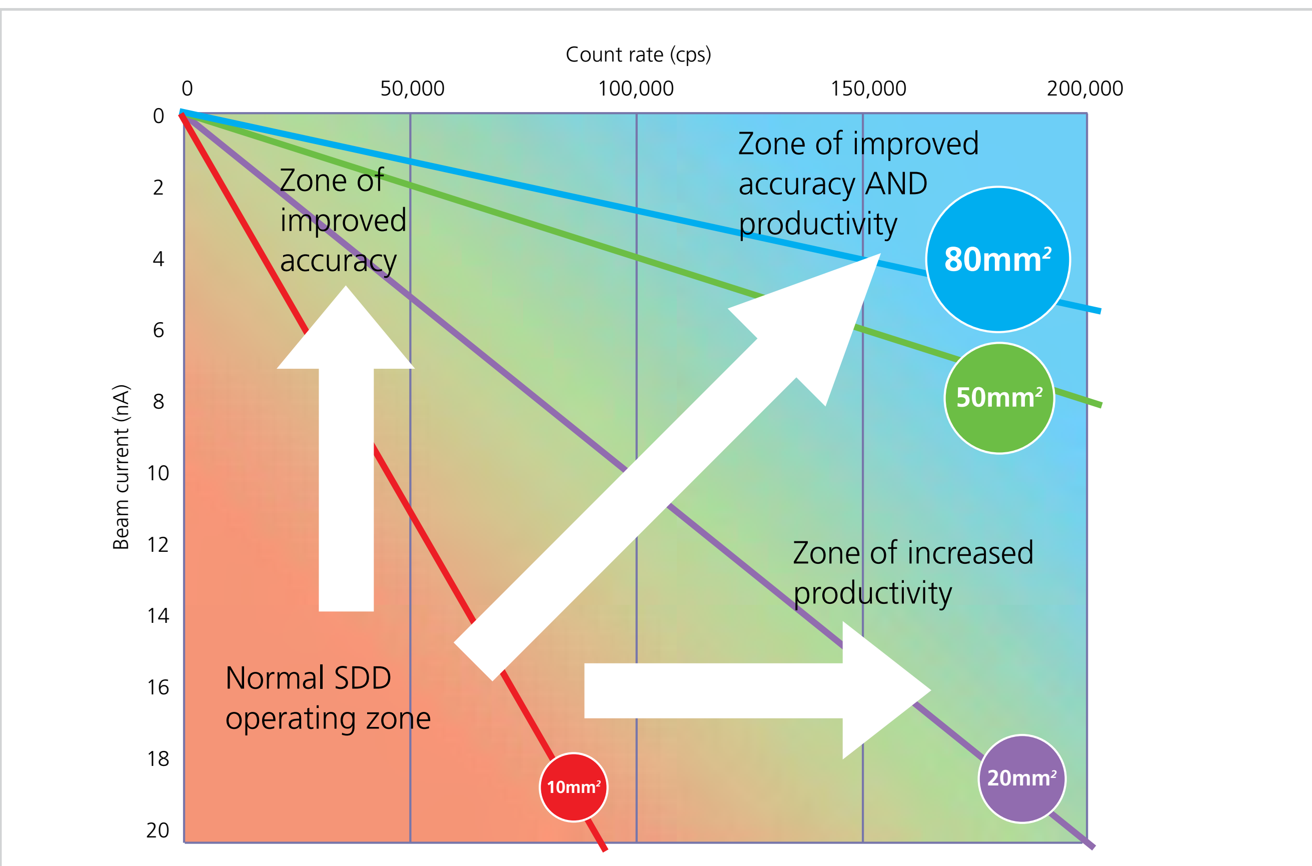


Fig. 18. Count rate vs beam current for different sensor sizes. At the same beam current a large area SDD sensor receives a much higher count rate than a 10 mm^2 sensor.

