QUANTAX FlatQUAD

- EDS for SEM with the XFlash® FlatQUAD

Innovation with Integrity

EDS

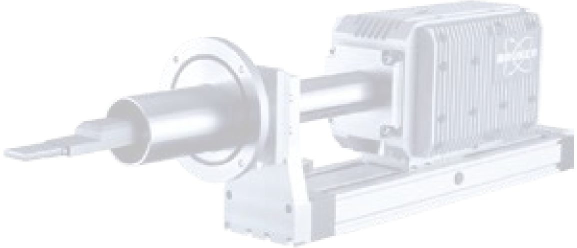
Maximum Efficiency in X-ray Detection...

QUANTAX FlatQUAD is the EDS microanalysis system based on the revolutionary XFlash® FlatQUAD. This special annular four-channel silicon drift detector is inserted between SEM pole piece and sample, achieving maximum solid angle in EDS. In combination with the ESPRIT analytical software suite QUANTAX FlatQUAD provides previously unheard of mapping performance, even for the most difficult samples.

Employing latest detector technology

The XFlash® FlatQUAD, the core of QUANTAX FlatQUAD, is based on a novel detector concept. This includes positioning the detector from the side between pole piece and sample. Therefore, the detector is mounted on a horizontal port on the SEM chamber. Conventional detectors, which rarely extend beneath the pole piece require an inclined port. To ensure compatibility with many different SEM types, the detector can be precisely positioned in X, Y and Z direction.

The four independent silicon drift detector chips of the XFlash® FlatQUAD are arranged annularly around a hole in the detector module. The primary electron beam passes through this opening. The materials the detector is constructed from, were chosen to avoid influences on the electron beam. This design as well as the intention to keep the detector finger as thin as possible require a new method to prevent backscattered electrons from reaching the detector chips: The detector is equipped with special polymer windows of varying thickness. They absorb the backscattered electrons while allowing X-rays to pass through. The polymer windows are mounted in a slider which permits changing them without affecting the vacuum. This enables SEM acceleration voltage changes while the detector is in measurement position.



...with the XFlash® FlatQUAD

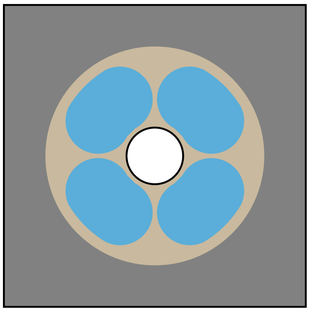
Best solid angle in EDS for SEM

The position and size (4 × 15 mm^2 active area) of the detector chips provide the largest solid angle for X-ray collection in a SEM. Depending on the specific geometrical conditions more than 1 sr is possible in combination with a high take-off angle of 60° or more.

No compromise between count rate and energy resolution

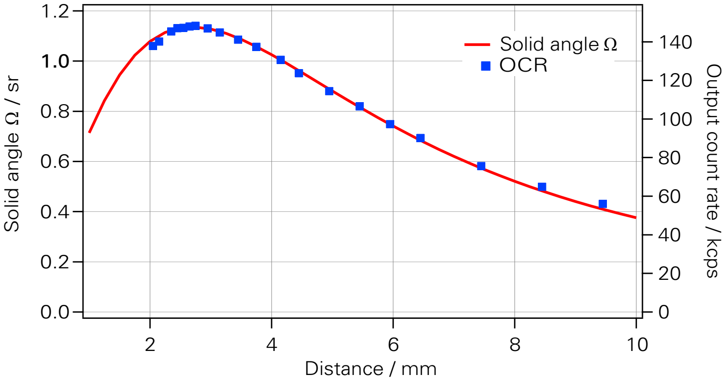
The collection efficiency can lead to ultra-high count rates. All four detector chips are therefore equipped with separate signal processing channels. This allows input count rates (ICR) of up to 4,000,000 cps and a combined output count rate (OCR) of up to 1,600,000 cps. Bruker's expertise in SDD technology makes XFlash® FlatQUAD available with an excellent energy resolution of 126 eV at Mn Kα and 100,000 cps input count rate (51 eV at C K and 60 eV at F K). Resolution classes of 129 and 133 eV are also available.

Detector layout



This schematic shows the annular arrangement of the four detector segments. Their active area is approximately kidney-shaped to provide optimum solid angle and detector resolution at the same time. The anode and on-chip preamplifier are positioned to one side out of the irradiated area for improved performance. The segments are protected by an on-chip 1 µm polymer window.

