Synchrotron Radiation Spectroscopies on Solids, Surfaces, and Interfaces

Giancarlo Panaccione

Lab. TASC INFM -CNR
Advanced Photoemission Experiment (APE) beamline @
ELETTRA storage ring
Trieste (Italy)

E-mail: panaccioneg@elettra.trieste.it

Main argument of the course:

Characterization and analysis of solids and surfaces with Synchrotron Radiation

1. Basics of Synchrotron Radiation (why, what, where, who)

   Beamlines, Devices
   Material Science and SR

2. Electron Spectroscopies with X-rays **INTENSITY OF SR**

   Interaction of radiation with matter
   Absorption and Photoemission (bulk and surface)
   Applications to Model Systems

3. Polarization dependent Spectroscopies **SYMMETRY OF SR**

Chirality and Dichroism
Measure and analysis of Spin
Nanoscience and magnetism with SR
Hot topics in Low dimensional Magnetism

4. Beyond the surface (UPS, XPS, HX-PES) **TUNABILITY OF SR**

Analysis of Correlated systems with SR
Surface sensitivity: advantages and problems
Volume sensitive photoemission

Objective :

Design, prepare, 'virtually perform' a specific experiment at SR source

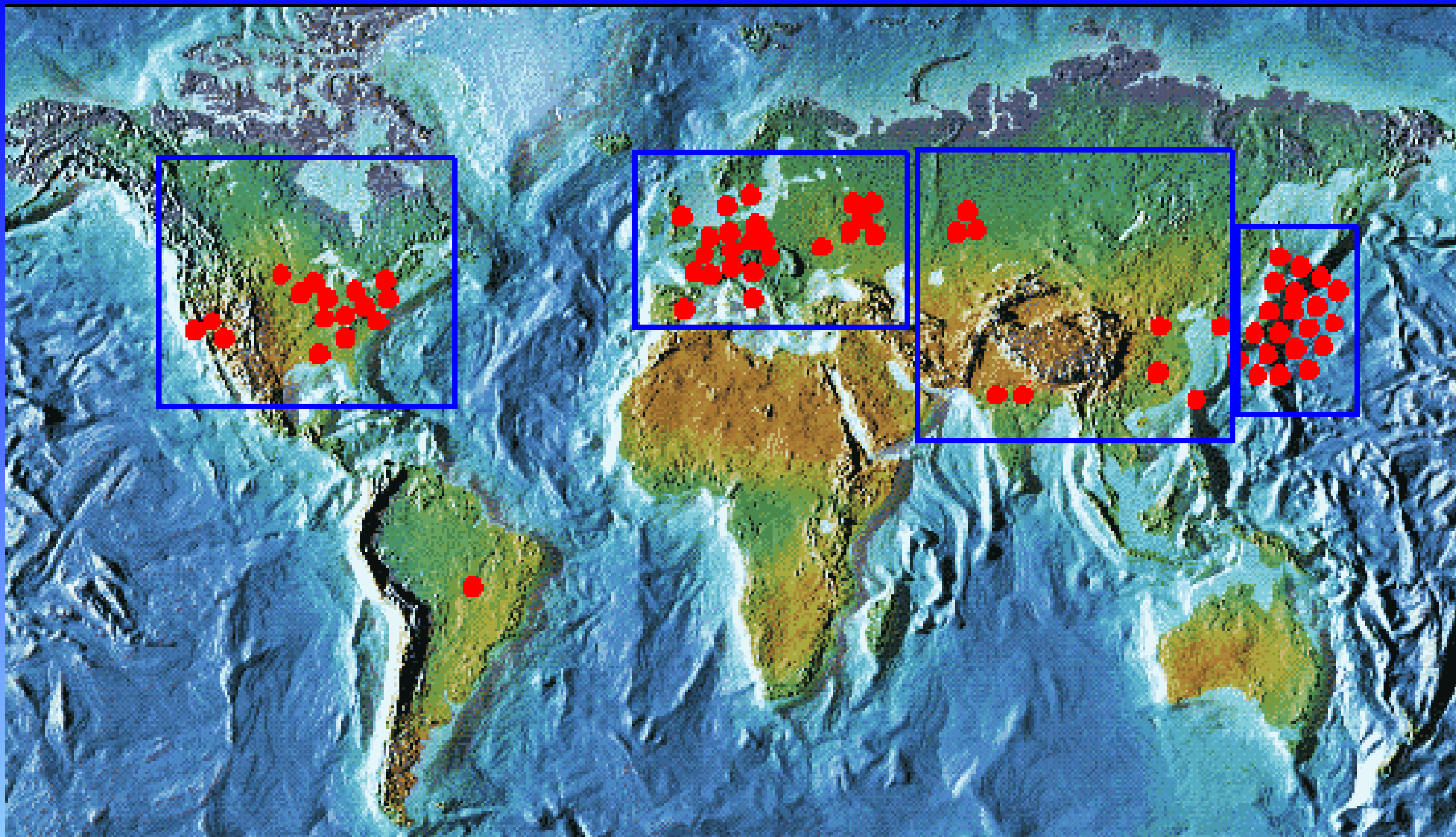
1) Subject of the experiment
   Electronic, Structural, Chemical, Magnetic properties of a solid

2) Choice:
   Synchrotron source, beamline, spectroscopy, experimental setup

3) Preparation:
   Samples, Parameters, Environmental Conditions

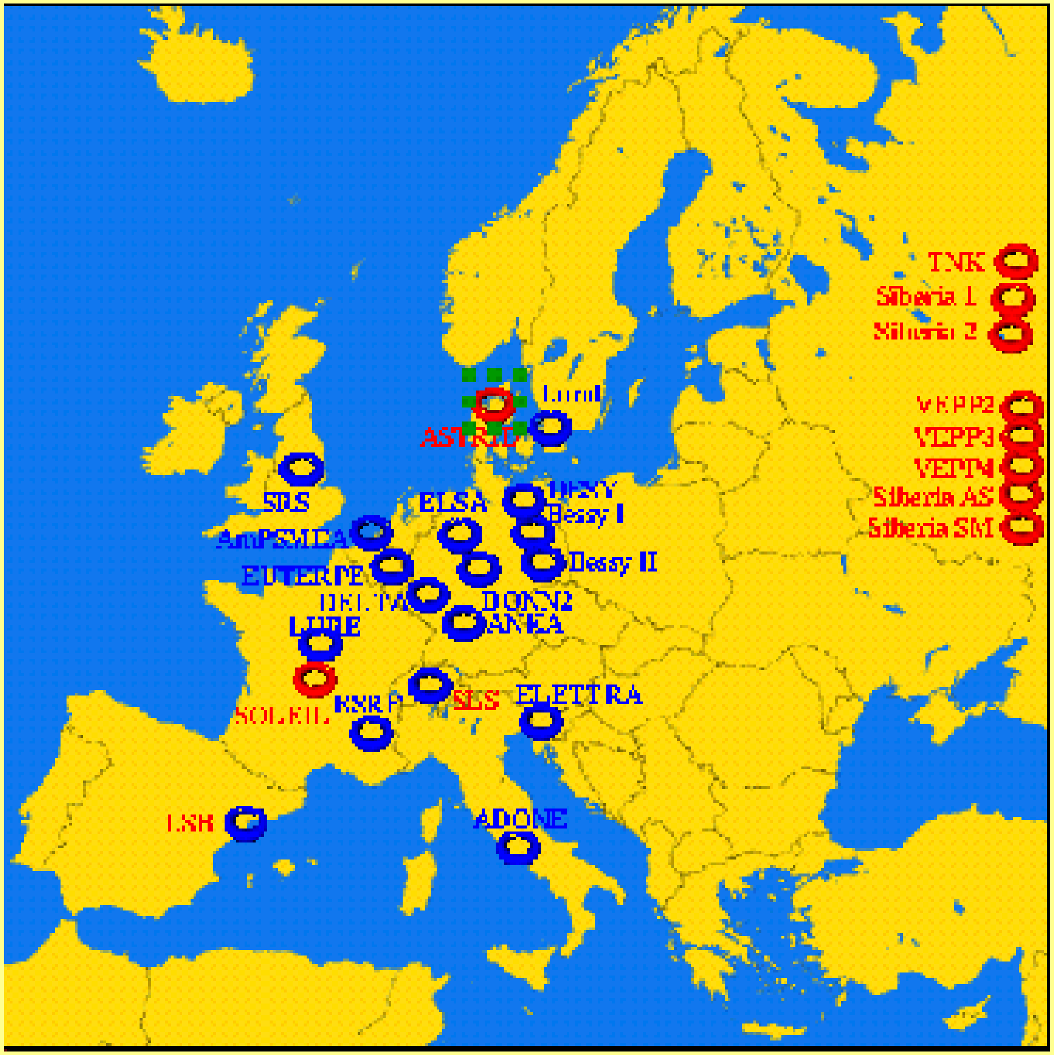
4) Measurements and Analysis

Tutorials, Seminars, Practicals on data analysis



Les installations de RS en 1997

Asie + Amérique du Sud : ~ 10
Amerique du Nord : ~ 20
Europe : ~ 20 Japon : ~ 20



	En exploitation	En construction, tests	En projet
Allemagne	2	3	1
Danemark	1		1
Espagne			1
Europe	1		
France	1		1
G.B	1		1
Italie	1		
Pays Bas	2		
Russie	8		
Suède	2		
Suisse			1

ELETTRA
Trieste (Italy)

European Synchrotron Radiation Facility (ESRF)
Grenoble, France

Canadian Light Source Inc.
University of Saskatchewan
101 Perimeter Road
Saskatoon,
Saskatchewan S7N 0X4

www.lightsource.ca

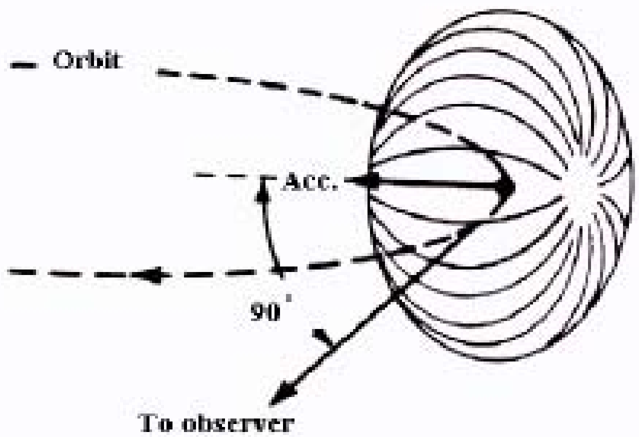
SOLEIL, Paris, France

Homework n. 1

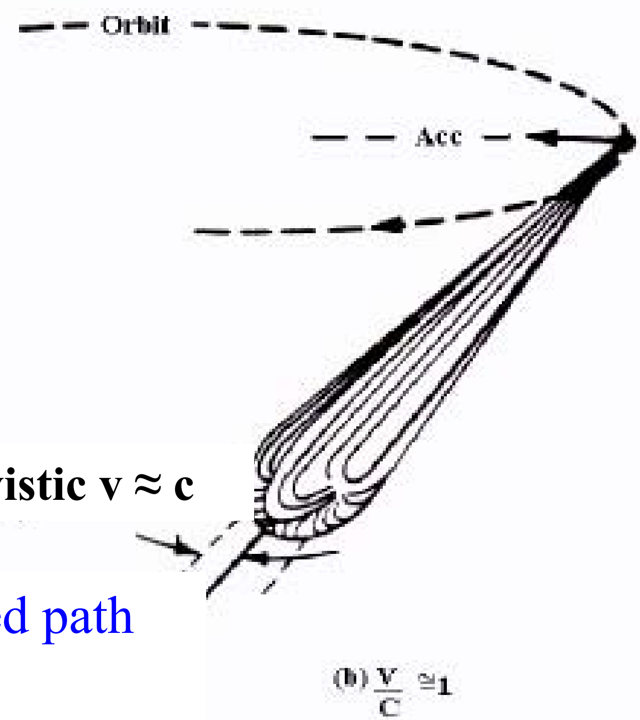
Find and list main differences
www.lightsources.org

Spring8,
Hyogo , Japan

Production of Synchrotron Radiation: relativistic electrons



Non relativistic v<<c



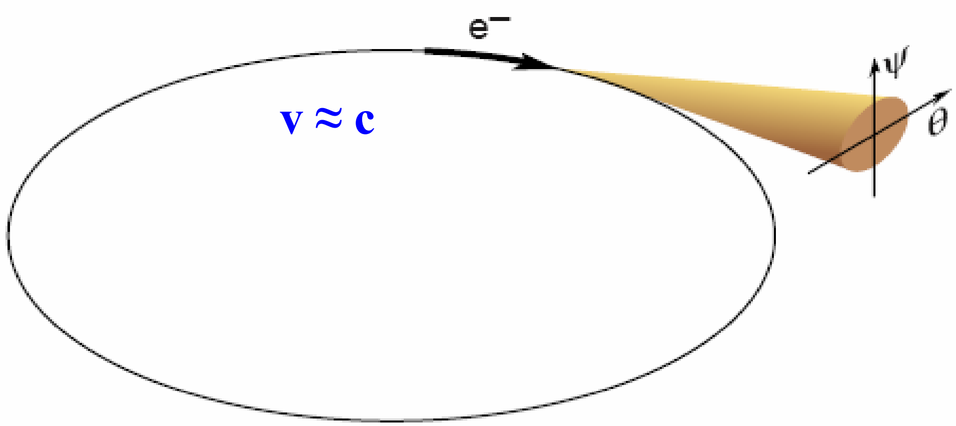
Relativistic v ≈ c

A charged particle constrained to move in curved path experiences a centripetal acceleration.
Due to this acceleration, the particle radiates energy according to Maxwell equations.

As the speed of the particle **approaches the speed of light**, the radiation pattern is distorted by relativistic effects and changes to a narrow cone of radiation, called Synchrotron Radiation.

Lesson II, next Tuesday. Production of Synchrotron Radiation

Production of Synchrotron Radiation: relativistic electrons



Synchrotron radiation

$$\gamma = \frac{1}{\sqrt{1-\frac{v^2}{c^2}}} = \frac{1}{\sqrt{1-\beta^2}} \; ; \quad \beta = \frac{v}{c}$$

$$E_e = \gamma m c^2, \quad \boldsymbol{p} = \gamma m \mathbf{v}$$

$$\gamma = \frac{E_e}{mc^2} = 1957\, E_e(\mathrm{GeV})$$

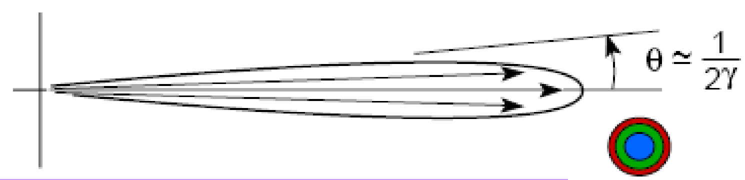
$$\hbar\omega \cdot \lambda = 1239.842 \text{ eV} \cdot \text{nm}$$

$$1 \text{ watt} \Rightarrow 5.034 \times 10^{15} \lambda[\text{nm}] \frac{\text{photons}}{\text{s}}$$

Frame moving with electrons

Lorentz Transformation

Laboratory frame of reference



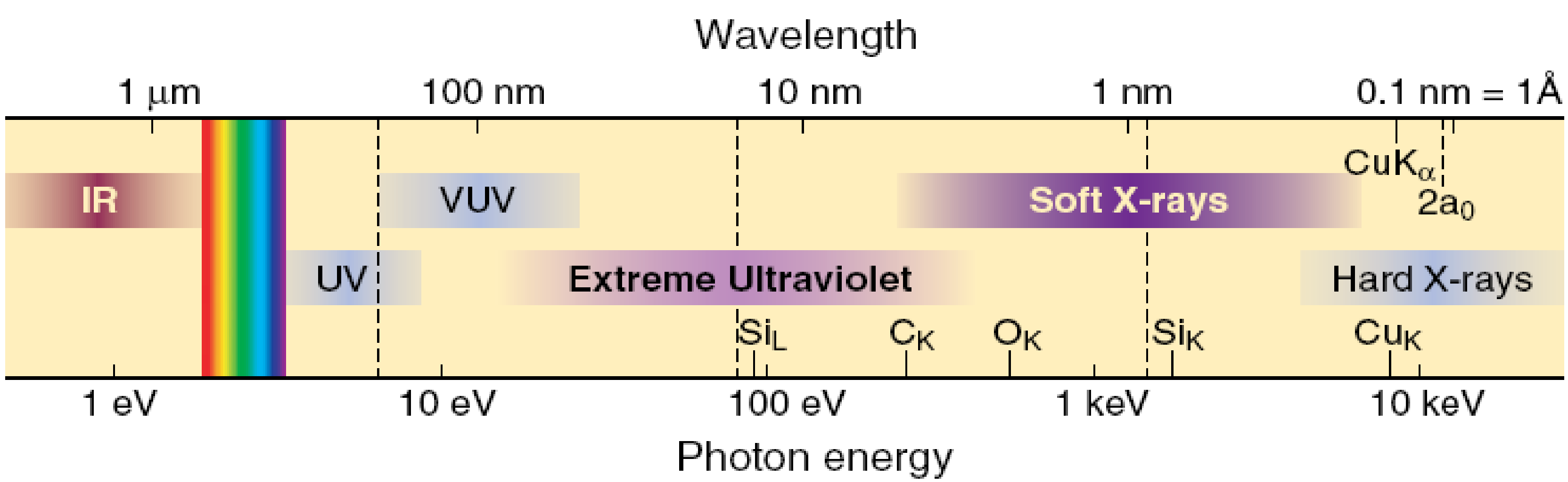
Homework n. 2

Only electrons radiate?
What about ions? Positrons?

