Synchrotron light sources:

amazing present and future.
Some applications using X-rays

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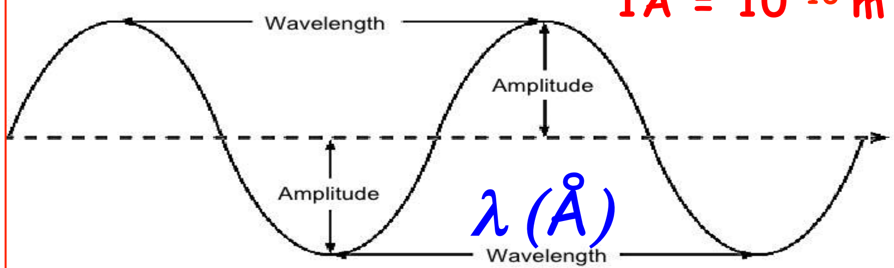
Outline

- Units
- Light, light sources, brightness and X-rays
- Synchrotron light
- Main properties
- Sources of Synchrotron Radiation
- Short history
- Present and future
- Some applications using X-rays

Reminder on some units used

- **Unit of Wavelengths** (λ): Angstrom - Å



$1 Å = 10^{-10}$ m

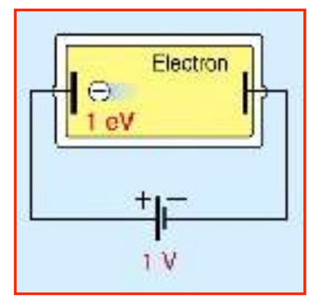
1 nm $= 10^{-9}$ m

$1 Å = 0.1$ nm

- **Unit of Energy:** electronvolt - eV

1 eV $= 1.602 \times 10^{-19}$ Joules

Energy gained (or lost) by the charge of a **single electron** moving moving across an **electron potential difference of one volt**.



GeV $= 10^9$ eV   MeV $= 10^6$ eV

- **Unit of time:** seconds - s   ps $= 10^{-12}$ s fs $= 10^{-15}$ s

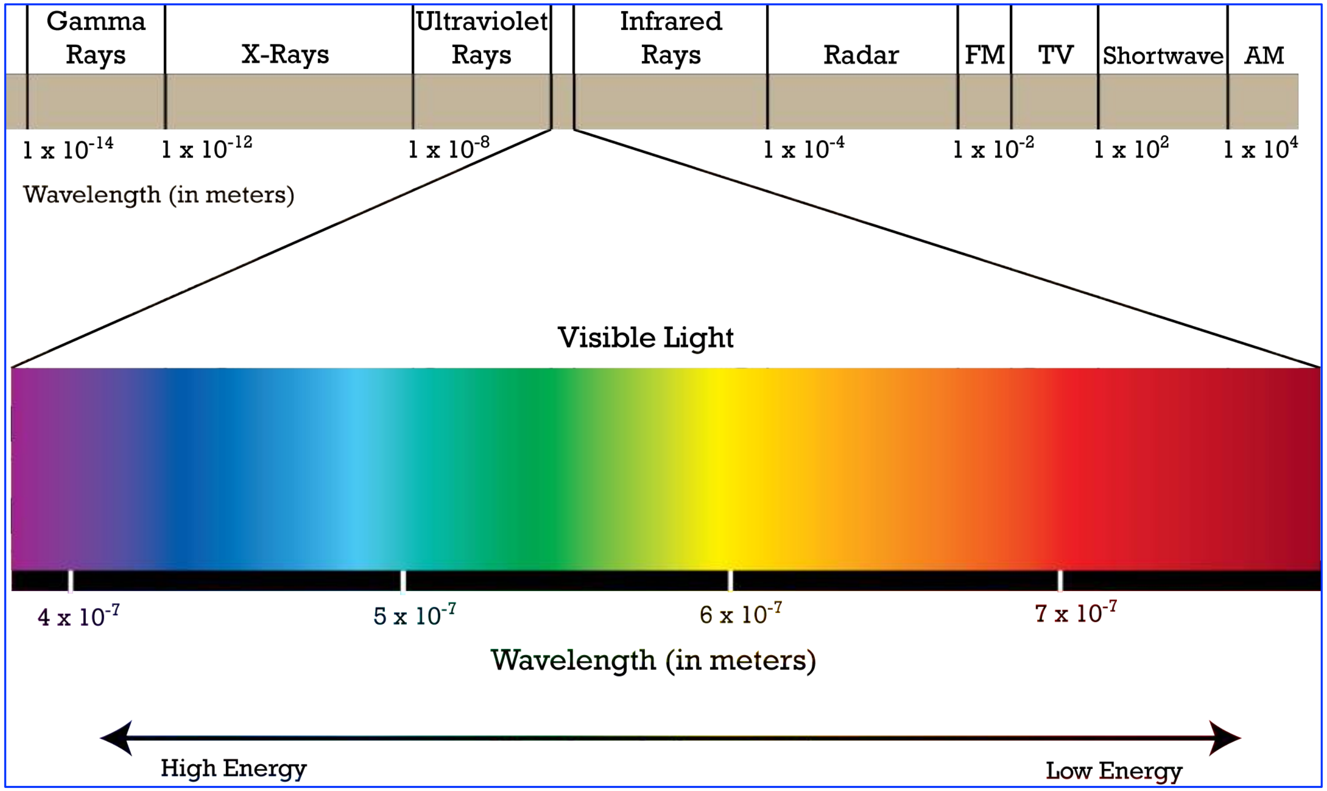
Light

Full featured double rainbow in Wrangell-St. Elias National Park, Alaska.

Visible Light

Visible light is only a ***tiny slice of the electromagnetic spectrum***. The entire electromagnetic spectrum of light is huge, spanning from ***gamma rays on one end to radio waves***.

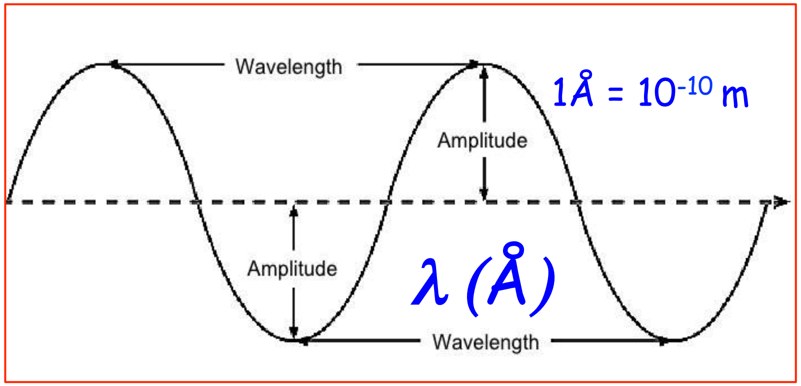
Visible Light is the light we can see using our eyes. This tiny human spectrum encompasses a very specific range of wavelengths from about:
380 nm to 780 nm.

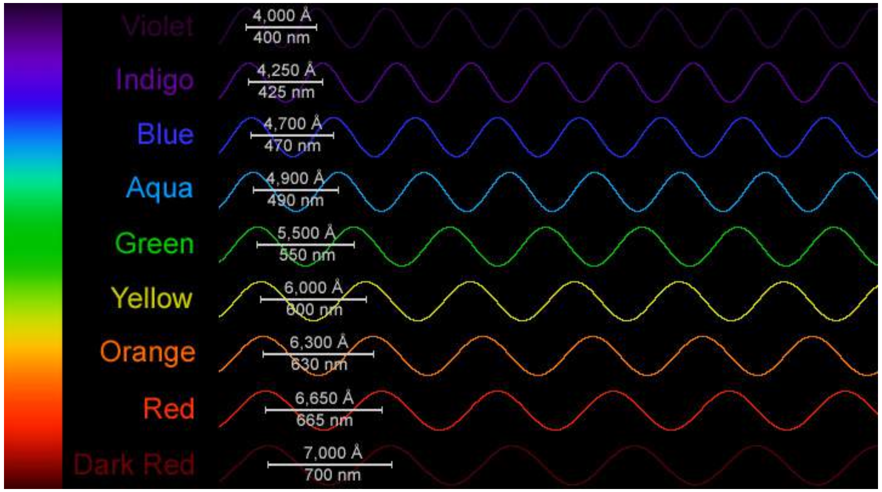


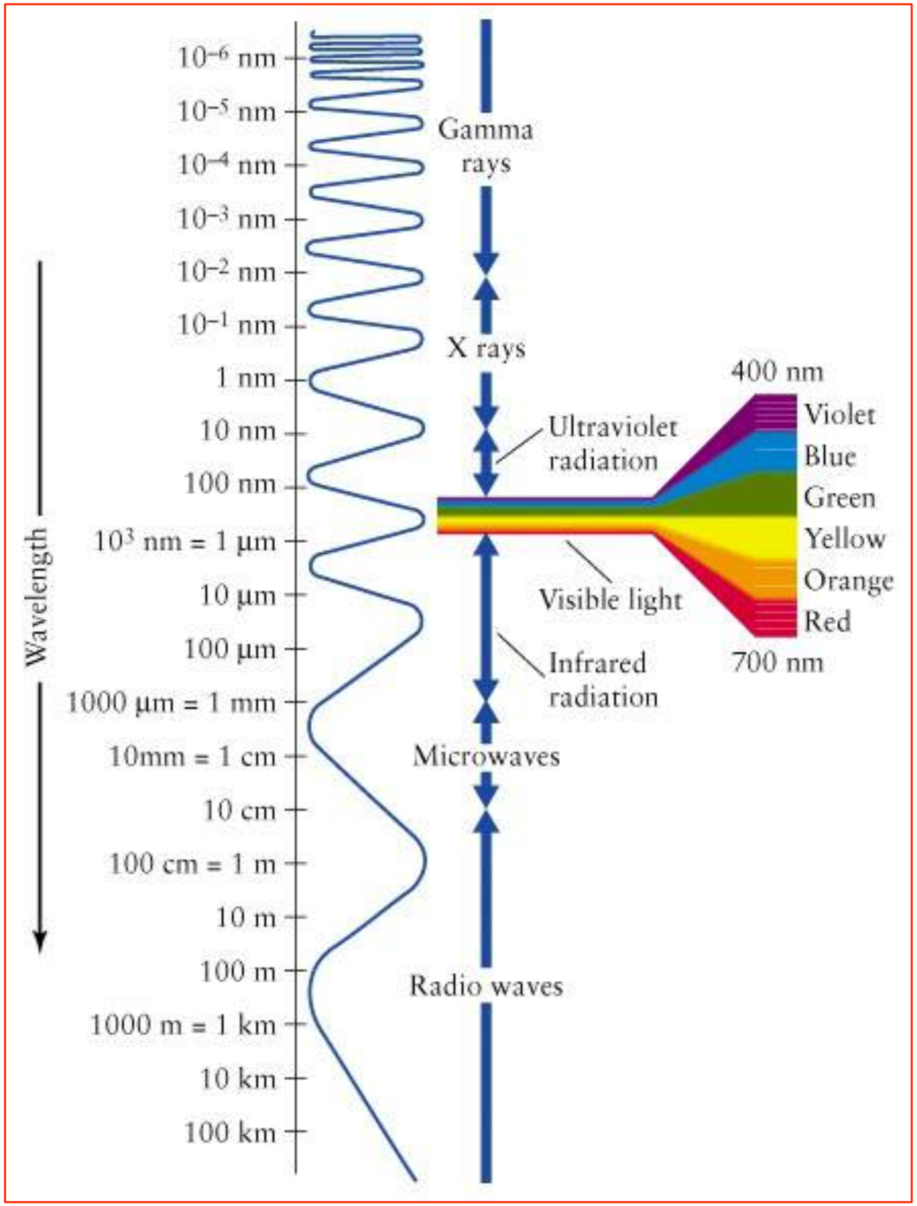
Physiologically we see these frequencies because the photoreceptors in our retinas are sensitive to them. When photons of light hit the photoreceptors it creates an electrochemical signal which is the first step in a fascinating process which ultimately results in us seeing colors.

