



Photon metrology using Synchrotron light sources

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Outline

Synchrotron radiation Light Sources Beamlines – controlling the beam Photon metrology – basic characterization **Defect metrology** Pump and probe techniques XAFS metrology **Response functions** Surface topology and metrology



- High brightness and high intensity, 10 orders of magnitude more than that of X-rays produced in conventional X-ray tubes.
- The production mechanism is unique in that it can be precisely described.
- High level of polarization (linear or elliptical).
- High collimation, small angular divergence of the beam.
- Low emittance the product of source cross section and solid angle of emission is small.
- Wide tunability in energy/wavelength by monochromatization (from sub-eV up to MeV).
- High brilliance, exceeding other natural and artificial light sources by many orders of magnitude.
- Pulsed light emission with durations of 1 ns or less

esa Synchrotron (Ivaneko-Pomeranchuk) radiation

Classically an electron moving in a magnetic field will execute a spiral trajectory and radiate as a dipole. The emission is isotropic at the Larmor frequency

$$v_L = \frac{eB}{2\pi m_o c} = 2.8 \text{ MHz per Gauss} ,$$
 $F = q (\vec{E} + \vec{V} \times \vec{B})$

where *B* represents the magnetic field component perpendicular to the particle velocity vector.

If the electron is non-relativistic, the radiation is isotropic and is emitted only at the Larmor frequency. This is known as cyclotron radiation. In the relativistic case, synchrotron radiation is emitted in a relativistically narrow cone of angle, $\theta \sim \gamma^{-1}$, where γ is the particle energy in units of its rest energy (typically $10^3 - 10^4$). The frequency distribution is no longer discrete as in the non-relativistic case, but is an asymmetric distribution with a maximum of the envelope at

$$v_m = 2/3\gamma^2 v_L$$

or in terms of energy
 $E_m = 5 \times 10^{-9} \gamma^2 B$ (keV)

D. Iwanenko, I. Pomeranchuk, On the maximal energy attainable in betatron, Phys. Rev., 65 (1944) 343

J. Schwinger, "On the Classical Radiation of Accelerated Electrons", Phys. Rev., **75** (1949)1912.



Synchrotron sources

Crab Nebula



First light observed 1054 AD

"Song Shi", official annals of the Song dynasty, chapter 12.

GE synchrotron, New York State



First light observed 1947 AD Elder et al., "Radiation from Electrons in a Synchrotron", Phys. Rev., 71(1947) 829



The Crab pulsar



Chandra (0.3-3) keV

Hubble (optical)

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The Crab pulsar



Chandra (0.3-3) keV

Hubble (optical)

NDIP Lyon 2011



Practical implementation

Synchrotron radiation is electromagnetic radiation produced by relativistic charged particles accelerated in circular orbits



Synchrotron radiation is emitted tangentially at each magnetic device in the form of horizontal layers (shown in yellow) with very little divergence. It can extracted via windows situated regularly along the length of the ring. Each beam feeds into a measurement station dedicated to a particular material analysis technique



Light sources

At present, there are over 50 synchrotron light sources operating world wide which can be broadly grouped into three categories, or generations.

<u>1st generation synchrotron radiation light sources</u> which were operated partially for synchrotron radiation and partially for other programs, such as high energy physics. Typically the light source was parasitic on the primary program. Examples include the CHESS facility at Cornell, USA and DORIS at DESY, Germany.

<u>2nd generation synchrotron radiation light sources</u> which are dedicated synchrotron radiation facilities but are not designed for low emittance or with straight sections for insertion devices. Examples include the Daresbury SRS in the UK and HASYLAB at DESY.

<u>3rd generation synchrotron radiation light sources</u>, which are dedicated synchrotron radiation facilities designed for low emittance and with many straight sections for incorporating insertion devices. Examples include Soleil & ESRF in France, the ALS in the USA, BESSY II in Germany and Diamond in the UK.

