New Large Area Silicon Drift Detectors - Fast Analysis without Compromise

Clair Collins, Neil Rowlands, Peter Statham,
and James Holland
Oxford Instruments, High Wycombe, Bucks, England
neil.rowlands@oxinst.com

Introduction

Recent advances in silicon drift detector (SDD) design have set a new benchmark for Energy Dispersive X-ray spectroscopy (EDS). Not only do these detectors offer all the benefits users have come to expect from SDD—high count rates, liquid nitrogen-free analysis and excellent resolution—but large active areas and unique technology allow the user to collect EDS data at normal imaging beam currents and lower accelerating voltages in seconds.

Energy Dispersive Spectrometry (EDS) has been used for many years to analyse the chemical composition of materials. Historically, EDS detectors used a bulk silicon crystal drifted with lithium. Although such Si(Li) detectors had exceptionally good performance, they had limited count rate capability and operated at very low temperatures thus requiring cooling with liquid nitrogen.

In the last few years, new EDS detectors have emerged that do not require liquid nitrogen to cool them. These semiconductor devices, known as silicon drift detectors (or SDDs) were first manufactured in the 1980s for radiation physics, however, recent advances in fabrication methods have meant that they have been developed to become a viable alternative to Si(Li) detectors in a number of SEM EDS applications.

In recent months, new large area EDS SDDs have emerged that offer even greater benefits for micro- and nano-analysis. They combine for the first time the potential for fast analysis and high productivity with operation at low beam currents. They are also ideal for conditions requiring low accelerating voltages that have not previously yielded enough counts for fast mapping or analysis.

How SDDs Work

The silicon drift detector (SDD) is fabricated from high purity silicon with a large area contact on the entrance side and on the opposite side a central small anode contact, which is surrounded by a number of concentric drift electrodes.

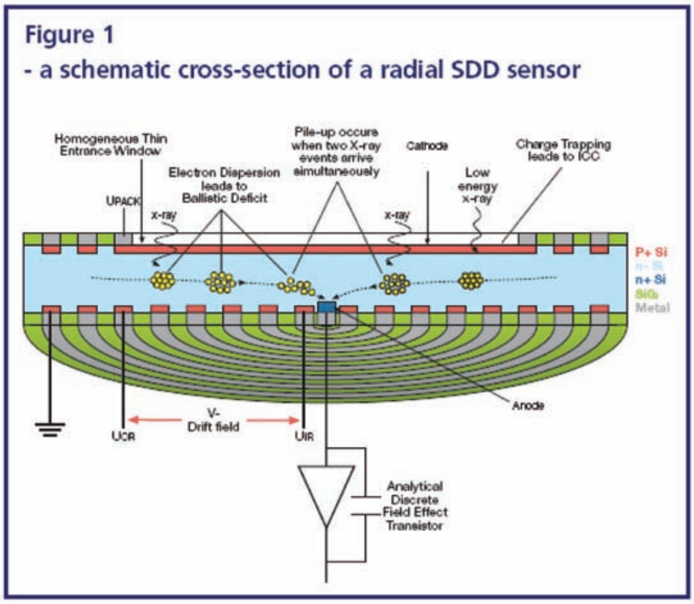


Figure 1. A schematic cross section of a radial SDD detector.

When a bias is applied to the SDD detector chip and the detector is exposed to X-rays it converts each X-ray detected to an electron cloud with a charge that is proportional to the characteristic energy of that X-ray. These electrons are then "drifted" down a field gradient applied between the drift rings and are collected at the anode.

Unlike a Si(Li) detector, however, the size of the anode on an SDD is small in comparison with the entrance contact. This results in lower capacitance and lower voltage noise. Therefore, short time constants can be used to minimize the effect of leakage current so that higher temperature Peltier cooling can be used instead of LN_2.

It also means excellent resolution is achieved even at short process/shaping times and at count rates much higher than conventional Si(Li) detectors.