The solution to extend the benefits of SDD to a wide range of applications is to fabricate larger detectors which can collect more X-rays under the same SEM operating conditions. When Si(Li) detectors are made with larger areas, as the area increases, the anode capacitance and noise increases. As a result, with Si(Li) detectors 30 mm^2 has so far proved to be the about the limit for premium analytical performance.

However, in SDD, instead of the whole back surface of the sensor being the anode (as it is with Si(Li)), the drift field brings the electrons to a very small anode, thus drastically reducing readout capacitance (Fig. 19). Furthermore, the SDD anode capacitance is not affected by the area so the design offers the potential for much larger sensors that offer good resolution.

The drawback with large area SDDs is the increased leakage current and the increased distance required to drift the signal charge cloud towards the readout anode. The leakage current increase can be nullified by increased cooling and the drift distance is minimised by use of a central anode. A central anode arrangement is more challenging with an integrated FET that sits inside the anode ring due to the need to protect the integrated FET from X-ray bombardment and avoid charge losses for X-rays that fall at the centre of the device on top of the FET. These issues are avoided by using an external FET arrangement where the SDD anode is simply a small central electrode and sensors up to 100 mm^2 in size are available that offer identical resolution and analytical performance to equivalent 10-30 mm^2 sensors. A practical limit to detector size is the diameter of the EDS detector tube that goes through a port on the microscope column. In addition, the design of the magnetic electron trap becomes more challenging when the open area is larger. Although in principle a smaller detector placed closer to the specimen can provide equivalent solid angle, at closer distances there is more risk of damage and interference with microscope imaging performance.