Considerable effort is now underway developing fourth-generation light sources, which will most likely combine a hard X-ray (wavelength less than 1Å) free-electron laser (FEL) with a very long undulator in a high-energy electron linear accelerator. Such a device would have a peak brightness many orders of magnitude beyond that of the third-generation sources, as well as pulse lengths of 100 fs or shorter, and be fully coherent.



Diamond Light Source, Harwell, UK





Each generation differs from the previous generation by innovation and is improved by at least an order of magnitude in performance, usually quantified by the flux and the brilliance of the source. The flux is defined as

$$\Phi = \frac{N_p}{0.1\% \, mrad}$$

(photons/(s, 0.1 % energy spread, mrad horizontally)

where N_p is the number of photons emitted per second for a given stored beam current.

The brilliance, *B*, is the peak flux density in phase space,

$$B = \frac{N_p}{0.1\%, mm^2, mrad^2}$$

The flux is a function only of the electron current and energy, while the brilliance takes into account the phase space defined by diffraction effects and the electron beam emittance.





Properties of the beam

The beam emerging from the bending magnets is known as "white light" and has a well defined energy spectrum extending from the microwave through to the hard X-ray regions of the electromagnetic spectrum. The spectrum is usually characterized by its critical energy, E_c , which is defined as the energy at which half the radiant power is carried by photons above E_c . The critical energy is given by

 $E_{\rm c}({\rm keV}) = 0.665 E^2/\rho$,

where *E* is electron beam energy in GeV and ρ is the bending radius. Critical energies generally ranges from ~10 keV for second generation machines to ~50 keV for third generation machines (usable end energy ~4E_c).

White light direct from an extraction point is extremely intense with a brilliance of

- ~ 10¹⁵ photons s⁻¹mm⁻²mrad⁻² (second generation machines)
- ~ 10²⁰ photons s⁻¹mm⁻²mrad⁻² (third generation machines)

For comparison a rotating anode X-ray generator has a brilliance

~ 10⁹ photons s⁻¹mm⁻²mrad⁻²



<u>Bottom line</u>: White light fluxes at a detector can be up to10¹⁴ photons cm⁻²

which will seriously melt it – count rates need to be in the range $\sim 10^2 - 10^5$ photons s⁻¹.



Flux reduction strategies

Absorbers	pros	cons	
	simple	difficult to control harmonic amplification	
Collimation/ Aperture reduction			
Pinholes Knife edges	very precise simple to implement	diffraction, difficult to align, leak above ~20 keV	
Monochromaterizing			
	very effective reduces flux by many orders of magnitude	usefulness and energy range depend on quality of monochromater	
Detuning the rocking curve			
	very effective also suppresses harmonics	need MOSTAB	
Low current			
circulate less electrons	the best simple beam decay correction	affects whole light source users become unfriendly	



Harmonics



Since $2d\sin\theta = n\lambda$

the monochromator transmits not only the desired fundamental energy (n=1), but also higher harmonics of that energy.

Si crystals with a diamond structure (space group Fd3m(Oh7)) will not produce harmonics that satisfy the eq. h+k+l=n, where *n* is twice an odd number.

Common methods of reducing harmonic Xray content include

- detuning the second crystal
- using a harmonic rejection mirror
- work at a high enough energy

Typically, when two Si(111) crystals are detuned by 50% on the rocking curve, the intensity of the third harmonic is reduced by a factor of 10^3 .



Beamline layout



Beamline X-1 HASYLAB, DESY Hamburg

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HASYLAB measurements

Preamp -

Slits

Beam

Detector

6

CdZnTe reference detector

_ Laser ref. (detached)

0

23

BAYY DETECTOR

TOOTAX W

2

Mechanical interface

Y stage



HASYLAB measurements

Slits

14-38

SiC Detector

an a

BSI

Cd Zn Ring Det X-Y stage

Ring Detector

CdTe reference Detector

beam

3

0

9



Insertion devices – Wigglers and Undulators



For K << 1 the oscillation amplitude of the motion is small and the radiation displays interference patterns which lead to narrow energy bands. If K >> 1 the oscillation amplitude is bigger and the radiation contributions from each field period sum up independently, leading to a broad energy spectrum. In this regime the device is no longer called an *undulator*, it is called a *wiggler*.