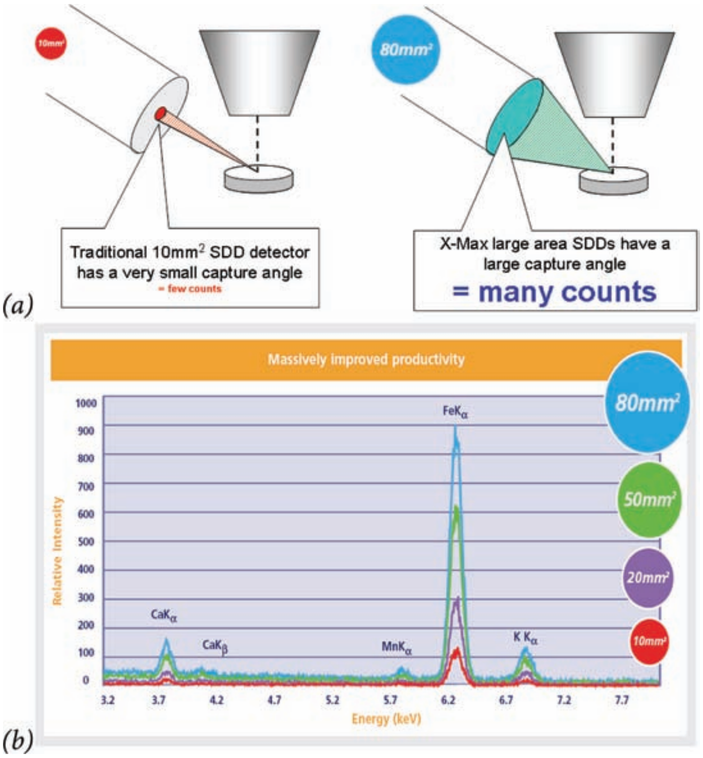


Figure 2. The benefits of large solid angle over small solid angle. (a) A larger capture angle means more counts collected without needing to change collection conditions. (b) Four spectra taken under exactly the same collection conditions showing how the number of counts collected by each detector increases as the size of the detector increases.

The Limitations of Small Area SDDs

The major advantages of the original SDDs were that no liquid nitrogen was required to cool them and that there were no moving parts to cause vibration. The capability to count at faster rates than Si(Li) detectors also promised increased productivity and faster data collection. However, with the true active area of conventional SDD sensors limited to a maximum of $30mm^2$, high-count rates could only be achieved under the following conditions:

1. That enough beam current could be generated without severely defocusing the probe (e.g. when working at low accelerating voltages).
2. That the sample itself produced enough counts (particularly light element samples).

3. That the sample could handle the high beam currents required without becoming damaged.

Because of these limitations, many of the initial adopters of the new SDD technologies found they were unable to work as effectively as they might have wished.

Large Area SDDs

With the latest innovations in manufacturing processes, it has now become possible to manufacture larger area (up to 80mm^2 active area) silicon drift sensors with excellent energy resolution. These new large area sensors have been used for the first time by Oxford Instruments in their X-Max detectors.

The key benefit of these detectors is that these SDD sensors, with active areas an order of magnitude greater than that previously achieved, can still be fitted in the same sized tube as a 10mm^2 SDD detector. Therefore, even an 80mm^2 detector can be positioned at the same sample to detector distance as a conventional 10mm^2 SDD. This increases the collected count rate massively without even having to change the operating conditions. This enables effective analysis at lower beam currents, lower accelerating voltages and at higher magnifications than has previously been possible.

Figure 2a is a schematic illustrating how increased area provides more counts and Figure 2b shows four spectra taken on an almandine garnet sample using a nominal 10mm^2 SDD and the new 20mm^2, 50mm^2, and 80mm^2 X-Max detectors. The spectra were recorded under exactly the same collection conditions and there is clearly no compromise in resolution as the sensor size increases.

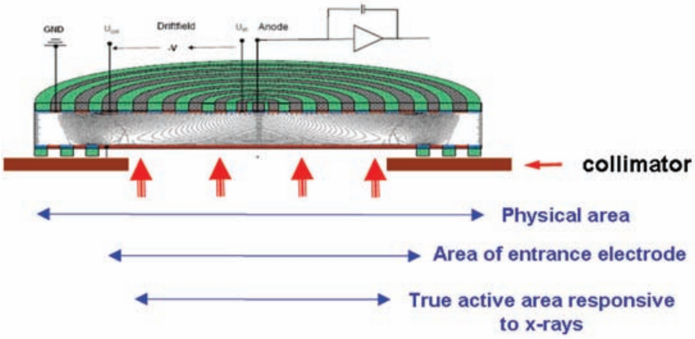


Figure 3. A schematic illustrating the use of collimation to improve spectral performance and the effect this has on active area.

