ESRF

Beam Monitoring Applications for diamond at synchrotron X-ray sources & Characterization of diamond: tools at ESRF

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Experiments Division

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This talk...

1. ESRF and synchrotons elsewhere

2. CVD diamond for beam *intensity* and *position* monitoring at ESRF

3. X-ray Microbeam Radiation Therapy *dosimetry*

4. Tools at and around ESRF for diamond characterization and device tests

budget

France	27.5 %
Germany	25.5 %
Italy	15 %
United Kingdom	14 %
Spain	4 %
Switzerland	4 %
Benesync (Belgium, Netherlands)	6 %
Nordsync (Denmark, Finland, Norway, Sweden)	4 %
Portugal	1 %
Israel	1 %
Austria	1 %
Czech Republic	0.38 %
Hungary	0.20 %

73 MEuros

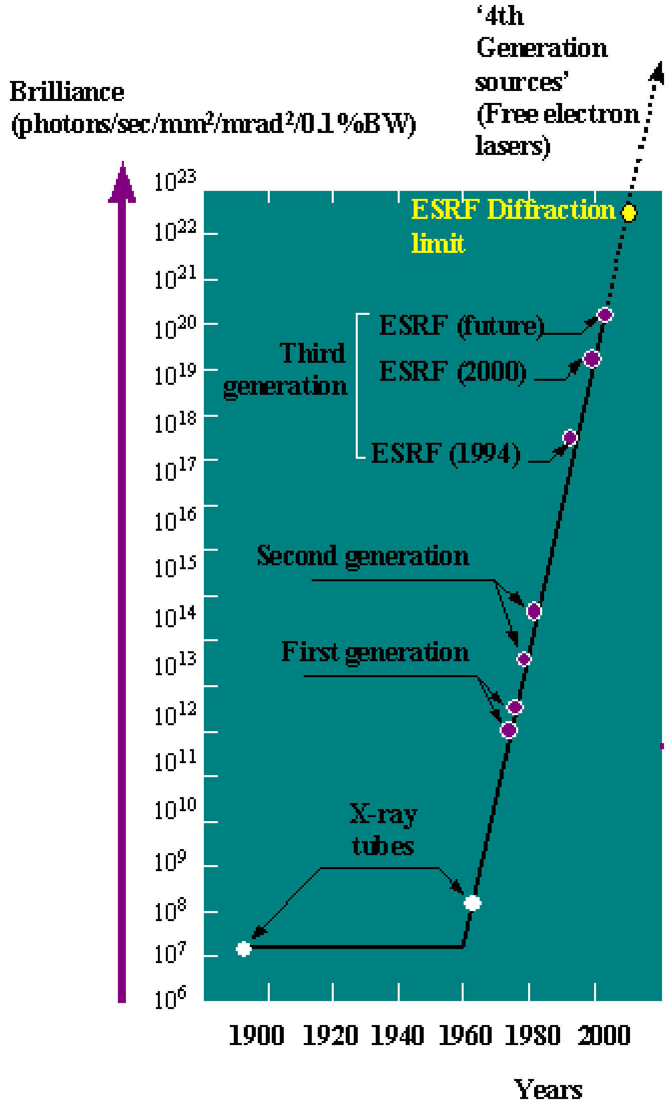
ESRF 'Public' Beamlines

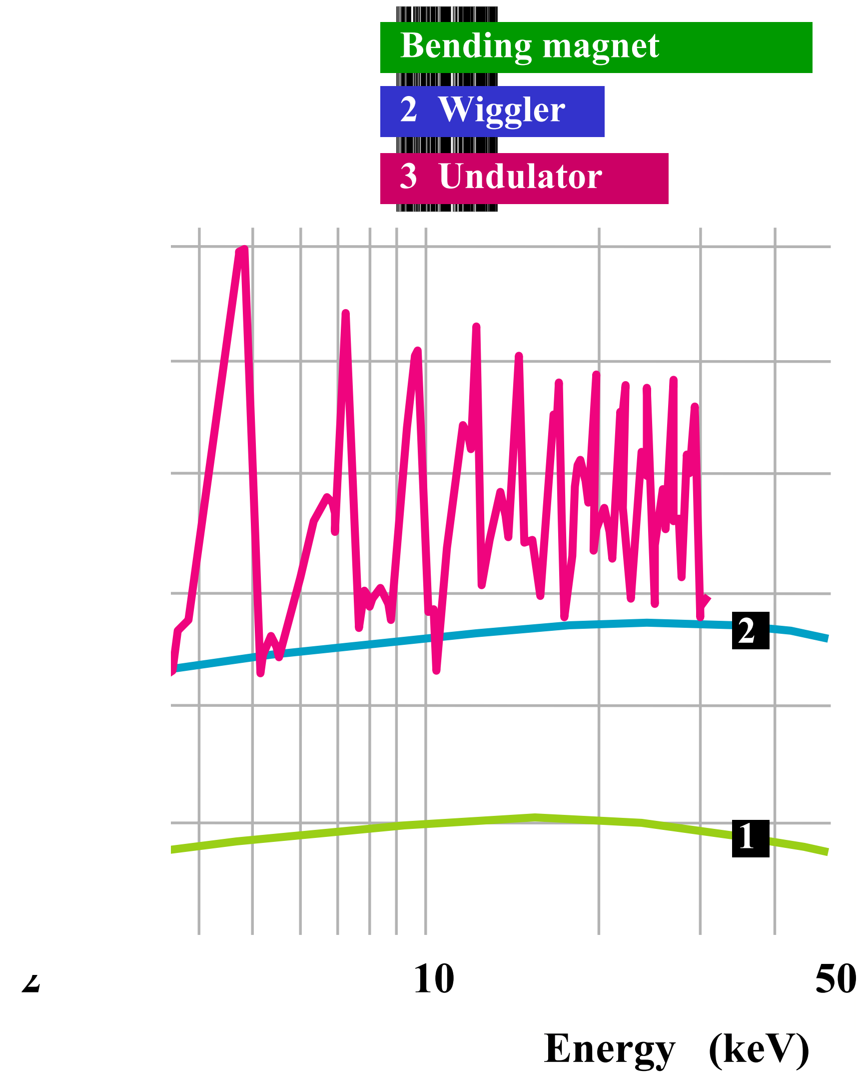
ID1	Anomalous scattering
ID2	Small-angle scattering
ID3	Surface diffraction
ID8	Spectroscopy using polarised soft X-rays
ID9	Biology / High pressure
ID10	Multipurpose
ID11	Materials science
ID12	Circular polarisation
ID13	Microbeam
ID14	Protein crystallography
ID15	High energy
ID16	Inelastic scattering
ID17	Medical
ID18	Nuclear scattering
ID19	Microtomography - Topography
ID20	Magnetic scattering
ID21	X-ray microscopy
ID22	Microfluorescence
ID24	Dispersive EXAFS
ID26	Spectroscopy on ultra-dilute samples
ID28	Inelastic scattering
ID29	Biology MAD
ID30	High pressure
ID32	Surface EXAFS - Photoemission
BM5	Optics
BM16	Powder diffraction
BM29	Absorption spectroscopy

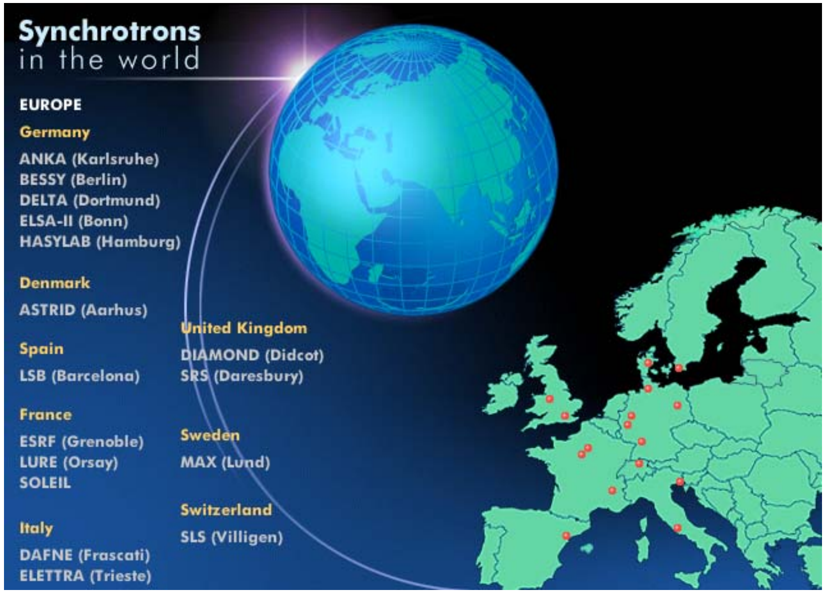
ESRF 'Collaborative Research Group' Beamlines

BM1	Swiss-Norwegian	Absorption and diffraction
BM2	D2AM (France)	Materials science
BM7	GRAAL (Italy/France)	Gamma spectroscopy
BM8	GILDA (Italy)	Absorption and diffraction
BM14	MAD (UK)	Biology (MAD)
BM16	MAD (Spain)	Biology (MAD)
BM20	ROBL (Germany)	Radiochemistry
BM25	SPLINE (Spain)	Absorption and diffraction
BM26	DUBBLE (Netherlands, Belgium)	Multipurpose
BM28	XMAS (UK)	Magnetic scattering
BM30	FIP (France)	Protein crystallography
BM30	FAME (France)	Environment (EXAFS)
BM32	IF (France)	Surfaces and interfaces

ESRF source characteristics






Synchrotrons
in the world
EUROPE
Germany
ANKA (Karlsruhe)
BESSY (Berlin)
DELTA (Dortmund)
ELSA-II (Bonn)
HASYLAB (Hamburg)
Denmark
ASTRID (Aarhus)
Spain
LSB (Barcelona)
France
ESRF (Grenoble)
LURE (Orsay)
SOLEIL
Italy
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ELETTRA (Trieste)
United Kingdom
DIAMOND (Didcot)
SRS (Daresbury)
Sweden
MAX (Lund)
Switzerland
SLS (Villigen)


Synchrotrons
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NORTH AMERICA
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Synchrotrons
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South Korea
PLS (Pohang)
China
IHEP (Beijing)
NSRL (Hefei)
Inde
INDUS (Indore)
Japan
HSRC (Hiroshima)
NANO-HANA (Chiba)
NEW SUBARU (Himeji)
PHOTON FACTORY (Tsukuba)
SPRING8 (Himeji)
UVSOR (Okazaki)
VSX (Tokyo)
Singapore
HELIOS 2 (Singapour)
Taiwan
SSRC (Hsinchu)
Australia
BOOMERANG

Applications of CVD diamond at ESRF

Beam *intensity and position* monitoring:

monochromatic (...and white?) beams, 5 ~ 100 keV

i. solid state ionization chamber
ii. bulk optical fluorescence

Microbeam radiation *dosimetry*

pink beam (peak flux at ~100keV)

i. solid state ionization chamber

Synchrotron Beam Monitoring: requirements

Synchrotron beam size at samples from
~100µm to <1µm (~10nm coming?)

required beam stability ~10% of beam fwhm

beam intensity measurements require
<1%...0.1% (relative) accuracy & linearity, for
sampling times <0.1 ... 10 secs

ESRF Insertion Device beamlines:

~10^9 photons/1µm^2/s ... 10^{13}photons/(100µm)^2/s
=> max. power: ~ mW (monochromatic beam)

but -> 100W/mm^2 (white beam) !!

why diamond?

