Supporting Information for Publication

STEM EDX Nitrogen Mapping of Nanoinclusions in Milky Diamonds from Juina, Brazil, Using a Windowless Silicon Drift Detector System

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Abstract

Supporting information includes more details on the ChemiSTEM™ Technology. Detailed information are given on the windowless design of silicon drift detectors (SDD), their placement within the transmission electron microscopy (TEM) column and differences between conventional Si(Li) detectors.

Table of Content

Chapter S-1 ChemiSTEM™ Technology

ChemiSTEM Technology consists of the FEI proprietary X-FEG high brightness Schottky field emission source and the FEI-designed Super-X™ system (Figure S-1). The compact design of the new developed SDDs allows integration up to four SDDs within the STEM column (Figure S-2). ChemiSTEM Technology includes fast mapping electronics capable of 100,000 spectra/second in EDX spectrum imaging. The integration of multiple SDDs results in a solid angle of 0.9 steradian (sr).

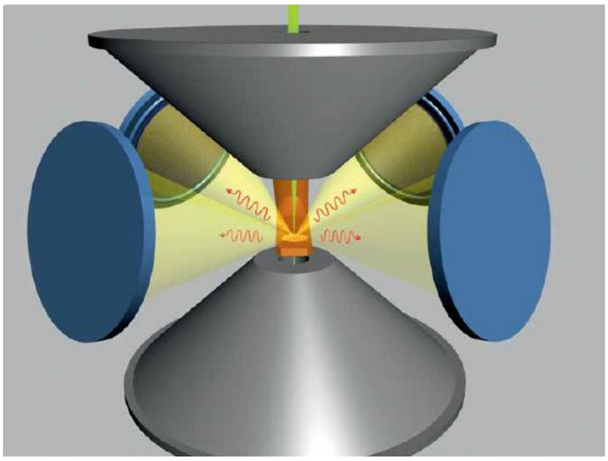

Figure S-1. Schematic illustration of ChemiSTEM™ design (Image courtesy of P. Schlossmacher, FEI Company). Four SDDs are symmetrically arranged around the optical TEM column.

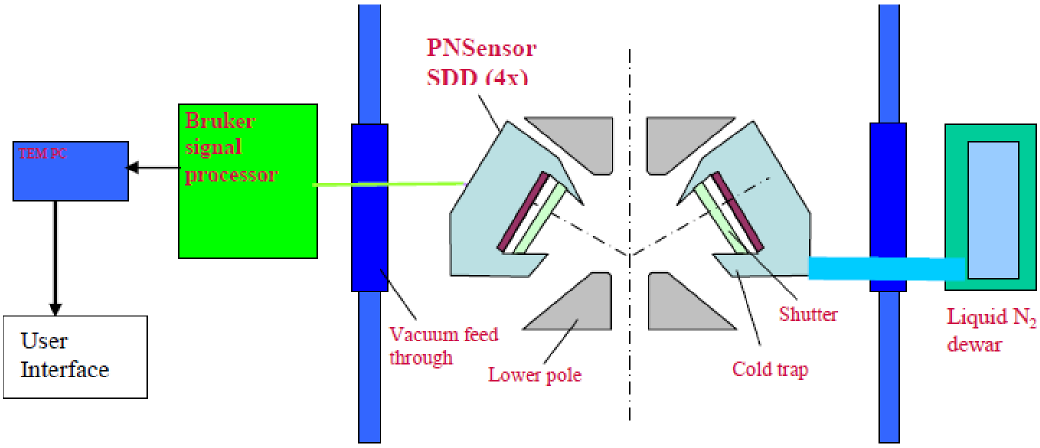

Figure S-2. Cross section through the TEM column near the objective lens[2] (Image courtesy of P. Schlossmacher, FEI Company). Two PNSensor designed SDDs are mounted around the specimen.

Chapter S-2 Comparison of the efficiency of a Super-X detector system with a conventional Si(Li) detector

S-2.1 Tilt response

Specimen tilting was a big disadvantage concerning single Si(Li) detector. Comparison of measured X-ray count rates with Super-X system (0.9 sr solid angle) over a tilt range from -25° to +25° and X-ray count rates detected by a conventional single Si(Li) detector (0.3 sr solid angle) clearly demonstrate the capabilities of the Super-X detector (Figure S-3). Super-X count rate always exceeds that of the single Si(Li) detector. Maximum count rate of the Super-X detector is achieved at zero degree tilt angle and never decreases more than 20 % over the entire tilt range. Maximum count rate of the Si(Li) detector can be observed at 20° tilt angle. Shadowing occurs at lower degrees and negatively affects the count rate. However, this is a general problem with single detectors, whether SDD or Si(Li) detector.