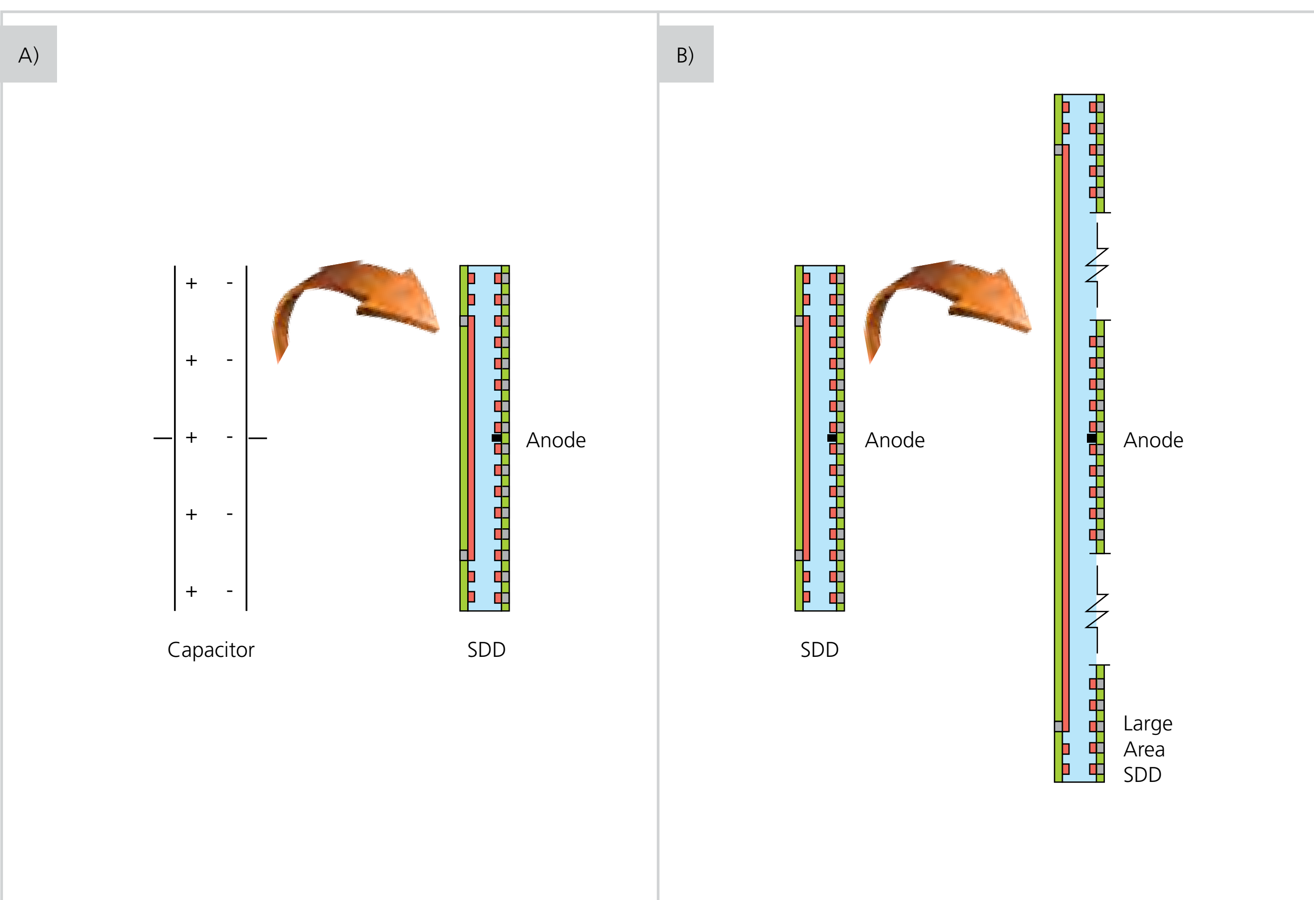


Fig. 19. A) The capacitance of an EDS detector is proportional to the area of its electrodes. In the case of SDD the front surface being its cathode and its anode being a small area on the back surface where the drift field is focussed. B) If the area of the detector is increased without changing the size of the anode then the same capacitance and resolution performance with count rate can be achieved.

Section 5: Challenges of Analysis at Very High Count Rate

SDD technology is far better suited to high count rate analysis than Si(Li) and examples have been shown of accurate quantitative analysis at input rates of up to 200,000 cps [1] and X-ray maps collected at input rates in excess of 500,000 cps [2]. However, at these count rates additional effects need to be considered.

5.1. Ballistic deficit

The electron cloud generated by the incoming X-rays spreads out before it reaches the anode. This is a problem for SDD because of the relatively slow speed at which the electron clouds are drifted to the anode for signal collection. The longer the path distance between the point where the X-ray is absorbed and the anode, the more the electron cloud spreads out. When the cloud gets close to the anode it begins to induce an output signal but the full signal step is not achieved until the whole cloud is collected; the larger the cloud, the longer the time. As a result, the rise time of the signal pulse depends on the original point of interaction in the detector. (Fig. 20).