The definition of Active Area

There is no official standard for quoting SDD sensor sizes and many areas are quoted as "nominal". The physical area of silicon that is covered by electrodes is one measure of detector area but that is obviously going to exceed the area that is effective for detection.

A more relevant measure is the area of the electrode through which the X-rays enter. However, because of the complex electrostatic field in an SDD, when an X-ray photon enters the peripheral regions of this contact, some of the liberated charge spills away to the side and does not reach the anode. Such events either produce low energy tailing on peaks or generate increased background. Therefore, to improve spectral performance, a collimator is placed close to the sensor to prevent X-rays reaching these "partially dead" zones (Fig.3). Thus, the collimator aperture reduces the effective area of the detector—it is only within this aperture that X-rays are detected.

X-Max large area SDDs are always specified in terms of the real collimated area rather than other measures that would exaggerate the sensitivity. Thus, X-Max 80 has a true active area of 80mm^2, and an X-Max 50 and X-Max 20 have active areas of 50mm^2 and 20mm^2 respectively.

Performance without Compromise

Before X-Max, very large SDD detectors had not been used because of peak broadening and peak shift due to effects such as ballistic deficit and problems of incomplete charge collection (ICC) at low energies that severely restricted the analytical capabilities of these detectors.

In the X-Max detector, ballistic deficit effects are eliminated by employing a symmetrical detector to minimise variation in transit time across the device and using a digital pulse processing method that is insensitive to rise time variations. An improved entrance contact is used that minimises the effects of reduced field in the larger diameter detectors so that good low energy performance is maintained.

Larger area detectors invite higher count rates and introduce more opportunity for individual X-ray photon induced charge clouds to be in the device at the same time. Electronic pile-up hardware inspectors can go some way to eliminating the effects of two X-ray photons being measured as a single "sum" event at higher energy but are not perfect.

However, after careful characterisation of the imperfections in the pile up inspector for the X-Max detectors, an additional sophisticated software correction scheme is used to remove residual sum peaks and pile-up continuum and correct for losses from peaks so that peak area ratios are preserved for accurate quantitative analysis [1].

The shot noise associated with detector leakage also increases with detector size but this can be controlled by increasing the cooling power. X-Max has been designed to achieve the necessary temperatures without using any auxiliary fans or cooling water that could add vibration. Thus, these detectors are the first SDD detectors to offer the same resolution and analytical performance for all sizes.

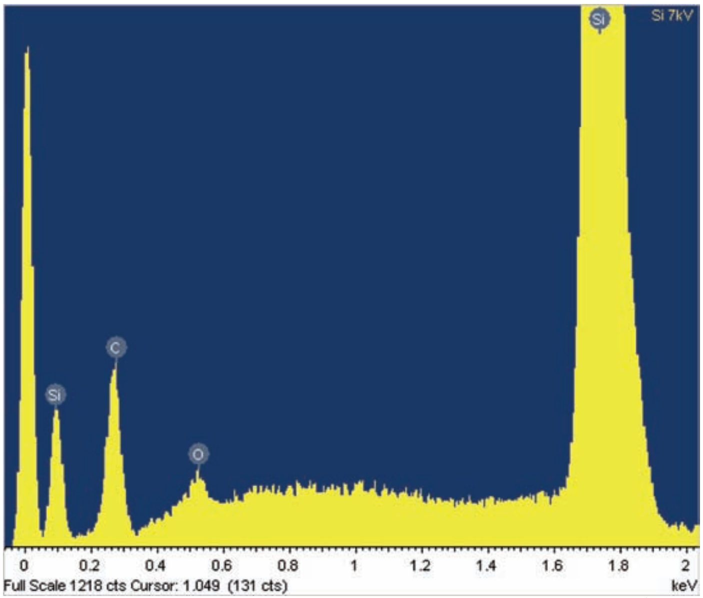
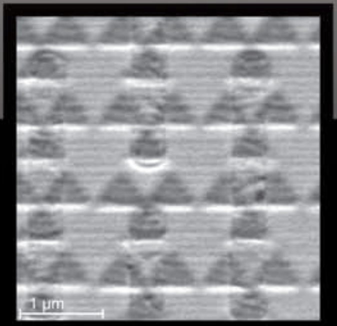


