Tutorial VV:

Neutron and X-rays — Sources, Instrumentation, and Scattering

Sunday, November 25
1:30 pm - 5:00 pm
Hynes Convention Center, Level 2, Room 208

Instructors:
Klaus-Dieter Liss, Australian Nuclear Science and Technology Organisation
Rozaliya Barabash, Oak Ridge National Laboratory

Tutorial VV:

Part 1:
Sources, Instrumentation, Basic Diffraction Techniques

Klaus-Dieter Liss
Australian Nuclear Science and Technology Organisation

ansto
Nuclear-based science benefiting all Australians

Tutorial VV:

Part 2:
Advanced Diffraction: Understanding Local Structure by Probing Reciprocal Space

Rozaliya Barabash
Oak Ridge National Laboratory

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The intention of the tutorial is to give the attendees an awareness of and fundamental background on evolving sources, new and developing methods, unfamiliar concepts, and the ability to view diffraction methods in a larger concept.

Topics

Sources, Instrumentation, Basic Diffraction Techniques — Klaus-Dieter Liss

Advanced Diffraction: Understanding Local Structure by Probing Reciprocal Space — Rozaliya Barabash

The course will cover modern and evolving neutron and hard X-ray sources, including nuclear reactors and spallation sources, X-ray tube, synchrotrons, free-electron lasers and energy-recovery linacs. Applications and instrumentation will be focused on diffraction methods, examining overlapping or combined techniques such as simultaneous spatial or spectroscopic information. The concept of state-of-the-art diffraction will be introduced, treated in reciprocal space, and displayed on comparable scales throughout the different kinds of radiation and techniques such as powder diffraction, single-crystal diffraction, pair distribution analysis, reciprocal space mapping, etc. Interrelations between different kinds of defects and their diffraction patterns will be discussed.

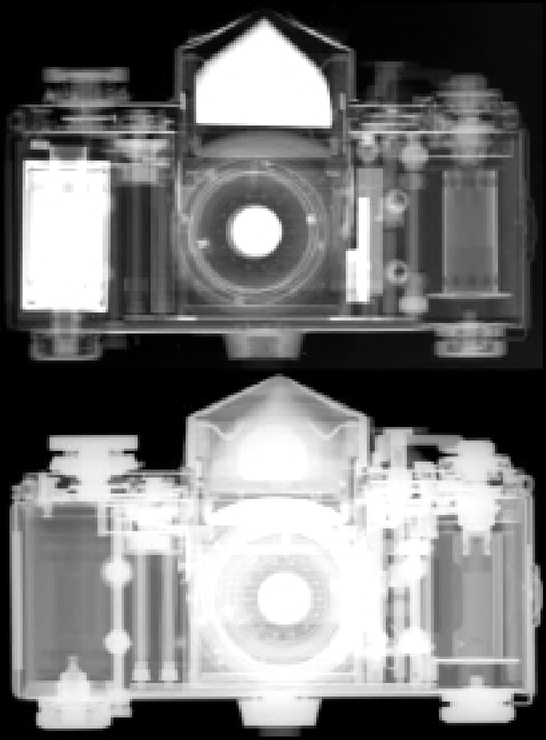
Exotic cases and transition regions, such as dynamic versus kinematic theory will be explored.

Overview – Part 1

- Neutrons and X-rays
  - properties, complementarity
- Characteristics of sources
- Neutron sources
  - nuclear reactor, spallation source
- X-ray sources
  - synchrotron radiation, emerging sources
- Diffraction
  - Laue equation, reciprocal space
  - selected diffraction techniques
- Conclusion - Part 1
  - further lookup and reading

Neutrons and X-rays

- similarities:
  - penetrating radiation
    - tomography
    - radiography
  - wavelength of the order of atomic distances
    - diffraction as a wave
    - probes for atomic scale structures
  - spectroscopic aspects
    - probes for excitations in matter

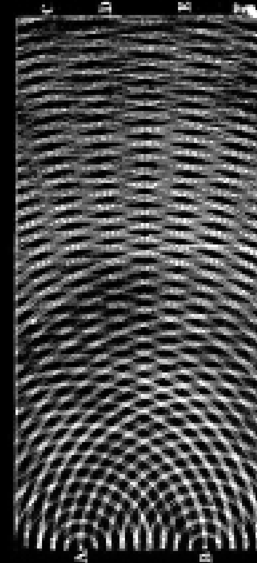


Radiograph of an analog camera by neutrons (top) by X-rays (bottom)

Courtesy of Eberhard Lehmann, PSI

http://www.psi.ch/niag/what-is-neutron-imaging

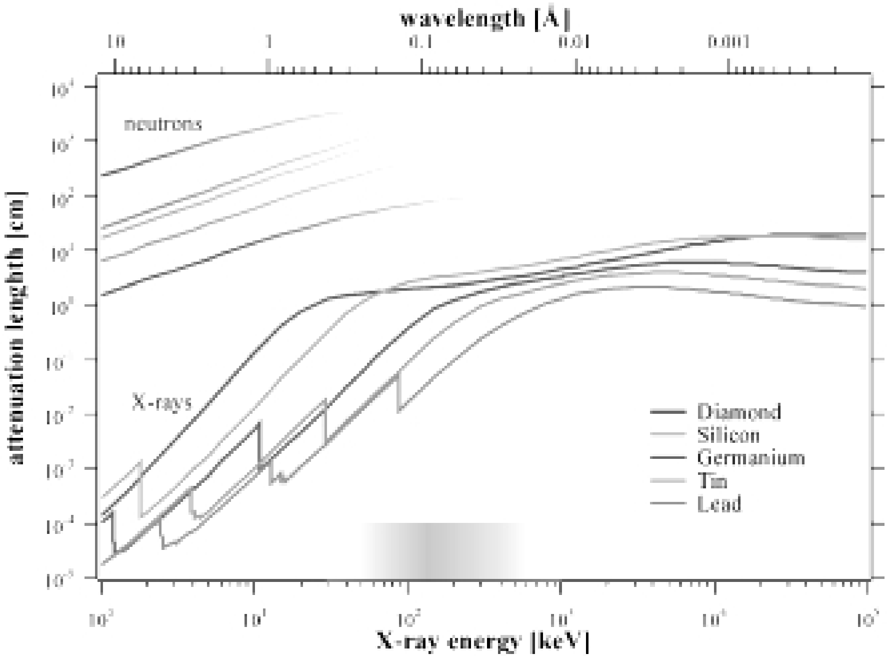
Neutrons and X-rays



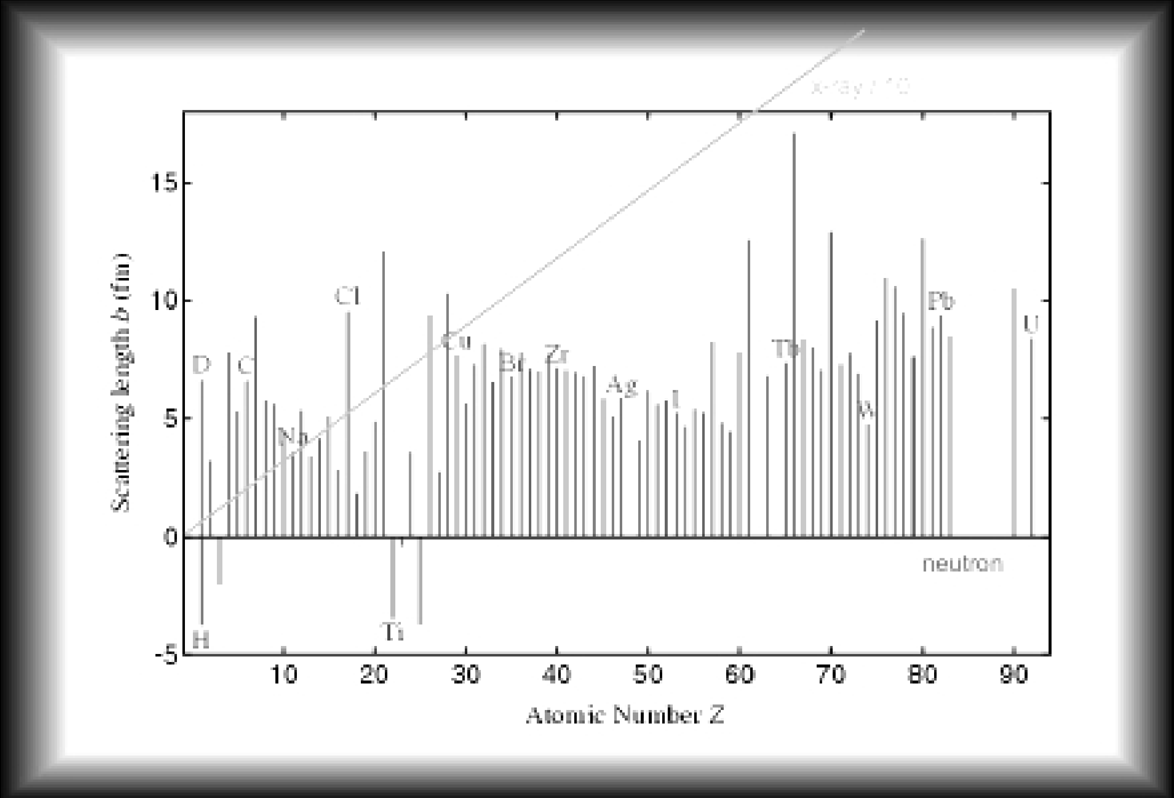
- complementarity:
  - scattering contrast
    - X-rays scatter on electron charge distribution
      - electro-magnetic interaction
    - neutrons scatter on nuclei and magnetic moments
      - strong interaction and magnetic interaction
  - wavelength of the order of atomic distances
    - diffraction as a wave
    - probes for atomic scale structures
  - spectroscopic aspects
    - probes for excitations in matter
      - neutrons more sensitive to collective excitations, phonons, magnons
      - X-rays more for electronic excitations, fluorescence, asorption ...

Figures: top – Young's double slit experiment (source: wikipedia)
bottom: interference of 2 spherical water waves (photo: Liss, Cooper Creek)

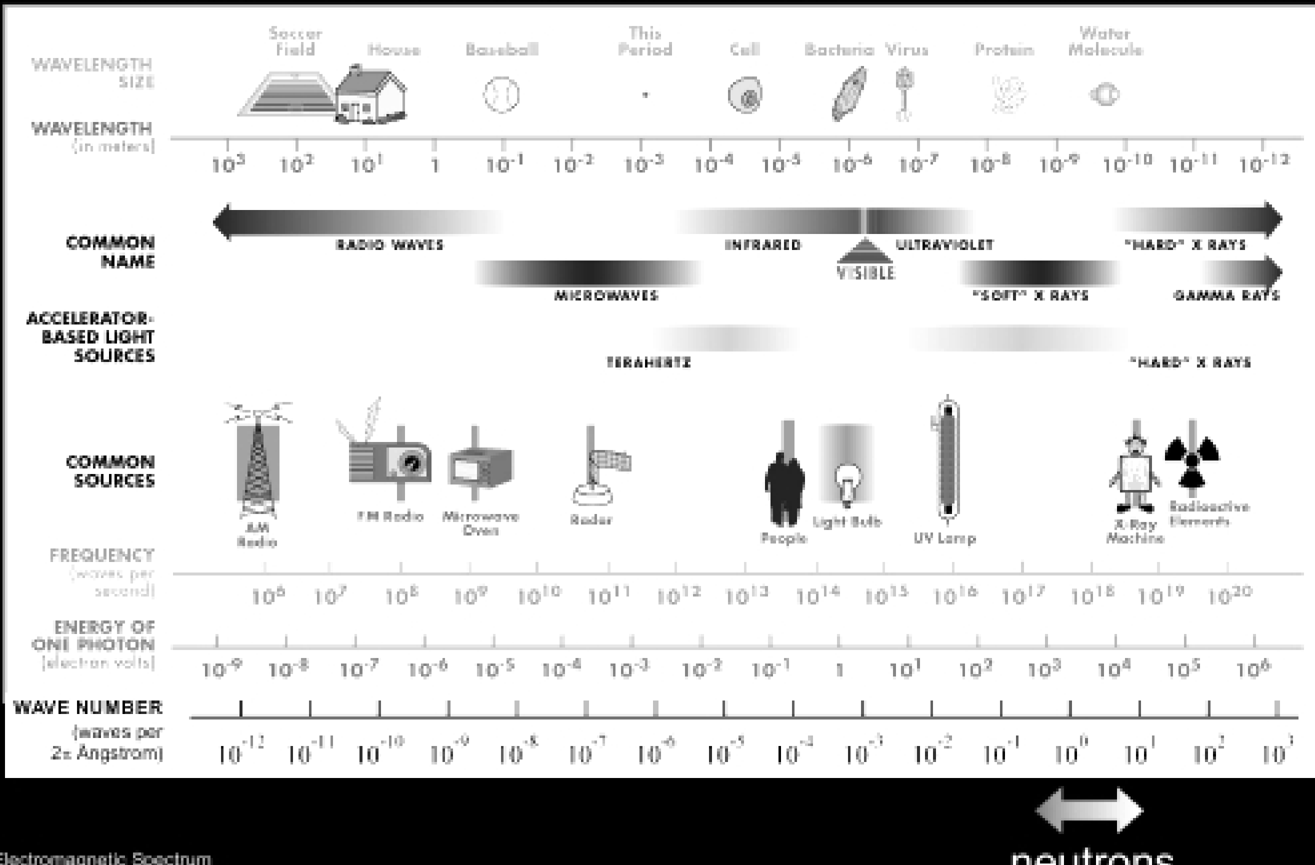
Attenuation lengths



Scattering amplitudes



THE ELECTROMAGNETIC SPECTRUM



Electromagnetic Spectrum
Courtesy: Advanced Light Source

Neutrons and X-rays

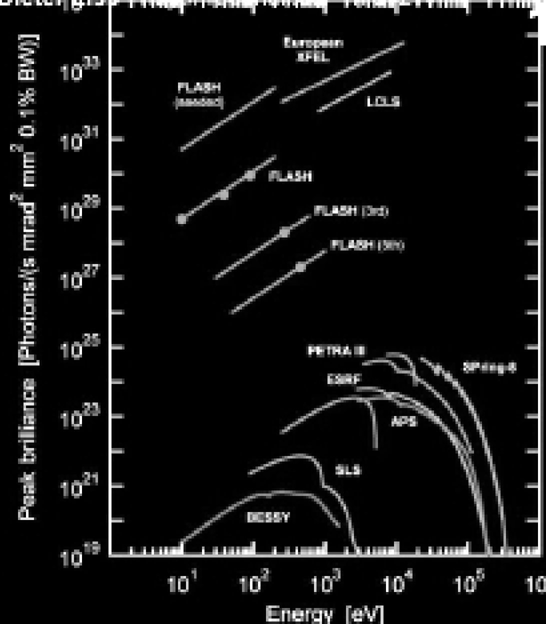
probe	**neutrons**	**X-rays (synchrotron)**
scattering contrast coherent scattering length	varies statistically for each isotope b_c [fm], (+,-)	proportional to electron density $Z\ r_e$ [fm], (+)
magnetism coupling	very sensitive spin coupling, vectorial	very weak second order, interference terms
brilliance beam size / time resolution	low cm / sec, min, h	high μm / ps, μs, s
structural determination light elements in heavy matrix	good grain statistics advantages for light elements / H,D	very high resolution H,D not seen, heavy elements predominant
contrast variation	through choice of isotope	at resonances
collective excitations phonons, magnons	very sensitive, high energy resolution at large momentum transfers	difficult to achieve only in first Brillouin zone
incoherent spectroscopy	n activation analysis	fluorescence, absorption Compton scattering
interaction	nuclear magnetic	electro-magnetic
dispersion-relation $E = \hbar\ \omega(k)$	quadratic ($E = \hbar^2 k^2 / 2m$)	linear ($E = \hbar\ c\ k$)