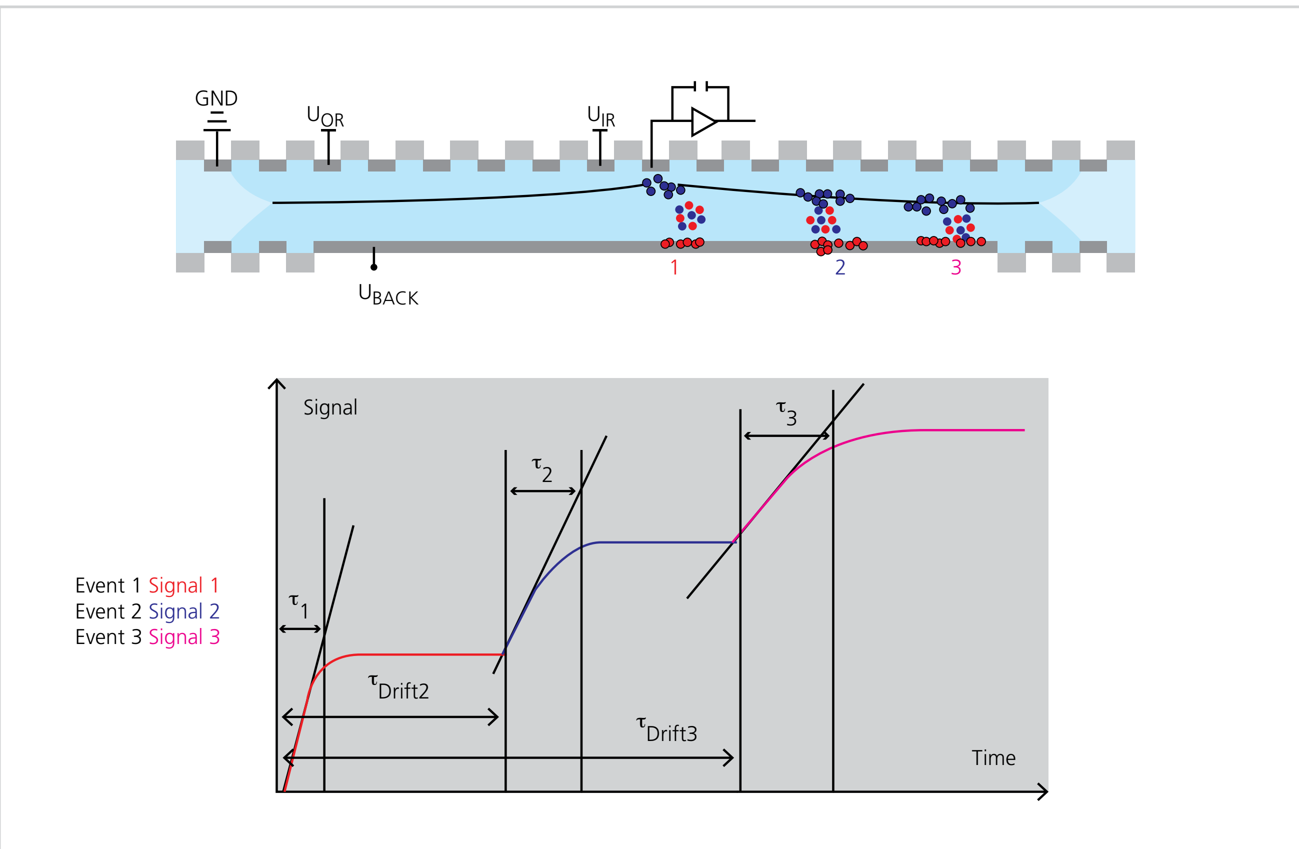


Fig. 20. Electron drift distance and rise time in an SDD detector.

- *X-ray 1 is registered immediately at the input of the pre-amp, therefore the rise time (τ_1) of the signal is short as the drift distance to the anode is very small*
- *X-ray 2 electrons have to drift a longer distance towards the anode and rise time (τ_2) is longer due to diffusion of the charge cloud on its way to the anode*
- *X-ray 3 has the longest drift path and hence the largest diffusion of the charge cloud and the longest signal rise time (τ_3)*

(Image courtesy of KETEK GmbH)

To achieve very high count rates, the signals have to be measured fast using a short processing time. If too short a process time is used, the pulse may not reach its full peak height so there is a deficit in the measurement referred to as 'ballistic deficit' (Fig. 21).

