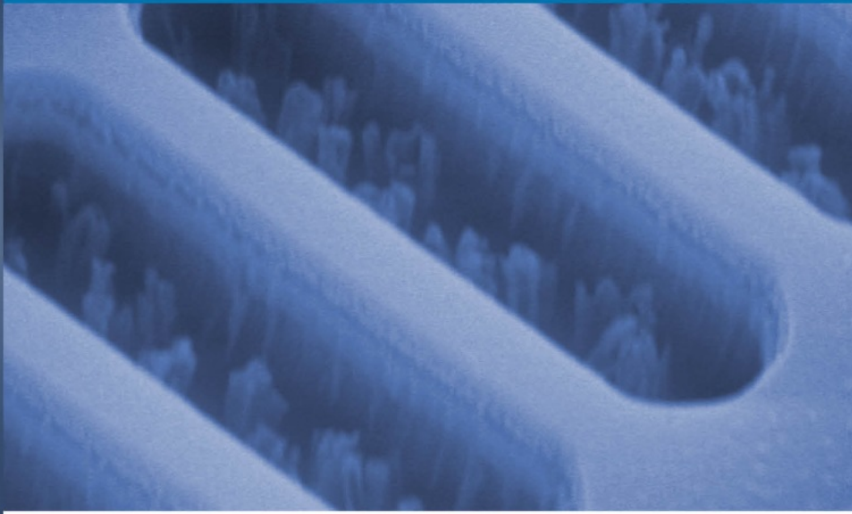

XFlash® FlatQUAD

SD³ detector for energy-dispersive spectrometry

Reference Manual

EDS

Innovation with Integrity

Bruker Nano GmbH
Am Studio 2D
12489 Berlin

Tel.	+49 (30) 670 990-0
Fax	+49 (30) 670 990-30
E-mail	service-berlin@bruker-nano.de
Internet	www.bruker.com

We have checked the contents of this manual for agreement with the hardware and software described. Since deviations cannot be precluded entirely, we cannot guarantee full agreement. However, the data in this manual are reviewed regularly and corrections will be included in subsequent editions.
Suggestions for improvement are welcome.

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Contents

1 Introduction

With the innovative XFlash® FlatQUAD detector Bruker Nano GmbH provides a versatile semiconductor X-ray detector of the latest generation utilizing the novel functional principle of a side-entry silicon drift detector with droplet shaped sensors (SD^3). The XFlash® FlatQUAD detector is easy to install and use, especially in conjunction with an XFlash® signal processing unit (SVE).

The detectors are maintenance-free and compact. However, there are certain operational guidelines that have to be observed. The advice and warnings given in this manual should be strictly adhered to in order to avoid dangerous situations or damage. Moreover, this manual gives information about optimizing the performance of the XFlash® FlatQUAD detector as well as an overview over the various options provided by Bruker Nano GmbH.

2 Specification

2.1 Mechanical Characteristics

The XFlash® FlatQUAD detector consists of a four-element detector module and a preamplifier case. Both parts are connected to each other through the detector finger.

The preamplifier case forms a heat sink to dissipate the heat generated both in the preamplifier and the detector. The detector finger length is designed to the dimensions of the respective SEM specimen chamber. The four-element detector module itself is housed in a sandwich like flat case. The detector windows are located in the bottom plate of the flat part of the case and grouped around a central whole, which acts as an aperture for the SEM electron beam. With this setup X-rays emerging from the illuminated specimen can be detected over a wide and uniform solid angle.

The manual movement stages in y and z direction and a motorized drive in x direction are located outside of the specimen chamber. They allow adjusting of the detector with respect to the pole piece of the SEM and the beam center. Moreover, the x-drive allows bringing the detector into a parking position whenever it is not used.

Beware!

During adjustment and use of the detector the front end must neither touch the pole piece nor the specimen or any other part. The detector finger is susceptible to damage.

Also take special care when moving any other detectors within the specimen chamber or the microscope sample stage.