Figure 4. A pure silicon spectrum taken on an X-Max 80mm^2 detector at 7kV for 30 seconds. The noise peak and SiL$_l$ peak can be clearly distinguished, indicating excellent low energy performance.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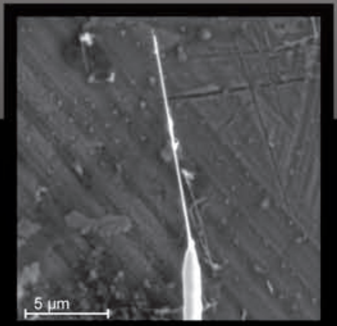
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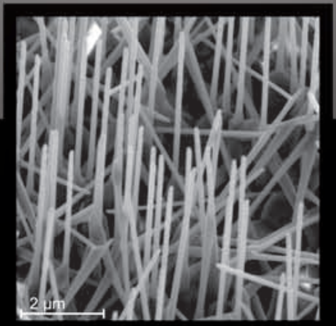
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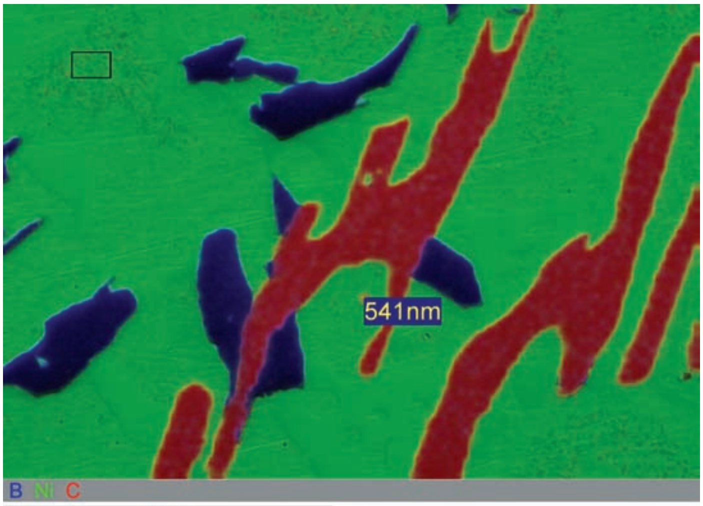


Figure 5. Composite mix of three X-ray EDS maps showing carbon (red) and boron (blue) precipitates in a nickel matrix (green). Collected on a nominal 10mm^2 SDD detector at 5kV, 7kcps, for 200 minutes.

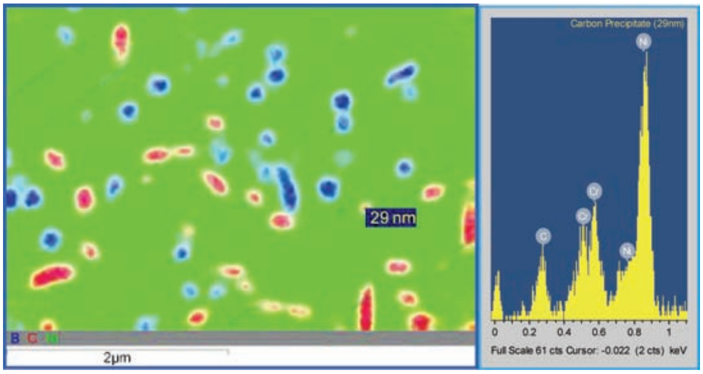


Figure 6. Spectrum image of the magnified area show in the black box in Figure 5. Analysed at 5kV, 13kcps, for 90 minutes. The corresponding X-ray spectrum was reconstructed from the 29nm particle and is equivalent to 0.5 seconds of spectrum acquisition.

Low energy performance

Figure 4 shows an X-Max 80mm^2 spectrum taken from a pure silicon sample. The spectrum has been expanded to show the very low energy lines next to the noise peak. The SiL_l line at 0.09keV is clearly evident and distinct from the noise peak next to it. Only detectors with very low noise and excellent low-energy performance are able to distinguish between the two peaks.

