Novel Radiation Hard
CVD Diamond Detectors for Hadron Physics

4th NoRHDia Workshop @ GSI

June 8 – June 10 2008

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

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

Need for *continuous measurement* **(→ permanent in-beam element)**

Position / Vector

Required beam stability ~10% of beam size typically 1 ~ 200µm,
nanofocusing goals (already ~100nm routine at ID22-ESRF)

Measurement bandwidth ≥1kHz (e.g. accoustic vibrations)

Intensity: (relative) accuracy & linearity requirement ≤ 0.1%

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

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

→ max. *absorbed* power: ≤ mW monochromatic beam
→ *>10W in 'white' beam applications !!*

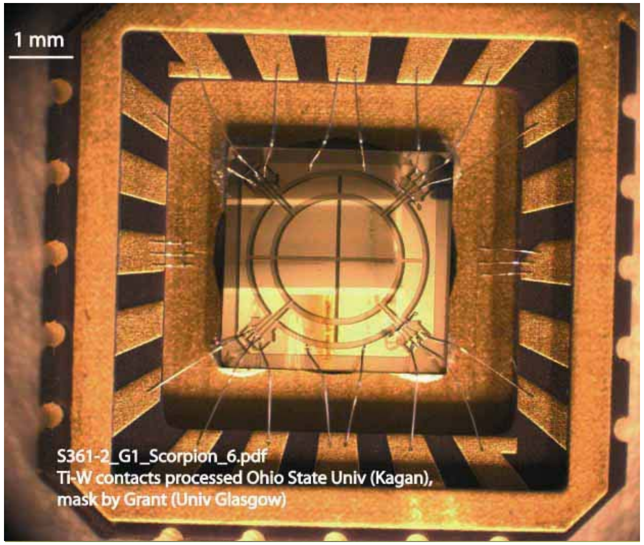
- thin plate diamond sample with ‘X-ray transparent’ metal electrode contacts e.g. <100nm Cr, Ti, … Ni, Al
- diamond bulk acts as solid state ‘ionization chamber’ in X-ray microbeam
  electron thermalization range ~ microns
- current signal readout ‘DC’ or RF (synchrotron clock frequency / n bunches)

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?

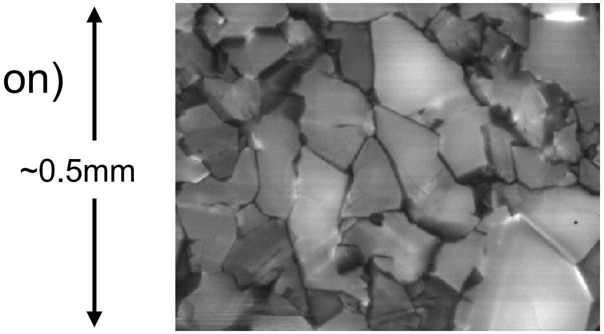
Z = 6 → low specific X-ray absorption / beam scattering
short 'hot electron' range at high energies

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)

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



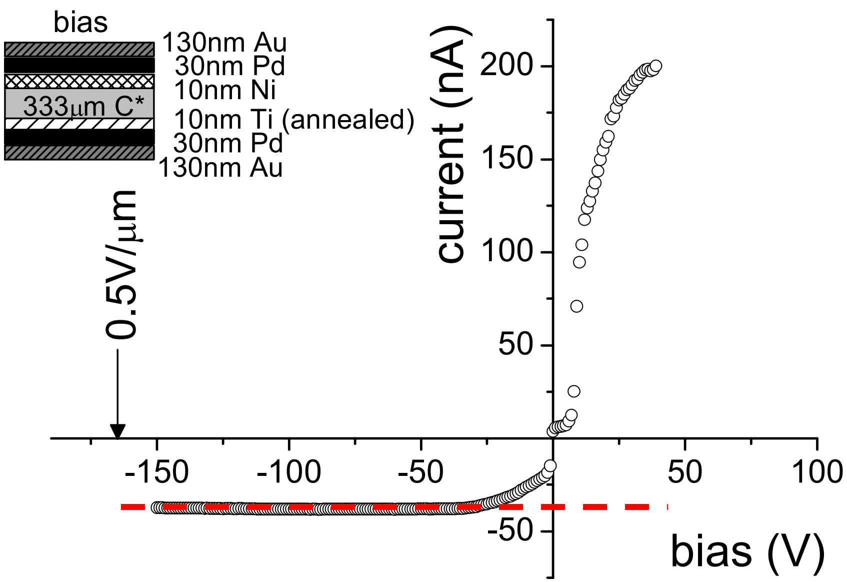
Tromson et al, CEA-LIST

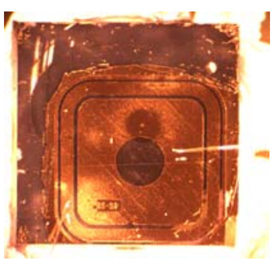
Single Crystal gives:
X-optics surface quality (<1nm) possible
(beam coherence preservation)
Uniform electrical response:
charge-carrier lifetime (E6 ELSC material) ~0.1 - 1μs
→100% charge signal collection over ~mm distances

X-ray beam I-V response, ‘good’ metal contacts

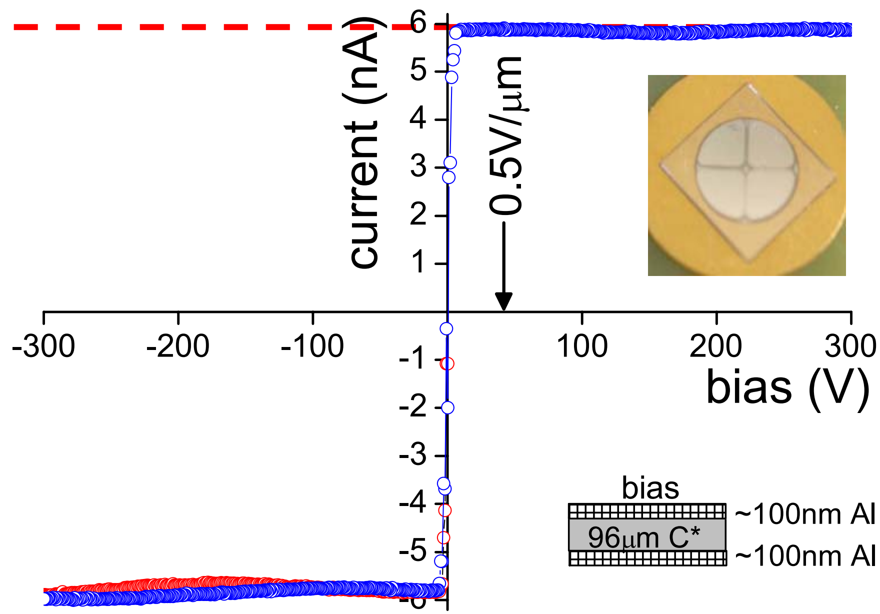
S-C E6 material I-V curves *under steady-state X-ray beam illumination (6 ~7keV)*

Lift-off litho’ evaporated contacts
Glasgow University





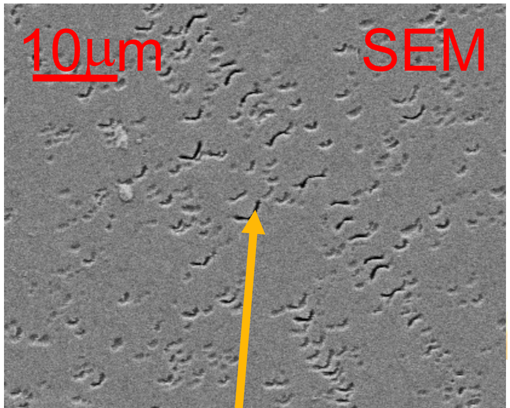
Shadow mask, sputtered contacts
GSI Darmstadt



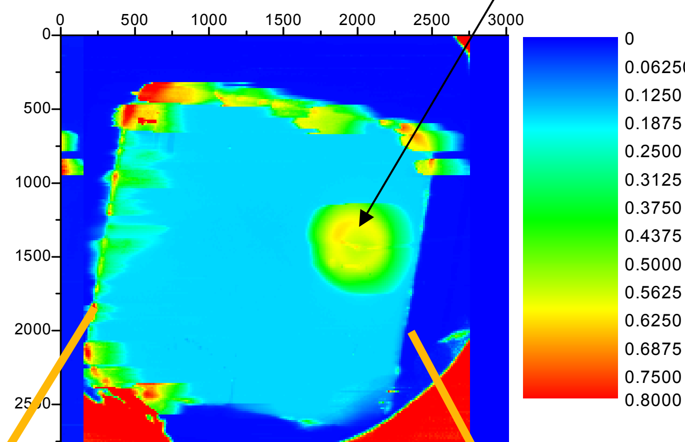
Si beam flux calibration (vacuum)
→ $\varepsilon_{Diamond}$ = 13.05 +/-0.2 eV/e-h pair
(ESRF, MI-885)

contact-edge hot defects and surface processing

Resin wheel polish 'tadpoles'