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Injector

ASTRID-2

Booster ring

Beamlines

RF cavity

Storage ring

Bending magnet

Undulator

Transfer beamline

esa Enhancing the energy range - Insertion devices



ESRF High energy spectra taken on beamline ID15

- Gamma-ray energies achieved using 2 insertion devices (asymmetrical multipole wiggler (AMPW) & superconducting wavelength shifter (SCWS)
- Energy range from 30 keV to 1 MeV
- Difficult to separate the monochromatic beam from white light above 800 keV



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Examples - basic detector characterization

800



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SiC detector – low energy response





SiC array development





diode construction

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esa Count rate distribution at 12 keV – quick look



Single point resolution 1.7 keV

BESSY II SiC efficiency measurements 12th Dec 2006



esa Efficiency and depletion depth determination

(data corrected for dead layers, air path, window thickness and dead time) (harmonics can be used)





Pump and probe techniques

Investigation of polarization effects in compound semiconductors

Defined as a time dependent variation in the detectors properties, such as count rate, charge collection and resolving power. Seems to be correlated with

- material properties (purity, stoichiometry)
- temperature
- high resistivity
- dielectric constant
- Correlated with the total energy deposition per unit time
- Particularly acute in groups II-VI and I-VII compounds (TIBr, CdTe, CdZnTe, Hgl₂)

Its treatment has been largely anecdotal and qualitative.

Recently, a 1 D semiempirical model was proposed by Bale and Szeles, Phys. Rev. B **77**, 035205 (2008) in which polarization was presumed to be caused by deep hole trapping

Based on "pump and probe" measurements Kozerezov et al have developed a full 3 D analytical model of polarization (Kozorezov et al. "*Polarization effects in thallium bromide x-ray detectors*", J. Appl. Phys., **108** (2010) 064507)



Polarization in TIBr X-ray detectors

which are 1 y, TIBr ph Br crystals

2.8 x 2.8 .x 1 mm³ TIBr planar detector Au contacts $V_b=840V$ T=-30-+23°C

Polarization effect in TIBr





Left: composite energy loss spectra covering the energy range 10-100 keV. Right: a series of "snapshots" of the 60 keV lineshape with exposure time under a constant illumination of 10³ photons s⁻¹.

The pump and probe technique





Evolution of peak parameters

60 keV pump and probe



Kozorezov et al. "High resolution study of polarisation effects in thallium bromide X-ray detectors", RTSD 2011



Kozorezov et al. "High resolution study of polarisation effects in thallium bromide X-ray detectors", RTSD 2011

Simulation of line-shape

$$Q = Q(\mathbf{x}_a) = -eN_0 \exp\left(-\frac{t^*(\mathbf{x}_a)}{\tau_e}\right) - eN_0 \int_0^\infty dt' \left[\frac{1}{\tau_e} \exp\left(-\frac{t'(\mathbf{x}_a)}{\tau_e}\right) \frac{z_e(t', \mathbf{x}_a)}{L} - \frac{1}{\tau_h} \exp\left(-\frac{t'(\mathbf{x}_a)}{\tau_h}\right) \frac{z_h(t', \mathbf{x}_a)}{L}\right]$$

$$S(\mathbf{x}_a, Q) = \frac{1}{\sqrt{2\pi}\sigma(\mathbf{x}_a)} \exp\left[-\frac{(Q - Q(\mathbf{x}_a))^2}{2\sigma^2(\mathbf{x}_a)}\right]$$



$$S(Q) = \int_0^L \mathrm{d}z P_s(z, E) S(z, \mathbf{r}_a, Q)$$

Line shapes near the pinch point



RTSD R16-3



Preliminary polarization results

Observations

- The spatial extent of the polarized region is much larger that the initial pump region.
- In TIBr and CdZnTe, the overall lineshape is very sensitive to the hole $\mu\tau$ product
- While peak channel and CCE, recovery on time scales of hours, spectral resolution and particularly the line shape recover on time scales of days.