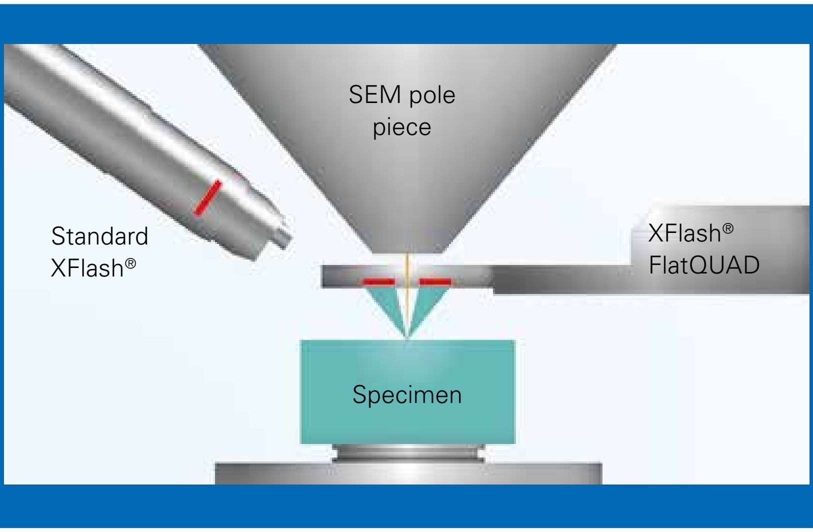
Solid angle and OCR as a function of the detector-sample distance



Plot of the output count rate (OCR) that can be achieved on copper at 5 kV accelerating voltage and 1 nA beam current and the according detector solid angle, theoretically calculated according to Nestor J. Zaluzec, *Detector Solid Angle Formulas for Use in EDS*, Microsc. Microanal. 15 (2009), 93

Functional principle

The XFlash® FlatQUAD is a side entry detector. It is positioned between SEM pole piece and sample. The primary electron beam can pass through the center hole around which the four detector segments are arranged. The solid angle for radiation collection that can be achieved depends on the distance between detector and sample. It can be varied by using the Z drive of the SEM stage. A set of included electron absorbing polymer windows can be changed in situ.



Application Examples

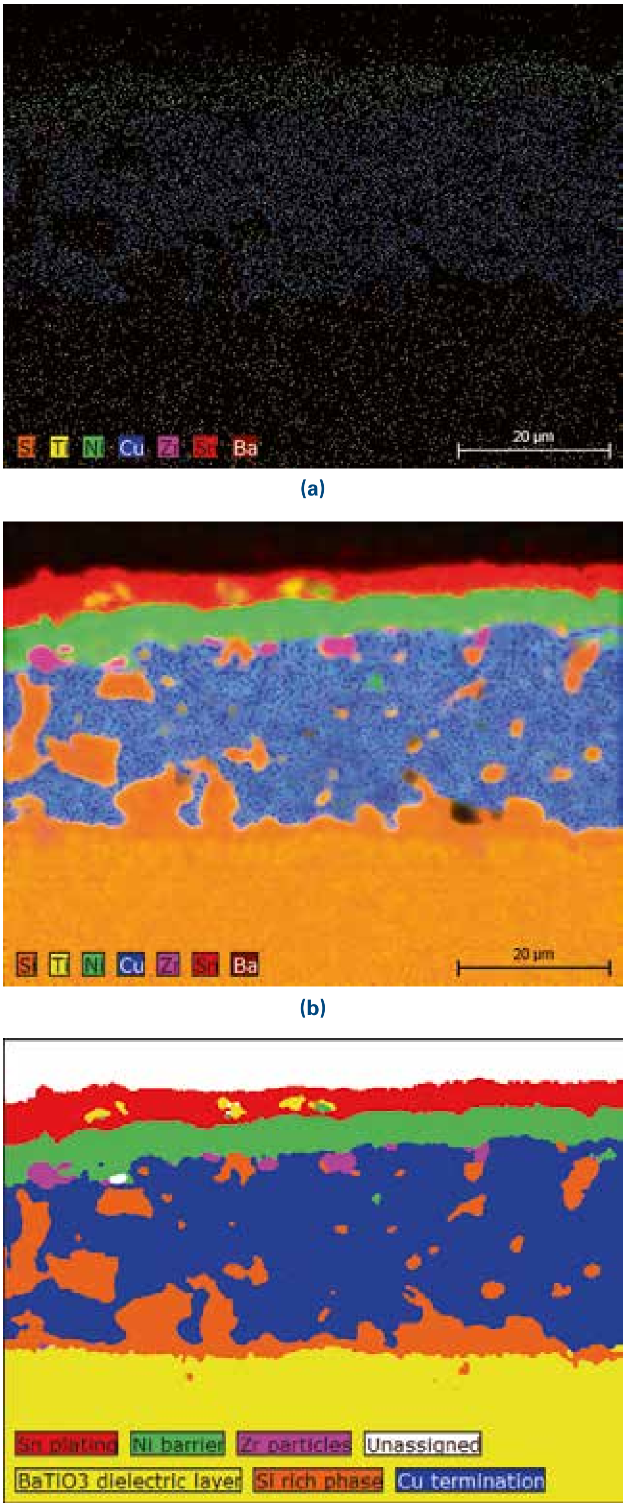
QUANTAX FlatQUAD is an EDS micro- and nano-analysis system that performs where conventional systems reach limitations:

- Extremely fast mapping at highest output count rate, using only moderate beam currents
- Analysis of beam-sensitive materials at low to extremely low beam currents (< 10 pA), e.g. of biological or semiconductor samples
- Investigation of samples with topography, avoiding shadowing effects
- Analysis of nanoparticles and nanostructures at low kV and highest magnification
- Measurement of thin samples (e.g. TEM lamellae) and other specimens with low X-ray yield.

Fast mapping

QUANTAX FlatQUAD can operate at up to 100× the speed of conventional SDD-based EDS systems. This enables map acquisition with excellent statistics in seconds without compromising SEM performance parameters.

Analysis of a ceramic capacitor



(a)

(b)

(c)

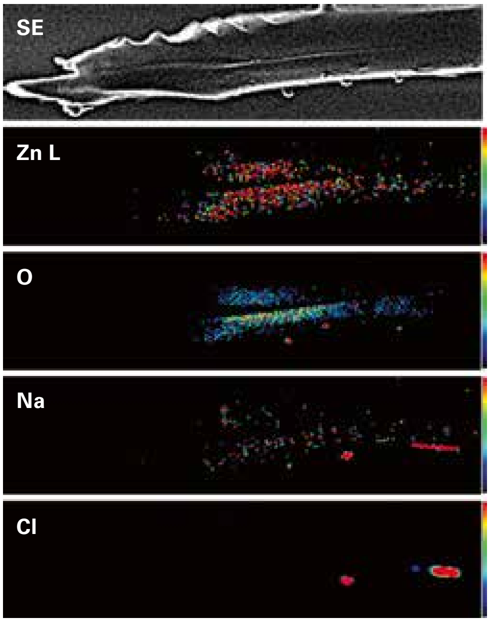
(a) Map of a multilayer ceramic capacitor containing Si, Ti, Ni, Cu, Zr, Sn and Ba obtained with a conventional EDS detector, showing very poor statistics. Size 512 × 384 pixels, HV 10 kV, acquisition time 170 s, input count rate (ICR) 260 cps, 41,000 counts total.
(b) Same specimen mapped under identical conditions using the XFlash® FlatQUAD, ICR 28,000 cps, 4,800,000 counts total.
(c) Results obtained with the XFlash® FlatQUAD can be used for further processing, e.g. chemical phase analysis as shown here.

Analysis of beam-sensitive and non-conducting samples

Thanks to the extremely large solid angle of the XFlash® FlatQUAD, QUANTAX FlatQUAD supports convenient analysis of samples that are difficult or even impossible to investigate using conventional systems, as it already starts performing with beam currents in the pA range:

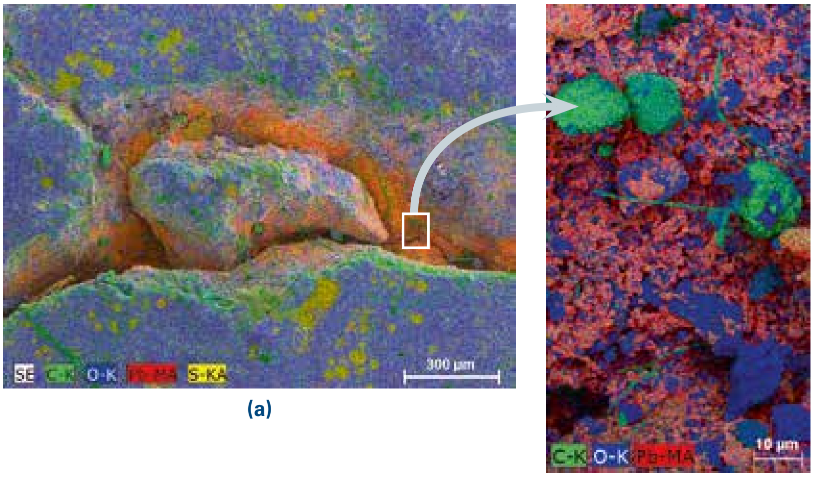
- Sensitive samples can be analyzed without electron beam damage.
- Non-conducting specimens can be investigated under high vacuum without carbon coating. Compared to low vacuum analysis, this approach reduces hydrocarbon contamination and avoids beam skirting effects.
- Samples with low carbon content can be analyzed, as carbon deposition caused by the electron beam is drastically reduced and result falsification avoided.
- Valuable samples (e.g. cultural heritage objects) can be analyzed in their original state without destructive sample preparation.

Ovipositor of a parasitoid wasp



The study of the ovipositor (sting and egg layer) of the parasitoid wasp *monolexis fuscicornis* was carried out under low vacuum (20 Pa, 5 kV, 1.8 nA, 20 kcps, 320x240 pixels, 460 nm pixel size, 31 min). (Sample courtesy: G.R. Broad, The Natural History Museum, London, UK). It reveals reinforcement by ZnO biomineralization and contamination with NaCl. Sufficient data quality allows of overlapping element lines (Zn-L, Na-K) deconvolution using an automatic routine.

Historic stony meteorite “Mocs”



(a)

(b)

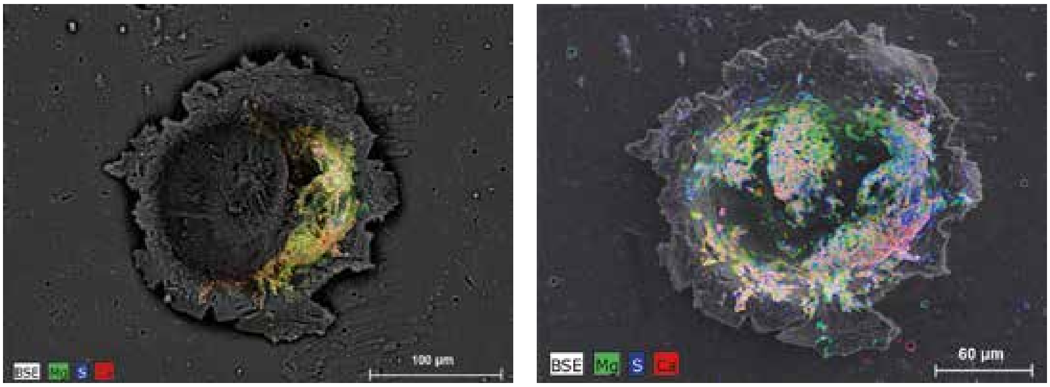
The analysis of a historic stony meteorite “Mocs” that fell on 3 February 1882 was carried out under high vacuum and ultra low beam current without conductive coating (6 kV, < 10 pA, 2 kcps). (Sample courtesy: L. Ferrière, Natural History Museum, Vienna, Austria)
(a) Composite EDS element map and SE micrograph overlay. Overlapping peaks (Pb-M, S-K) were deconvolved with an automatic routine. It allows to discriminate sulfides (yellow) from lead contamination. The latter is a result of old polishing (800x600 pixels, 2 µm pixel size, 17 min).
(b) Composite EDS map of the area highlighted in (a) showing carbon features <300 nm indicating soot contamination by heating with coal-fired furnaces (130 nm pixel size, 5h 10 min).

Analysis of samples with complex topography

Standard EDS detectors are at a disadvantage when it comes to mapping samples with topography, as their view from one side restricts them from detecting radiation from all parts of the sample – shadowing occurs.

The XFlash® FlatQUAD with its 4 detector segments positioned directly above the specimen is affected far less by this phenomenon. This permits complete investigation of all sample areas hit by the electron beam.