2.2 Cooling System

The cooling system of the XFlash® FlatQUAD detector consists of two stages. The first thermoelectric cooler (Peltier element) is located at the joint between the flat detector case and the detector finger. It provides cooling for the complete front part. The second stage (at the far end of the detector finger) has the function to remove the heat, which is generated by the first thermoelectric cooler and the other parts of the detector module. The preamplifier case functions as a heat sink for dissipating the all generated heat by unforced convection. Thus completely vibration free cooling is provided for all parts of the detector.

The complete front- part of the XFlash® FlatQUAD detector is cooled. In contrast to a standard XFlash® detector with a hermetically sealed detector crystal, the cooler must only be operated while the specimen chamber is evacuated.

Beware!

Switch off the cooling circuit and allow approx. 15 minutes for warming up of the detector to at least +15 °C before venting the specimen chamber or opening the chamber door. In addition, cooling must only be started after the SEM is evacuated. Cooling down to a temperature of -25 °C takes approx. 15 minutes.

2.3 Detector Window

The detector comes equipped with either a polymer window or a polymer-coated beryllium window of 12 µm thickness. It is integrated into the zirconium collimator.

Warning!

Beryllium is extremely fragile and toxic. Fragments of a broken detector window must not escape into the environment, and must not be swallowed or inhaled.

For the polymer window a thin foil is used (thickness 1 µm) which allows a maximum excitation of ≤6 keV.

For higher voltages two additional windows with different thicknesses mounted on a slider can be positioned below the sensor elements. The window changer is operated manually. The gear is located at the heat sink and can be operated in-situ under vacuum conditions. Placing these windows beneath the sensors will result in different spectroscopic properties (depending on the thicknesses of the additional windows) as listed below:

Internal window (1 µm) only allows HV of ≤ 6 keV

Additional 2 µm window allows HV of ≤12 keV

Additional 6 µm window allows HV of ≤20 keV.

Beware!

Never use higher HV values than the ones given for a specific window combination as this can damage the detector.

If the window is set incorrectly, electrons with high energy can pass through the window and impact on the detector chip. This causes spectrum distortion with a higher background and leads to inaccuracy in quantitative analysis. Moreover, the detector can be damaged over time.

The polymer X-ray window is also transparent to light. Therefore, the detector must only be used in complete darkness.

2.4 Output Signals

The standard detector control unit is the SVE 6. The detector communicates with the control unit via following signals:

The detector preamplifier output signal is a positive pulse signal with a rise time of approx. 70 ns to 90 ns. Subsequent voltage steps form a ramp, which is automatically reset when the maximum output voltage is reached. The output impedance is 50 Ohms. The gain is fixed at 5 mV/keV; external means for calibration which can correct gain errors of approx. +/-5 % are recommended.

In addition, a temperature signal output is provided. It monitors the chip temperature and is used in the SDD temperature control with the supply unit. The temperature signal voltage has a gain of ~55 mV/K and an offset of +7.44 V at 0 °C.

The pin assignment of the all-in-one detector connector is stated in Appendix A2.

2.5 Signal Processing Considerations

All four SD^3 detector elements and preamplifiers are functionally independent from each other. Likewise four separate signal-processing chains are necessary to process the individual signals. The SD^3's of the XFlash® FlatQUAD detector exhibit very low series noise due to the extraordinary features of these detector elements, i.e., the cut off frequency of the noise is much higher than usual for semiconductor X-ray detectors. This allows for remarkably short filter times in the range of 0.1 µs to 1 µs.

The optimum filter time depends on the application (for there is usually a compromise between resolution and throughput). The optimum filter time is influenced by the operation temperature of the detector and the input pulse density. Short filter times are suitable for high count-rate applications or high temperature environments. Usual filter times in the range of 1 µs deliver optimum resolution as long as detector chip cooling is sufficient (below 20 °C) and low to moderate count rates are provided.

The detector works with pulsed reset feedback. The Signal Processing Unit (SVE 6) support this through a reset trigger and adjustable inhibit time as well as a gated base-line restorer (BLR). The SVE 6 comes optimally adapted to the signal conditions of the XFlash® FlatQUAD detector.