Conclusions

- The pump and probe technique is an ideal technique to investigate dynamic processes and their evolution.
- The primary cause of polarization effects is due to the charging of deep traps by one of the carriers.
- The build-up of space charge perturbs of the internal electric field affecting the collection of the other carrier.
- At the "pinch" point the induced field due to the space charge is equal to the applied field resulting in a catastrophic loss of performance
- De-polarization proceeds through thermal recombination and cannot be effectively achieved through modulation of the bias voltage as previously thought.

<u>Next steps</u> – use 3D analytical model to derive hole mu-tau products, trap densities, occupancy rates and ionization energies \rightarrow Identify the particular trap(s) involved.



Defect metrology



Crystal defects - primer

Twins	 grain boundary defect, in which a crystal is joined to its mirror image – size up to ~ ~mm
Dislocation	 line defect that may run the length of the crystal – size up to ~mm
Grain boundarie	es – Boundary between two crystals in a polycrystalline solid size up to ~ ~mm
Voids	– macroscopic holes in the lattice - size up to ~mm
Inclusions	- regions of a different phase, size 1-10's of micron
Precipitates	- small regions of a different phase, size up to ~micron



Macroscopic defects



a) Optical images of two $50 \times 50 \text{ mm}^2$, 3 mm thick slices of a CdZnTe crystal grown by the High Pressure Bridgman method. Numerous grain boundaries and twins are apparent in the image. *b*) The crystals count rate response, measured with a ⁵⁷Co radioactive source is shown in the lower images, illustrating poor charge collection at the grain boundaries.





Crystal defects

The X-ray world with finer spatial resolution







FIG. 4. (Color online) X-ray maps evaluated for the same area of the device, but with different spatial resolutions: 10×10 , 20×20 , 100×100 , and $200 \times 200 \ \mu m^2$.



Crystal defects

Finer spatial resolution



IR and X-ray mages of Te inclusions/precipitates measured in a 1-mm thick CZT crystal. The lower images are X-ray maps when of the crystal when operated as a simple planar detector. The dark spots in this case correspond to a drop in the detector response, demonstrating the link between precipitates and poor device performance. The scans were performed by using a $10 \times 10 \ \mu m^2$, 85 keV X-ray beam.

IR transmission microscopy

IR system allows us to take "in-depth" images of Te inclusions and collapse them on a single plane. We can do a 3-D reconstruction of images. We can also measure sizes and concentration of Te inclusions.



Brookhaven Science Associates U.S. Department of Energy

Bolotnikov et al., Extended Defects in CdZnTe Radiation Detectors, IEEE Trans Nucl. Sci, 56 (2009) 1775.



X (mm) C. Hansson, ESA internal progress report 2011

Small area/ high resolution scans - R Large

esa



- 24 inclusions were observed in region LR2 (5) and LR3 (19)

- 18 inclusions were investigated with respect to spectral response.

C. Hansson, ESA internal progress report 2011



see poster by Hansson



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Basic spectral response (60keV)



The basic response when monitoring the spectral evolution across the inclusion was a reduction in peak centroid up to the centre and a recovery when moving out of the inclusion.



R2 inclusion 1



C. Hansson, ESA internal progress report 2011



X-ray mapping using synchrotron radiation provides a very powerful technique to investigate crystal defects

- Inclusion/precipitates acquire a space charge and modify the local electric field
- Measured spectrum within an inclusion shifts to lower energies
- Counting efficiency and FWHM of the photopeak remain largely unchanged, however
- CCE's are reduced by ~10%
- Double peak structure seen at high energies (depths)
- No correlation between peakshift and inclusion size
- Near field devices could act as X-ray microscopes
- Next steps: await input from UL

X-ray measurements with CVD material (MTPVT) – expect only voids





1 inch right circular crystals coupled to XP2060B 10-stage PMTs



Scintillator studies - non-proportionality



Owens et al., *The hard X-ray response of Ce-doped lanthanu halide scintillators*, Nucl. Instr. Meth.,**574** (2007) 158

Khodyuk et al., *Improved scintillation proportionality and energy resolution of LaBr3:Ce at 80K*, Nucl. Instr. Meth., **642** (2011) 75.