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

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

-> fast pulse response (<nsec in practical device)

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

-> high heat load, 'white' beam monitoring?

why now?

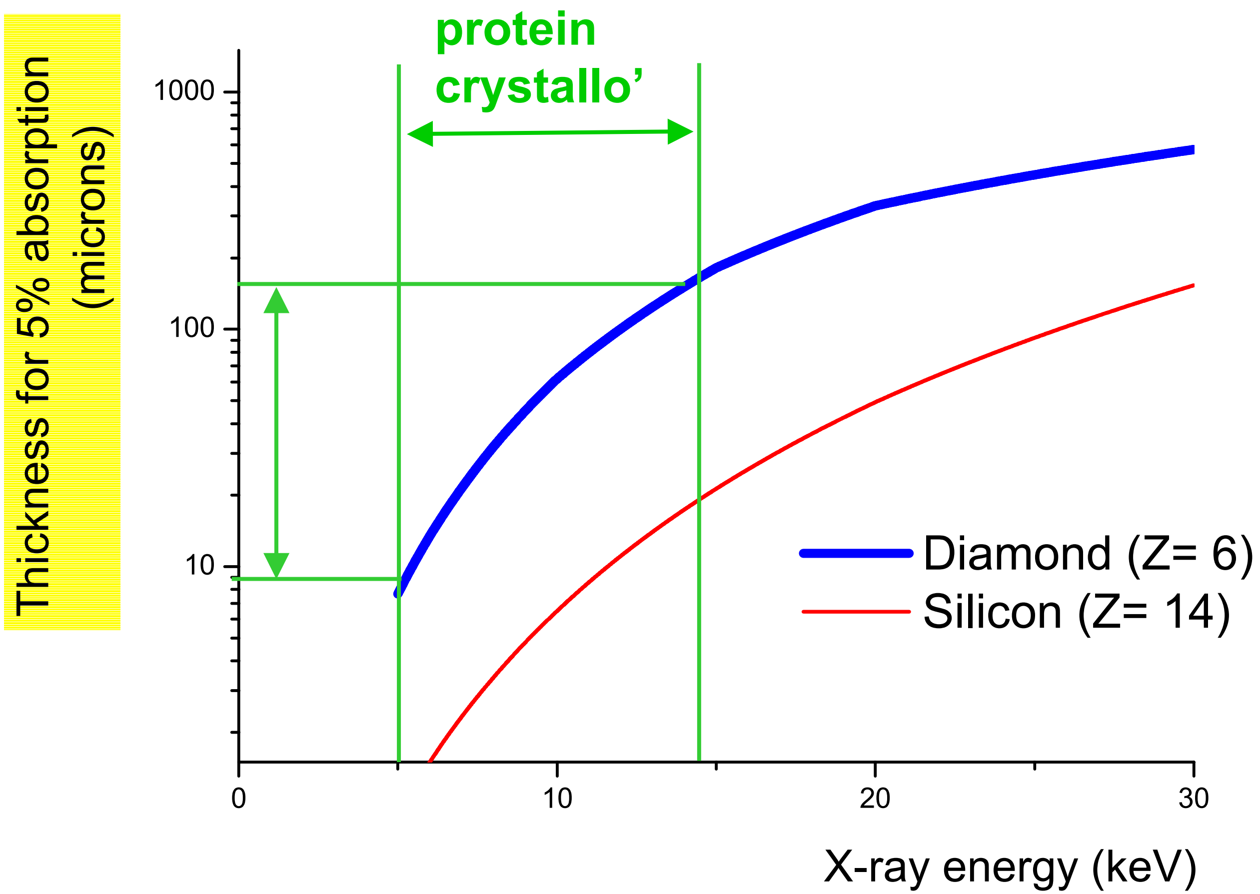
single crystal CVD grown material becoming available (2003+) with charge-carrier lifetime > 1μsec --*Element-6* *

'4th generation' FEL X-ray sources: no other materials can withstand pulse heating/ablation energies

*Isberg et al., Science 297 (2002) 1670

material absorption limitations

0,5keV ← ESRF beamlines → 500keV

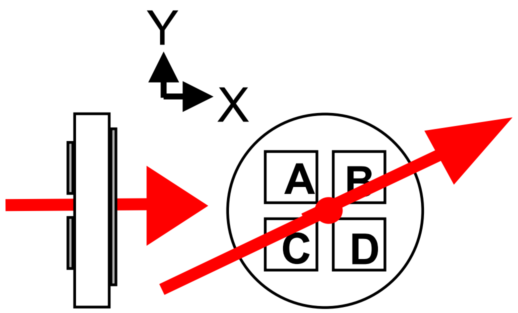


CVD diamond beam monitors: possible methods

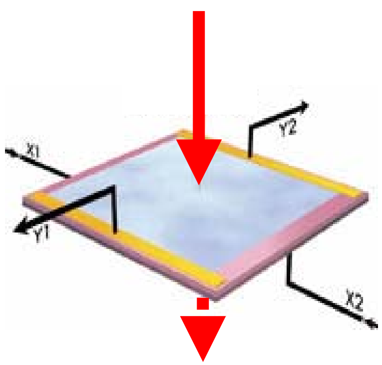
Beam intensity: use crystal ~ 50μm thick with transparent electrode contacts Ti, Mo, Al, (Pt, Au?)

Diamond bulk acts as an 'ionization chamber'

Beam Position: quadrant motif -> beam 'centre of gravity' by weighting four electrode currents A, B, C, D.



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

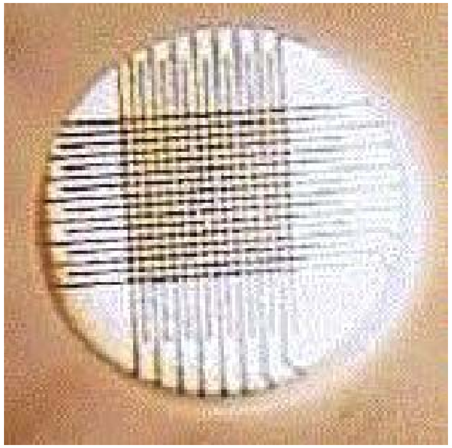


$$X = \frac{X1-X2}{X1+X2}$$
$$Y = \frac{Y1-Y2}{Y1+Y2}$$

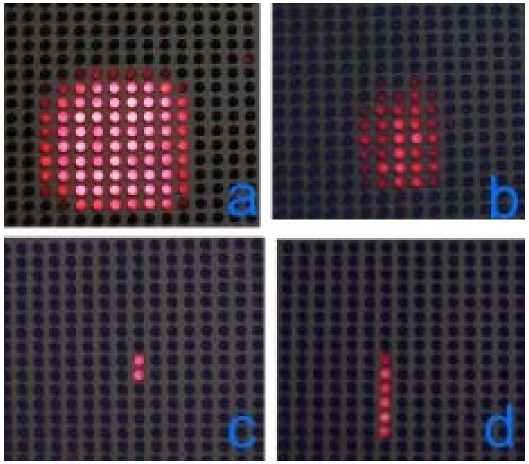
resistive surface contacts
- insensitive to beam size
- large linear spatial response

but lose fast time response (RC limit)

multiple electrode structures

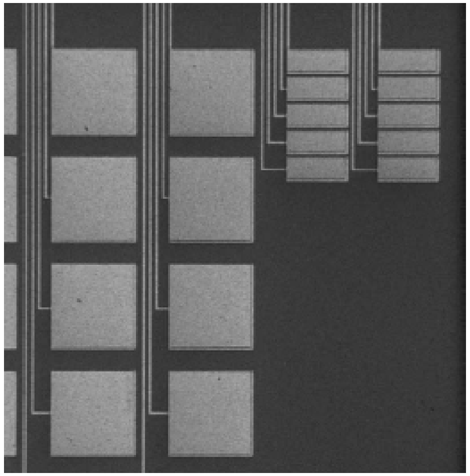


Two sided 200nm Al strip contacts on 175 μm GE *polyxtal* diamond substrate. Bias applied sequentially at 3kHz; parallel current readout ->175μm x 175μm pixels



Response to 'pink' *white beam* undulator radiation after 13mm of Al

Deming Shu, et al., APS-Argonne Nat. Lab. Report c. 2000



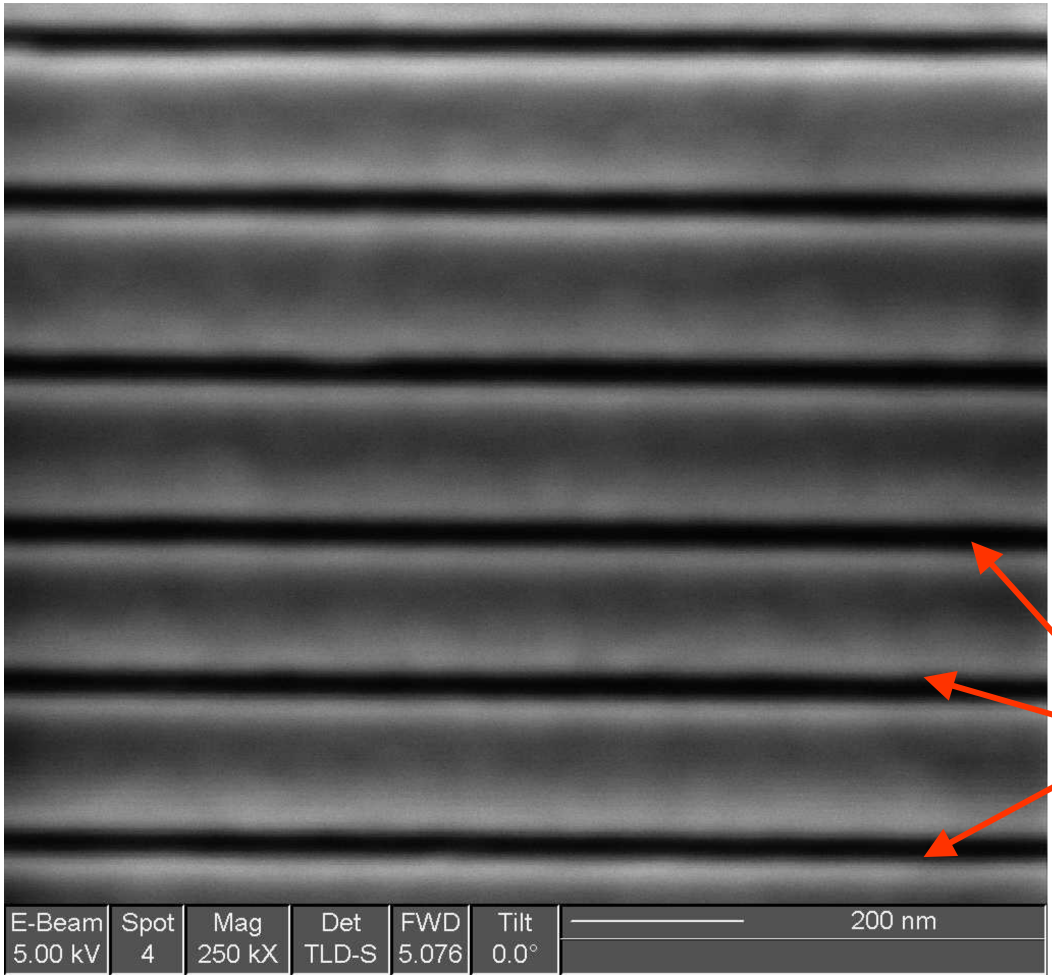
SLS-PSI PX beamline profile monitor. Pixel structures on a 1.3 μm *polyxtal* thick membrane. Small pixels are 110 x 290μm^2

