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

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D. Twitchen, H Godfried
Ph. Bergonzo, M. Nesladek
P. Muret, M. Wade
M. Mermoux
Ch. Nebel
J. Butler

J. Härtwig, P. van Vaerenbergh, R. Barrett, H. Gonzalez, G. Naylor, I. Snigireva, A Rommevaux, ID02/ID19/ID21 staff

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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?…

Transmission 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, ΔE/E ~ 10^{-4}
→ max. absorbed power: < mW (monochromatic beam)

but >10W in ‘white’ beam applications!!
…why diamond?

$Z = 6 \rightarrow$ low specific X-ray absorption / beam scattering

High charge carrier saturation velocity ($\sim 3 \times 10^7$ cm/s) and low dielectric constant (5.5)

$\rightarrow$ fast pulse response ($\sim 1\text{ nsec in } 50\mu\text{m thick device}$)

synchrotron ‘pulse by pulse’ analysis

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

$\rightarrow$ low leakage currents at high temperature,

high heat load ‘white’ beam monitoring possibility

why single crystal material?

*beam coherence* preservation:

no grain-boundary artifacts

$\rightarrow$ minimal bulk scattering

X-optics surface quality

charge-carrier lifetime $\sim 1\mu\text{s}$

$\rightarrow$ 100% charge signal collection over $\sim \text{mm distances}$

Polycrystal sample, on same scale

Single crystal CVD material

Courtesy Ph. Bergonzo E Berdermann/E6
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
Methods of sample analysis tested:

**Material:**
- Raman confocal micro scanning, visible/UV microscanning fluorescence spectrometry
- cryogenic fluorescence spectrometry, visible, UV, electron excitation
- electron spin resonance: impurities at (sub)-ppB level
- X-ray topography; X-ray small angle scatterinng
- AFM and laser optical interferometry

**Device:**
- 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

White light

Crossed polarizers

‘fingerprint twirls’ are artifacts in image display only

Raman* intensity (arbitrary scale)

Raman* center frequency (cm\(^{-1}\))
n.b. not precisely calibrated!

Raman* width (cm\(^{-1}\))

Courtesy Michel Mermoux LEPMI-ENSEEG, Grenoble Dec 2003
Apollo Diamond
sample No.2186

Crossed polarizers

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

Diffuse region
NVO centers at 636nm
Strip-like features --!? H3 centers at 503nm

Raman* intensity (arbitrary scale)

Raman* center frequency (cm⁻¹)
! System not precisely calibrated!

Raman* width (cm⁻¹)

Courtesy Michel Mermoux LEPMI, Grenoble Dec 2003
Cryogenic fluorescence spectrometry:
E6 70310 and Apollo 2186 Samples

E6 #70310
excitation: 15kVe-beam, spot size: 8 (53 nA),
slits: 0.05, integration time: 10s
temperature: 5K

Apollo #2186
excitation: 15kVe-beam, spot size: 8 (53 nA),
slits: 0.05, integration time: 0.5s
temperature: 5K

A band
H3
N-V

Courtesy P. Muret, M. Wade
LEPES-CNRS Grenoble
**ESR measurements:**
Sumitomo and E6 CVD samples

(a) SumiCrystal (b) e6, scan times is 15 times longer than that of (a).
P1 center in SumiCrystal at $10^{14} \text{cm}^3 \sim 10^{15} \text{cm}^3$.
P1 center in E-Six is under detection limit ($<10^{14} \text{cm}^3$).
Carbon dangling bond defect (at $g=2.0026$) $\sim 5 \times 10^{14} \text{cm}^3$
“NV− and VH− were not observed…”

Courtesy Ch. Nebel, H. Watanabe AIST Nov 2005

*sample MDS-5 delivered ESRF March 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
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*

ESRF 02-12-05 A sample, front & readsides

‘thick’ sample ~350µm

*courtesy Herman Godfried*
E6 Ascot sample after *scaithe polish* at E6-Cuijk*

E6 sample MDS-5, *thinned to 40\(\mu m\)*

*courtesy Herman Godfried*
AFM ESRF sample MDS1 (Mar05), thickness 350 µm, E6 resin wheel polish then scaife and superpolish by J Butler-NRL