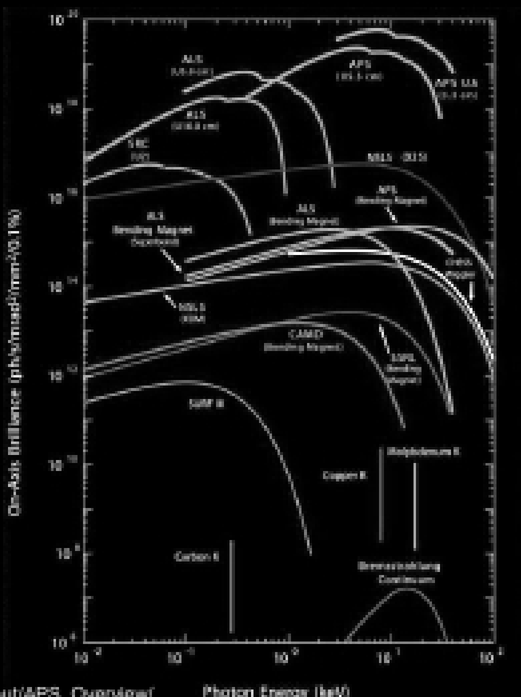
Brightness & Fluxes for Neutron & X-Ray Sources

	Brightness *(s^{-1} m^{-2} $ster^{-1}$)*	*dE/E* *(%)*	*Divergence* *($mrad^2$)*	*Flux* *(s^{-1} m^{-2})*
Neutrons	10^{15}	2	10 x 10	10^{11}
Rotating Anode	10^{16}	3	0.5 x 10	5×10^{10}
Bending Magnet	10^{24}	0.01	0.1 x 5	5×10^{17}
Wiggler	10^{26}	0.01	0.1 x 1	10^{19}
Undulator (APS)	10^{33}	0.01	0.01 x 0.1	10^{24}

after
Roger Pynn: *Neutron Scattering, Lecture 1*, Los Alamos
http://www.physics.indiana.edu/~p637/notes/

X-ray Brilliances





source:
Advanced Photon Source
http://www.aps.anl.gov/About/APS_Overview/

source:
DESY – Flash Facility Information
http://hasylab.desy.de/facilities/flash/

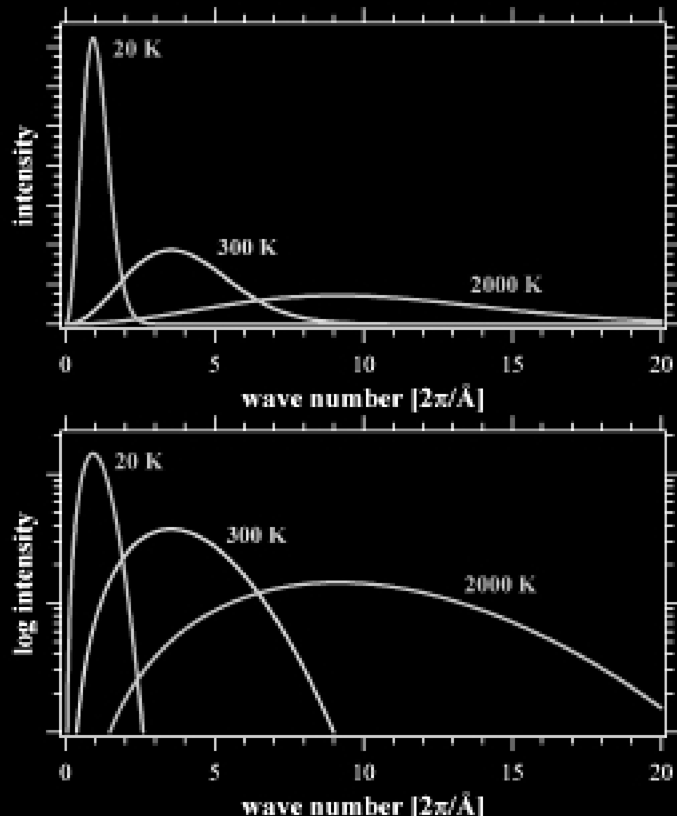
Neutron Temperature

- moderated neutrons
  - Maxwellian distribution
    - cold neutrons ~20 K
    - thermal neutrons ~300 K
    - hot neutrons ~2000 K

$$f(v)=\sqrt{\frac{2}{\pi}\left(\frac{m}{k_B T}\right)^3}\, v^2 \exp\left(-\frac{m v^2}{2 k_B T}\right)$$

$$v=\frac{\hbar}{m}k$$

$$k=2\pi/\lambda$$



Reactor vs Spallation Neutrons

- Reactor
  - continuous flux
  - stable source
  - typical angular dispersive
  - good high-Q resolution

- Spallation
  - pulsed beam
  - time of flight experiments
  - high peak flux
  - but poor peak shape

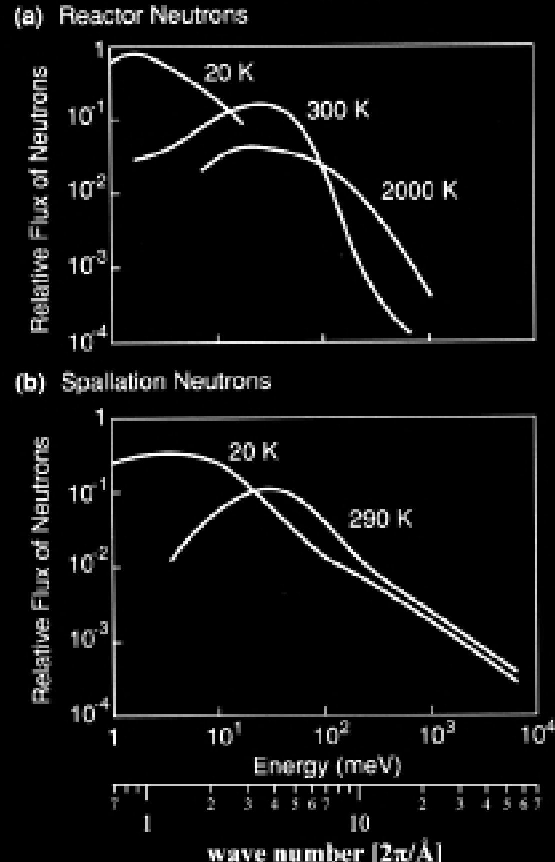
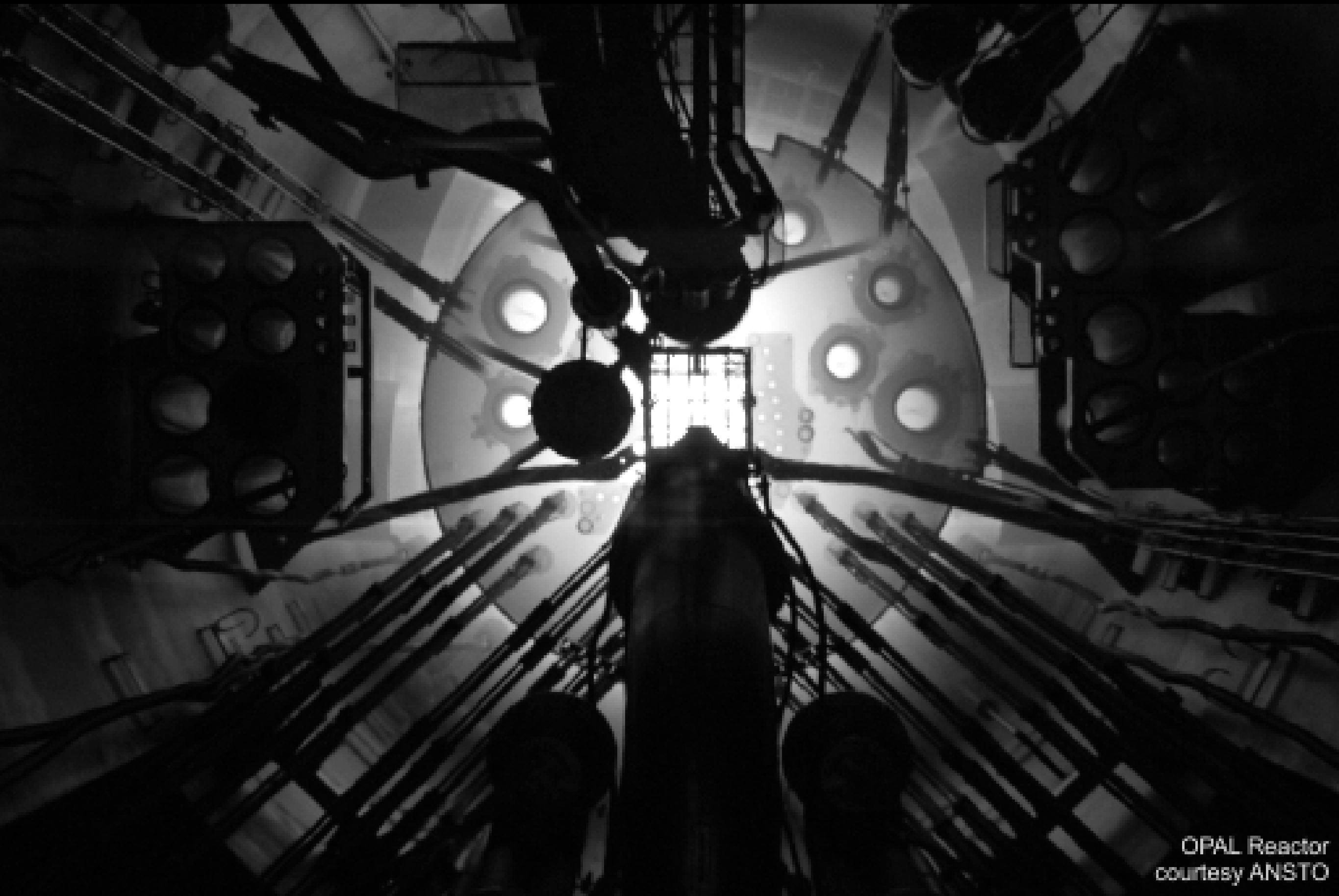