2.6 Power Consumption

The detector and preamplifier require a dual power supply of ±15 V to ±25 V (at approx. 150 mA). The voltages are internally stabilized but in case of excessive RF-noise on the supply lines there is a risk of cross-coupling to the signal stages.

The Peltier cooler is supplied preferably via a current controlled source. Care must be taken not to exceed the maximum recommended current for this detector (see the label on the electronics case or the detector data sheet). The current requirements are normally in the range of 1.5 A to 2 A with a maximum voltage drop of 12 V.

Beware!

Overloading the cooler with an excessive current can lead to severe damage of the detector.

The voltages and currents mentioned above are fed via the all-in-one detector connector as stated in Appendix A2. The SVE 6 supplies all of the necessary voltages along with temperature monitors and control circuitry for the detector temperature.

3 Maintenance and Troubleshooting

3.1 Maintenance

The XFlash® FlatQUAD detector is completely maintenance-free, apart from occasional cleaning of the outside casing.

Never open the preamplifier case because severe damage can occur and the warranty will be void. Under no circumstances dismantle the detector assembly. Any measurements on live or disconnected parts of the circuitry (with the exception of specially marked test points) can damage the detector (ESD).

3.2 Cleaning the Detector Window

Beware!

Never touch the sensors. Do not allow them to become damp or exposed to any noxious agents. Prevent the detector from contamination at any time.

A contaminated detector window can usually not be cleaned. Even trying to blow-off dust or using a fine brush, common for cleaning optical instruments, can break the extremely thin window foil. This applies to both the on-chip and the additional detector foils.

3.3 Checking the Electronic Parts

If there is no or a faulty output signal first make sure that the detector is subjected to radiation. Check cables, connections, and the power supply lines. Also check the operation of the cooler and the heat sink via the monitored chip temperature.

The output signal can be checked using a conventional oscilloscope (connected to the monitor output of the amplifier of the signal processing unit or the optional signal output on the XFlash® FlatQUAD detector). Normally, a ramp voltage superimposed by wide-band noise of approx. 5 mVpp to 10 mVpp along with some DC offset is observed. The positive going voltage steps in presence of X-radiation (approx. 30 mV step height at 5.9 keV), can be measured using an appropriate trigger mode.

3.4 Electromagnetic Interference and Noise Pick-up

Because of the signal voltages being extremely low, there is always a risk of noise pick-up from electromagnetic fields (CRT-monitors, electric motors, RF-broadcast, etc.) or ground loops. The differential signal transmission used by all parts minimizes this effect but cannot guarantee total noise prevention if the environmental conditions are too noisy.

Noise pick-up can lead to poor energy resolution and count rate loss. In extreme cases the signal processing unit can be completely blocked (100 % dead time) and no signals can be processed.

Try one or all of the following measures to eliminate the issue:

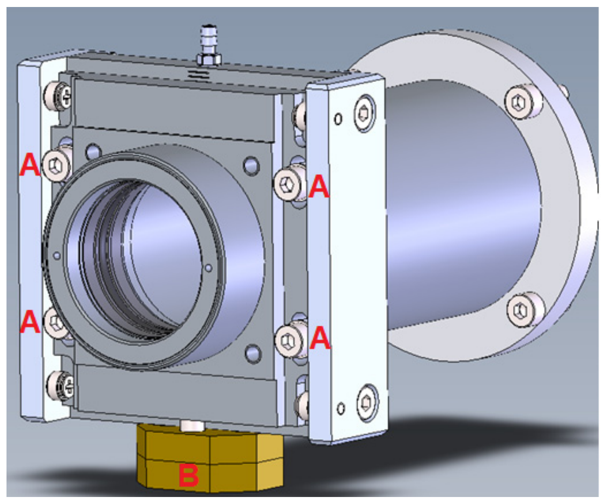
- use a short detector connection cable (1.5 m)
- apply additional shielding
- use additional ground cables to provide low ohm ground connection between any parts including the processing unit and computer
- use the same AC-power wall outlet for all components of the measurement system
- use an AC-power line filter or an isolating transformer
- place interfering devices (e.g., CRTs) as far as possible from the detector and the signal processing unit.