SLS work continuing with FP6 'BioxHit' funding

(Automation for Protein Crystallography)

C. Schulze-Briese et al., NIM A 467–468 (2001) 230–234

Fine structures for sub-micron beam monitoring



Lithography tests:

18μm micron thick, natural diamond, heavily-boron-doped

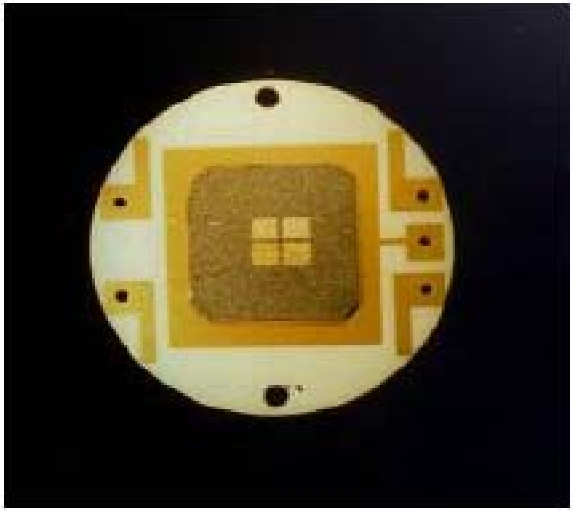
~30Å Mo + 3000Å Pt metal overlayer

focussed ion beam etched trenches, 30nm wide, (10:1 etch aspect ratio)

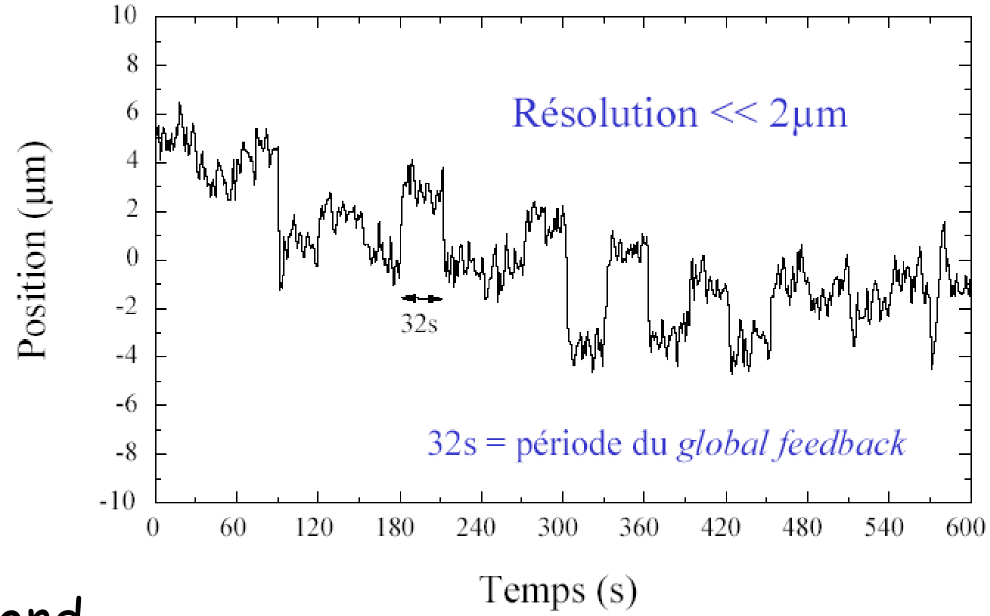
courtesy Dan Pickard, Stanford Univ. & Chris Kenney, Molecular Biology Consortium

BPM 'feasability tests'

ESRF ID12A, test of Quadrant Position Monitor



polycrystalline CVD diamond
film grown on silicon substrate



courtesy Ph. Bergonzo, CEA

Problems of *polycrystalline* diamond films:

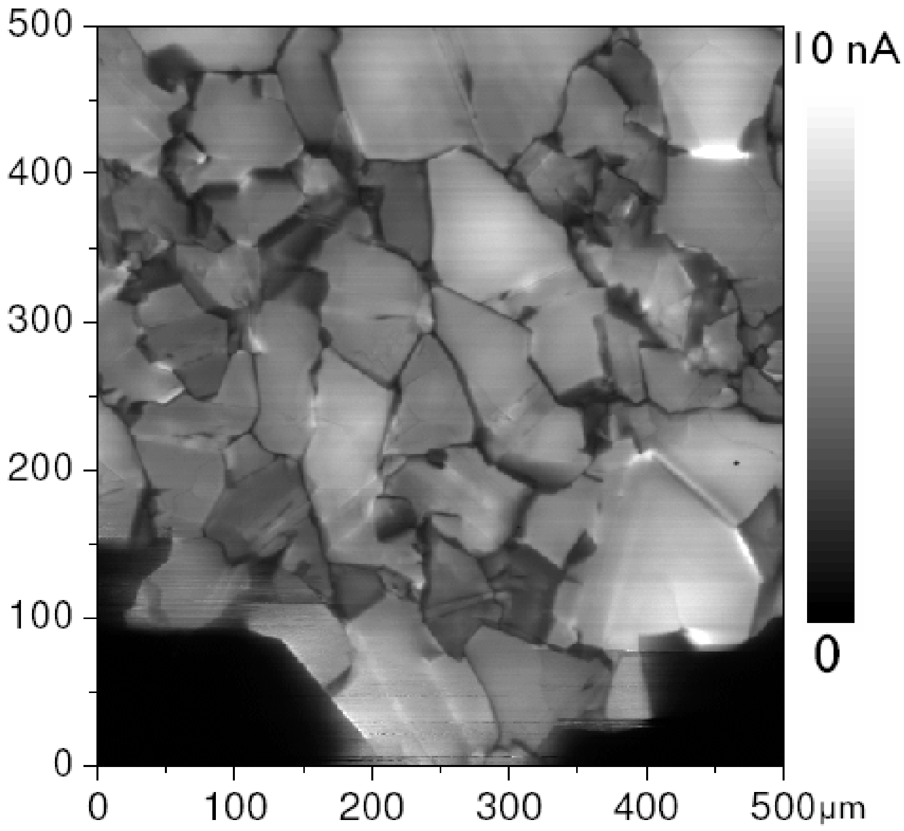
-maximum charge carrier charge collection distance ~250μm

-time lag and hysterisis in 'dc' current measurements

('trap priming' ~10secs in ~10^{13} X-photon PX beamline)

- severe *grain-boundary* response artifacts

Spatial response, polycrystalline film CVD diamond



courtesy Ph. Bergonzo, CEA

ESRF ID21 microscopy beamline

raster scan, 1μm beam at 5keV

~10^9 photons/sec.

Current signal 'contrast' with

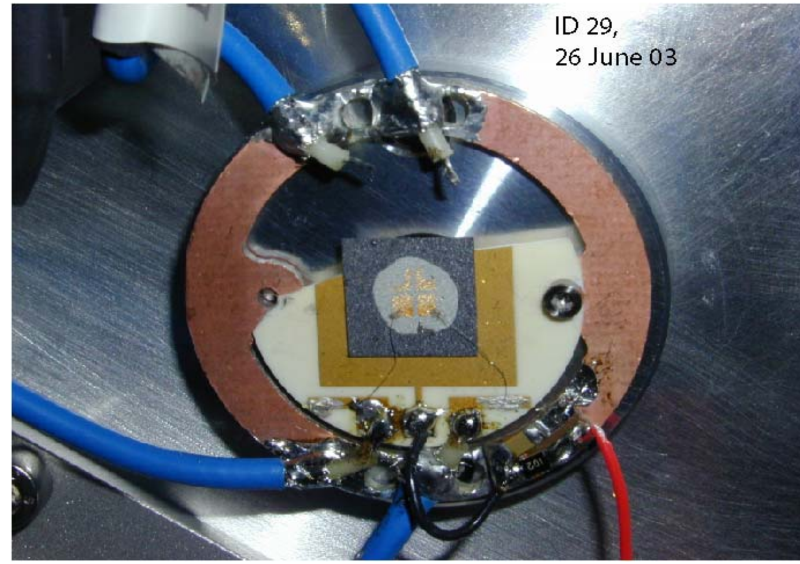
crystal grain boundaries

beam monitoring?

beam width >100μm ~ OK

beam width <100μm ✗

CVD diamond film RF signal tests, ID29 PX beamline ESRF



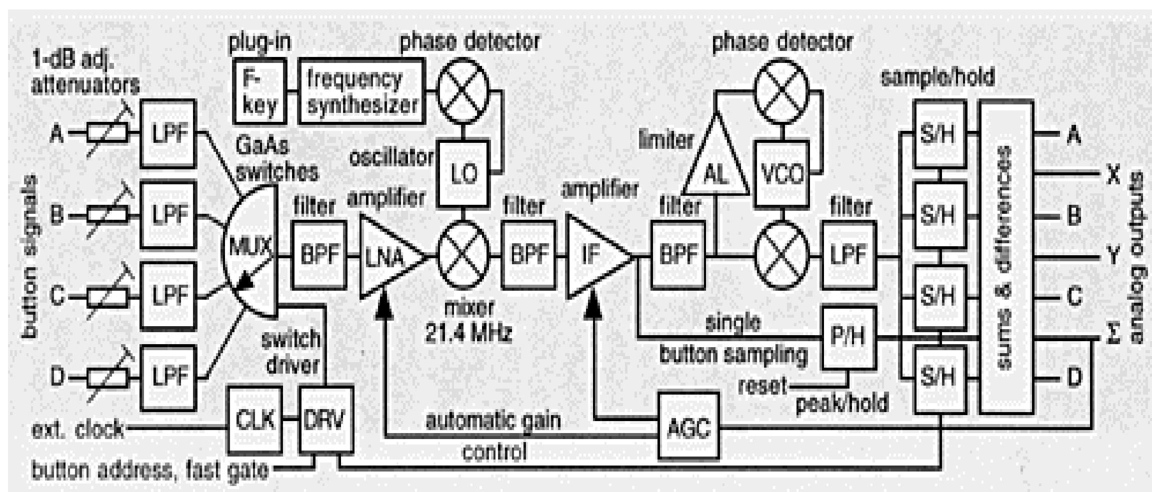
Concept: use phase sensitive detection, locked to 350MHz time structure of ESRF X-ray beam

-> mitigate lag, 'dc' drift effects?