Examples of the use of large area SDD detectors in X-ray mapping.

Typically, when EDS detectors are used, the SEM probe size has to be increased to generate enough beam current and this gives a poor electron image. However, with the increased solid angle offered by the X-Max large area SDDs high count rates can be achieved under standard SEM imaging conditions. Furthermore, when low accelerating voltages are used to minimise the X-ray interaction volume to analyse small features, and the X-ray yield drops off dramatically, it is particularly useful to have a large area detector that will still achieve an acceptable count rate under these low-yield conditions. With the increased count rates offered by large area SDDs, spectra may be collected in a fraction of the time required previously, and mapping can often be used rather than selected point analysis.

1. Effect of larger area in detecting smaller features

Figure 5 demonstrates the high-count rates achieved with a nominal 10mm^2 SDD The map shows well resolved features at the micrometer level as well as particles as small as 500nm, but it still took 3 hours to map this data at the probe size conditions needed to resolve the features.

Using an X-Max 80, a smaller area (see box in fig. 5) was mapped on the same sample at a higher magnification and at a much lower beam current. It can be seen in Figure 6 that precipitates as small as 30nm are clearly observed and this data was acquired in less than half the time needed to gather a map at lower magnification on the 10mm^2 detector. The spectrum taken from the 29nm particle also clearly indicates the presence of carbon and demonstrates the excellent low energy detection that is achieved with the large area device.

2. Comparison of maps collected at constant beam current

To illustrate the effect of solid angle on spectrum image results, two maps were collected under exactly the same beam current conditions. The first map (Fig. 7a) was collected on a nominal 10mm^2 SDD and the second map (Fig. 7b) was collected on an 80mm^2 X-Max large area SDD detector.

The benefit of the larger active area is evident by comparing the two maps. While the phases in Figure 7a are reasonably well defined, the greater collection efficiency in Figure 7b vastly increases the number of counts collected in the X-ray map. The result is easy identification of separate phases and finer details in the maps. Furthermore, with large recorded counts, there are sufficient statistics to perform detailed quantitative analysis of selected areas from spectral data stored in the spectral data cube.

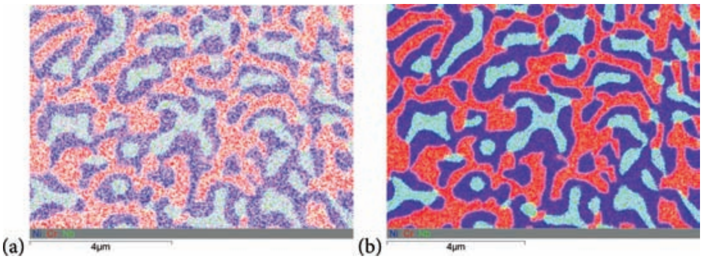


Figure 7. Two spectrum images of a spinner bowl material, both collected at the same beam current (3nA) using (a) a nominal 10mm^2 SDD and (b) the large area 80mm^2 X-Max SDD detector.