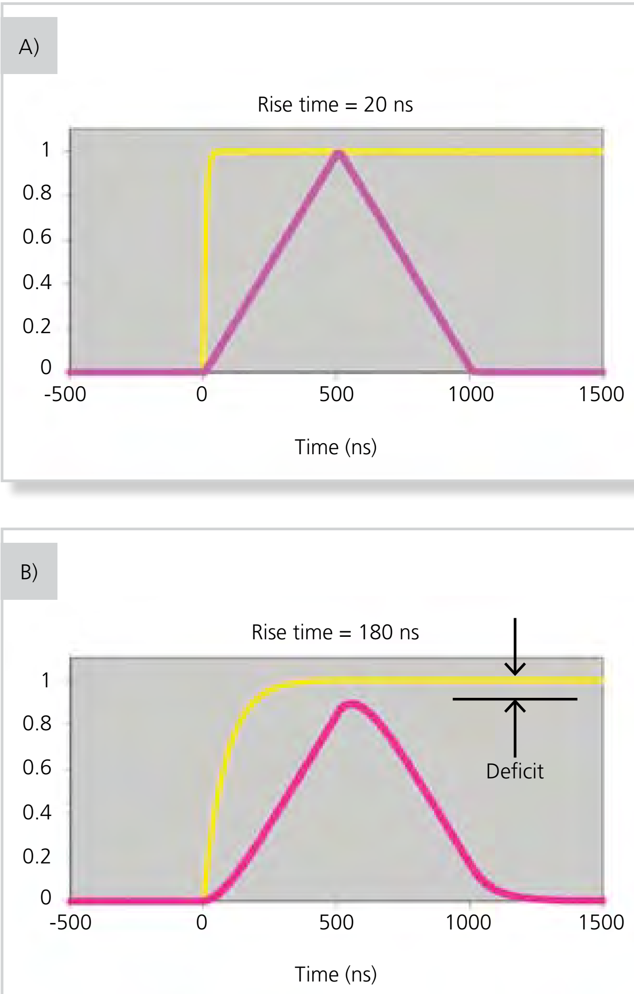


Fig. 21. Signal waveforms with A) 20 ns and B) 180 ns rise times for a process time of 500 ns. The yellow line represents the step output from the FET preamplifier into the pulse processor. If a triangular pulse shaper is used to measure the height of the step the output would be the pink line. Ballistic deficit occurs in B) where the extended rise time causes the output of pulse shaping to be lower than the signal itself.

Ballistic deficit therefore becomes an important issue as the process time decreases, the mode of operation for SDD at high count rate. Because the pulses from a large area detector will have a mixture of rise times, the resulting effect will be similar to incomplete charge collection, with tailing on the low energy side of peaks. The distortion of peak shape away from the ideal model will reduce the accuracy of spectrum processing, particularly for overlapping peaks.

Therefore, to run large area SDD detectors at high count rates the issue of ballistic deficit must be accounted for. The problem is reduced by limiting the path distance of drift to the anode by using a symmetrical design. It is also possible to use special pulse processing techniques to make sure the measurement is insensitive to variations in rise time. Although this limits the maximum speed it does ensure that good analytical performance is maintained.

5.2 Pulse pile-up

Pulse pile-up occurs when pulses from two X-rays arrive so close together that the voltage signals are not discriminated by the pulse processor as separate signals. The result is that the two X-rays are measured as if they were one and a count is added to the spectrum in the channel that is the sum of the energy of the two X-rays. This is most commonly seen in the spectrum as a sum peak which appears at the energy which is the sum of two major peaks at lower energy. In Fig. 22a, a spectrum of Al_2O_3, for example we see a sum peak at 2.97 keV which has been labelled as Ag whereas this is actually the sum peak of AlK + AlK. We also see a sum peak at 2.01 keV which has been labelled as P but is actually the sum peak of OK + AlK. Pulse pile-up can occur with any pair of pulses and continuum X-rays can pile-up with characteristic X-rays to produce a 'pile-up continuum' on the high energy side of major peaks. Pile-up of continuum X-rays with other continuum X-rays can cause the background to extend beyond the Duane Hunt limit or landing energy of incident electrons. The continuum artefacts can affect the accuracy of spectrum processing, particularly for algorithms that use background modelling rather than background filtering. Uncorrected pulse pile-up can cause a wide variety of analytical errors including element misidentification, inaccurate peak ratios and quantitative results which change with count rate.

Pulse processors usually use a pile-up inspection channel to ensure that only one photon is measured at a time. The inspection channel is designed to sense the arrival of every incoming event. The circuit has a time constant that determines the shortest time between events that can be recognised. If the time constant is short, noise levels will be high and the inspector will miss low energy events but will be very effective at spotting high energy events that are close together. If the time constant is long it will see all low energy events but will be unable to distinguish between closely arriving events. If any events are missed, either low energy events or events too close together, the inspector will not be able to veto the measurement so pile-up artefacts will appear. Since a compromise is involved, pulse processors should ideally have more than one of these circuits, each with a different time constant to ensure efficient pulse pile-up protection over the full range of detectable energies.