figure after: Roger Pynn: *Neutron Production, Lecture* Los Alamos

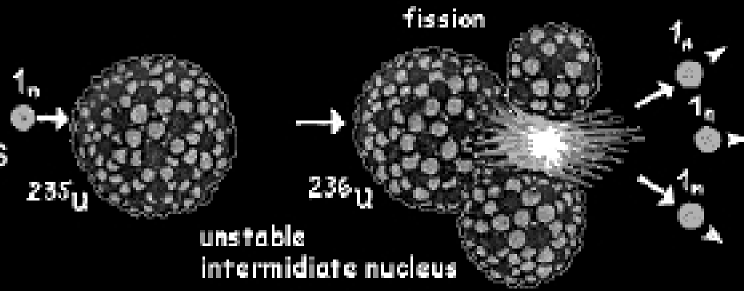
Neutron Source: Nuclear Reactor

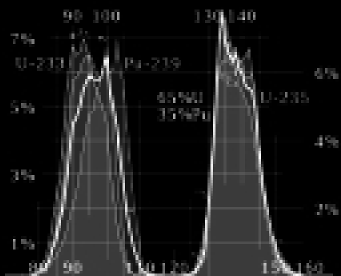


OPAL Reactor
courtesy ANSTO

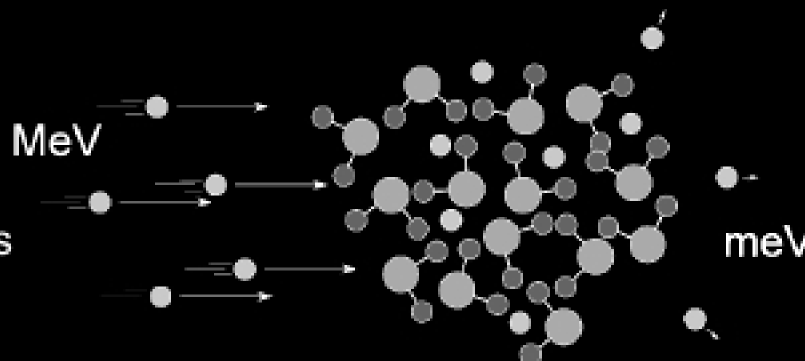
Neutron Source: Nuclear Reactor

- neutron production
  - fission
    - high energy neutrons



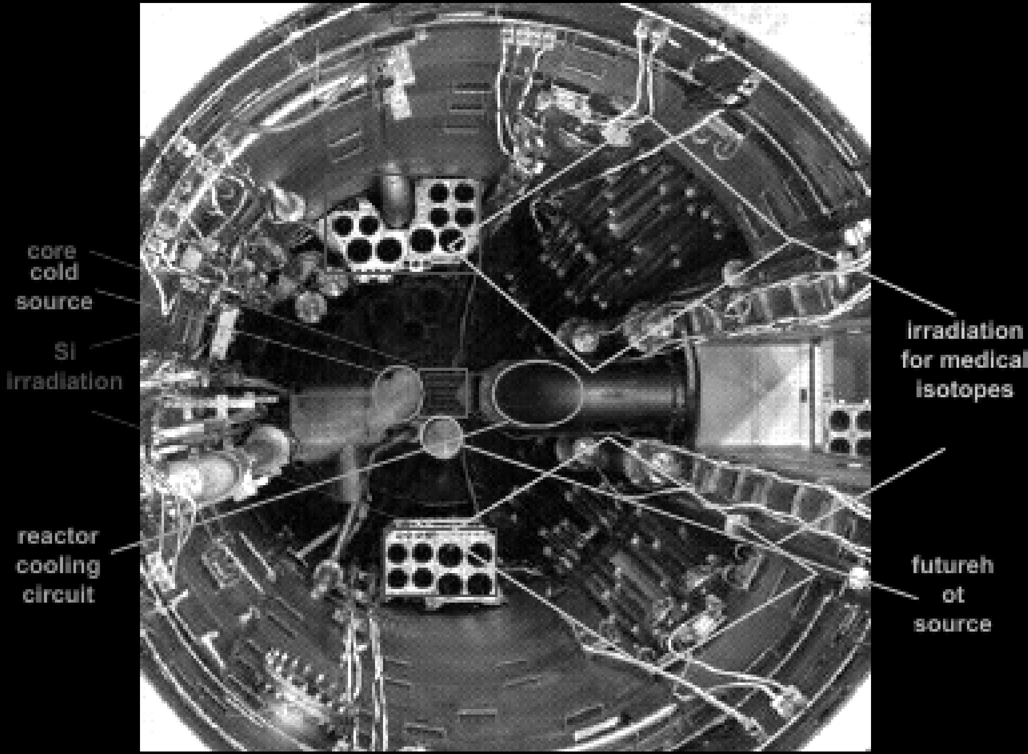


  - moderation
    - thermalized neutrons



the H_2O Billiard

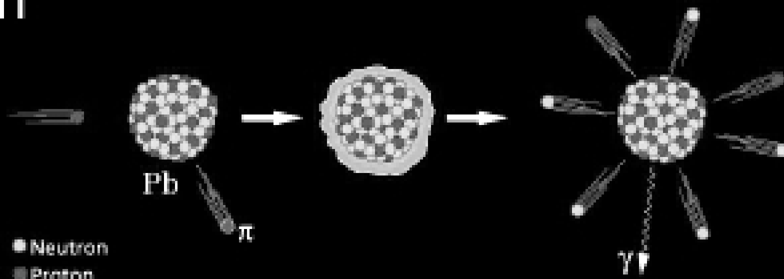
OPAL Reactor, Australia

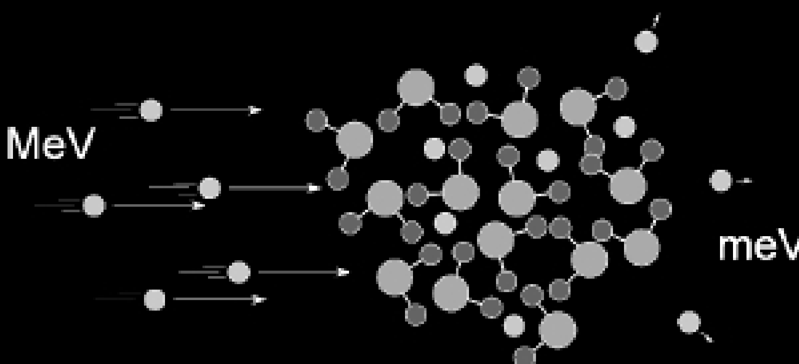


OPAL Reactor
courtesy ANSTO

Neutron Source: Spallation

- neutron production
  - spallation
    - high energy protons
    - evaporate neutrons
    - short pulses
  - needs accelerator
  - moderation
    - pulse shape
    - compromise

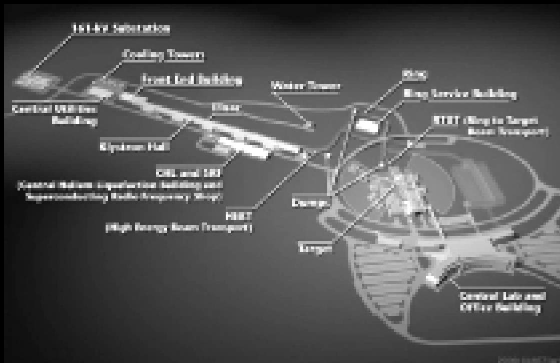


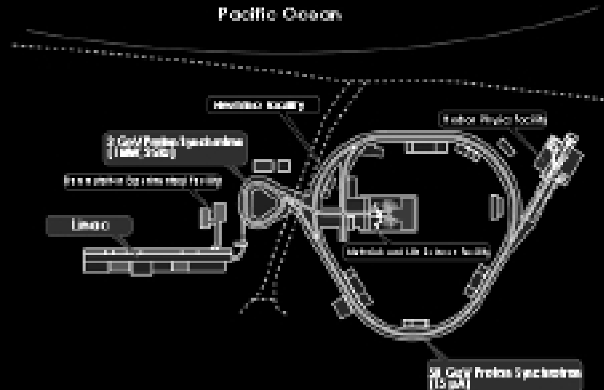


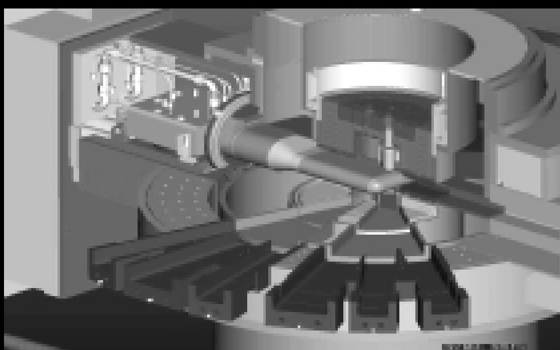
the H_2O Billiard

image source (top):
Paul Scherrer Institute
http://www.psi.ch/media/neutron-source-sinq

Spallation Source







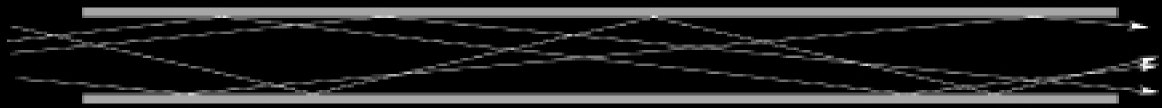
Spallation Neutron Source, SNS
courtesy SNS

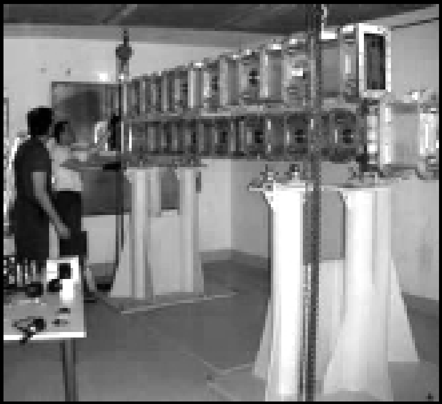
J-PARC, Japan
courtesy J-PARC

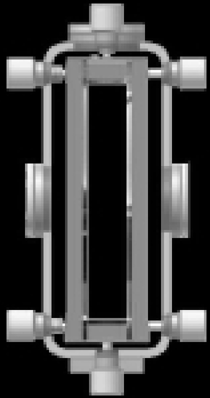
Reactor or Spallation Source?

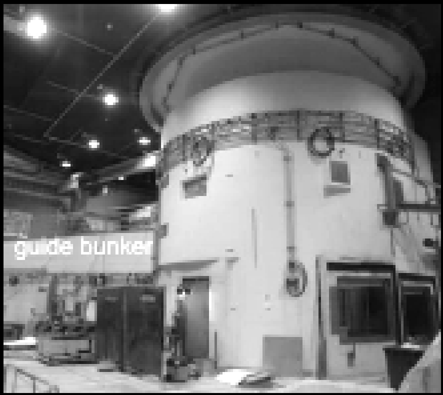
Short Pulse Spallation Source	Reactor
Energy deposited per useful neutron is ~20 MeV	Energy deposited per useful neutron is ~ 180 MeV
Neutron spectrum is "slowing down" spectrum – preserves short pulses	Neutron spectrum is Maxwellian
Constant, small $\delta\lambda/\lambda$ at large neutron energy => excellent resolution especially at large Q and E	Resolution can be more easily tailored to experimental requirements
Copious "hot" neutrons=> very good for measurements at large Q and E	Large flux of cold neutrons => very good for measuring large objects and slow dynamics
Low background between pulses => good signal to noise	Pulse rate for TOF can be optimized independently for different spectrometers
Single pulse experiments possible	Neutron polarization has been easier

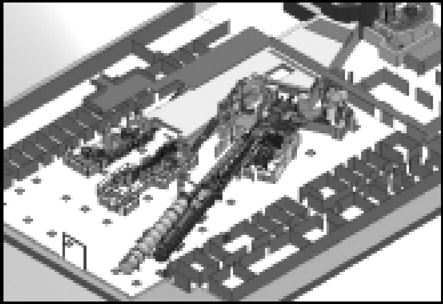
Neutron Transport - Guides





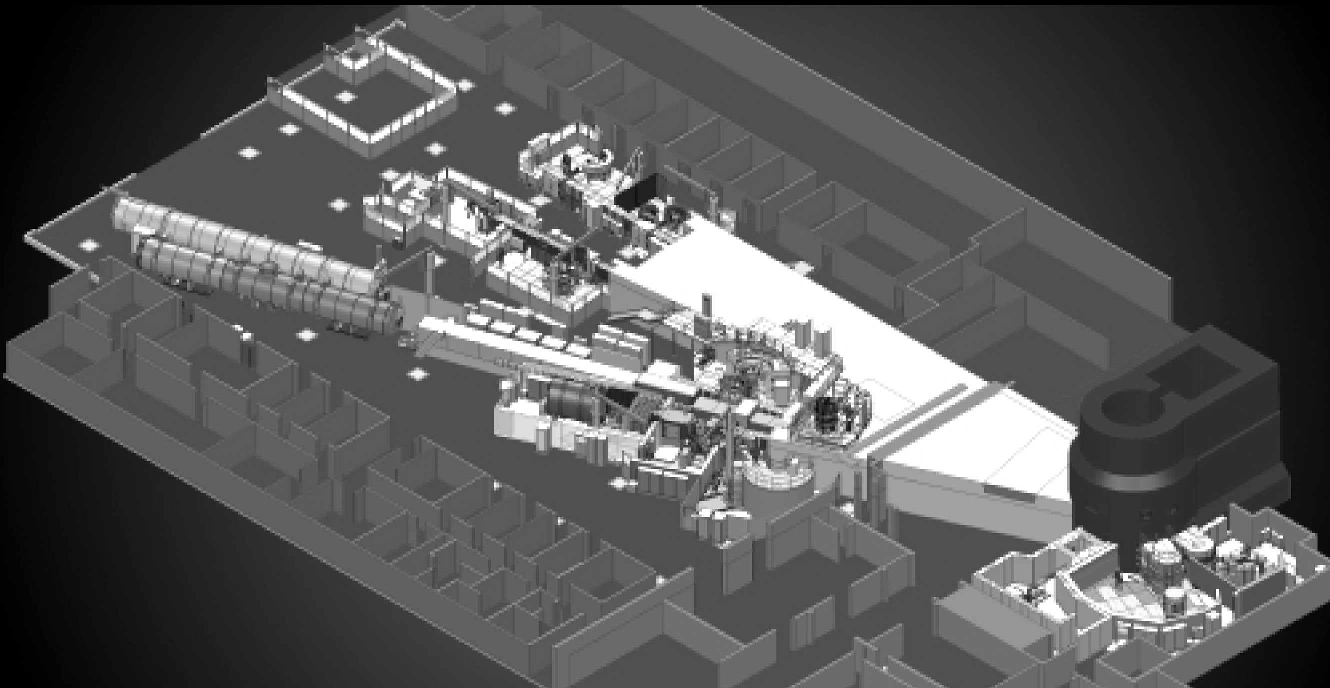






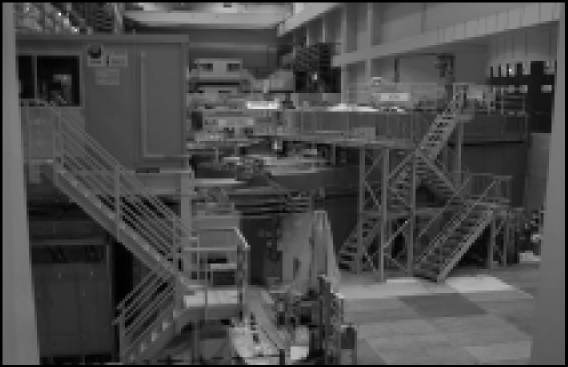
OPAL Reactor
courtesy ANSTO

OPAL Reactor Facility



OPAL Reactor
courtesy ANSTO

Pictures at Neutron Sources

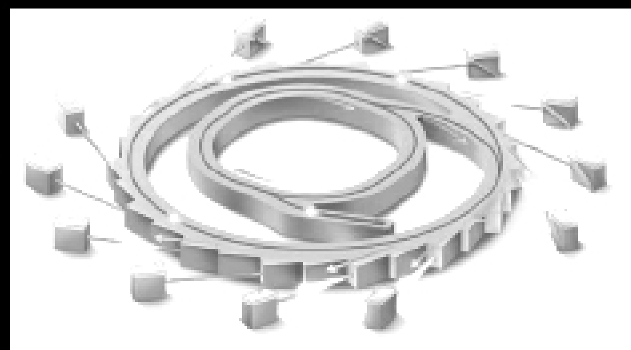


OPAL, Australia
courtesy: ANSTO

J-PARC, Japan
photos: Liss

Synchrotron Radiation



APS; NSLS-II
courtesy: Argonne NL; Brookhaven NL

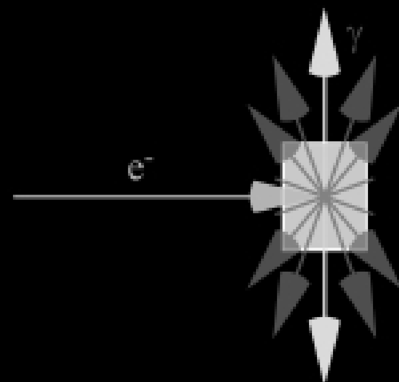
ESRF; AS
courtesy: ESRF; Australian Synchrotron