Basics of Synchrotron Radiation 1 (a bit of history)

X-Ray dates http://xdb.lbl.gov/Section2/Sec_2-2.html

1909: Barkla and Sadler discover characteristic x-ray radiation (1917 Nobel Prize, Barkla)
1912: von Laue, Friedrich, and Knipping observe x-ray diffraction (1914 Nobel Prize, von Laue)
1913: Bragg, father and son, build an x-ray spectrometer (1915 Nobel Prize)
1913: Moseley develops quantitative x-ray spectroscopy and Moseley's Law
1916: Siegbahn and Stenstrom observe emission satellites (1924 Nobel Prize, Siegbahn)
1921: Wentzel observes two-electron excitations
1922: Meitner discovers Auger electrons
1924: Lindh and Lundquist resolve chemical shifts
1927: Coster and Druyvesteyn observe valence-core multiplets
1931: Johann develops bent-crystal spectroscopy



Basics of Synchrotron Radiation 2

http://www.lightsources.org/cms/ In section history

>1920 Interest in the radiation as an energy-loss mechanism

1930-40 Betatron, i.e. X-rays production via electron accelerators

1940-41 Kerst (Univ. Illinois) first 2.3 MeV betatron
General Electrics

1944 Ivanenko and Pomeranchuk (USSR) ,calculations of energy losses due to radiating electrons

1945 Schwinger (US), theory (non-relativistic) on dipole Radiation of circular trajectories (not published)
1965 Nobel Prize for relativistic electrodynamics

1949 Sokolov and Tersov (USSR) quantum calculation

However these early betatrons (General Electrics) did not have transparent vacuum tubes.

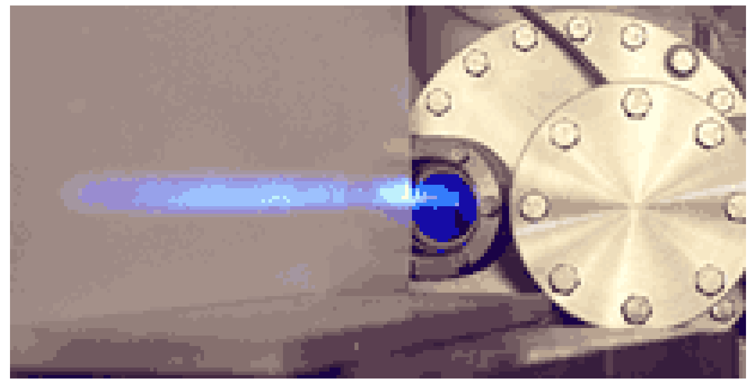
Basics of Synchrotron Radiation 3

http://www.lightsources.org/cms/ In section history

24 April 1947, H. Pollock, R. Langmuir, F. Elder and A. Gurewitsch saw a gleam of bluish-white light emerging from the transparent vacuum tube of their new 70 MeV electron synchrotron at General Electric's Research Laboratory, Schenectady, New York.

Synchrotron radiation had been seen.

First visible light for machine testing NSLS (US), 1982.

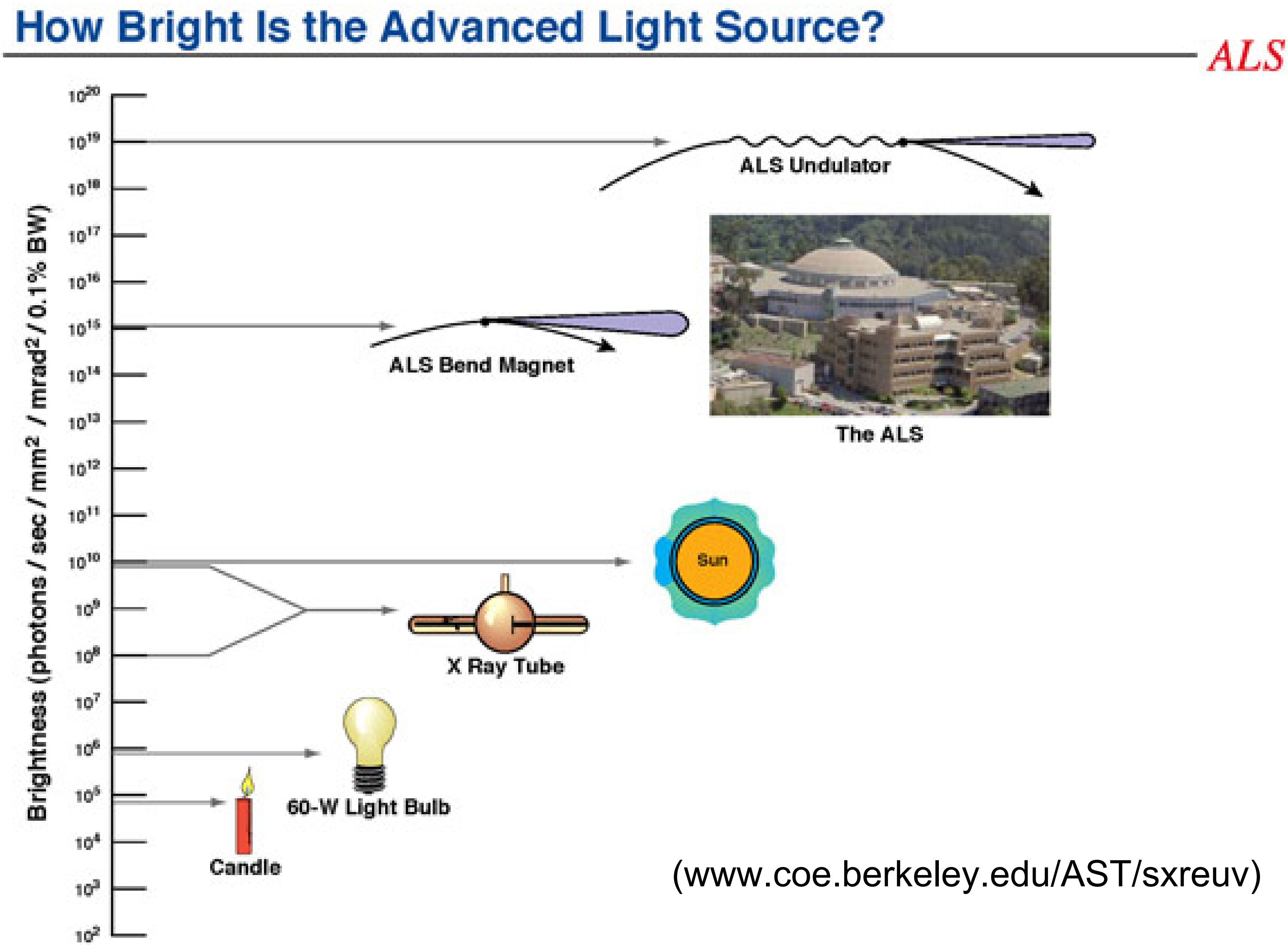


Homework 3
Three Generations of Synchrotrons

1) Parasitic
2) Storage Rings
3) Dedicated Sources

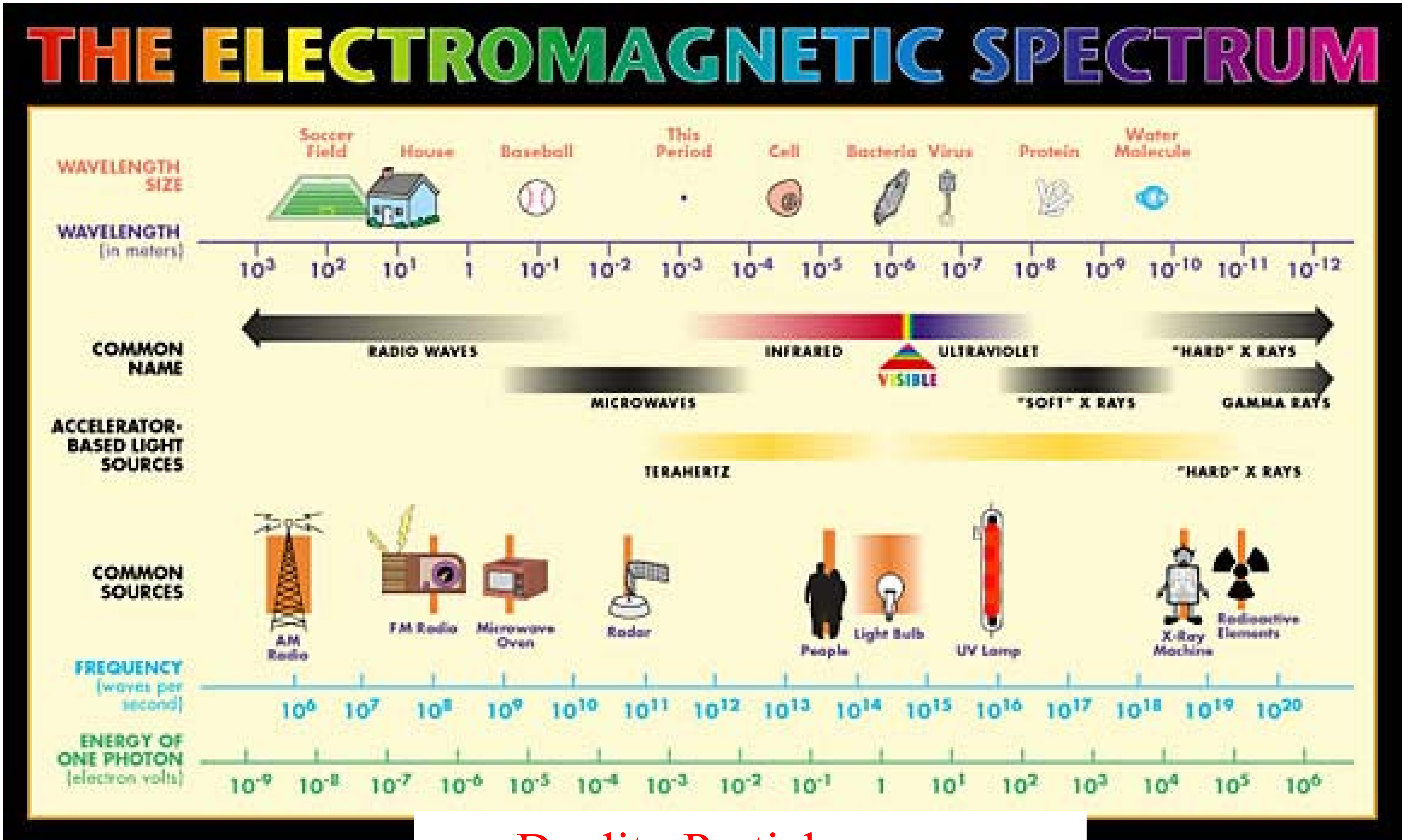
What about fourth generation?

Why Synchrotron Radiation? 1



(www.coe.berkeley.edu/AST/sxreuv)

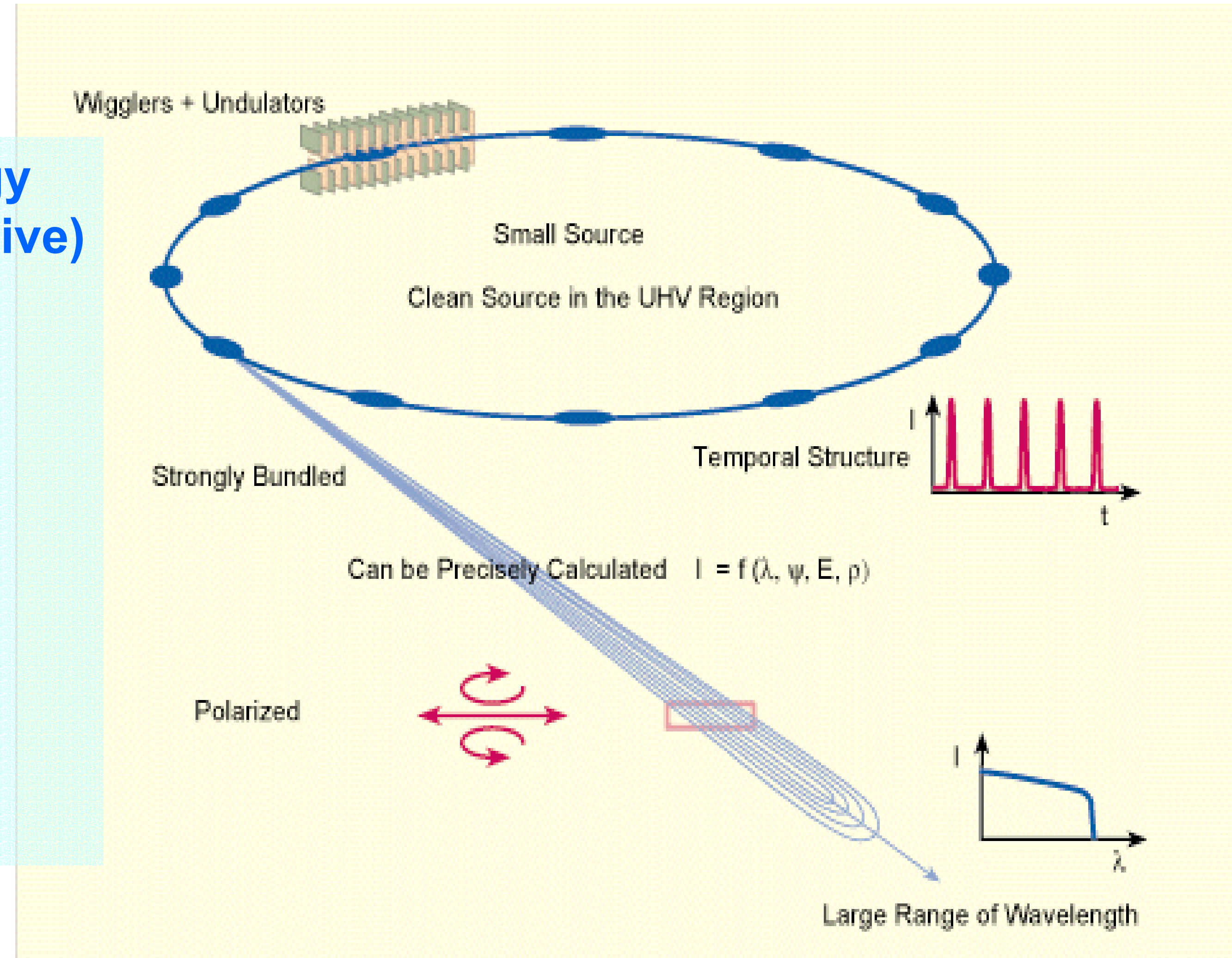
Why Synchrotron Radiation? 2



Duality Particle-waves
Extremely broad spectral range
Vision of the 'invisible'

Characteristics of Synchrotron Radiation

- **Tunable Energy (Element Selective)**
- **High Intensity**
- Time Structure
- **Polarization**
- Coherence
- Focalisation