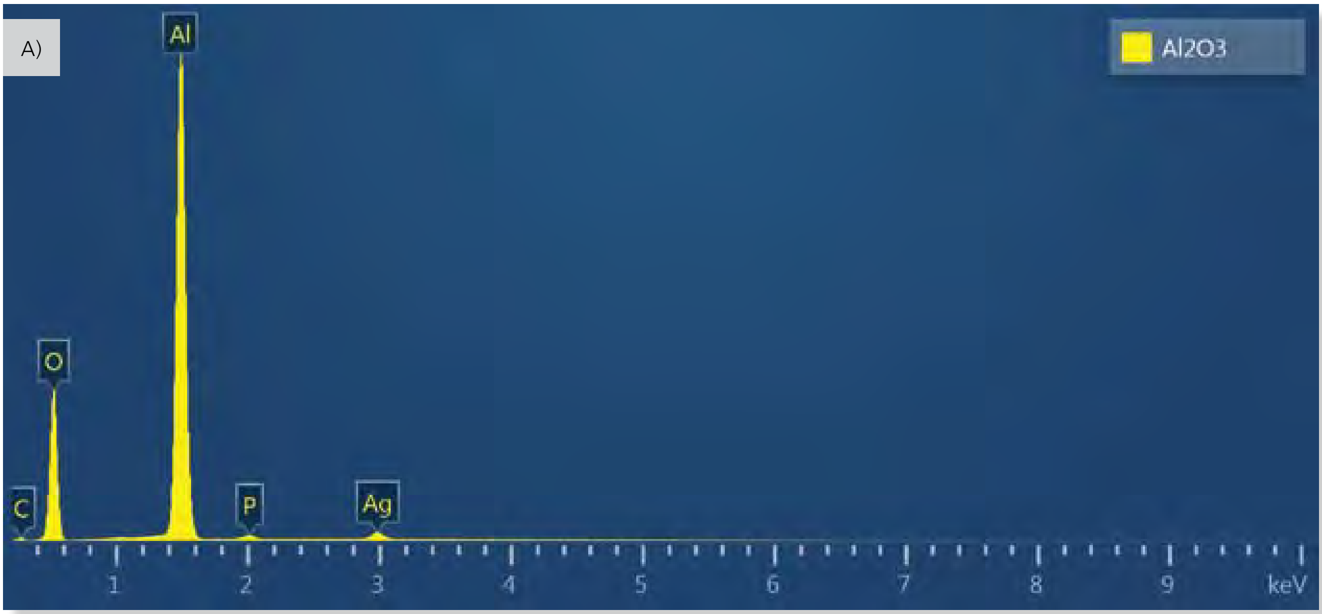
Therefore the result of efficient pulse inspection is to provide a spectrum with no pile-up artefacts. With SDD detectors working at up to 100x greater count rate than was typical for Si(Li) detectors, some pile-up artefacts are inevitable in the spectrum even if there is an electronic pile-up inspector.

A second approach, in addition to pile-up inspection, can be used to correct for any pile-up in the spectrum. This ‘pile-up correction’ needs to identify all pile-up events, remove them from the spectrum, and put them back in the correct spectrum channels. A successful algorithm to fulfil this task has been developed [3], which uses a detailed model of the pile-up inspection circuits and a predictive statistical approach to determining the pile-up in a spectrum. Using this algorithm pile-up events can be detected and corrected in the spectrum even while the spectrum is being acquired (Fig. 22b).

5.3 Evaluating the analytical capabilities of an EDS system at high count rates

To determine the amount of pile-up in a spectrum, the effectiveness of pulse pile-up correction software, or other potential high count rate issues, the best method is to collect two spectra from your sample, one at 1,000 cps where there should be no significant high count rate artefacts, and one at the count rate used for analysis. If the spectra are overlaid, any effects such as sum peaks, background or peak shape changes and extension of the background to energies higher than the electron beam energy, should be obvious.