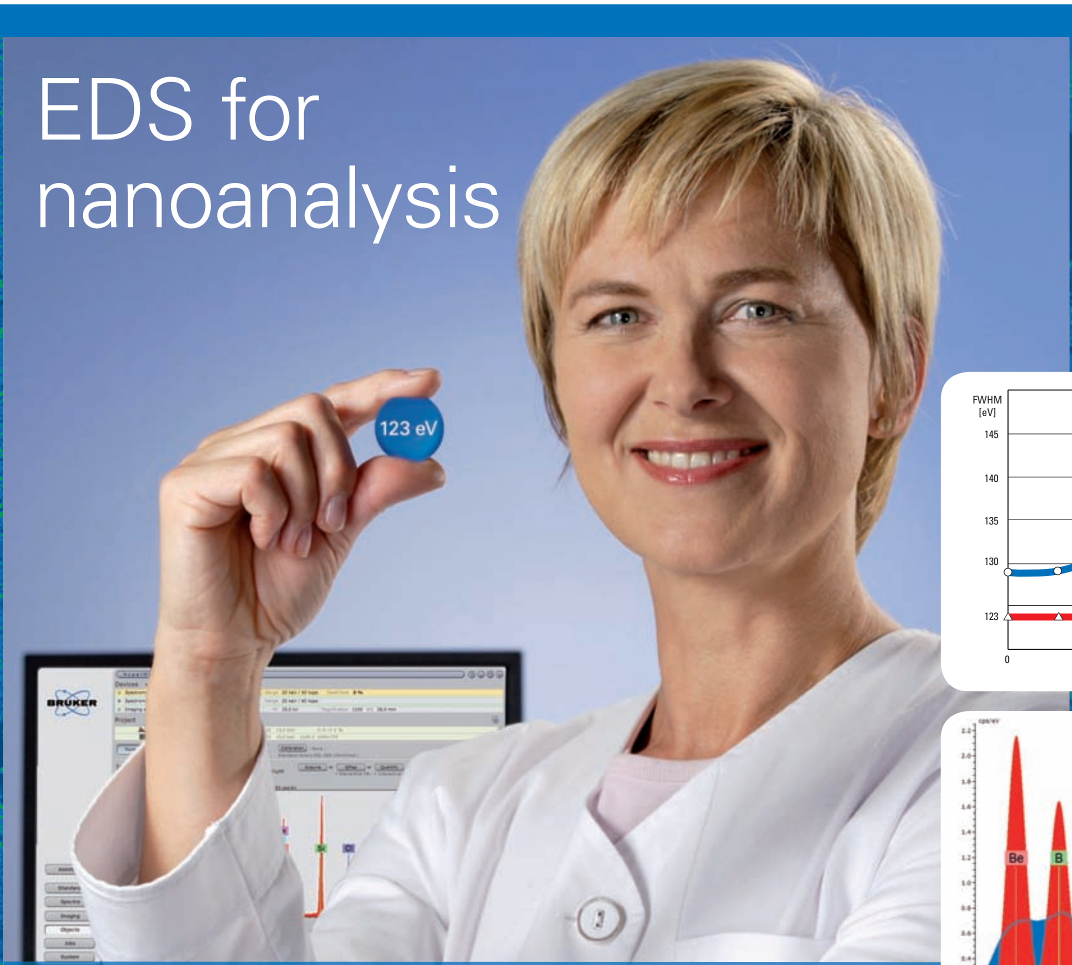
Summary

This new generation of large area SDDs offers significant advantages over previously existing technology. Advances in detector technology, signal processing and software have enabled large area detectors to be produced that offer performance that was previously only possible with much smaller SDDs.

Consequently, low kV mapping of light elements, fast and accurate quantitative analysis, and acquisition of large spectrum images or data-cubes may be achieved in far less time than was previously attainable. The improved sensitivity facilitates analysis of very fine particles, both on solid substrates and on transmission stages in the SEM. In addition, the ability to work at lower beam currents reduces specimen contamination and allows higher resolution electron images to be acquired simultaneously with X-ray data. ■

References

[1] P.J.Statham, Microchimica Acta, 155, 289-294, 2006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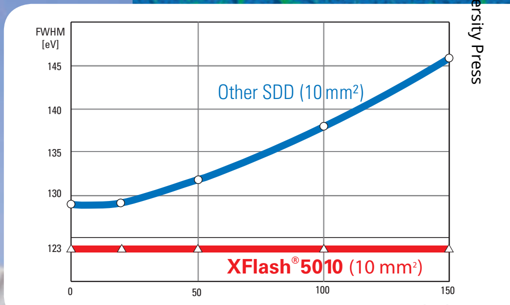
FWHM [eV]
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140
135
130
123
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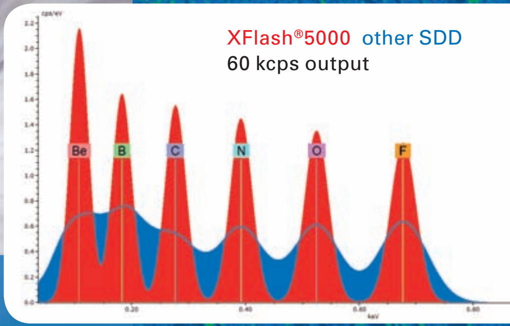
50
100
150
Input count rate [kcps]
Other SDD (10 mm²)
XFlash® 5010 (10 mm²)


XFlash®5000 other SDD
60 kcps output
Be
B
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