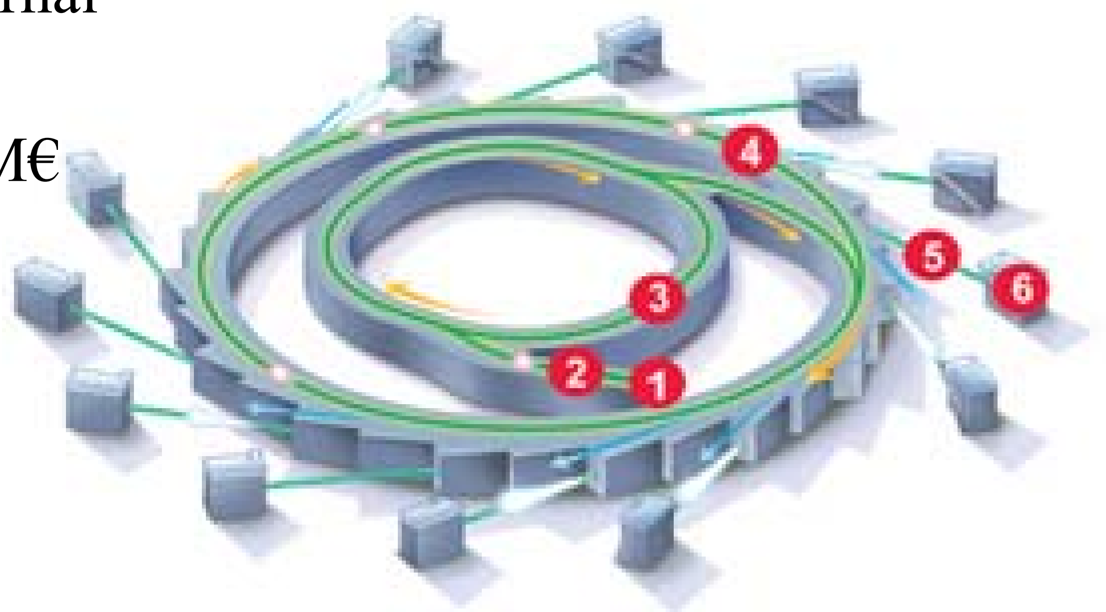
Why Synchrotron Radiation? **Facilities for users**

Costs, <u>average 1000/4000 euro/hour</u>

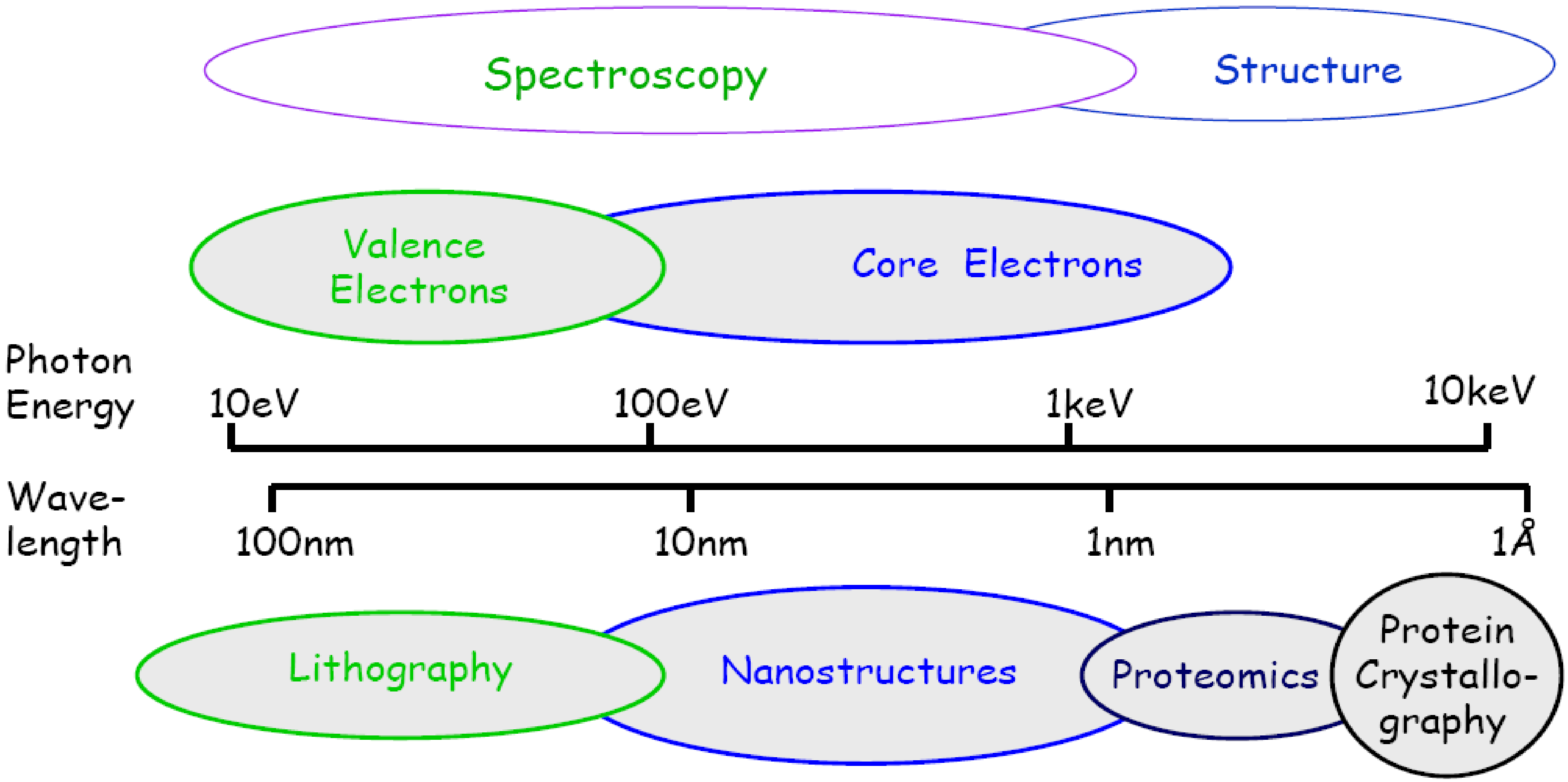
<u>Experiments have time limit, fixed time window</u>

1. electron gun
2. linac
3. booster ring
4. storage ring
5. beamline
6. end station

- 70% beamtime open to external users
- Program committee, external members
- Annual Budget: 30/200 M€
- 700/2000 users/yrs
- 50% users from abroad
- Staff: 400 persons
- 24/24 h, >5000 hrs/yr



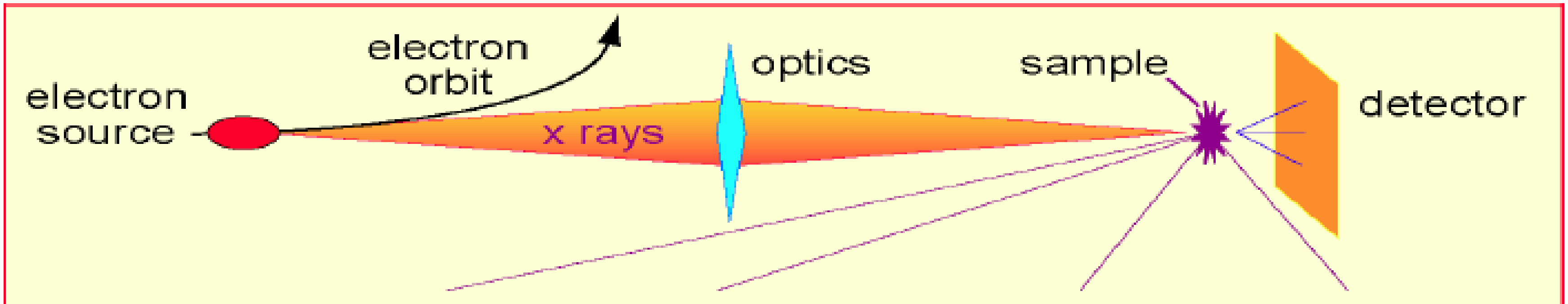
Science with Light Sources



Adopted from: Franz Himpsel, CMMP '07

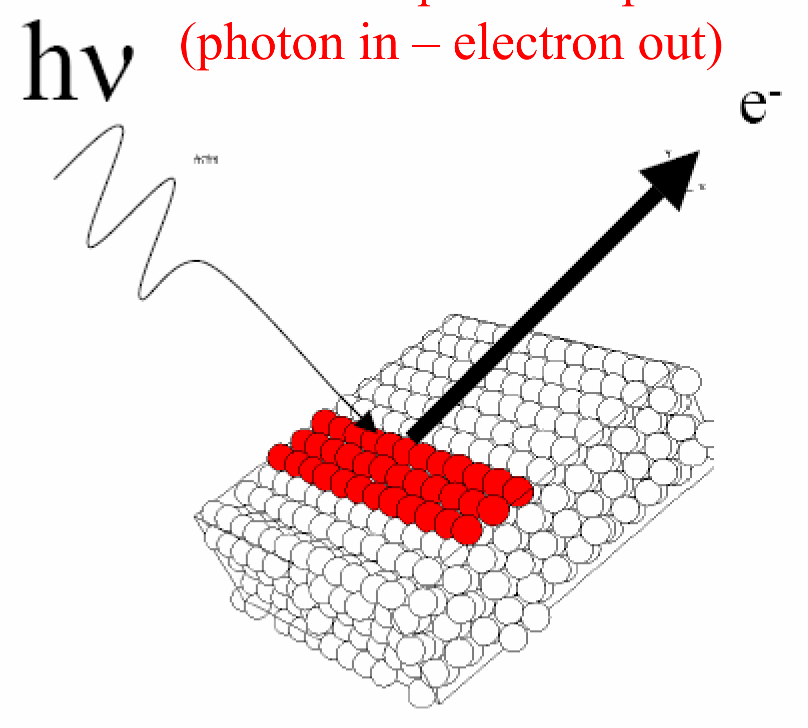
Z. Hussain, UCB lecture, 2007

What experiment at Synchrotron Radiation?



SPECTROSCOPY	SCATTERING	IMAGING	DYNAMICS
Energy	*Momentum*	*Position*	*Time*

Electron Spectroscopies
(photon in – electron out)



Photon (probe mode):

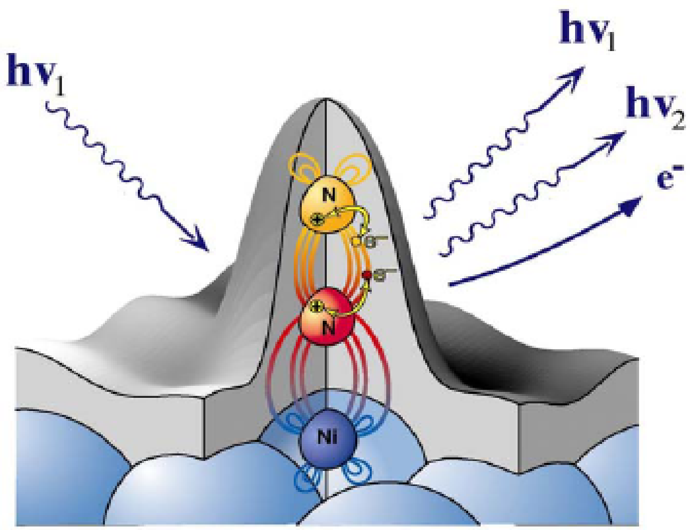
- Energy
- Polarisation

Electron (detection mode):

- Energy
- Spin
- Angle

Photon Interaction

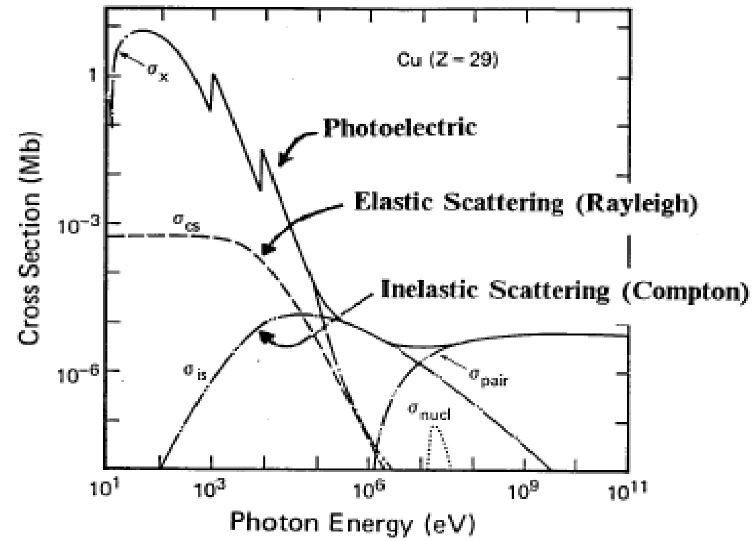
Incident photon interacts with electrons
Core and Valence



