CVD Single Crystal Diamond

synchrotron X-ray beam monitoring and surface characterization tests at ESRF

J Morse, M Salomé, E Mathieu — ESRF - Grenoble

E Berdermann, M Pomorski — GSI - Darmstadt

J Grant, W Cunningham — University of Glasgow

C Kenney — Mol. Bio. Consortium - Stanford

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M. Mermoux	LEPMI Univ. J. Fourier (Grenoble)
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J. Butler	Naval Research Lab' (Washington)
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Talk Overview

1. Synchrotron X-ray beam monitoring requirements, and why single crystal diamond?

2. Surface and bulk characterization tests of CVD diamond sample material

3. CVD device ‘XBIC’ mapping tests at ESRF-ID21

Synchrotron X-ray Beam Monitoring: what is needed?…

Transmissive sensors → permanent, in-beam measurement of:

Intensity: typical need is 1%…0.1% (relative) accuracy & linearity
for *sampling* times ~msec … ~1 secs

Position / Vector (& Profiling):

Synchrotron beam size at samples is now
~1…~100μm , with 50nm FWHM achieved
Required beam stability ~10% of beam size
Profiling matrix ~10 x 10 points ?

X-ray fluxes:

~10^8 photons/1μm^2/s ... 10^{13}photons/(100μm)^2/s
photon energies ~ 1 … 50 keV, $\Delta E/E \sim 10^{-4}$

→ max. *absorbed* power: < mW (monochromatic beam)

but >10W in 'white' beam applications!!

…why diamond?

Z = 6 → low specific X-ray absorption / beam scattering

High charge carrier saturation velocity (~3×10^7cm/s) and low dielectric constant (5.5)

-> fast pulse response (~1nsec in 50µm thick device)
synchrotron 'pulse by pulse' analysis

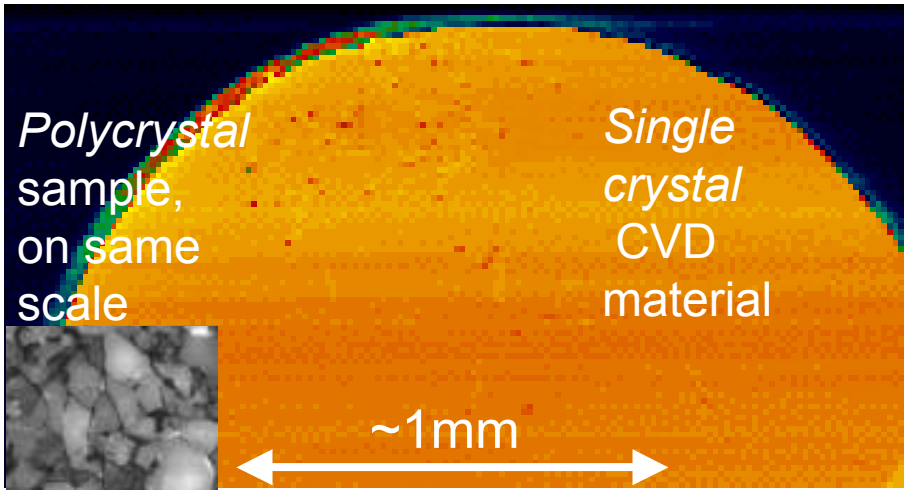
wide bandgap energy (5eV), excellent thermal/mechanical properties

-> low leakage currents at high temperature,
high heat load 'white' beam monitoring possibility

why *single crystal* material ?

beam coherence preservation:
no grain-boundary artifacts
→minimal bulk scattering
X-optics surface quality

charge-carrier lifetime ~1µs
→100% charge signal collection over ~mm distances)



Courtesy Ph. Bergonzo E Berdermann/E6

1. Synchrotron X-ray beam monitoring requirements, and why single crystal diamond?

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3. CVD device ‘XBIC’ mapping tests at ESRF-ID21

Methods of sample analysis tested:

Material:

BULK

Raman confocal micro scanning, visible/UV microscanning fluorescence spectrometry

cryogenic fluorescence spectrometry, visible, UV, electron excitation

electron spin resonance: impurities at (sub)-ppB level

SURFACE

X-ray topography; X-ray small angle scatterinng

AFM and laser optical interferometry

Device:

BULK & SURFACE

XBIC (X-ray induced current) mapping: near surface or bulk charge injection

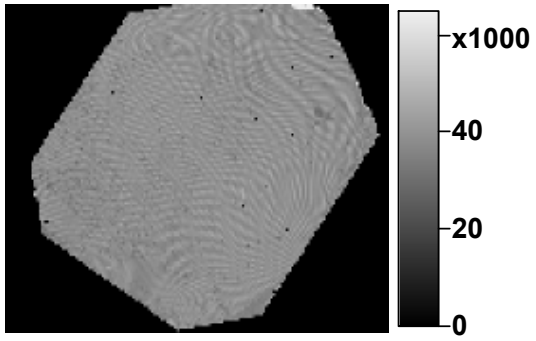
Scanning electron microscopy EBIC: near surface charge injection (radiation aging tests?)

Microscanning Raman

E6 sample 70310

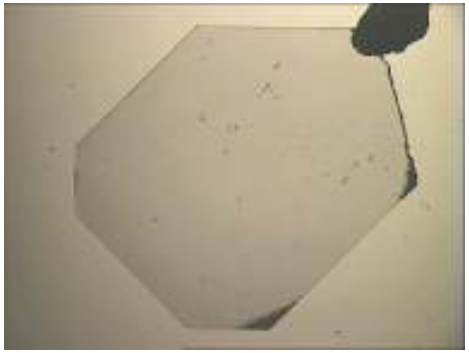
*Raman maps made with sampling ~2μm lateral spatial resolution, at 50μm raster grid

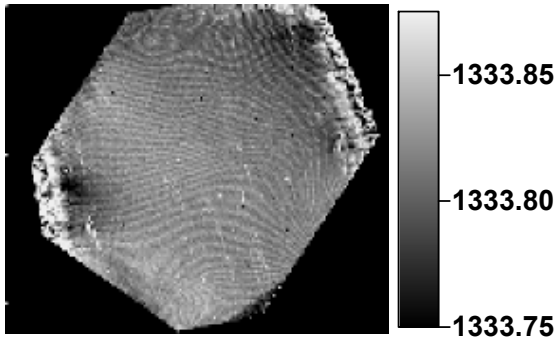
‘fingerprint twirls’ are artifacts in image display only



Raman* intensity (arbitray scale)

White light

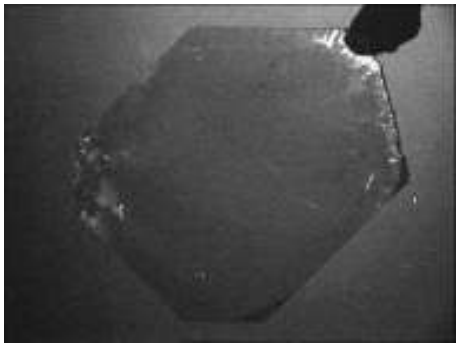


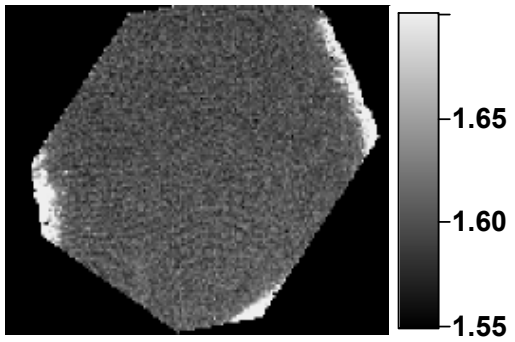


Raman* center frequency (cm^{-1})

n.b. not precisely calibrated!

Crossed polarizers



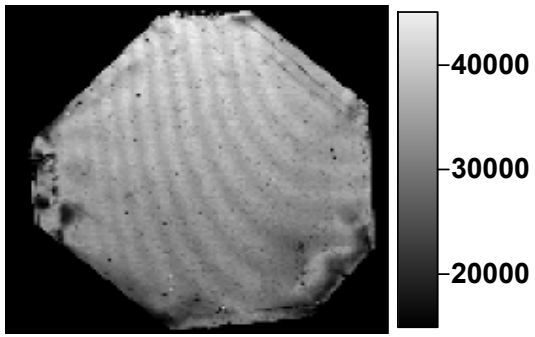


Raman* width (cm^{-1})

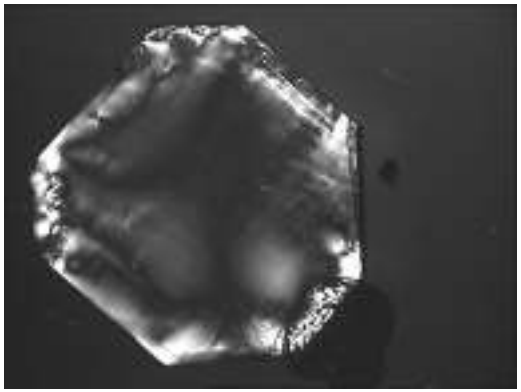
Courtesy Michel Mermoux LEPMI-ENSEEG, Grenoble Dec 2003

Apollo Diamond

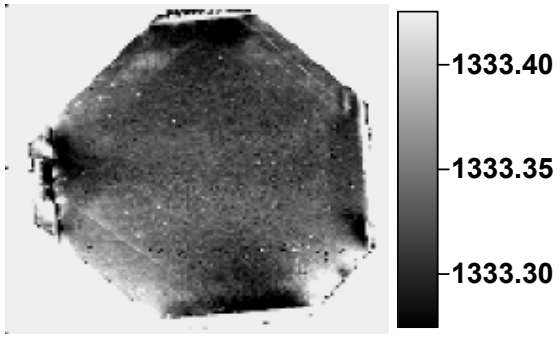
sample No.2186



Raman* intensity
(arbitray scale)

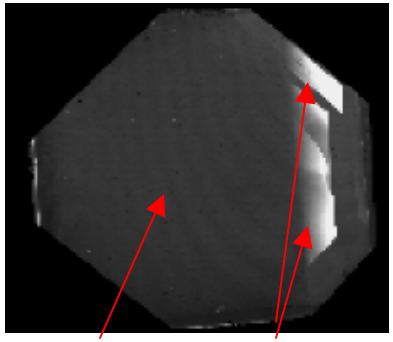


Crossed
polarizers



Raman* center
frequency (cm^{-1})

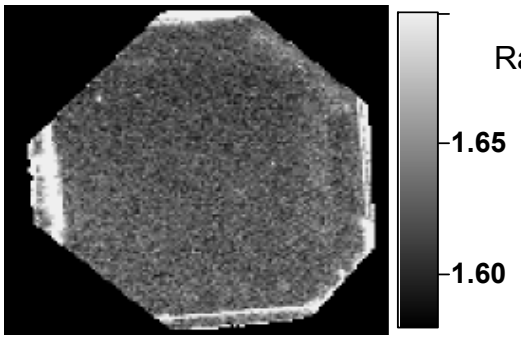
! System not precisely
calibrated!



Fluorescence emission
(argon-ion laser excitation
488nm)
Arbitrary intensity scale

Diffuse region
NVO centers
at 636nm

Strip-like features --!?
H3 centers at 503nm

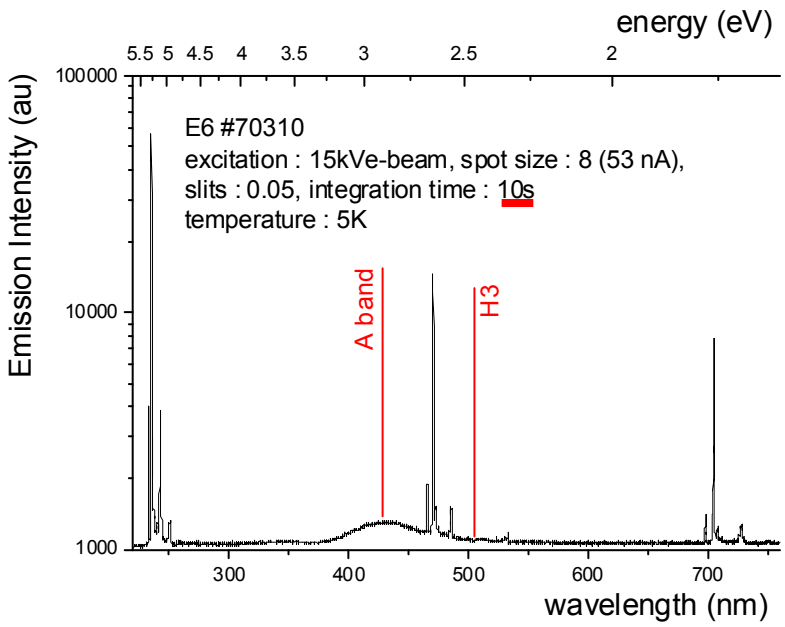


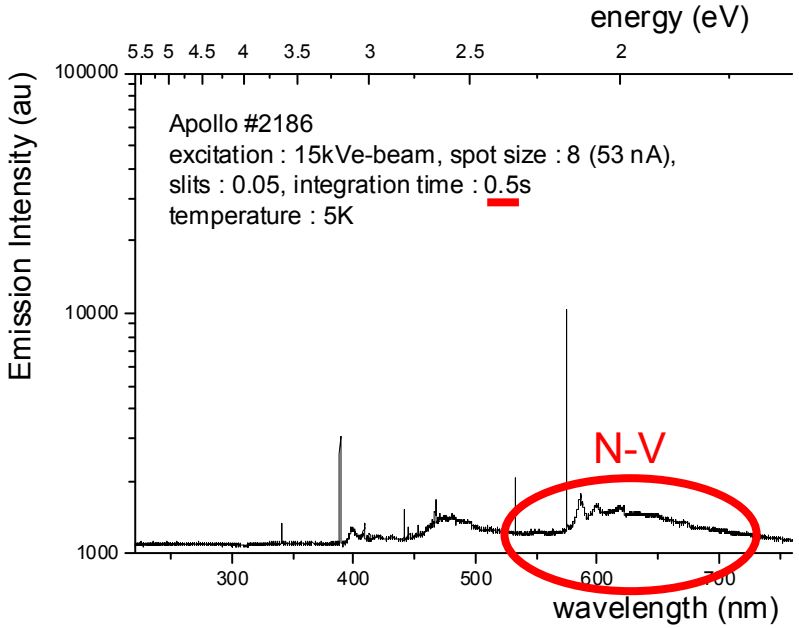
Raman* width (cm^{-1})

Courtesy Michel Mermoux LEPMI,
Grenoble Dec 2003

Cryogenic fluorescence spectrometry:

E6 70310 and Apollo 2186 Samples





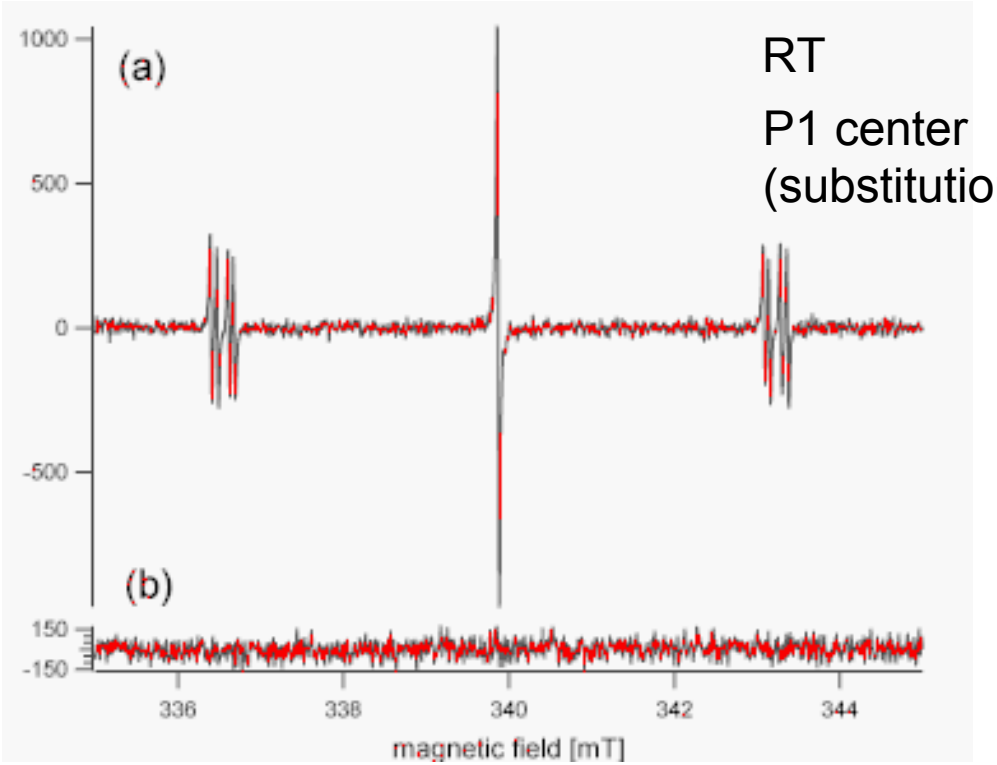
Courtesy P. Muret, M. Wade
LEPES-CNRS Grenoble

ESR measurements:

Sumitomo and E6 CVD samples

Sumitomo

E-Six*



RT

P1 center
(substitutional nitrogen)

*sample MDS-5
delivered ESRF
March 2005

(a) SumiCrystal (b) e6, scan times is 15 times longer than that of (a).

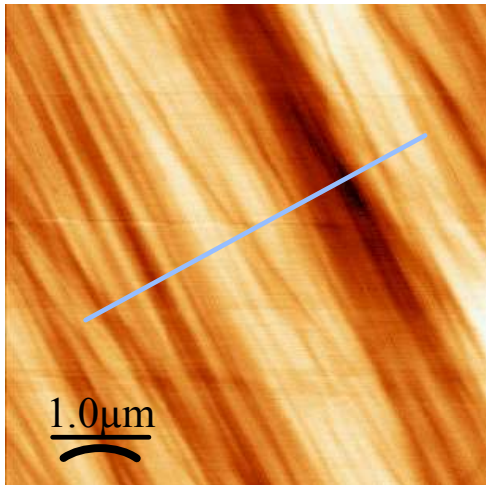
P1 center in SumiCrystal at $10^{14}/cm^3 \sim 10^{15}/cm^3$.
P1 center in E-Six is under detection limit ($<10^{14}/cm^3$).