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Khodyuk et al., Improved scintillation proportionality and energy resolution of LaBr3:Ce at 80K, Nucl. Instr. Meth., 642 (2011) 75.

esa Non-proportionality - K-dip spectroscopy (TU Delft)

Can obtain high accuracies down to 30eV. More accurate than CCT below 3keV



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Khodyuk, et al. Nonproportional scintillation response of NaI:TI to low energy x-ray photons and electrons, J. Appl. Phys., 107(2010) 113513 2 European Space Agency



XAFS structure



Little evidence for extended fine structure suggesting that the bulk of the structure arises from the local coordination environment. Lanthanum halides crystallize with hexagonal symmetry around a. It is assumed that when doped, the Ce atoms uniformly replace the La atoms—and this could be a source of disorder leading to a suppressed EXAFS signal.

NDIP Lyon 2011 Owens et al., The hard X-ray response of Ce-doped lanthanum halide scintillators, Nucl. Instr. Meth., 574 (2007) 158 European Space Agency



X-ray Absorption Spectroscopy and Metrology



XAFS and metrology





The Broad Band X-ray Telescope (BBXRT) was flown on the space shuttle Columbia (STS-35) on December 2-11, 1990, as part of the ASTRO-1 payload. The flight of BBXRT marked the first opportunity for performing X-ray observations over a broad energy range (0.3 to 12 keV) with moderate energy resolution (150 eV at 6 keV). The BBXRT consists of a pair of coaligned thin foil conical X-ray mirrors, with a cryogenically-cooled, Si(Li) spectrometer at the focus of each.



X-ray Absorption Fine Structure



FIG. 1.—Measured BBXRT spectrum of the Crab Nebula. The solid line is the best-fit absorbed power law through the data points. The bottom panel shows the residual count rate after the underlying continuum has been subtracted, illustrating dramatically the effects of XAFS. Features at energies appropriate for the Si, Al, and O edges can be delineated clearly in the residuals.

Owens et al., The Effect of X-Ray Absorption Fine Structure in Soft X-ray Astronomical Telescopes, Ap. J., **476** (1997) 924 NDIP Lyon 2011

esa What is X-ray Absorption Fine Structure?

X-ray Absorption Fine Structure (XAFS) occurs due to interference effects as photoelectrons leave the surface of a material following photoabsorption. The interference depends on the interatomic distance between the atom that ejected the photoelectron and the nearest neighbour atom, and to lesser extents the next nearest neighbour and other atoms.





XAS spectra are usually divided in three energy regions

Edge – the edge including the white line

XANES – X-ray Absorption Near Edge Structure attributed to multiple scattering processes leading to structure within about 50 eV of the edge. Much larger signal than EXAFS

Sensitive to electronic structure and symmetry

EXAFS – Extended X-ray Absorption Fine Structure attributed to single scattering processed leading to oscillatory structure up to ~few hundred eV above the edge. Influence over larger energy ranges than XANES

Sensitive to bond distances, coordination numbers and local disorder



Post BBXRT – The X-ray observatories



XMM-Newton:

- Energy range 0.1 15 keV
- \cdot Mirror area 0.4 m²
- Focal length 7.5 m
- Spatial resolution 15" HEW
- Energy resolution 130 eV @ 5.9 keV
- Δ E/E = 500 @ 0.5 keV
- Limiting sensitivity: 10⁻¹⁵ erg cm⁻² s⁻¹



Chandra:

- Energy range 0.1 10 keV
- Mirror area 0.08 m²
- Focal length 10 m
- Spatial resolution 0.5" HEW
- Energy resolution 130 eV @ 5.9 keV
- ∆E/E = 400-1000 @ 0.5 keV
- Limiting sensitivity: 10⁻¹⁶ erg cm⁻² s⁻¹



X-ray transmission, absorption and reflection



FIG. 3.—The measured CCD quantum efficiency for 1–4 pixel events. For comparison, we show also individual discrete line measurements (*open circles*) along with Monte Carlo calculations (*lines*). The inset shows an expansion in the region of the Si K-edge. As expected, the quantum efficiency above the edge shows considerably more structure than that expected from silicon alone, showing clear evidence of a strong SiO₂ component. For comparison, we show also in the region of the edge predicted from standard atomic and nuclear data tables and on newly measured cross sections.