4 Installation

4.1 Mechanical Installation

The detector is shipped pre-mounted to a motorized x-drive unit (in and out) and manual y- and z-drive units. All linear stages are part of the adaptor of the regarding SEM. The x- and y-drive units allow moving of the detector both while the microscope chamber is vented and while the detector is under vacuum. Only the z-adjustment is not possible with SEM under vacuum.

Coarse optimization of the pole piece to detector distance (z-position) can be done through the z-drive unit under optical control at the microscope flange adaptor. This adaptor is microscope specific, the picture is an example only.



To do the alignment of the z-position, loosen the four screws (A) by a ½ turn and use the 3 mm Allen Key to adjust the height through screw (B). Tighten the four screws (A) again while checking the distance between pole piece and detector.

2 mm and 3 mm Allen Keys are required for positioning the horizontal (x) and lateral (y) adjustment screws (refer to the drawing in Appendix A2). Coarse adjustment should be made under direct visual control, preferably with an open chamber door.

Fine adjustment should be done with SEM image focussed onto the detector back side with smallest possible magnification. Align the aperture hole to the center of the SEM image with the screws for x- and y-movement.

After alignment the acquisition position must be initialized in the ESPRIT software (System, Data, Initialize acquisition position).

Exercise extreme caution with the detector tip. It may never be touched by hand or any mechanical component.

For fine adjustments under vacuum the use of a chamber scope is strongly recommended. In any case, take extreme care when moving the specimen stage toward the detector.

During shipment the detector is protected by a plastic cap or a metal cap with pump connection. Whenever possible, use the cap to cover the detector window when not in use (especially during storage or transportation). A replacement cap can be provided by Bruker Nano GmbH if the original cap is lost.

4.2 Connecting the SVE 6

The signal processing unit SVE 6 includes the complete detector supply and all the operating circuitry required. To operate the XFlash® FlatQUAD detector, simply connect it to the SVE 6 using the cables provided with the detector.

5 Operating Considerations

5.1 General Safety Advice

Caution!

Before venting the SEM the detector has to be warmed up (T >15 °C). Make sure to switch off the cooling ~ 15 min before venting.

Caution!

Make sure that the appropriate window is inserted before the HV is set.
Internal window: for HV ≤ 6 kV
2 µm window: for HV ≤ 12 kV
6 µm window: for HV ≤ 20 kV.

Caution!

When the detector is not in use always have the 20 kV window inserted. The detector must be fully retracted to avoid collision. Switch off the cooling.

5.2 Cooling Startup

Beware!

Make sure the specimen chamber is under vacuum before starting detector cooling, e.g., before switching on the SVE 6.

The XFlash® FlatQUAD detector is operational as soon as the cooling temperature is reached (after 10 – 15 min). However, the first few hours following each restart it may display a minor drift of signal gain and resolution.

Prior to high precision measurements or a final calibration, the detector should be allowed to settle for a couple of hours.

Switch off the cooling circuit and allow approx. 15 minutes for warming up of the detector to at least +15 °C before venting the specimen chamber or opening the chamber door.

5.3 Cooler Operating Modes

The cooler can be operated in constant current mode or in thermostat mode using the incorporated temperature sensor for feedback.

The lowest detector temperature (consequently the best energy resolution) can be achieved in constant current mode (for maximum recommended current see the label on the electronics case or detector data sheet).

For optimum stability of the detector parameters the temperature of the detector element are factory set to a fixed value using the thermostat mode. Because of the necessary margin for thermostat action not quite the full cooling power is available in this mode.

At a set detector temperature of -20 °C to -25 °C, temperature stability is maintained over the full environmental temperature range, provided the detector is held under vacuum (<30 Pa residual pressure).

The user can switch between modes by setting either “max cooling” or “thermostat mode” in the software.

5.4 Set Detector Operating Temperature

The nominal temperature in thermostat mode and the current temperature are factory settings.

The detector element can be used within a wide temperature range beginning at room temperature down to -30 °C. The detector temperature and the characteristics of the signal processing unit determine the achievable energy resolution.