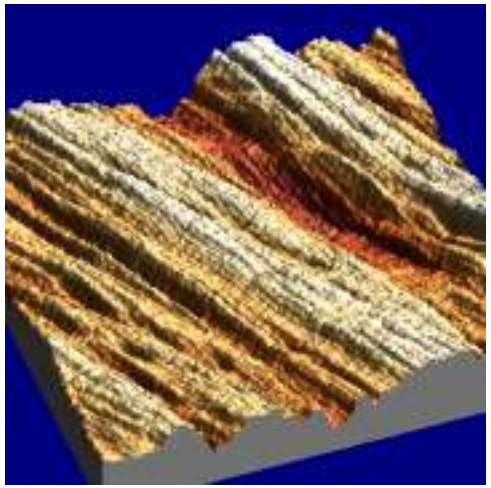
Carbon dangling bond defect (at g=2.0026) ~5×10^{14}/cm3
"NV^- and VH^- were not observed…"

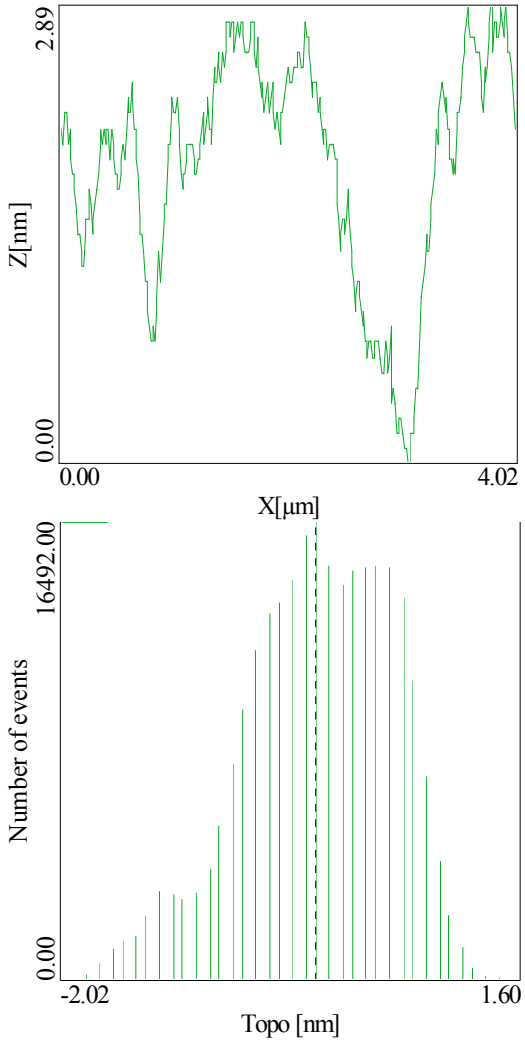
Courtesy Ch. Nebel, H. Watanabe AIST Nov 2005

Atomic Force Microscopy:

E-Six Ascot sample: *resin wheel* polished





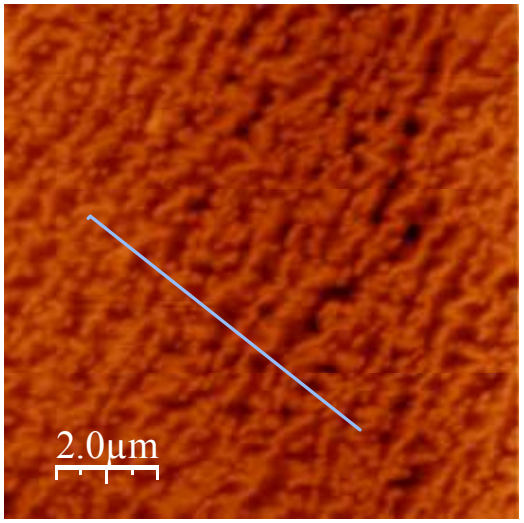


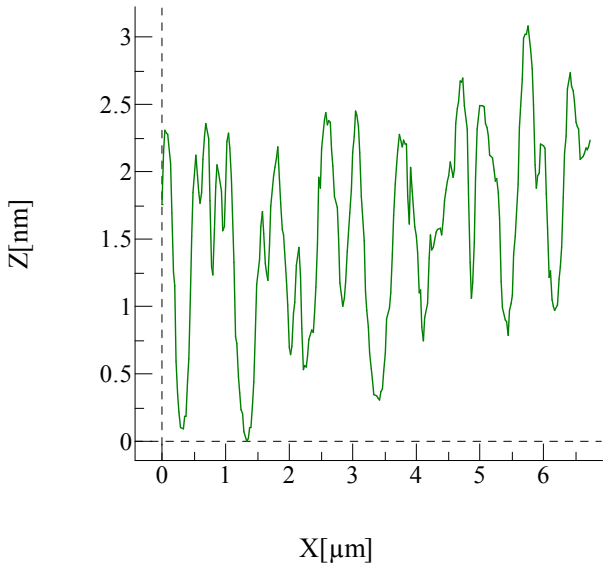
MDS3.015

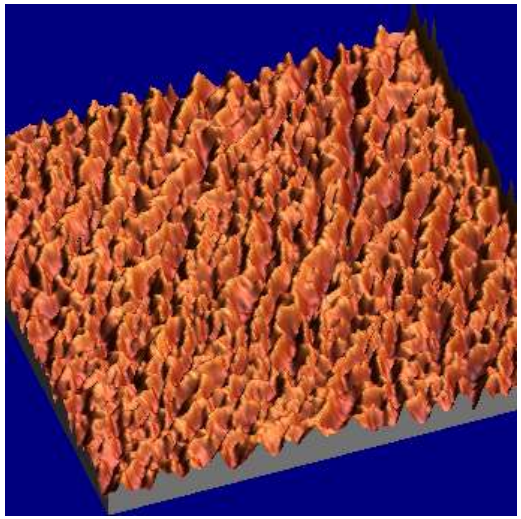
Side 1

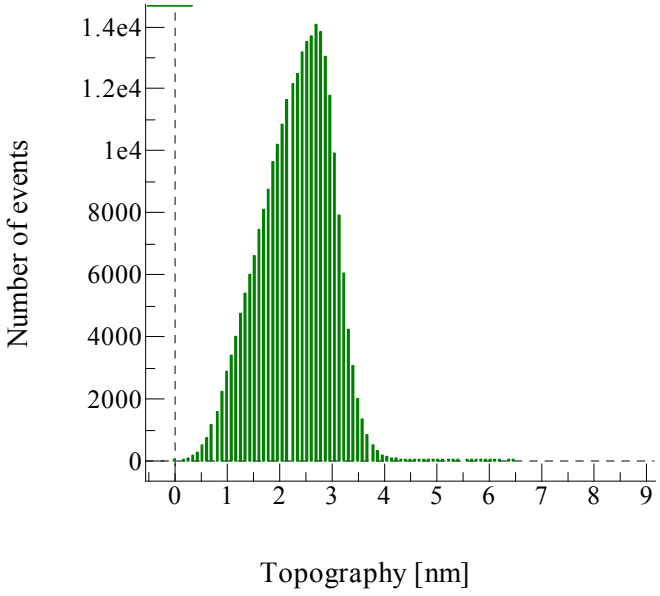
rms 0.60nm

E6 Ascot: *resin wheel* polished surface ??







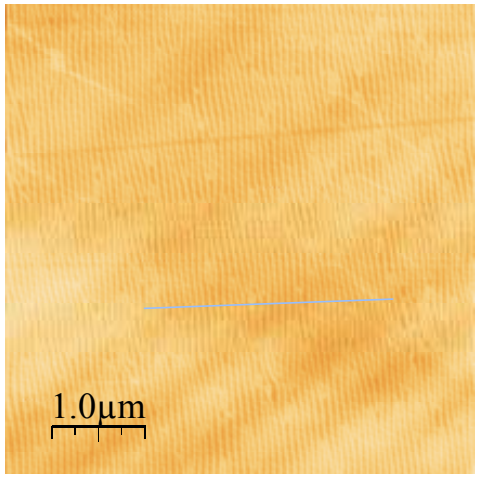


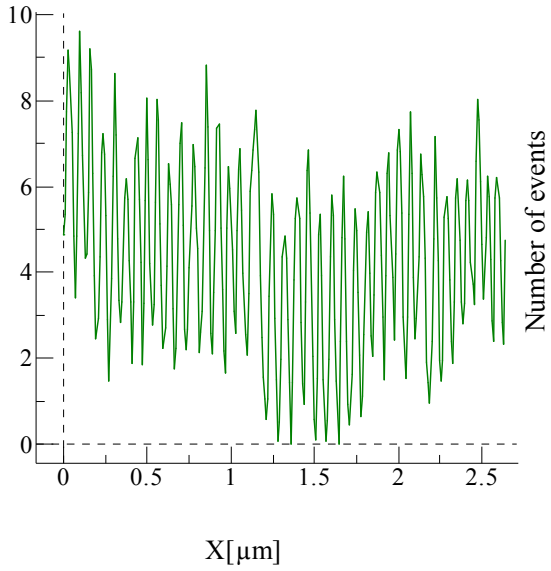
MDS-5-b.004

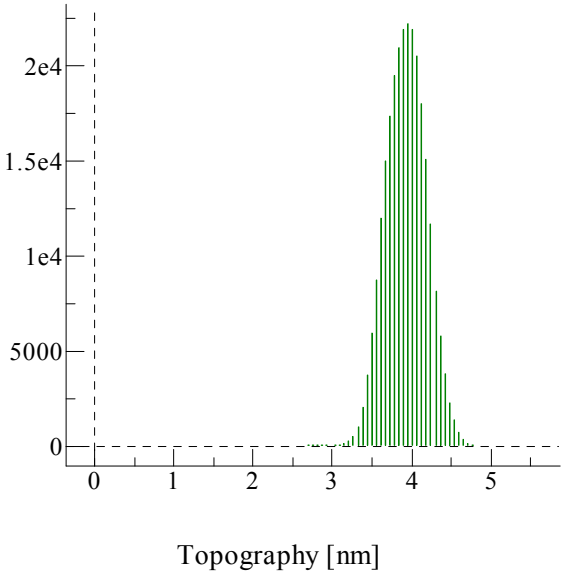
Side 1

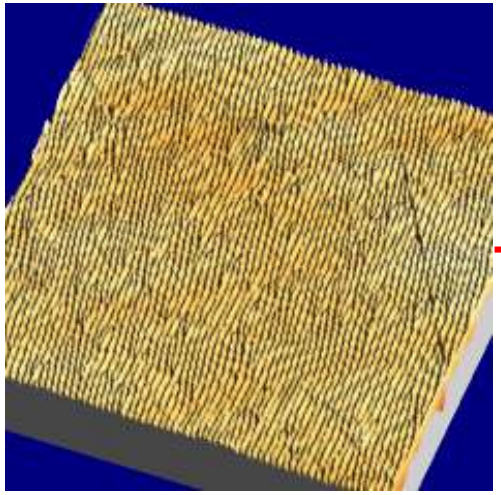
0.69nm rms

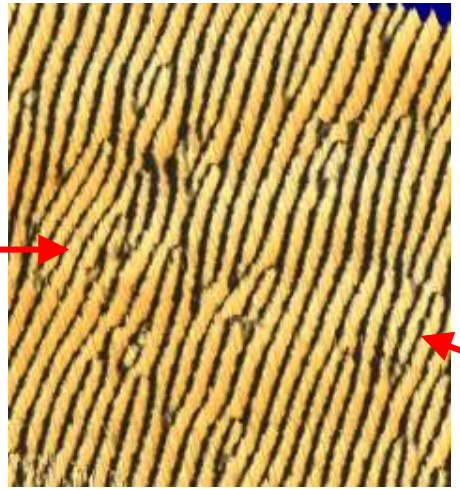
E6 Ascot: surface treatment ??











MDS3.004

Side 2

rms roughness
0.27nm

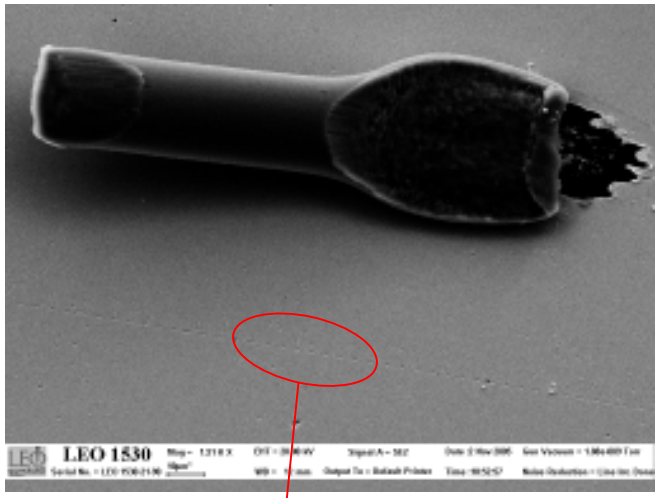
ridge pattern
period 71nm

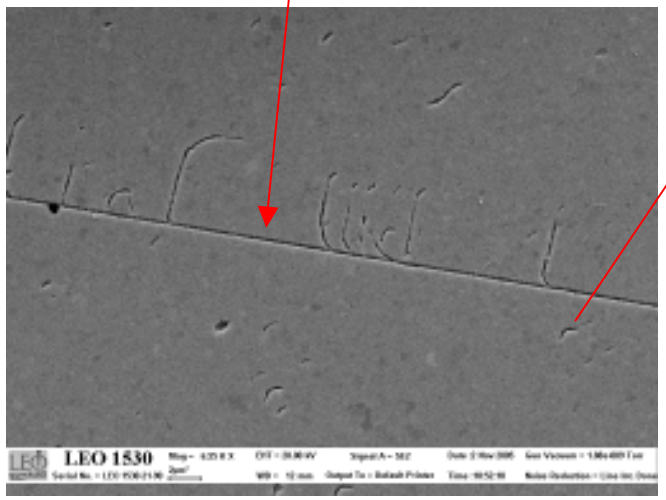
3D view

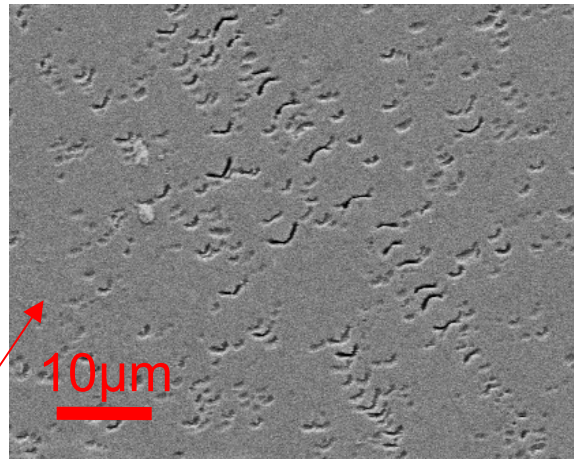
Zoom

E6 Ascot: resin wheel polish *'tadpoles'*

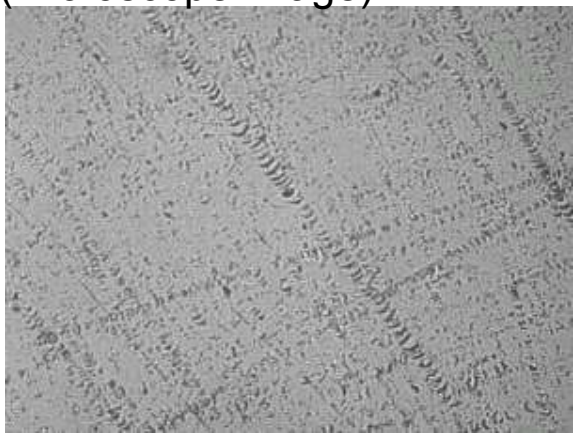
secondary electron microscopy at 20kV on Ni/Pd/Au coated surface



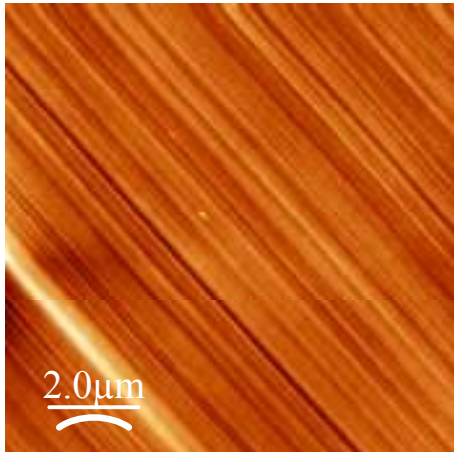




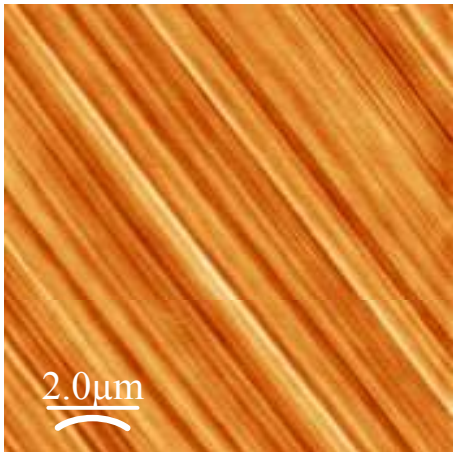
Pomorski, Nohrdia talk 2005, annealed 600°C/30mins naked sample (microscope image)