Light and wavelengths

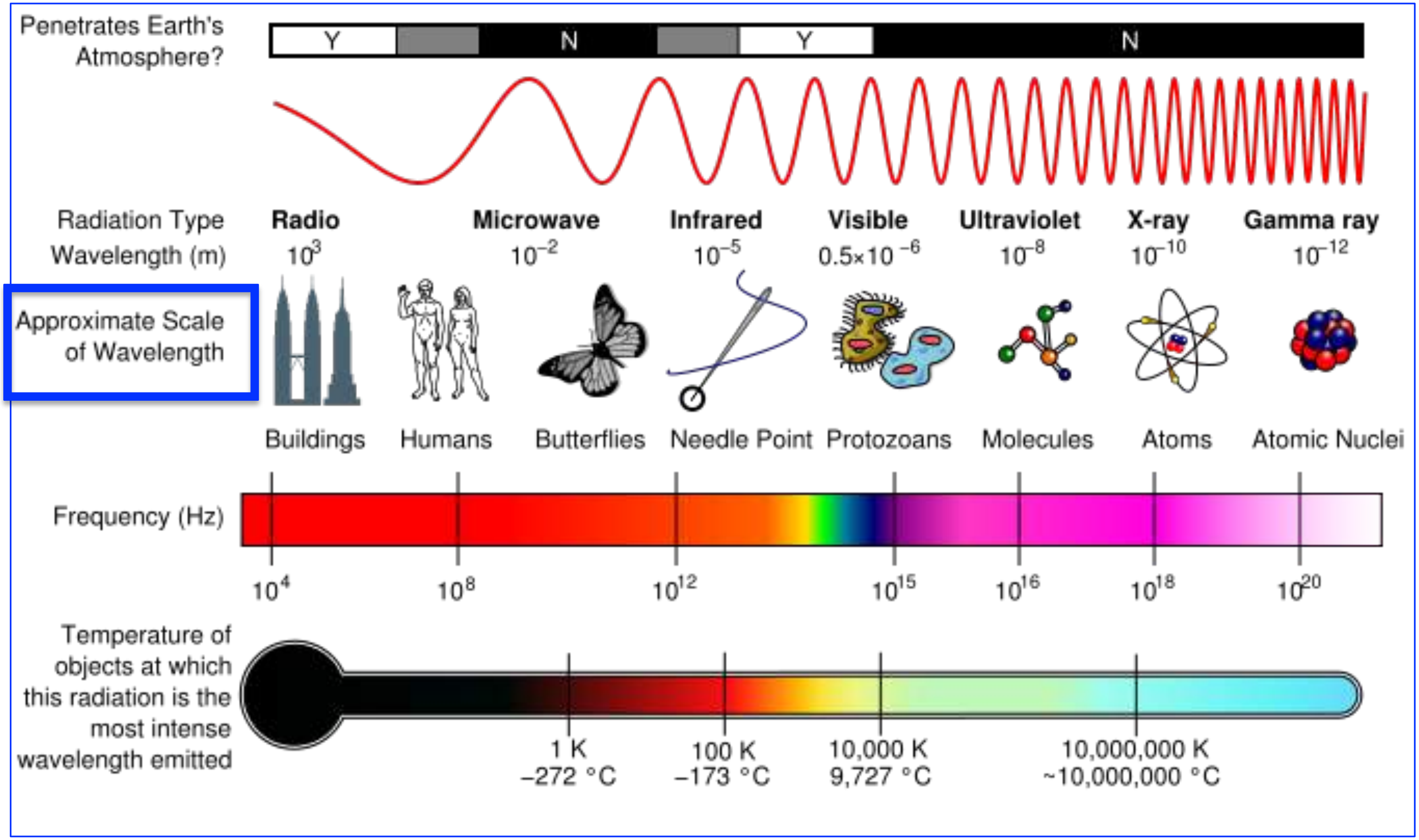
Light travels as wave of energy. Waves of light have different ***wavelengths*** (the distance between the top of one wave and the top of the next). ***Different colors of visible light have different wavelengths.***







Electromagnetic Spectrum



The ***wavelength*** (λ) and ***frequency*** (ν) ***of light are strictly related***: the higher the frequency the shorter the wavelength! This is because ***all light waves move through vacuum at the same speed*** (*c* = speed of light) and the equation that relates wavelength and frequency for electromagnetic waves is: $\lambda\nu = c$ $E = h\nu$

Light sources

Fire *is not a very useful light source to see small details because its emitted power is* ***spread in all directions****!*

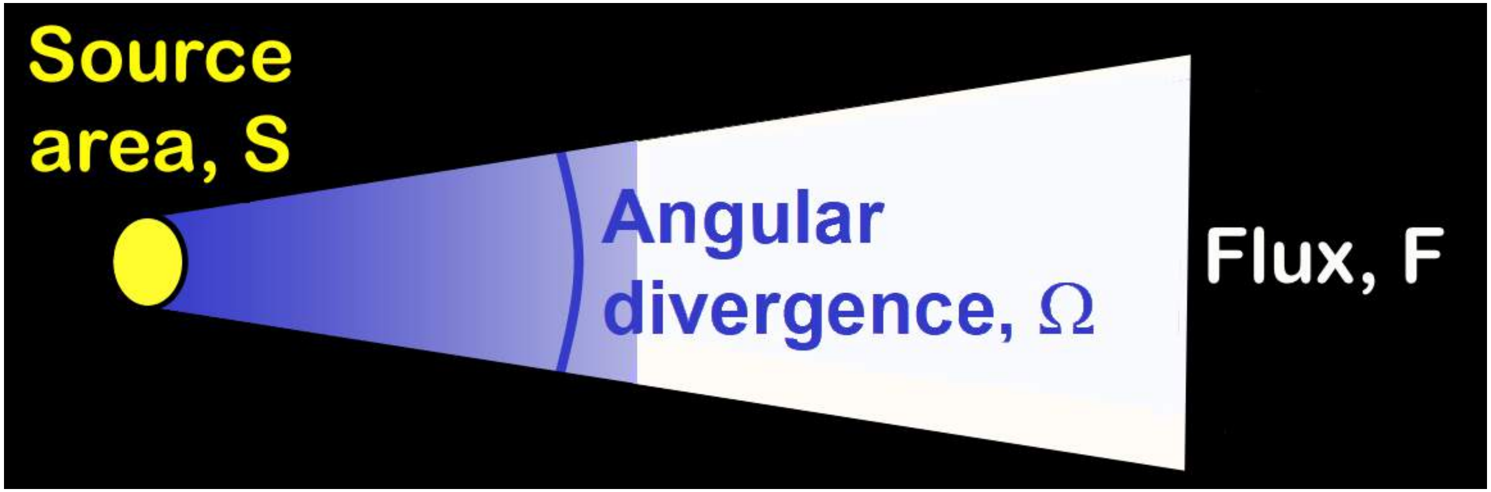
A torchlight is more adequate because due to its small size the emission is ***concentrated within a narrow angular spread: this a "bright" source****!*

Synchrotron radiation is a very bright light source *that, as will be shown, gives us the chance to* ***study also things that we cannot "see " with our eyes using not visible light but X-rays****!*

Light sources and brightness

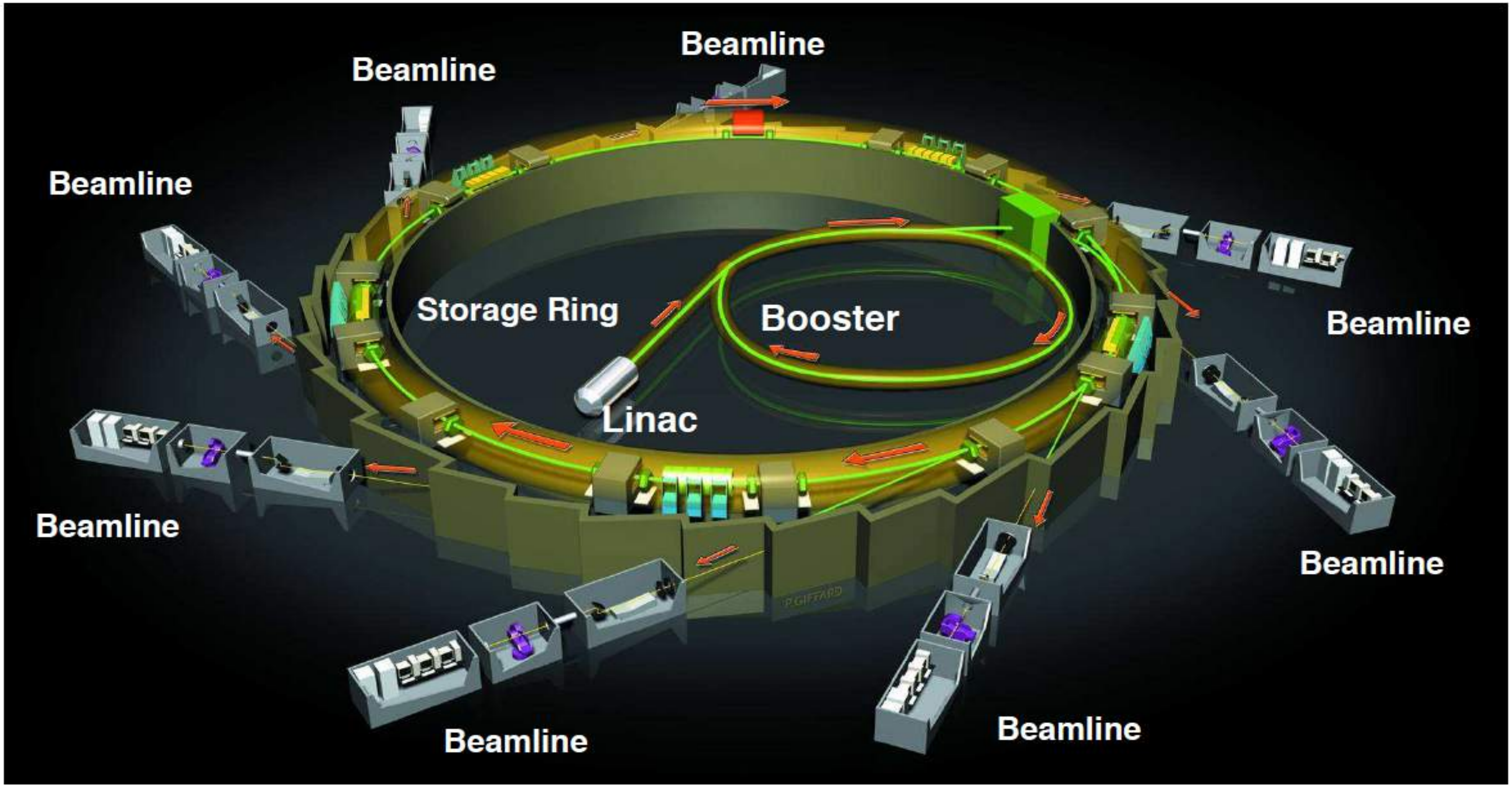
When interested in nm scale details BRIGHTNESS becomes fundamental.