FIG. 5.—Predicted JET-X mirror reflectivity for an angle of incidence of 27:375 based on single reflectivity Au measurements. For comparison, we show also calculated values based on the calculations of Cromer & Liberman (1970).

The Effect of X-Ray Absorption Fine Structure in Soft X-ray Astronomical Telescopes, Ap. J., **476** (1997) 924

Apparent discrepancy between measured and tabulated M absorption edge energies, Ap.J., **468** (1996) 451

Woo, EXAFS and XANES: New Astrophysical Tools to Study the Solid State Structure of Interstellar Grains, Ap. J., **447** (1995) L129

esa Understanding the response function, spectral unfolding



FIG. 6.—The full-up effective area for the JET-X telescope. Two curves are given one for which the detailed edge structure was input from direct measurement (*thin line*) and the other in which the edge shapes were calculated using cross sections (*thick line*).

Fig. 7.—Measured energy-loss spectrum for a 10^5 observation of a 10 millicrab source. The solid line shows the folded source model through the measured response function. The residuals (i.e., measured spectra – model spectra) shown below are for two cases: (a) where the data have been unfolded using the CAFS response function and (b) where the data have been unfolded using the Cassical response function (see text).





Figure 4: Spectra (*upper panels*) and residuals in units of data/model ratio (*lower panels*) when the Crab spectra of Obs.#0160960401 (*left panel*) and 0160960601 (*right panel*) are fit with a photoelectrically absorbed power-law (cf. Tab. 2). For plotting purposes only, spectra are rebinned in such a way that each spectral channel has got a signal-to-noise >10.



Launch 10th December 1999

esa

XAFS, surface metrology and process control



Simplified cross-sectional view

Single pixel of an EEV large area $20.7 \times 27.6 \text{ mm}^2$; multi-pixel (768 × 1024) three-phase front-illuminated MOS X-ray CCD From the depletion region outwards: SiO₂ 850Å (100%) passivation layer, Si₃N₄ (850Å) dielectric layer, SiO₂ 8000Å (50%), Si 1700Å (P3), thermal oxide (100Å)

A. Owens, Nuclear Instruments and Methods in Physics Research A 526 (2004) 391-398



Step 1- Measure the quantum efficiency

Low current run - full area illumination





SRS beamline 3.4, InSb[111]





Step 2 – measure the near edge cross sections



Fig. 1. The experimental configuration of beamline 3.4 at the Daresbury SRS.







Fig. 3. The derived linear attenuation coefficients across the Si K-edge. For Si, the letters c and a refer to crystalline and amorphous. We also show the 'classical' Si curve based on the calculation of Cromer and Liberman (1970).

2000

NDIP Lyon 2011 A. Owens et al., Near K-edge linear attenuation coefficients for Si, SiO₂ and Si₃N₄ Rad. Phys. & Chem., 65 (2002) 109. European Space Agency



Step 3 – fit the quantum efficiency



Simplified CCD geometry assumed in the model.

Comparison of the thicknesses of surface structures specified by the manufacturer and those derived by best-fitting the measured quantum efficiency

Structure	Manufacturers spec. (µm)	Best fit (µm)
P1, P2	0.65 ± 0.13	0.51 ± 0.10
P3	0.17 ± 0.03	0.26 ± 0.07
Vapox + LTO	1.2 ± 0.24	1.06 ± 0.11
P3 SiO ₂	$< 0.01^{a}$	0.11 ± 0.04

^a Re-measured to be 0.1 μ m.

Precisions of 1%

Factor of 2 better than process control

Combined with XPS, possible to tomographically isolate features