X-Ray Sources

- Bremsstrahlung
  - X-ray tube
- bending magnet
- wiggler
- undulator
- free-electron laser

X-Ray Sources

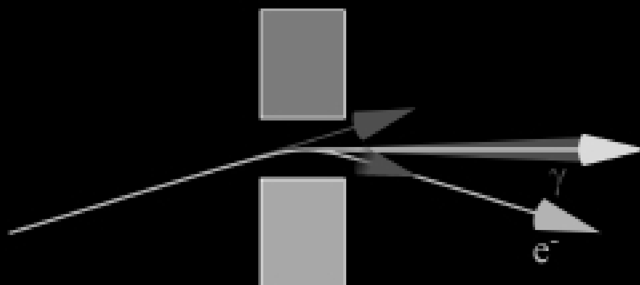
- Bremsstrahlung
  - electron tube
  - anode radiation
  - longitudinal acceleration (brems)
  - + characteristic lines



Bremsstrahlung
continuous spectrum
+
characteristic lines

X-Ray Sources

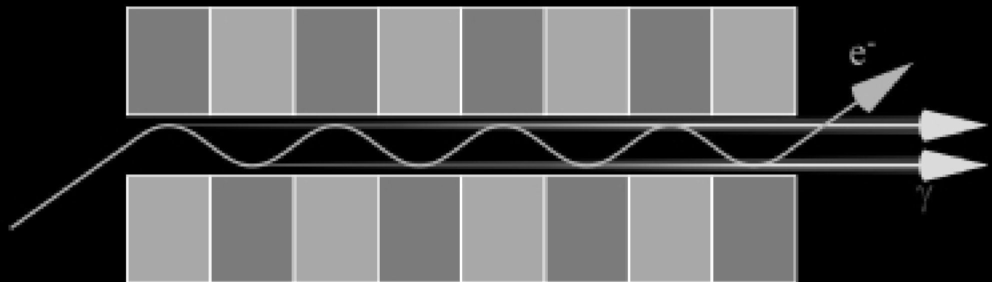
- Bending magnet
  - radial acceleration
  - relativistic transformation
    - forward direction
  - polarized



Bending Magnet: Intensity I ~ 1
continuous spectrum

X-Ray Sources

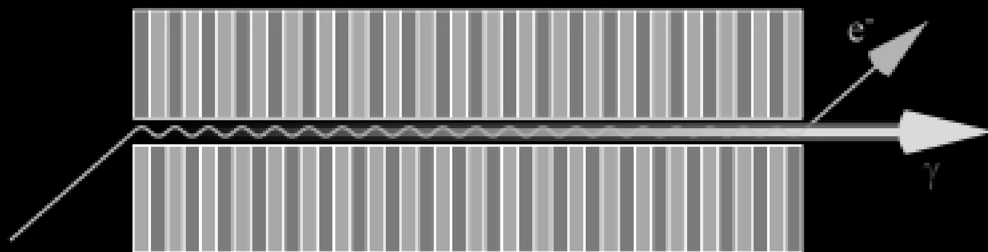
- Wiggler insertion device
  - many bends
  - incoherent summation of intensities



Wiggler: Intensity I ~ N
continuous spectrum

X-Ray Sources

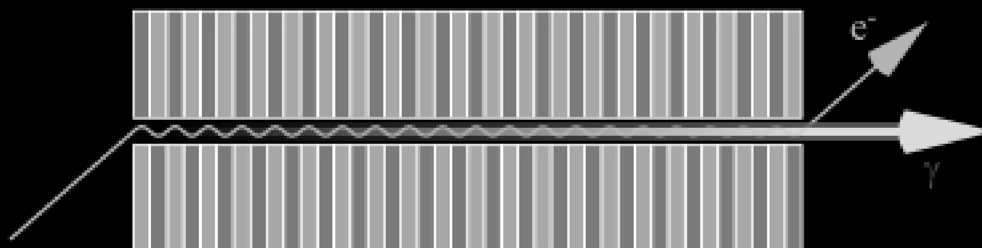
- Undulator
  - small e-beam amplitude
  - coherent superposition
  - sharp line spectrum + harmonics



Undulator: Intensity I ~ N^2
incoherent electrons; discrete lines in energy

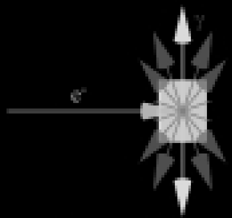
X-Ray Sources

- Free-electron laser
  - very long undulator
  - X-ray wave couples to electrons
    - microbunching
  - e + γ coherent superposition
    - coherent emission of light

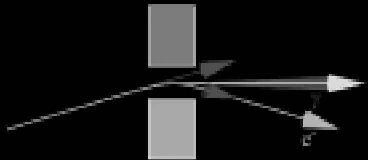


Free Electron Laser: Intensity I ~ exp(L)
coherent electrons; very few energy modes

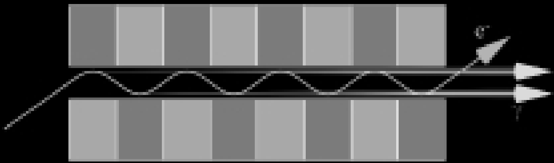
X-Ray Sources



Bremsstrahlung
continuous spectrum
+
characteristic lines



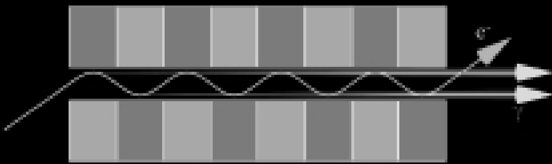
Bending Magnet: Intensity I ~ 1
continuous spectrum



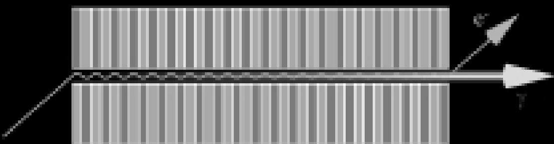
Wiggler: Intensity I ~ N
continuous spectrum

- Bremsstrahlung
  - electron tube
  - anode radiation
  - longitudinal acceleration (brems)
  - + characteristic lines
- Bending magnet
  - radial acceleration
  - relativistic transformation
    - forward direction
  - polarized
- Wiggler insertion device
  - many bends
  - incoherent summation of intensities

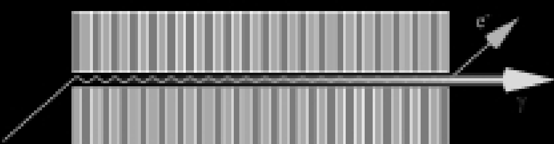
X-Ray Sources – Insertion Devices



Wiggler: Intensity I ~ N
continuous spectrum



Undulator: Intensity I ~ N^2
incoherent electrons; discrete lines in energy



Free Electron Laser: Intensity I ~ exp(L)
coherent electrons; very few energy modes

- Wiggler
  - large e-beam amplitude
  - incoherent superposition
  - broad, white spectrum
- Undulator
  - small e-beam amplitude
  - coherent superposition
  - sharp line spectrum + harmonics
- Free-electron laser
  - very long undulator
  - X-ray wave couples to electrons
    - microbunching
  - e + γ coherent superposition
    - coherent emission of light

Typical Synchrotron Lattice

ADVANCED PHOTON SOURCE

BEAM ACCELERATION & STORAGE SYSTEM

(A) ELECTRON GUN

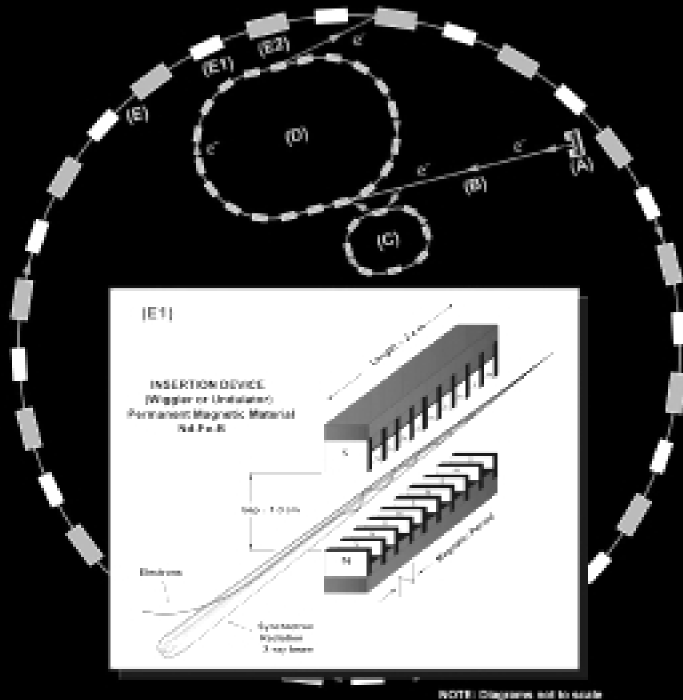
(B) ELECTRON LINEAR ACCELERATOR
Output energy: 325 MeV

(C) ACCUMULATOR RING

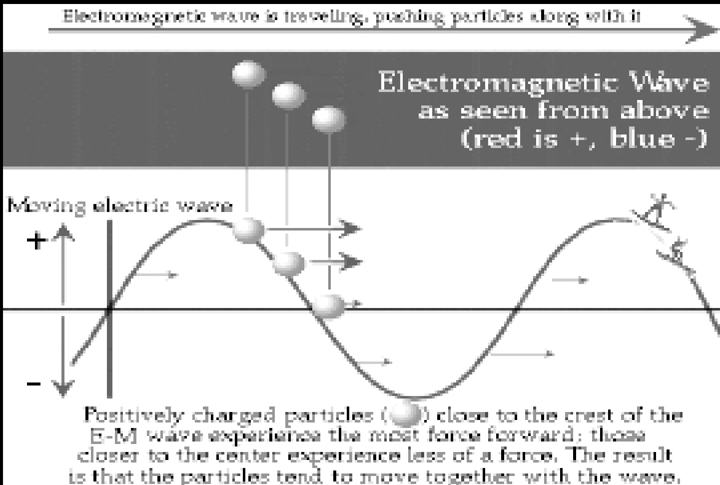
(D) BOOSTER SYNCHROTRON
Nominal extraction energy: 7 GeV

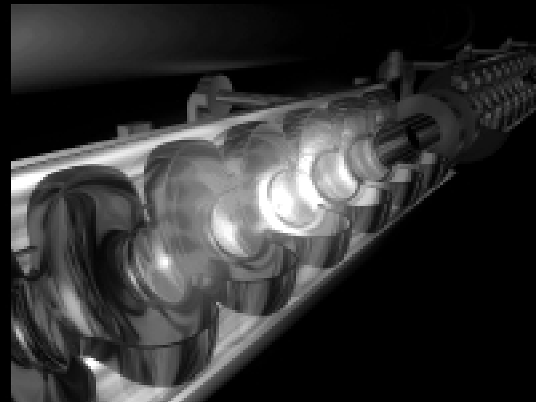
(E) STORAGE RING
Nominal energy: 7 GeV

(E1) INSERTION DEVICE
(E2) BENDING MAGNET



Accelerator and Devices





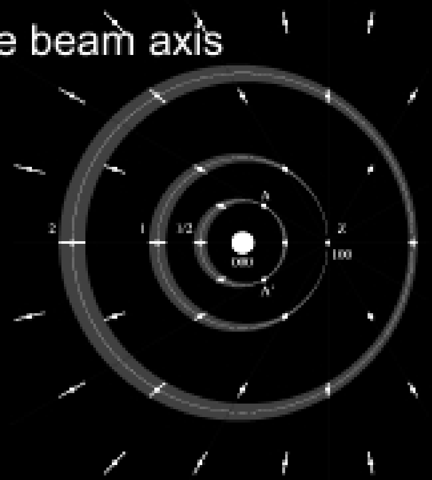
courtesy: USA

surfer: Kilian Liss

courtesy DESY

Characteristics of Synchrotron Rad.