Micro impact crater

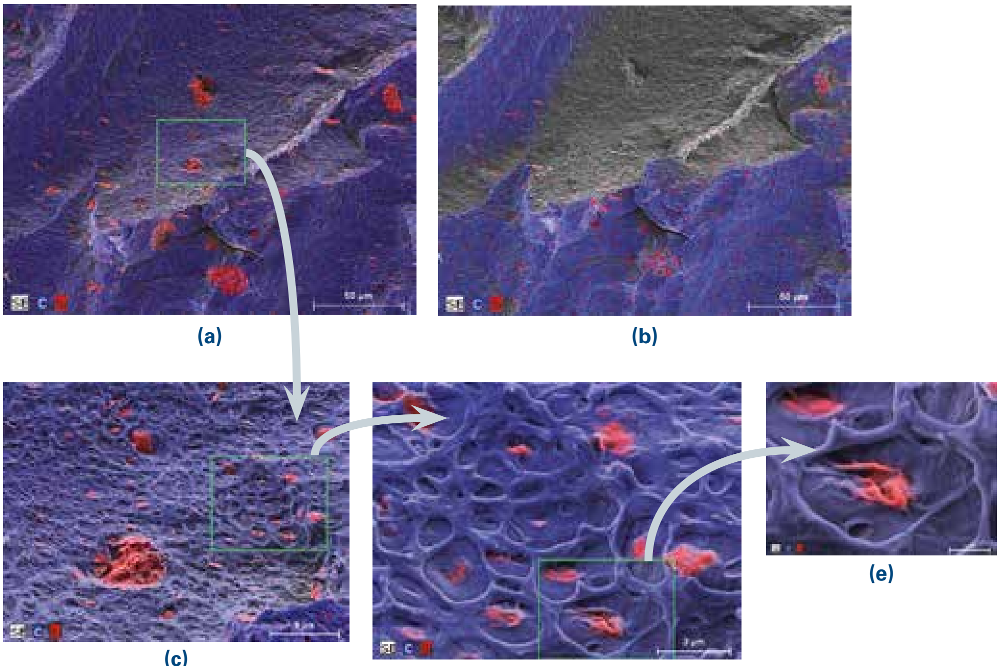


(a)

(b)

(a) Overlay of backscattered electron image and map of Mg, S, and Ca of an artificial impact micro crater created to support understanding of those created during the NASA Stardust mission, obtained with a conventional SDD on a 35° port. Only part of the impacted particle residue is visible (20 kV, 3 nA, 15 h 54 min,1024 x 768 pixels, 380 nm pixel size). (Sample courtesy: A. Kearsley, Natural History Museum, London, UK)
(b) Same crater mapped with the XFlash® FlatQUAD. No shadowing, the complete particle residue is mapped (6 kV, 3 h 55 min, 175 kcps, 800 x 600 pixels, 380 nm pixel size).

Polymer with nano clay particles



(a)

(b)

(c)

(d)

(e)

(a) SE image and C and Si map overlay of a low density polyethylene composite with organoclay (organically modified montmorillonite) nanoparticles, obtained with the XFlash® FlatQUAD. The clay particles have formed large agglomerates (Sample courtesy: D. P. da Silva Dalto, Federal University of Rio de Janeiro, Brazil). All maps acquired with 3 kV, 220 pA beam current, 300 s acquisition time. The XFlash® FlatQUAD produced an ICR of 16 kcps (30 mm^2 detector: 0.5 - 1.4 kcps).
(a) Size 1024 × 768 pixels, pixel size 224 nm.
(b) Same sample region mapped with a 30 mm^2 detector, shadowing occurs.
(c) Sample detail as indicated in (a), 45 nm pixel size.
(d) Detail from (c), 24 nm pixel size.
(e) Detail from (d), 14 nm pixel size.