P Bergonzo, J Bergoz, K Unser, B Shepard, J Morse

Electronics already developed for synchrotron *electron* beam submicron position sensing

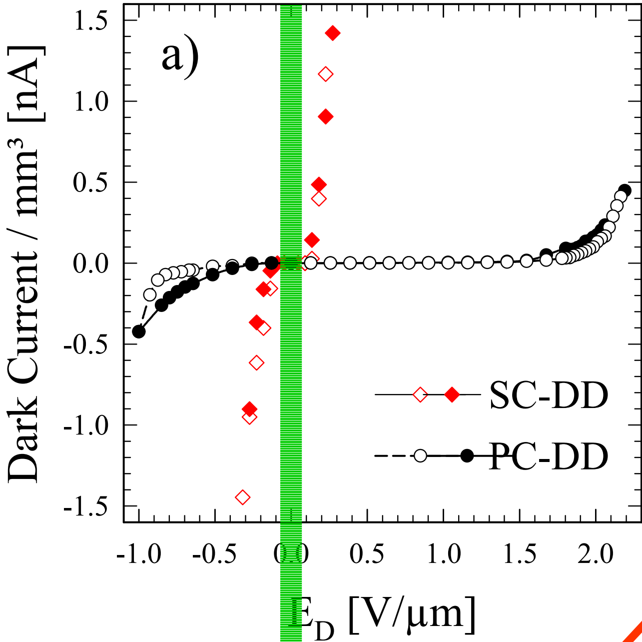
(Bergoz Instruments)

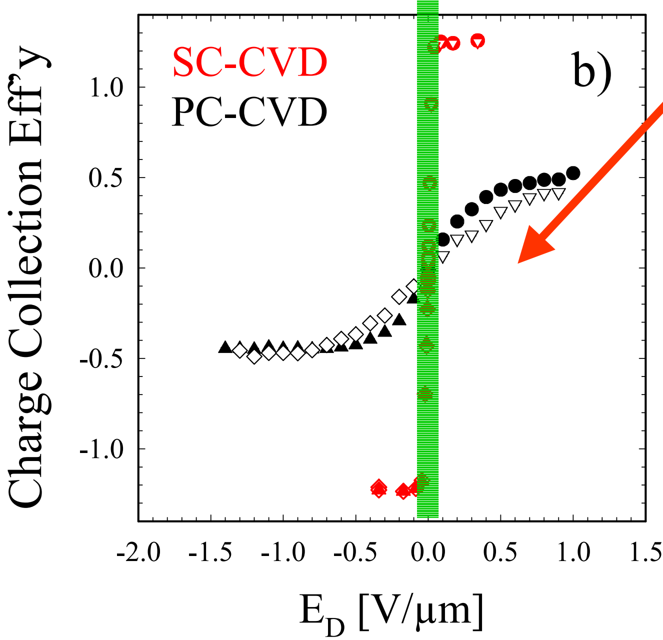


Charge collection

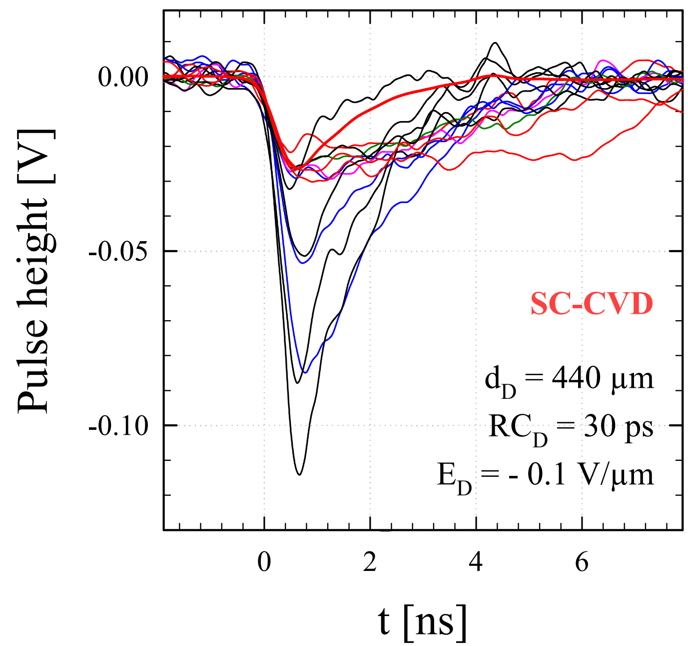
single crystal, CVD grown diamond
(E6 'electronic grade'sample)

operating E-field ~ 100 x less than for polycrystalline material





Am^{241} alpha signals (5.5 MeV)



courtesy E Berdermann (Surface and Bulk Defects in CVD Diamond Films, IX, Hasselt 2004)

ESRF tests on *single crystal* high purity CVD grown diamond

(Element-6 electronic grade sample)

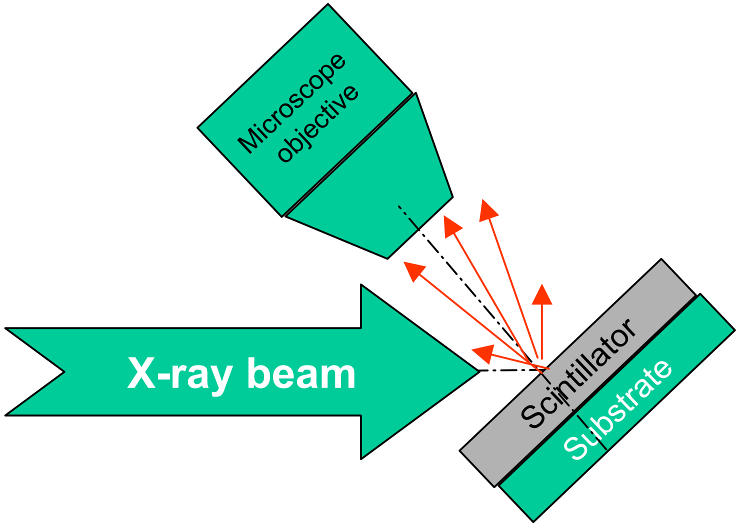
- 100μm thick polished sample, 'cleaner' than Berdermann-GSI sample (strain, as shown by Raman micromapping & X-ray topography)

- electrical response uniformity >> better than polycrystalline material over 5 x 5 mm^2 area (scanning <1μm X-ray beam)

- no observation of lag or 'trap priming' on <1sec timescale in 5keV, 1μm X-beam (10^6 ...10^9 X/sec)

- good correlation between topography defect map and dc electrical response to 5keV X-rays

Scintillant Screen X-Ray Beam Position Monitors



O Hignette, ESRF → SESO/Oxford-Danfysik licence

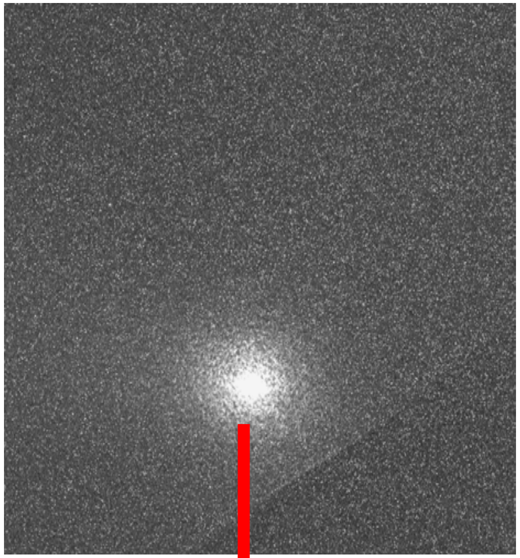
Currently most wide spread method of beam monitoring:
gives shape information and ~100nm CofG sensitivity.

existing granular-phosphor screens are X-beam opaque,

-> intermittent use only with insertion/retraction mechanics

need for radiation hard, thin film, *X-ray transmissive* scintillator

CVD diamond as a thin film, 'bulk' scintillator?



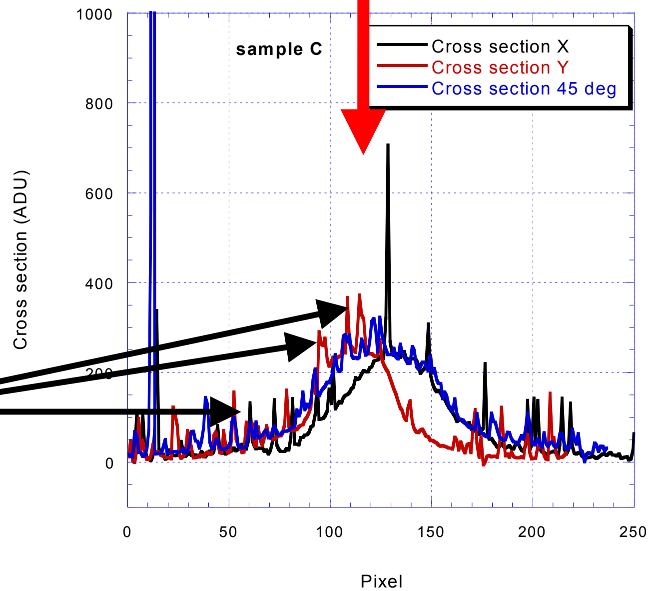
'accidental' nitrogen impurity in polycrystalline material
-> visible light fluorescence

poor light yield (~0.1 visible photon/ absorbed keV); rapid radiation damage (~1 week)

but

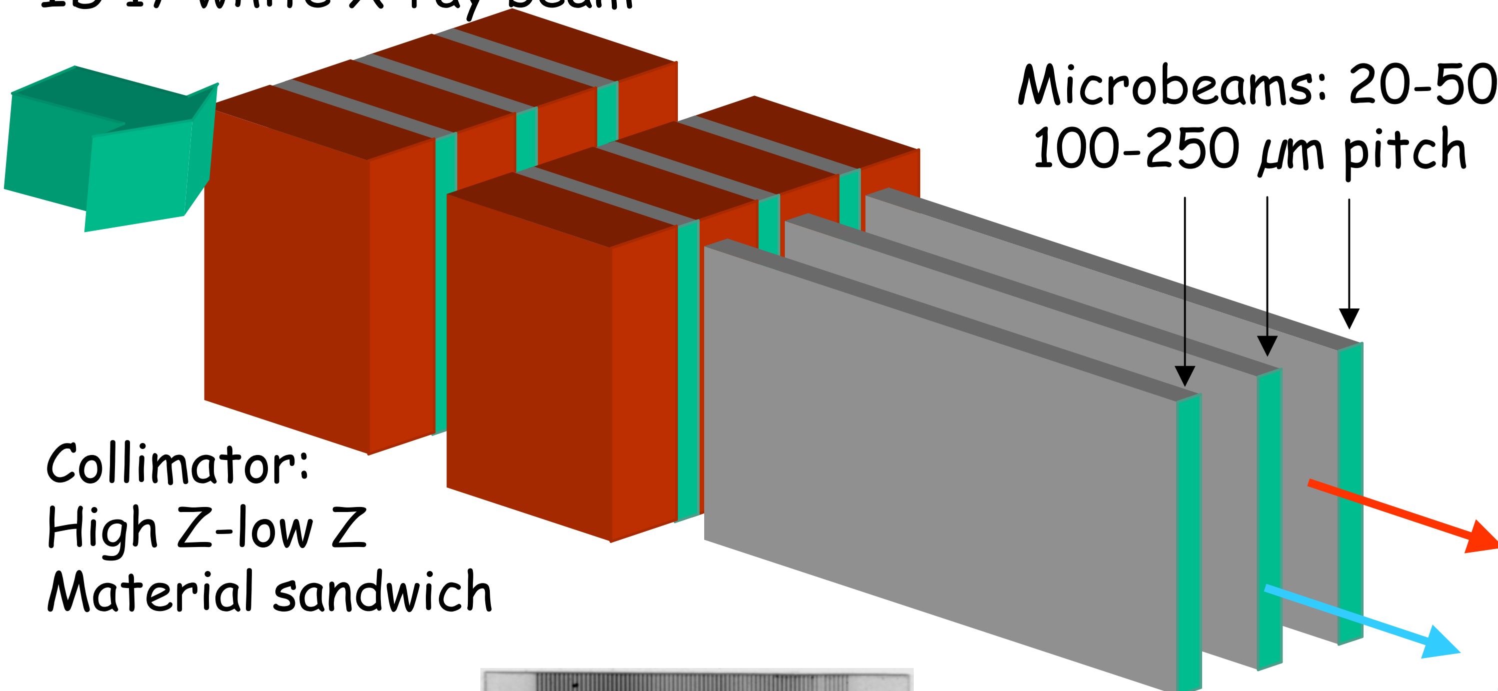
already in use for crude, 'pink' beam-status monitors at ESRF