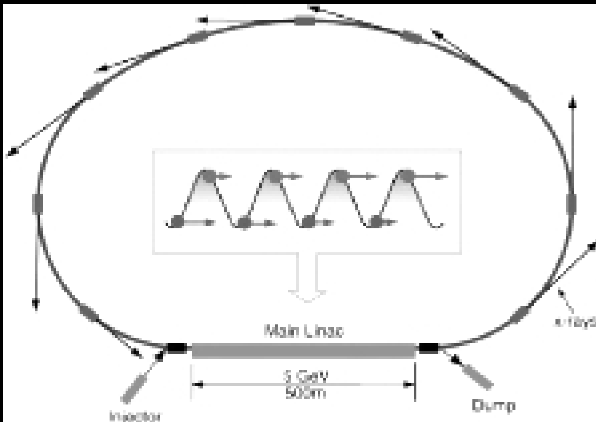
- tunability
  - select your Energy according to your problem
  - measure energy-dispersive
  - spectroscopy
- polarization
  - light is highly polarized
    - depending on projection of the orbit to the beam axis
    - linear in orbital plane
    - circular out of plane
      - useful for magnetic coupling
    - circular by helical insertion devices
- small angular source size
  - no dispersion in diffraction
  - high lateral coherence

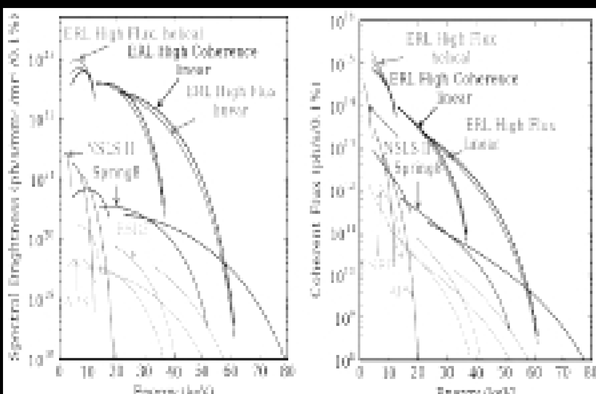


Limitations of conventional SR

- Emittance:
  - phase space volume element occupied by electrons
  - few (1-100) nanometer * radian (nm rad) in each dimension
- Bunch Length:
  - length in space and time
  - 50 - 100 picoseconds (ps)
  - 1.5 - 3 centimeter (cm)
- Thermalization of Bunches:
  - even shorter and lower emittant bunches spread out in phase space
  - limited by a pouch of the RF cavities
  - laterally confined by lattice of the storage ring
  - synchrotron oscillations are vibrations of the trajectory around the mean trajectory of a perfect beam
  - new bunches are injected into a neighboring pouch and then cooled by damping into the mean trajectories
  - it takes many milliseconds to thermalize the beam

Future: Energy Recovery Linacs

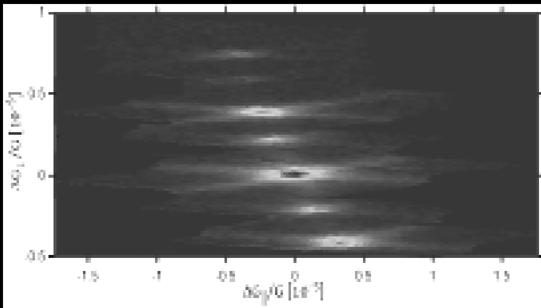




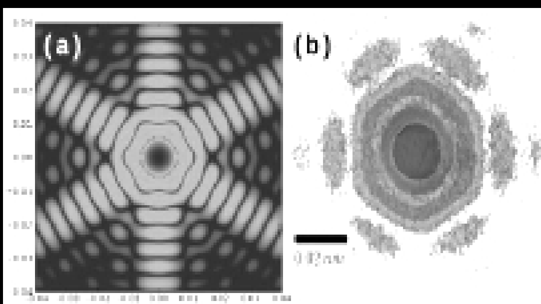
Don Bilderback, et. al.
figures courtesy: CHESS

- ERL principle
  - start with low emittant bunch
  - only one or few turns
  - give bunch energy back to accelerator
  - dump the degrading bunch
  - start over with new bunch
- advantages
  - no time to thermalize
  - keep low emittance
  - short time pulses (100 fs)
  - high coherence
  - bunch not re-used
    - can be used degradably

Beam Coherence



diffraction with conventional synchrotron X-rays
courtesy: K.-D. Liss et al

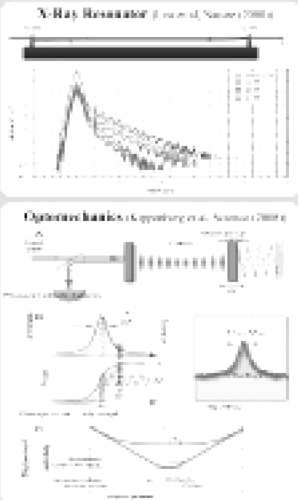


diffraction with coherent synchrotron X-rays
courtesy: V. Favre-Nikolin

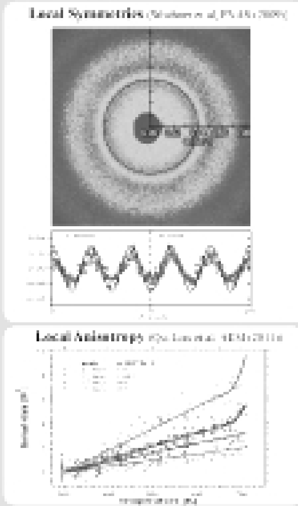
- small electron emittance
  - high X-ray beam coherence
- limitations by
  - repulsion of electrons
  - thermalization of bunches
- dependencies
  - electron Energy
  - synchrotron lattice
  - damping

Potential Applications of New Sources

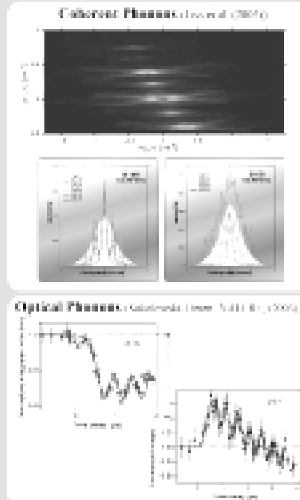
Application in Quantum Nanoscience



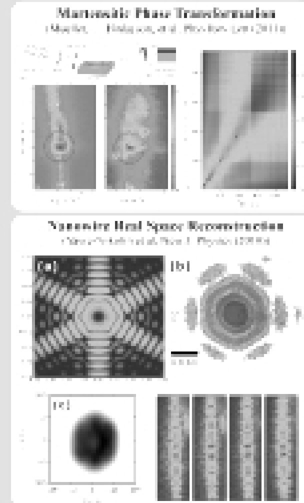
Application for Amorphous Materials



Application in Time Resolved Processes

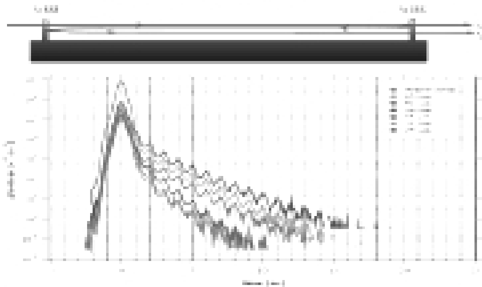


Coherent X-ray Scattering

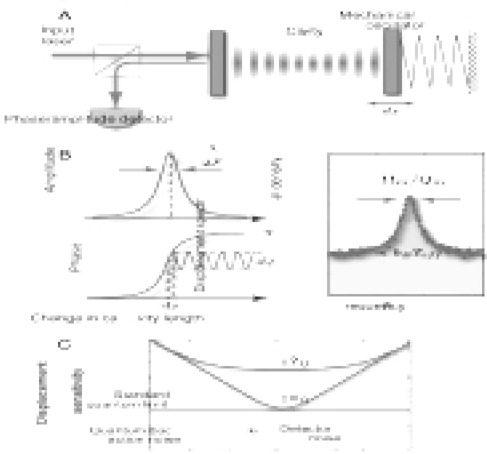


Application in Quantum Nanoscience

X-Ray Resonator (Liss et al, Nature (2000))

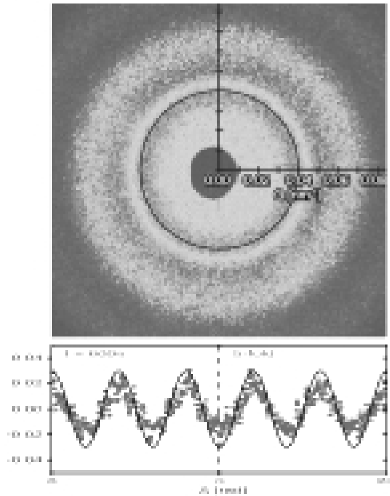


Optomechanics (Kippenberg et al, Science (2008))

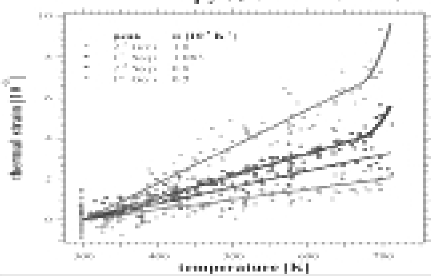


Application for Amorphous Materials

Local Symmetries (Wochner et al, PNAS (2009))

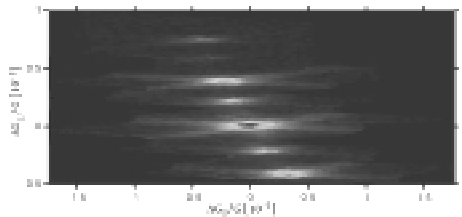


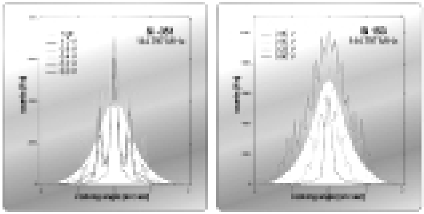
Local Anisotropy (Qu, Liss et al, AEM (2011))



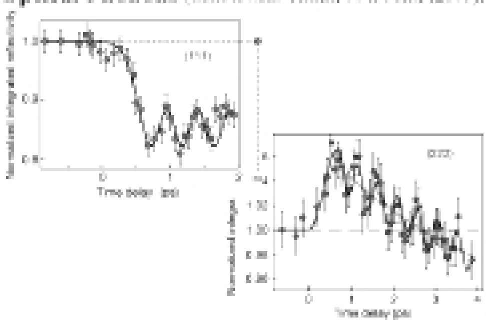
Application in Time Resolved Processes

Coherent Phonons (Liss et al, (2003))





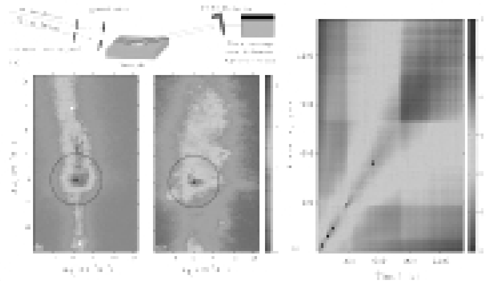
Optical Phonons (Sokolowski-Tinten, NATURE, (2003))



Coherent X-ray Scattering

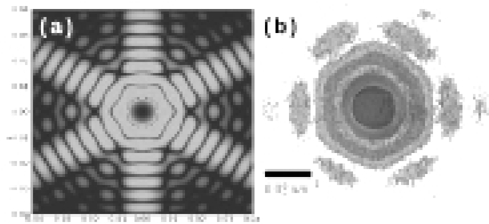
Martensitic Phase Transformation

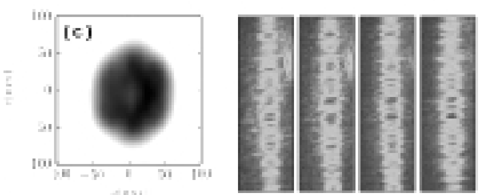
(Mueller, ... Finlayson, et al, Phys Rev Lett (2011))



Nanowire Real Space Reconstruction

(Favre-Nikolin et al, New J. Physics (2010))



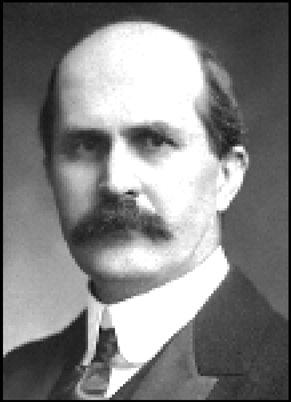


Diffraction

- 2012 - 100 years X-ray diffraction
  - Nobel Laureate:

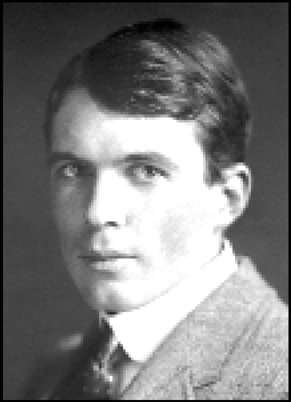
Max von Laue

1914



Sir William
Henry Bragg

1915



William Lawrence
Bragg

1915

Diffraction

- interferences on scale of wavelength
  - atomic distances in crystals
- Bragg's law
  - interpretation in real-space
- Laue equation
  - uses reciprocal space
- Fourier transforms
  - exp(i (ωt – kr))

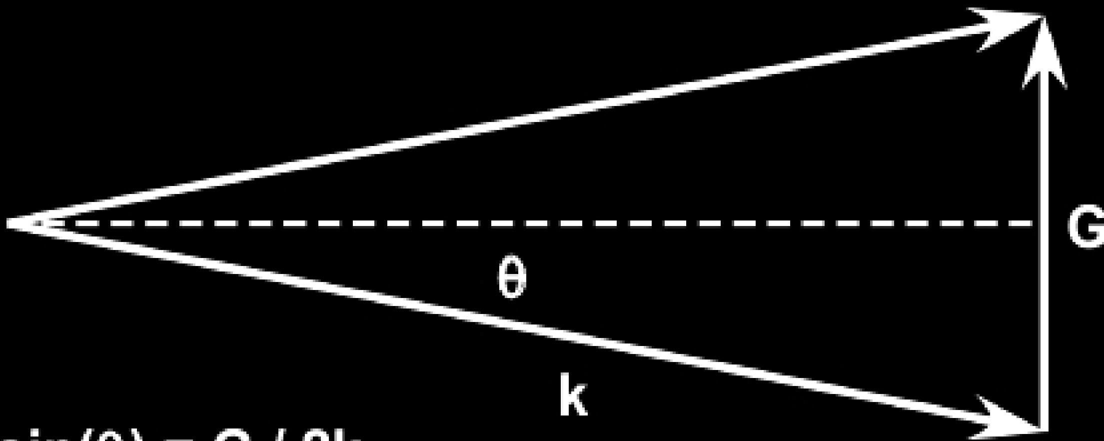
- Note:
  - dependency on wave vector k: diffraction
  - dependency on frequency ω: spectroscopy

