Synchrotron X-ray Beam Tests
of Poly- and Single-Crystal Diamond at ESRF

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Talk Outline

1. X-ray Synchrotron beam monitoring requirement and diamond device approach

1. why diamond?

2. ESRF test results on SC (and PC)

*Development Issues*

-- surfaces, contacts
-- threading dislocations
-- RF electronic readout
X-ray Synchrotrons, beam monitoring goals

Need for \textit{continuous measurement} \((\rightarrow \text{permanent in-beam element})\)

Position / Vector

- Required beam stability \(\sim 10\%\) of beam size typically \(1 \sim 200\mu m\), \textit{nanofocusing goals} (already \(\sim 100\)nm routine at ID22-ESRF)
- \textit{Measurement bandwidth} \(\geq 1kHz\) (e.g. acoustic vibrations)

Intensity:

- (relative) accuracy & linearity requirement \(\leq 0.1\%\)

Timing:

- e.g. synchronization of laser with X-rays in pump-probe experiments
- electron-photon bunches \(50 \sim 100\)psec (MHz to 352MHz rates)

\textit{device}...

- minimal beam interference (absorption, scattering, coherence loss)
- reliability with zero / low maintenance
- compatibility with beamline design (size, vacuum...)
- cost??

\(\rightarrow\) max. \textit{absorbed} power: \(\leq mW\) \textit{monochromatic beam}

\(\rightarrow\) \(>10W\) \textit{in ‘white’ beam applications}!!
Diamond X-Ray BPMs: Principle

- thin plate diamond sample with ‘X-ray transparent’ metal electrode contacts e.g. <100nm Cr, Ti, … Ni, Al
- diamond bulk acts as solid state ‘ionization chamber’ in X-ray microbeam
  electron thermalization range ~ microns
- current signal readout ‘DC’ or RF (synchrotron clock frequency / n bunches)

Beam Monitoring: position and intensity

multiple electrode designs, e.g. simple quadrant motif, diffusion splitting of charge

→ weighting of electrode currents A, B, C, D gives beam ‘centre of gravity’
→ sum of currents gives beam intensity

Packaged device tests ID09B
why single crystal diamond?

Z = 6 → low specific X-ray absorption / beam scattering
short ‘hot electron’ range at high energies

high electron AND hole saturation velocities (150µm/ns), low dielectric constant (5.5)
fast pulse response (<1nsec in 50µm thick device)
→ synchrotron beam ‘pulse by pulse’ analysis possible

wide bandgap (5.5eV), stable and insulating O-terminated surface,
excellent thermal/mechanical properties
→ ‘low’ leakage currents at temperatures up to ~400°C
high heat load ‘white’ beam monitoring possibility (??)…

Polycrystalline material
grain-boundary artifacts
→ bulk scattering, ‘powder’ X-ray diffraction
→ limited charge transport (incomplete charge collection)

Single Crystal gives:
X-optics surface quality (<1nm) possible
beam coherence preservation
Uniform electrical response:
charge-carrier lifetime (E6 ELSC material) ~0.1 - 1µs
→ 100% charge signal collection over ~mm distances

~1mm
Polycrystal sample, image from XBIC mapping in X-ray beam

~0.5mm
Tromson et al, CEA-LIST
S-C E6 material I-V curves *under steady-state X-ray beam illumination (6 ~7keV)*

Lift-off litho’ evaporated contacts
Glasgow University

Shadow mask, sputtered contacts
GSI Darmstadt

\[ \begin{array}{c}
\text{bias} \\
333\mu m C^*
\end{array} \]

\[ \begin{array}{c}
130nm \text{ Au} \\
30nm \text{ Pd} \\
10nm \text{ Ni} \\
10nm \text{ Ti (annealed)} \\
30nm \text{ Pd} \\
130nm \text{ Au}
\end{array} \]

\[ \begin{array}{c}
\text{current (nA)} \\
0.5V/\mu m
\end{array} \]

\[ \begin{array}{c}
\text{bias (V)} \\
-150, -100, -50, 50, 100
\end{array} \]

\[ \begin{array}{c}
\text{current (nA)} \\
0.5V/\mu m
\end{array} \]

\[ \begin{array}{c}
\text{bias (V)} \\
-300, -200, -100, 100, 200, 300
\end{array} \]

Si beam flux calibration (vacuum)

\[ \varepsilon_{\text{Diamond}} = 13.05 \pm 0.2 \text{ eV/e-h pair} \]

(ESRF, MI-885)
Resin wheel polish ‘tadpoles’

- 10μm SEM

- XBIC map, 6 keV X-rays
- Ni-TiC contacts

- hot spot around Al wedge bond

- Guard ring
electrode

- 75μm
- 50x reflect microscope

left
right
Area response uniformity ‘good’ contact

50μm thick sample, bias 50V, 50nm sputtered Al-Al contacts (GSI Target Lab.).
1μm-step raster scan with beam probe 0.3 x 1.1μm² at 6keV (ID21 beamline)

Row 40 signal variation **0.103%** (1σ)

Signal variation during mesh scan
- linescan at column 50, rows 0-100

~ 20 minutes
Ni/Pt/Au and Ti(annealed)/Pt/Au contacts, fabricated at Stanford NanoFab’ Facility (Chris’ Kenney)

50, 20, 10 and 5μm quadrant isolation gaps

E6 MDS2 & MDS3 samples, Achard-CNRS ATJ30 sample
Failed processing of Ni-TiC contacts

E6 Sample MDS-2, I-V under steady state X-ray beam

bad Ni-TiC contacts
Sample thickness ~350µm

electrometer saturated

Sample thickness ~350µm

A lot of hysteresis

-26nA
SEM-EBIC measurements:

E6 SC material, Stanford processed Ni-TiC electrodes

Secondary electron emission contrast

!! Surface probe 1 ~ 2 µm
Signal ‘linearity’ with beam intensity

E6-70310 thickness 100μm single crystal
20nm+20nm Cr, Au contacts - GSI
‘dc’ quadrant currents measured with electrometers
bias 50V

Incident beam flux (X-ray/sec in 0.4 x 1.2μm² fwhm spot)
20% diamond absorption, 4 bunch synchrotron fill pattern with 2.8μs orbit
→ <<1 … ~15 X-ray photons / bunch
Position Response and beam size

For large beam (> 50µm), quadrant ‘crossover response’ is \( \sim \) line integral over the beam intensity profile.