hot spot around Al wedge bond



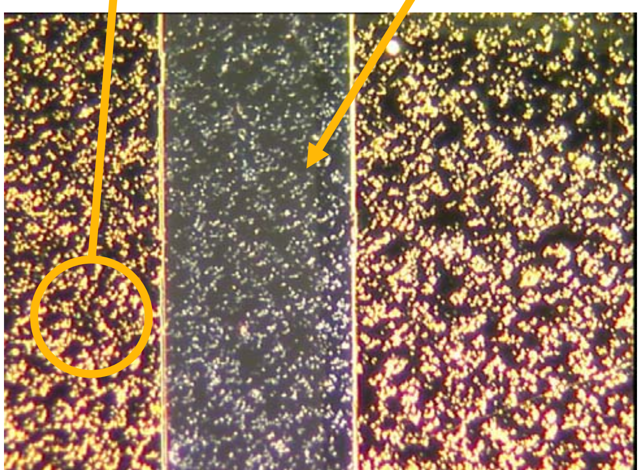
XBIC map,

6 keV X-rays

Ni-TiC contacts

left

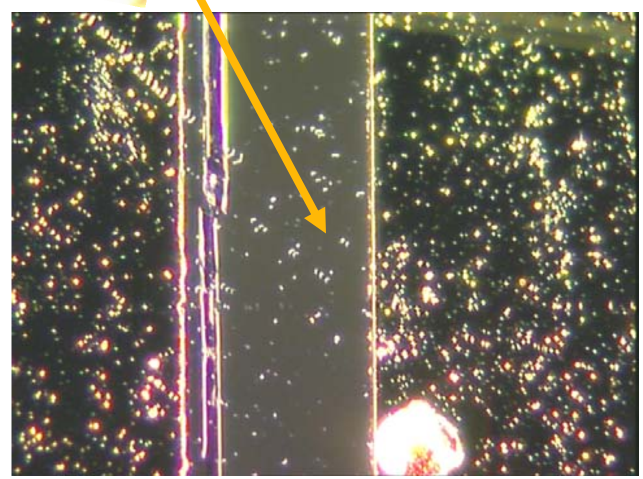
50x reflect' microscope



Guard ring

75µm

electrode

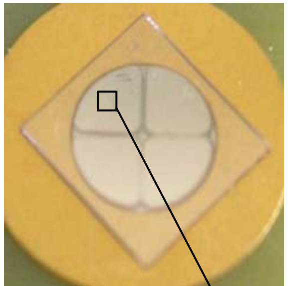


right

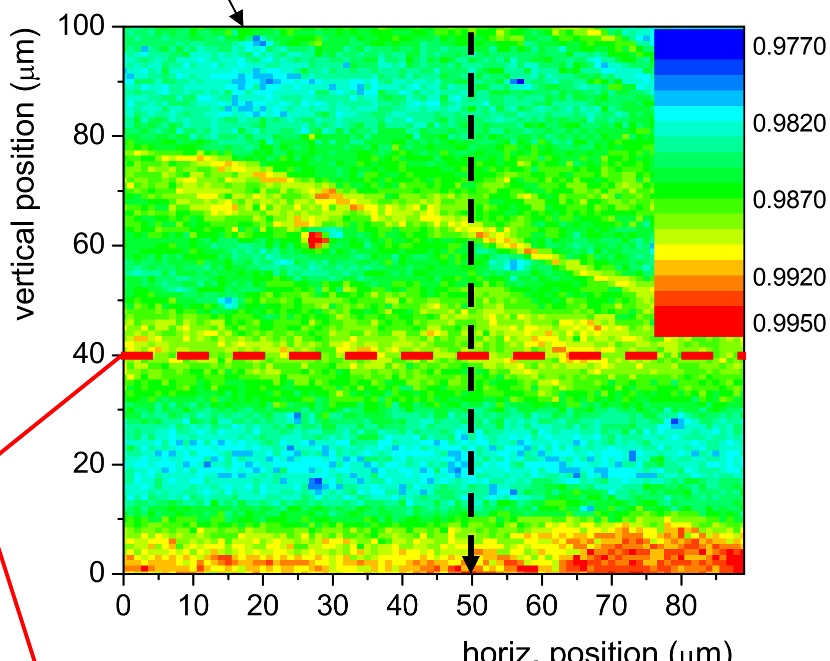
electrode

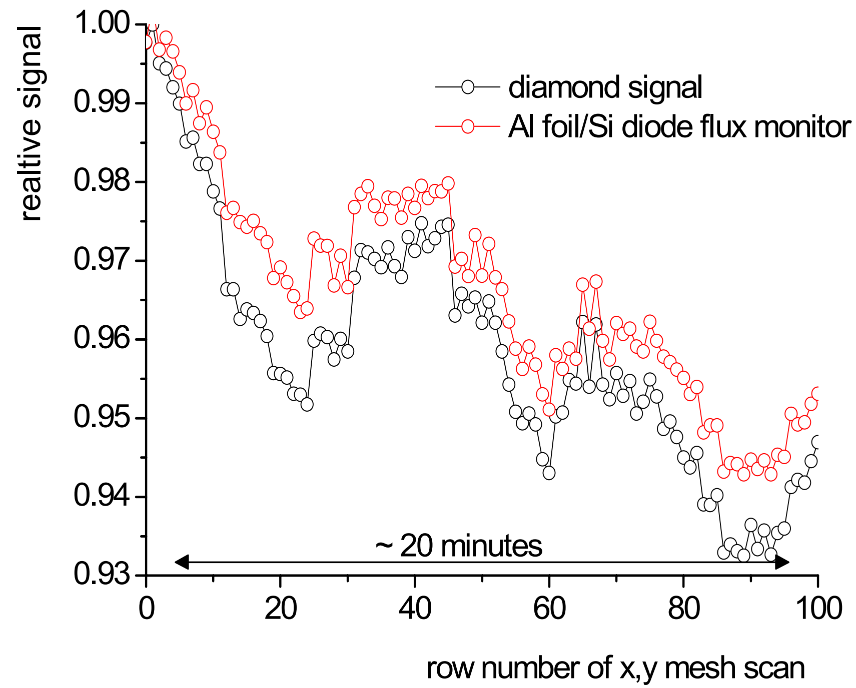
Guard ring

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 1.1μm^2 at 6keV (ID21 beamline)



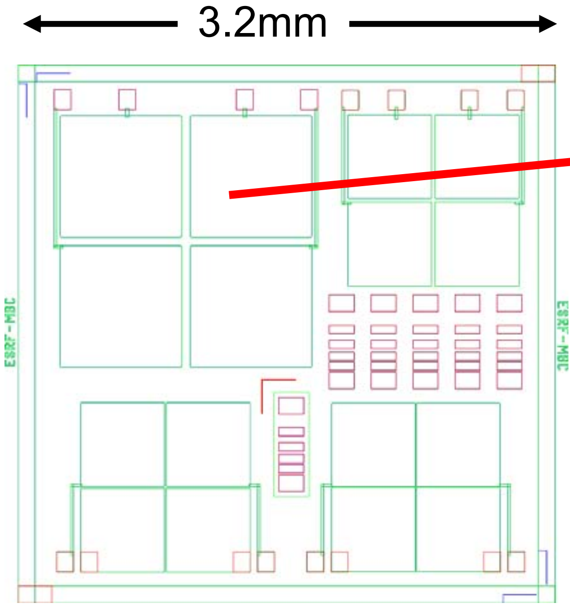


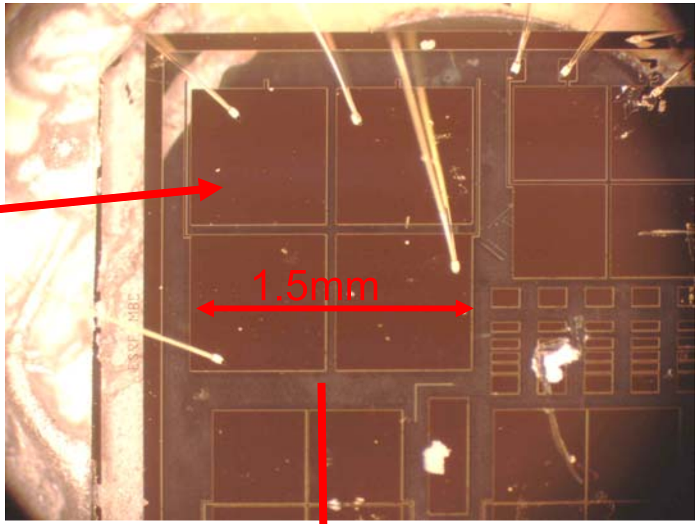
Row 40 signal variation **0.103%** (1σ)

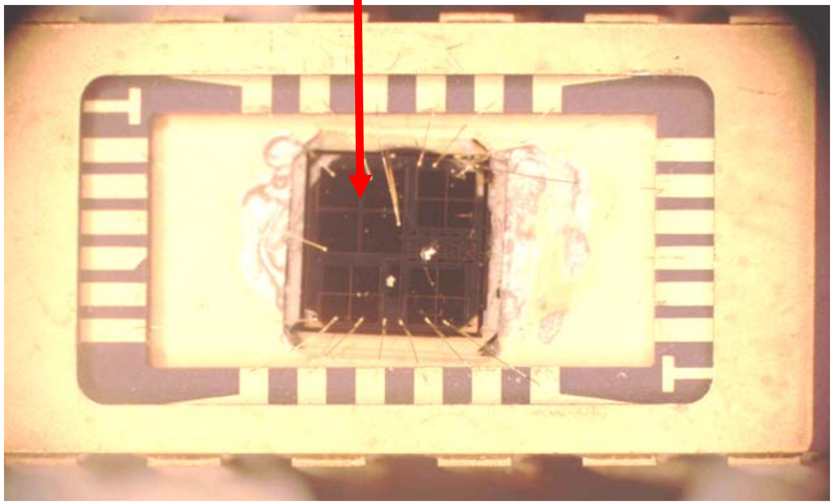
Signal variation during mesh scan

- linescan at column 50, rows 0-100

bad contacts (insufficient surface cleaning)