E6 Ascot sample after *scaithe polish* at E6-Cuijk*



elsc-a-1.003
0.71nm rms

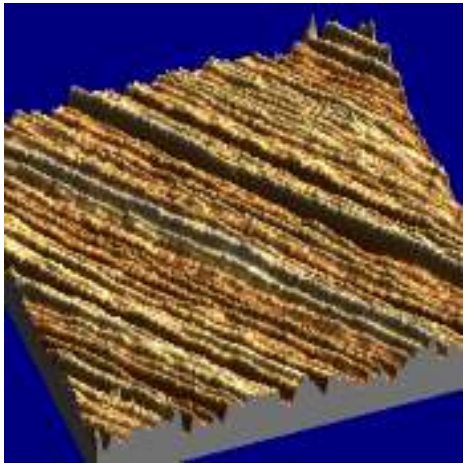


elsc-a-2.005
0.53nm rms

ESRF 02-12-05 A sample,

front & readsides

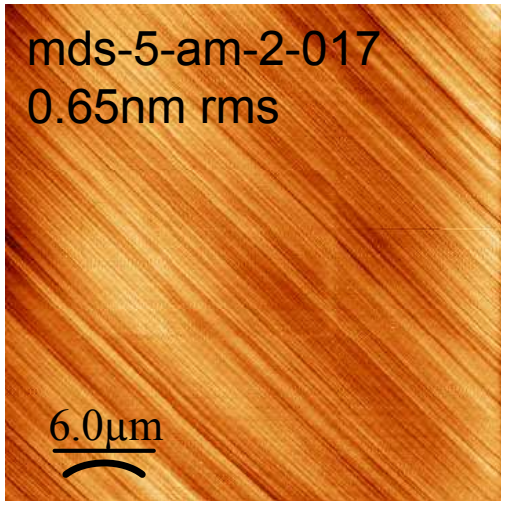
'thick' sample ~350μm



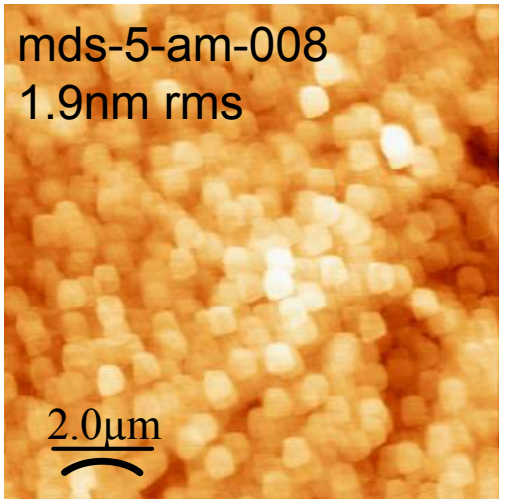
*courtesy Herman Godfried

E6 Ascot sample after *scaithe polish* at E6-Cuijk*

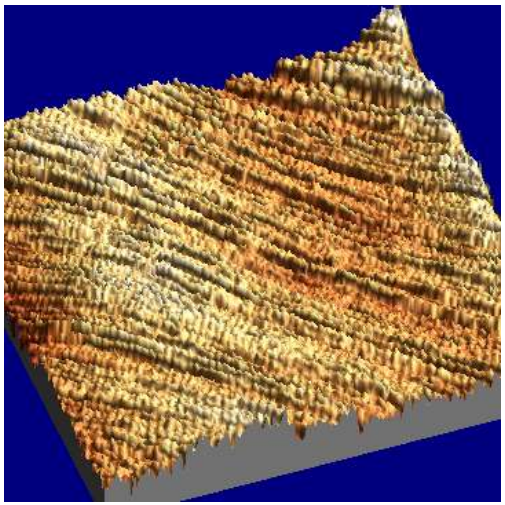
side 1



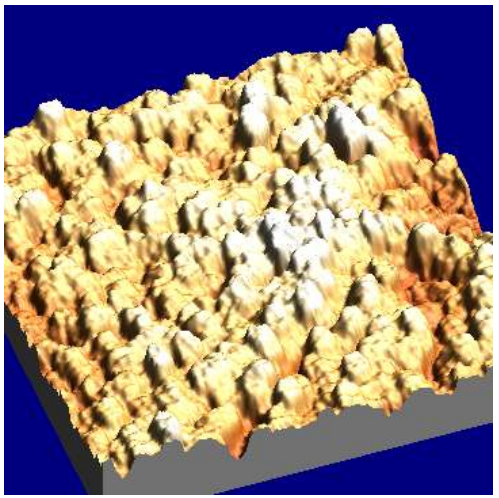
side 2



E6 sample MDS-5,
thinned to 40μm
at E6-Cuijk ~Mar 06



mds-5-am-2-017
0.65nm rms

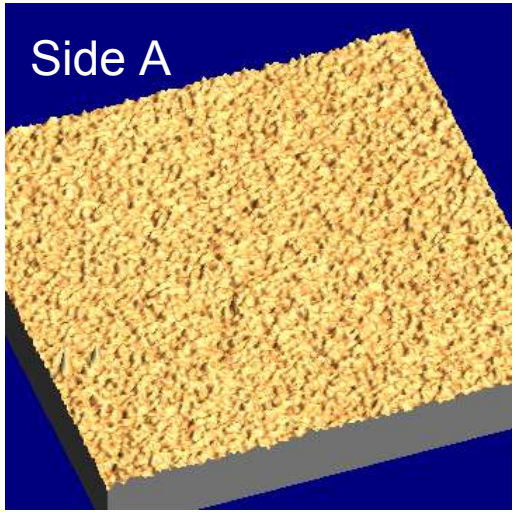


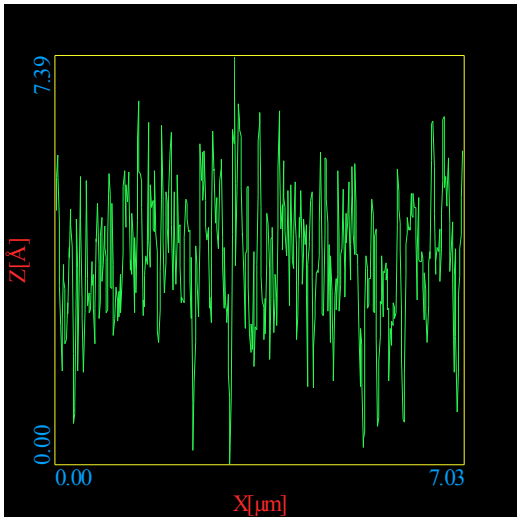
mds-5-am-008
1.9nm rms

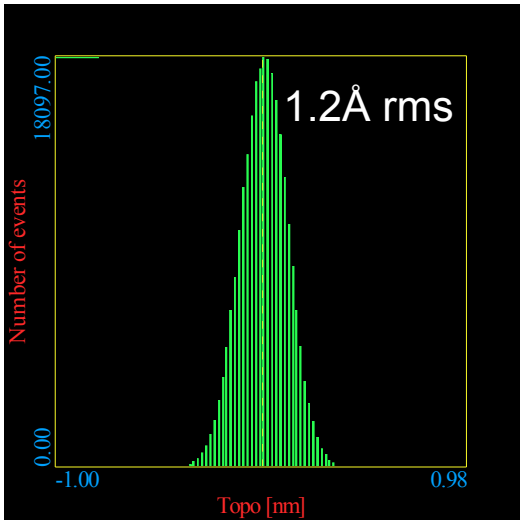
*courtesy Herman Godfried

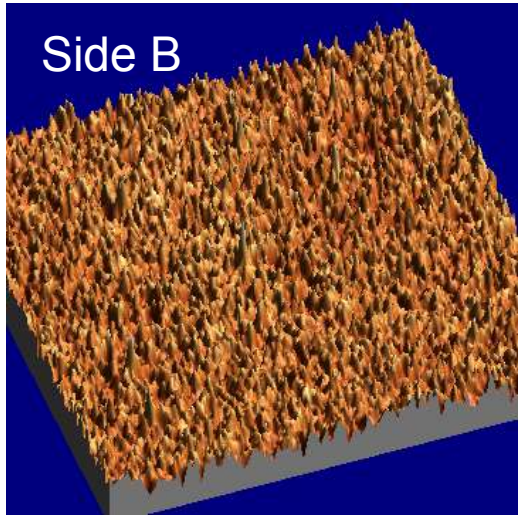
AFM ESRF

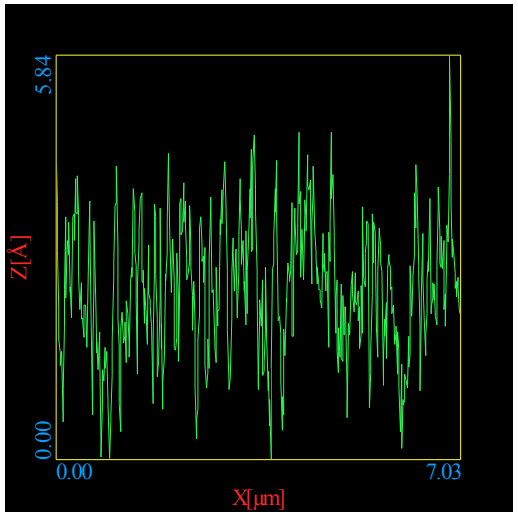
sample MDS1 (Mar05), thickness 350 µm, E6 resin wheel polish then scaife and superpolish by J Butler-NRL

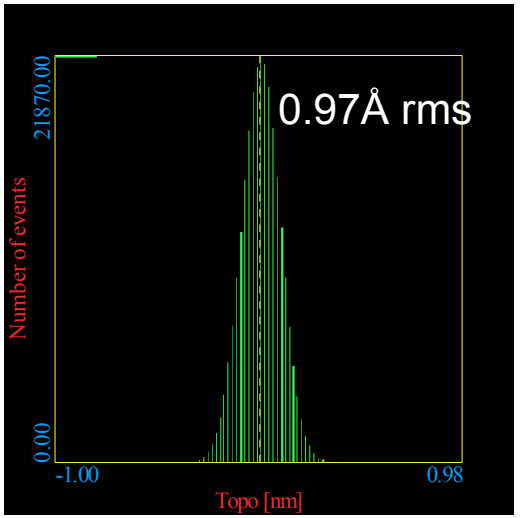








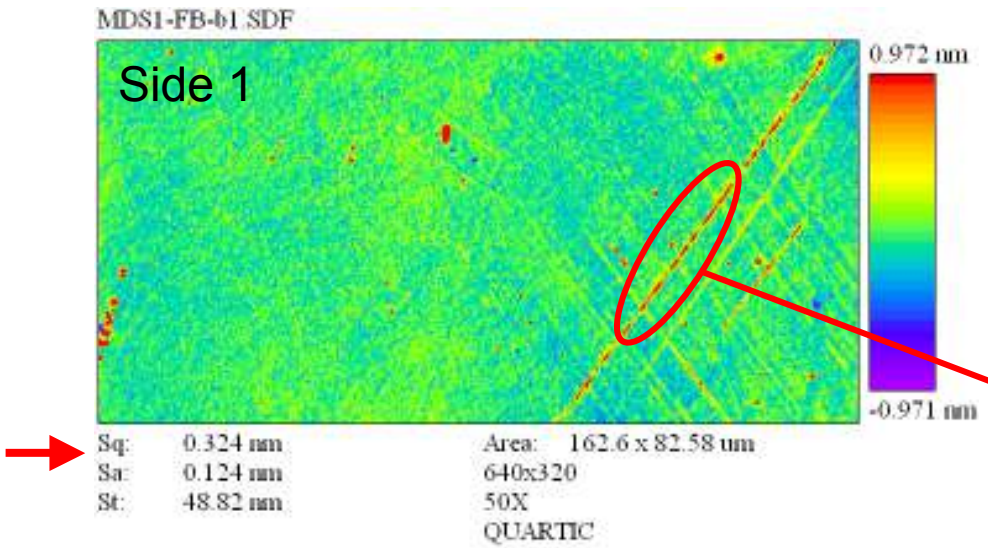


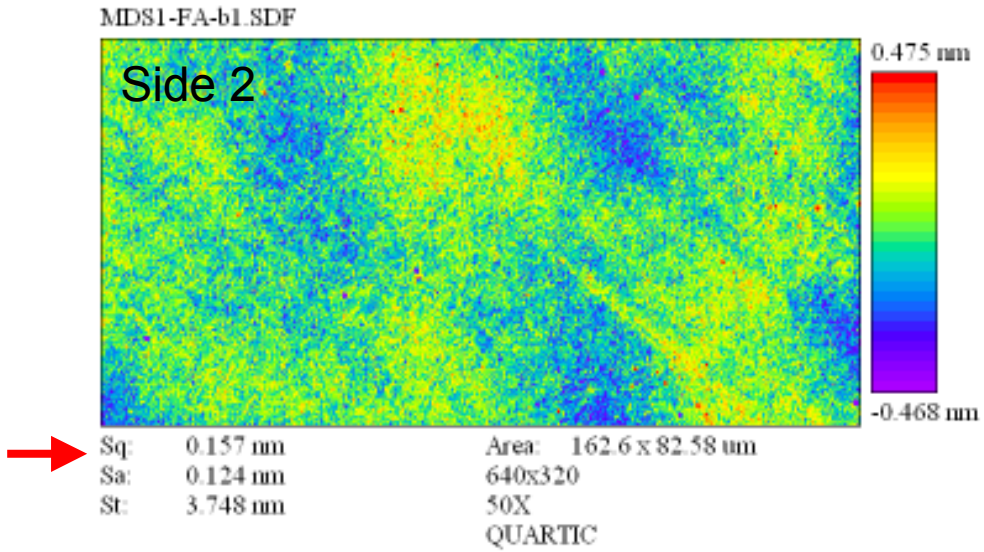


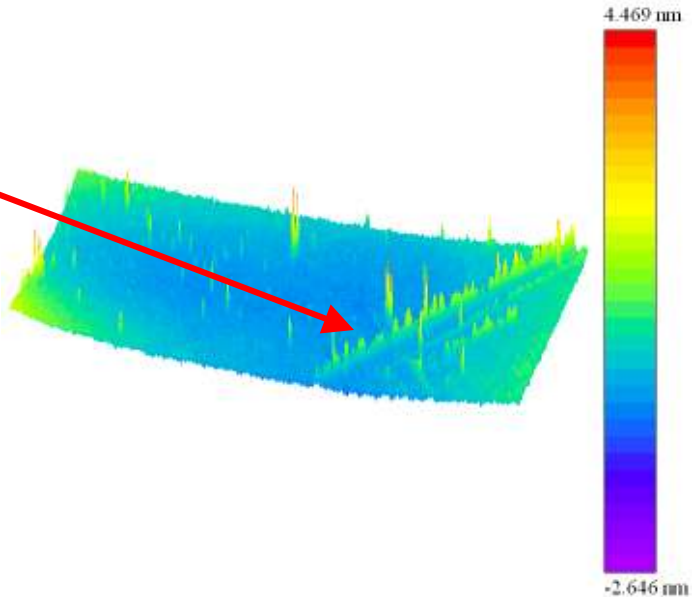
Laser Optical Interferometry

PROMAP 512 profilometer-ESRF

E6 sample MDS-1 (~ Mar05),
thickness ~350 µm,
resin wheel polish (E6 Ascot)
then *scaife and 'superpolish'* by
J Butler-NRL (~June 06)

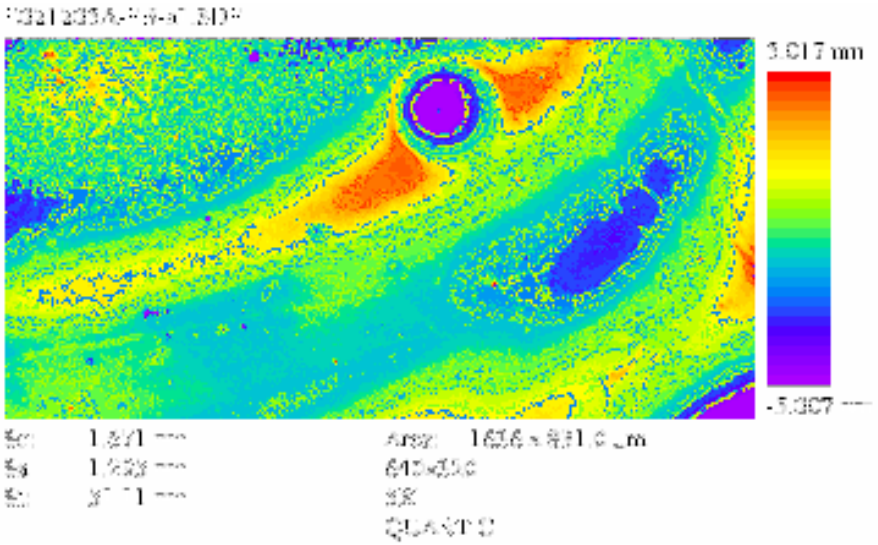




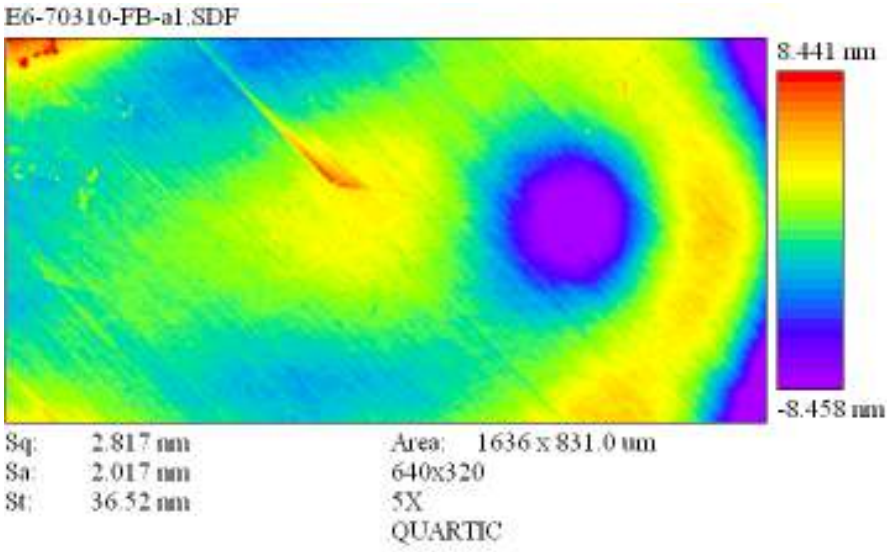


Laser Optical Interferometry

PROMAP 512 profilometer-ESRF



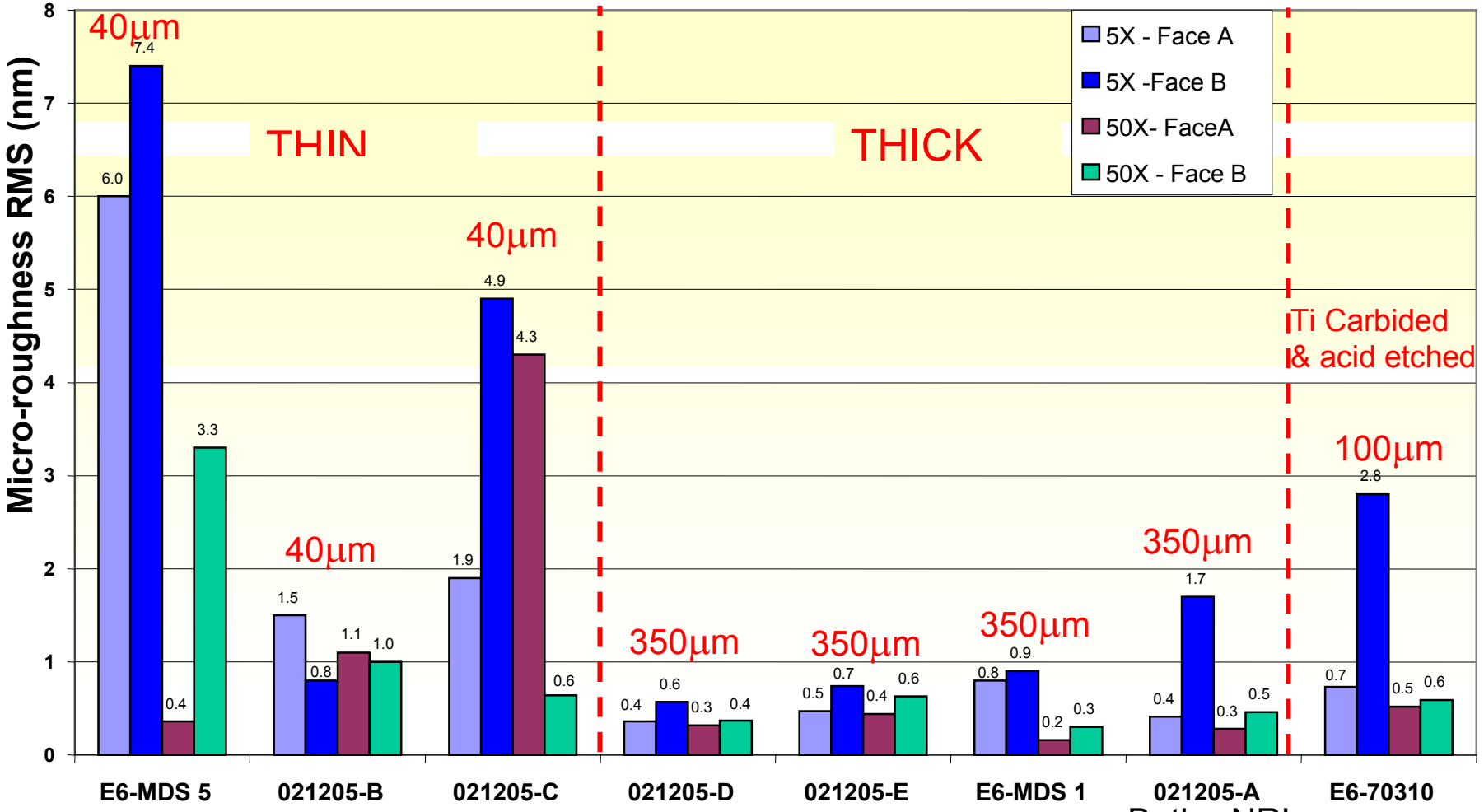
Element Six electronic grade CVD sample 02-12-05A, thickness ~350 µm
E6 resin wheel polish, then scaife and superpolish by J Butler-NRL



Element Six electronic grade CVD sample 70310 (~10-03), thickness 100µm
E6 scaife, metal carbided, stripped, then superpolished by J Butler-NRL