Quantitative analysis can be checked by comparing the quantitative analysis results of the two spectra. For a particular system and sample of interest the maximum useable count rate for accurate analysis can be determined by increasing the beam current and comparing spectra and quant results with the 1,000 cps result until unwanted artefacts or changes in results are seen. If the system is set up to terminate on a particular number of counts, rather than a set live time, then the results should be equivalent. The acquisition time will, of course, decrease as the count rate is increased.



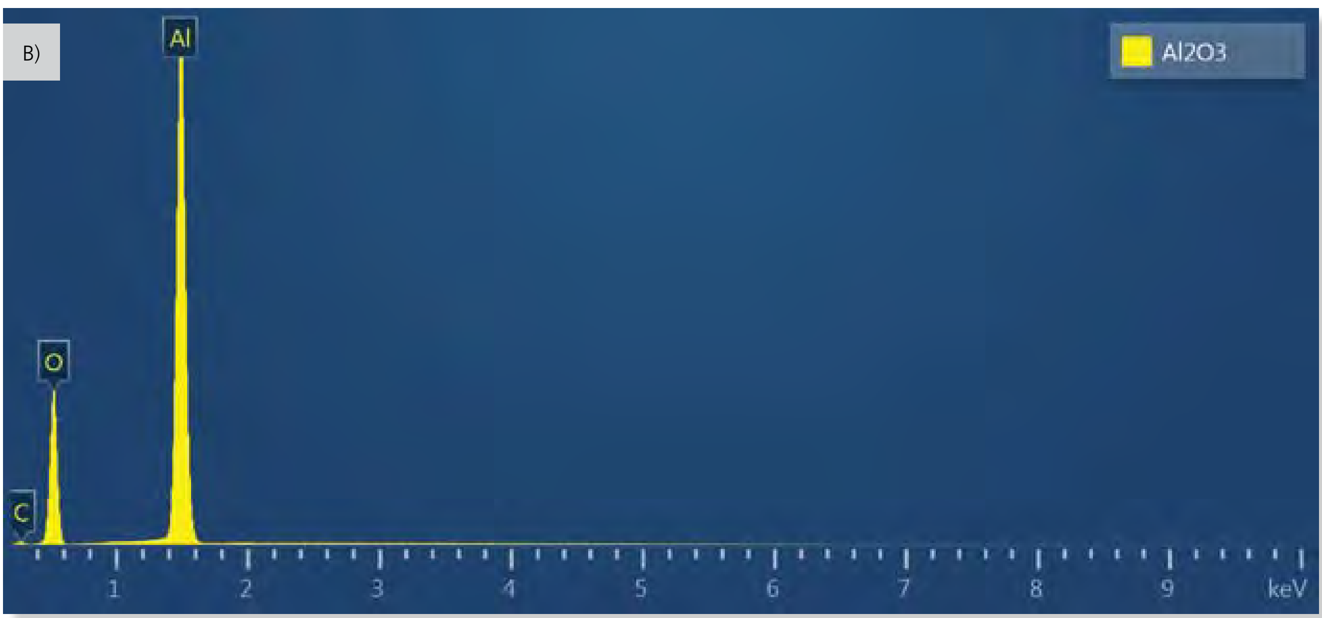


Fig. 22. A) Al_2O_3 spectrum collected at high count rate shows pulse pile-up artefacts with Al+O misidentified as P and Al + Al misidentified as Ag. B) same spectrum with pile-up correction algorithm applied and artefacts removed.

6.0 Summary

SDDs have improved the capability of EDS analysis in the SEM in many ways. For example:

- Best resolution and analytical performance can be achieved at much higher count rates and this results in faster analysis of elements and fast collection of X-ray maps
- Larger sensor sizes with the same analytical performance mean this enhanced productivity can also be achieved under normal microscope operating conditions
- Excellent low energy resolution is possible with very large sensor sizes to maximise count rate and peak sensitivity under low kV operating conditions or when analysing beam sensitive samples

SDDs are achieving their promise to revolutionise EDS analysis, however they remain only one part of the analysis chain. Pulse processor and software design is equally important to achieve a system that produces reliable results at high and low count rates.