For small beam (~µm), crossover response is convolution of photoelectron thermalization range (~1µm) and lateral charge diffusion (5~50µm) which occurs during drift of \( e^- \), \( h^+ \) charge carriers.
Position sensitivity and useful range

For small beam, response slope / sensitive range may be modulated over range 10~100µm by
- applied electric field (bias)
- inter-electrode gap / sample thickness

*limit for weak fields is charge recombination or drift trapping signal loss…*

100µm thick sample, 140µm quadrant spacing exaggerates weak field effect

nb. low frequency Electrometer current measurements
ID21 Vertical / horizontal position timescan

Al-Al contacted quadrant device, ESRF ID21 beamline (MI-885)

timescan V->F data, 1sec integrations

σ <15nm

σ <20nm
ESRF synchrotron in 4 bunch mode (ID21)

ID21: focused X-ray beam ~0.5 x 0.1 µm² and ~10² ph/pulse at 6keV

ID09B: ~50 x 100µm² chopped white beam, ~10⁵-⁹ ph/pulse at ~18keV

5% beam absorbed in diamond → ~1pC ... 10nC /pulse (1GeV ... 10TeV)
DSO signal after ~30db preamp gain

device layout not optimized for RF!

+150V/118µm Al-Al sputtered electrodes, *single crystal*
Timing of ESRF Synchrotron Pulses

E6 Sample MDS-2, ‘bad’ Stanford Ni-TiC contacts, DBAIII 2.3GHz preamp

*single crystal*

![Graph showing pulse separation](image)

**ESRF RF group data:**
- rev. freq. 355044Hz / 16 bunches
- \( \rightarrow \) interbunch period = 176.035ns

**measured:**
- mean = 176.036ns
- \( \sigma = 16 \text{ps} \)

**Lecroy DSO data post-analyzed with leading edge ‘discrimination’ normalized Individual to signal pulse integrals**
- \( \sigma \rightarrow 20 \text{ps for simple leading edge discrimination} \).
Charge drift and signal development
XBIC map, 5µm steps, 50ps time bins
+150V/118um Al-Al sputtered contacts, *single crystal*

0.5ns wide ‘gated integrations’,
time-slew of signal as function of beam position
(response of bottom right quadrant only is shown here !)
position response at ID09B (chopped beam)

\[ \sim 5 \times 10^7 \text{ ph/pulse (1kHz) at } \sim 18\text{keV, 5\% absorption in } \sim 350\mu\text{m thick diamond} \]

Single crystal sample, TiW contacts

Ad hoc test of single crystal OSU quadrant device

Very poor circuit layout & cabling of diamond support \( \rightarrow \) signal ‘bounce’
Position response
(motor scan of diamond)

timescan with beam initially centered on diamond

Beam found at: $(y,z) = (0.002, -0.004)$, size: $0.188 \times 0.069$ mm
Polychromatic beam, choppers in, ≈18keV X-ray beam U17-13, phg 7 pvg 0.7, pd2f counting: $1.24 \times 10^{11}$/sec
In beam spot ≈100 x 200µm$^2$ FWHM.

rise time: 235ps, area 5.4nVs, 
fall time 3.2 ns determined by circuit response(?)  Bias 500V / ≈300µm, 

Linearity check to ≈6 x10$^6$ photons absorbed/pulse) 

No reduction in signal response with overnight beam on sample
Measurements with GSI stripline mounted *polycrystalline* sample

- **2mV/2ns division**: monochromatic ~ $5 \times 10^6$ ph/pulse at 18keV, ~2% absorption (350µm thick diamond)

- **2mV/1µs division**: 24*8+1bunch hybrid mode

- **2mV/1µs division**: Signal constant over duration of heatload chopper pulse.

Scattered beam ‘powder diffraction’ rings arising from the ~10µm random orientation grains of the polycrystalline diamond plate.
conclusions – future plans

‘good’, flawless device fabricated on single crystal sample using ‘simple’ metal contacts…
but avoiding ‘significant’ local defects is not trivial

demonstrated (ID21 + ID09B tests):

- flux intensity: linearity - stability – area uniformity \(\sim 0.1\%\)\(^1\)
- position response: sensitivity \(\sim 10\text{nm}\)\(^*\)
- timing resolution / leading edge risetime \(\sim 20 / 250\text{ps}\)\(^**\)

rms figures
- \(^1\) single crystal samples only!
- \(^*\) for a 1\(\mu\text{m}\) beamsize. \textit{Accuracy is limited by lithography}
- \(^**\) circuit layout limited

present objectives:

- validate ‘industrial’ source(s) of reliable contact processing with precision lithography
  …on thinned (to <50\(\mu\text{m}\)) CVD plates
- implement electronic readout: multichannel electrometers or RF signal techniques

\textit{long-term radiation stability remains unproven, but X-ray induced damage can only occur at surfaces with low energy X-rays in a ‘perfect’ diamond crystal…}

\(\sim 5\text{ GigaGray, 16hour stability test made at ID21}\)
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*Element Six (Ascot & Cuijk), Diamond Detectors Ltd., LIMHP: material samples, surface polishing*