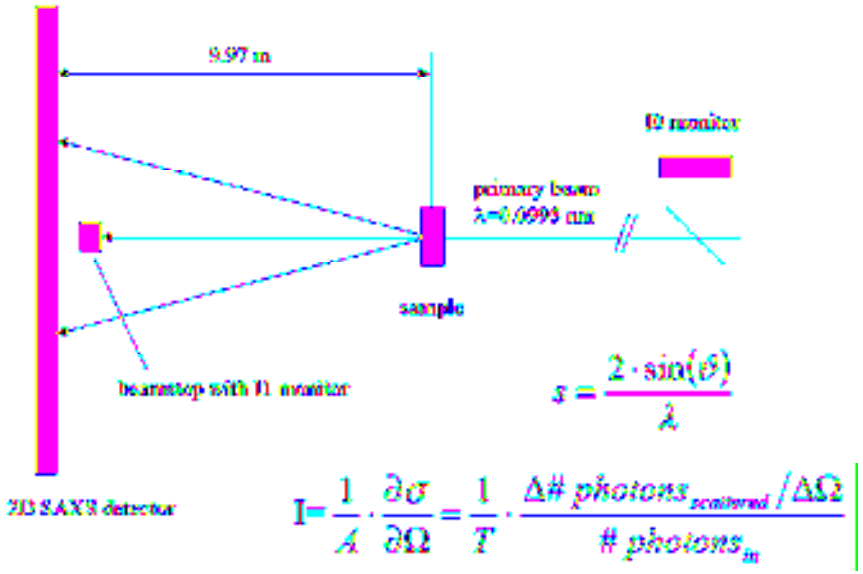
E6 CVD samples at ESRF:

summary of *optical interferometry*



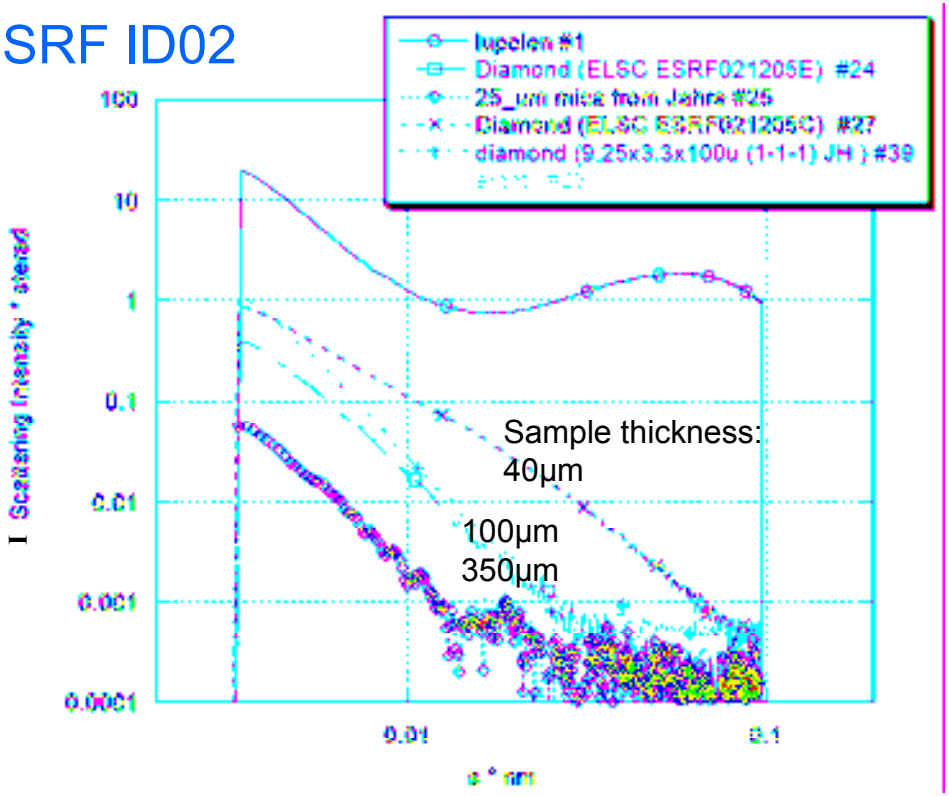
Courtesy A Rommevaux, ESRF

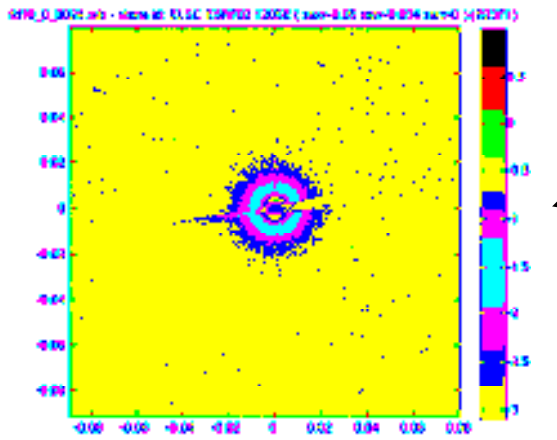
Small angle X-ray scattering at ESRF ID02



$$s = \frac{2 \cdot \sin(\theta)}{\lambda}$$

$$I = \frac{1}{A} \cdot \frac{\partial \sigma}{\partial \Omega} = \frac{1}{T} \cdot \frac{\Delta \#\ photons_{scattered} / \Delta\Omega}{\#\ photons_{in}}$$



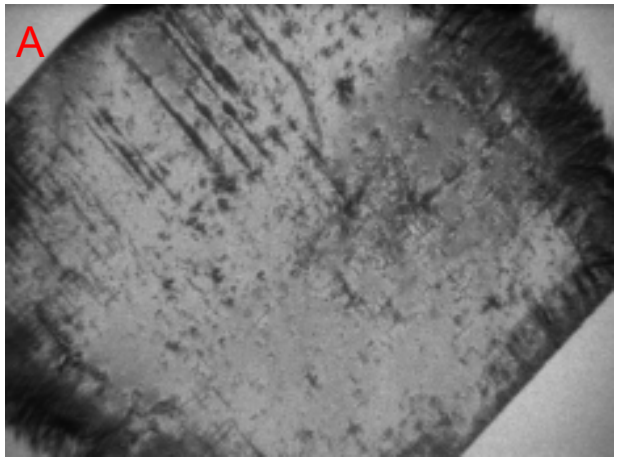


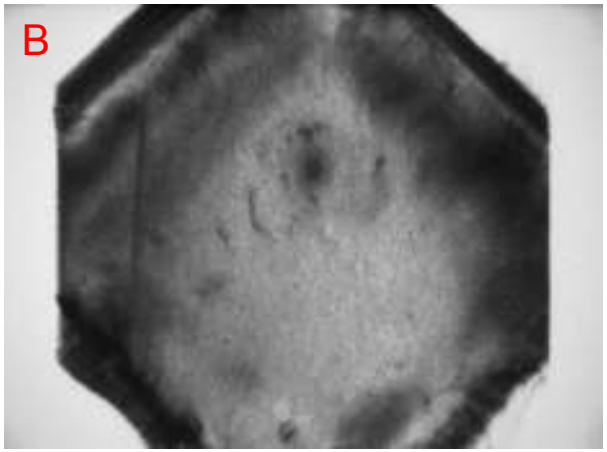
Azimuthally averaged scattering intenity I between α=120° to α=150°
i.e. outside the regions of slit scattering: data is sample-absent scattering subtracted.

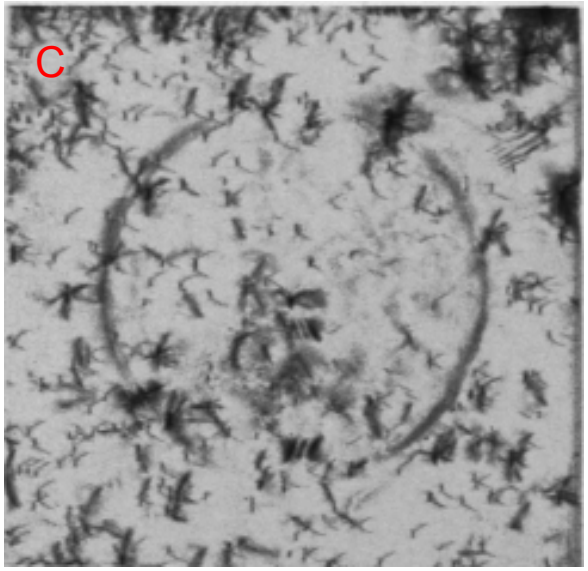
CVD diamond, electronic grade ELSC ESRF021205E	~350µm
CVD diamond, electronic grade ELSC ESRF021205C	40µm
JH sample HPHT diamond: 9.25x3.3x100µm (1-1-1)	100µm
Jahre Q41 mica	25µm
lupolen (calibration sample)	3300µm

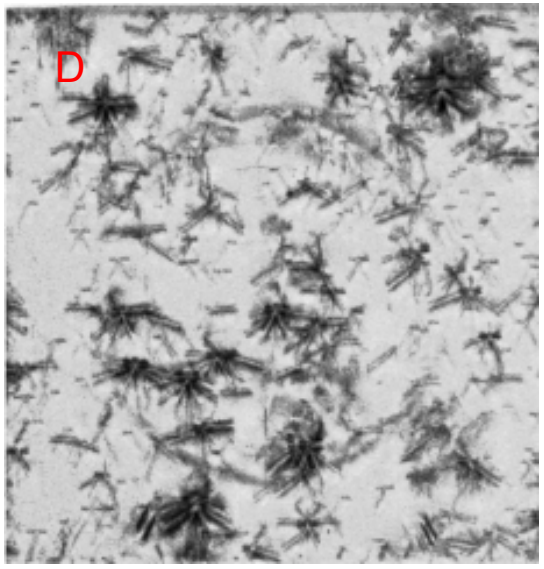
courtesy Boesecke, Panine May 06

Topography data, ESRF ID19









Topo courtesy J Haertwig,

(A) E6 Sample 70310, Laue

(B)

Apollo sample, Bragg

white beam

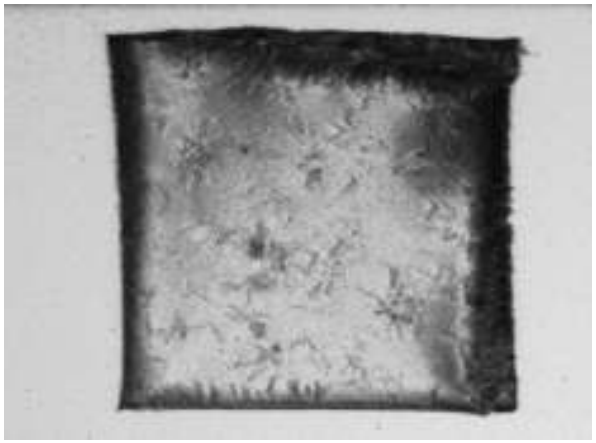
Dec 2003

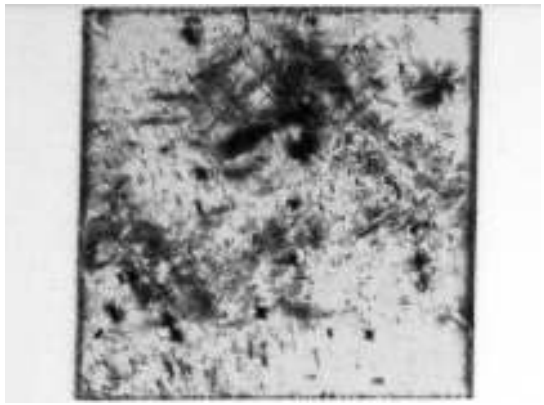
E6-Berdermann samples
C and D
Laue white beam

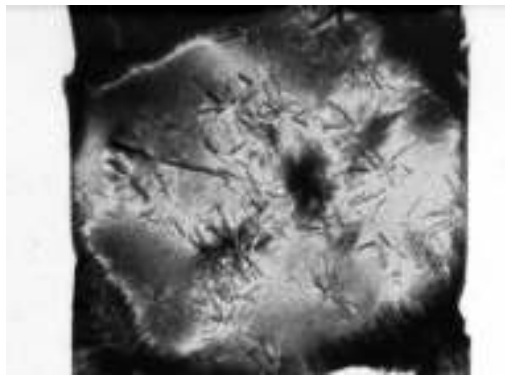
Oct 2004

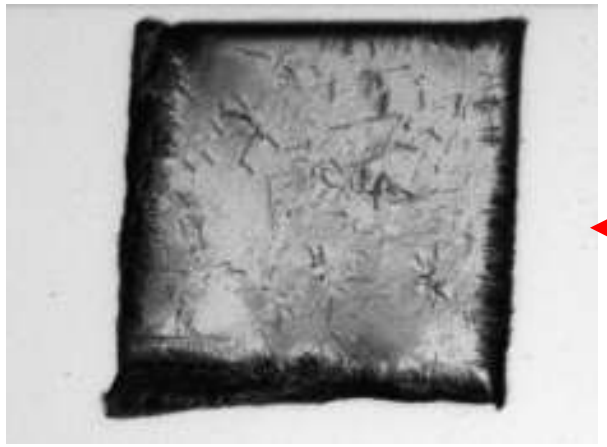
Topography data, E-6 ‘electronic grade’ CVD

Transmission (220 and 040) of E6 ‘electronic grade’ diamond plates MDS-1…5, ~3.6 x 3.6 x 0.35 mm^3 (May 2005)





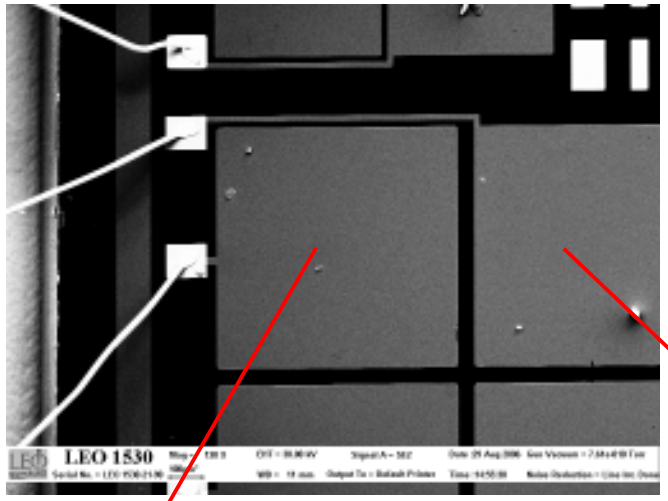




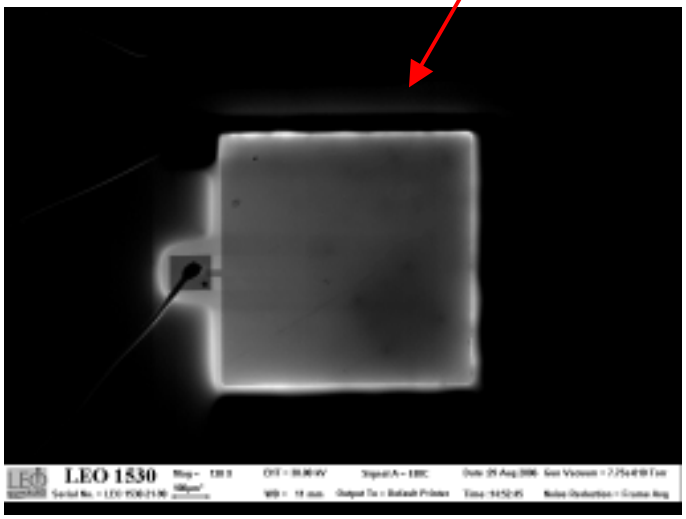
Same batch !

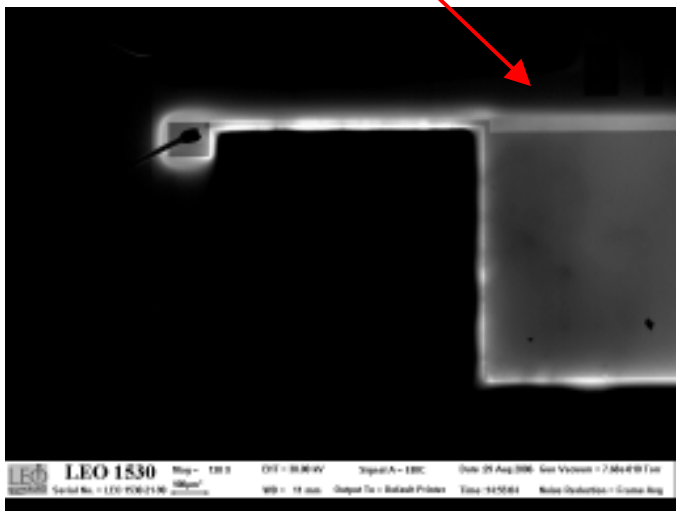
EBIC measurements:

E6 sample, Ni-TiC electrodes



Work just begun!
29 Aug 2006





1. Synchrotron X-ray beam monitoring requirements, and why single crystal diamond?

2. Diamond X-BPM tests at the ESRF ID21 X-ray Microscopy Beamline

3. CVD device ‘XBIC’ mapping tests at ESRF-ID21

Diamond ‘XBIC’ mapping at ESRF-ID21

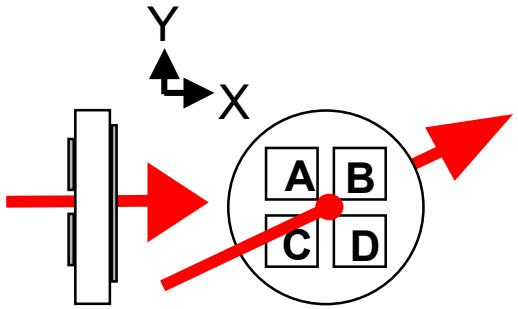
thin plate crystal sample with ‘X-ray transparent’ metal electrode contacts
Ti, Mo, …Ni, Al
diamond bulk acts as solid state ‘ionization chamber’
signal readout ‘DC’ or RF (synchrotron bunch clock frequency 352MHz)

Beam Monitoring application: position and intensity

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

→ beam 'centre' by weighting four electrode currents A, B, C, D

→ Sum currents ~ beam intensity (ε_D = ??)

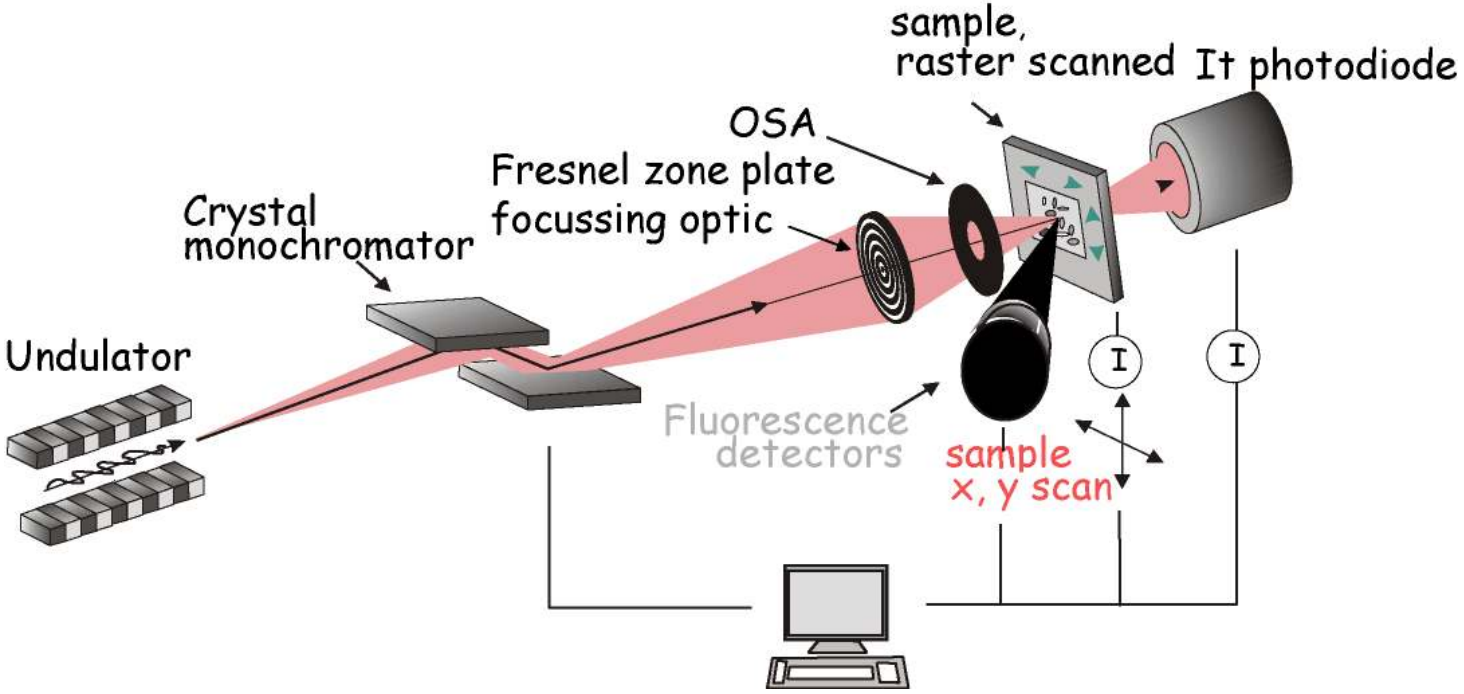


$$X = \frac{(A+C)-(B+D)}{A+B+C+D}$$

$$Y = \frac{(A+B)-(C+D)}{A+B+C+D}$$

ESRF-ID21 X-ray Microscopy Beamline

mapping of contacted CVD diamonds:



ESRF beam 50ps pulses at 3nsec-3μs intervals,
energy (ID21) 2 … 7.5 keV

Fresnel zone plate focuses the X-beam to a *sub-micron* probe. Unwanted diffraction orders from the zone plate are removed by a central stop and an order selecting aperture (OSA).