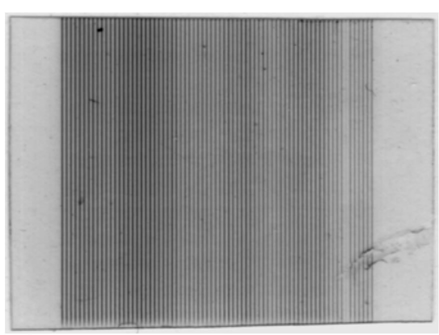
Tests on-going to optimize growth and/or doping for higher light yields in *single crystal* CVD diamond



as grown (one side polished) sample crystal-grain facet peaks

ESRF Microbeam Radiation Therapy





Microbeams pattern on gafchromic film

courtesy A Bravin, ID17 Medical Beamline, ESRF

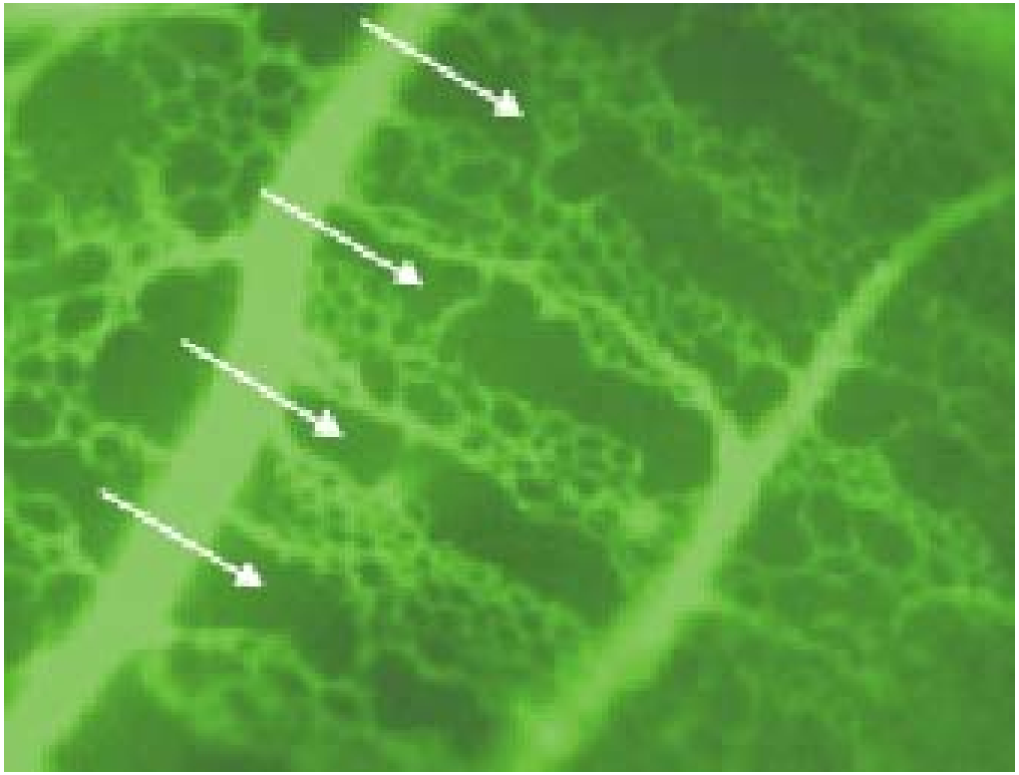
Microbeam in-vivo tests:

Chorio-Allantoic Membrane of chicken egg 24 h after after irradiation

X-ray microbeam irradiation (arrows) of 300Gy, 28 μm wide and 200 μm center-to-center distance.

larger vessels survive, but capillaries destroyed.

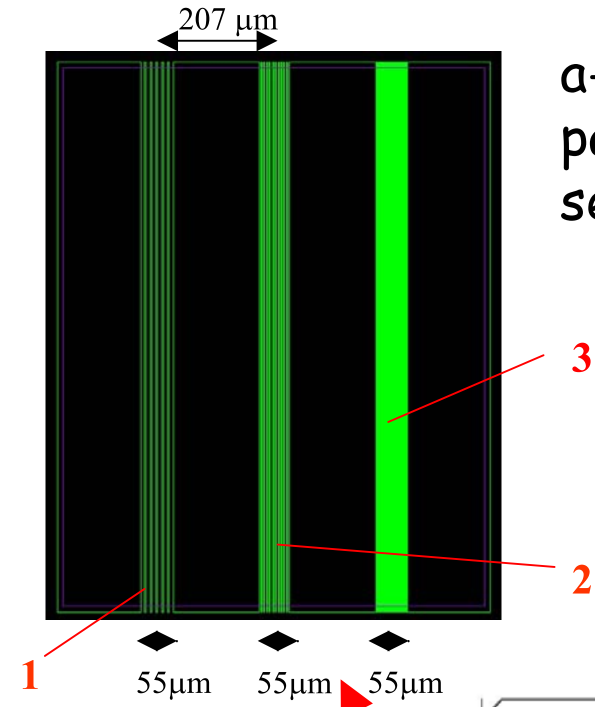
width of destroyed areas larger than micro-beams, but in some areas new capillaries bridge the gaps.



Courtesy H. Blattmann[1], W. Burkard[1], V. Djonov[2], M. Di Michiel[3] , E. Bräuer[3], J. Stepanek[1], A. Bravin[3], J.-O. Gebbers[4] and J.A. Laissue[5]
1 PSI, Villigen, CH, 2 Anatomisches Institut, Bern, CH, 3 ESRF, Grenoble F, 4 Inst. of Pathology, Luzern, CH, 5 Inst. of Pathology, Bern, CH

Microdosimetry:
'Mibedo' Test Structure

rad-hard CMOS ASIC (P Jarron et al., Cern) overlayered with 30 µm of a-Si:H (IMF-Neuchatel)



a-Si coated part of sensor

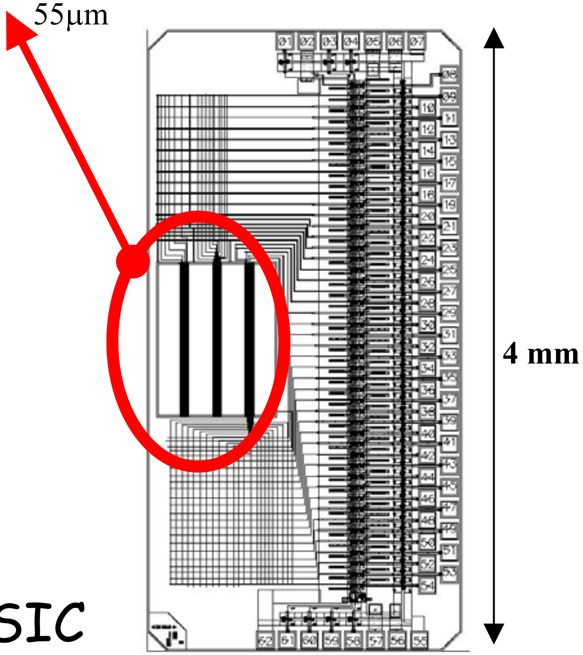
1: 5 strips with 10 µm pitch

2: 10 strips with 5 µm pitch

3: 25 strips with 2 µm pitch

-> similar strip structure + fan-out

in CVD diamond ?



CMOS ASIC

Characterization of material

X-ray topography at ESRF (ID19, BM5)*

Method based on Bragg diffraction applied to single crystal samples

Used to visualise *inhomogeneities in the crystal* on a micrometric scale...

dislocations, twins, stacking faults, growth sector boundaries, growth striations, inclusions, precipitates, ...

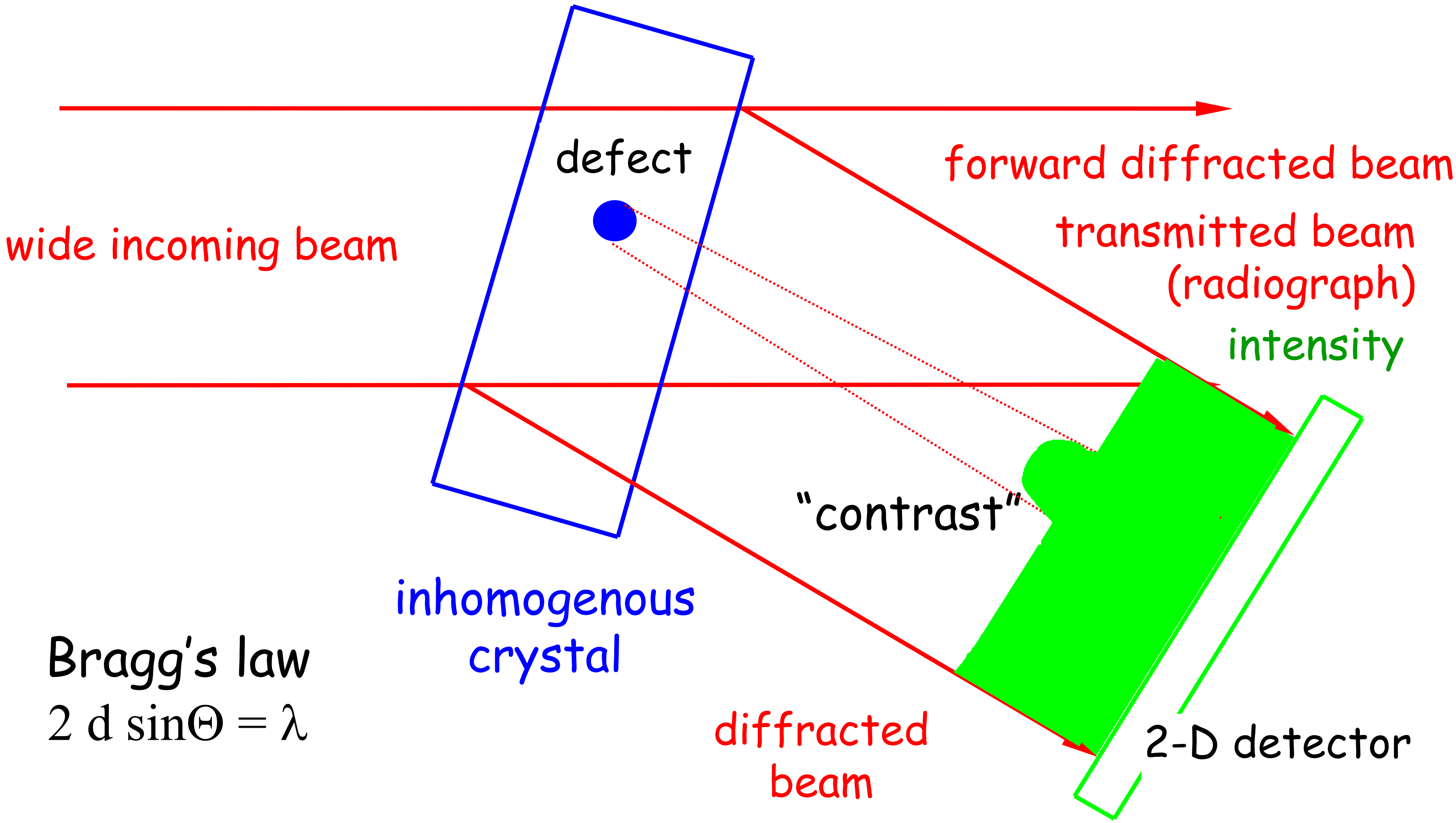
...and macroscopic deformations

bending, acoustic waves, heat bump...