Diffraction: Laue Equation

$$\vec{k}_f = \vec{k}_i + \vec{G}$$

$k, G \in Q$;	reciprocal space
$k = 2\pi/\lambda$;	wave vector
$G = 2\pi/d$;	reciprocal lattice vector
$Q = 4\pi/\lambda \sin((2\theta)/2)$;	Bragg's Law

Bragg's Law



$\sin(\theta) = G / 2k$

<=>

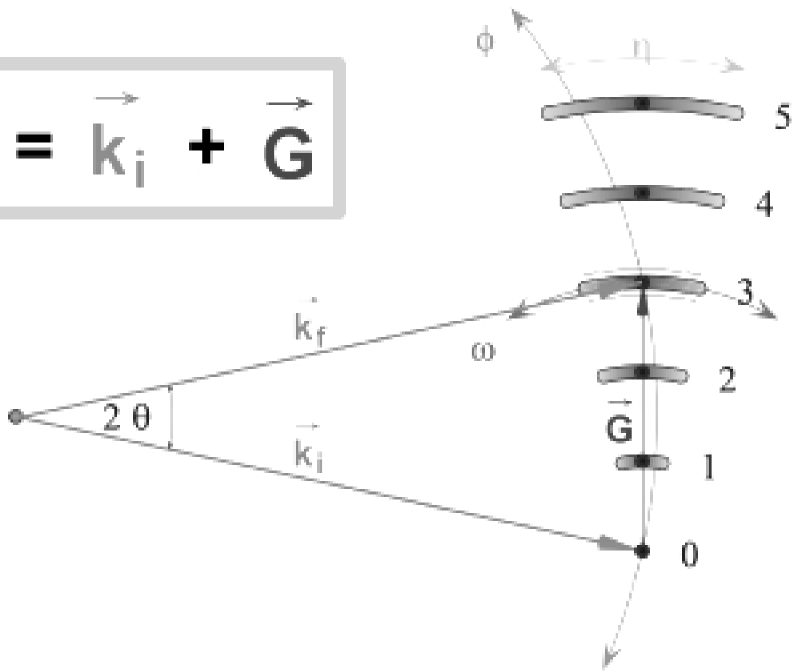
$\lambda = 2d \sin(\theta)$

$k = 2\pi/\lambda$; wave vector

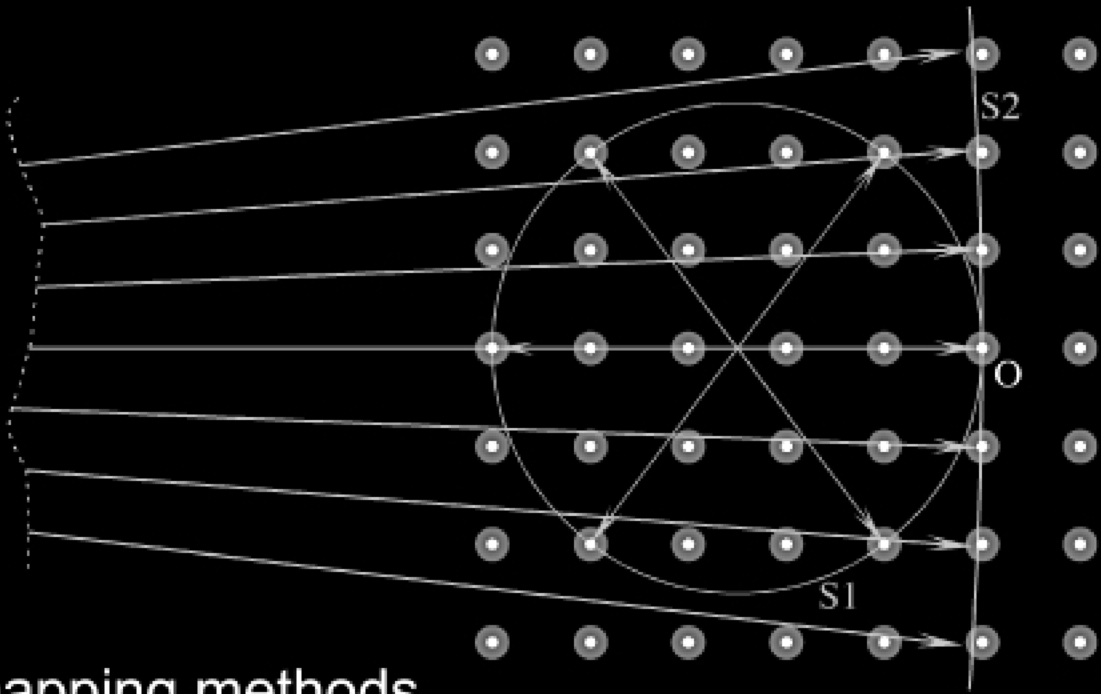
$G = 2\pi/d$; reciprocal lattice vector

Diffraction: Laue Equation

$$\vec{k}_f = \vec{k}_i + \vec{G}$$

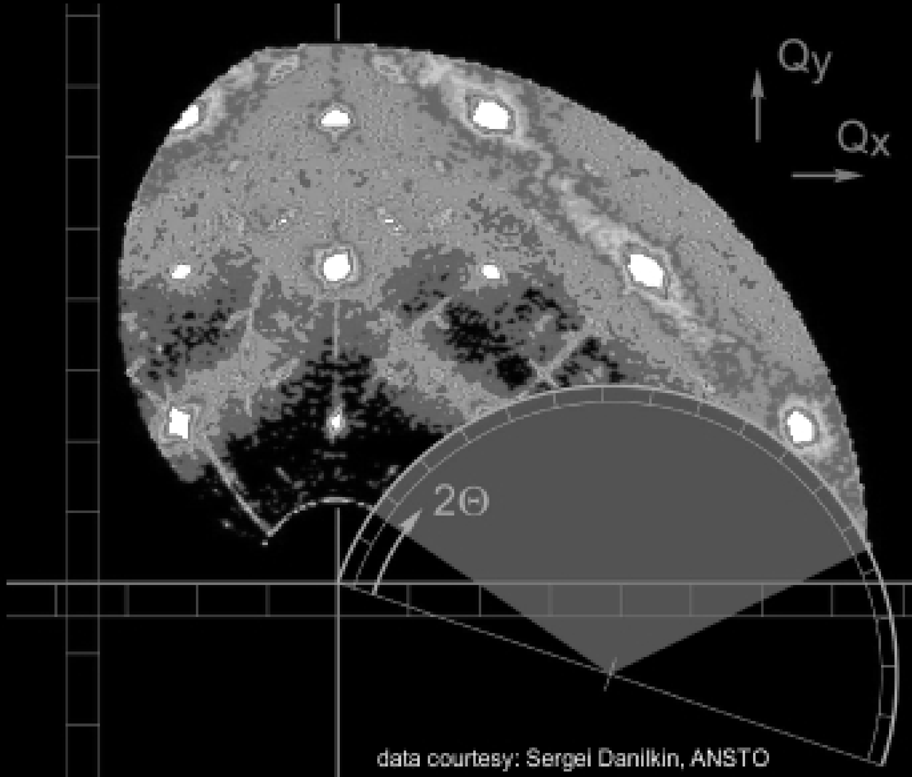


Ewald Construction

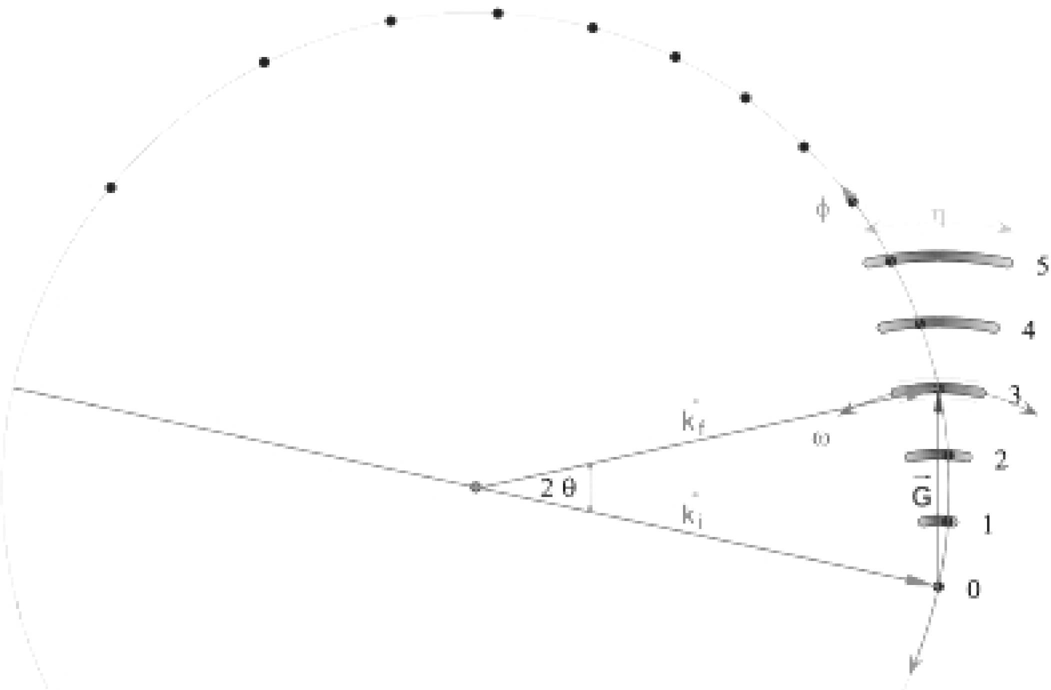


- mapping methods
  - angle dispersive
  - energy dispersive

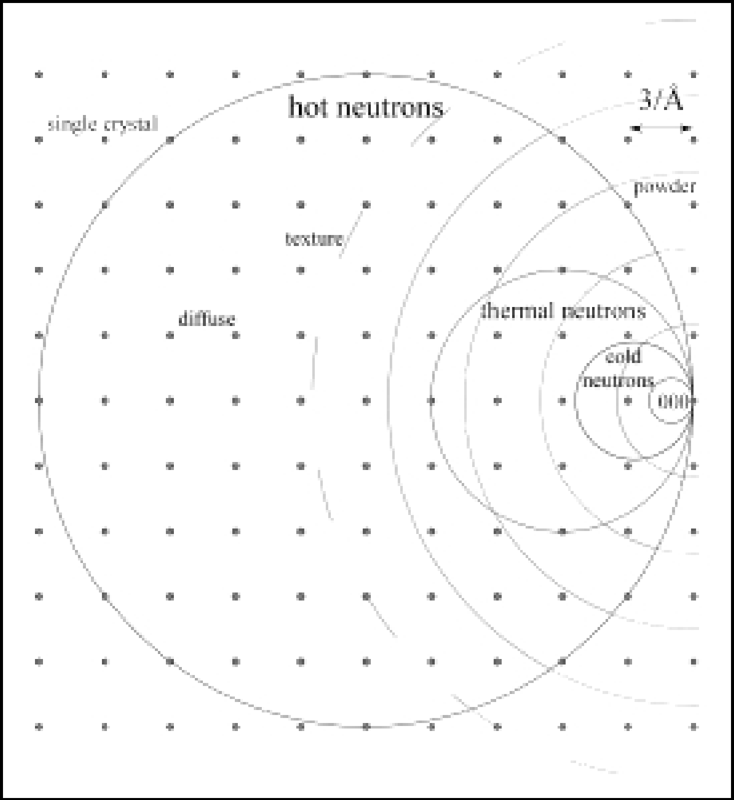
Single Crystal Diffraction



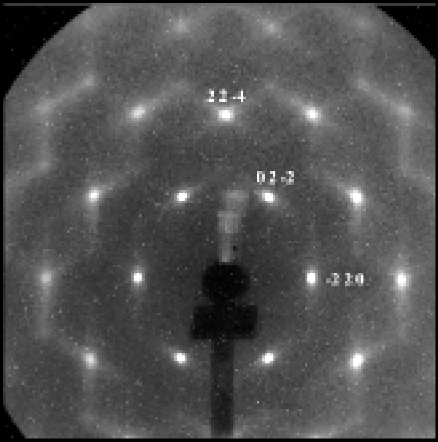
Diffraction: Powder Diffraction



Reciprocal Space Coverage

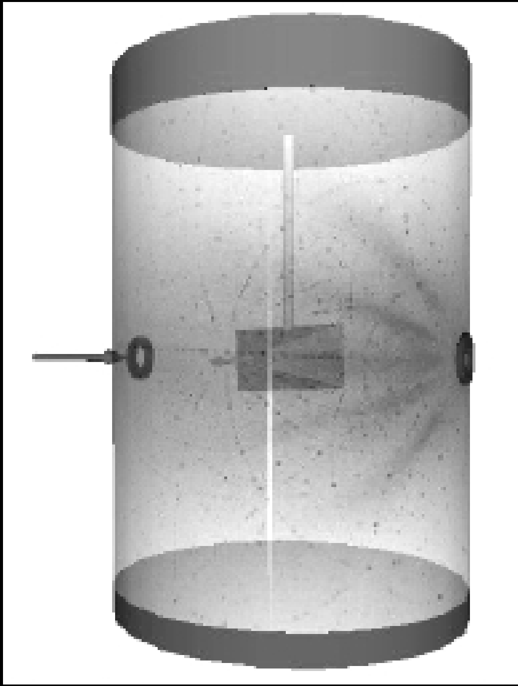


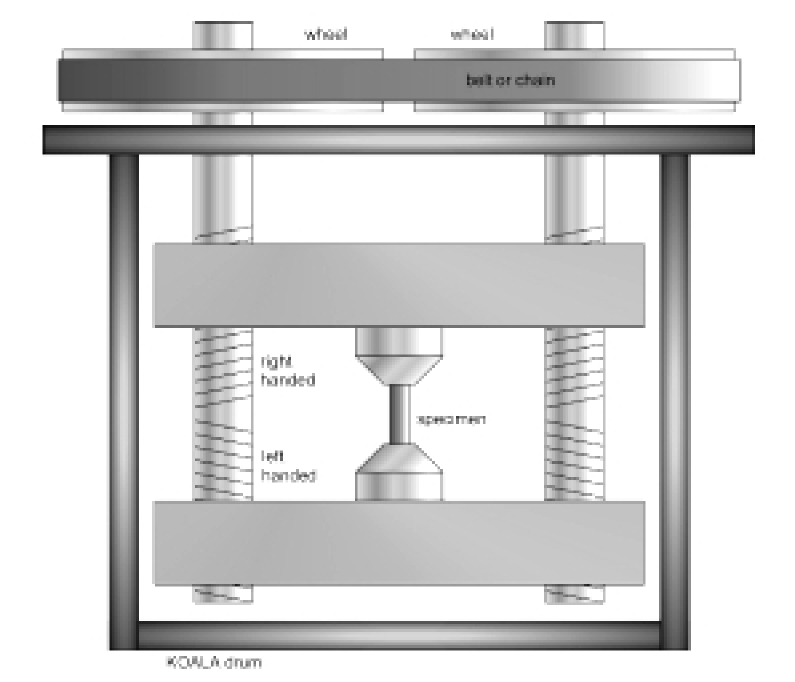
- reciprocal space:
  - sample dependent only
  - instrument independent
  - wavelength independ.
  - linearity and symmetry