The lower the operating temperature of the detector element the more electric power is required for the thermoelectric cooler. The excess power has to be dissipated via the detector casing (heat sink) without significantly heating it. Good convection and low environmental temperature are therefore mandatory to achieve very low detector temperatures (e.g. -30 °C). Operating temperatures around -20 °C are a good compromise.

5.5 Optimizing Cooling Performance

The maximum recommended cooling current may vary. The value of the maximum recommended current stated on the label on the preamplifier case or in the detector data sheet is also the value that ensures optimum cooling performance at normal conditions. However, under certain operating conditions (poorer or much better dissipation than normal) the optimum current may vary.

> **Beware!**
>
> Excessive cooler currents can lead to severe damage of the detector. The maximum recommended cooler current should never be exceeded by more than 20 %, not even temporarily.

Achieving optimum results from the cooling system requires a deeper understanding in how the thermoelectric cooler works.

By means of a current flowing through a cooling element (a stack of p- and n-semiconductors), a thermoelectric cooler transports an amount of heat (according to the applied current) from the so-called cold side to the warm side of the cooling element. This heat, along with a substantial amount of heat from the electric power dissipation in the cooling element, has to be removed from the warm side. Above a certain current level (the optimum current for the system), warming up dominates the cooling effect. Further increase of the cooling current leads to the opposite effect (i.e., it leads to higher temperatures than optimum). The optimum current depends substantially on how much heat can be dissipated from the warm side without significant increase in temperature.

Finding the optimum current, i.e., the lowest possible detector temperature is made difficult due to the fact that near the optimum it is unknown at first whether an increase or a decrease of the current yields better performance. In addition to that, after every change of the cooling current it is necessary to wait for at least half an hour until thermal equilibrium is reached.

Note

Because of the heat capacity of the assembly, increasing the current usually decreases temperature initially. This effect however reverses within a few minutes or more if the current is set too high, due to the delayed warming-up of all components.

The cooling current should never be changed by more than 10 % to 20 % at a time when trying to find the optimum current. Observe the detector temperature closely to avoid damage due to overheating.

5.6 Measures in Case of a Broken Detector Window

In case a detector window is broken, immediately switch off the complete detector supply (the signal processing unit, respectively). Never try to operate a detector with a broken window. Defective detector components can only be replaced at authorized Bruker Nano GmbH service locations.

Window fragments can get into the specimen chamber or spread over the work area. Especially for a Beryllium window it is essential to gather all fragments carefully, e.g., by means of adhesive tape.

Warning!

Beryllium is very toxic when swallowed or inhaled. In case of a breakage of a beryllium window, ensure that all fragments are carefully gathered and properly disposed of according to the relevant rules for toxic waste.

If not otherwise possible the remains of the window can be disposed of by sending them (securely packaged and labeled) to Bruker Nano GmbH along with the damaged detector.

5.7 Disposal

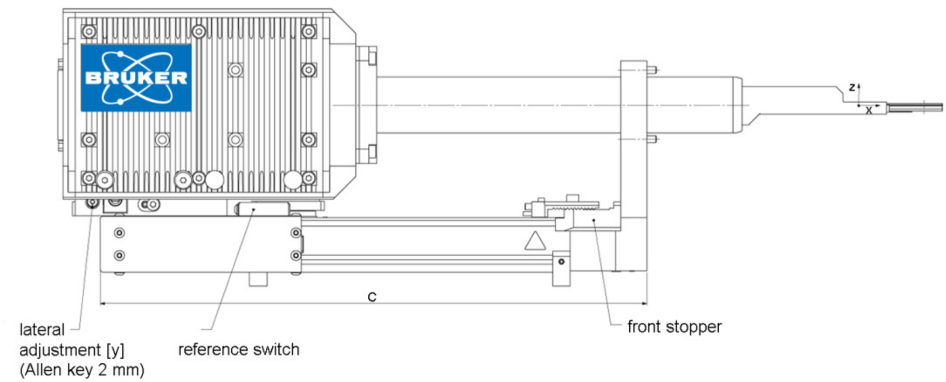
Detectors with Beryllium windows must not be handled by unauthorized personnel and must be marked as hazardous waste and disposed of accordingly. Worn out Detectors that have reached their end of life can be returned to Bruker Nano GmbH or your local service.