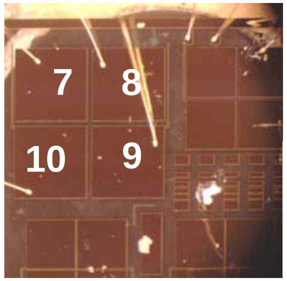
Ni/Pt/Au and Ti(annealed)/Pt/Au contacts, fabricated at Stanford NanoFab' Facility (Chris' Kenney)

50, 20, 10 and 5µm quadrant isolation gaps

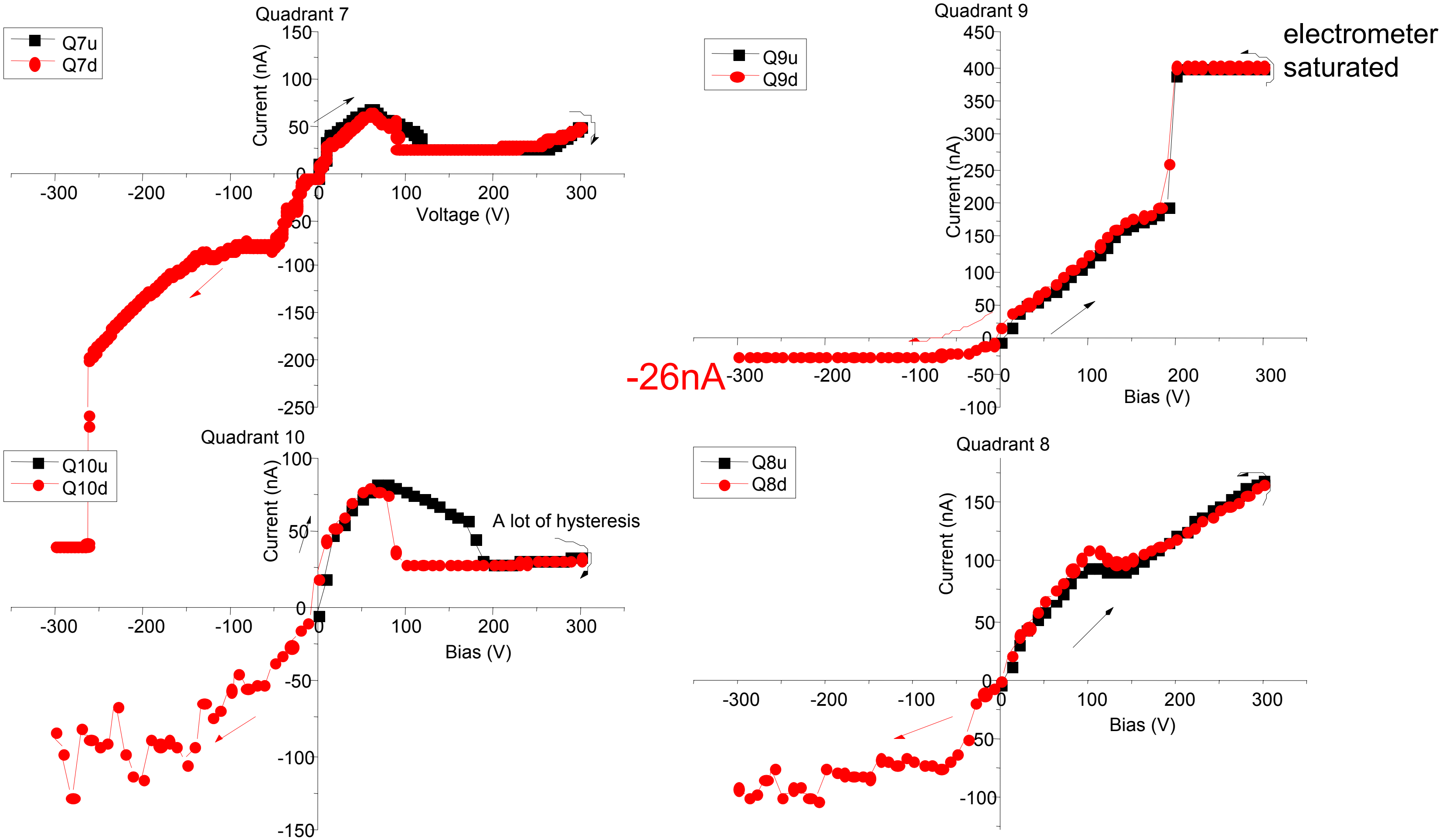
E6 MDS2 & MDS3 samples, Achard-CNRS ATJ30 sample

Failed processing of Ni-TiC contacts

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

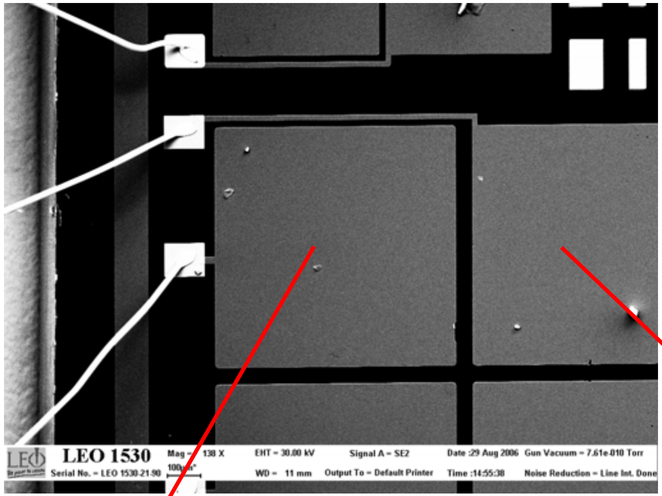


bad Ni-TiC contacts
Sample thickness ~350µm

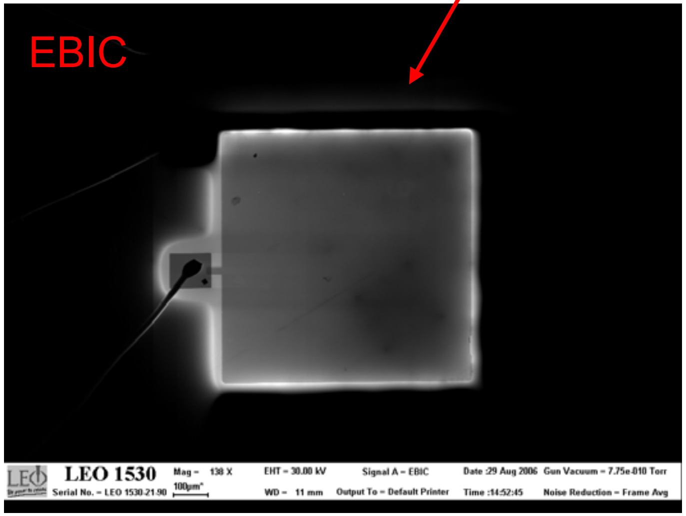


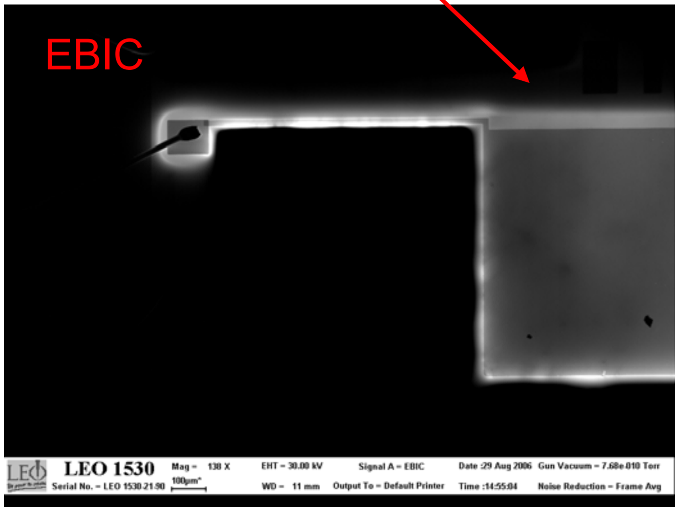
E6 SC material, Stanford processed Ni-TiC electrodes