diffuse X-ray scattering on Silicon

Neutron Laue Diffraction

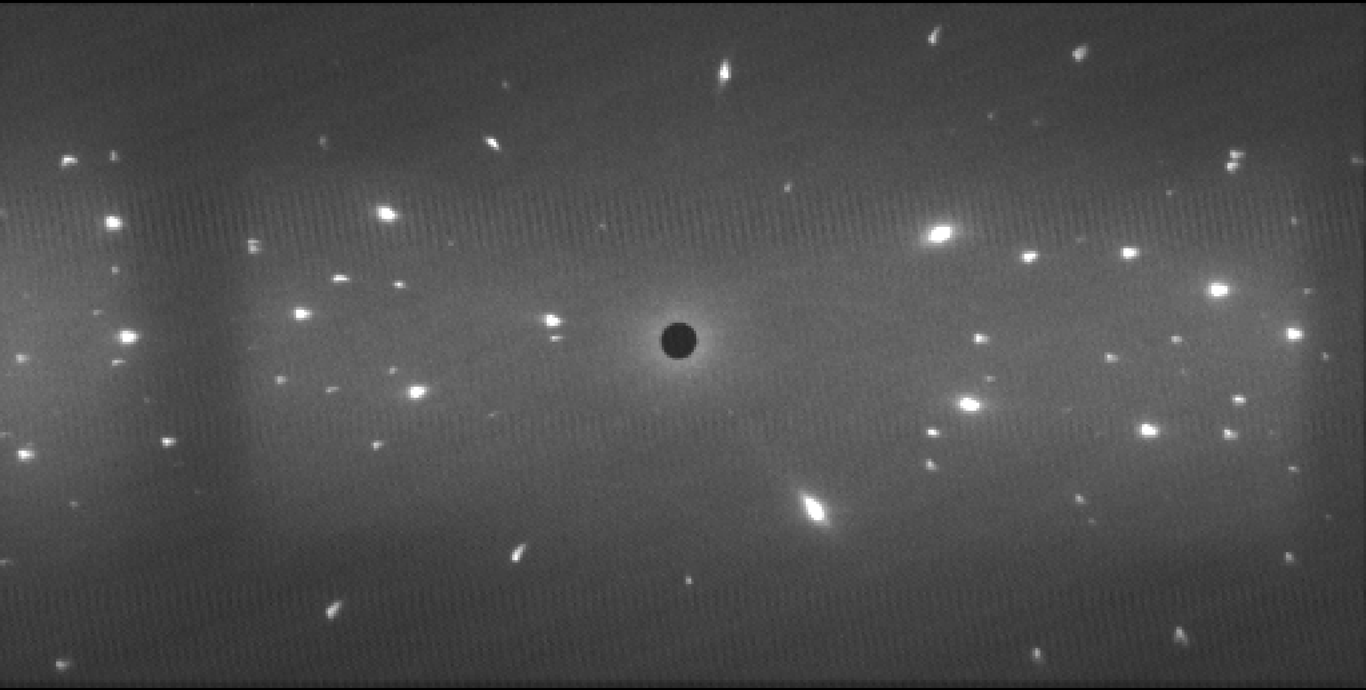




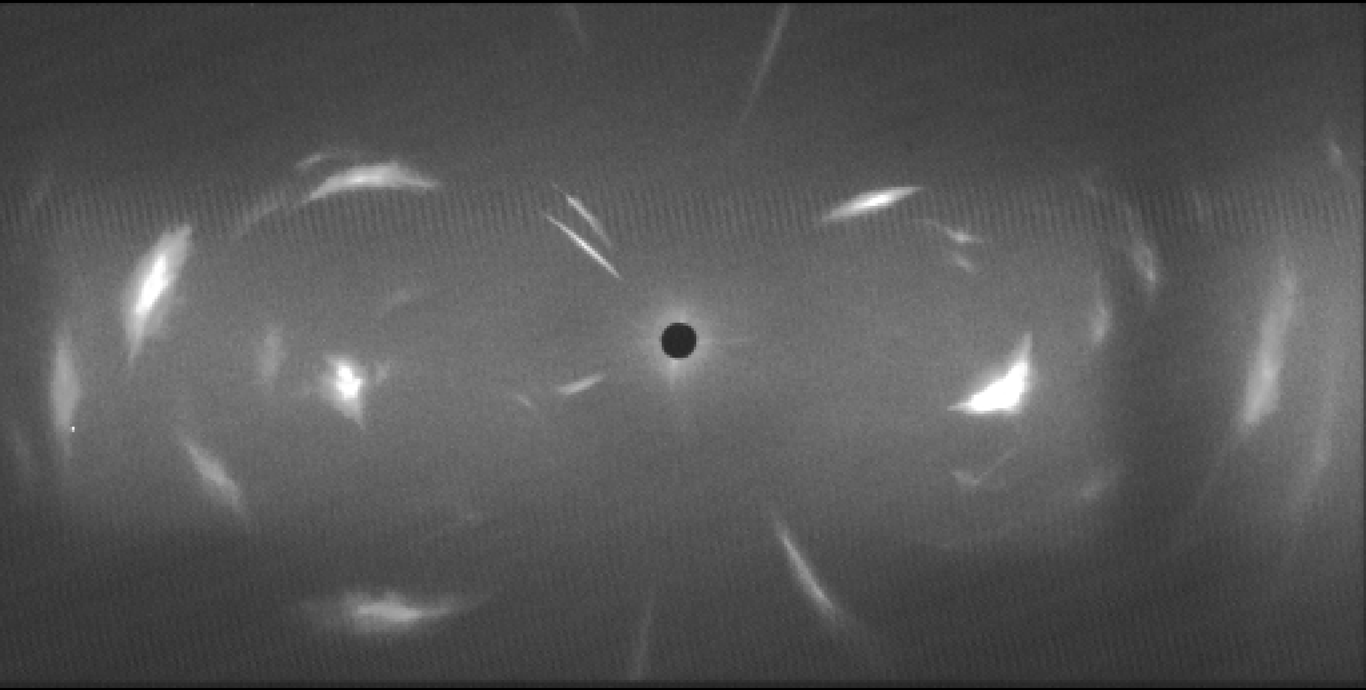
Koala Instrument @ ANSTO

figure: G.J. McIntyre et. al. Physica B 385-386 (2006)

Cu single crystal

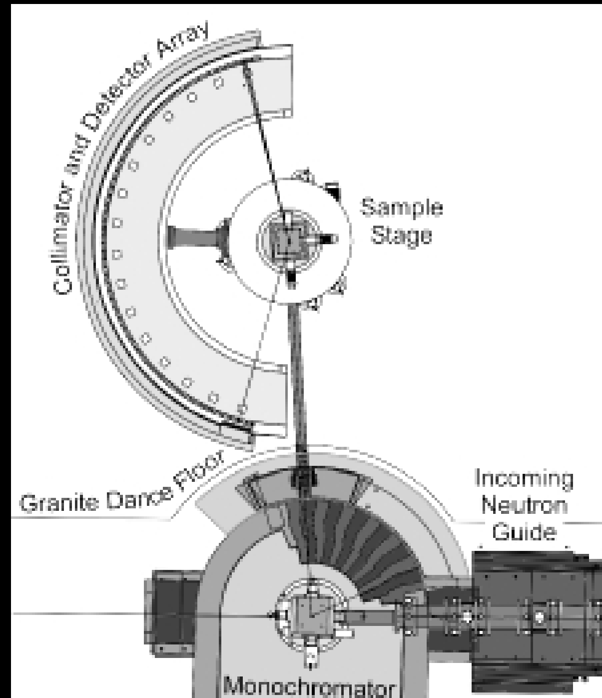


Cu single crystal - deformed



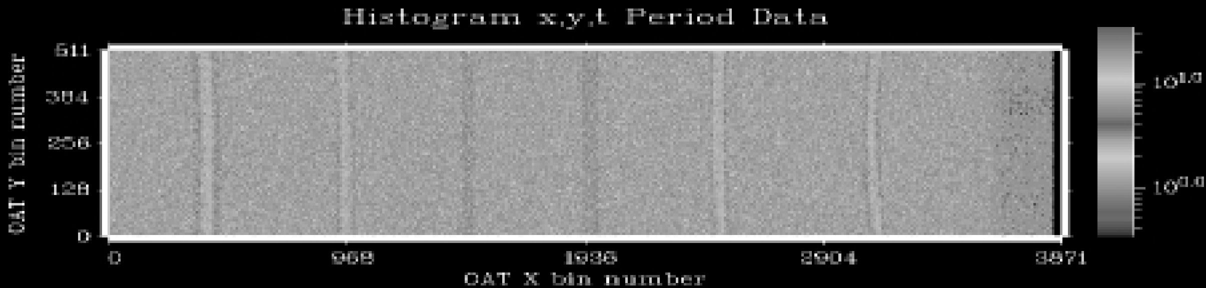
Angle Dispersive Diffraction

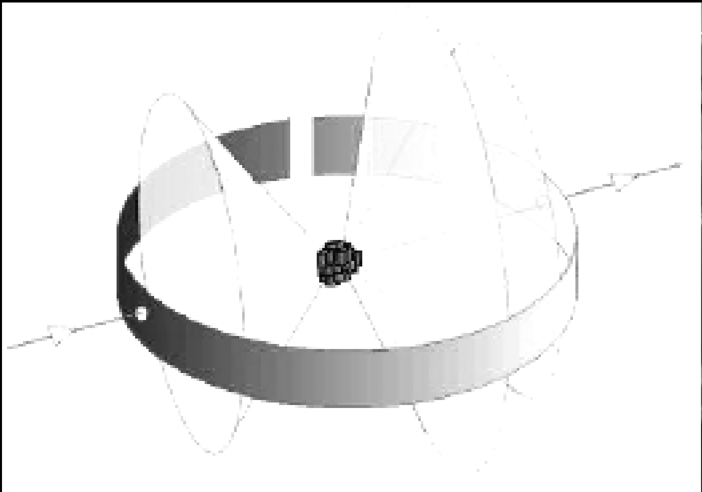
- conventional method
- continuous sources
- simple detectors

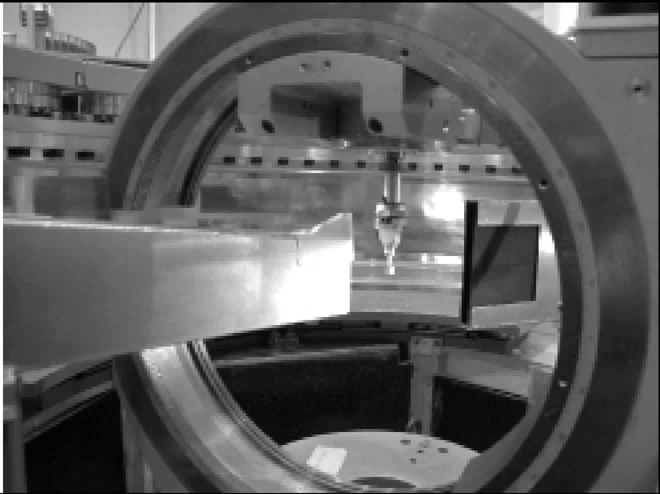


Echidna powder diffractometer
OPAL, Australia

Neutron Powder Diffraction





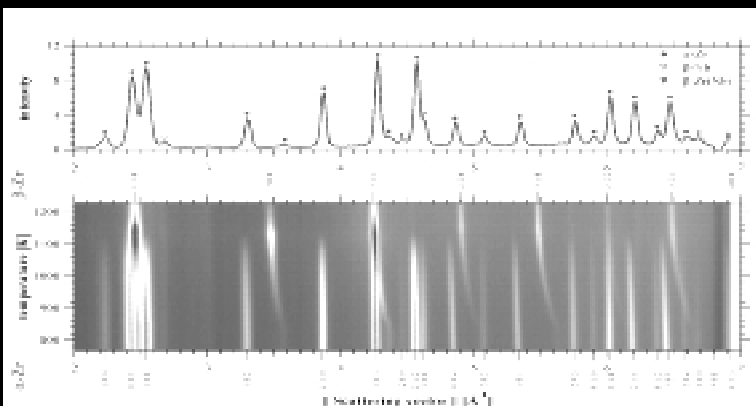


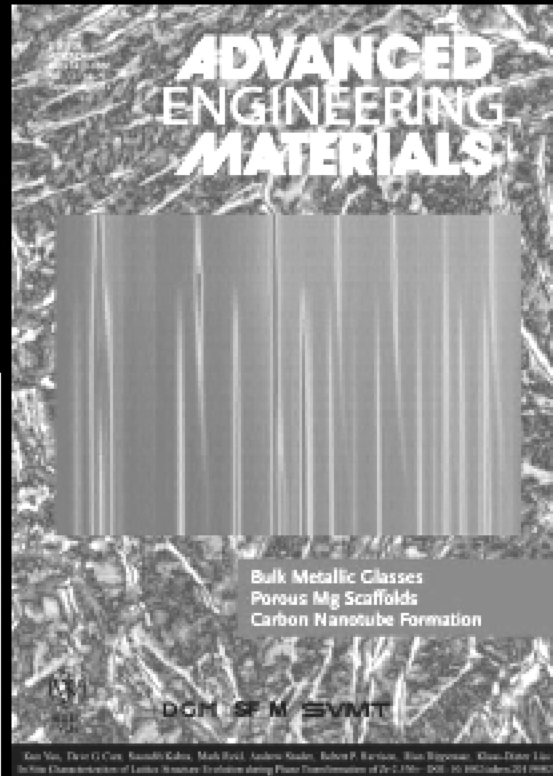
Eulerian Cradle on Wombat powder diffractometer
ANSTO, Australia