** courtesy Jurgen Haertwig*

principle of X-ray diffraction topography

for an extended, homogeneous, white or monochromatic beam



Bragg's law

$$2\, d \sin\Theta = \lambda$$

topograpy methods

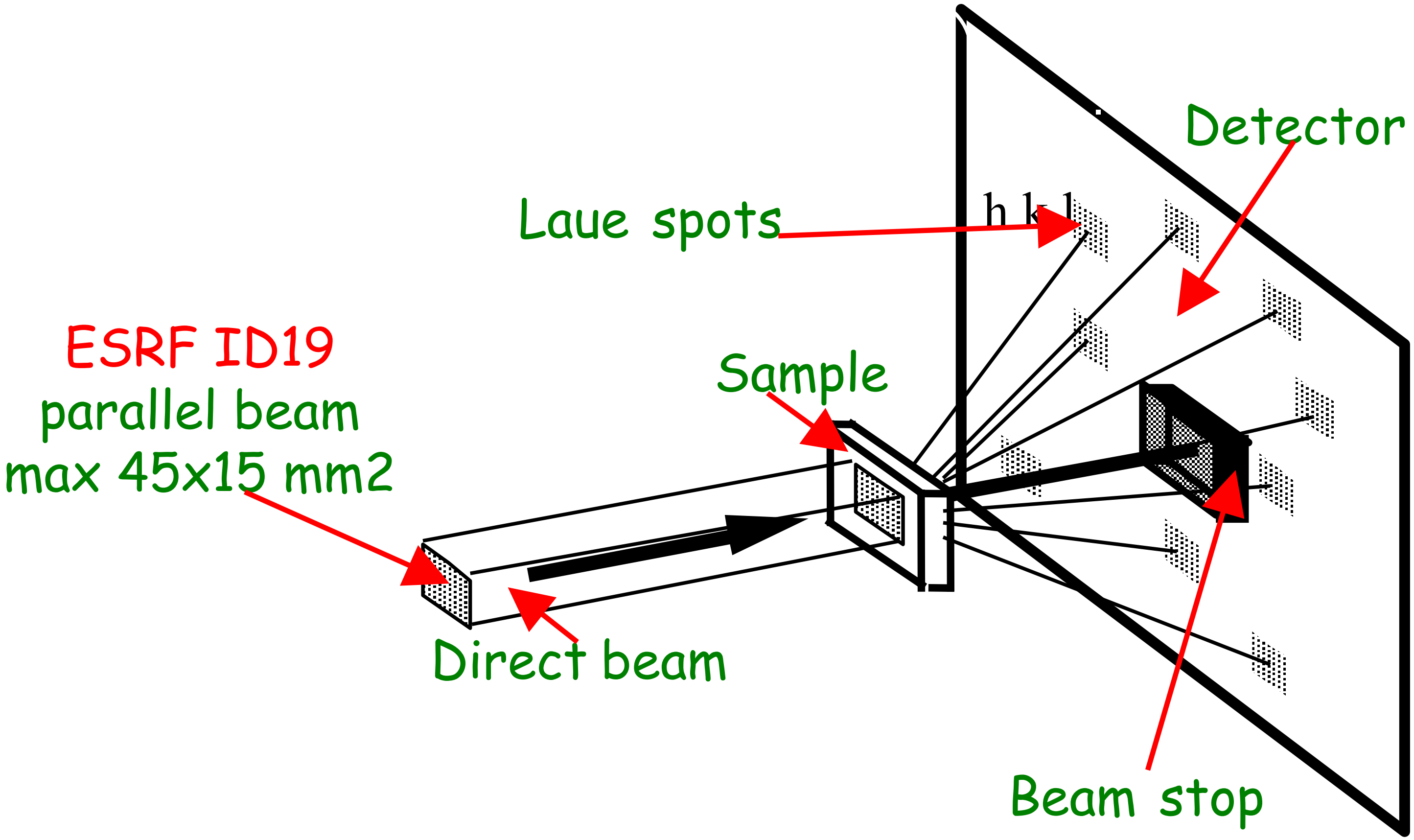
Reflection (Bragg) geometry - transmission (Laue) geometry

extended beam - limited beam

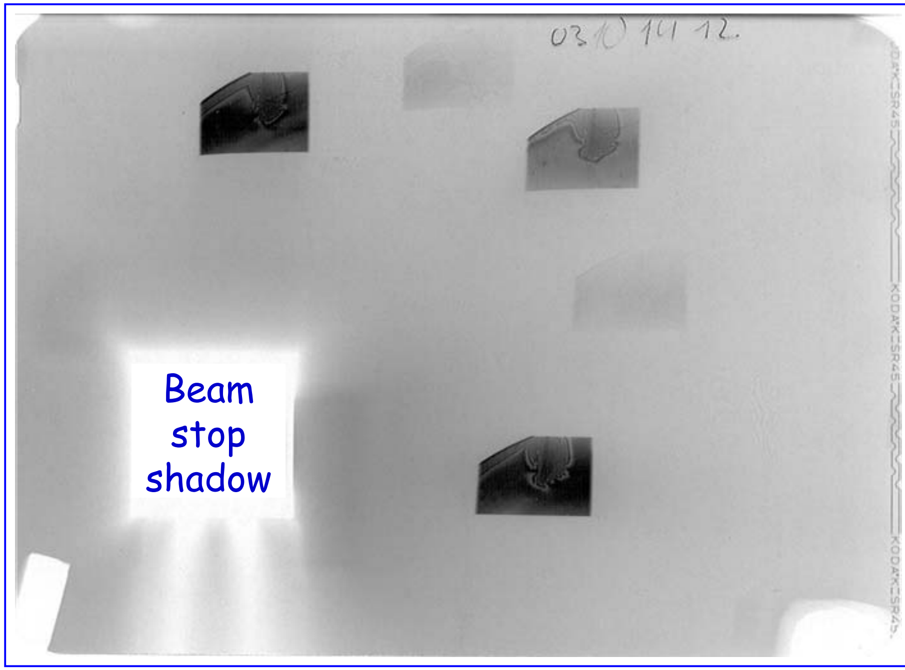
integrated wave topography - plane monochromatic wave topography