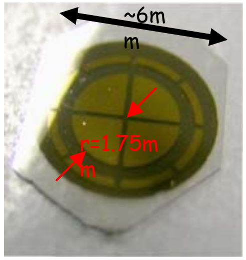
Secondary electron emission contrast



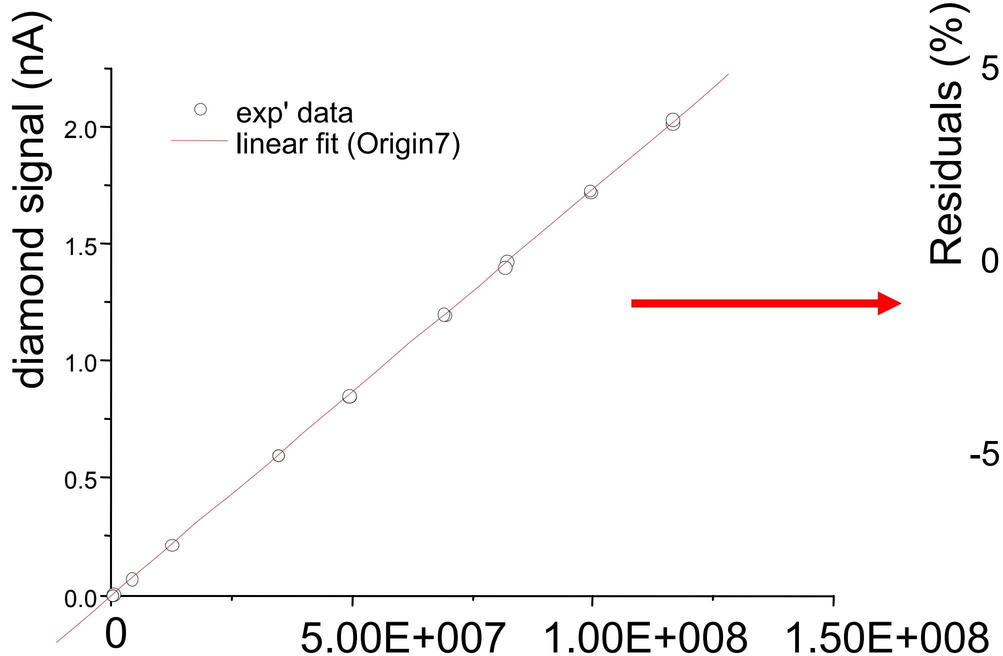
!! Surface probe 1 ~ 2 µm

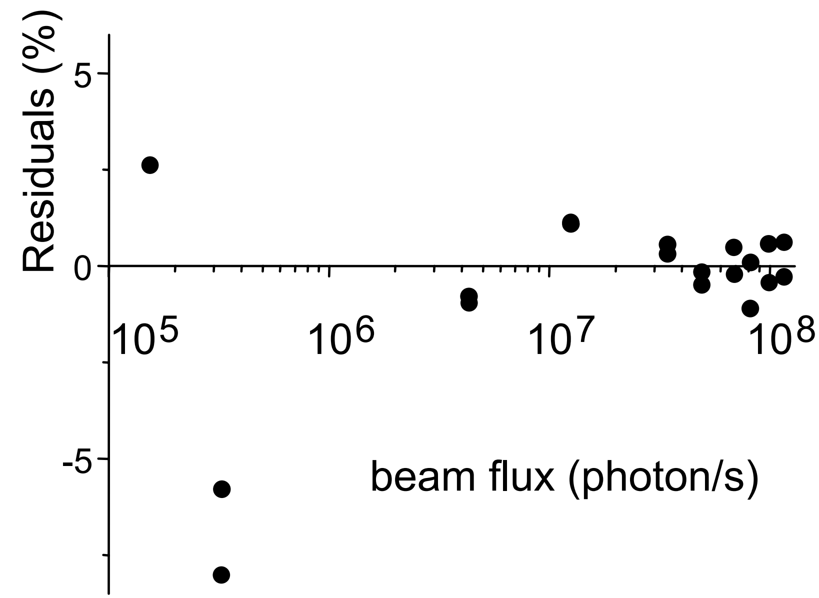






E6-70310 thickness 100μm *single crystal*
20nm+20nm Cr, Au contacts - GSI
'dc' quadrant currents measured with electrometers
bias 50V

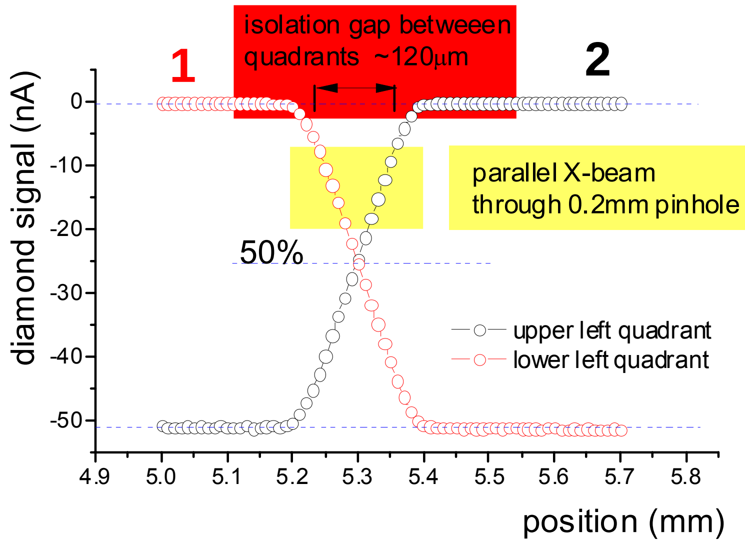




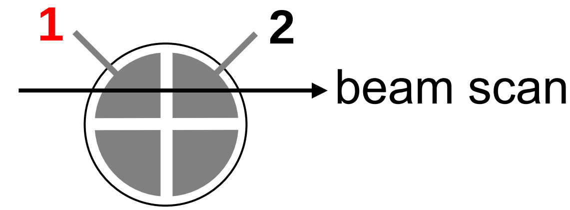
Incident beam flux (X-ray/sec in 0.4 x 1.2 μm^2 fwhm spot)

20% diamond absorption, 4 bunch synchrotron fill pattern with 2.8μs orbit

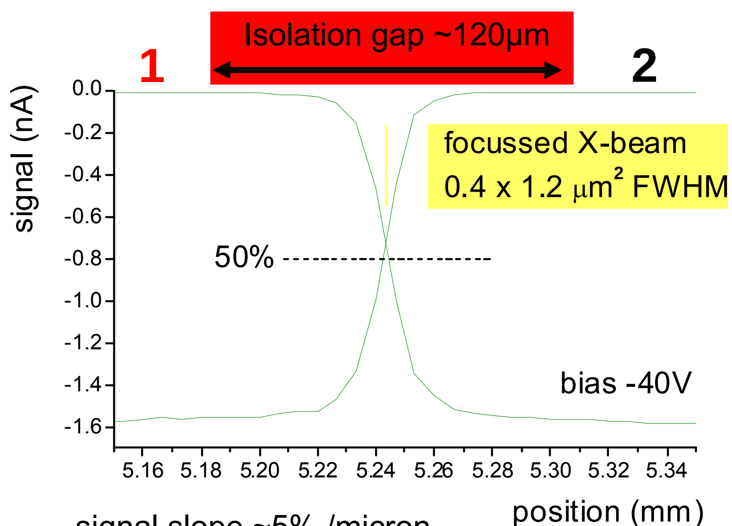
→ <<1 … ~15 X-ray photons / bunch



signal slope ~0.5% /micron

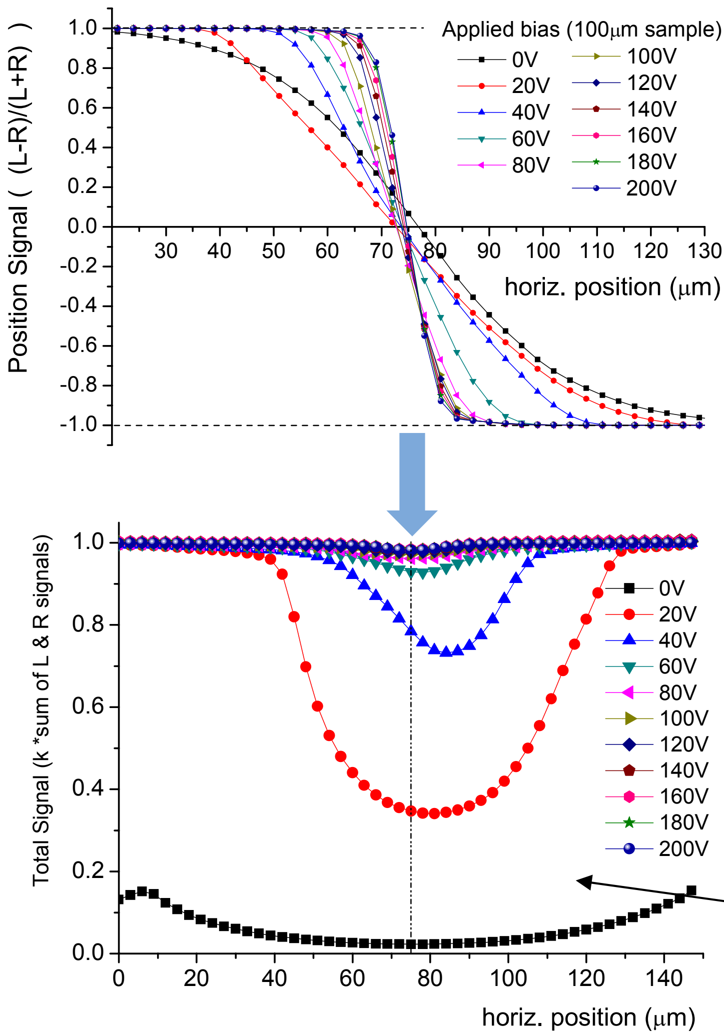


For large beam (> 50µm), quadrant 'crossover response' is ~ line integral over the beam intensity profile