A ***bright source*** is the one ***very effective in illuminating a specific target***. If the specific target is small a ***bright source*** has a ***small size with light emission concentrated within a narrow angular spread.***

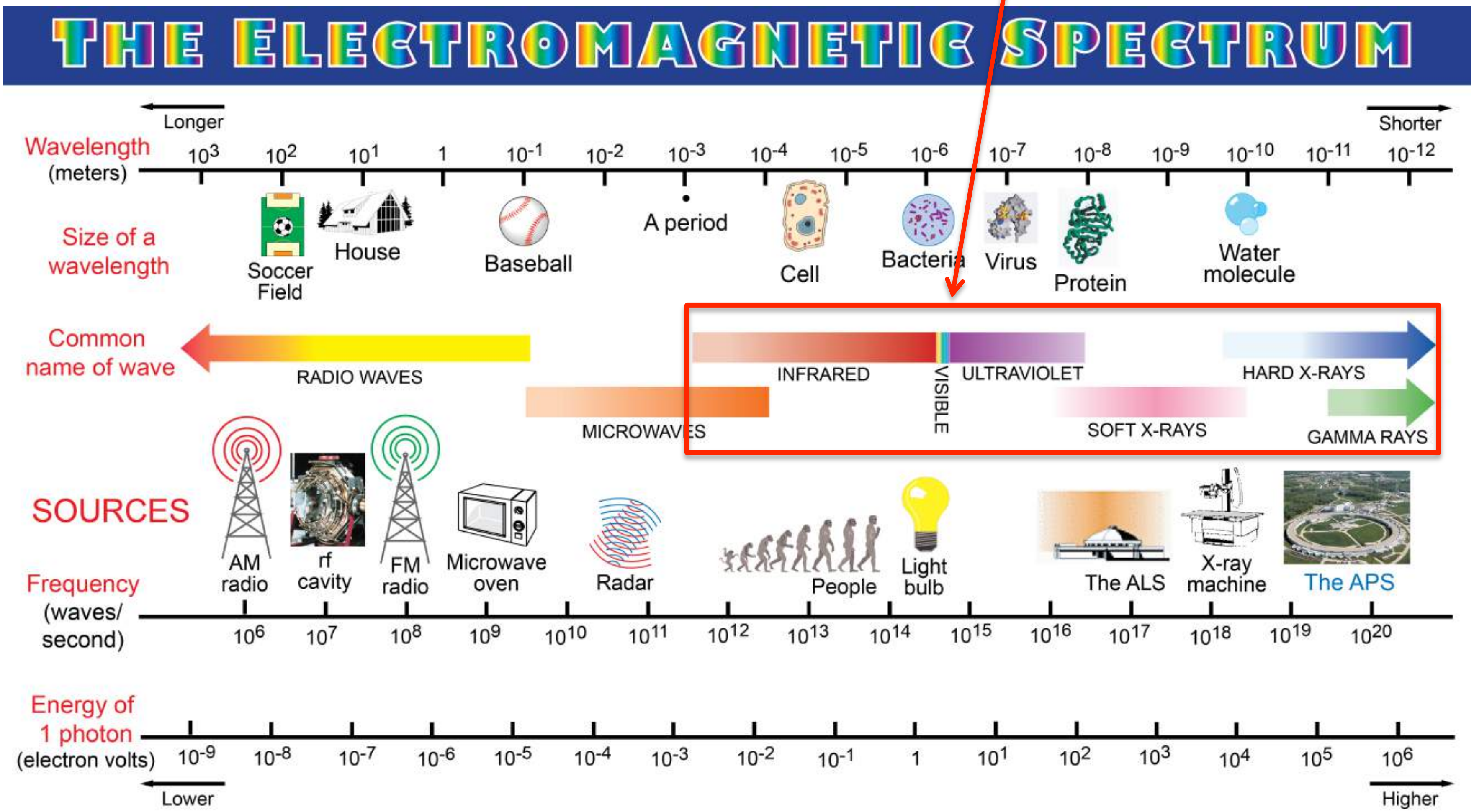


$$Brightness = constant \, \frac{F}{S \times \Omega}$$

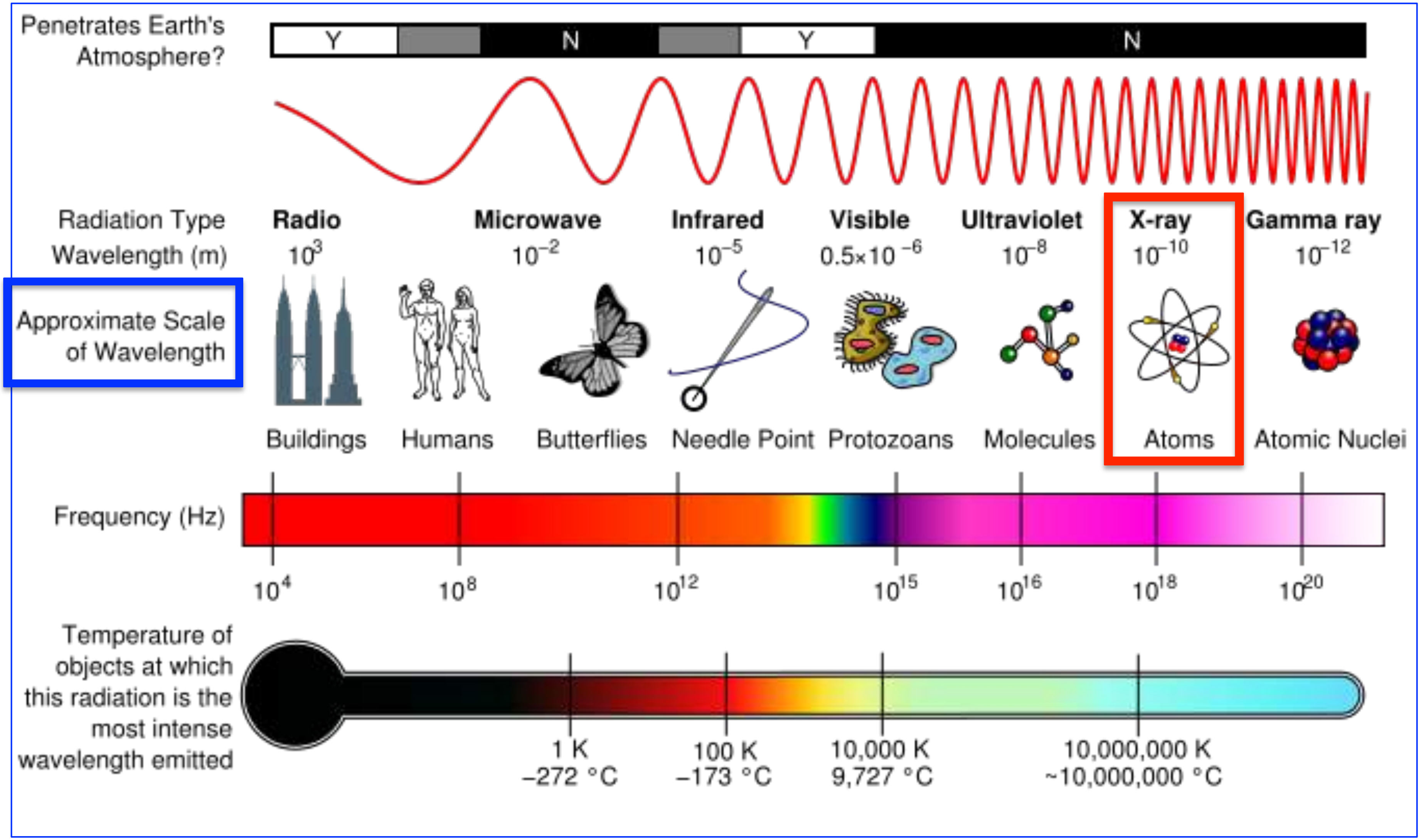
Accelerators are bright sources of synchrotron radiation



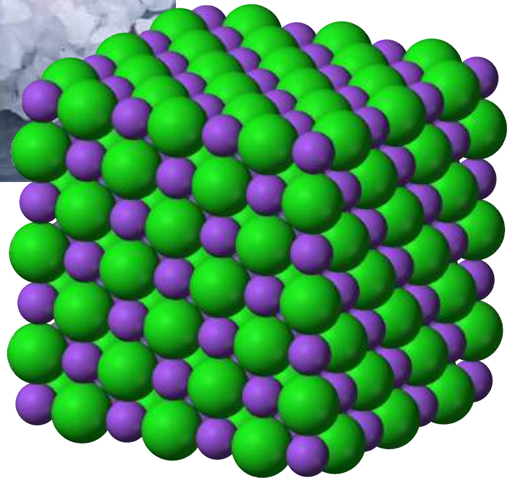
Spectral range covered by Synchrotron Radiation