→ surface or bulk charge injection

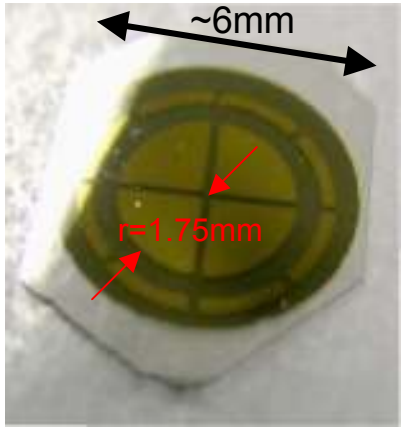
Single crystal quadrant X-ray mapping (XBIC)

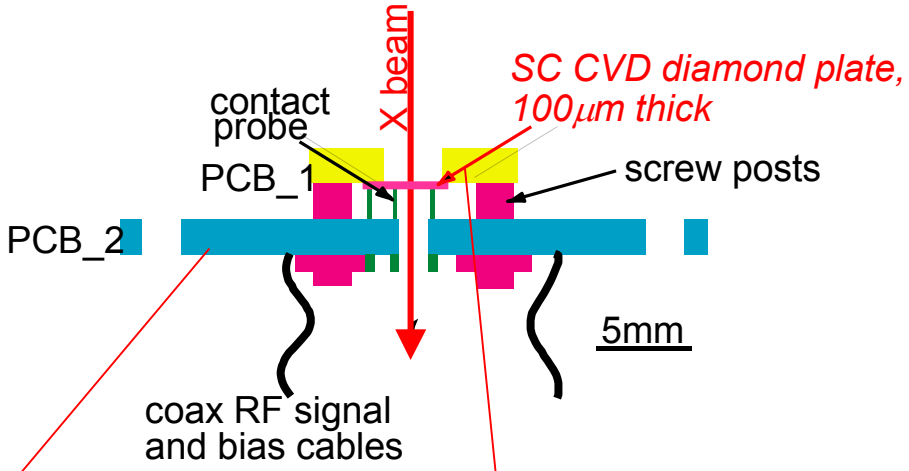
ESRF ID21 (May 2005)

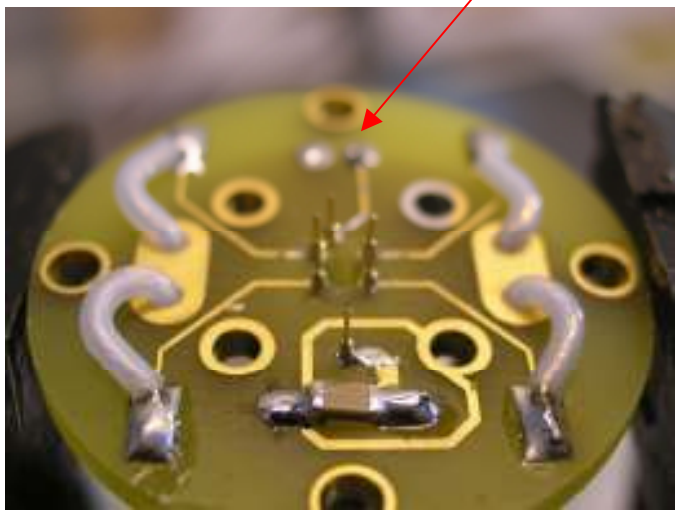
Element Six 'electronic grade' sample 70310 *thickness 100μm*

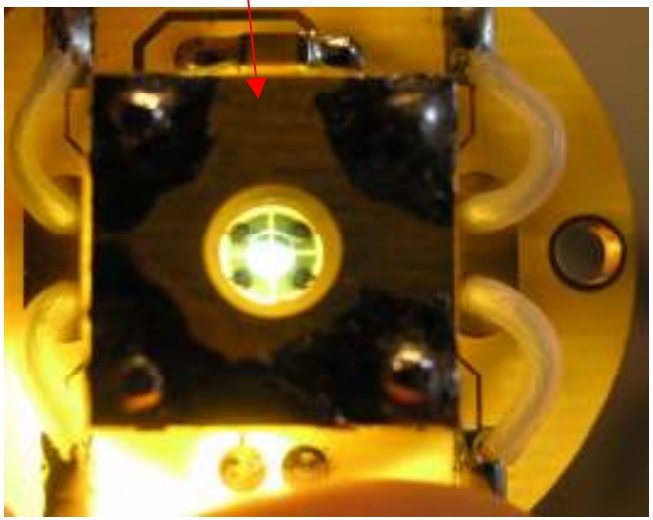
20nm+20nm sputtered Cr, Au contacts [GSI-Darmstadt Target Lab'.]

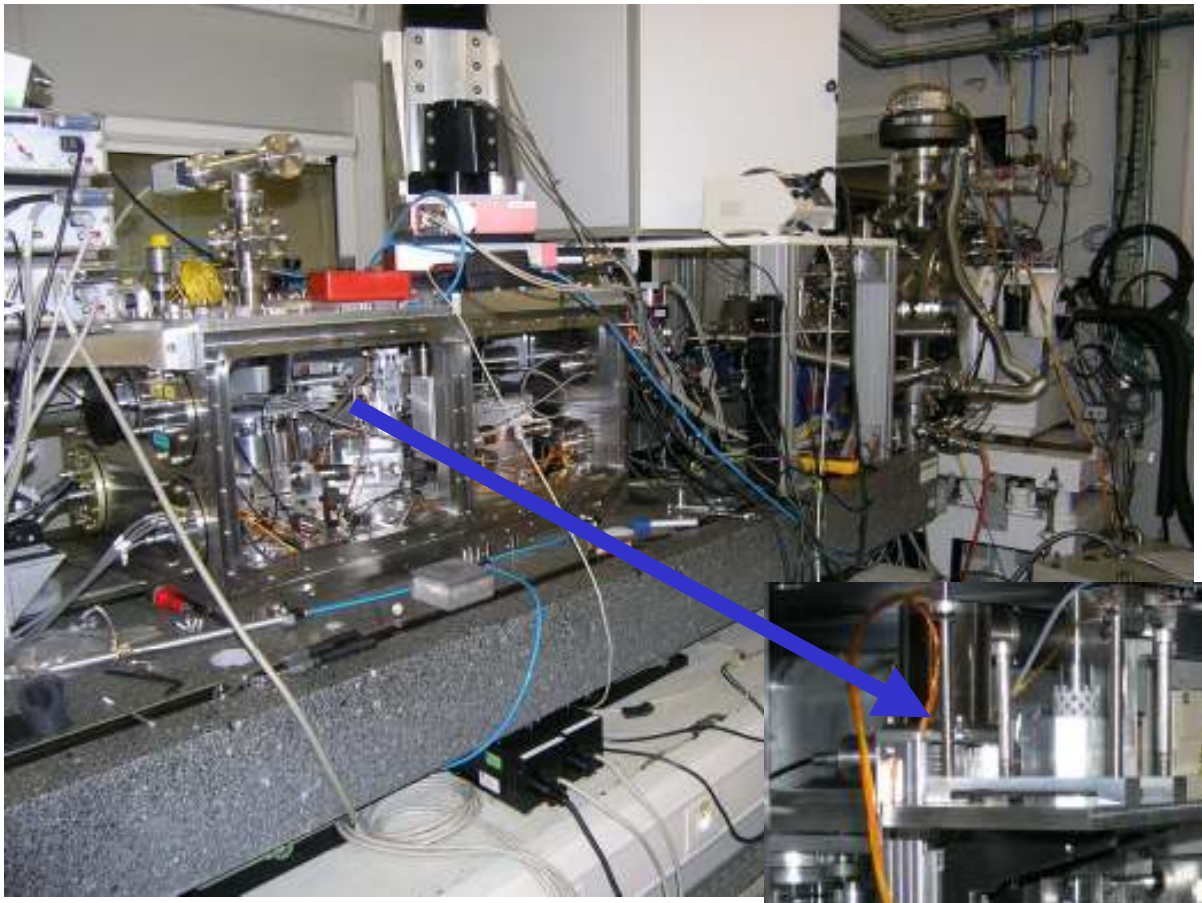
PCB sandwich assembly with sprung microprobes



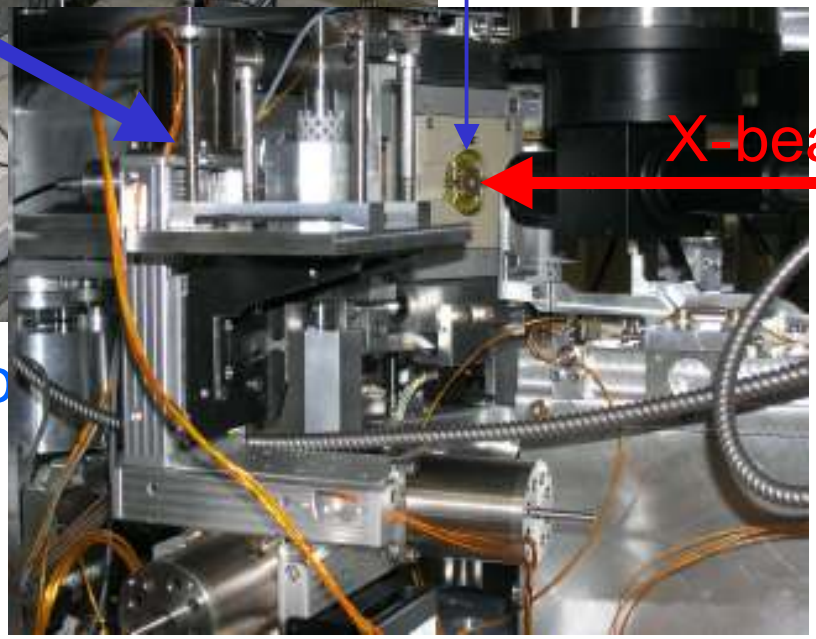








Diamond on submicron
x-y scanning stage

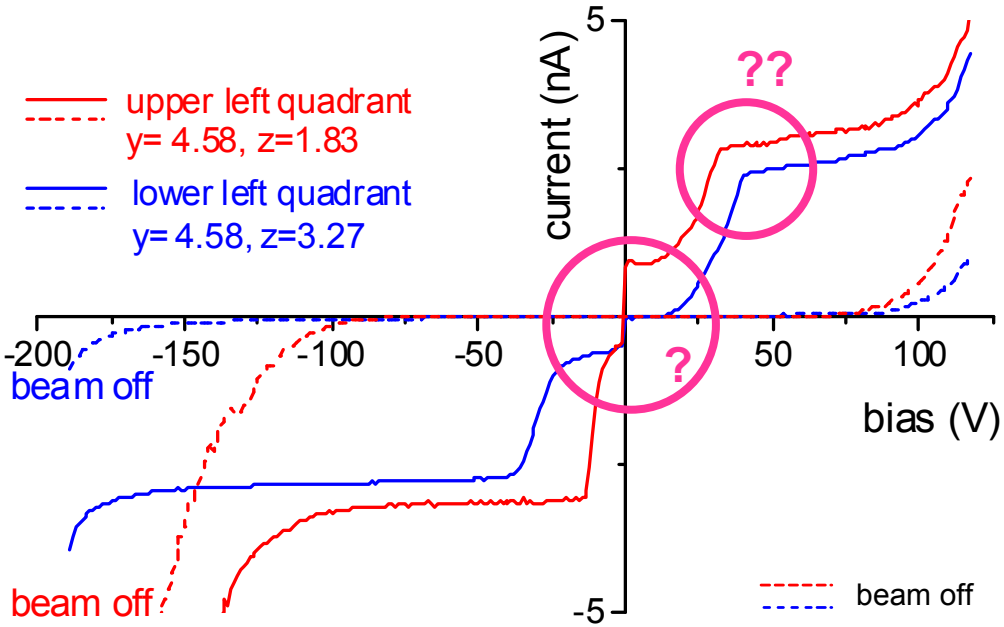


ESRF-ID21 X-ray Microscop

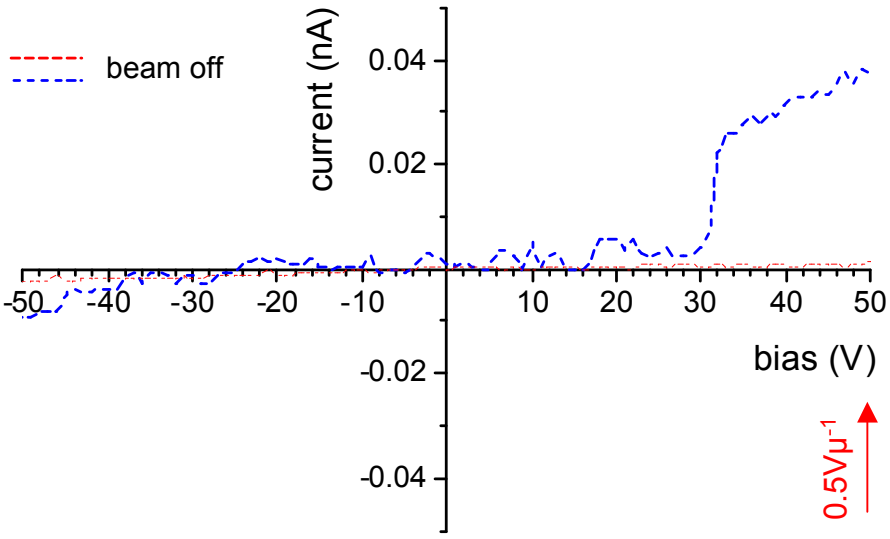
focus 1.2 x 0.4μ^2 FWHM,
~5x10^7 photos/sec at 7.2keV

I-V curves

Cr-Au contacted 100mm plate with/without beam

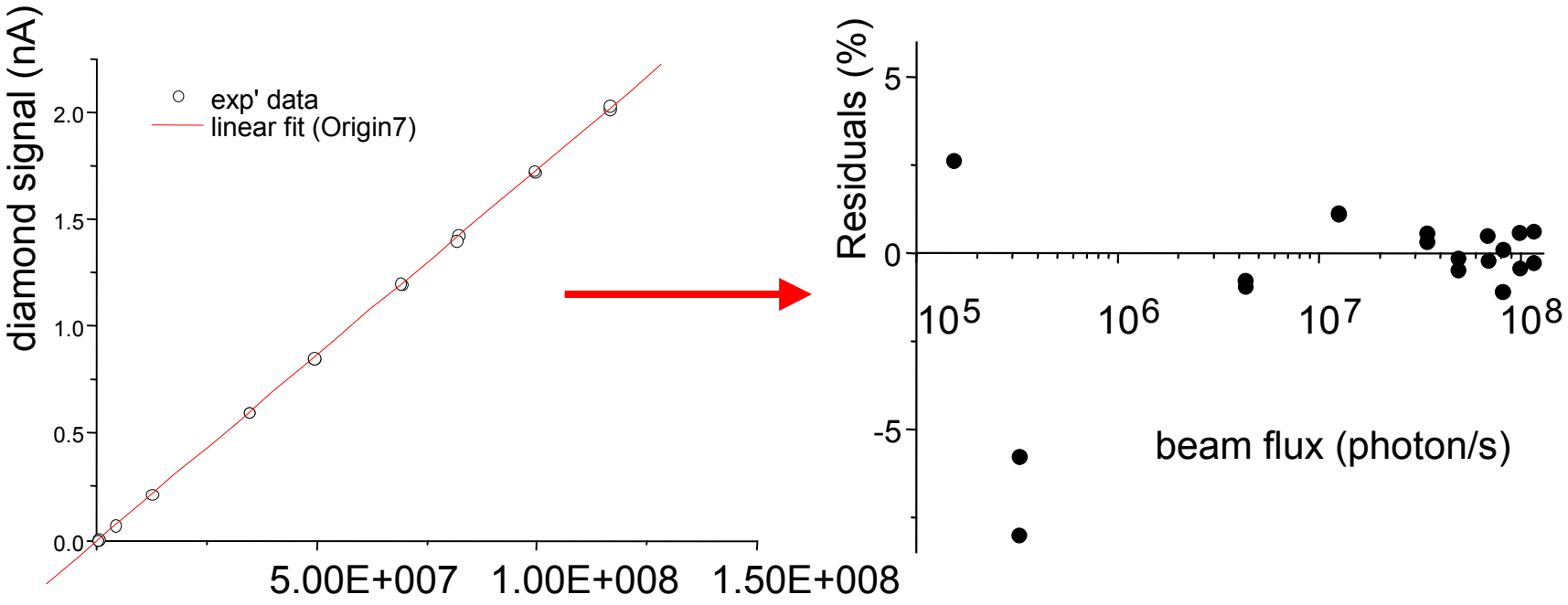


Detail of leakage currents (electrodes 2.5mm²)



beam focussed 1 x 0.4μ², absorbed ~2x10⁷ photos/sec at 7.2keV

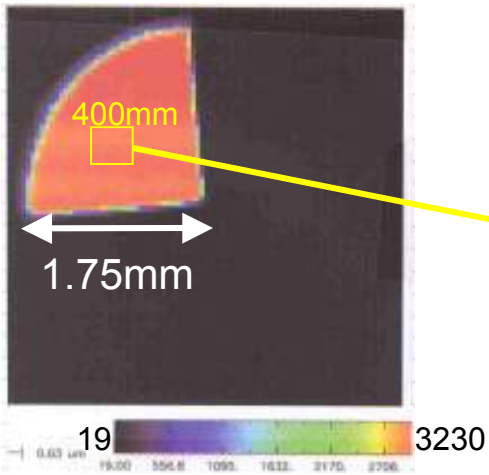
Signal linearity with beam intensity

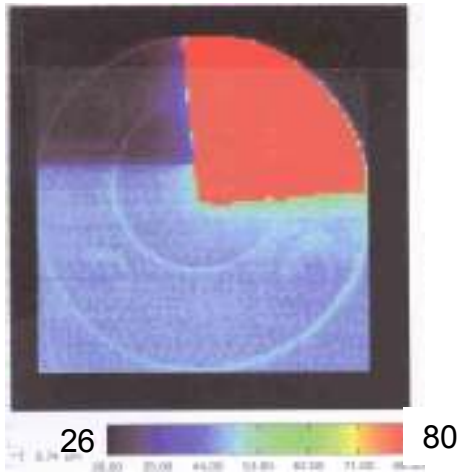


Incident beam flux (X-ray/sec in 0.4 x 1.2 μm^2 fwhm spot)

Quadrant isolation and uniformity of response

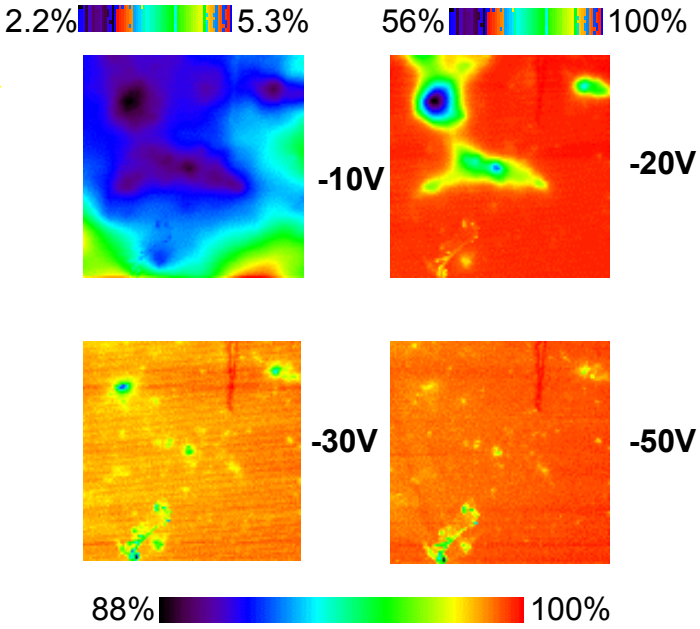
X-ray response ('dc' signal current) of individual quadrant electrodes as microbeam is raster-mapped over entire surface