white beam topography

most popular 'integrated wave technique' at synchrotrons



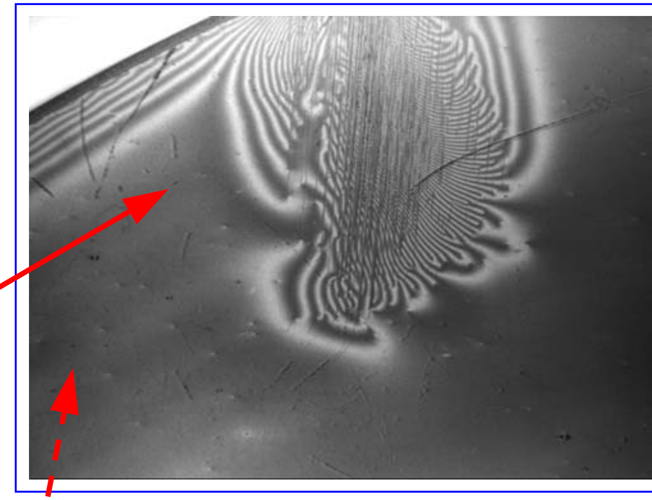
example



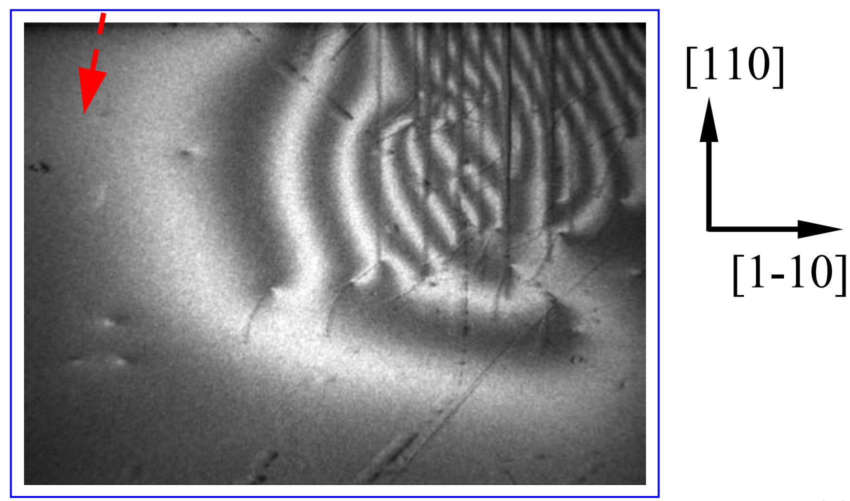
Film with Laue spots

part of a low dose SIMOX* wafer

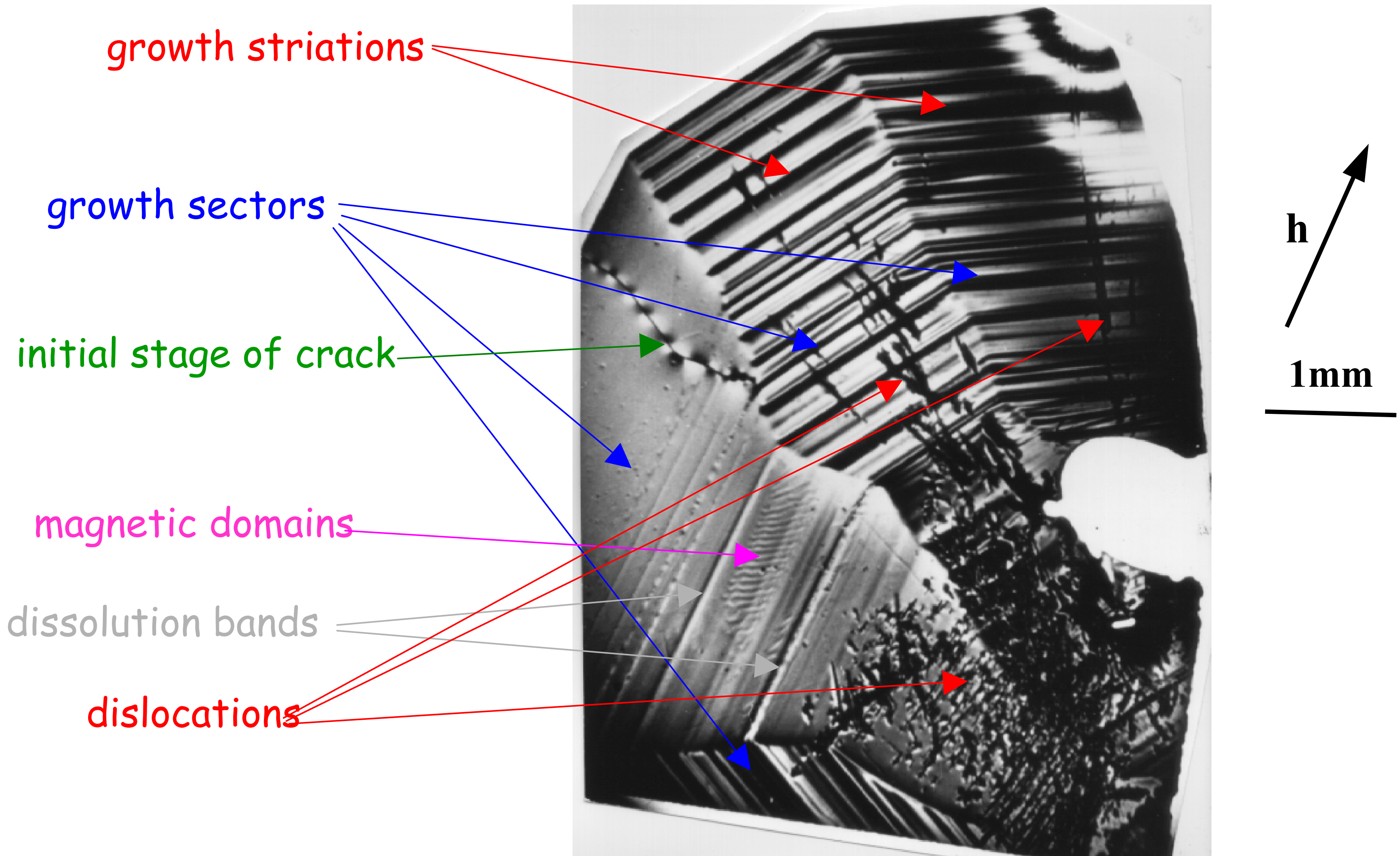
*Separation by IMplantation of OXygen



One individual “spot”
- a topograph



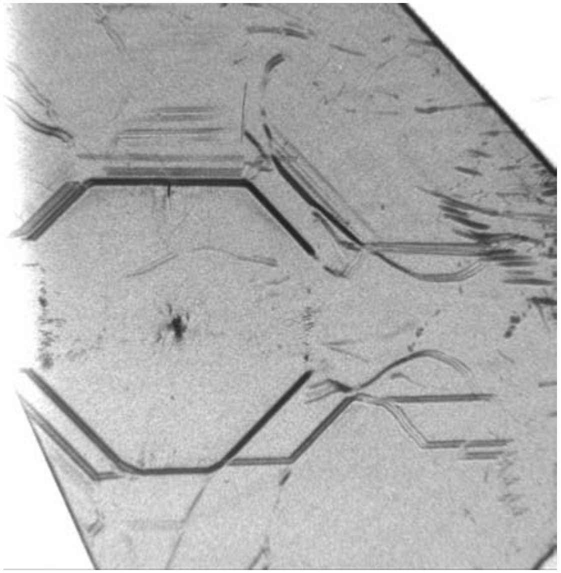
a nightmare topograph...

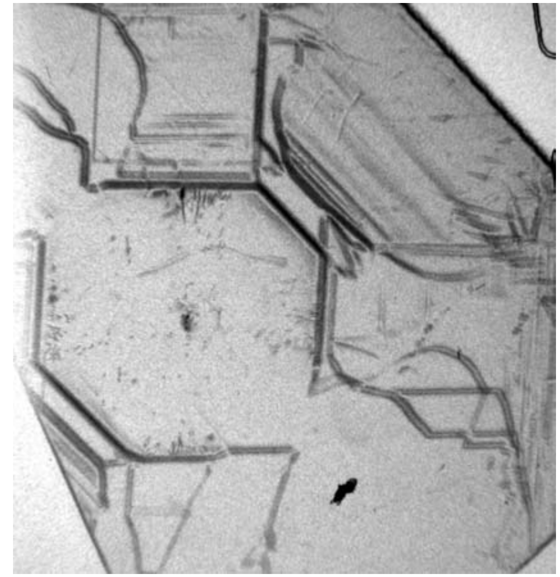


Transmission X-ray topograph of a flux grown Ga-YIG ($Y_3Fe_{5-x}Ga_xO_{12}$, x ≈1) crystal plate, $MoK_{\alpha 1}$-radiation (λ=0.709 Å), 44-4 reflection

HPHT synthetic diamond example

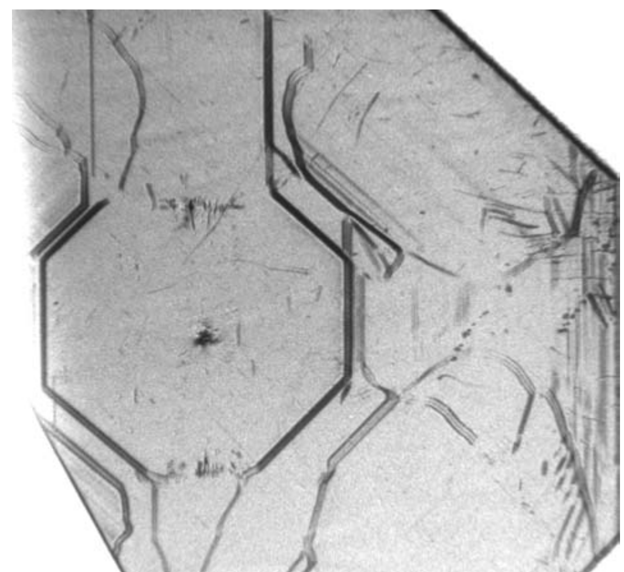
[220]





[400]

2 mm



$[2\bar{2}0]$

Ib diamond, 100-oriented

transmission geometry

Advantages

Simple set-up

in principle no sample orientation necessary
(no knowledge of sample orientation necessary, sample environment, crystal growth)

several reflections simultaneously (extinction of contrast)

all crystal parts (also misoriented ones) visible simultaneously

Drawbacks...

fluorescence radiation background (detector/film fogging)

sensitive only to lattice plane inclination ('misorientation' contrast)

limited sensitivity to weak distortions ($\sim 10^{-6}$)

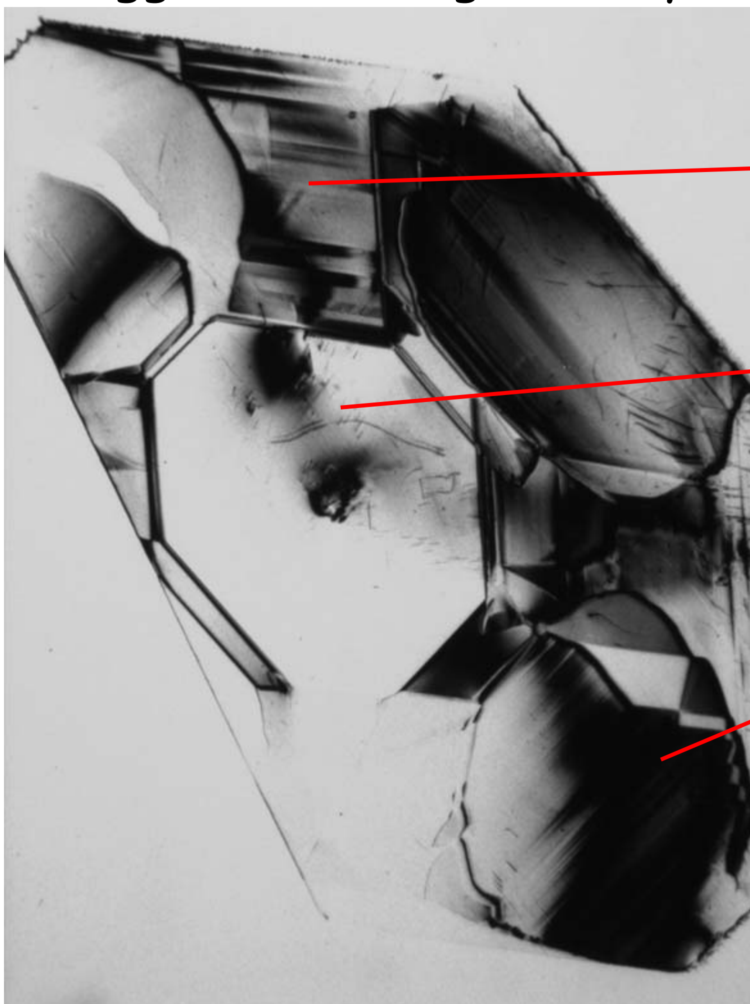
quantitative analysis complicated

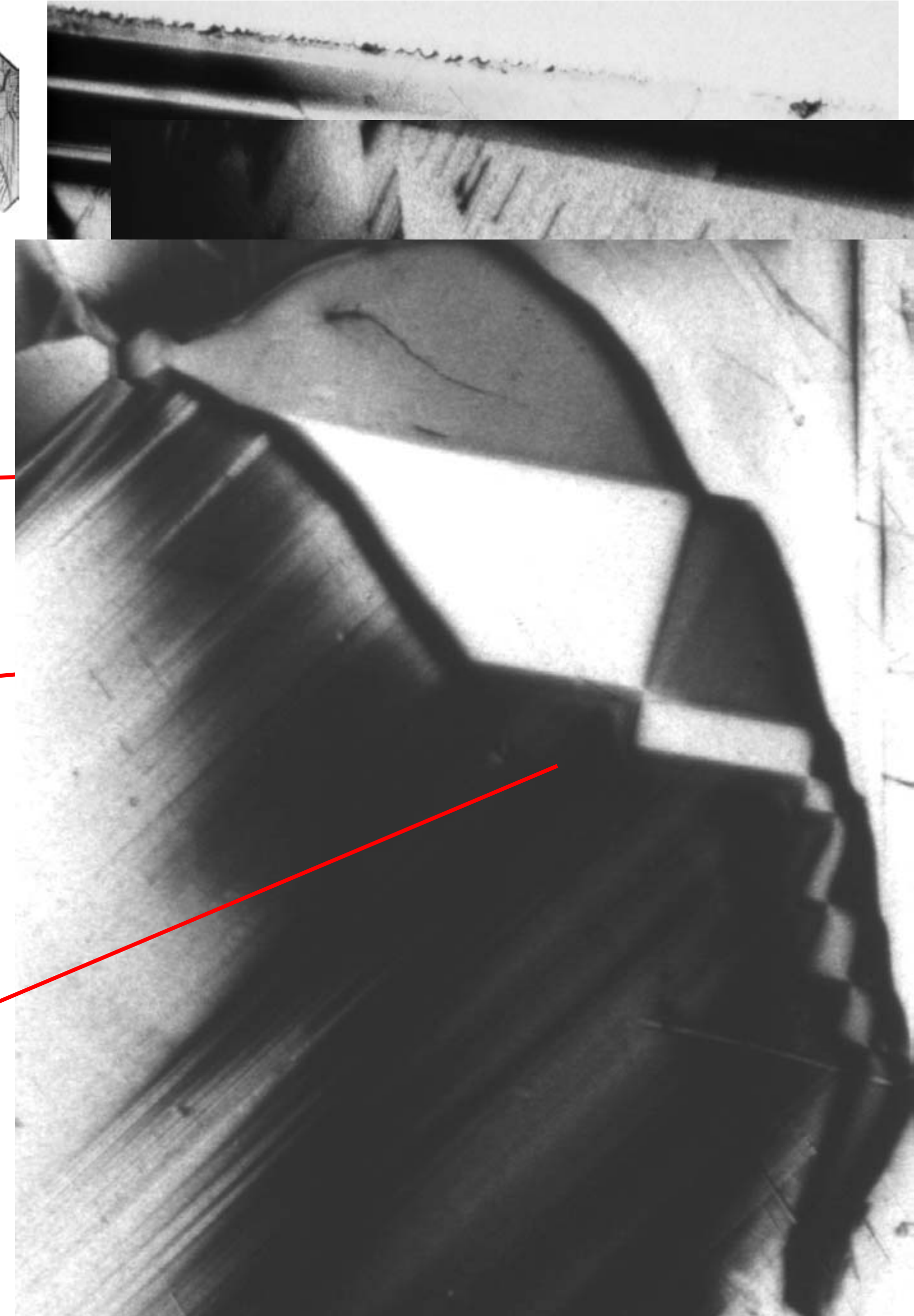
...Solution

use monochromatic and/or plane waves

plane wave topography (same HPHT diamond)