Electromagnetic Spectrum and X-rays

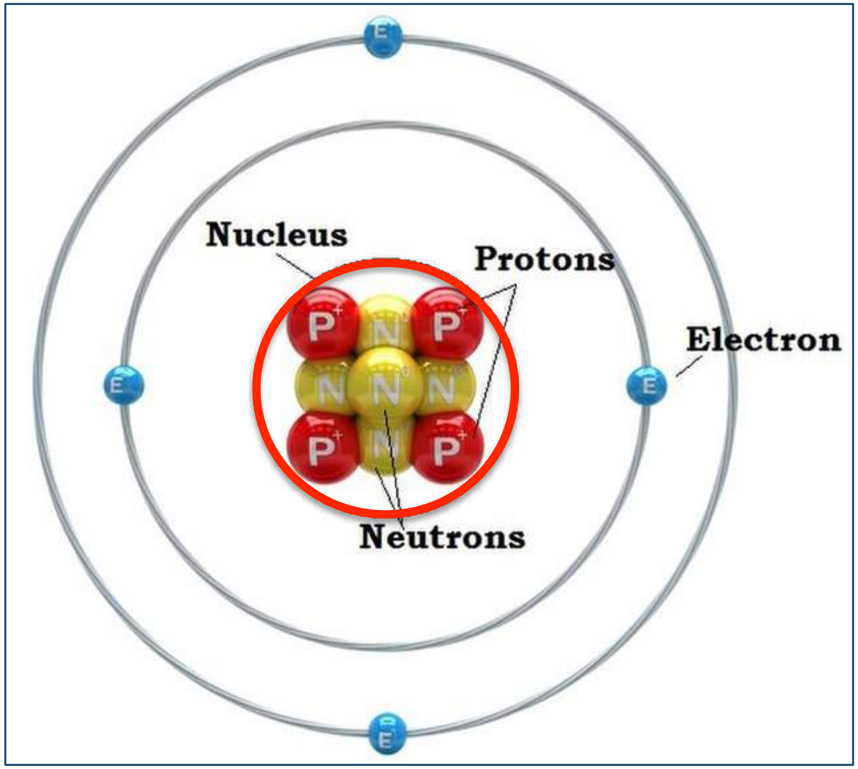


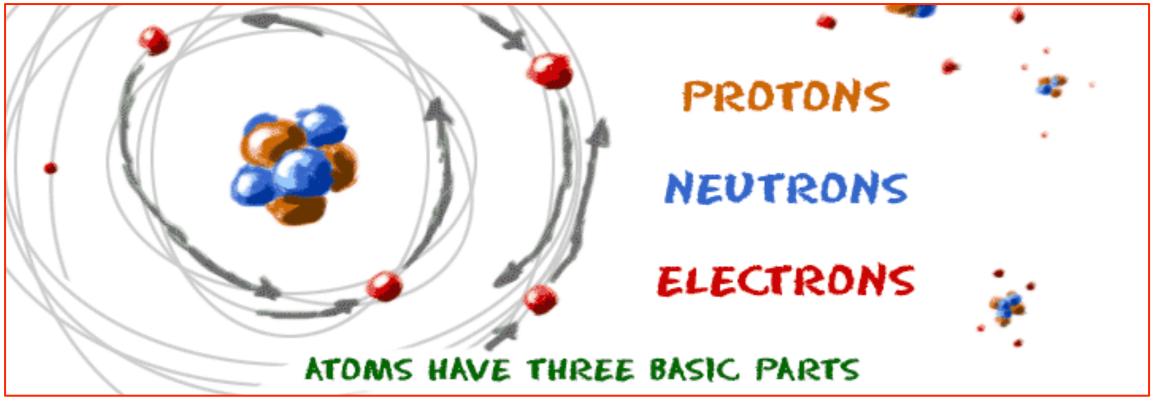
Atoms

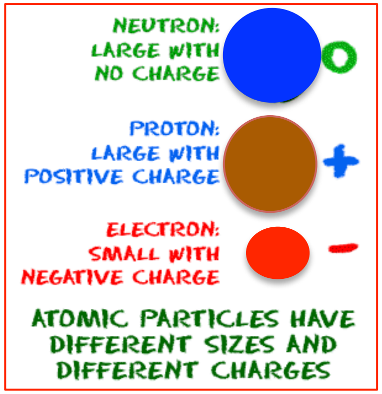


Crystal atomic structure of sodium chloride

NaCl - Na + Cl atoms

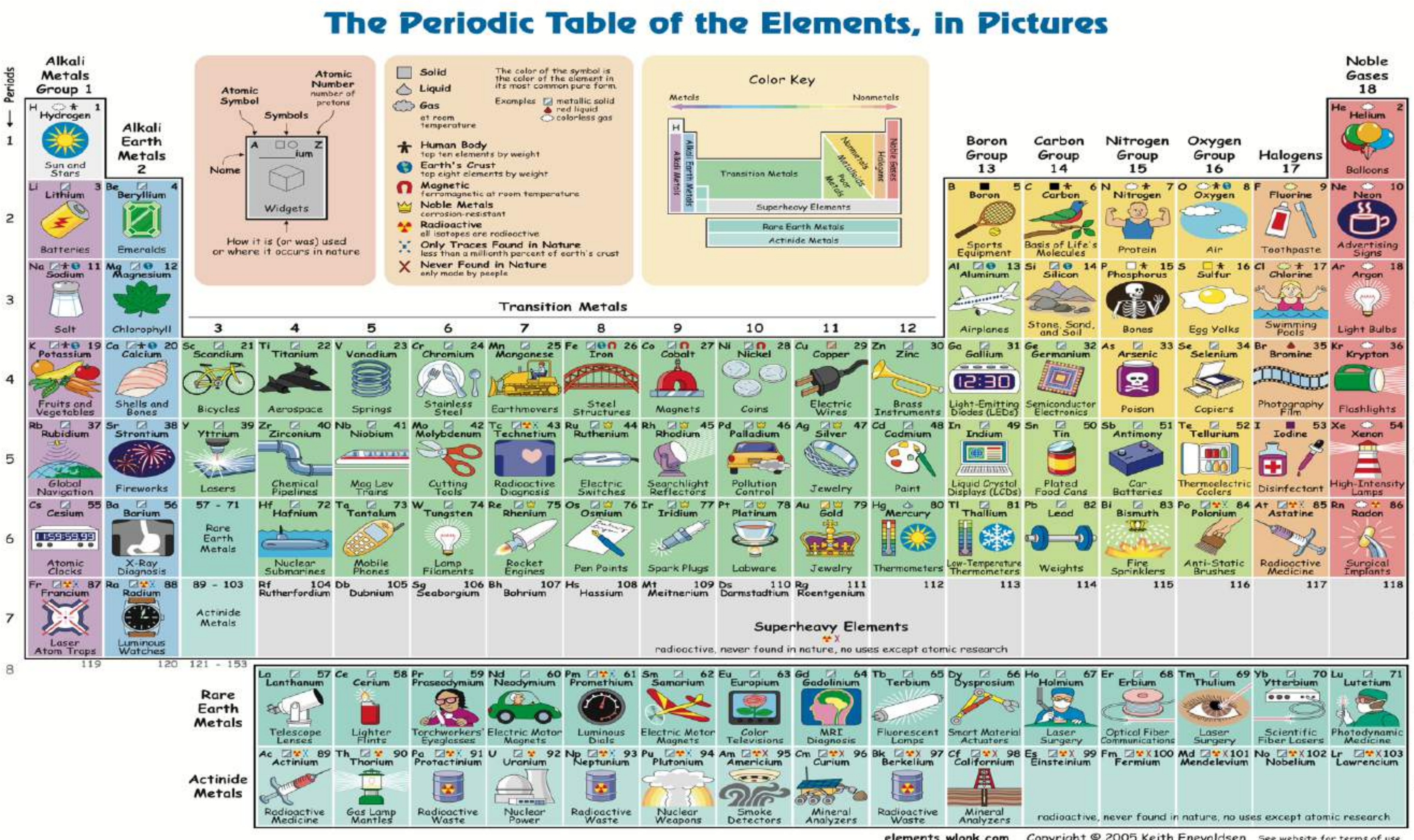






Atoms

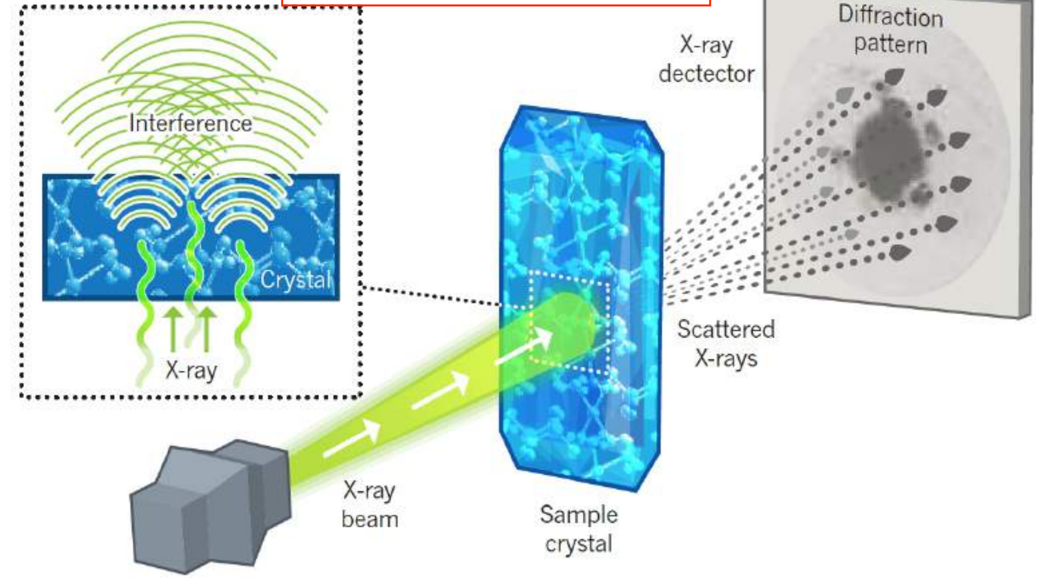
Matter is everything around us! All matter such as solids, liquids, and gases, is composed of atoms. Therefore, atoms are considered to be the basic building block of matter. From the periodic table, it can be seen that there are only about 100 different kinds of atoms. These same 100 atoms form thousands of different substances ranging from the air we breathe to the metal used to support tall buildings.



Selected by Z = atomic number = number of protons in the nucleus

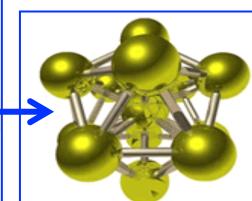
X-rays and atoms

N. Jones, Nature 505, 602 (2014)



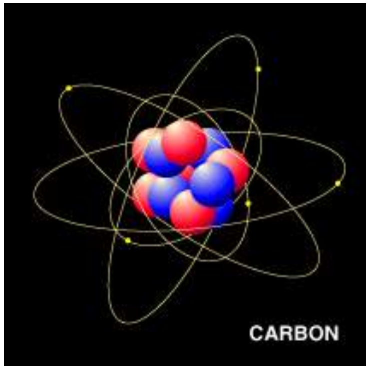
With X-rays we can study atoms because their wavelengths are of the order of 10^{-10} m or 0.1 nm or 1Å
Matter is composed of ATOMS!

Using X-rays we can study the atomic structure of materials. The atomic structure primarily affects the chemical, physical, thermal, electrical, magnetic, and optical properties.



Why is this important?

Atoms and X-rays

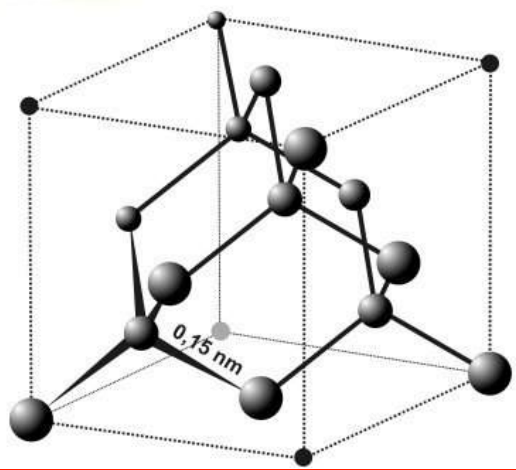


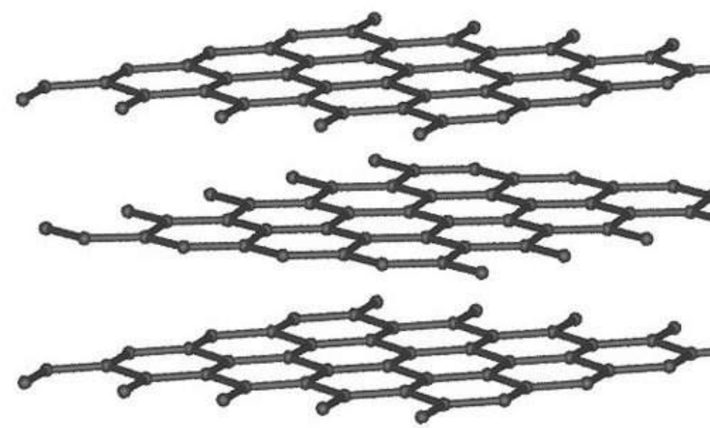
C atomic number Z = 6

Both diamond and graphite are made entirely out of carbon atoms!

The differing properties of graphite and diamond arise from their distinct crystal structures.

Graphite is opaque and metallic- to earthy looking, while diamonds are transparent and brilliant.