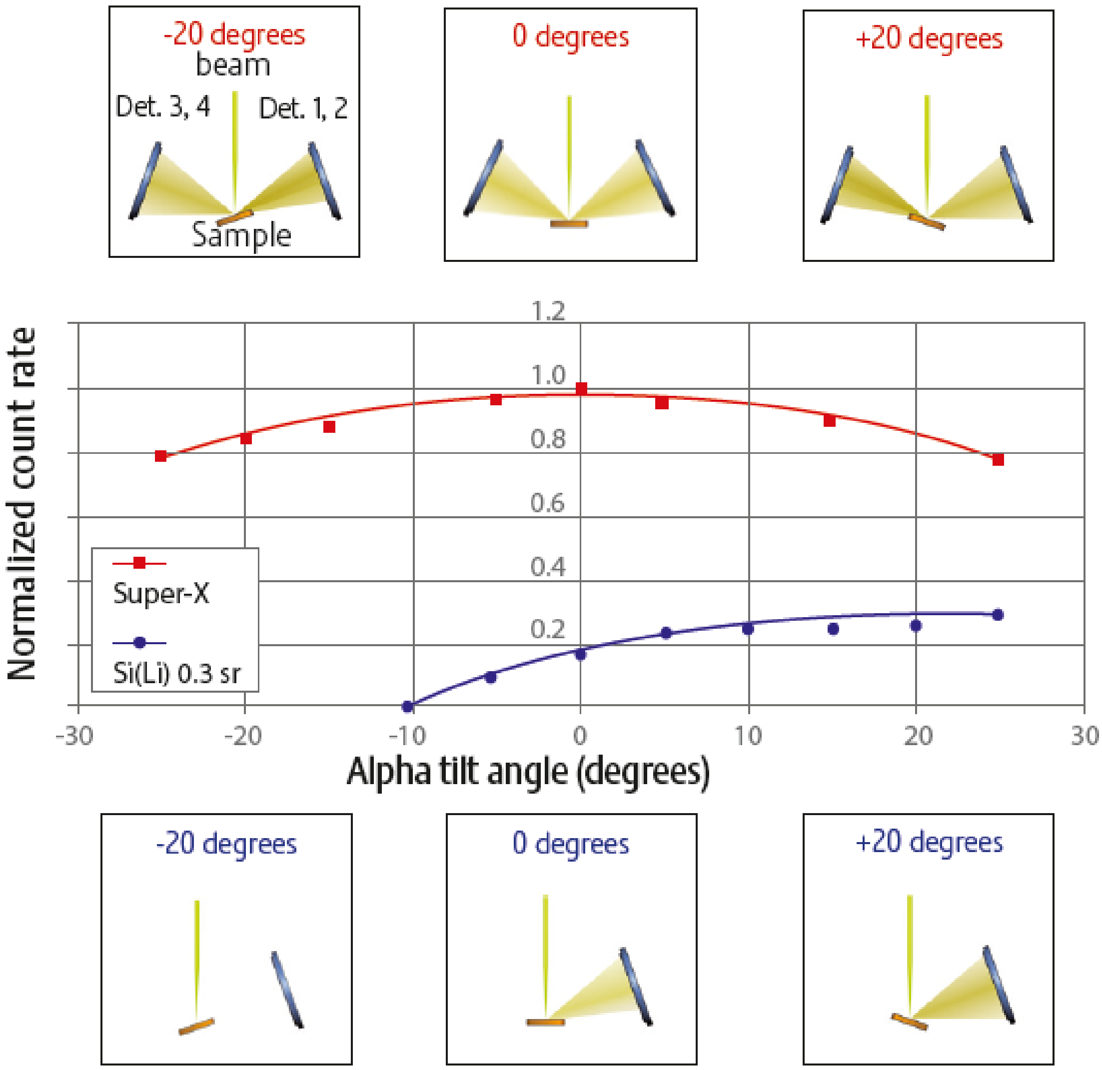

Figure S-3. Relative count rates of the Super-X system (red line) compared to that of a single Si(Li) detector (blue line) at different tilt angles[1] (Image courtesy of P. Schlossmacher1, FEI Company). Shadowing effects of the Super-X system and Si(Li) detector is presented above and below the count rate diagram respectively.