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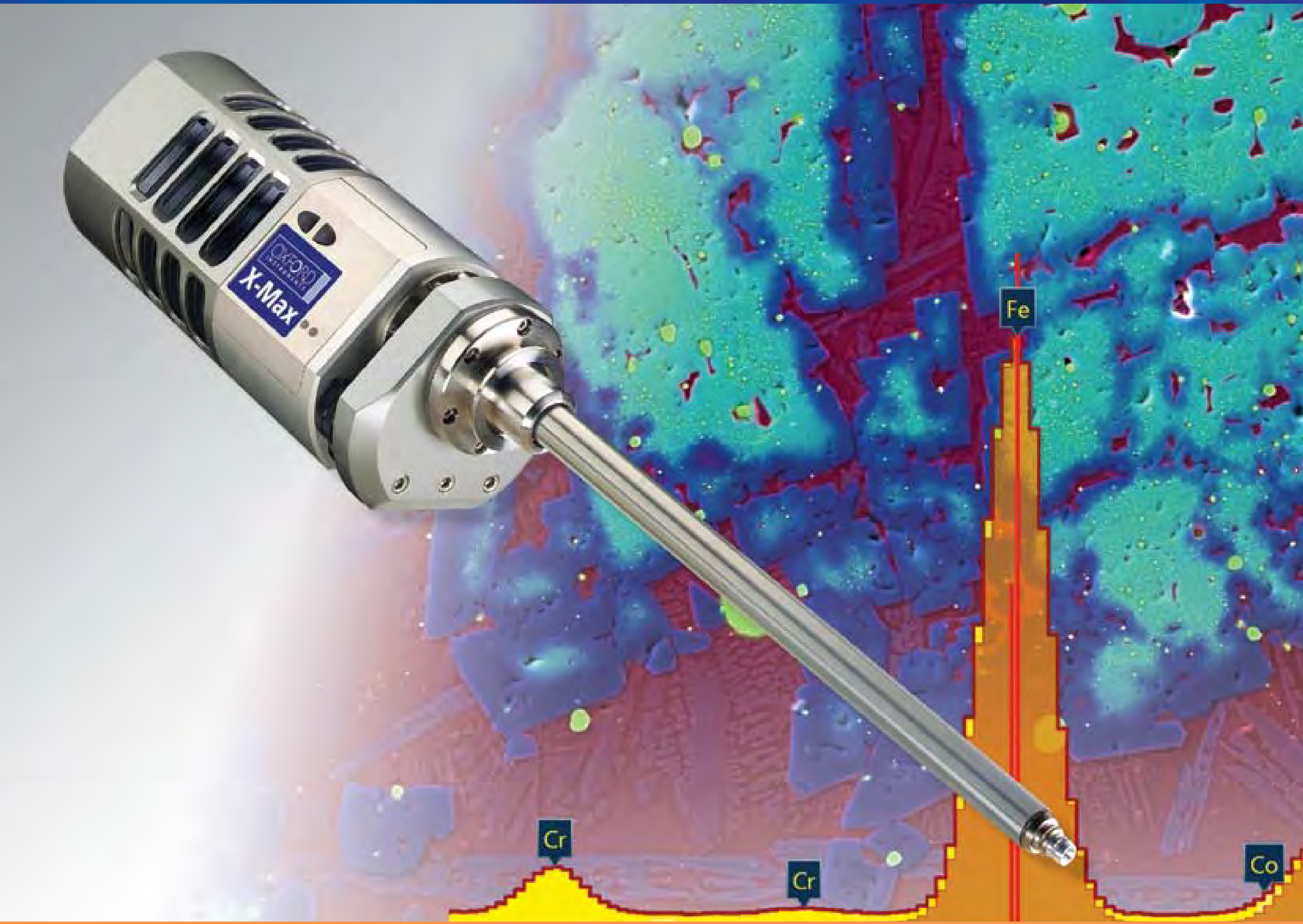

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