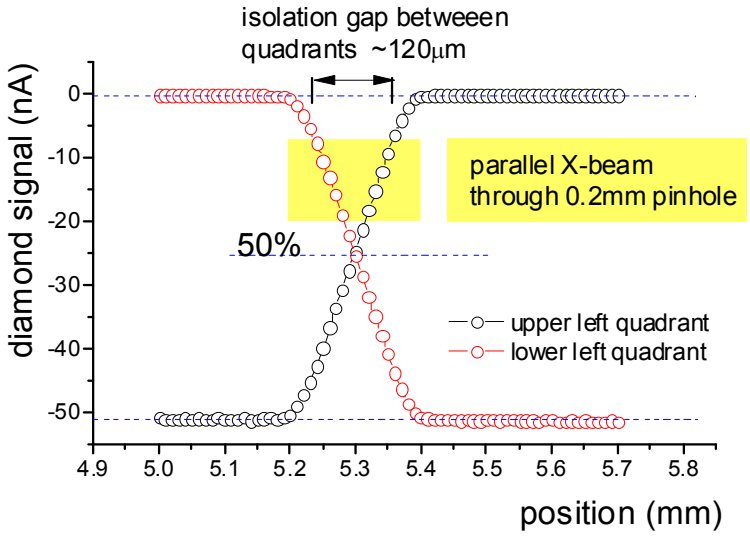
1 x 0.4μ², ~10⁸ photos/sec at 7.2 keV

effect of operating bias

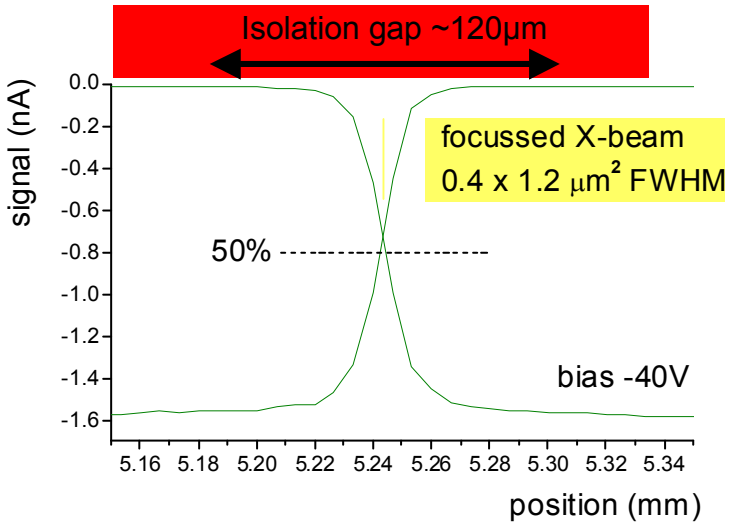


Charge collection →100% for bias >50V
localized surface (?) defects remain

Cr-Au contacted quadrant device: ‘dc’ measured position response



signal slope ~0.5% /micron



signal slope ~5% /micron

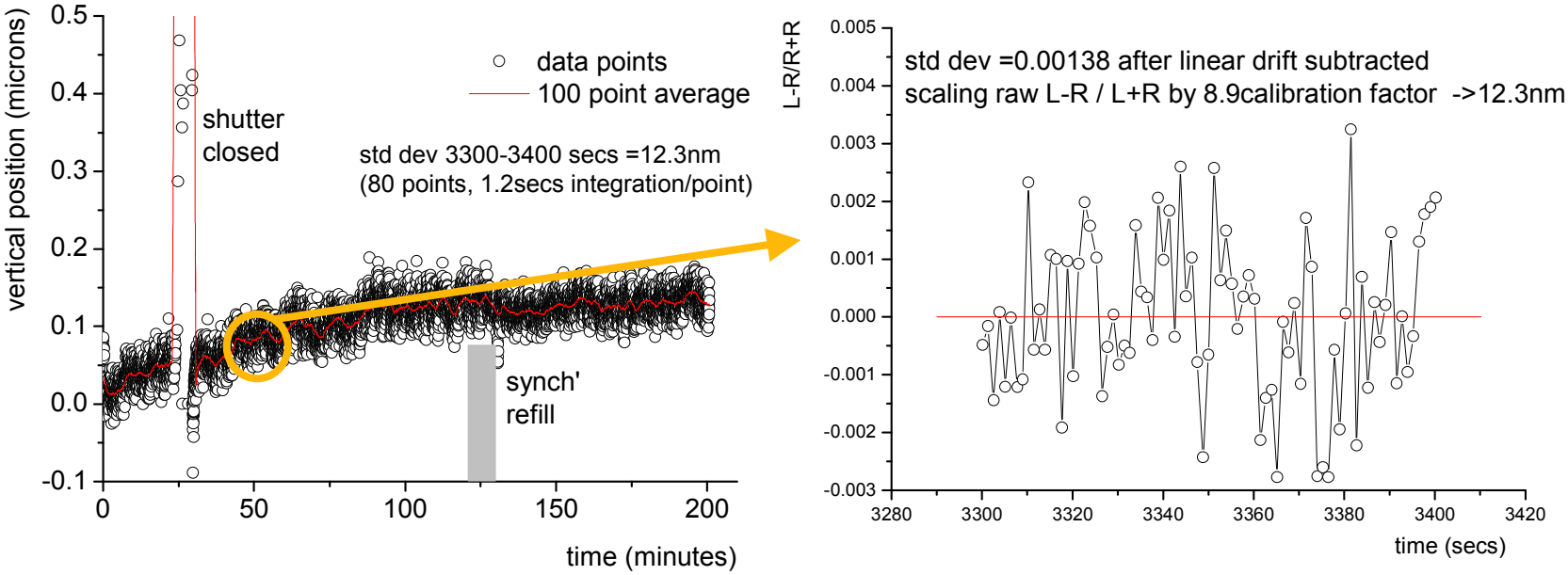
For large beam (> 50μm), device ‘crossover response’ is ~ line integral across the beam intensity profile

For small beam (< 10μm), crossover response is ~ convolution of photoelectron thermalization range and lateral charge diffusion ocurring during drift)…

for current integration mode!

‘dc’ measured position of beam: time scan at ID21

Cr-Au contacted quadrant, Oct 2005



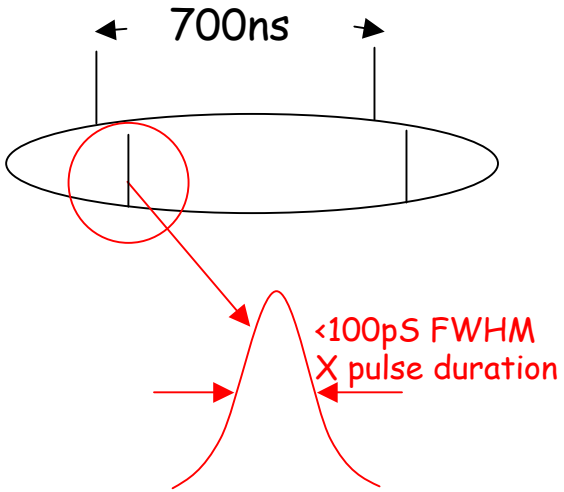
standard deviation 12.3nm calculated for 100 successive data points
(1sec V/F integrations at 1.2 sec intervals, linear drift term only subtracted) .

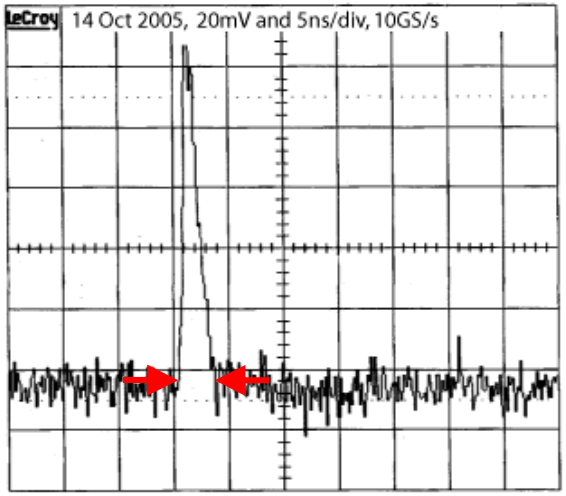
Contribution from electrometer + V/F noise is ~1.1nm
(inferred from observed, beam-off V/F count noise during shutter closed period).

.

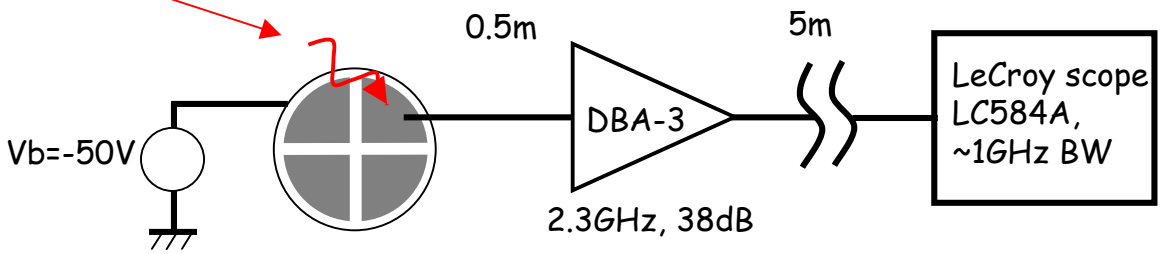
Diamond signal time response ‘single bunch’

ESRF synchrotron in
4 bunch mode





quadrant E70310, -50V bias 200um pinhole after 38db gain (DBAIII)

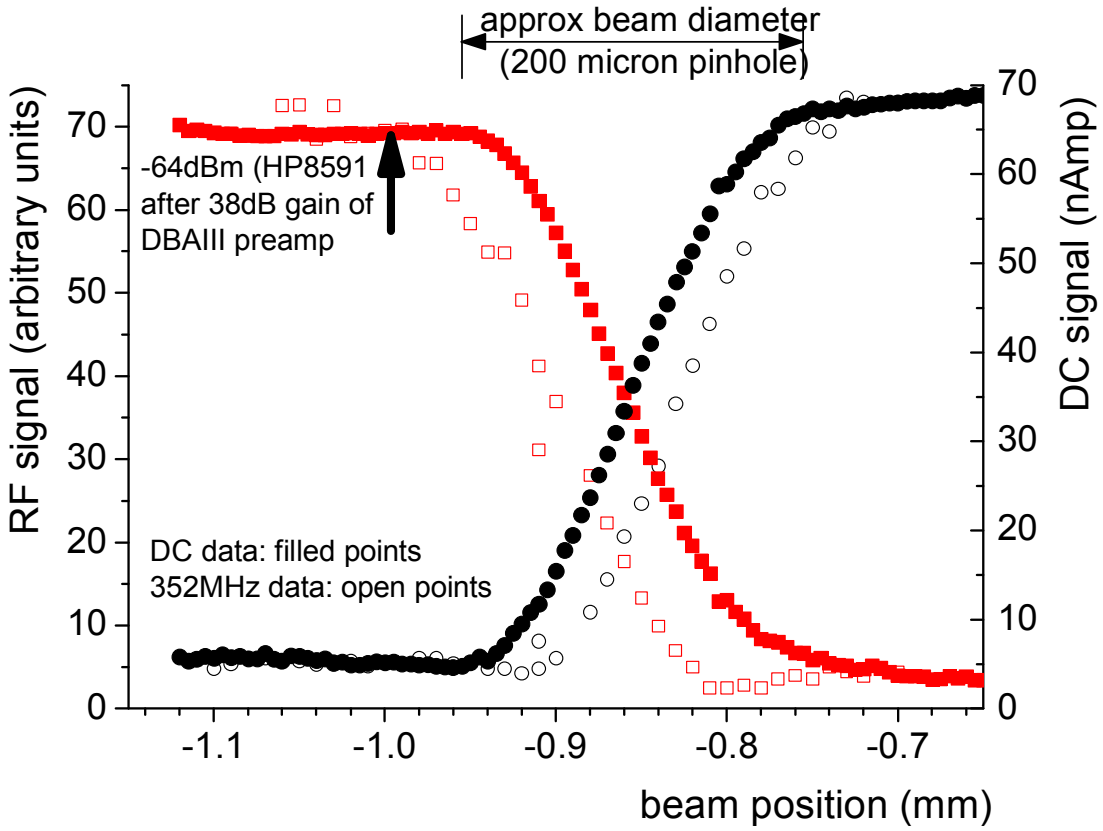


Signal response to crossing of *one* X-ray bunch

= absorption of ~400 photons at 7.2keV (~3MeV)

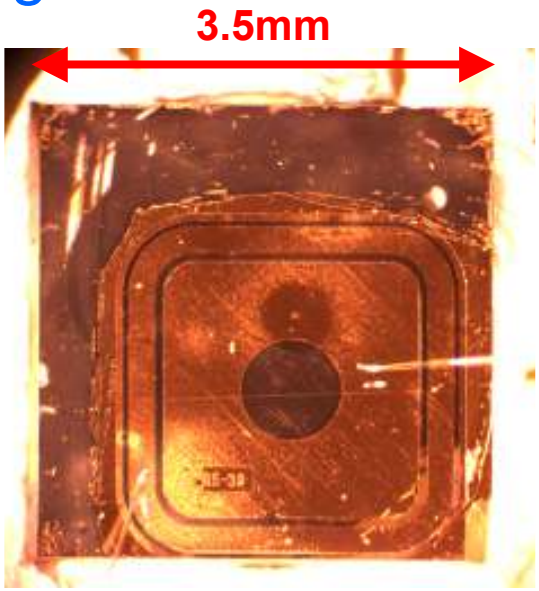
Linear fit to slope gives signal full base width 2.46ns, --> e- **drift velocity ~41µm/ns**

Comparison of RF and ‘dc’ electrometer position response



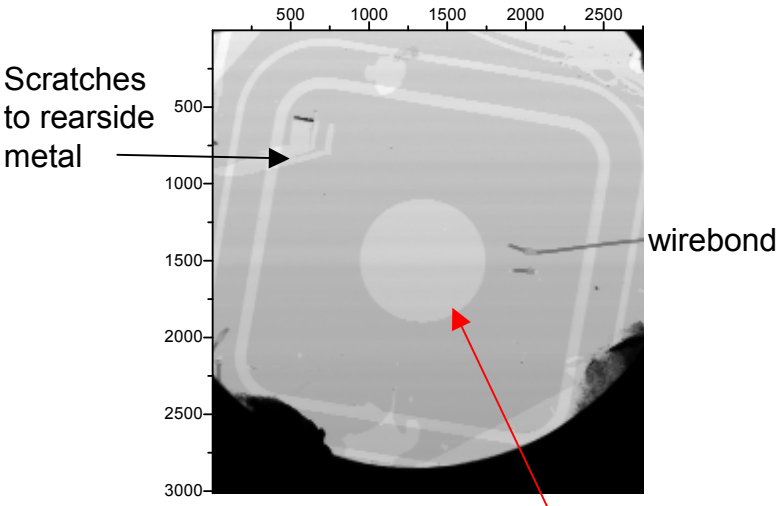
Beam pulse every ~730ns (1.37MHz), HP8591 **signal analyzer at 352MHz** with ~1kHz BW: measuring pulse signal power in *8th harmonic.*

Glasgow contacted device: X-ray response maps

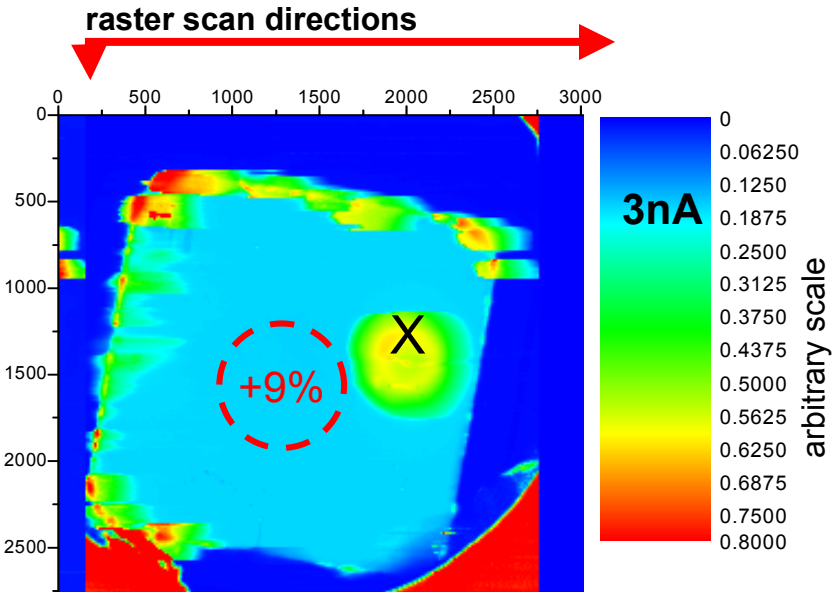


Contacts:
front Ni/Pd/Au 10/30/130nm
rear Ti(annealed)/Pd/Au 10/30/130nm

Signal current map:
response to scanning <1μm fwhm
X-beam at 12.5μm steps
'hot spot' 'X' under wire bond



Transmission 'radiograph'



(normalized to transmitted flux)

Central area Φ0.8mm
Ni/Au 5nm/5nm

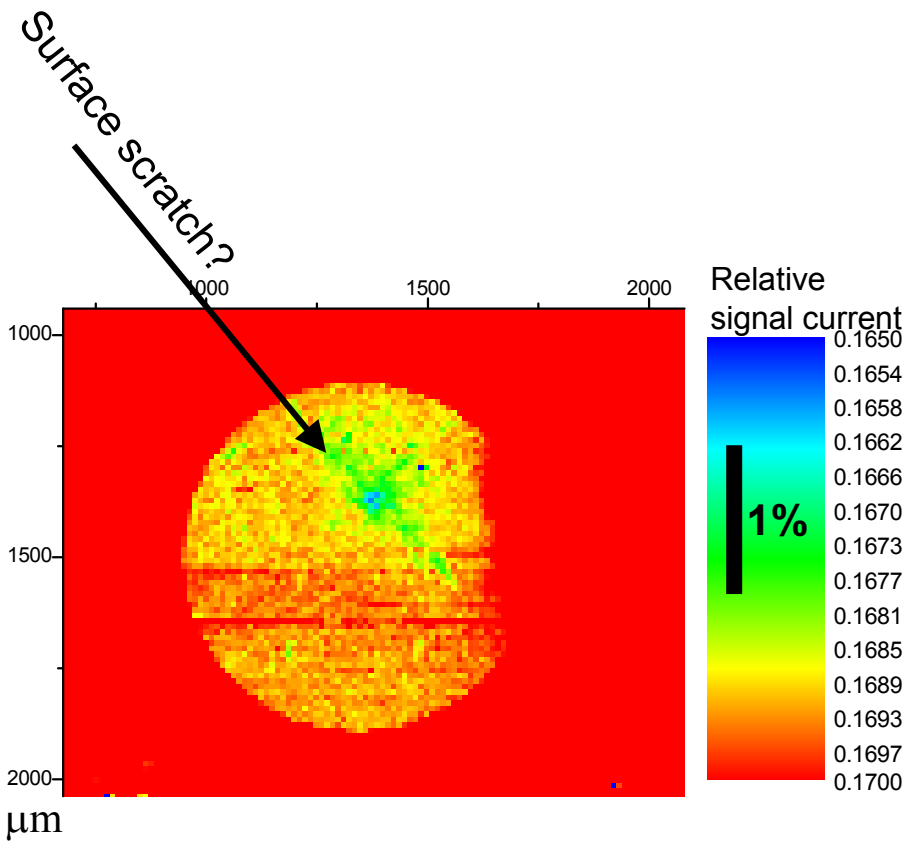
Glasgow Ni-TiC contacted device:

X-ray response uniformity

Thinned electrode central area
Φ0.8mm 5nm Ni / 5nm Au

response is ‘flat’ with 0.18% std. dev.