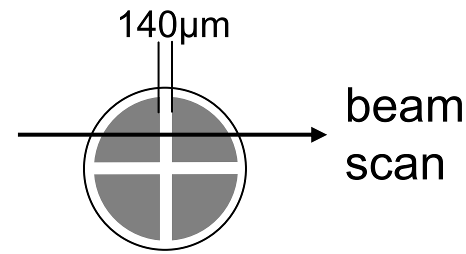


signal slope ~5% /micron

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



nb. low frequency Electrometer current measurements

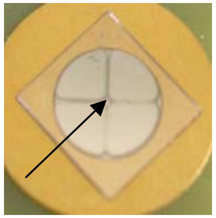


For small beam,
response slope / sensitive range may be modulated over range 10~100μm by
- applied electric field (bias)
- inter-electrode gap / sample thickness

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

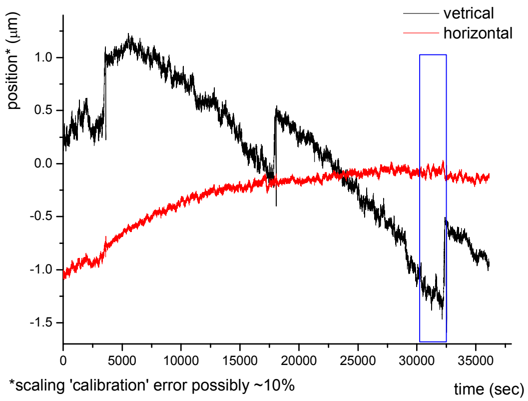
100μm thick sample, 140μm quadrant spacing
exaggerates weak field effect

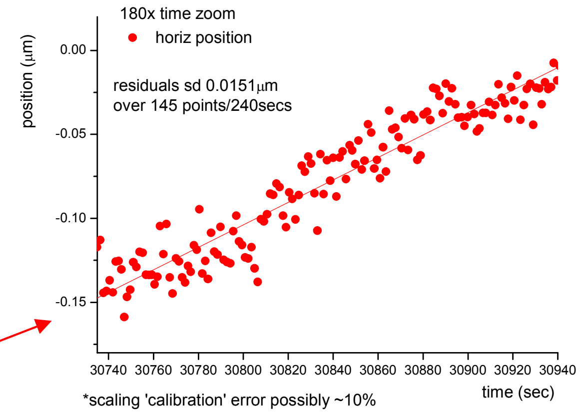
ID21 Vertical / horizontal position timescan



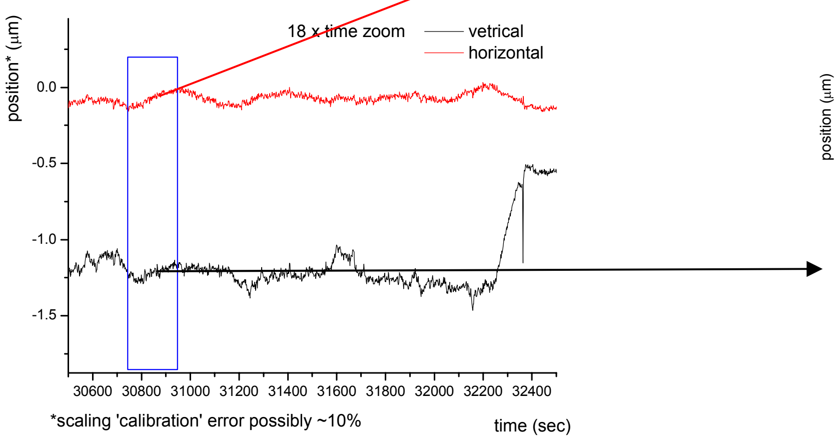
Al-Al contacted quadrant device,
ESRF ID21 beamline (MI-885)

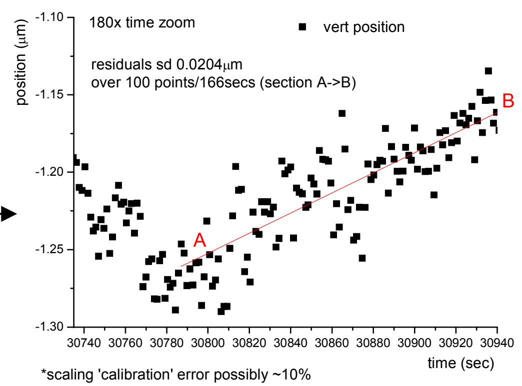
timescan V->F data, 1sec integrations





σ <15nm

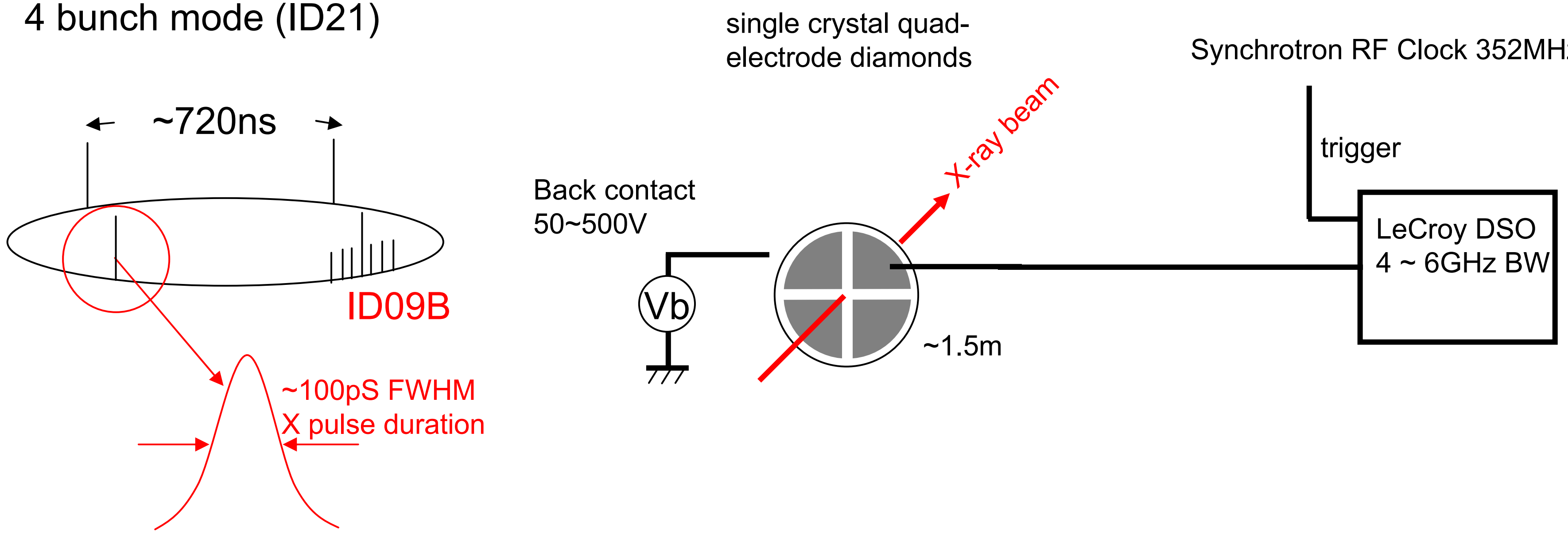




σ <20nm

Wideband measurements (ID09B, ID21)

ESRF synchrotron in
4 bunch mode (ID21)



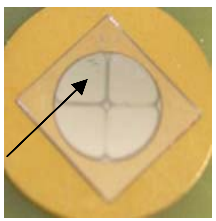
ID21: focused X-ray beam ~0.5 x 0.1 µm^2 and ~10^2 ph/pulse at 6keV