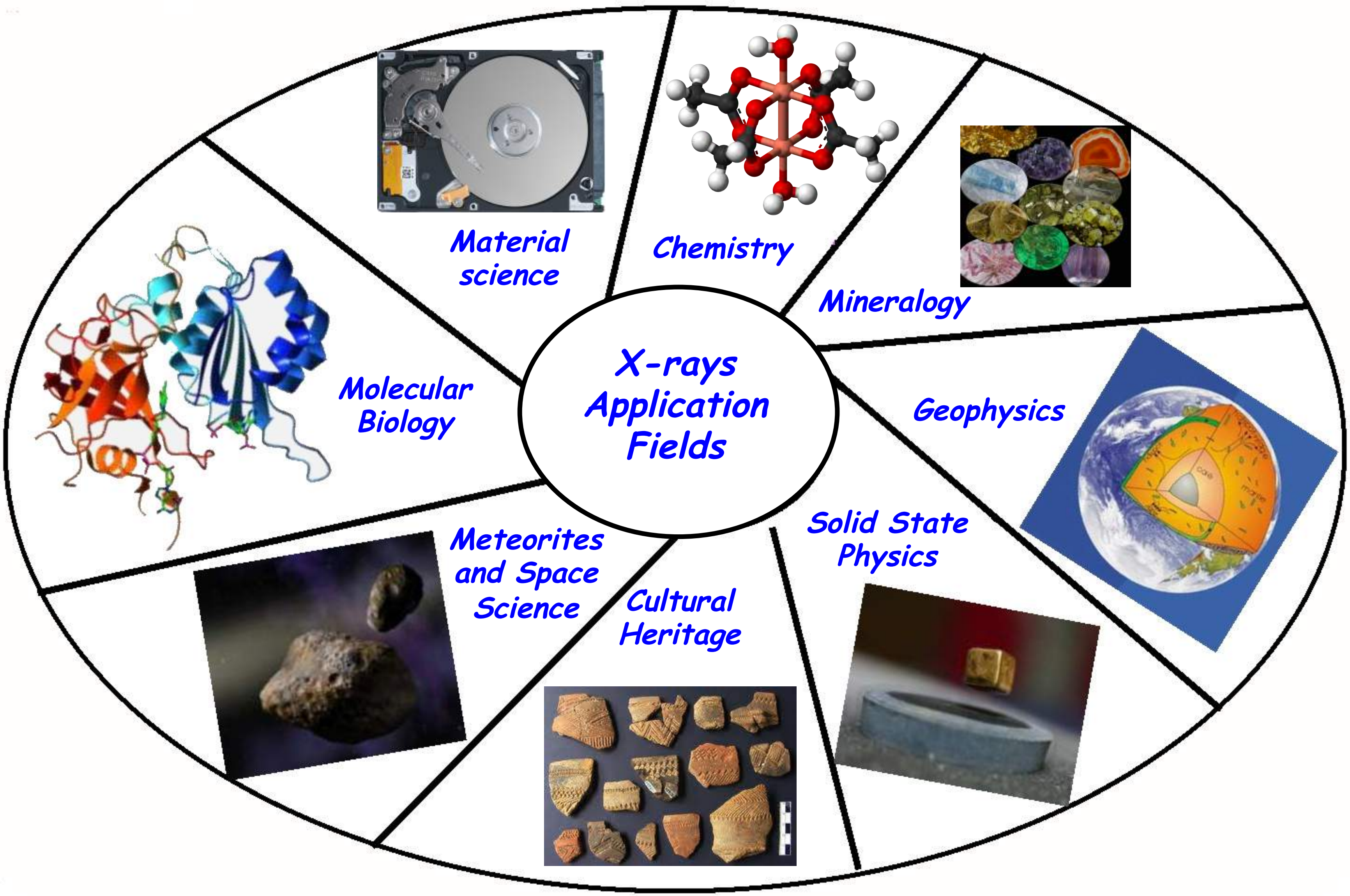




In ***graphite,*** the ***individual carbon atoms link up to form sheets of carbon atoms***. Within each sheet every carbon atom is bonded to three adjacent carbon atoms (***covalent bonds***) producing hexagonal rings of carbon atoms. ***Weak bonding forces called van der Waals forces hold the sheets together***. Because these ***forces are weak, the sheets can easily slide past each other***. The sliding of these ***sheets gives graphite its softness for writing and its lubricating properties***.

In diamonds, ***each carbon atom is strongly bonded to four adjacent carbon atoms located at the apices of a tetrahedron*** (a three-sided pyramid). The four valence electrons of each carbon atom participate in the formation of ***very strong covalent bonds***. These bonds have the same strength in all directions. ***This gives diamonds their great hardness.***

X-rays application fields



Bright X-ray source?

HE Particle accelerators

↓

Synchrotron light

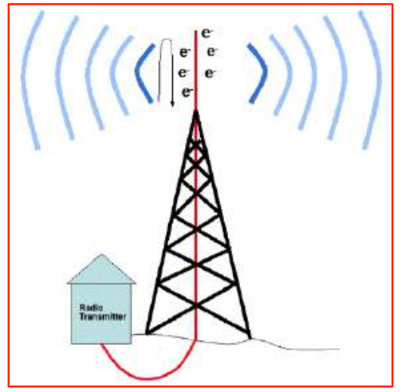
Answers to be given

- What is synchrotron light?
- How is it produced?
- History?
- Properties?
- Sources?
- How and why is it used?
- Applications?

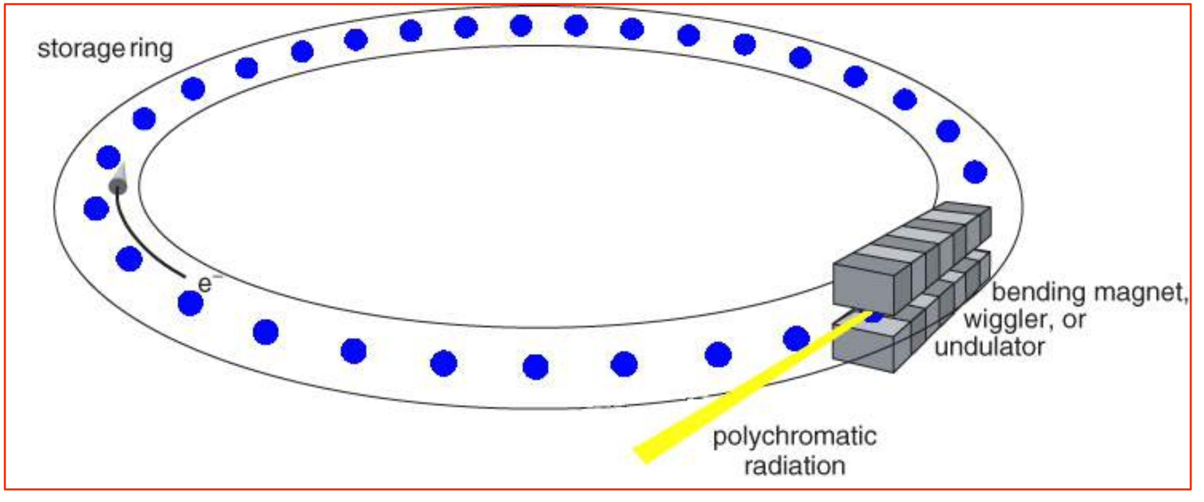
Synchrotron radiation

Accelerated charged particle, like e^+, e^- and ions, emit electromagnetic radiation.

v << c or β = v/c << 1

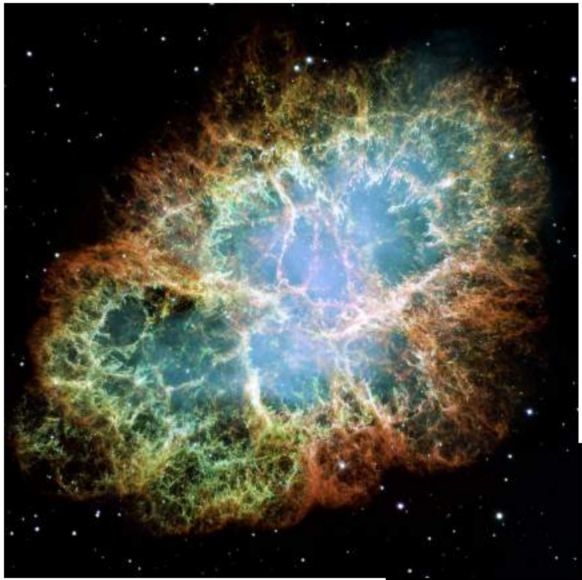


When charged particles, ***moving at relativistic speeds*** (v ≈ c), *are forced to* ***change the direction of their motion (acceleration)****, under the effect of magnetic fields,* ***in circular particle accelerators, like synchrotrons****, the radiation produced is called* ***synchrotron radiation***.



v ≈ c or β = v/c ≈ 1

Synchrotron light is present in nature

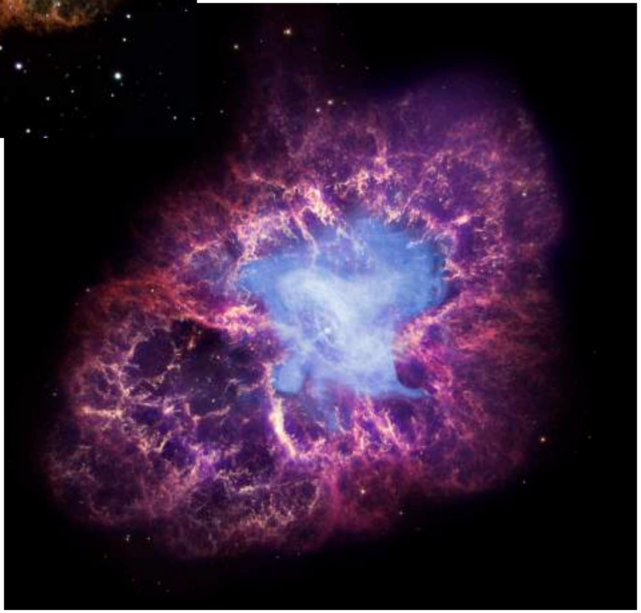


Synchrotron radiation is a very important emission process in Astrophysics.

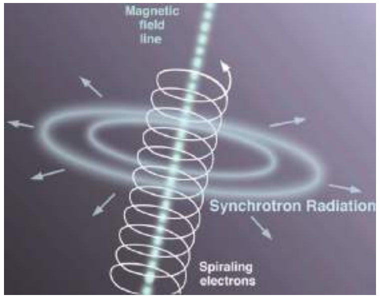
Crab Nebula: remnant of a supernova explosion seen on earth by Chinese astronomers in 1054, at about 6500 light years from Earth in the constellation Taurus !

The heart of the nebula is a rapidly-spinning neutron star, a pulsar, that powers the strongly polarised bluish 'synchrotron' nebula.

NASA Hubble Space Telescope image of the Crab Nebula (NASA, ESA and Allison Loll/Jeff Hester (Arizona State University)).