Side A

Side B
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

Micro-roughness RMS (nm)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Micro-roughness RMS (nm)</th>
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<tbody>
<tr>
<td>E6-MDS 5</td>
<td>6.0</td>
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<tr>
<td>021205-B</td>
<td>4.9</td>
</tr>
<tr>
<td>021205-C</td>
<td>4.3</td>
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<tr>
<td>021205-D</td>
<td>0.6</td>
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<tr>
<td>021205-E</td>
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<tr>
<td>E6-MDS 1</td>
<td>0.8</td>
</tr>
<tr>
<td>021205-A</td>
<td>1.7</td>
</tr>
<tr>
<td>E6-70310</td>
<td>0.7</td>
</tr>
</tbody>
</table>

- E6 scaife
- E6 scaife + Super polish
- Ti Carbided & acid etched

Courtesy A Rommevaux, ESRF
Small angle X-ray scattering at ESRF ID02

Azimuthally averaged scattering intensity $I$ between $\alpha=120^\circ$ to $\alpha=150^\circ$ 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

Sample thickness: 40µm 100µm 350µm

courtesy Boesecke, Panine May 06
Topography data, ESRF ID19

(A) E6 Sample 70310, Laue

(B) Apollo sample, Bragg white beam

Dec 2003

E6-Berdermann samples C and D
Laue white beam

Oct 2004

Topo courtesy J Haertwig,
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)
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 (εD = ??)
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

SC CVD diamond plate, 100µm thick
screw posts

c coax RF signal and bias cables

r=1.75mm

~6mm

PCB_2

X beam

PCB_1

contact probe
ESRF-ID21 X-ray Microscope

focus 1.2 x 0.4µ² FWHM, 
~5x10⁷ photos/sec at 7.2keV
I-V curves
Cr-Au contacted 100mm plate with/without beam

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 µm² 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

effect of operating bias

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

1 x 0.4µ², ~10^8 photos/sec at 7.2 keV
Cr-Au contacted quadrant device: ‘dc’ measured position response

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 occurring 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

700ns

<100pS FWHM
X pulse duration

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
Beam pulse every \(~730\text{ns}\) \((1.37\text{MHz})\), HP8591 signal analyzer at \(352\text{MHz}\) with \(~1\text{kHz}\) BW: measuring pulse signal power in \(8\text{th harmonic}\).
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 <1\(\mu\)m fwhm X-beam at 12.5\(\mu\)m steps
‘hot spot’ ‘X’ under wire bond

Raster scan directions

3nA (normalized to transmitted flux)
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
(333 µm thick, sample E6-MDS4)

I-V curve in X-ray beam (ID21, pinhole beam Ø100 µm, \(\sim 10^8\) ph/sec at 7.2 keV).

leakage current is \(\sim\) pA level for +/-150V bias.

Photoconductive gain

Si calibration \(\rightarrow \epsilon_D = 15.1\) eV / e-h pair
Surface damage and contact edge hot defects

Resin wheel polish ‘tadpoles’

SEM

left

Guard ring
electrode

50x reflect’ microscope

right

Guard ring
electrode

10μm

75μm

0.06250
0.12500
0.18750
0.25000
0.31250
0.37500
0.43750
0.50000
0.56250
0.62500
0.68750
0.75000
0.80000
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

A lot of hysteresis

-26nA

E6 Sample MDS-2, I-V curves
X-ray beam on

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

A lot of hysteresis

-26nA
E6 Sample MDS-3, I-V curves
X-ray beam on

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

Quadrant 19

Quadrant 20

Quadrant 21

Quadrant 22

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

Quadrant 19

Quadrant 20

Quadrant 21

Quadrant 22
Pulse response  MDS3 samples (July 06)

sample 1 (E6-MDS3), 200um pinhole beam at 7.2keV bias +150V on TiC contact

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…