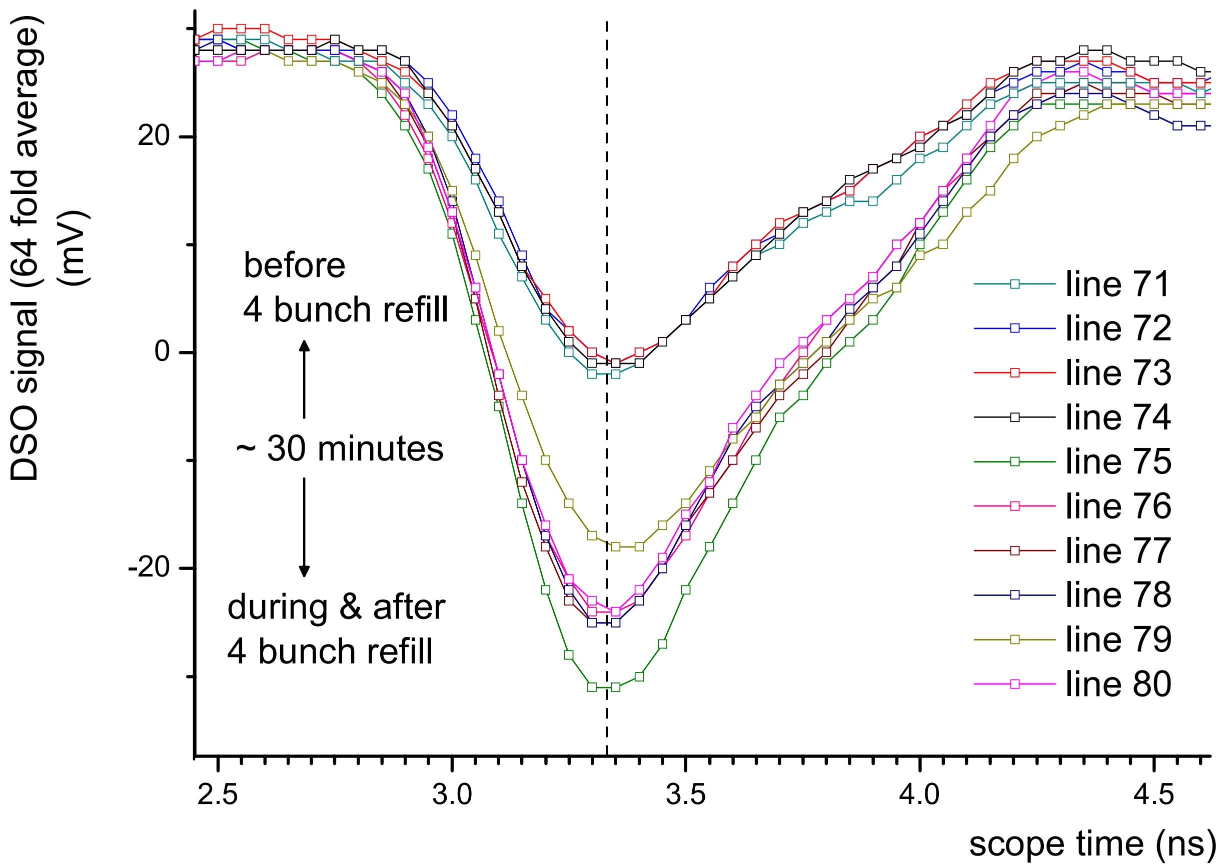
ID09B: ~50 x 100µm^2 chopped white beam, ~10$^{5-9}$ ph/pulse at ~18keV

5% beam absorbed in diamond → ~1pC … 10nC /pulse (1GeV … 10TeV)

DSO signal after ~30db preamp gain

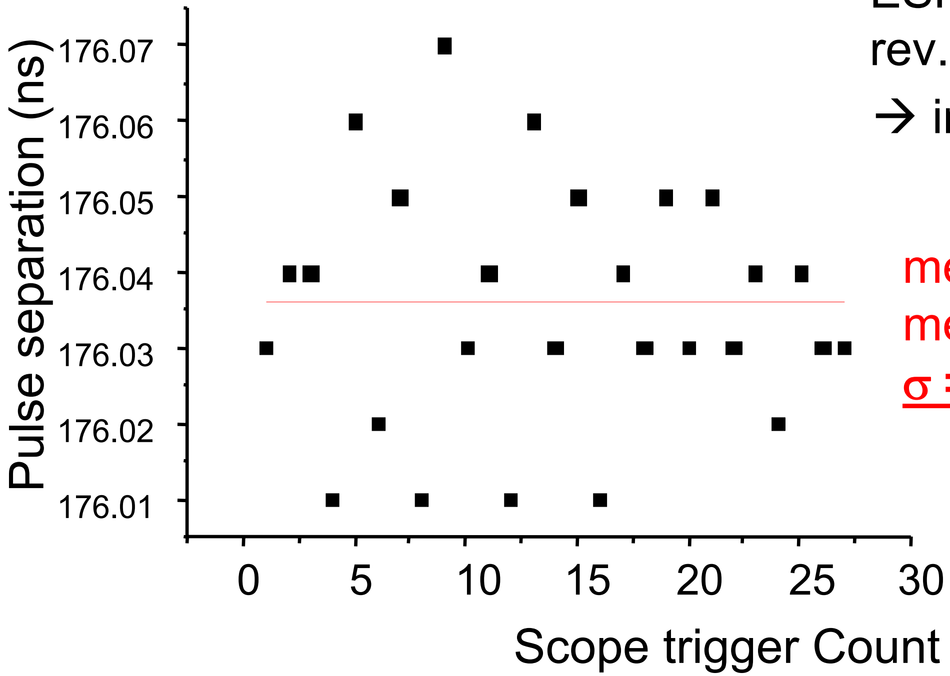
device layout not optimized for RF!





+150V/118µm Al-Al sputtered electrodes, *single crystal*

E6 Sample MDS-2, 'bad' Stanford Ni-TiC contacts, DBAIII 2.3GHz preamp
single crystal

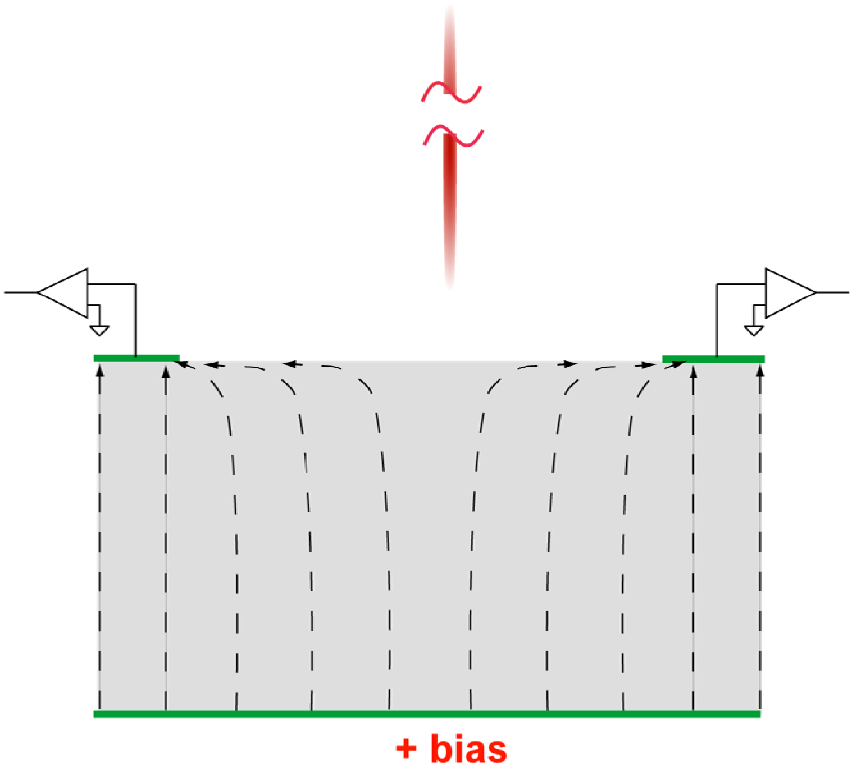


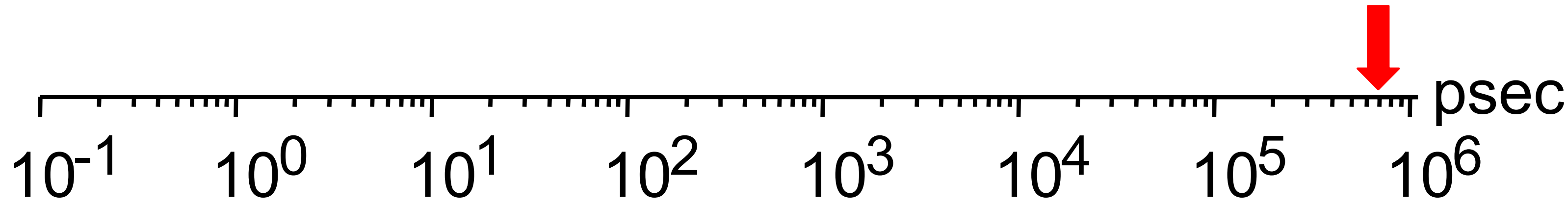
ESRF RF group data:
rev. freq. 355044Hz / 16 bunches
→ interbunch period = 176.035ns

measured:
mean=176.036ns
σ = 16ps

Lecroy DSO data post-analyzed with leading edge 'discrimination' normalized
Individual to signal pulse integrals
(σ→20ps 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*

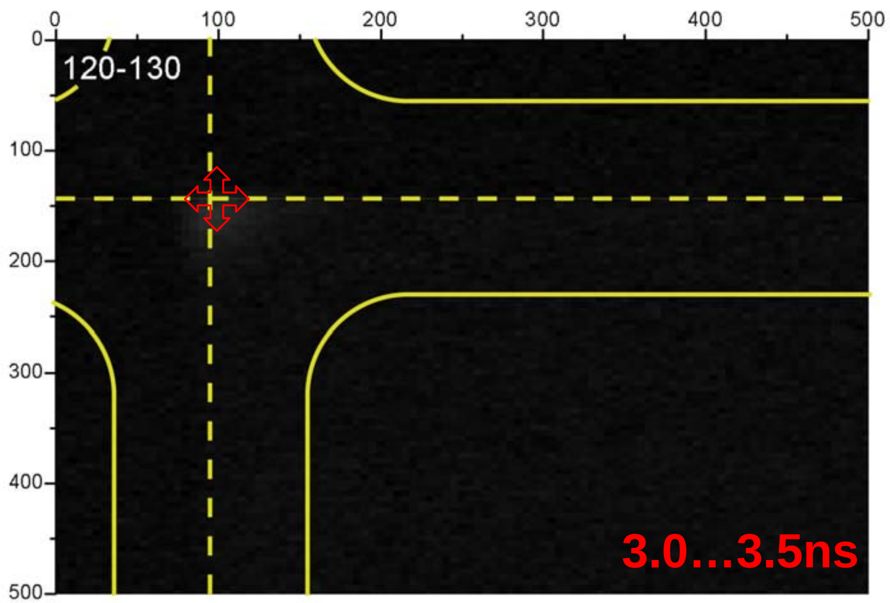
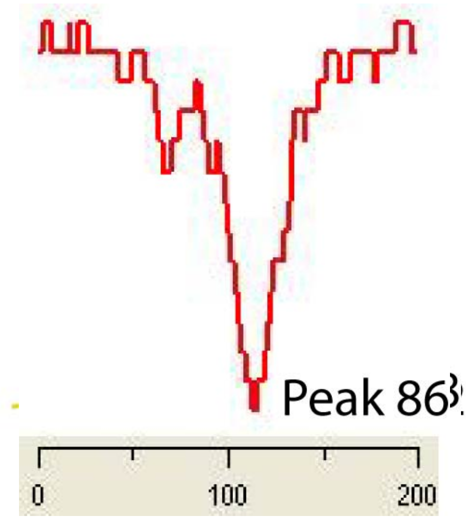