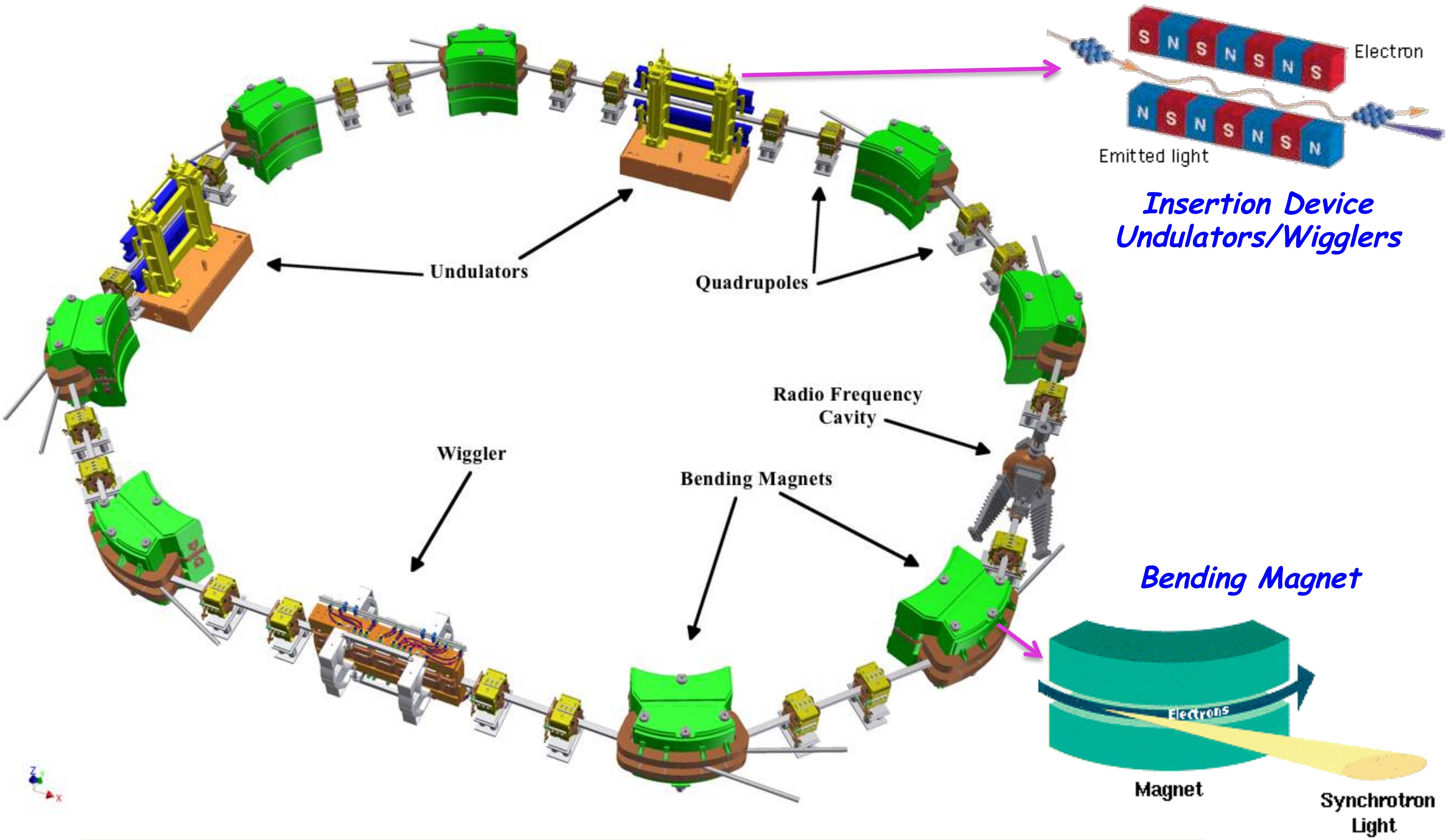
The *Crab pulsar* is *slowing at the rate of about 10^{-8} sec per day*, and the *corresponding energy loss agrees well with the energy needed to keep the nebula luminous*. Some of this luminosity takes the form of *synchrotron radiation, requiring a source of energy for accelerating charged particles*.



NASA's Great Observatories' View of the Crab Nebula X-Ray-blue: NASA/CXC/J.Hester (ASU); Optical-red and yelllow: NASA/ESA/J.Hester & A.Loll (ASU); Infrared-perple: NASA/JPL-Caltech/R.Gehrz (Univ. Minn.)

Composite image data from ***three of NASA's Great Observatories****. The* ***Chandra X-ray Observatory image*** *is shown in blue, the* ***Hubble Space Telescope optical image*** *is in red and yellow, and the* ***Spitzer Space Telescope's infrared*** *image is in purple. The X-ray image is smaller than the others because extremely energetic electrons emitting X-rays radiate away their energy more quickly than the lower-energy electrons emitting optical and infrared light.* ***The Crab Nebula is one of the most studied objects in the sky, truly making it a cosmic icon.***

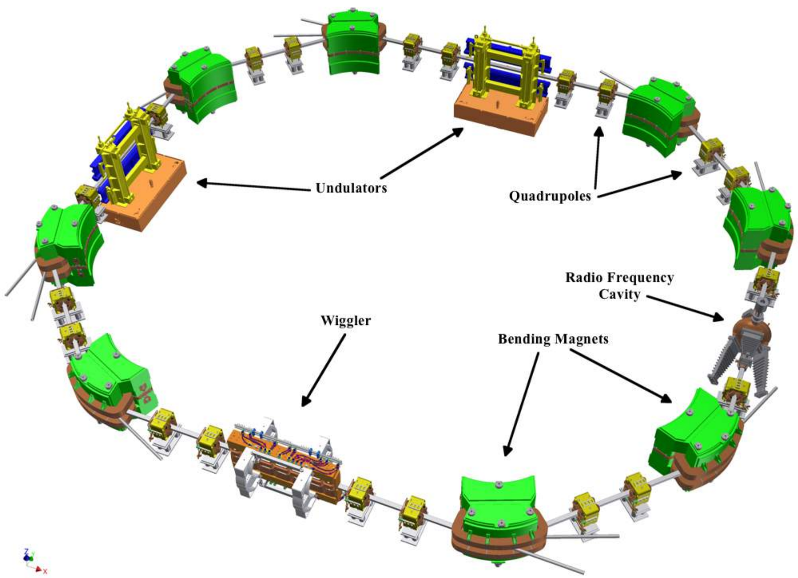
Synchrotron light artificially produced by circular particle accelerators

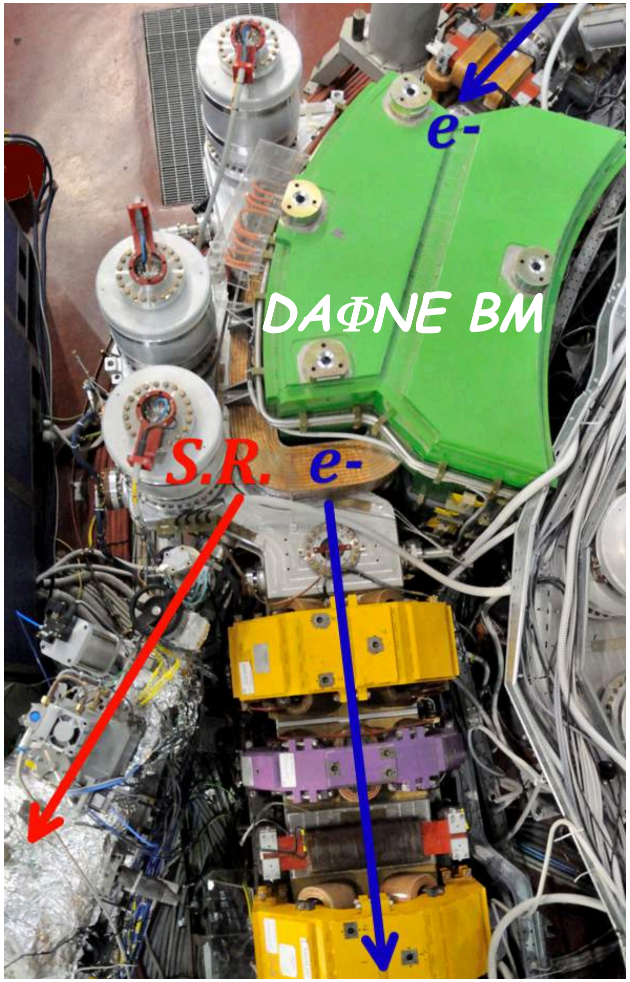


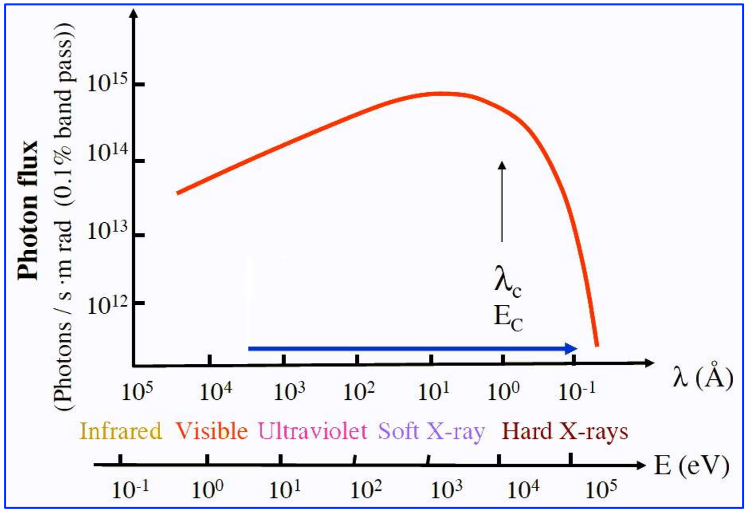
Bending magnets and insertion devices are components of the storage ring.

Bending magnets

Bending magnets are fundamental parts of the storage ring.

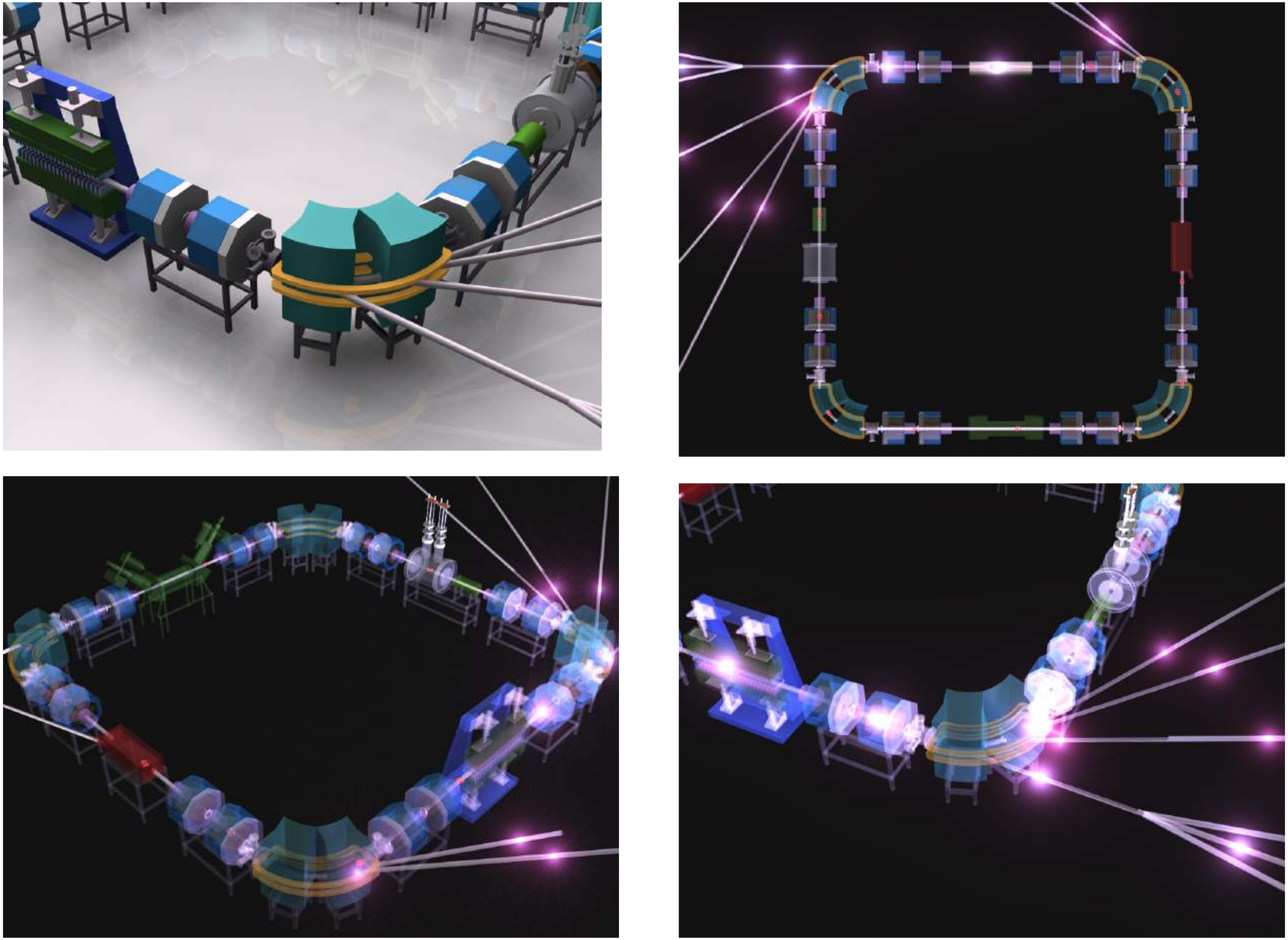






$$\varepsilon_c[keV] = 2.218\frac{E[GeV]^3}{R[m]} = 0.665 \cdot E[GeV]^2 \cdot B[T]$$