- Photon is
  - Adsorbed
  - Elastic Scattered
  - Inelastic Scattered

- Electron is
  - Emitted
  - Excitated
  - Dexcitated

Cross Sections



Stöhr, NEXAPS spectroscopy

Below 100 keV

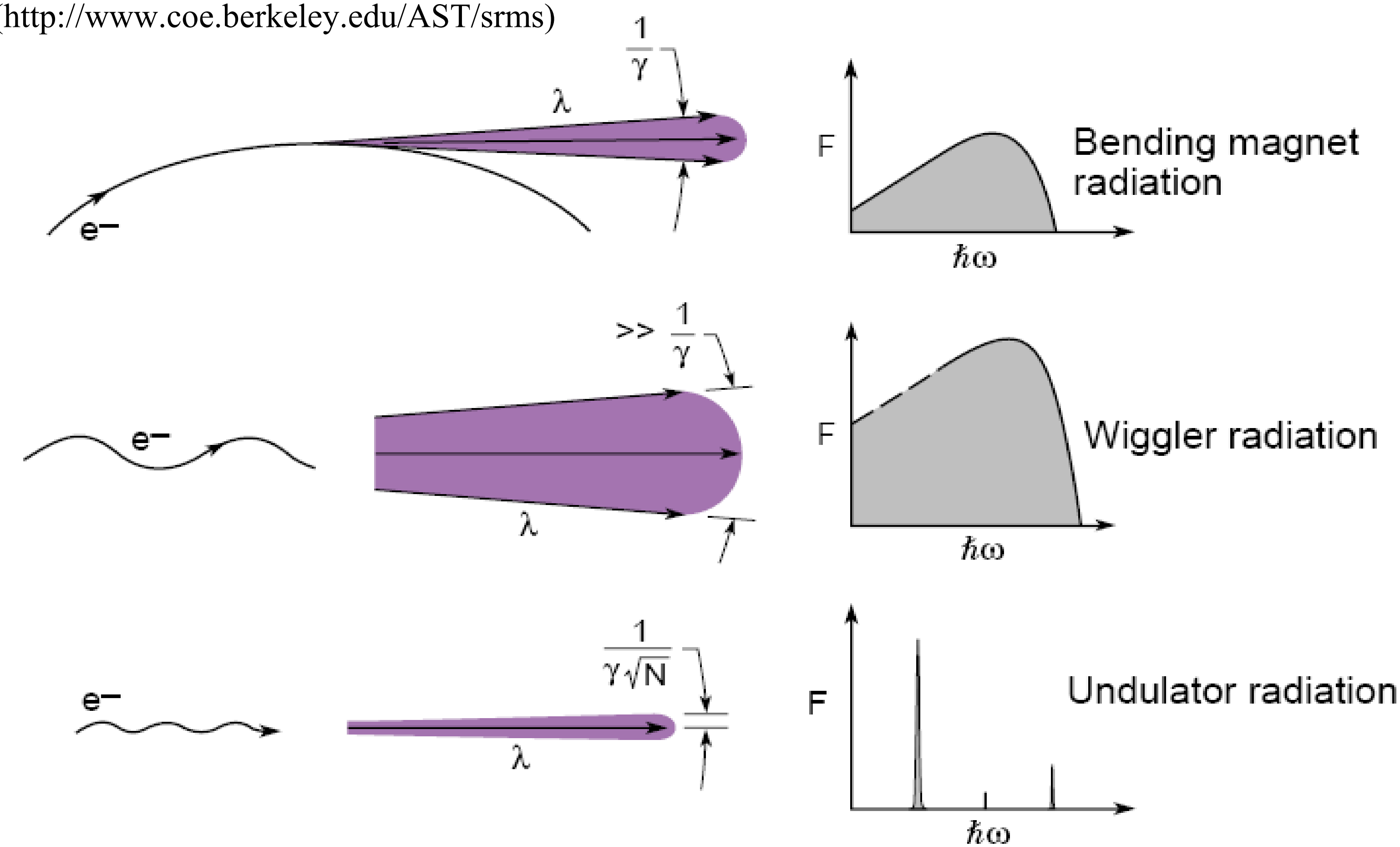
Photoelectric cross section dominates

Spectroscopy

A. Nillson, SSRL and Stockholm Univ.

Photon sources and insertion devices

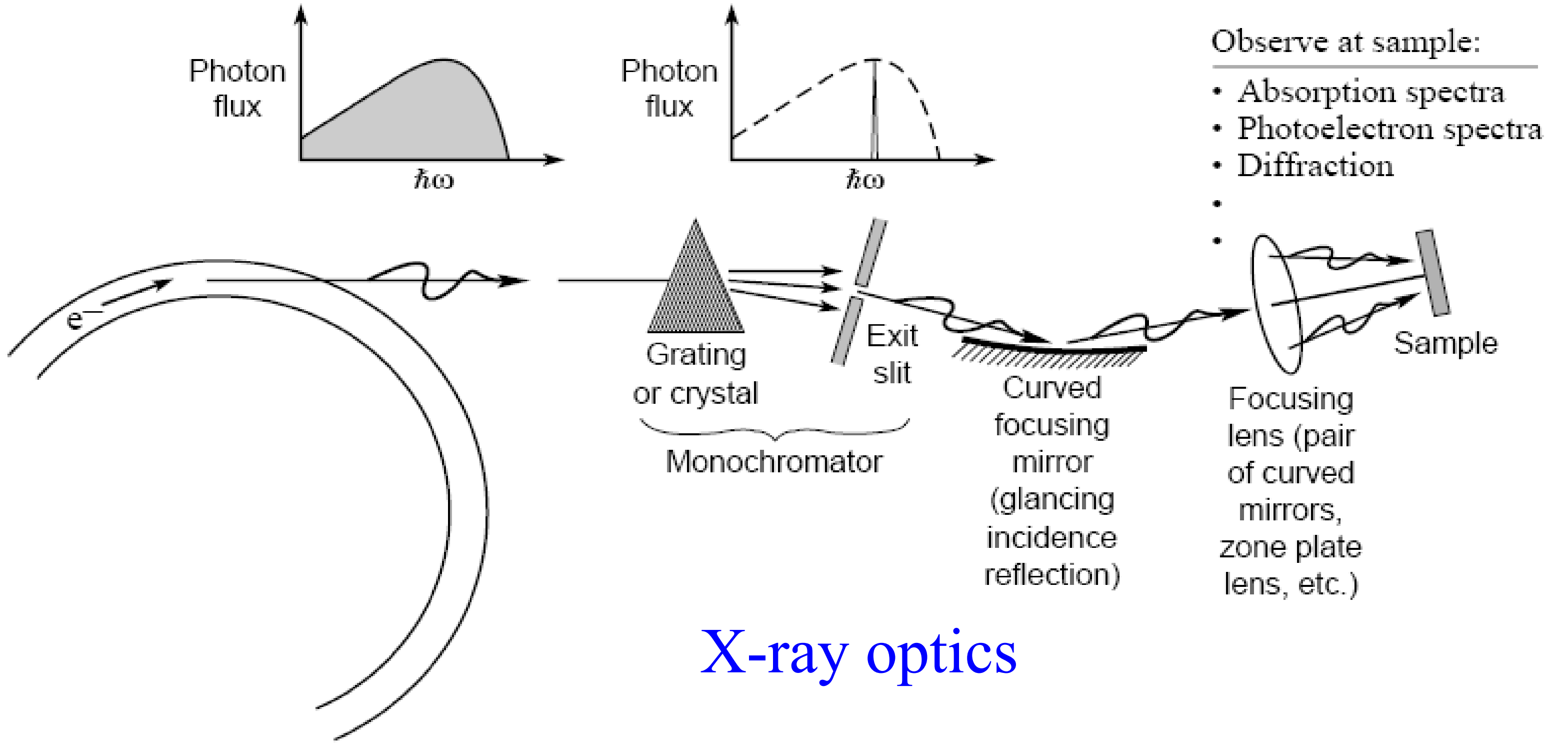
(http://www.coe.berkeley.edu/AST/srms)



Lesson II and III, next Thursday. Bending Magnets, Undulators, Wigglers

Beamline: from the source to the sample

(http://www.coe.berkeley.edu/AST/srms)



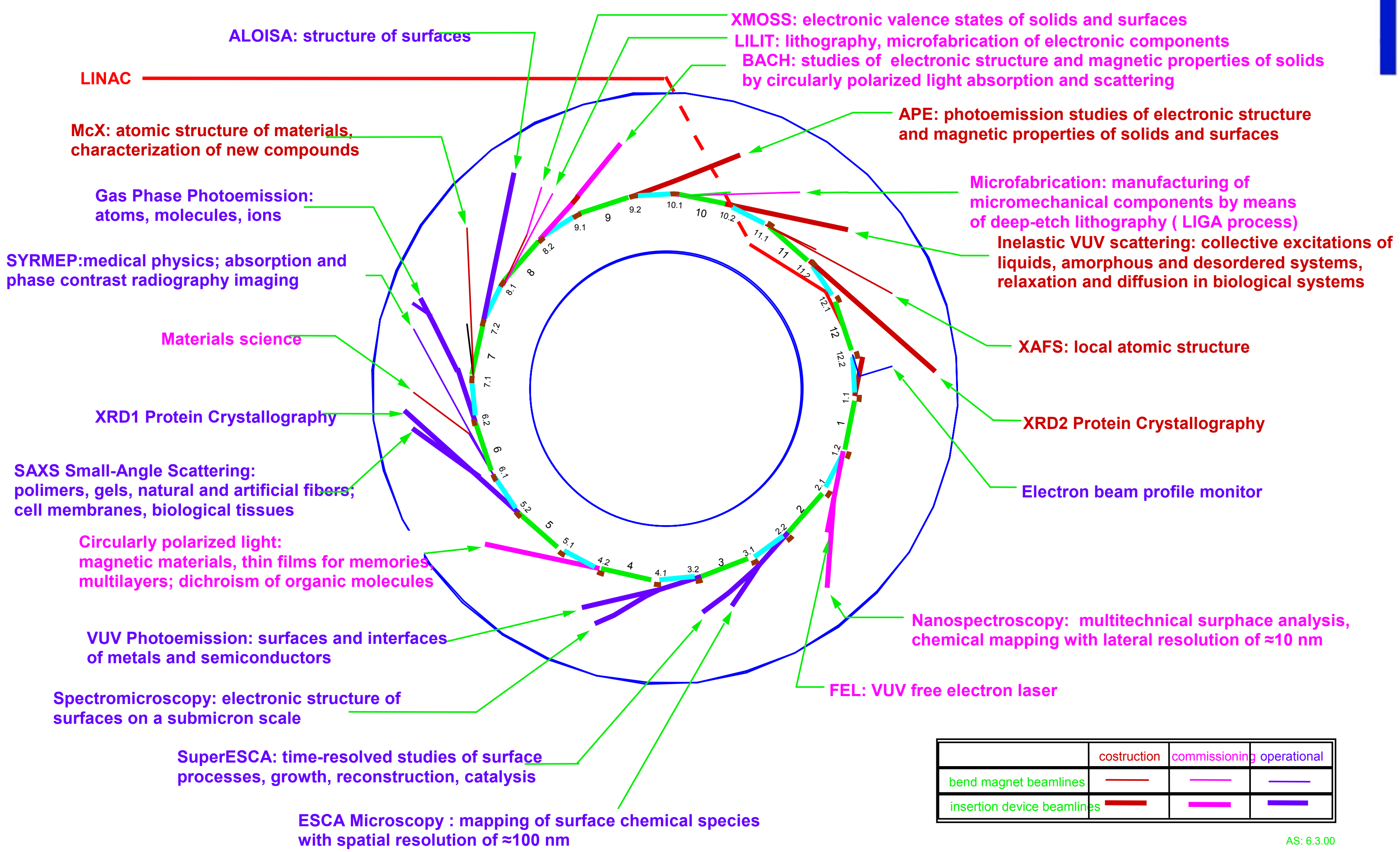
X-ray optics

Energy selection

Energy resolution vs. flux

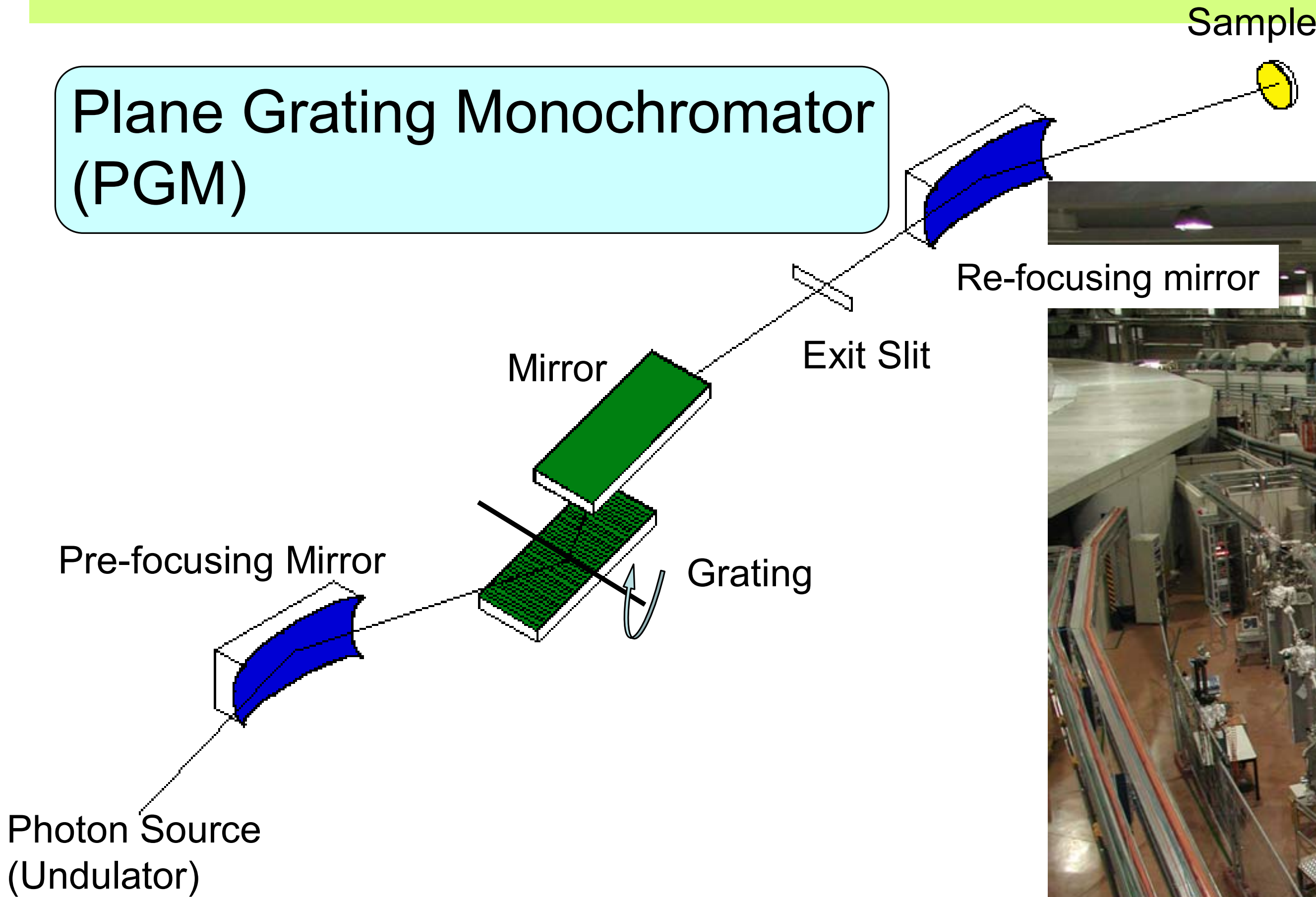
Focusing

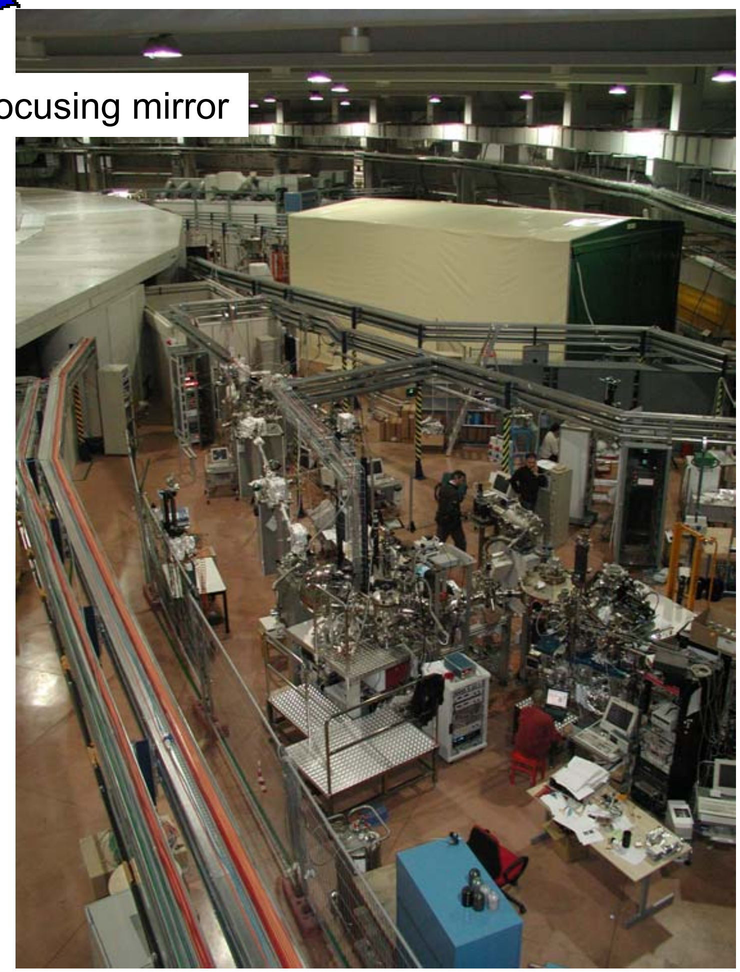
Beamlines and scientific activity



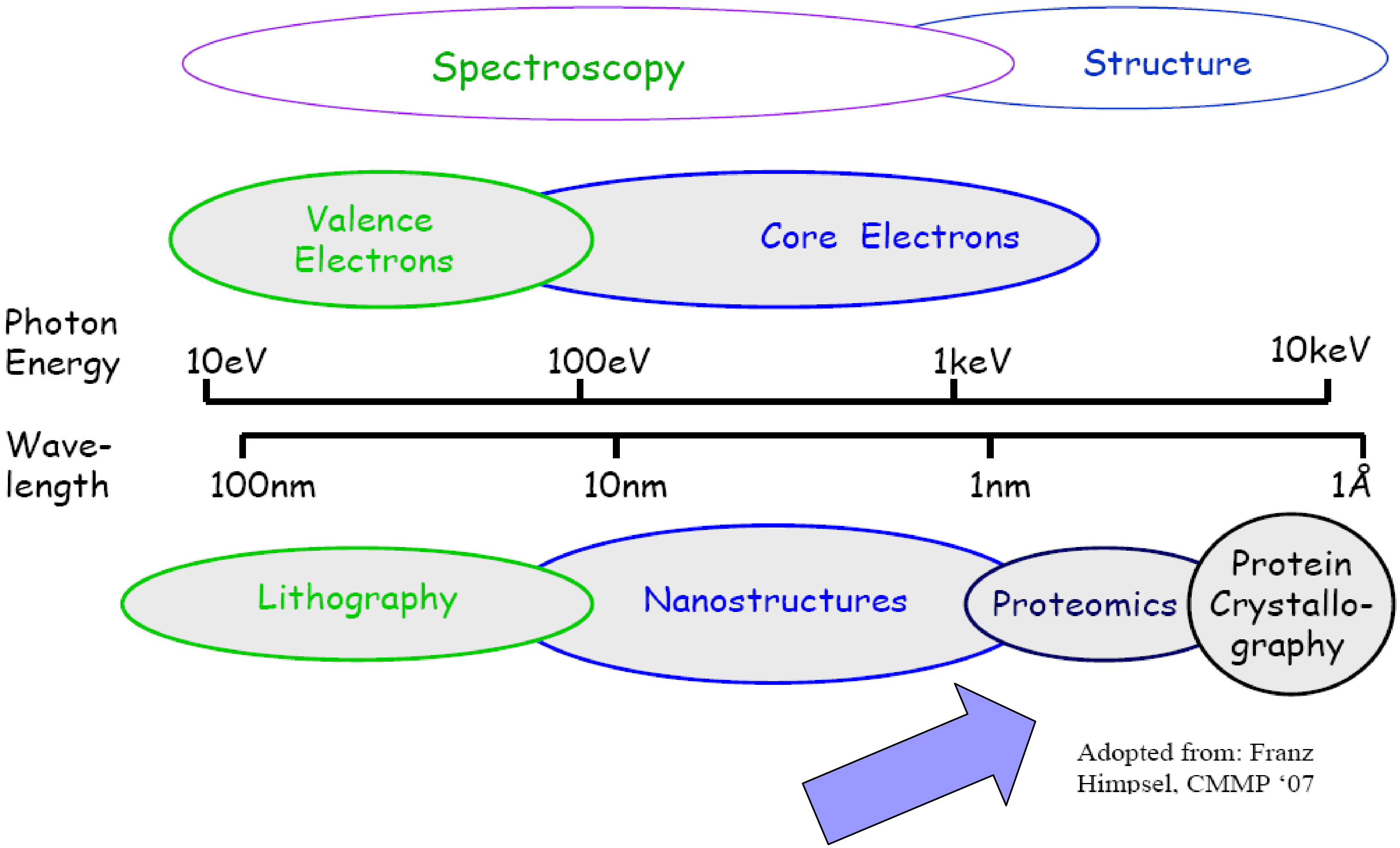
Beamlines and scientific activity

Plane Grating Monochromator (PGM)