In-situ neutron powder diffraction

- average over large volume → good grain statistics
- phase determination
- texture measurement
- from the bulk

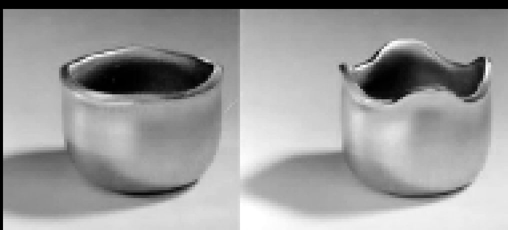
example : Zr-2.5Nb alloy





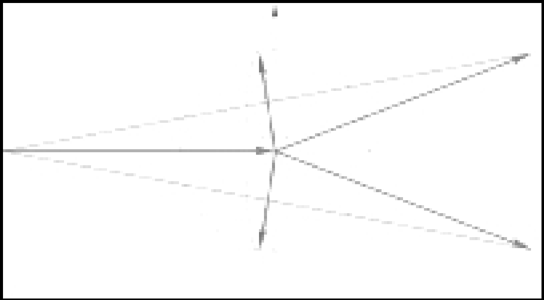
Texture

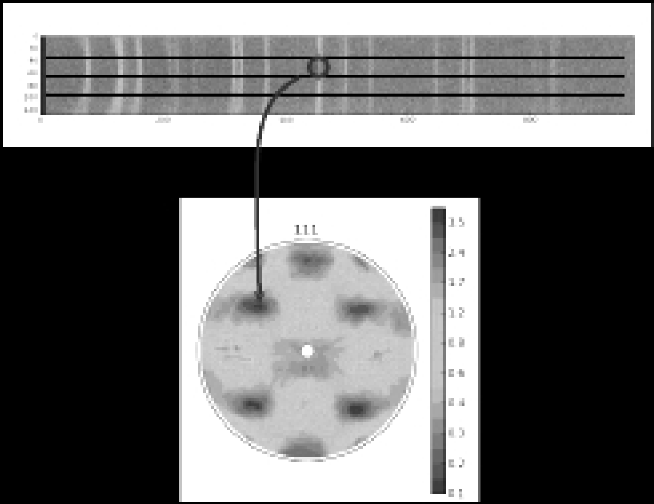
- Texture is Preferred Orientation of crystallites
- Textures are formed by plastic deformation processes
- Mechanical properties depend on texture
- Texture tells the history of a deformation process

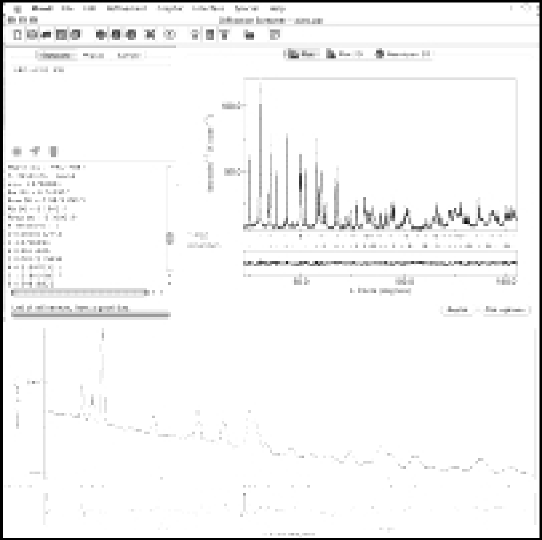


1970s can 2 made in Brisbane

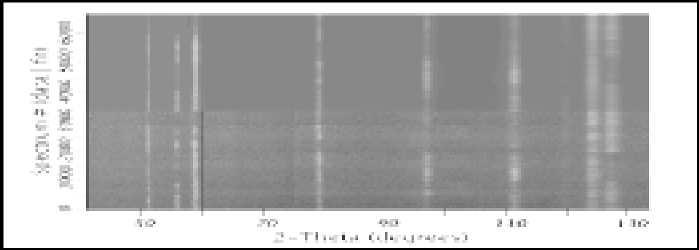
Mapping Pole Figures



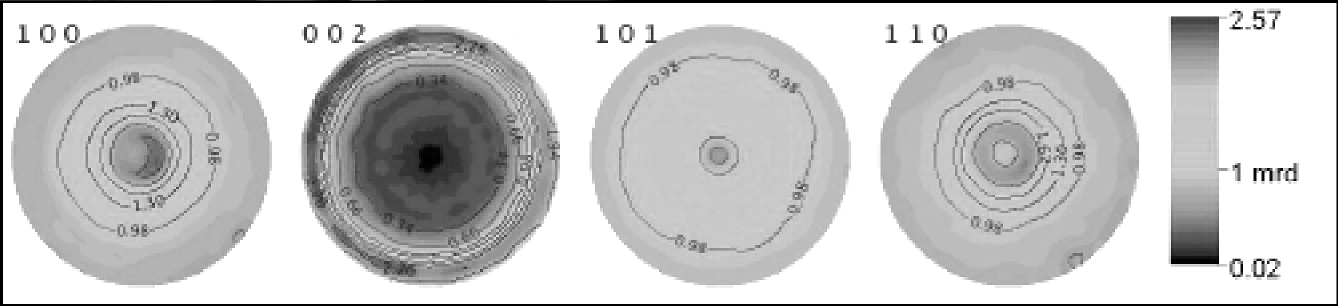


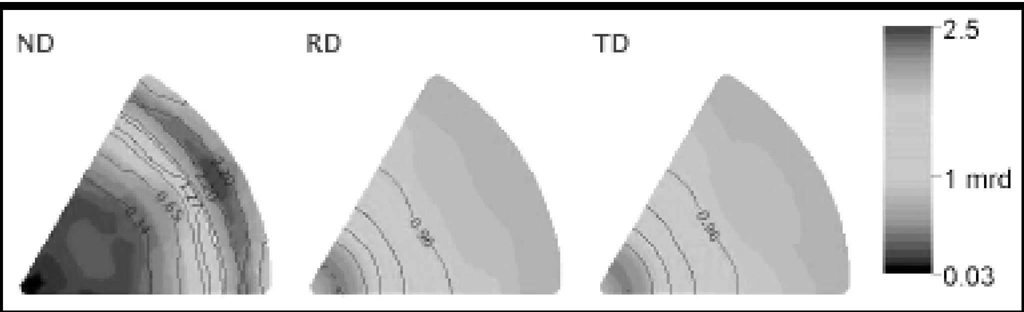


- Direct pole figure mapping
- ODF fitting with MAUD Rietveld package



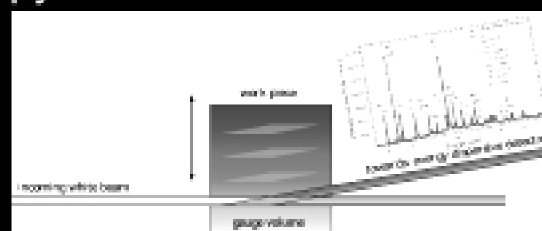
Texture of extruded Magnesium





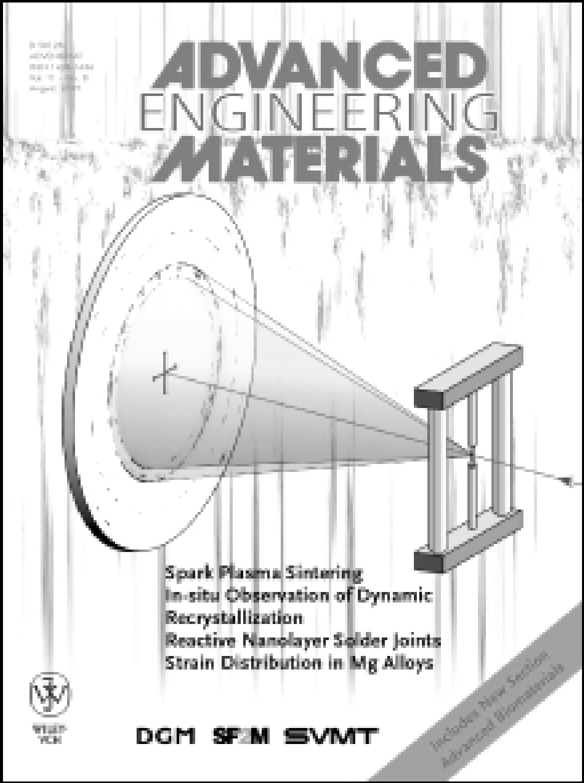
Energy dispersive measurements

- scan length of k-vector
  - neutrons: Time Of Flight analysis
    - pulsed spallation sources
    - beam chopping at reactor sources
  - synchrotron:
    - energy-dispersive solid state detector
- advantages:
  - full diffraction pattern in a single direction of observation
  - samples and detectors do not move
    - can be done with multiple detectors simultaneously
    - combine diffraction and spectroscopy
    - measure anisotropic properties

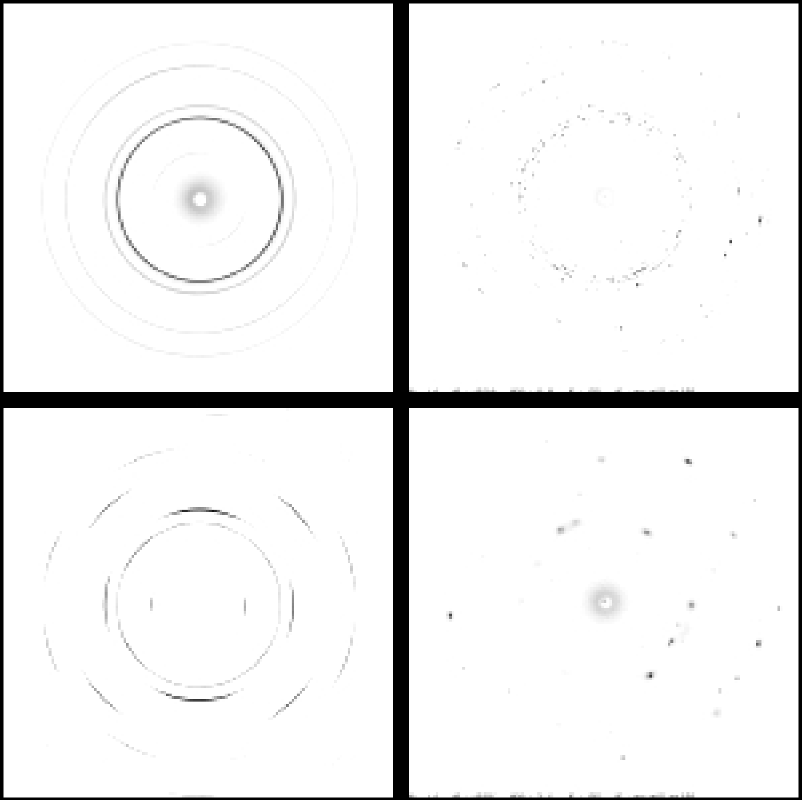


High Energy Synchrotron Radiation

- 90-100keV X-rays
  - ESRF, Grenoble, France
  - APS, Argonne, USA



Debye-Scherrer Rings (on TiAl)



Neutrons or Synchrotron?

OPAL, Australia

- Neutrons
  - different contrasts
  - good grain statistics
    - quantitative phase analysis
    - texture analysis
  - magnetic moment
  - hydrogen
- Synchrotron X-Rays
  - small, brilliant
  - single grain statistics
  - high time resolution
  - high reciprocal space resolution

ESRF, France

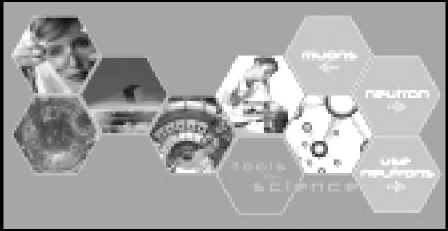
⇒ use X-rays, neutrons only when valuable!

Conclusion – Part 1

- complementary types of radiation
  - neutrons
  - X-rays
- modern sources
  - X-ray tubes
  - synchrotron storage rings
  - energy recovery linacs
  - free-electron lasers
  - reactor neutron sources
  - spallation sources
- diffraction
  - large volume vs small volume
  - cross sections and magnetism

Further Lookup and Reading

- lightsources.org
  - http://www.lightsources.org
- European Neutron Portal
  - http://www.neutron-eu.net/



- Neutronsources.org
  - http://neutronsources.org
  - (under construction)
- Komitee Forschung mit Neutronen
  - http://www.neutronenforschung.de
- 2 symposia in 2013
  - PRICM-8 – Hawaii, Aug 4-9
  - THERMEC – Las Vegas, Dec 2-6

Tutorial VV:

Part 2:
Advanced Diffraction: Understanding Local Structure by Probing Reciprocal Space

Rozaliya Barabash
Oak Ridge National Laboratory