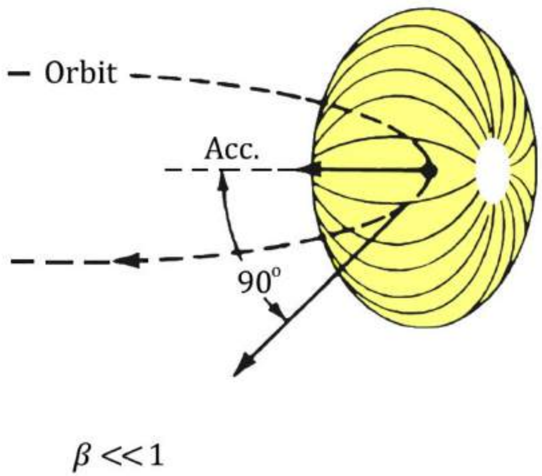
The critical energy, ε_c , divides the spectrum into two parts of equal radiated power: 50% of the total power is radiated at energies lower than ε_c and 50% at energies higher than ε_c .

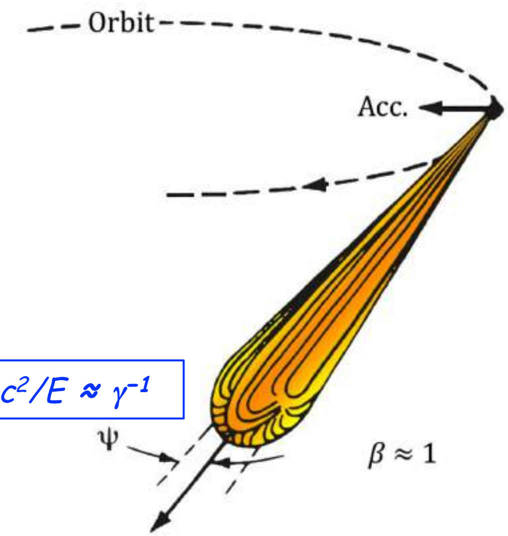


ASTRID (Aarhus - Denmark) http://www.isa.au.dk/animations/pictures/pic-index.asp

http://www.isa.au.dk/animations/Finalmovie/astrid_total_v2.mov

Synchrotron radiation: physics





$\psi \approx mc^2/E \approx \gamma^{-1}$

v << c or β = v/c << 1

$$P = 2\, e^2\, a^2/(3c^3) \quad [W]$$

P = total emitted power, *a* = acceleration

At low electron velocity (non-relativistic) the radiation is emitted in a ***non-directional pattern***.

1897 ***Lamor***: calculates power radiated by an accelerated charged particle
1898 ***Liénard***: extends the theory to relativistic particles in a circular path

v ≈ c or β = v/c ≈ 1

For a relativistic effect, when the speed of the emitting electrons increases to relativistic values (v ≈ c) the radiation pattern is compressed into a ***narrow cone in the direction of motion, resulting into an emission tangential to the particle orbit***.

$$P_{rad} = \frac{2}{3}\frac{Q^2 c}{R^2}\left[\frac{E}{mc^2}\right]^4$$

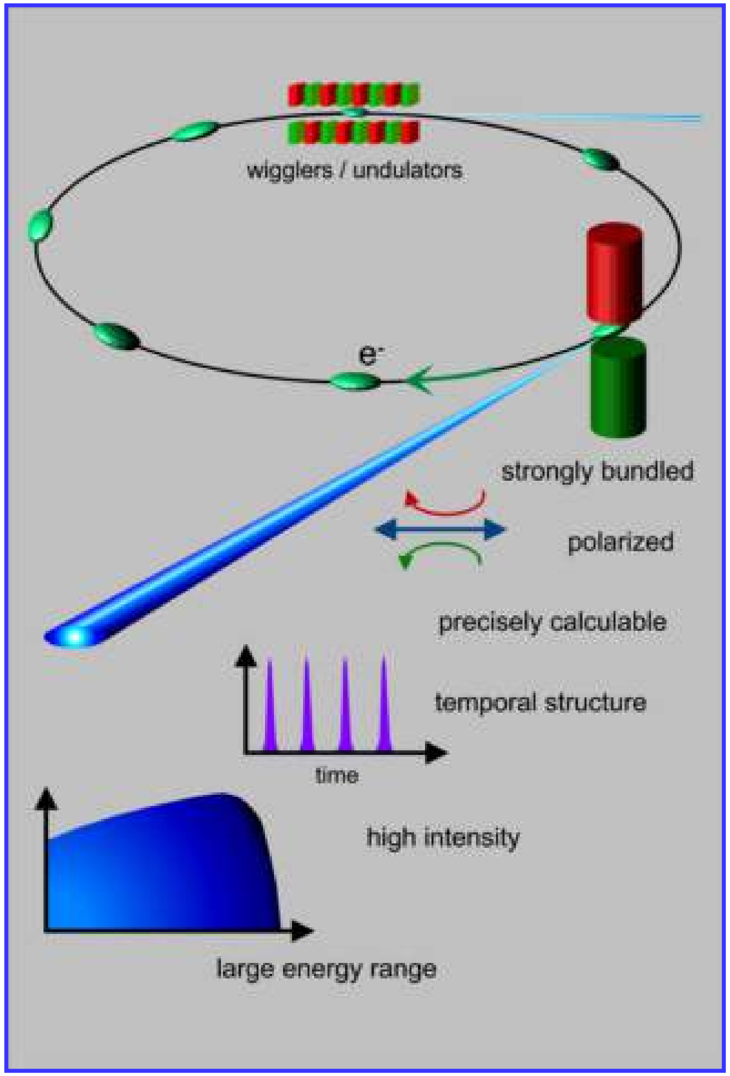
E = particle energy, ***m*** = mass, ***R*** = radius of curvature

1945 Schwinger: classical theory of radiation from accelerated relativistic electrons

Synchrotron Radiation Properties

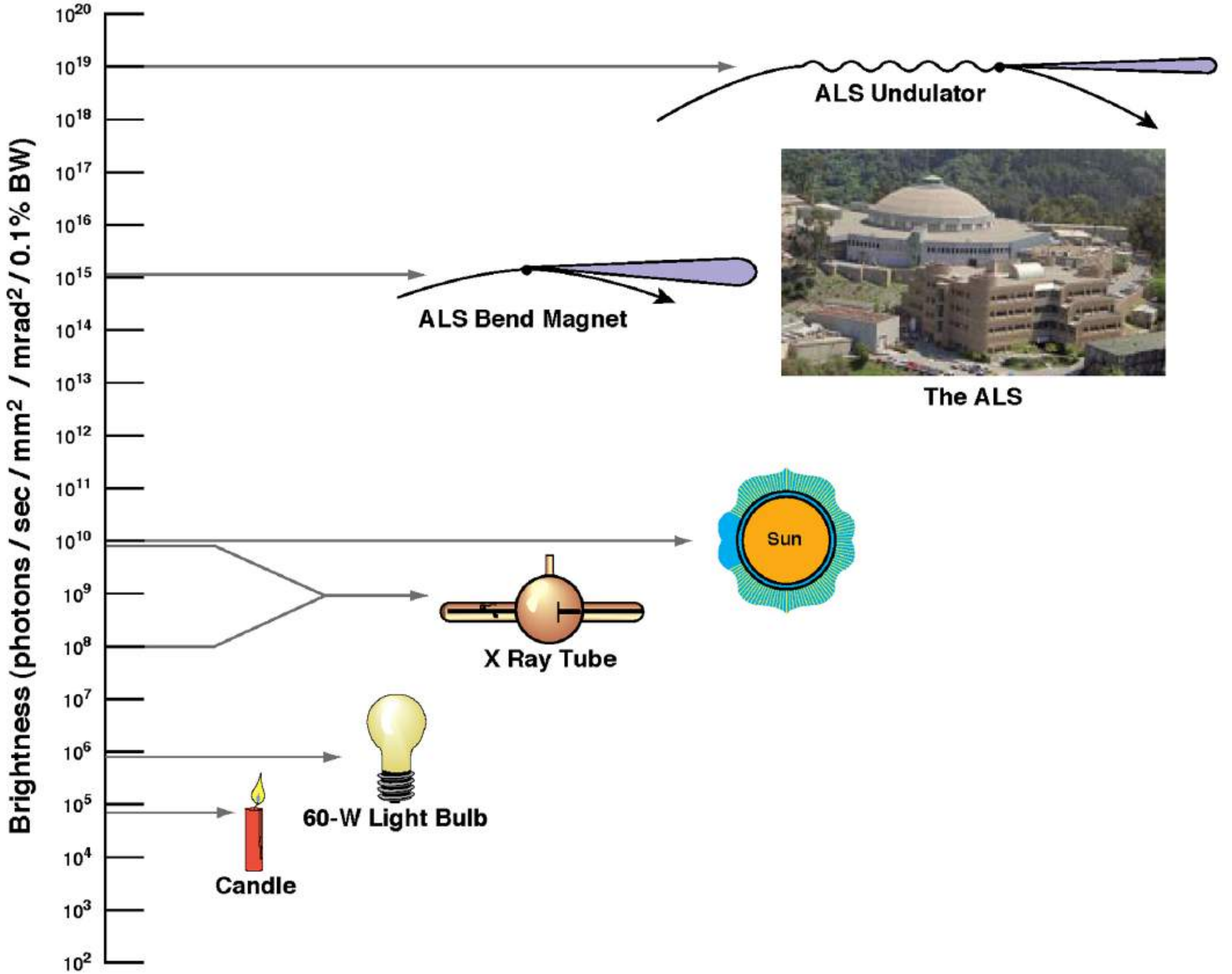
What makes synchrotron radiation interesting, powerful and unique?