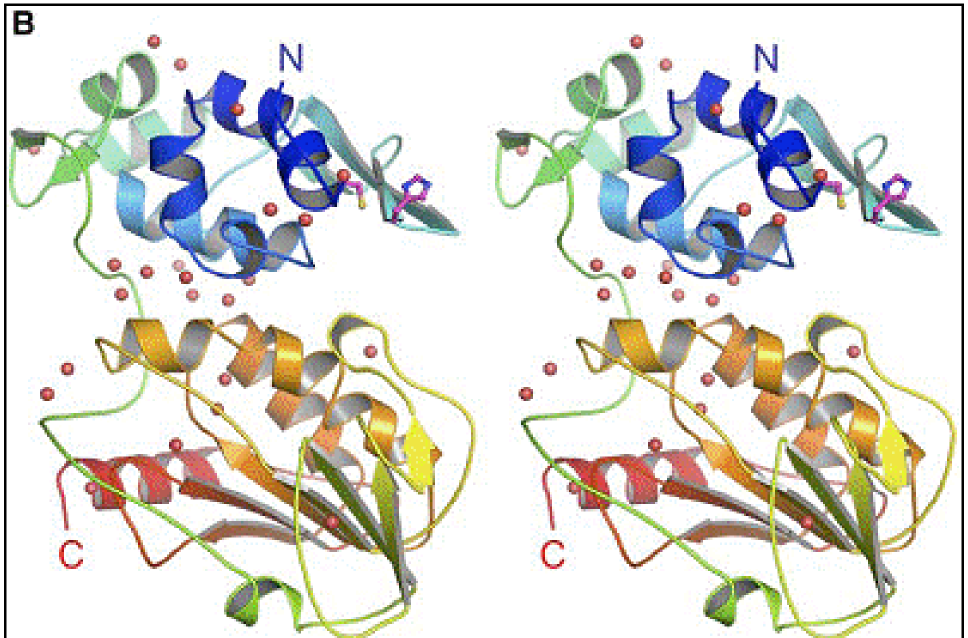
Science with Light Sources



Z. Hussain, UCB lecture, 2007

Example 1 : Protein Crystallography

Baton Rouge CAMD beamline (http://www.camd.lsu.edu/)



(B) Ribbon diagram of nsP2pro colored from blue (N terminus) to red (C terminus). red spheres correspond to bound water .

STEP 1. Background

Revealing the structure of a protein that the Venezuelan Equine Encephalitis (VEE) virus requires for replication.
VEE is a mosquito-borne virus found in Central and South America, and southern Texas. The virus was developed into a biological weapon during the Cold War.

STEP 2. Scope of the experiment

Developing an inhibitor of nsP2 protease to prevent the virus from replicating and causing encephalitis.

STEP 3. Preparation and Results

Dr. A. Russo, (Univ. of Texas at Galveston (UTMB), solved the structure of a protein called nsP2 protease.
It is an enzyme that divides a large viral protein into smaller segments at specific locations.
Only the smaller segments are active for the replication of the virus.

Example 2: Medical Diagnostic

(Syrmep medical beamline (ELETTRA-Italy), www.elettra.trieste.it)

Metastases formation

Increased details with image processing with SR

Earlier diagnose possible

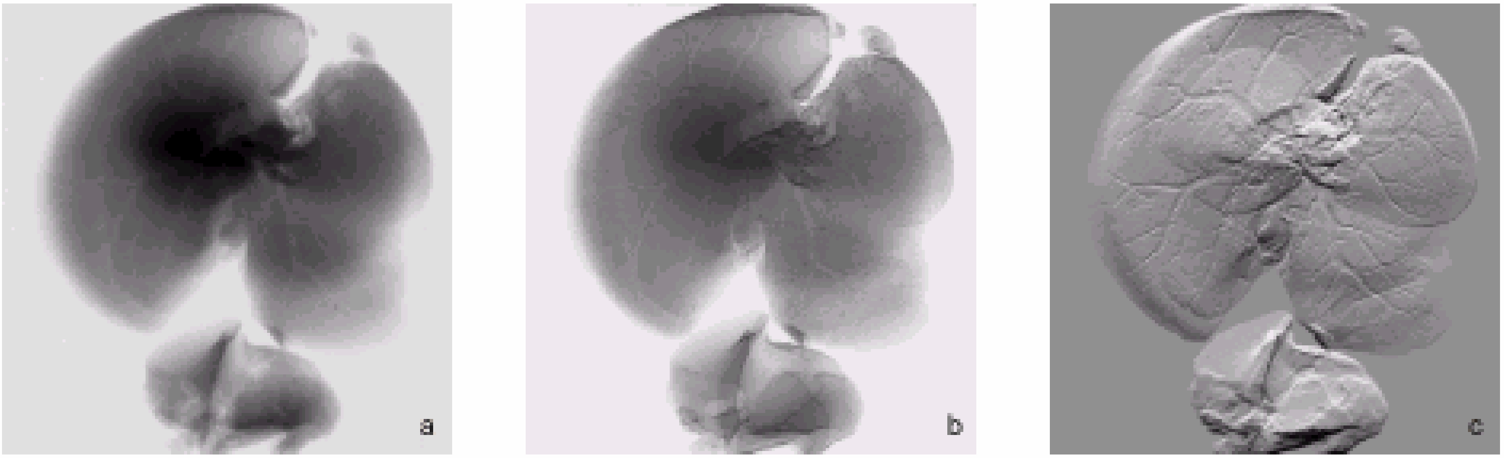
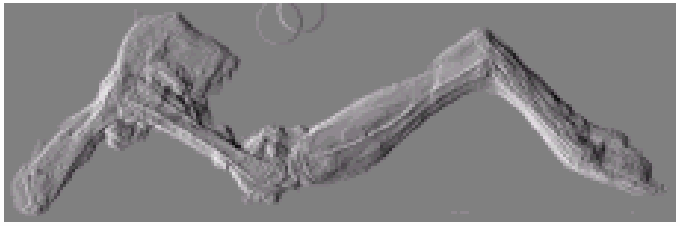


Figure 1. *DEI images of a preserved mouse liver. The first (a) shows a radiogram taken with no analyser crystal. This would be similar to, but somewhat better than an image taken with a conventional x-ray generator / film system. The next (b) shows the image taken with the analyser crystal at the peak of its reflectivity curve. Finally image (c) shows an image processed to show only the refraction changes.*

Images of mouse leg

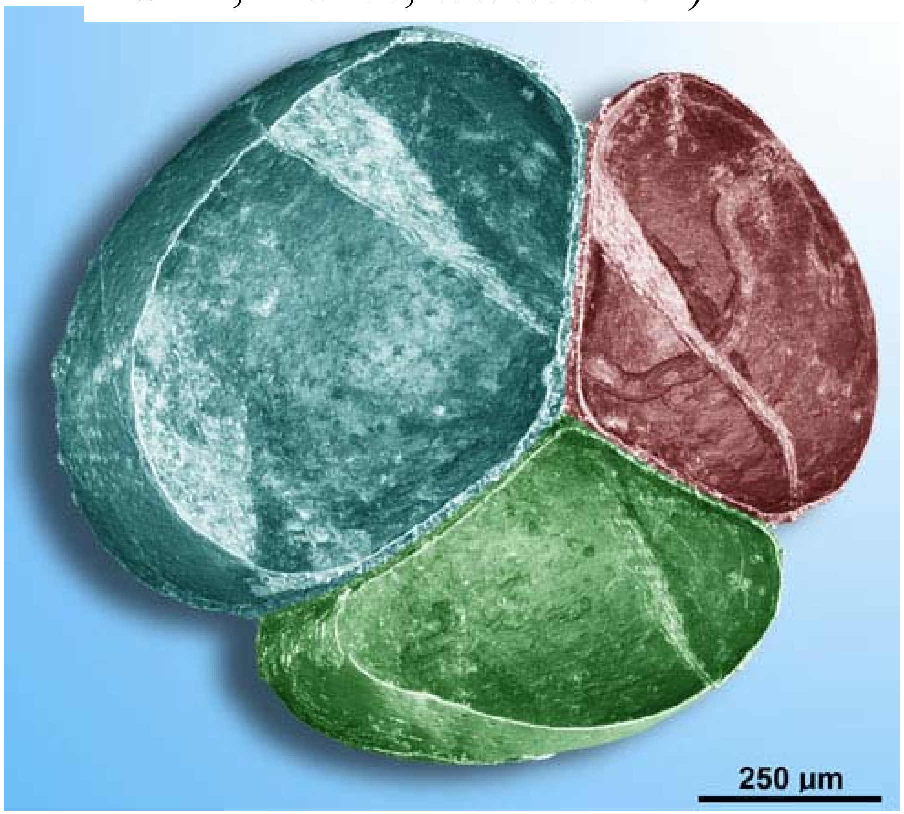
Conventional source



Synchrotron source

Example 3: Biology and Paleontology

(ID19 High-resolution Diffraction Topography Beamline, ESRF, France, www.esrf.fr)



Chinese embryo.
Different colours correspond to each of the 3 embryonic cells (blastomeres). Calculation of volume of each blastomere shows that the blue one is twice the volume of the others, strengthening the polar-lobe formation interpretation

STEP 1. Background

Origin of complex animals with symmetry in a central plane (bilaterian).
Embyonic development?

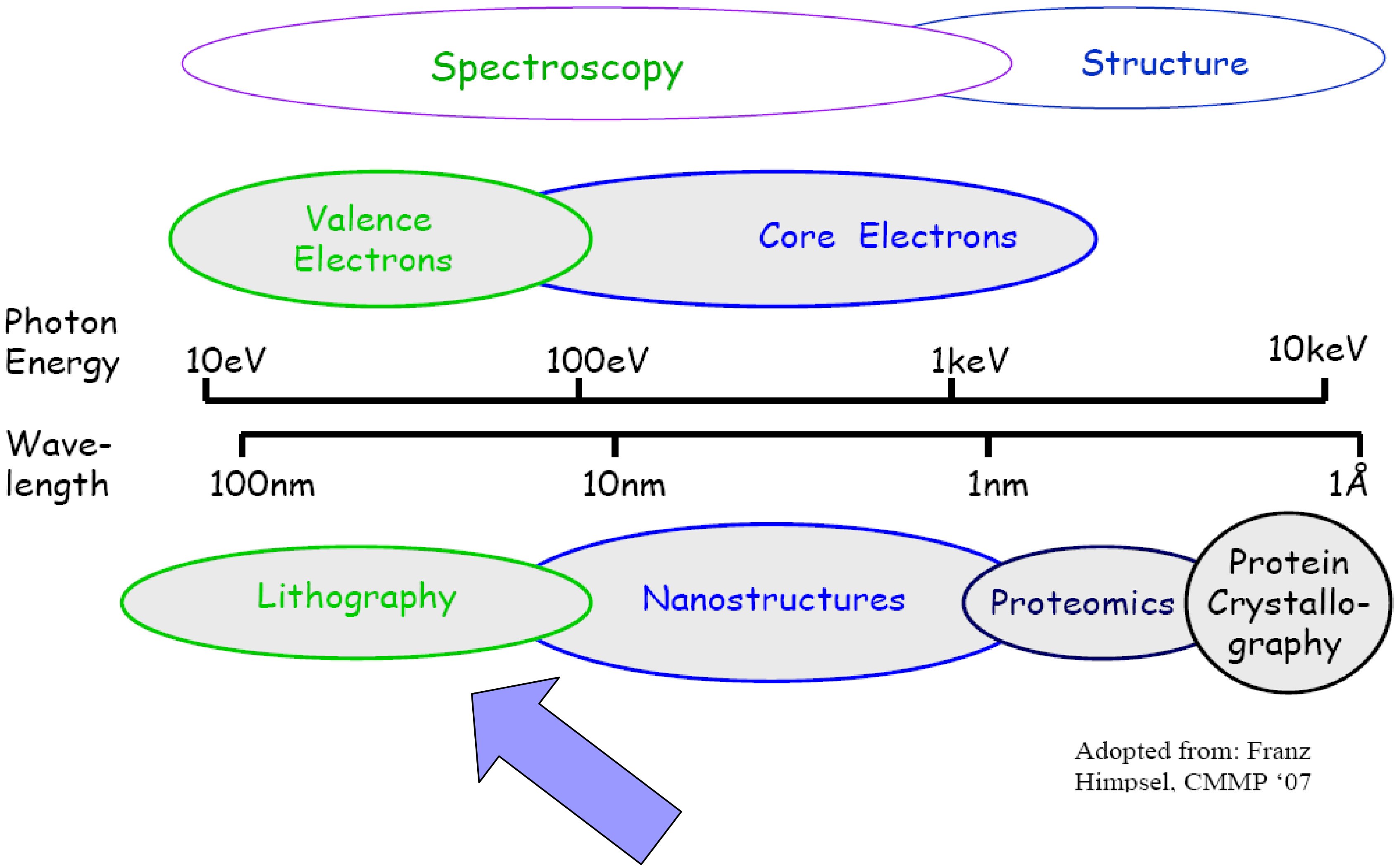
STEP 2. Scope of the experiment

To investigate the internal structures of fossilised embryos from China
in a non-destructive manner

STEP 3. Results

Striking resemblance between the cellular cleavage pattern in the fossil embryos and that of modern polar lobe-forming embryos (580 millions years ago). This is 40 million years earlier than previously thought.
J.Y. Chen et al. Science 312, 1644 (2006)

Science with Light Sources

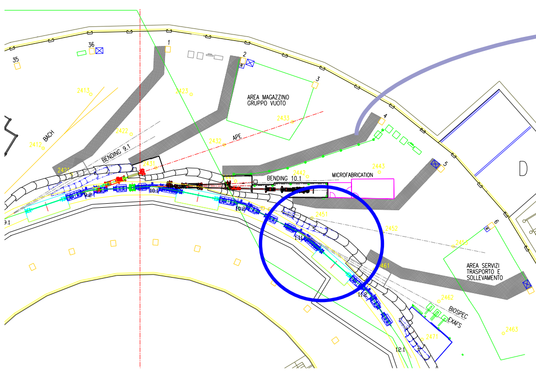


Adopted from: Franz Himpsel, CMMP '07

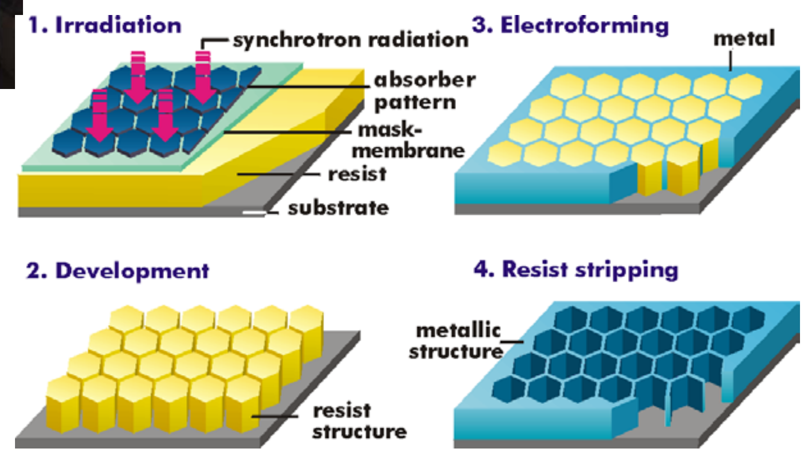
Z. Hussain, UCB lecture, 2007

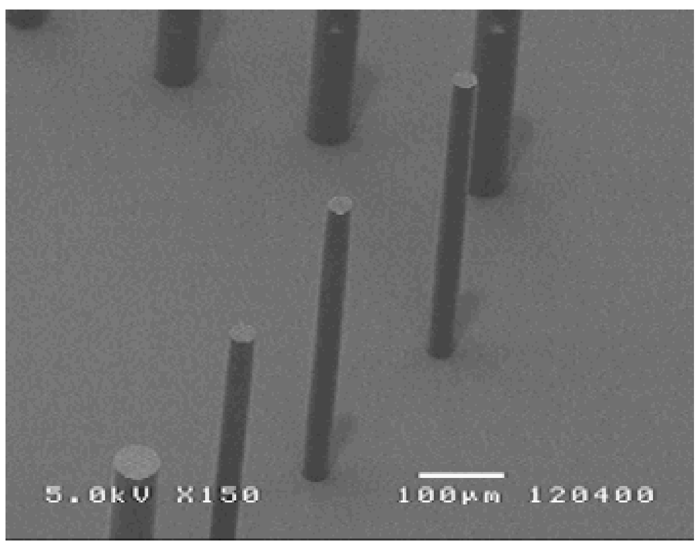
Example 4: Production of micro-nano objects