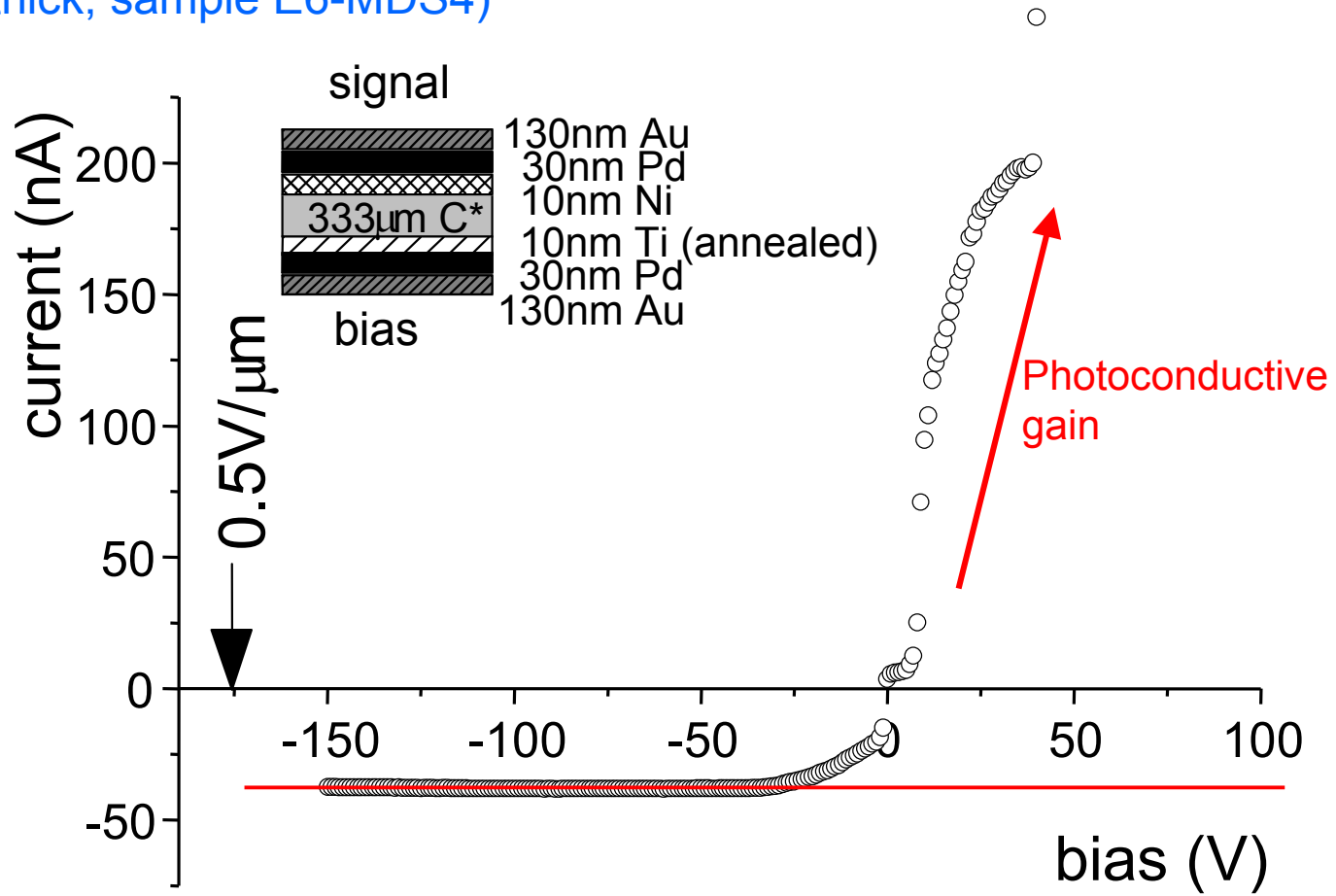
calculated for un-normalized data,
15 x 15 points at 12.5um steps,
<1μm² beam probe at 7.2keV



above map, data normalized to transmitted beam intensity
(to remove ‘top-bottom’ synchrotron beam decay artifact)

Glasgow contacted device

(333μm thick, sample E6-MDS4)



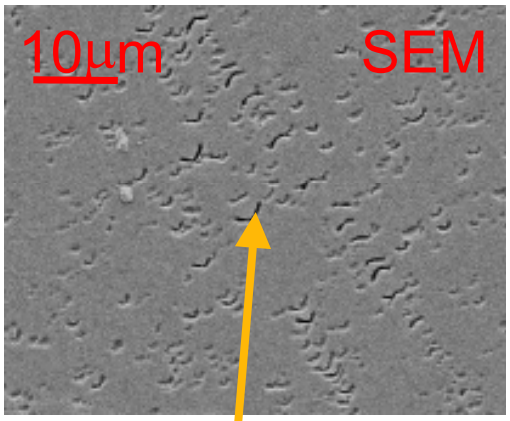
I-V curve *in X-ray beam* (ID21, pinhole beam Ø100μm, ~10^8ph/sec at 7.2keV).

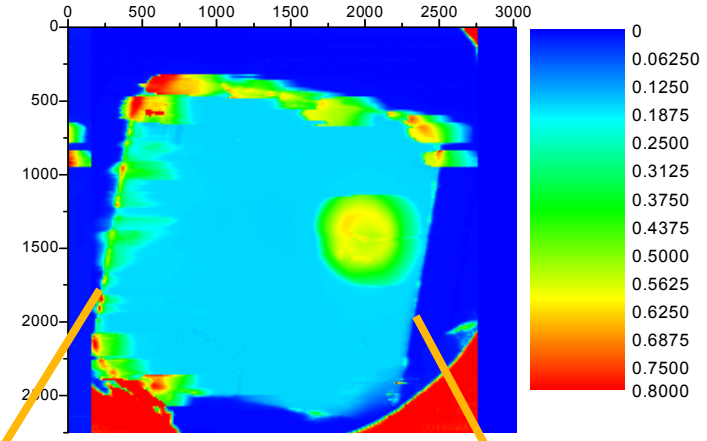
leakage current is ~pA level for +/-150V bias.

Si calibration → ε_D = 15.1eV / e-h pair

Surface damage and contact edge hot defects

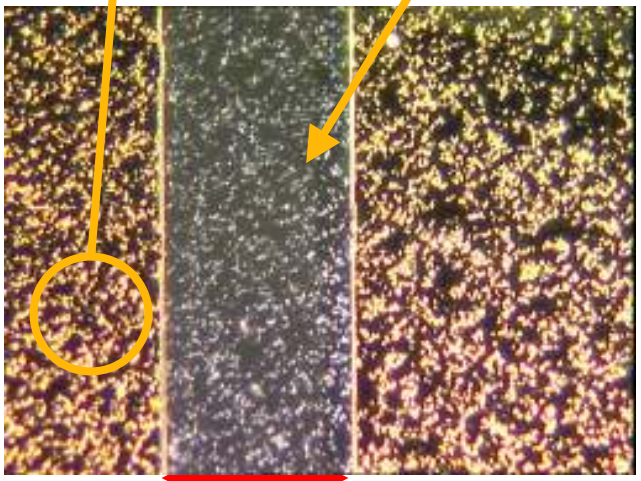
Resin wheel polish ‘tadpoles’





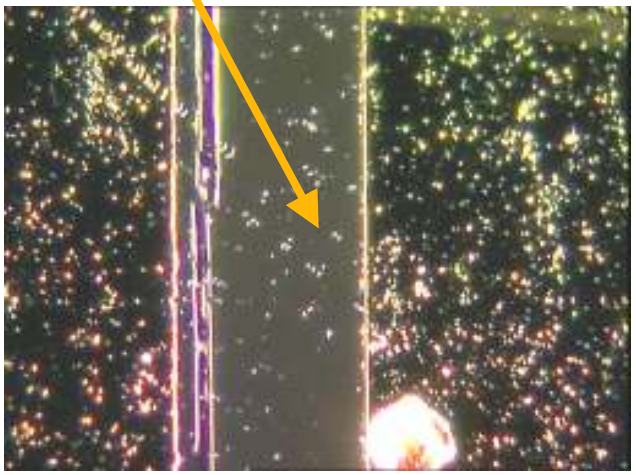
left

50x reflect’ microscope



Guard ring

electrode

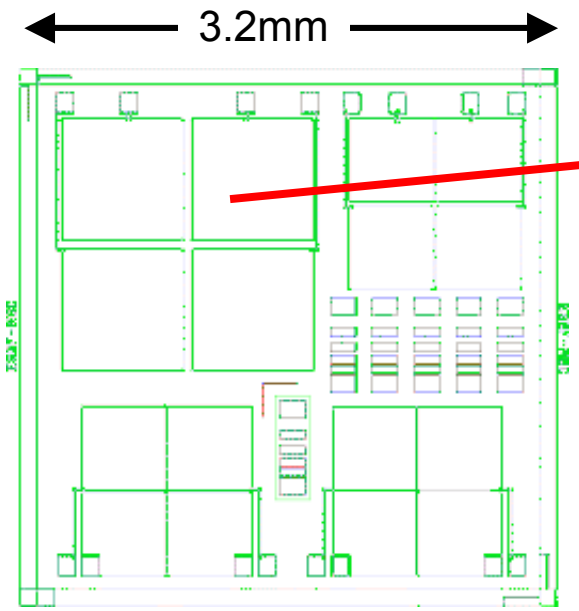


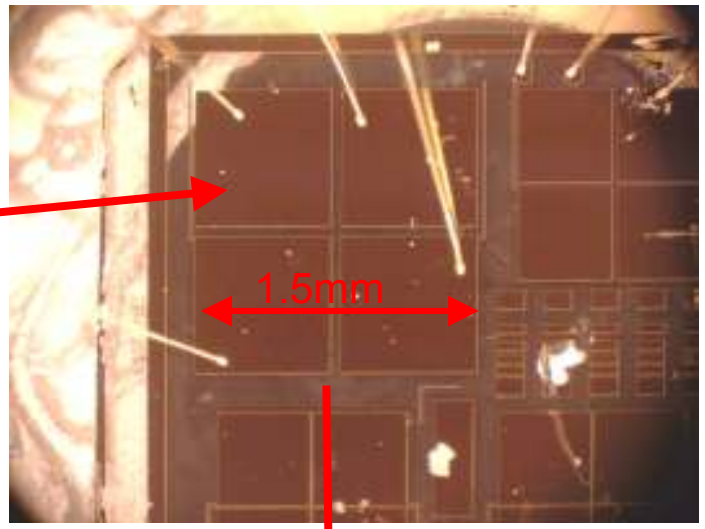
right

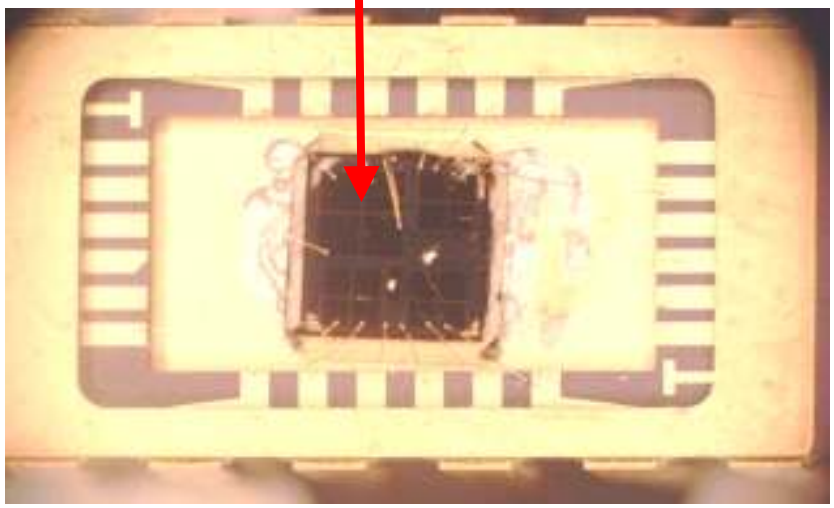
Guard ring

electrode

Beam XBIC tests DC and RF at ID21 (20/21 July 06)







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

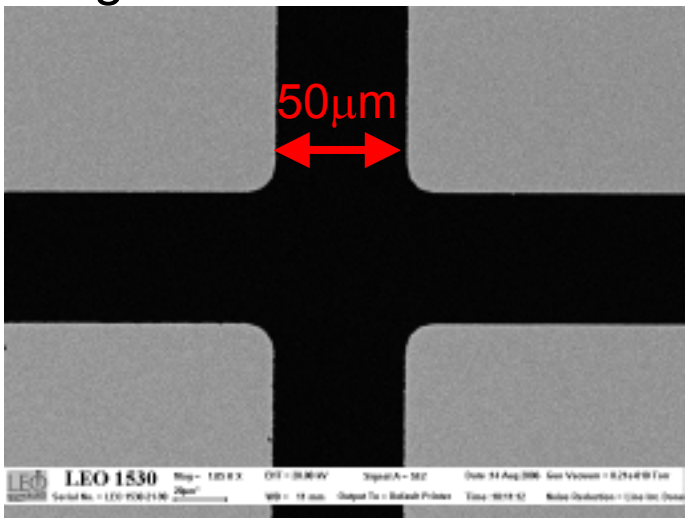
50, 20, 10 and 5μm quadrant isolation gaps

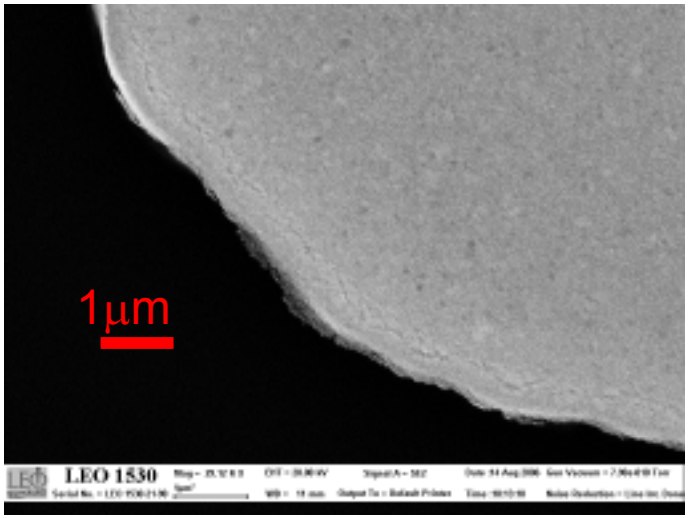
E6 MDS2 & MDS3 samples, Achard-CNRS ATJ30 sample

Lift-off lithography

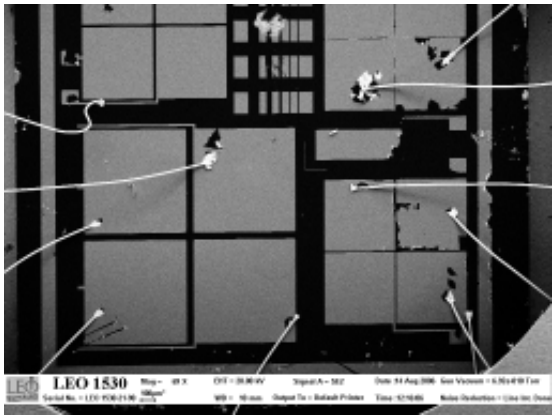
Ni/Pt/Au on ATJ30 scaife and MDS2 &3 resin wheel polished samples (July 2006)

the good…

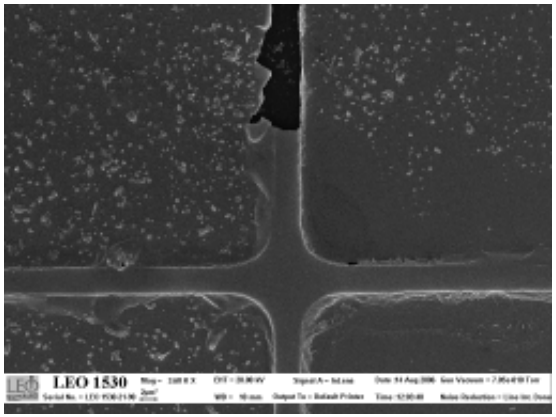


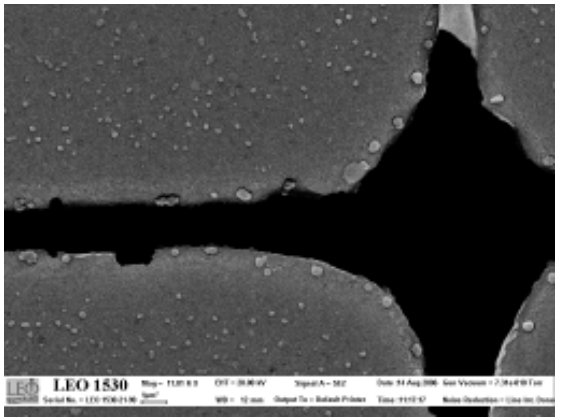


…the bad…



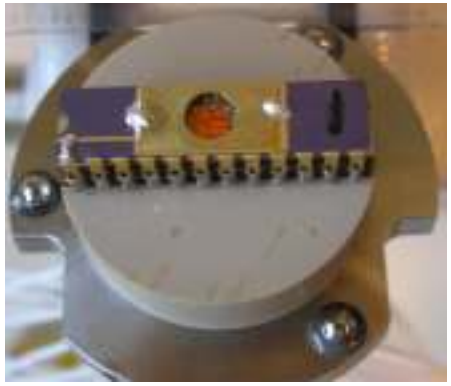
…and the ugly

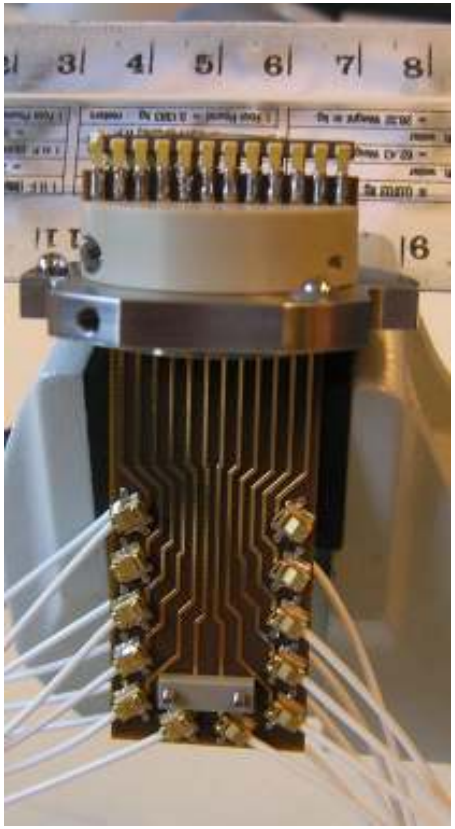




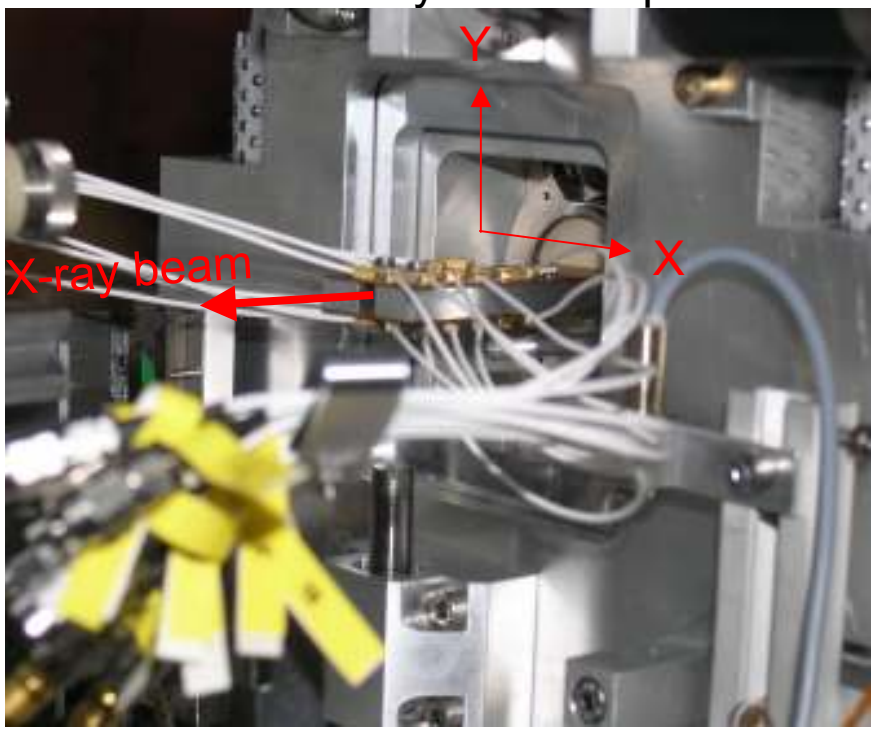
Sample mounting ID21

MDS-2, 3 and ATJ30 samples (July 06)