S-2.2 The effect of beam current on the input count rate

Beam current positively correlates with the input count rate[1]. This effect is particularly important for the Super-X detector system (Figure S-4). The use of the Super-X system with a X-field emission gun (X-FEG) allows beam currents more than 10 nA resulting in a count rate of more than 400 000 counts per second without lowering the energy resolution[1]. This is in contrast to the capacity of a single Si(Li) detector in combination with a Schottky-FEG.

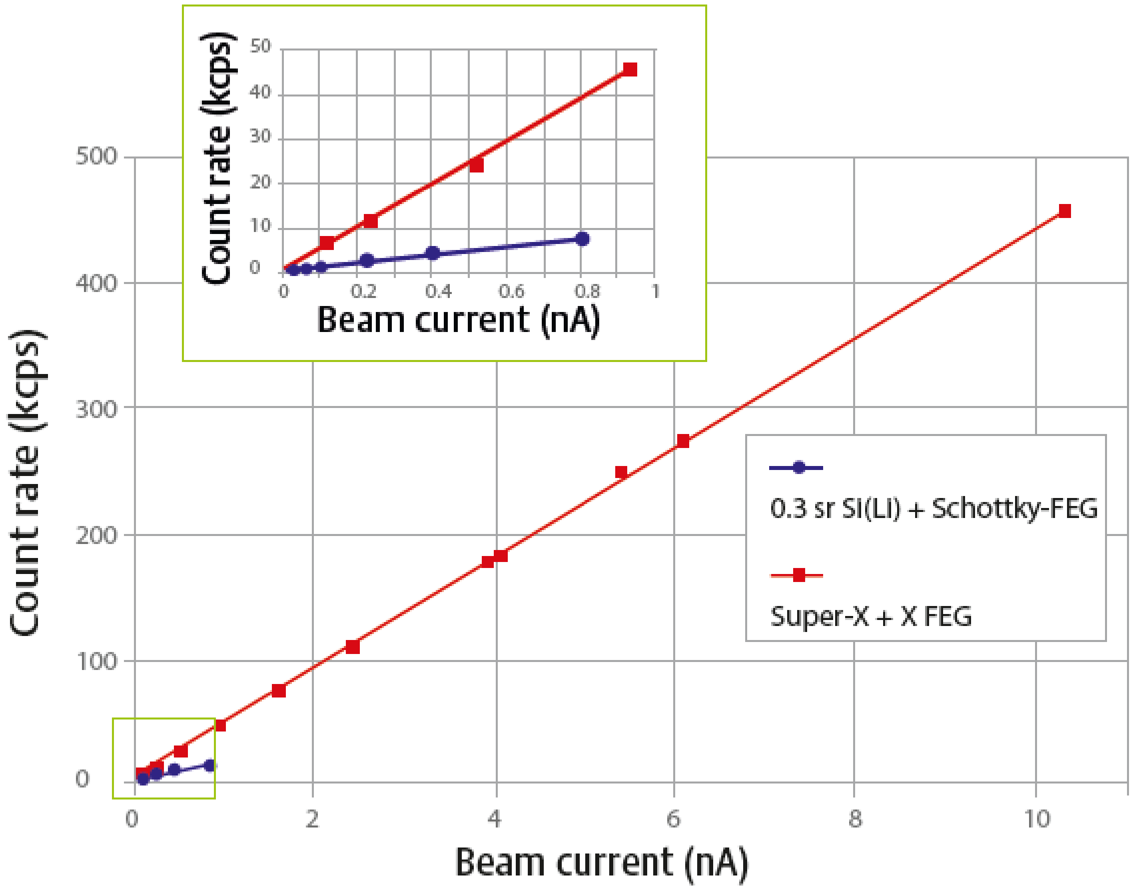

Figure S-4. Input count rate versus beam current for the Super-X detector system (red line) and a conventional Si(Li) detector (Image courtesy of P. Schlossmacher[1], FEI Company).

S-2.3 Detection of light elements

The windowless design of the Super-X detector system results in enhanced detection sensitivity for light elements such as nitrogen[1]. Figure S-5 demonstrate the advantage of the new system compared to a conventional single Si(Li) detector, especially in the low energy range (< 500 eV).