Bragg reflection geometry)





Advantages

sensitive to weak and strong distortions
(last example: from ~10^{-7} … ~ 10^{-5})

monochromatic beam images may show much more details than white beam topographs

contrasts 'easy' to calculate

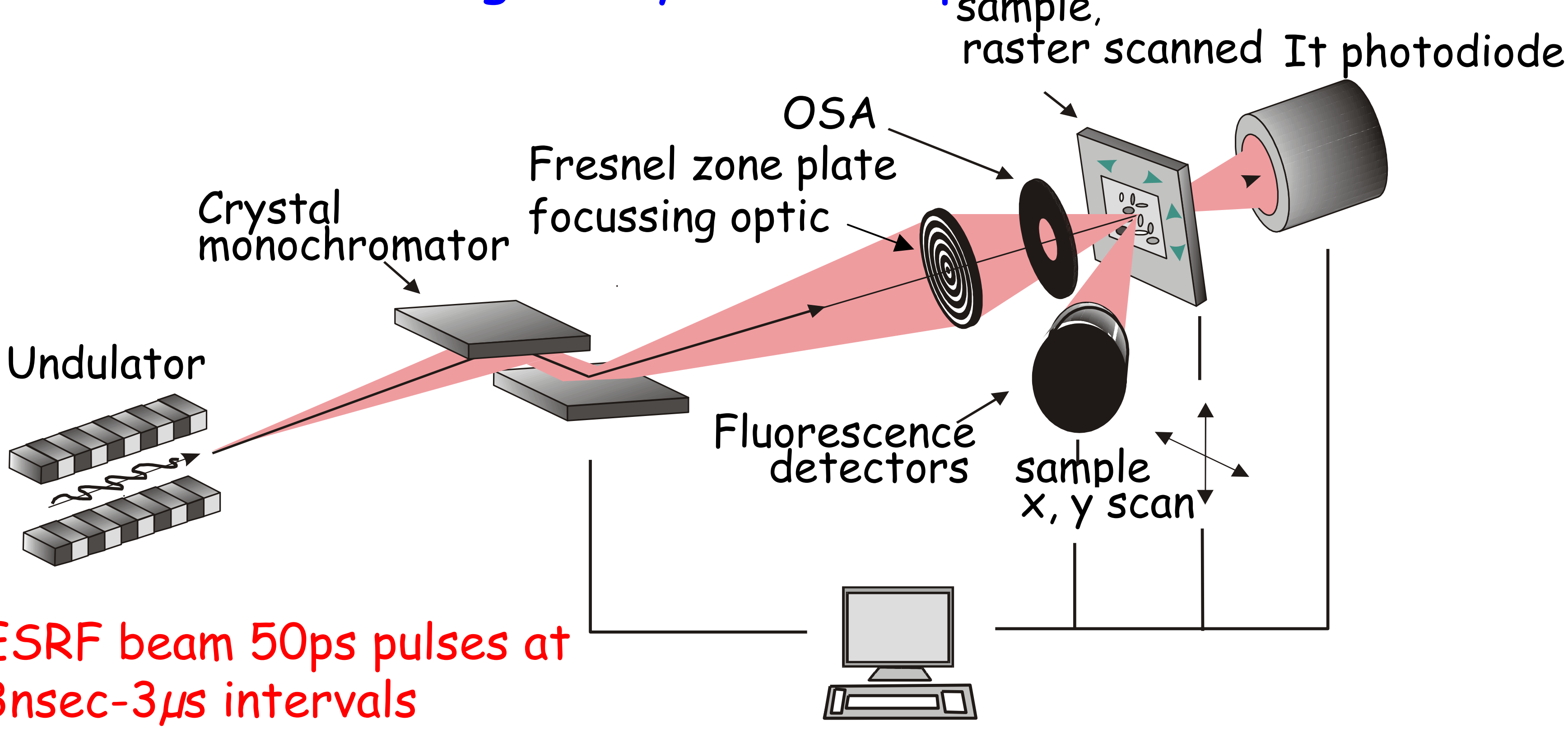
Drawbacks

only one reflection per crystal orientation

practical set-up more demanding

Characterization of 'material-detectors'

ESRF ID21 scanning X-ray microscope



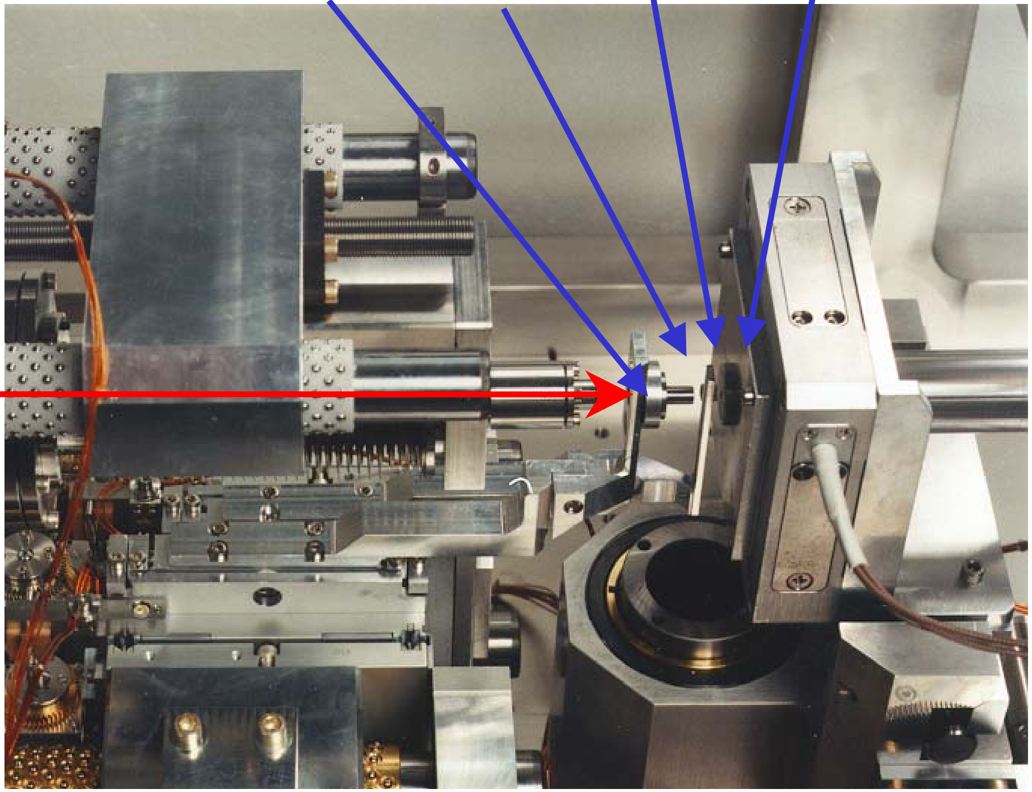
ESRF beam 50ps pulses at
3nsec-3μs intervals

A Fresnel zone plate focuses the 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).

→ direct charge/current probe of detector (2 - 6keV)

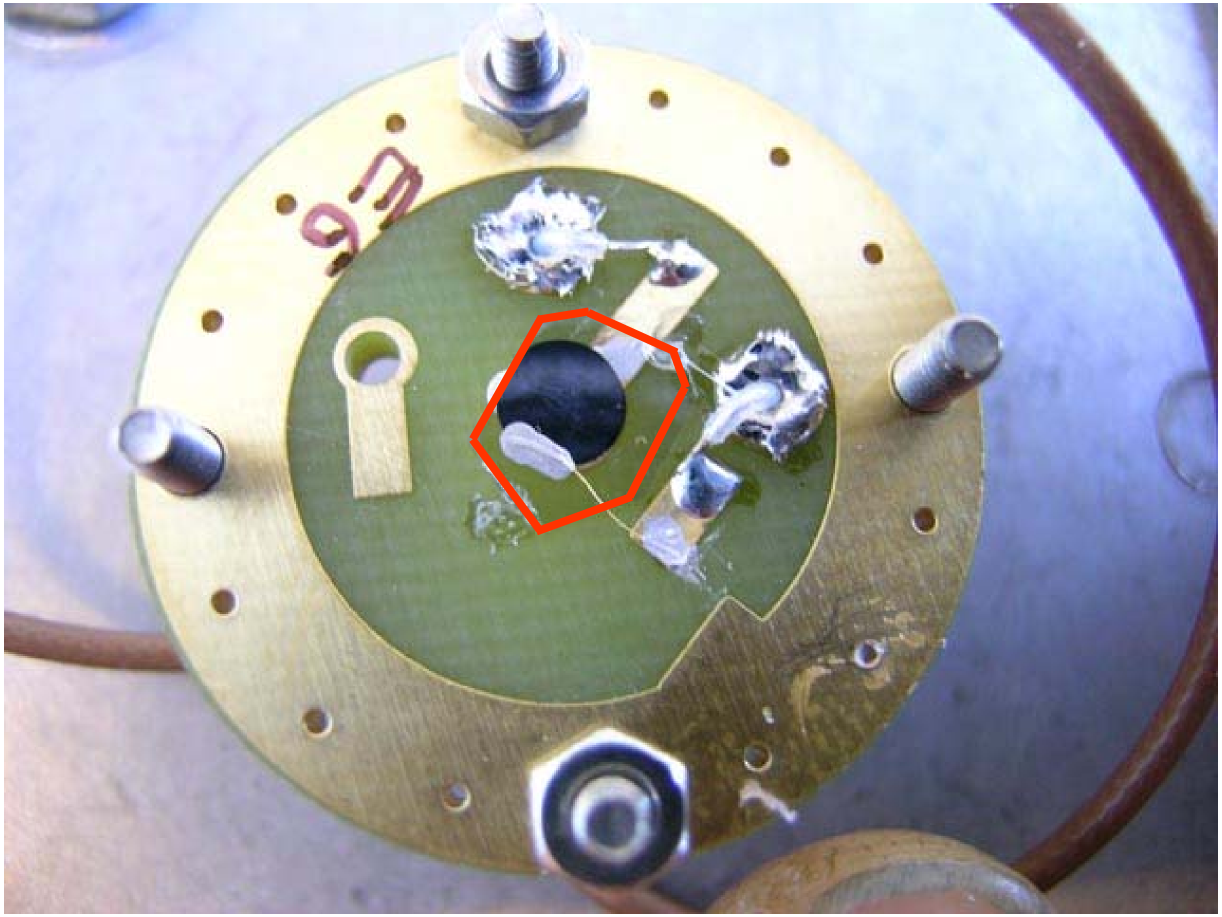
ESRF ID21 X-ray microscope

'limited access'



the entire microscope can be operated under high vacuum.

E6-70310 tests, ID21-ESRF May 18-19, 2004.



Φ4mm contacts 20nm Ti + 100nm Al (P. Muret, LEPES)
RF- PCB mount (Φ31mm) to suit ID21 sample holder

Other methods of analysis in Grenoble

Raman (confocal) micro scanning

Visible/UV microscanning fluorescence spectrometry

at LEPMI-ENSEEG

fluorescence spectrometry (cryogenic)

visible - hard UV excitation

at LEPES-CNRS

Summary/Conclusions

1. *single crystal* material overcomes some limitations of polycrystalline material, *but uniformity is still an issue*

   SC material is still in early test-evaluation phase

2. must develop/test surface preparation and improve 'primitive' electrical contact technologies

   radiation hardness ?

3. *ESRF* can provide exceptional characterization tools

   material: quality-homogeneity
   detector: efficiency & spatial-temporal response