(Deep X-ray Litography beamline (ELETTRA-Italy), www.elettra.trieste.it)



LIGA process

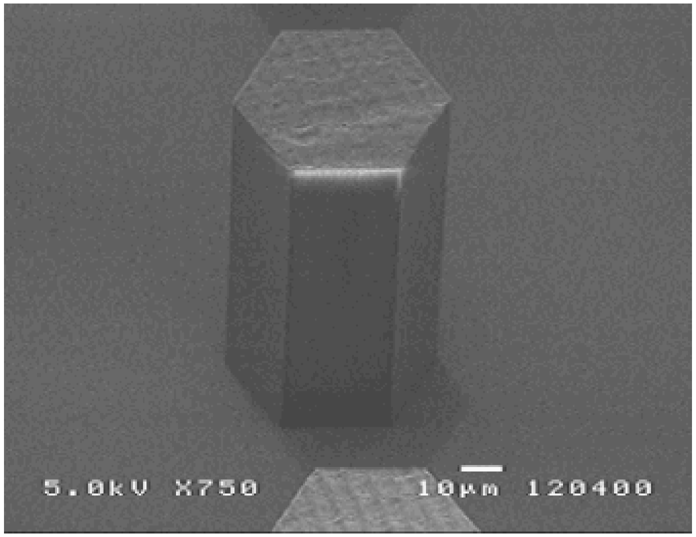


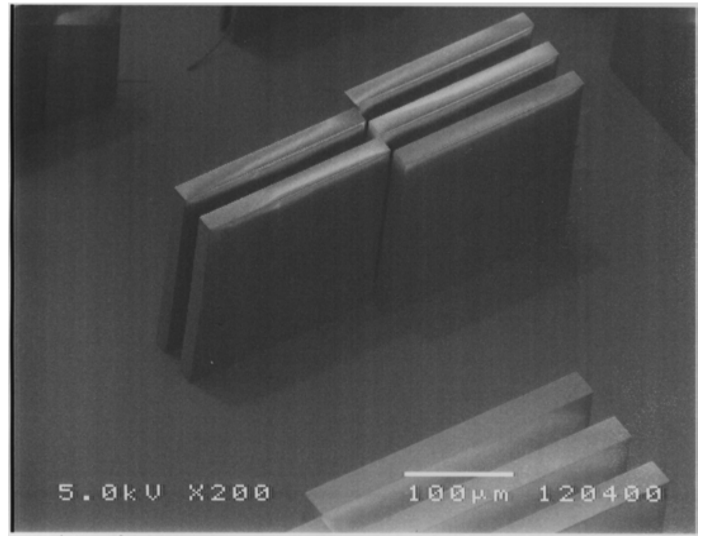


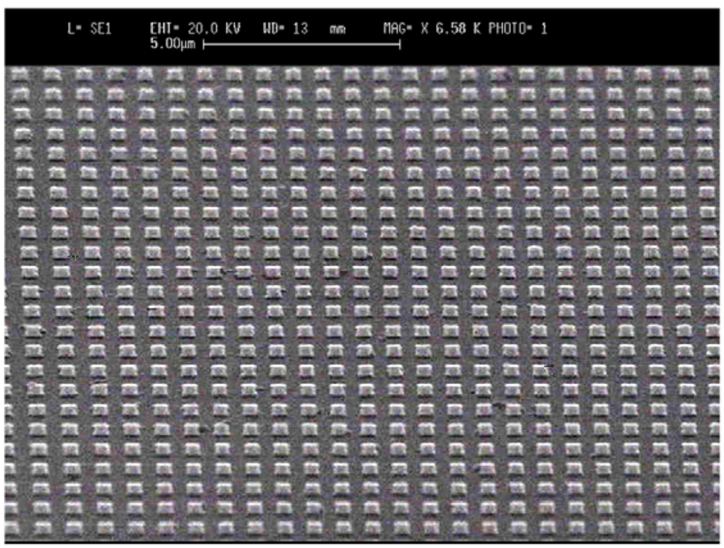
SEM images after LIGA process
PMMA structures

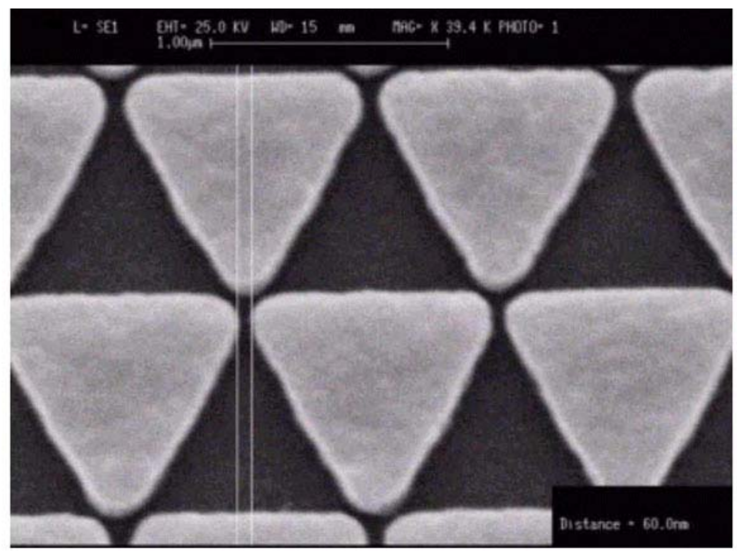
Example 4: Production of micro-nano objects

(Deep X-ray Litography beamline (ELETTRA-Italy), www.elettra.trieste.it)

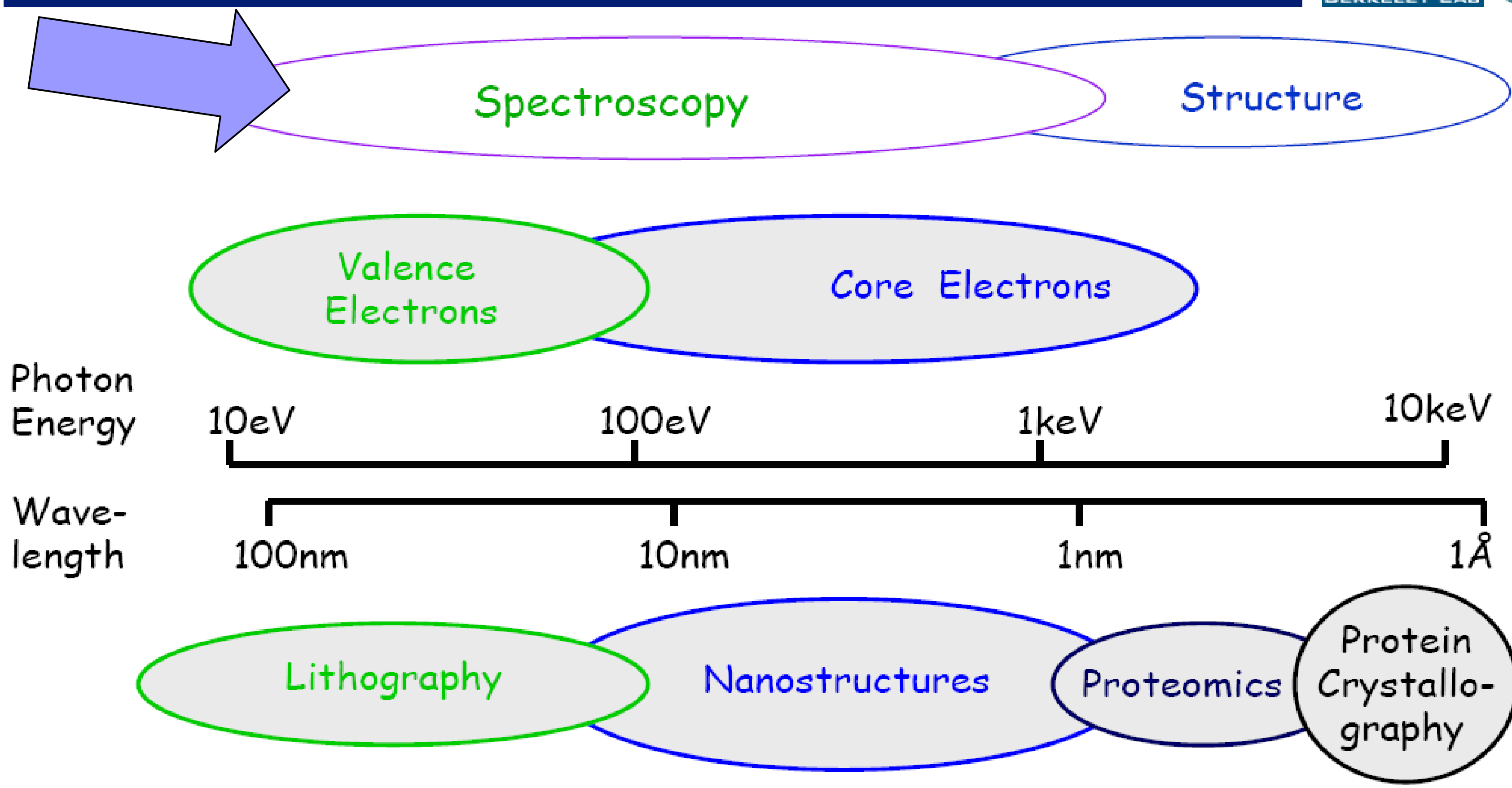








Science with Light Sources



Adopted from: Franz Himpsel, CMMP '07

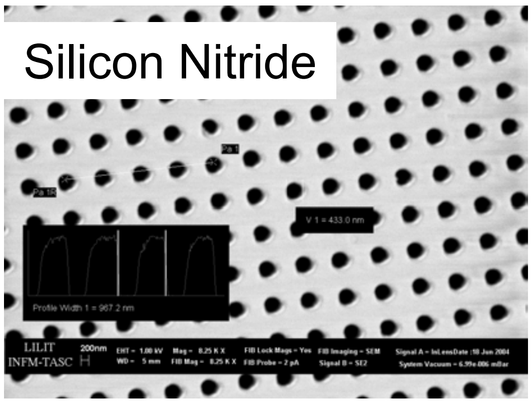
Z. Hussain, UCB lecture, 2007

Part 3 and Part 4. Spectroscopy:

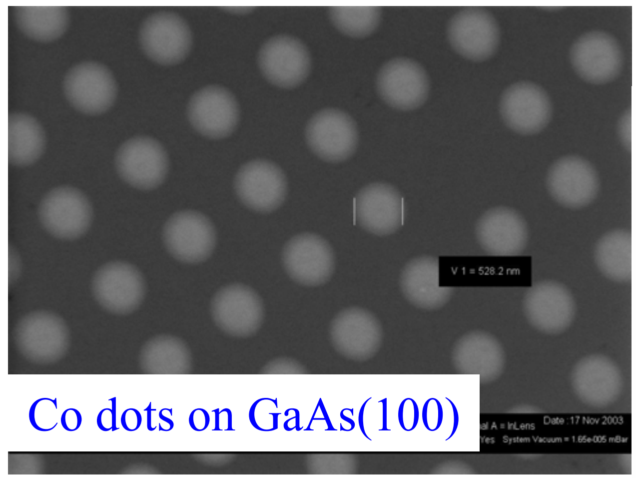
NANO = Lithography + Spectroscopy + Focalisation of SR

Aim = Study of the collective vs. local properties in laterally confined systems

Silicon Nitride

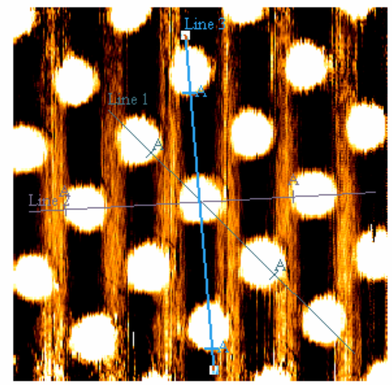


Diameter ~ 420nm Period ~ 1 micron

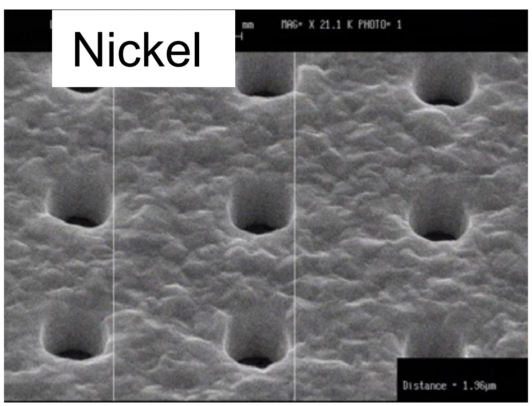


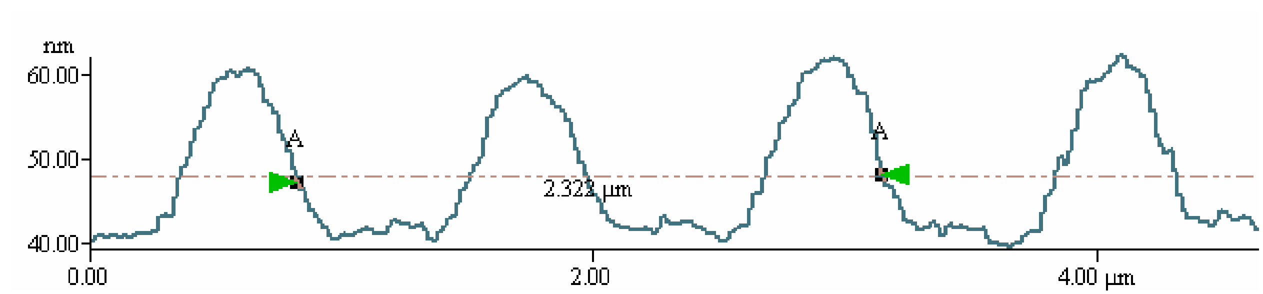
Co dots on GaAs(100)

Diameter 530 nm

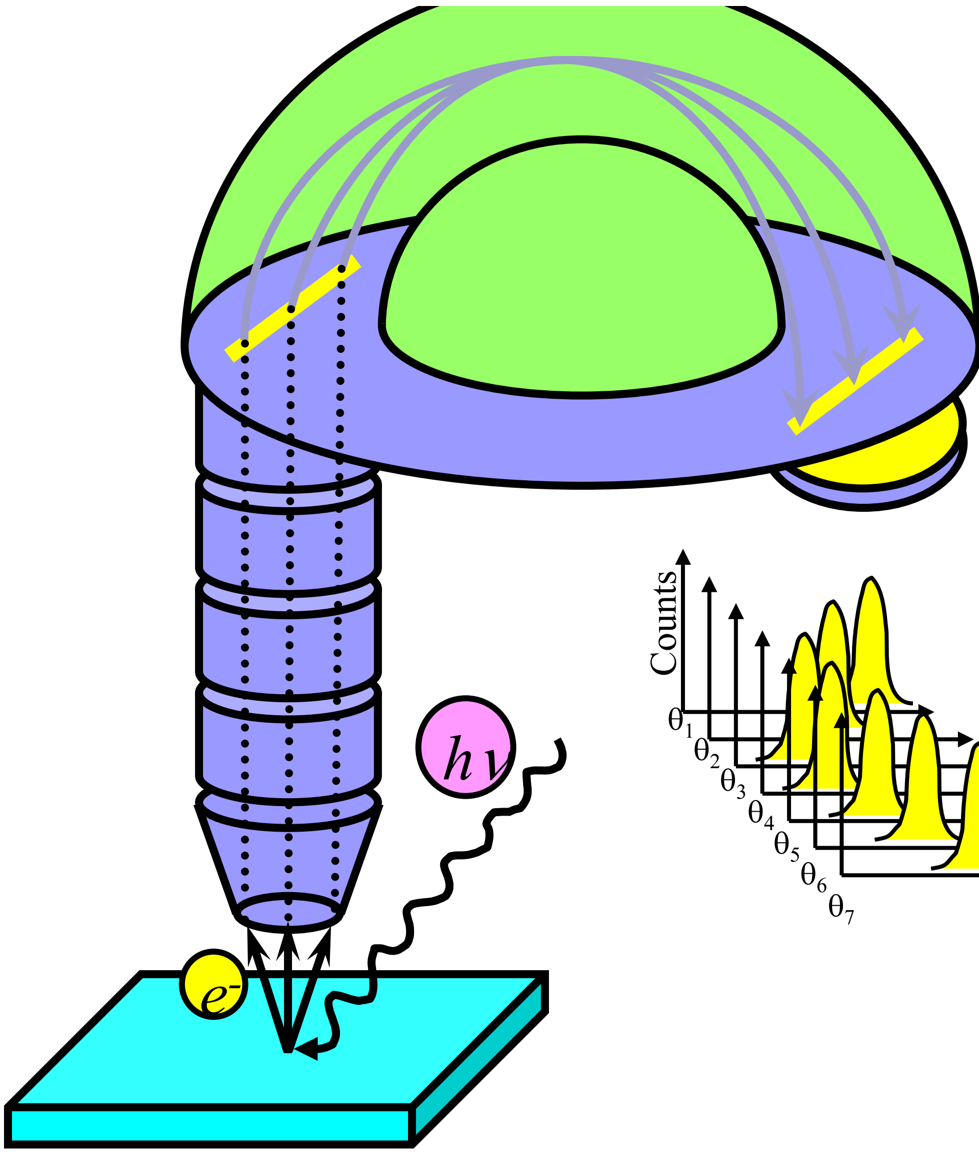


Nickel





Part 3 and Part 4. Spectroscopy:
Photoemission (Regimes of Photoemission, UPS, XPS…)