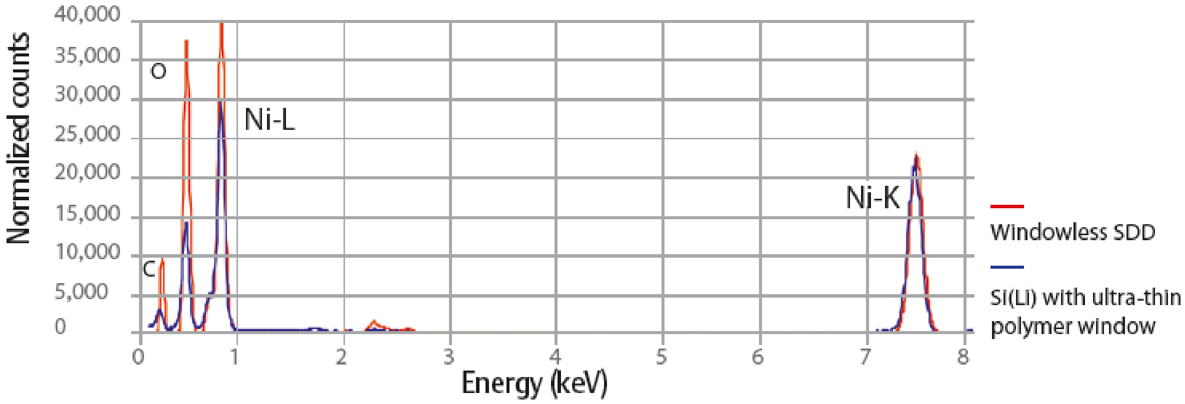

Figure S-5. Count rates detected by a windowless SDD compared to that of Si(Li) detector with ultra-thin polymer window (Image courtesy of P. Schlossmacher, FEI Company).

S-2.4 Energy resolution

Detector XFlash 5030 with an active area of 30 mm^2 achieves an energy resolution of ≤ 127 eV (Mn Kα) due to a special chip design with integrated charge amplifier. In contrast, energy resolution of a conventional Si(Li) detector decreases with increasing input count rate (Figure S-6).

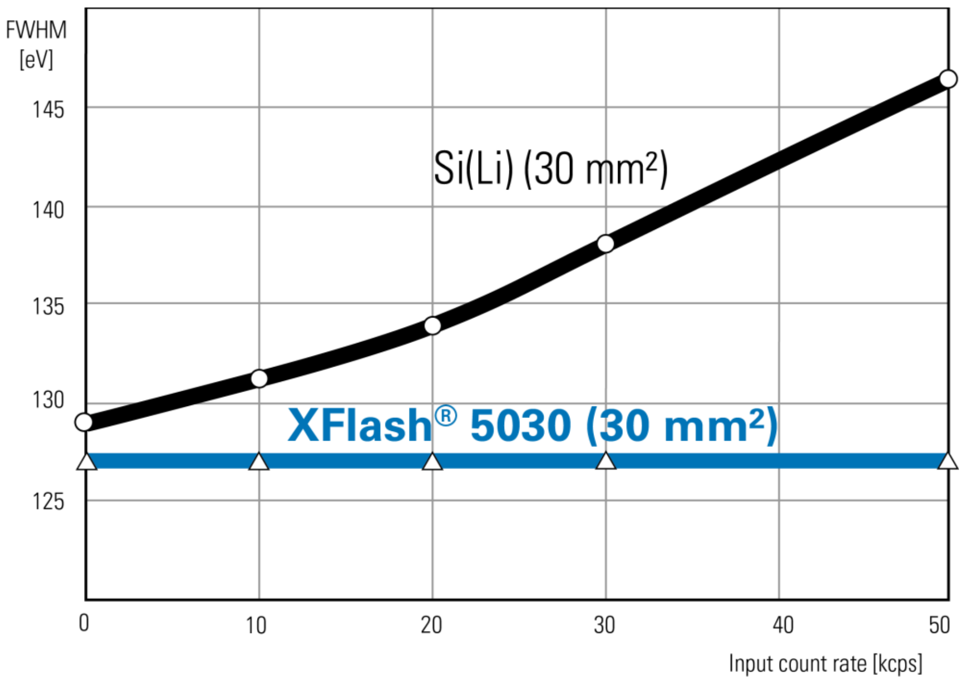

Figure S-6. Energy resolution at Mn Kα versus input count rate of SDD XFlash 5030 (blue line) and Si(Li) (black line) (Image courtesy of Bruker).

Chapter S-3 Results of Monte Carlo simulation of electron trajectory in solids ("CASINO")

We simulated electron trajectories in a diamond using Version 2 of Monte Carlo simulation of electron trajectory in solids ("CASINO") reprogrammed by Alexandre Réal Couture in 2000 under the supervision of Professor Dominique Drouin. The used spot size was 5 nm. The calculation was based on Rutherford formula. The result is shown in Figure S-7. The X-ray generation volume has a dimension of about 100 nm by 100 nm.