Image 2-scan1 p105

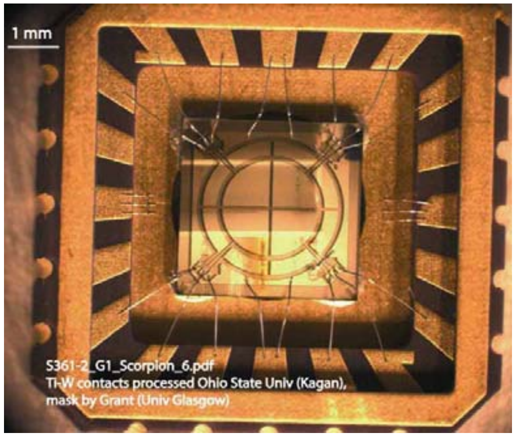


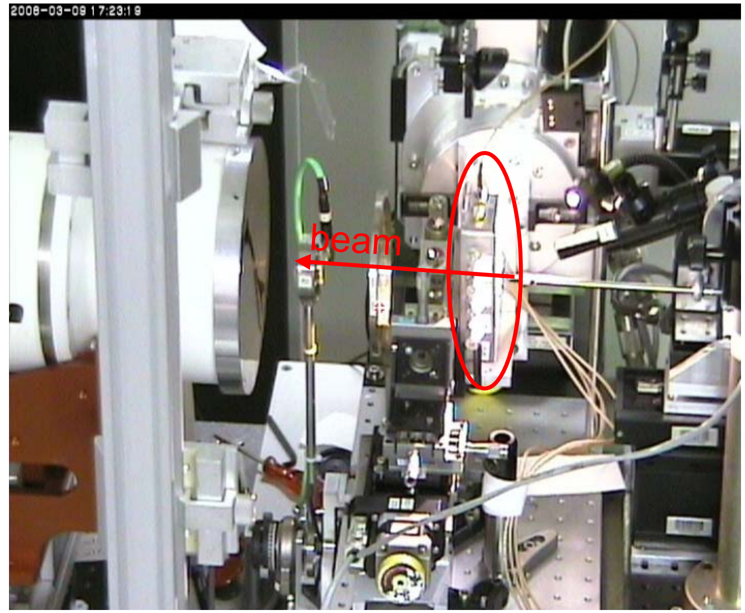
0.5ns wide 'gated integrations',

time-slew of signal as function of beam position
(response of bottom right quadrant only is shown here !)

‘position response at ID09B (chopped beam)

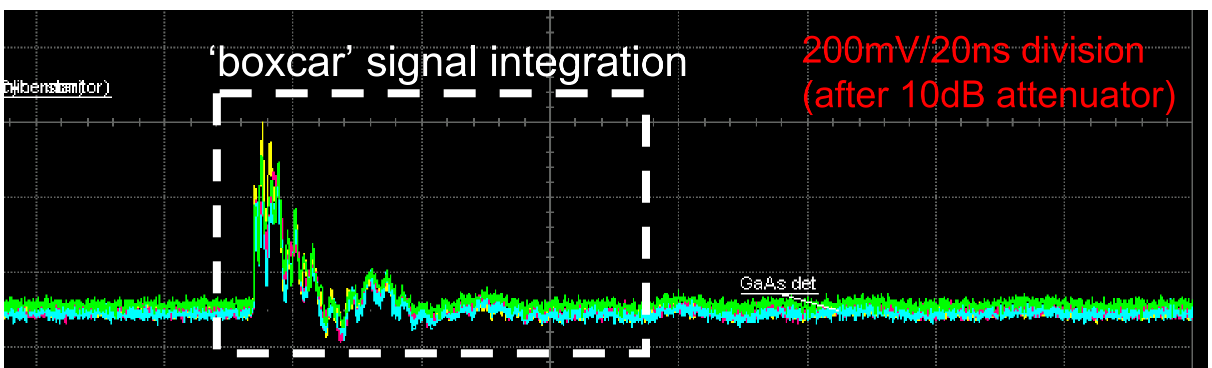
~5 x 10^7 ph/pulse (1kHz) at ~18keV, 5% absorption in ~350µm thick diamond





Ad hoc test of single crystal OSU quadrant device

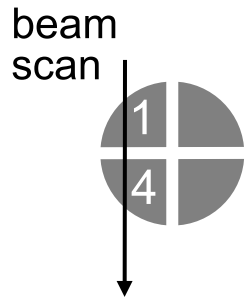
Single crystal sample,
TiW contacts

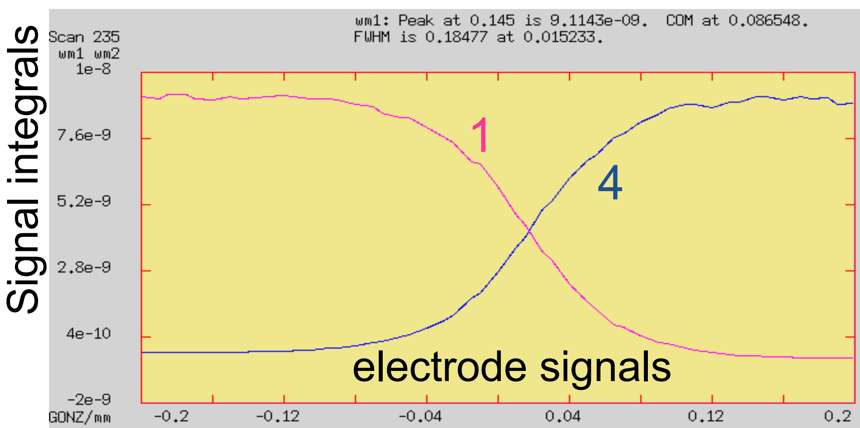


Very poor circuit layout & cabling of diamond support → signal ‘bounce’

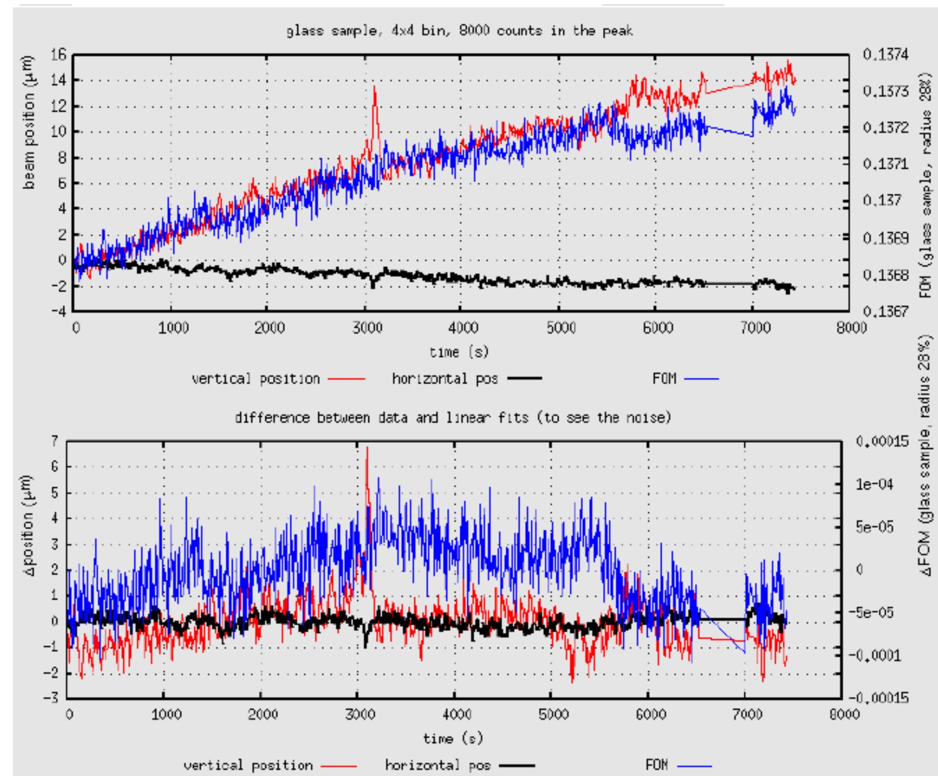
Position response

(motor scan of diamond)