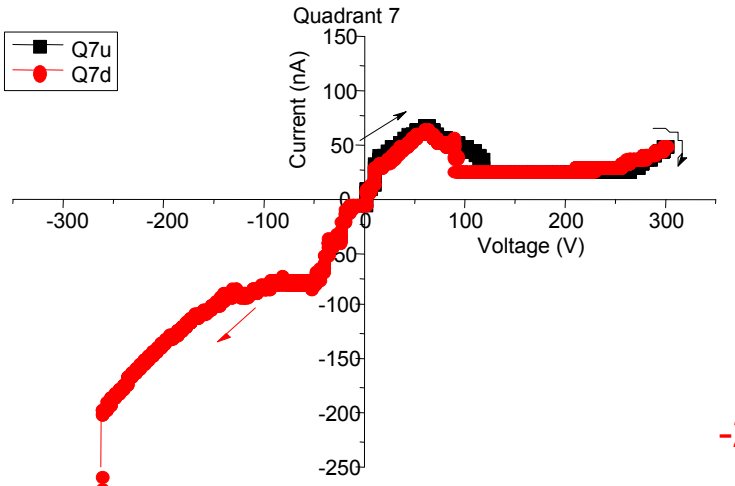
In situ on ID21 X-ray microscope

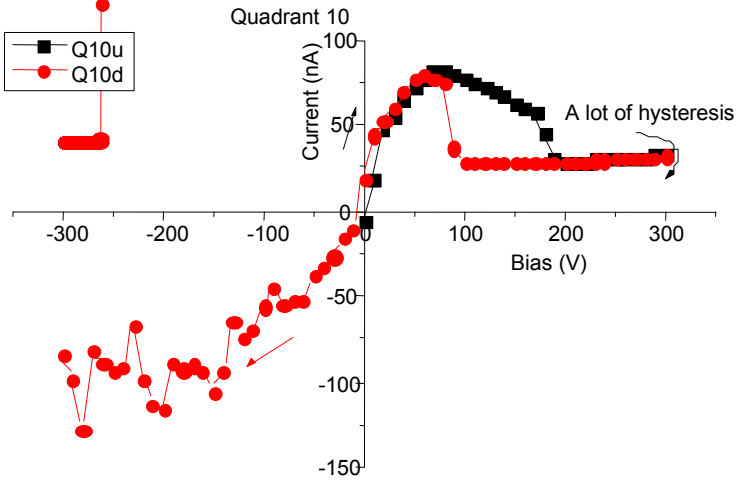


E6 Sample MDS-2, I-V curves

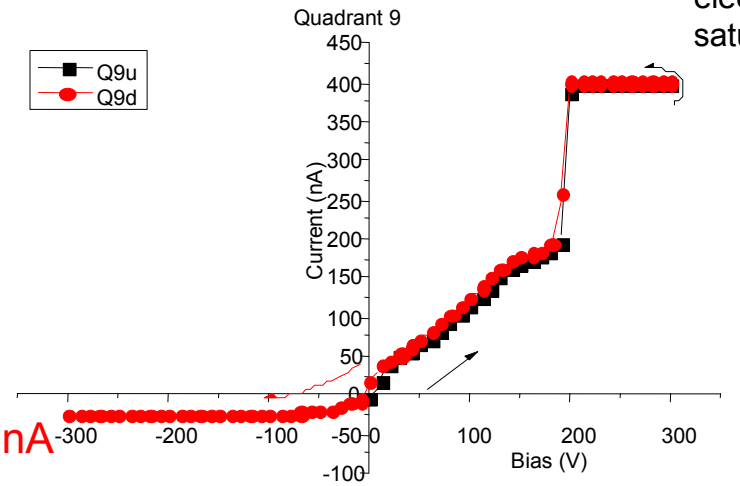
X-ray beam on

Ni-TiC contacts, *poor quality*
Sample thickness ~350μm

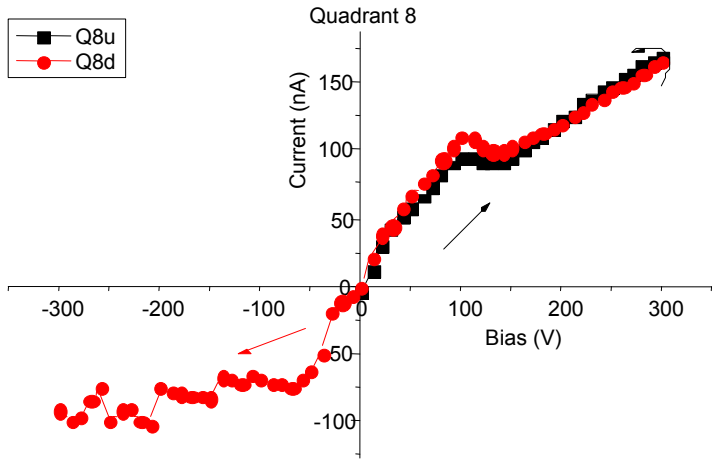




electrometer saturated



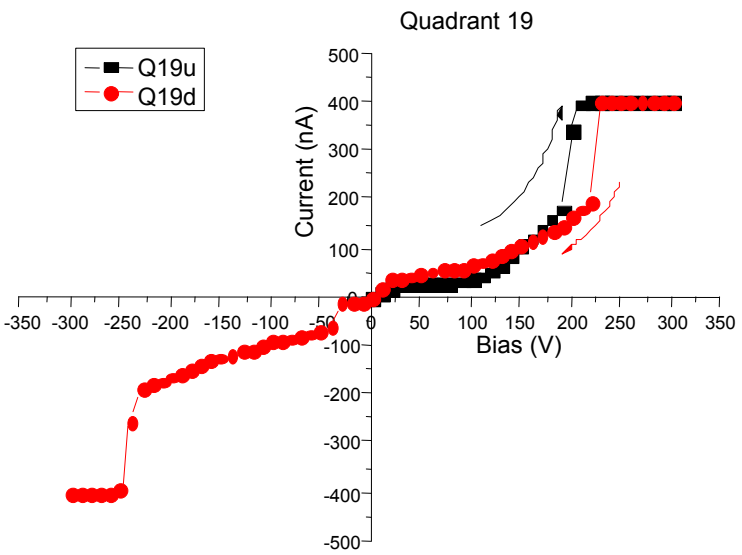
-26nA

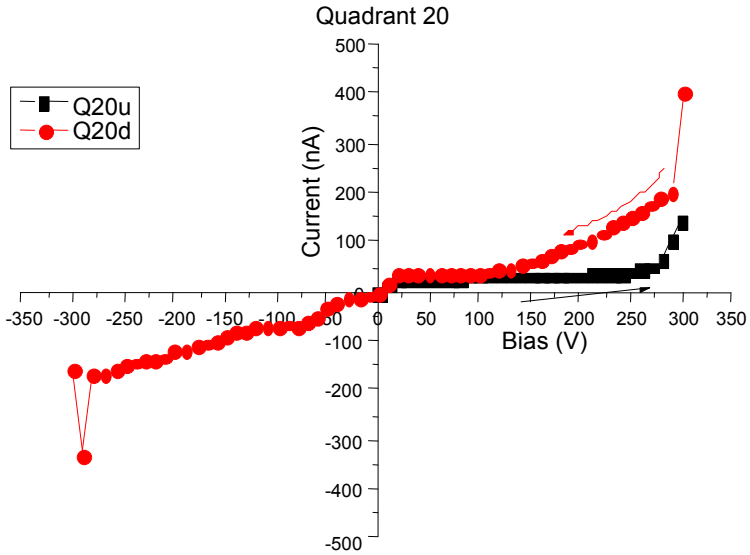


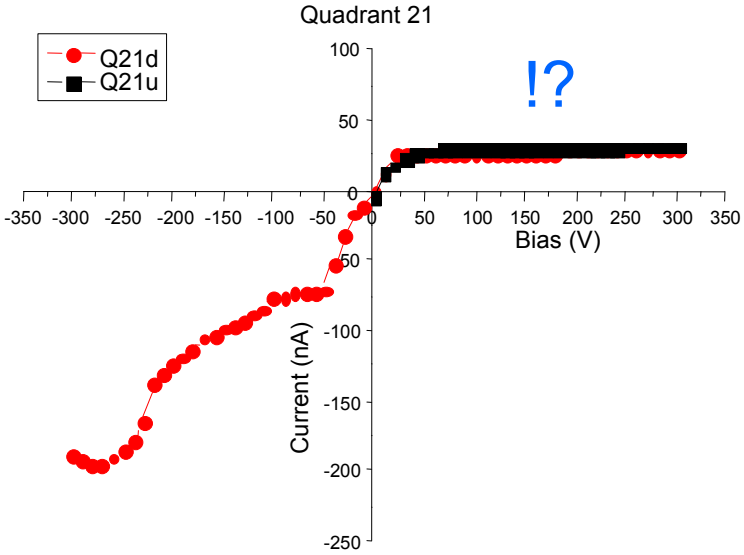
E6 Sample MDS-3, I-V curves

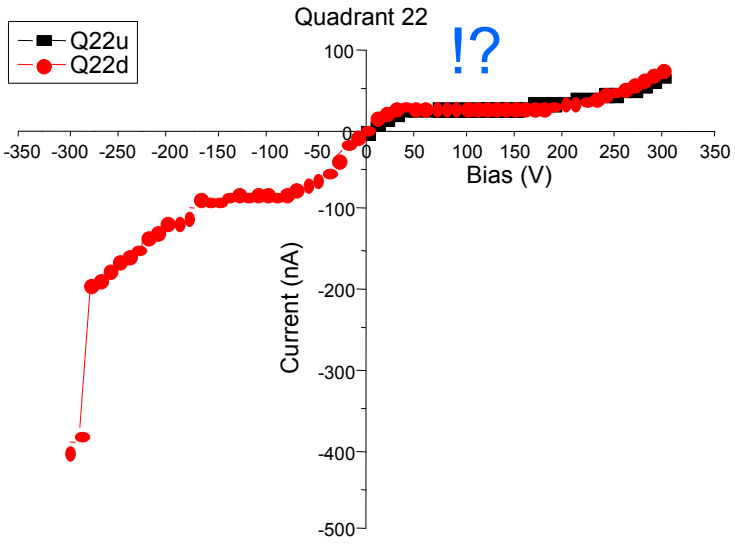
X-ray beam on

Ni-TiC contacts, *poor quality*
Sample thickness ~350μm

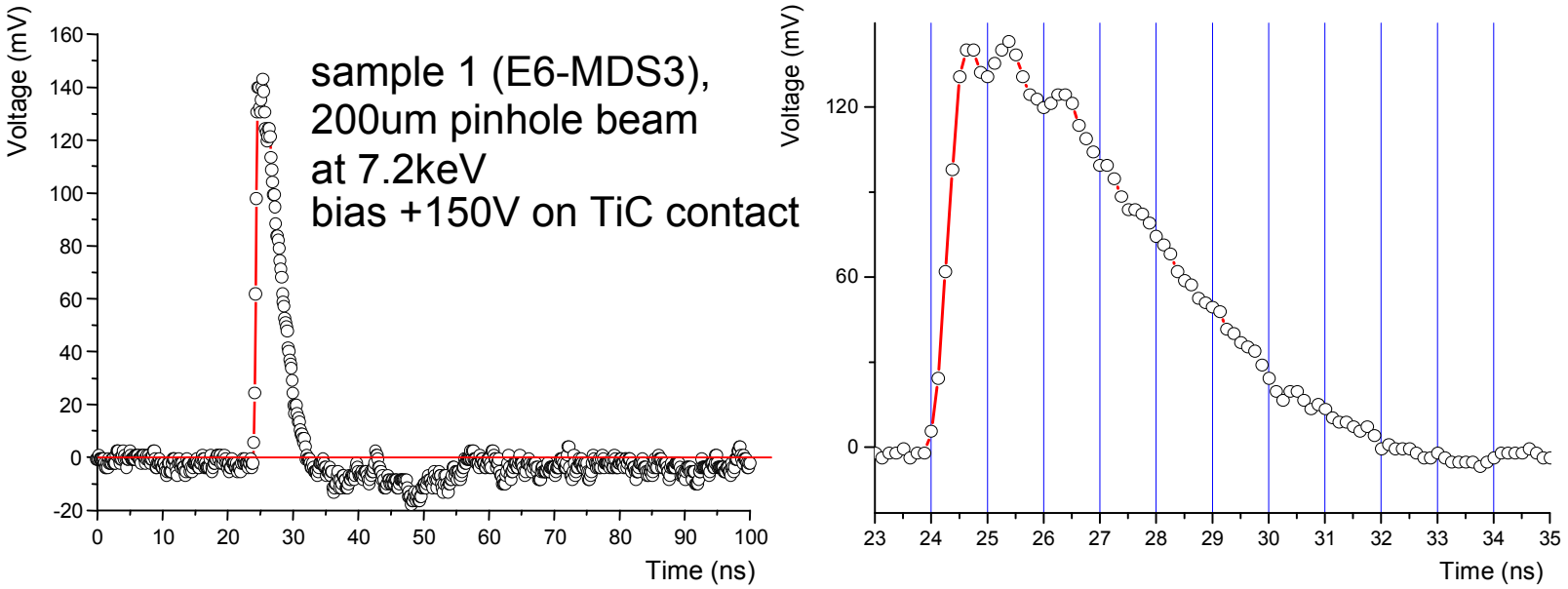








Pulse response MDS3 samples (July 06)



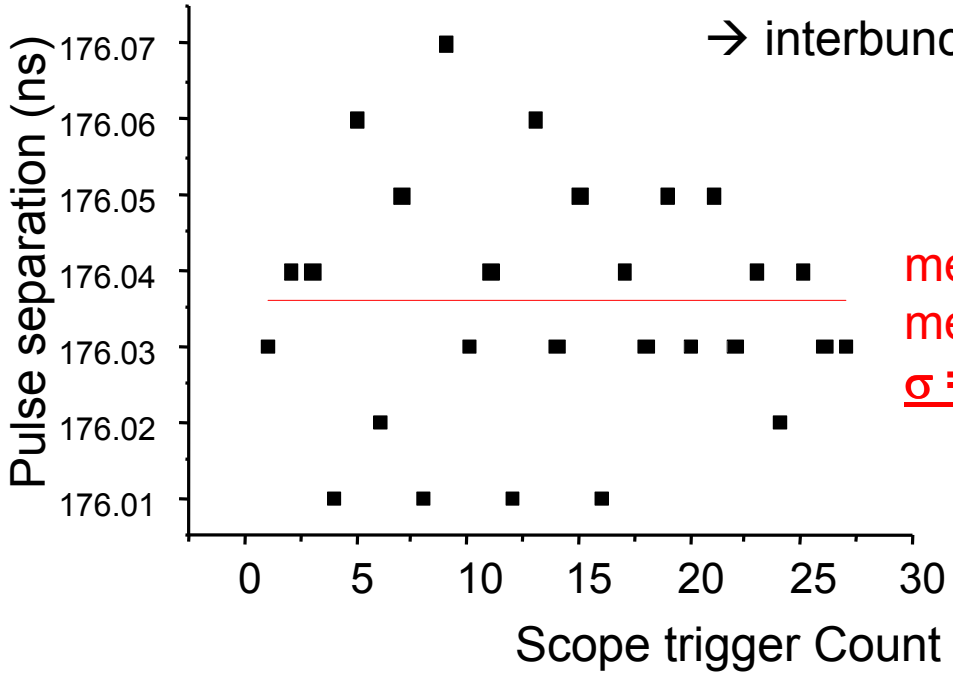
DBA3 +1GHz BW 8Gs/s Lecroy 584 scope

beam on quadrant 19,

(X-ray pulse duration 50~100ps, not measured)

Intervals between ESRF Synchrotron Pulses

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

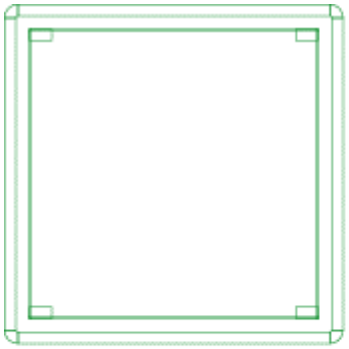


measured:
mean=176.036ns
σ = 16ps

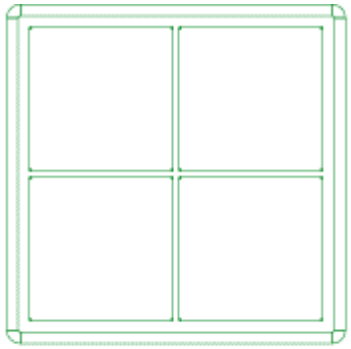
Lecroy scope data post-analyzed with leading edge 'discrimination', level normalized Individually to pulse integral (σ→20ps for simple leading edge discrimination).

Test designs for Glasgow-ESRF mask

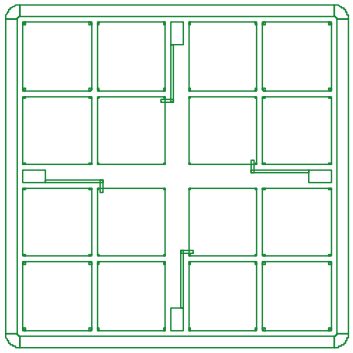
Simple pad and guard ring



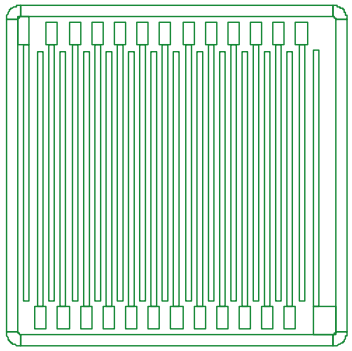
Quadrant



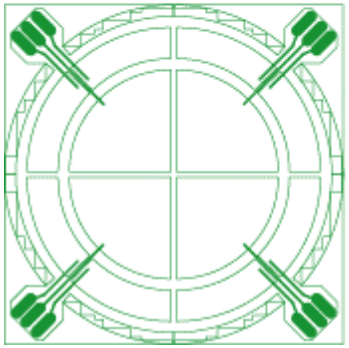
Quad-quadrant



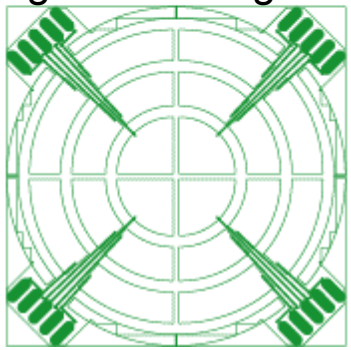
Strips



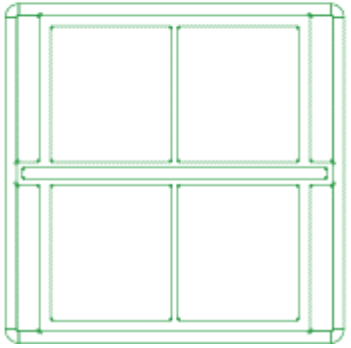
Quadrant and segmented ring



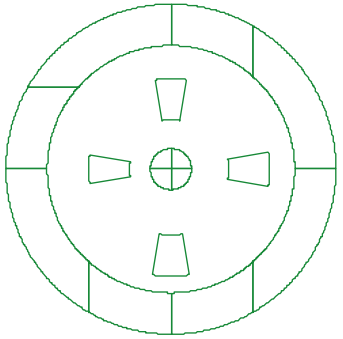
Quadrant and double segmented rings



RF phase signal readout (Naylor)



Contact impedance test (cf. Dynex Semicon')



3mm-side motifs, positive/negative mask sets in processing at Glasgow University (Sept 2006)

Conclusions:

AFM and interferometric profiling very useful and *accessible* tools, but give no information on subsurface damage

X-ray beam tests (topo', XBIC…) give much information but *limited access*

Solutions for good device fabrication demonstrated, but we still need

availability of state of art diamond surface finishing technologies

reliable/routine precision lithography and contact processing on (thinned) CVD plates

initial tests, diamond X-ray beam monitors work well --
no physics 'show stopper' problems encountered

but high intensity, long-term radiation & aging tests not yet done…