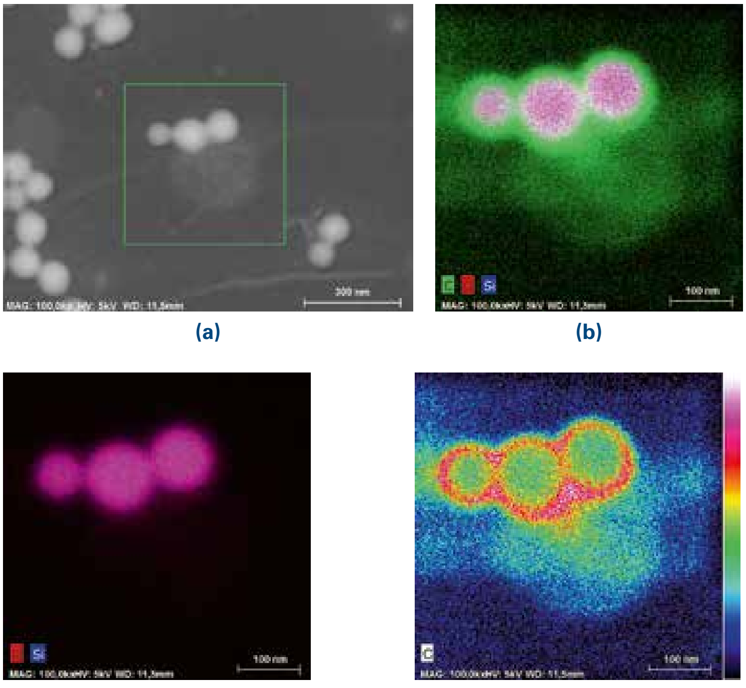
Meeting the Most Difficult Analytical Challenges

Specimens with low X-ray yield

Its excellent collection efficiency allows the XFlash® FlatQUAD to analyze samples that provide only low X-ray yield and still deliver excellent analytical results. Apart from biological samples these can be thin samples of all kinds.

Another broad field of application are nanoparticles. Here the analysis with SEM and QUANTAX FlatQUAD offers an additional benefit compared to other methods. Not only can compositional information be provided but nanoparticle agglomerates can also be resolved.

Nanoparticles



(a) (b) (c) (d)

(a) SE image of agglomerates of SiO_2 nanoparticles covered with an organic dye at roughly 100,000× magnification. The green rectangle indicates the area mapped with the XFlash® FlatQUAD. (Sample courtesy: S. Rades, K.Natte, T. Behnke, BAM Federal Institute for Materials Research and Testing, Berlin, Germany). The research leading to these results has received partial funding from the European Union Seventh Framework Programme (FP7/2007–2013) under grant agreement no. 263147 (NanoValid – Development of reference methods for hazard identification, risk assessment, and LCA of engineered nanomaterials).
(b) Mixed element map of an agglomerate of 3 nanoparticles. Size 250 × 250 pixels, HV 5 kV, beam current 520 pA, acquisition time 391 s.
(c) Mixed map of Si and O, measurement parameters as before.
(d) Single element map of carbon in false color coding to emphasize the intensity of radiation, measurement parameters as before. The organic dye covering the nanoparticles can be clearly recognized by its reddish/pinkish color, indicating the highest carbon intensity in the map. Also, it seems that dye has leached from the particles, spreading on the substrate, recognizable as a greenish patch close to the particles. This patch is also visible in (a) and (b). It is most likely an effect of preparing the sample onto a carbon TEM membrane.

Technical specifications of the XFlash® FlatQUAD

Parameter	Specifications
Energy resolution	Energy resolution of 126 eV at Mn Kα, 51 eV at C Kα, 60 eV at F Kα Also available: 129 eV at Mn Kα, 58 eV at C Kα, 66 eV at F Kα 133 eV at Mn Kα, 65 eV at C Ka, 73 eV at F Kα Stated in compliance with ISO 15632:2012 and guaranteed at 100,000 cps input count rate
Element range	Boron (5) to americium (95), further details see table below
Input count rate	Up to 4,000,000 counts per second (cps)
Output count rate	Up to 1,600,000 cps
Active area	60 mm^2 (4 × 15 mm^2)
Cooling	Liquid nitrogen-free Peltier cooler, vibration-free
Operating pressure	Max. 30 Pa permissible during detector operation
Detector mount	Horizontal port with 33 mm minimum diameter required (fixed Y and Z position), larger diameter to make use of detector XYZ adjustment
Window changer	3-position window changer with 2 polymer windows and vacuum passthrough, see also table below
Interference	Minimal interference with SEM through use of non-magnetic materials

Polymer window options for the XFlash® FlatQUAD

Window type	Total thickness / µm	Maximum SEM high voltage / kV	Low energy performance
On-chip, fixed	1	6	B, C, N, O, F detectable
2 µm polymer window	1 + 2	12	B, C, O, F detectable, N near LOD*
6 µm polymer window	1 + 6	20	F near LOD

*LOD: Limit of detection

For further information scan the QR code or visit **www.bruker.com/quantax**

Bruker Nano GmbH
Berlin · Germany
Phone +49 (30) 670990-0
Fax +49 (30) 670990-30
info.bna@bruker.com

www.bruker.com