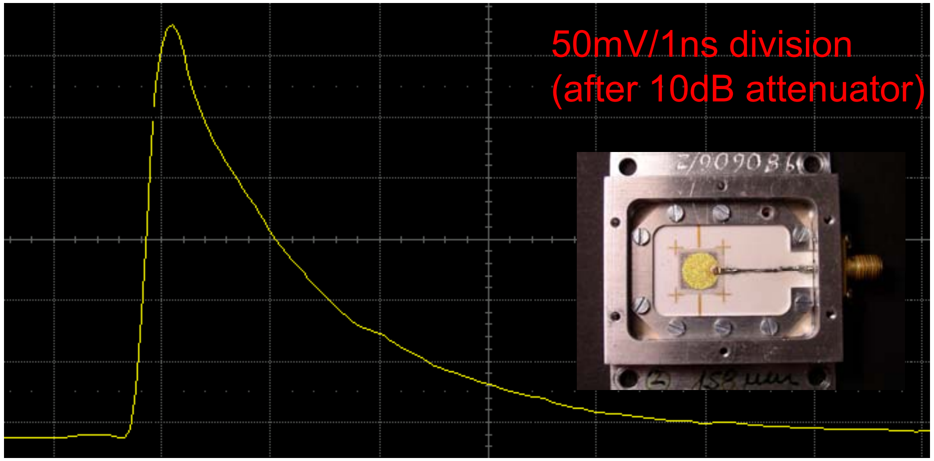
timescan with beam initially centered on diamond

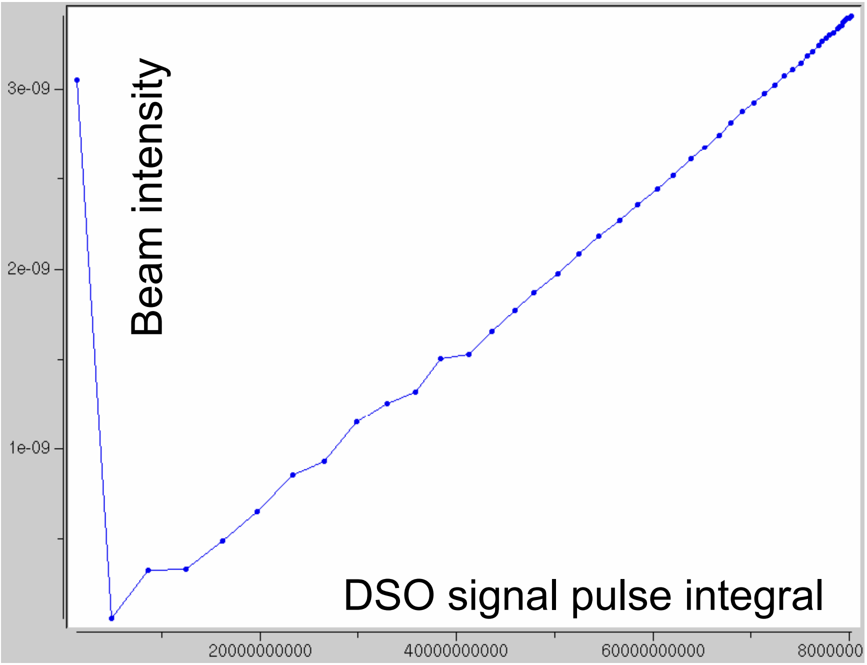


Beam found at : (y,z) = (0.002,-0.004), size: 0.188 x 0.069 mm

linearity, stripline mounted GSI sample

'old' E6 *polycrystalline* material, Cr-Au contacts on unpolished surface





Polychromatic beam, choppers in, ~18keV X ray beam
U17-13, phg 7 pvg 0.7, pd2f counting: 1.24×10^{11}/sec
In beam spot ~100 x 200μm^2 fwhm.

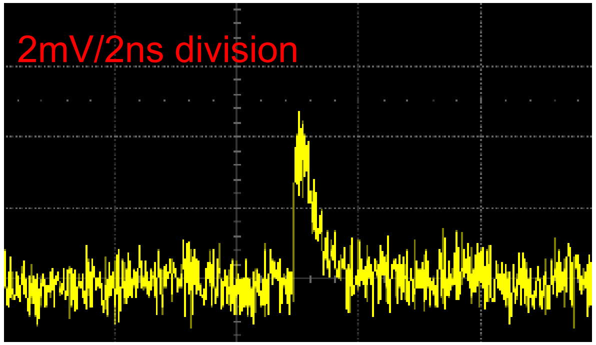
rise time: 235ps, area 5.4nVs,
fall time 3.2 ns determined by circuit response(?) Bias 500V / ~300µm,

Linearity check to ~6 $\times10^6$ photons absorbed/pulse)

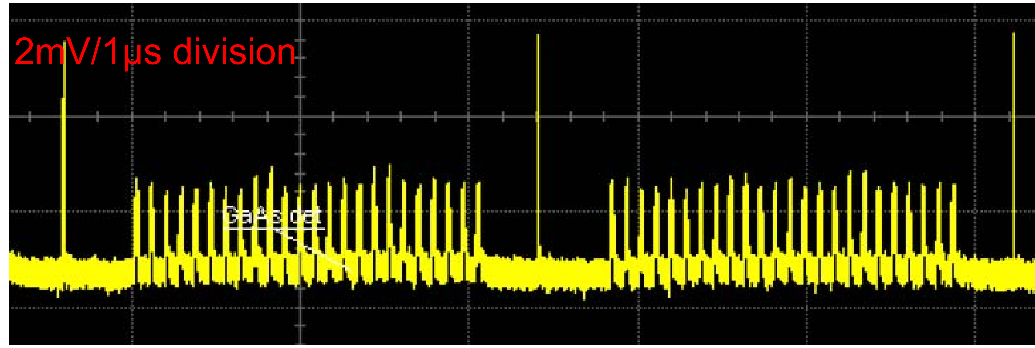
No reduction in signal response with overnight beam on sample

ID09B beamline time structure

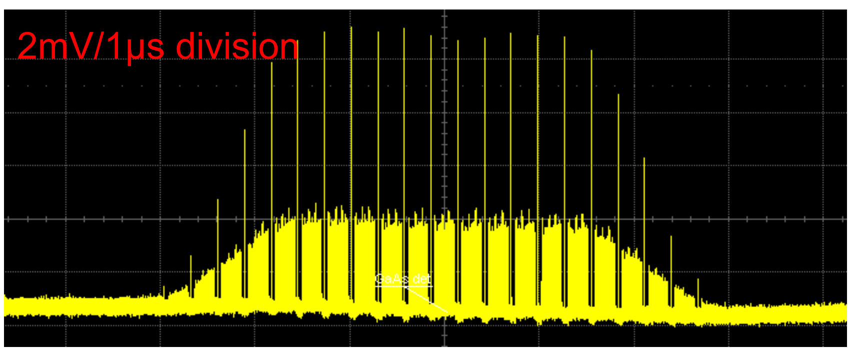
Measurements with GSI stripline mounted *polycrystalline* sample



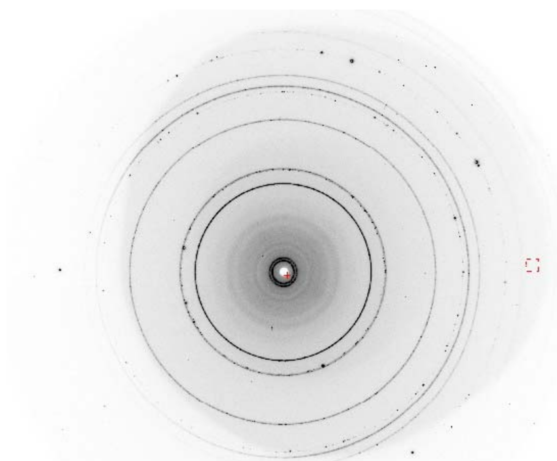
monochromatic ~ 5x10^5 ph/pulse at 18keV
~2% absorption (350µm thick diamond)



24*8+1bunch hybrid mode



Signal constant over duration of heatload chopper pulse.



Scattered beam ‘powder diffraction‘ rings arising from the ~10µm random orientatation grains of the polycrystalline diamond plate '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	~0.1%[1]
position response: sensitivity	~10nm*
timing resolution / leading edge risetime	~20 / 250ps**

rms figures

[1] single crystal samples only !

* for a 1µm beamsize. *Accuracy is* limited by lithography

** circuit layout limited

present objectives*:*

→ *validate 'industrial' source(s) of reliable contact processing with precision lithography*

…on thinned (to <50µm) CVD plates

→ *implement electronic readout: multichannel electrometers or RF signal techniques*

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…

(~5 GigaGray, 16hour stability test made at ID21)

acknowledgements

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H. Kagan	Ohio State University
Ph. Bergonzo	CEA-LIST (Saclay)
M Nesladek	Institute of Physics, Prague
P. Muret	SC2G Institut Néel (Grenoble)
M. Mermoux	LEPMI Univ. J. Fourier (Grenoble)
J Butler	Naval Res. Labs. (Washington DC)
(Ch. Kenney)	Mol. Biology. Cons. (Stanford)
M Salomé, J. Härtwig, L Descamps, I. Snigireva, A Rommevaux (E Mathieu, H. Gonzalez, G. Naylor…)	ESRF (Grenoble)

…

Element Six (Ascot & Cuijk), Diamond Detectors Ltd., LIMHP:
material samples, surface polishing