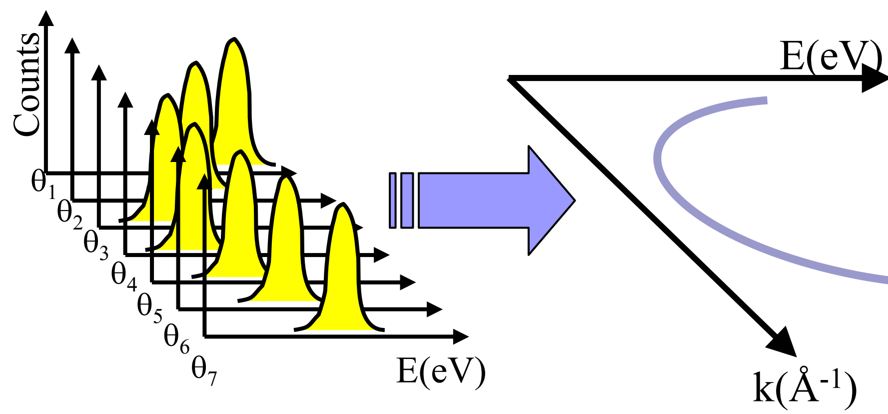


1) High Energy Resolution

1 – 10 meV @ Kinetic Energy < 50 eV

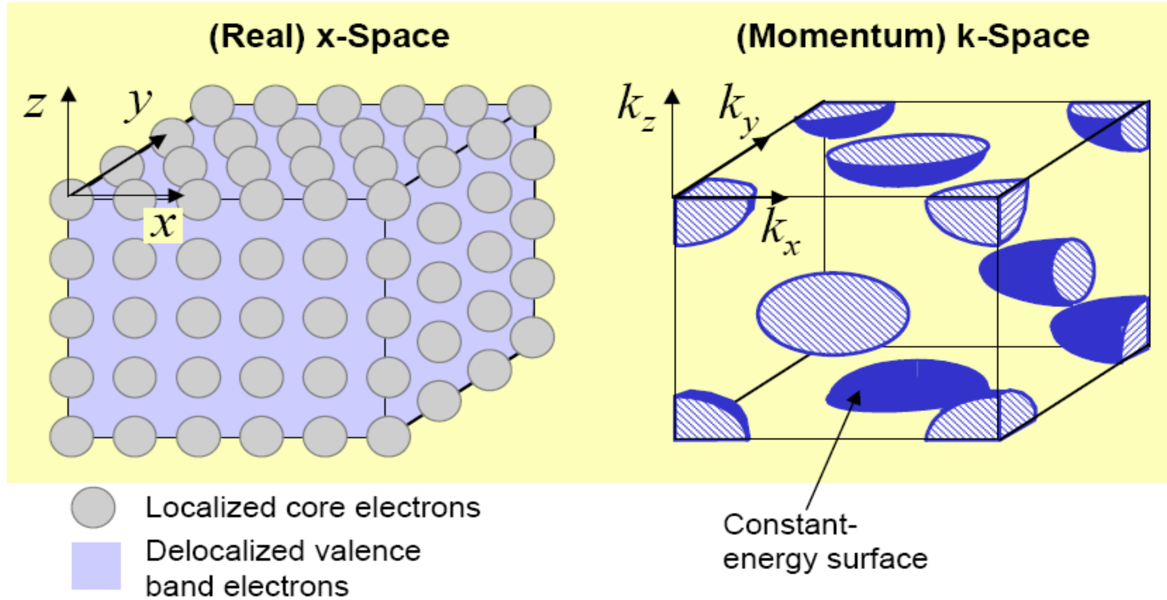
~ 100 meV @ Kinetic Energy > 500 eV

2) E = E(**k**)

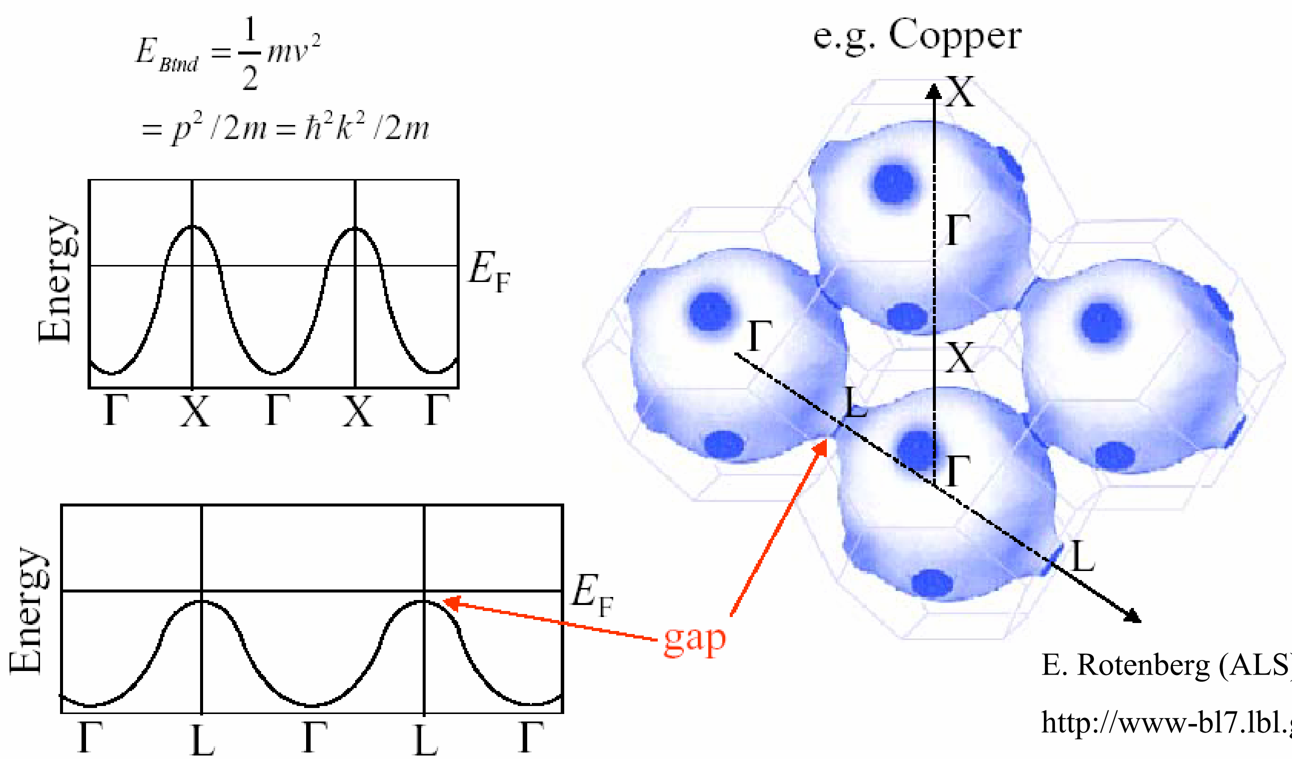


Homework n. 4
Regimes of Photoemission
UPS, XPS ??

Band structure via Photoemission



$$E_{Bind} = \frac{1}{2}mv^2$$
$$= p^2/2m = \hbar^2 k^2/2m$$

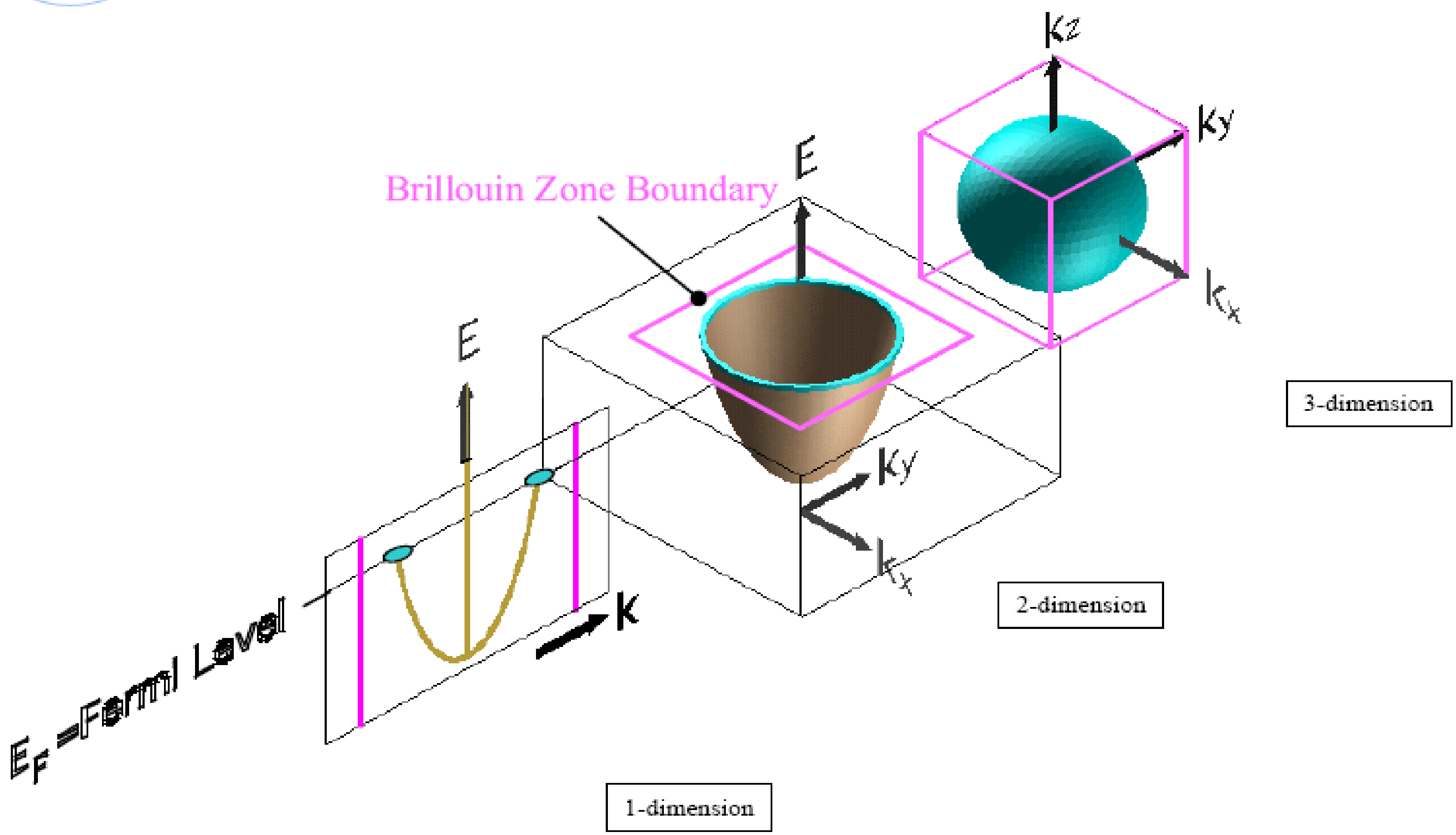


E. Rotenberg (ALS)

http://www-bl7.lbl.gov/BL7/who/eli/SRSchoolER.pdf

The Fermi Surface

ALS



2001 Berkeley-Stanford Summer School

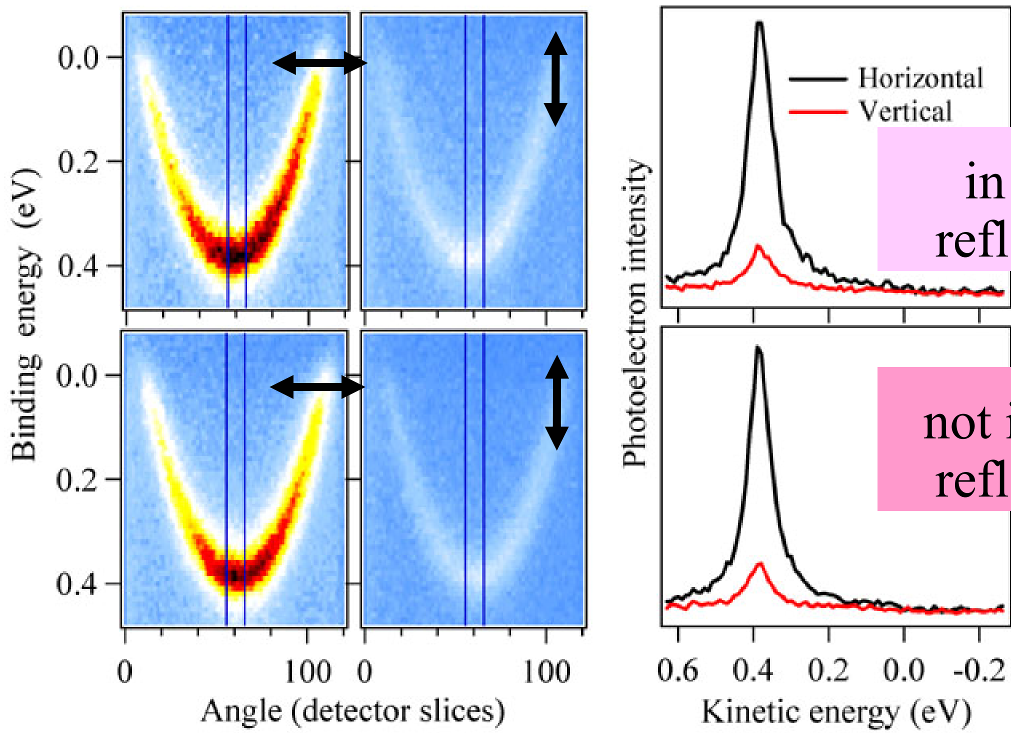
Eli Rotenberg

From http://www-bl7.lbl.gov/BL7/who/eli/eli.html, Tutorial on photoemission

Angular Resolved PES

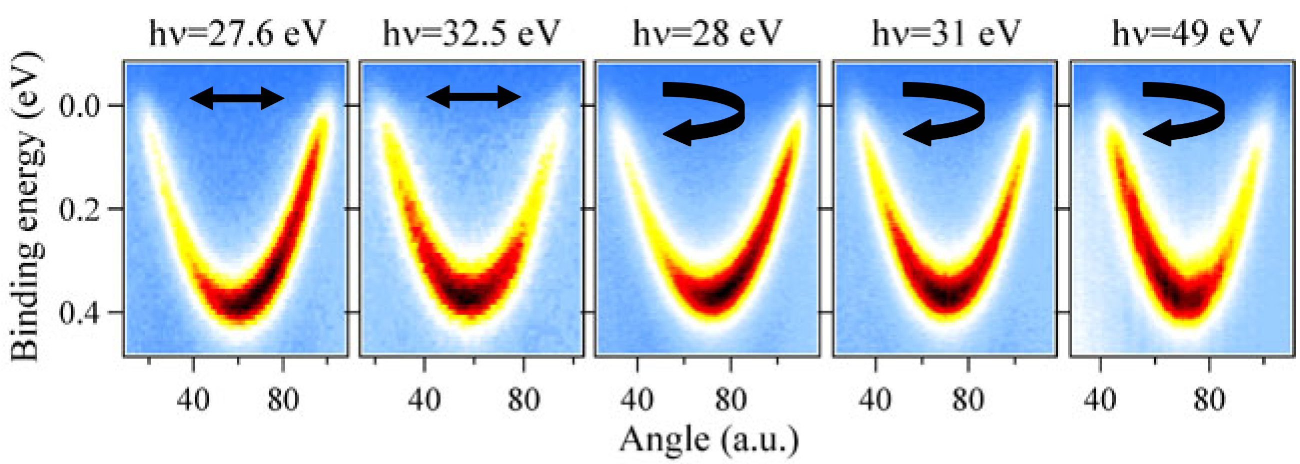
Cu(111) surface state

Matrix Element + Polarization dep.