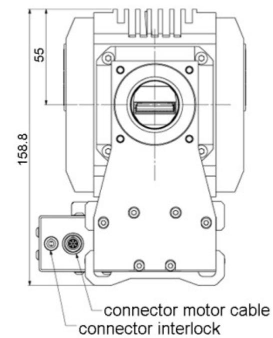
Appendix

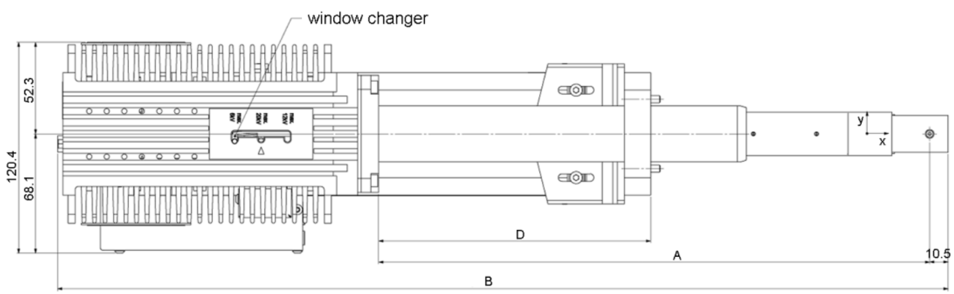
A1 Characteristics

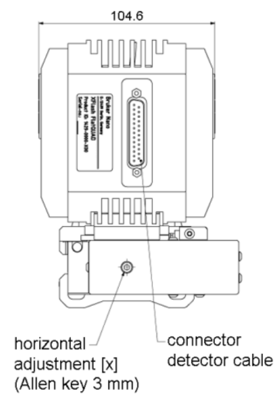
Detector	Type	Silicon Drift Detector (SDD)
	Active Area	4 x 15 mm^2
	Silicon Thickness	0.45 mm
Window	Window Material	12 µm Beryllium with polymer coating or 1 µm internal polymer window plus two additional windows (2 µm or 6 µm)
	Collimator Material	Zirconium
	Radiation Entry	perpendicular
	External Pressure Range	0 bar … +1.2 bar (-15 psi … +3 psi)
	Internal Pressure Range	<0.3 mbar when in operation
	Maximum Slew Rate	±0.3 bar/s (5 psi/s)
Power Supply	Supply Voltage	±15 V … ±24 V, 50 mA
	Auxiliary Power (Fan)	12 V (±5 %), max. 100 mA
Preamplifier	Output Signal	positive, 5 mV/keV, ramp type
	Rise Time	70 ns ± 20 ns
	Ramp Size	< ±10 V
	Output Impedance	50 Ω
	Recommended Shaping Time	0.1 µs … 1 µs, depending on application
	Max. Input Count Rate (ICR)	4 x 1,500,000 cps (limited by signal processor)
Cooler	Max. Cooling Current	2.0 A
	Cooling Modes	Constant current; thermostat
Environmental	Temperature (Operation)	0 … 35 °C
	Temperature (Storage)	-10 °C … +55 °C
	Max. Relative Humidity	90 % RH

A2 Mechanical drawing









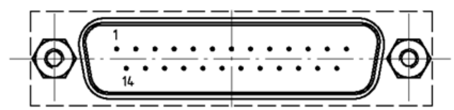
Detector	Length A	Length B
FlatQUAD 300	314	507,5
FlatQUAD 350	364	557,5

Support	Length C	Length D
FlatQUAD Support 300	313,6	155
FlatQUAD Support 350	363,6	205

A3 Pin Assignments

Detector connection

Pin	Signal
2, 15	GND
1, 14	+19 V
3, 16	-19 V
6, 7, 18	Peltier +
8, 19, 20	Peltier – (GND)
17	Temperature signal
5	Temp. GND
4	HV ON
13 / 25	Signal 1 + / -
12 / 24	Signal 2 + / -
11 / 23	Signal 3 + / -
10 / 22	Signal 4 + / -
9, 21	SGND



Connector:
SUB-D, 25 pole, male