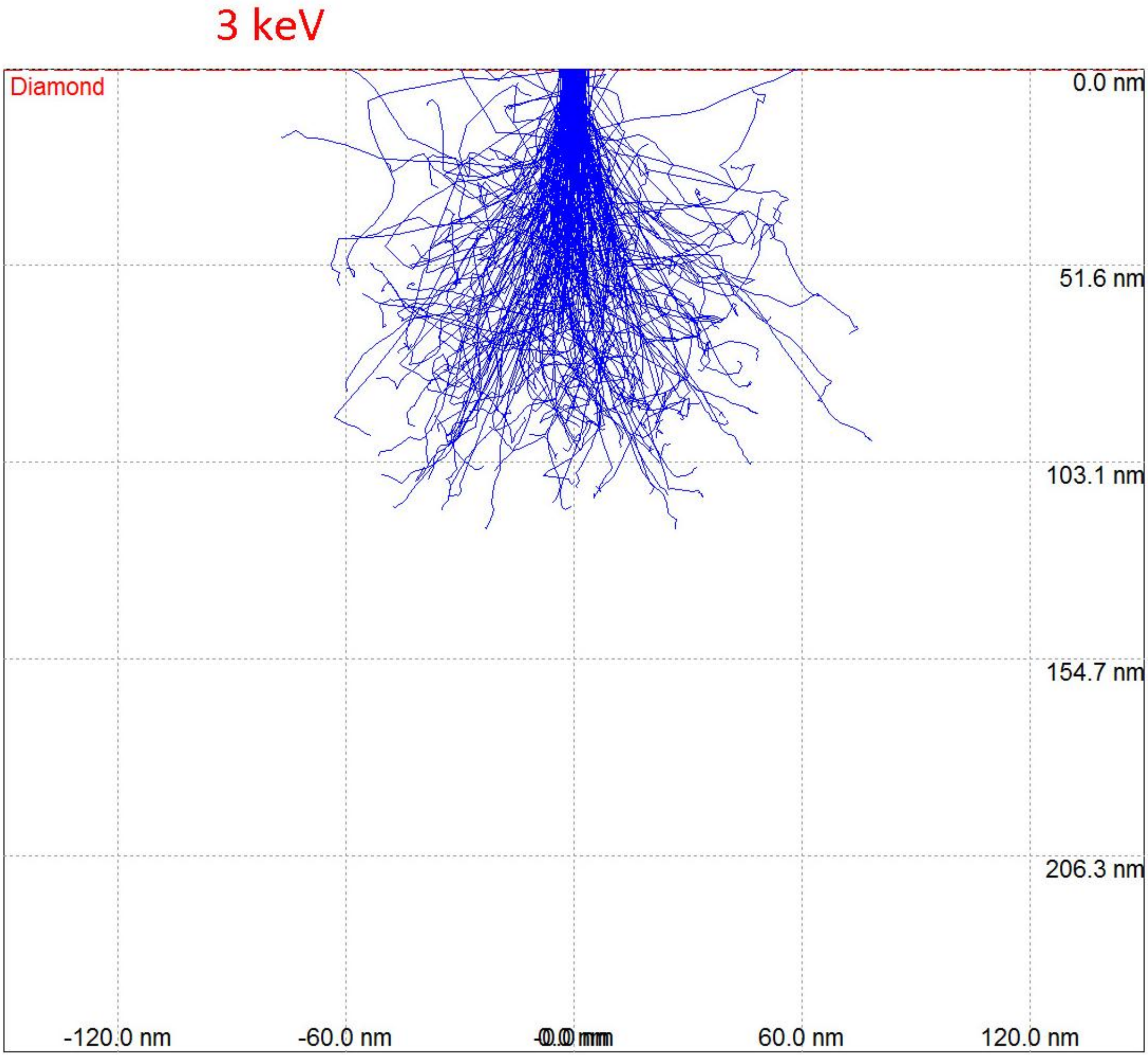

Figure S-7. Volume of generated X-rays calculated using Monte Carlo simulation for diamond at 3 keV.

References

(1) Schlossmacher, P.; Klenov, D.O.; Freitag, B.; von Harrach, H.S. *Microscopy Today* **2010 a**, 18, 14–20.

(2) Schlossmacher, P.; Klenov, D.O., Freitag, B.; von Harrach, S.; Steinbach, A. *Microscopy and Analysis* **2010 b**, 24, 5–8.