in the crystal reflection plane

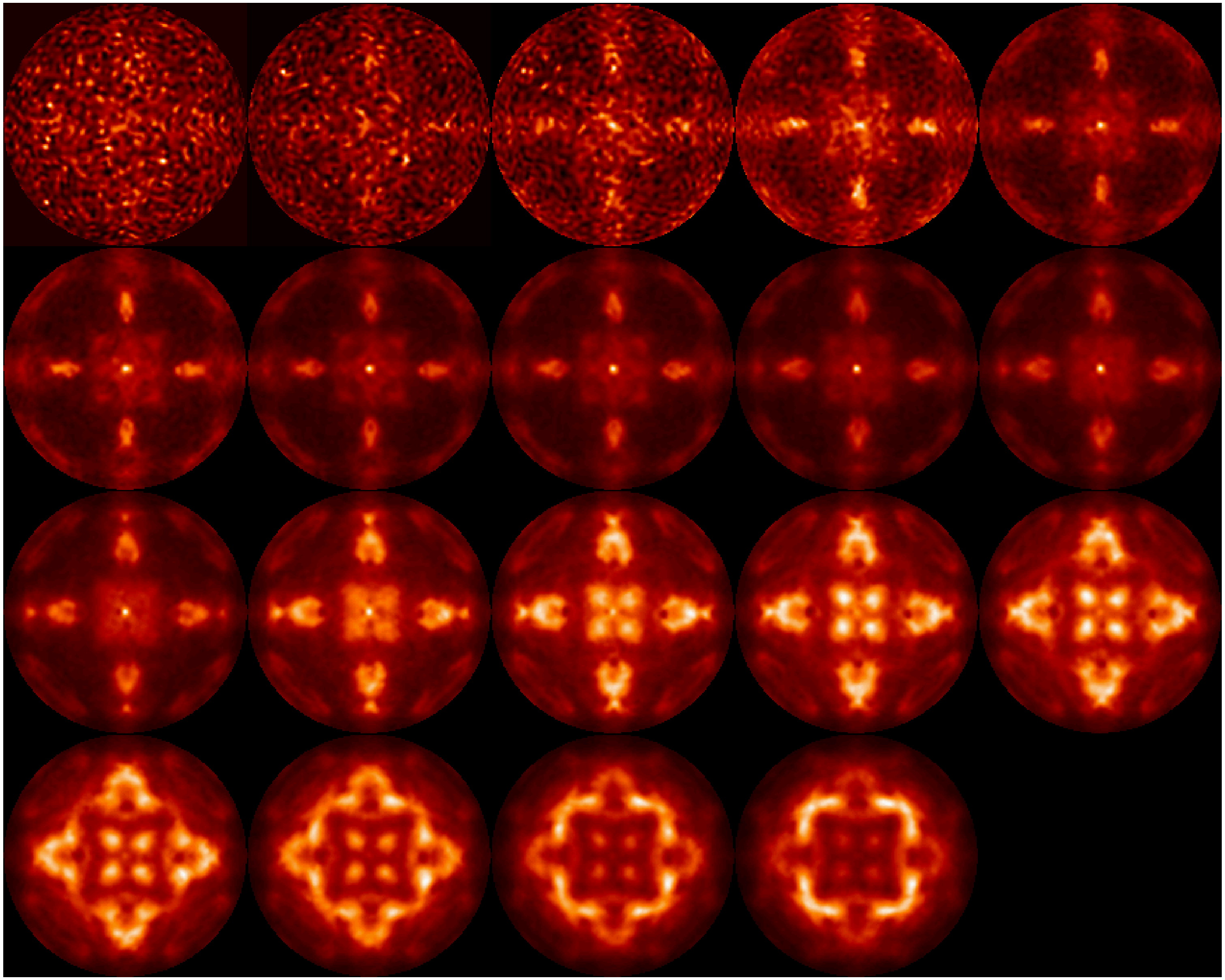
not in the crystal reflection plane



FERMI SURFACE MAPPING
Fe(001) single crystal

40 eV

step
25 meV



2D (and < 2D…) Surface science and Magnetism
What kind of control is mandatory?

Growth

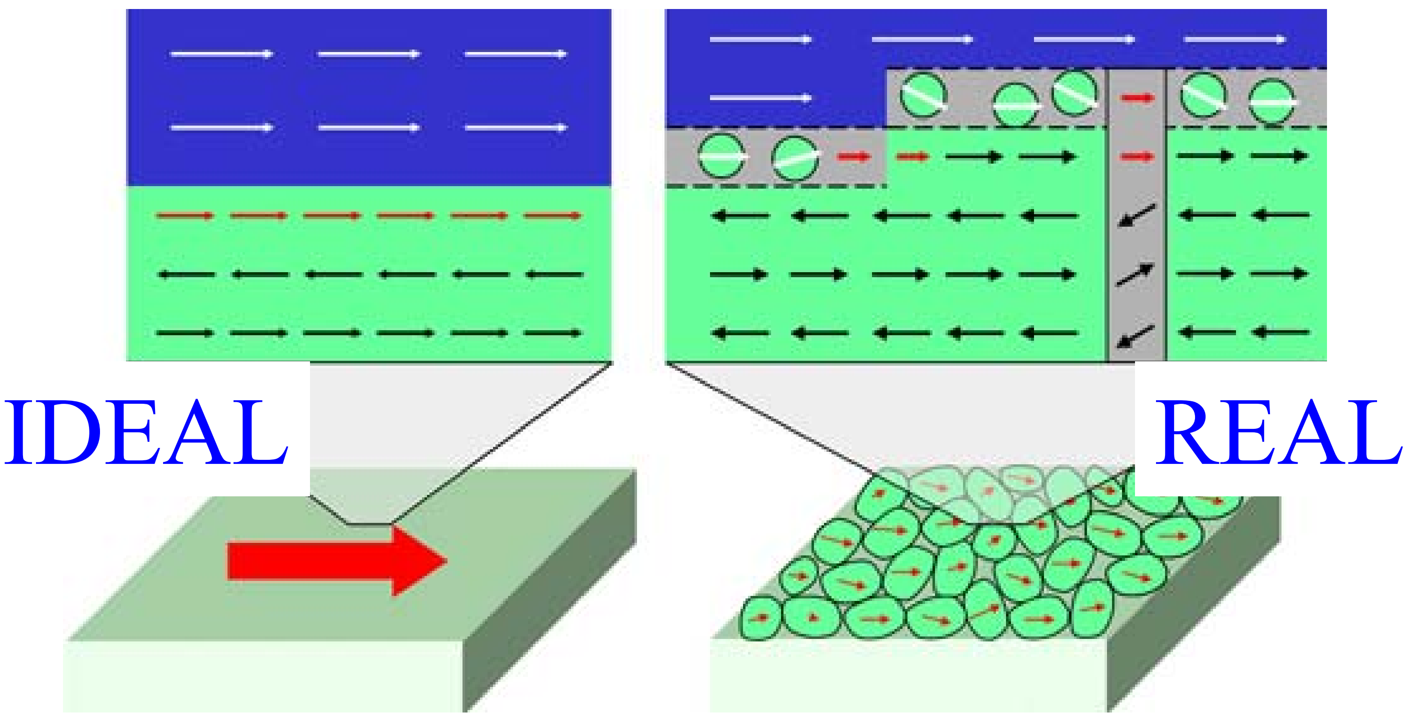
Chemical Sensitivity

Sample Prep. + **Measure**

Probe s… ng … arization)

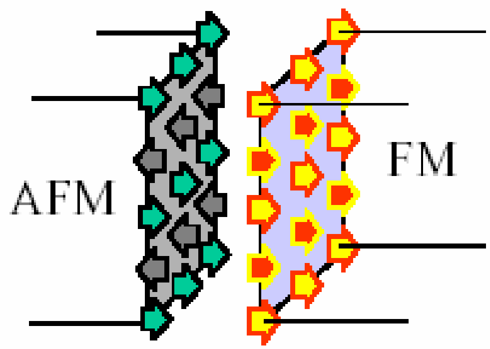
Magnetic coupling
Relaxation/Expansion/Contraction

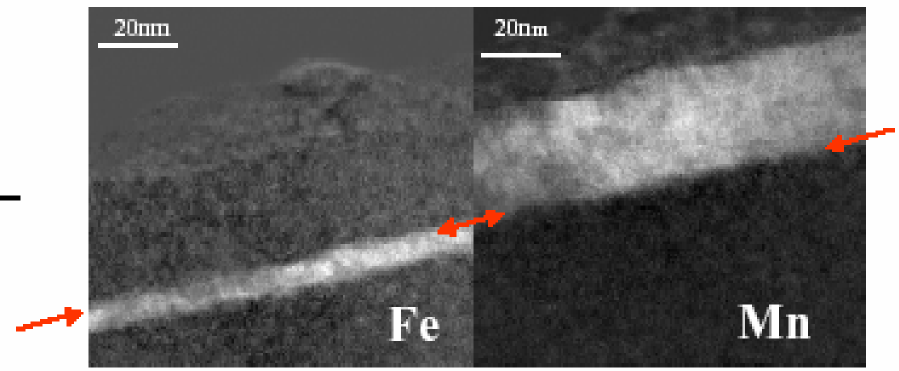
M = M (H, T)
Spin + Time + Lateral Res.

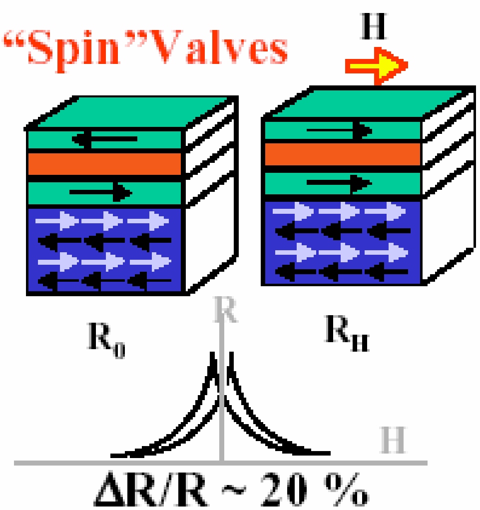


Part 3 and Part 4. **Interface and growth control.**
Model systems: Epitaxial AFM/FM interface

Frustration , Interface and Proximity Effects

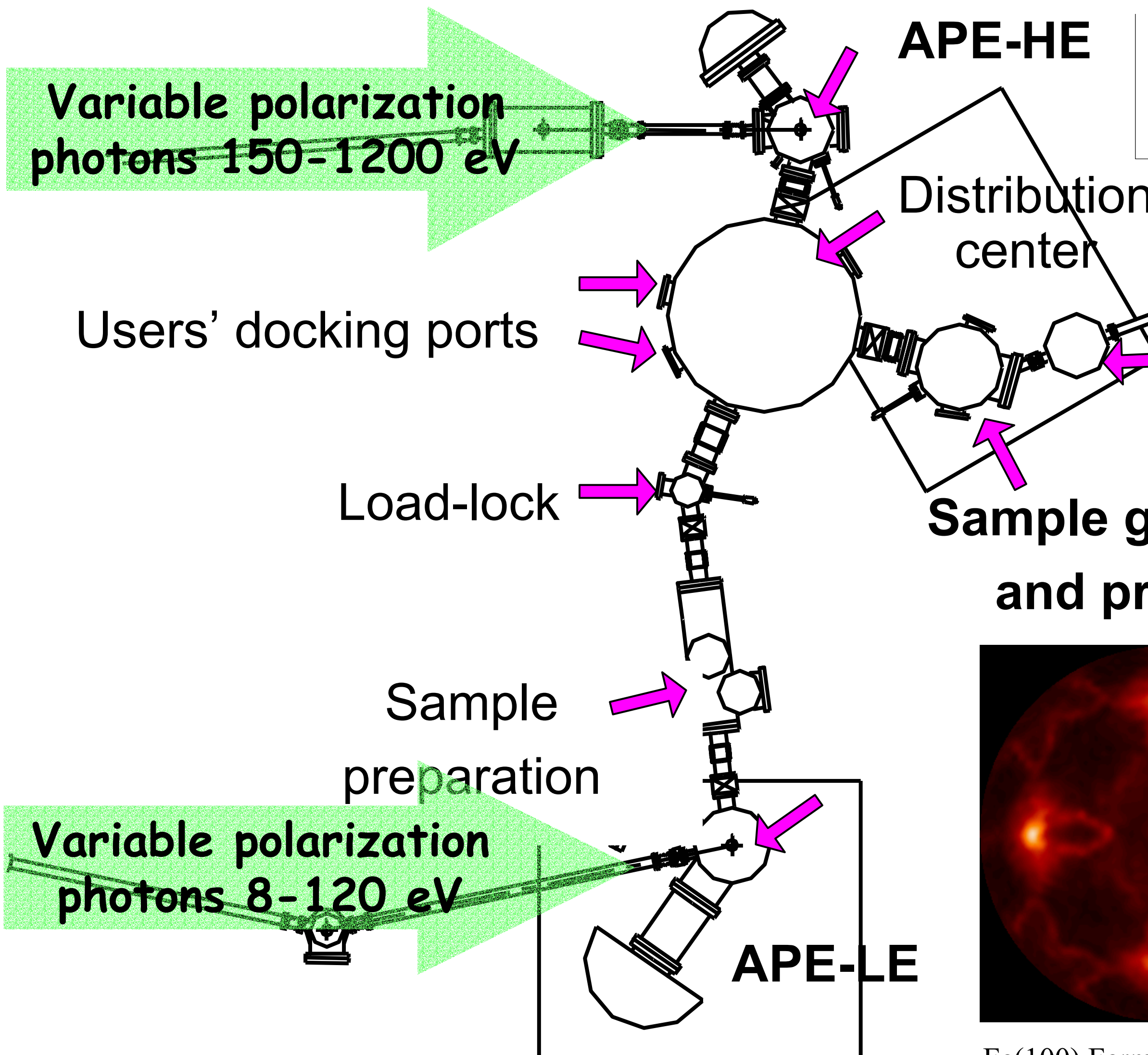


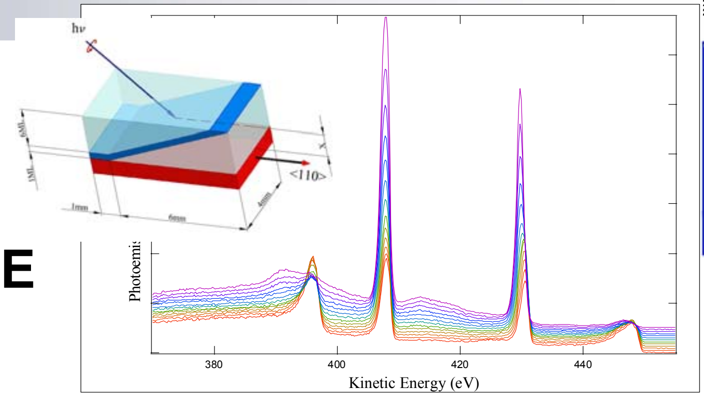




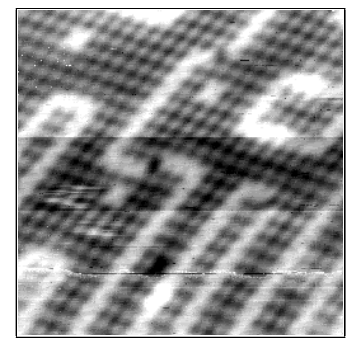
- Phenomenon poorly understood
- Interface effects dominate
- Need element-specific probes
- Growth of ideal structures - epitaxy
- Technology

Electron Spectroscopy with SR = CONTROL

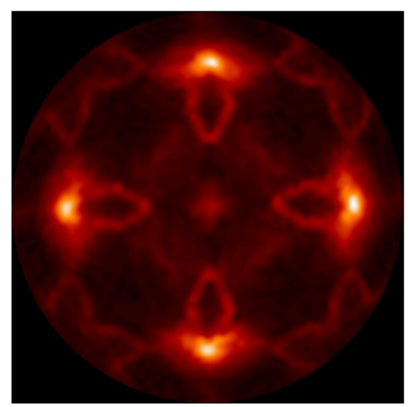




XPS vs. thickness in wedge sample



S segregation on Fe(100)



Fe(100) Fermi surface

Useful References

http://www.lightsources.org/cms/

http://www.iucr.org/cww-top/his.sync50.html

http://xdb.lbl.gov/Section2/Sec_2-2.html (x-ray data booklet)

Book and review articles

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(www.coe.berkeley.edu/AST/sxreuv)

2) H. Munro, "Synchrotron Radiation Research in the UK," *J. Synch. Rad* **4**, Part 6 (1997) 344.
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3) E.-E. Koch, D. E. Eastman, and Y. Farges, "Synchrotron Radiation—
A Powerful Tool in Science," in *Handbook on Synchrotron Radiation*,
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1983, pp. 1-63.

4) T. Sasaki, "A Prospect and Retrospect—the Japanese Case,"
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