- ***Very high flux and brightness (with undulators)*** highly collimated photon beam generated by a small divergence and small size source (partial coherence)
- ***Broad spectral range (tunability)*** which covers from microwaves to hard X-rays: the user can select the wavelength required for experiment- *continuous (Bending Magnet/Wiggler) - quasi-monochromatic (Undulator)*
- ***Small source size***
- ***Collimated beams***
- ***High stability (submicron source stability)***
- ***Pulsed time structure*** - pulsed length down to tens of picoseconds allows the resolution of processes on the same time scale
- ***Polarization*** *(linear, circular, elliptical with Insertion Devices)*
- ***High vacuum environment***

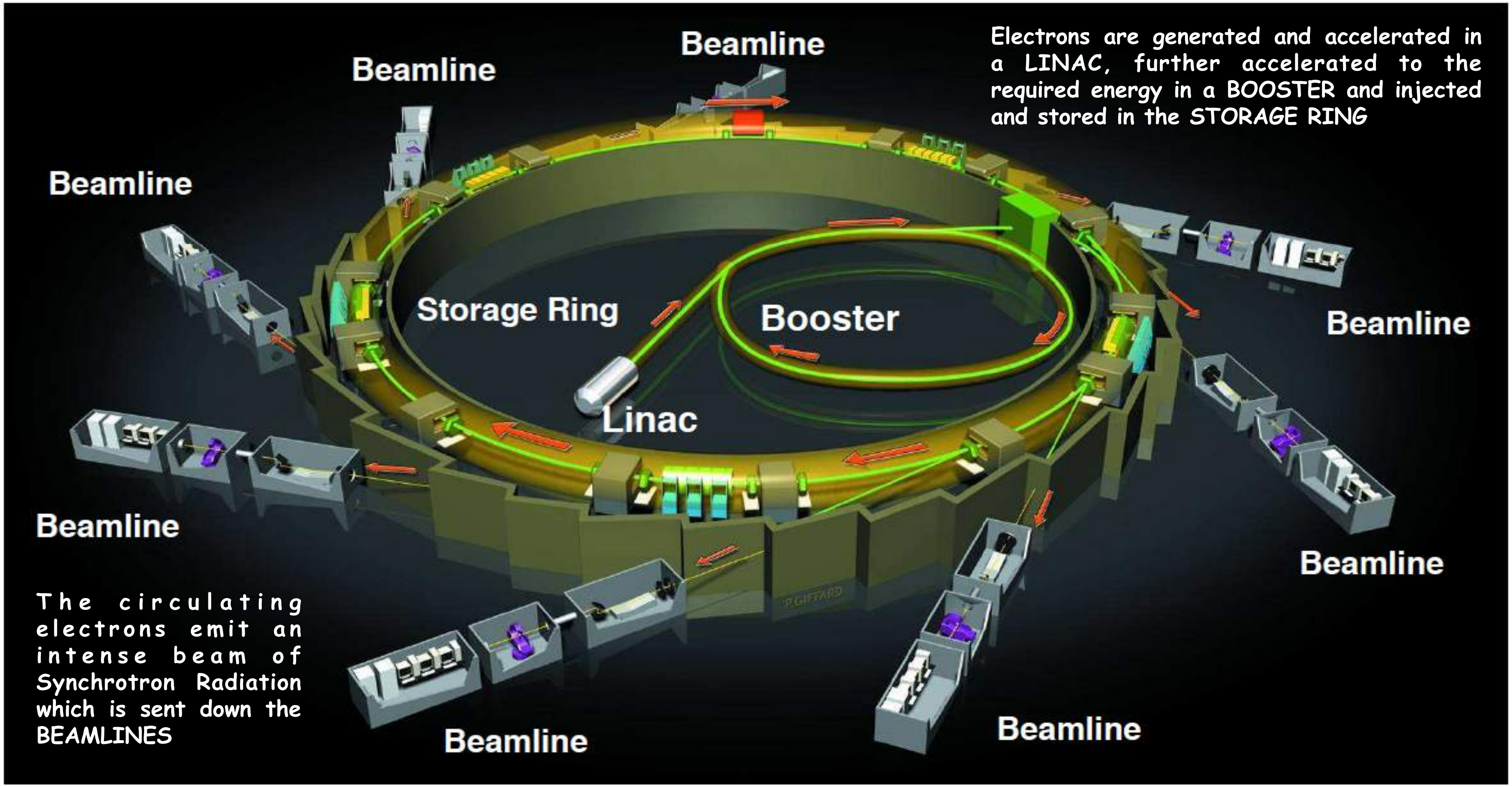


How Bright Is the Advanced Light Source?

ALS



Schematic view of a Synchrotron Radiation facility



As a function of the energy range to be used each beamline must be optimized for a particular field of research.

Beamline schematic composition:

- ***Front end***
- ***Optical hutch***
- ***Experimental hutch***
- ***Control and computing***

The ***front end*** isolates the beamline vacuum from the storage ring vacuum; **defines the angular acceptance of the synchrotron radiation** via an aperture; blocks(beam shutter) when required, the x-ray and Bremsstrahlung radiation during access to the other hutches.

Short history

Synchrotron Light Short History and Name

4th gen. - LINAC based accelerators
FELs

3rd* gen. ultimate storage rings like MAX IV (Sweden) near future

3rd gen. dedicated storage ring ESRF (France)
1994

Brightness increase

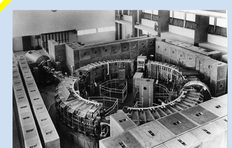
2nd gen. dedicated storage ring SRS (UK)
1981

1st gen. dedicated ring Tantalus I (USA)
1968

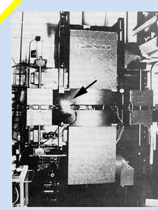
Storage rings development
1960s

ADA - B. Touschek - LNF

Parasitic use of electro-synchrotrons
1961



First observation of synchrotron radiation
1947



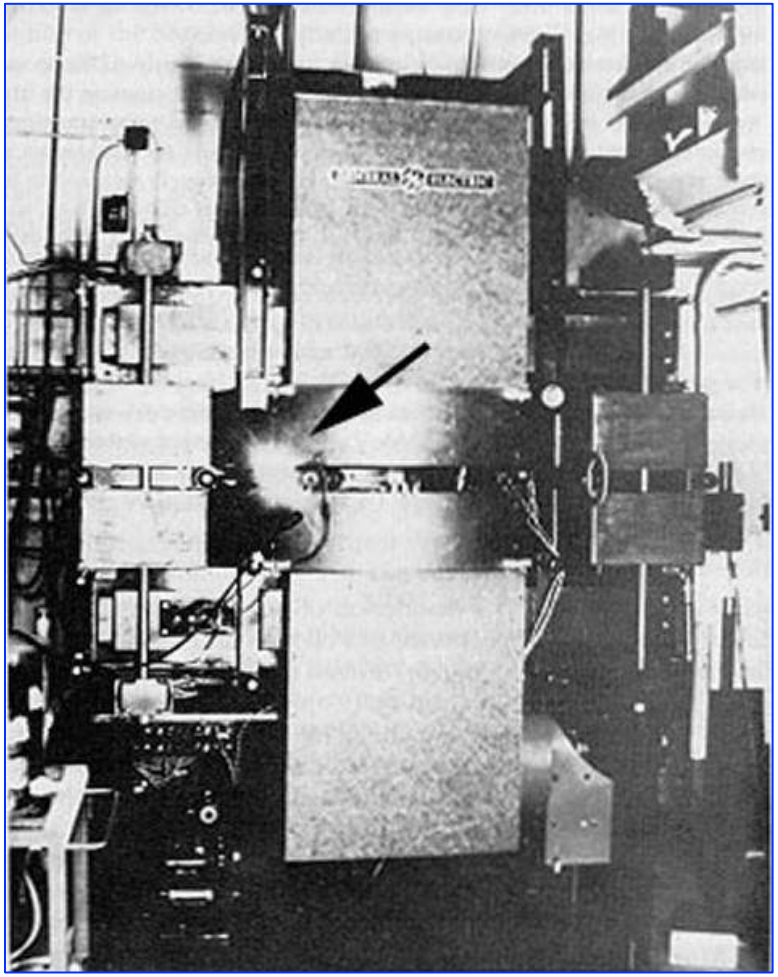
General Electric Res. Lab. - 70 MeV Electro-Synchrotron (N.Y. USA)

Proof of concepts, tests of theories
1897-1946

J. Schwinger Nobel Prize 1965 Classical Relativistic quantum field theory

Synchrotron radiation: history

First generation: parasitic operation and storage rings



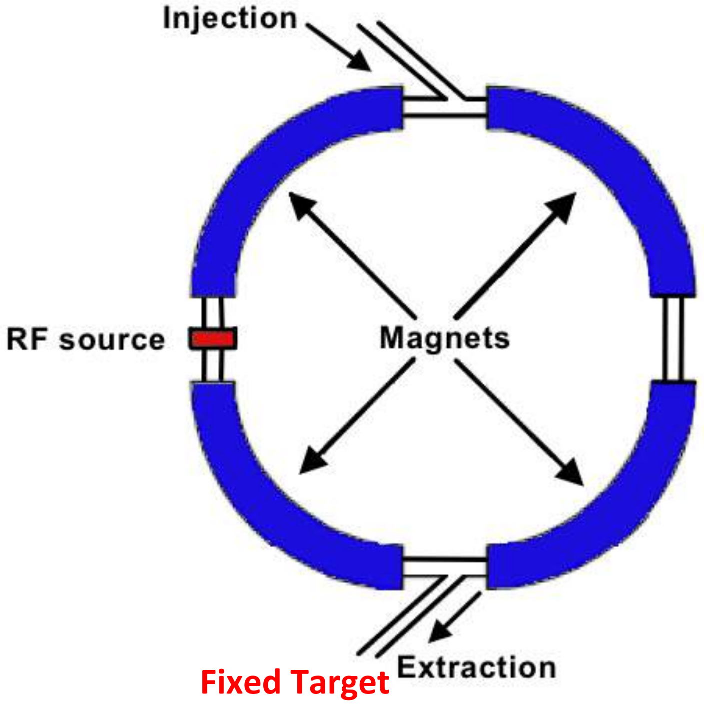
1947 ***General Electric Res. Lab. - 70 MeV Electron Synchrotron – N.Y. USA***

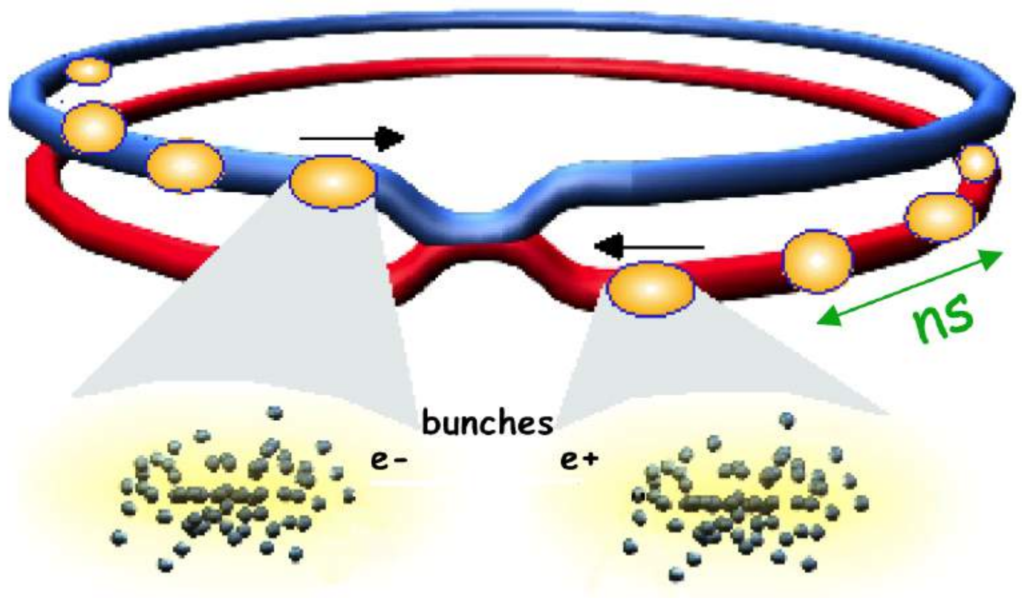
Starting point: Proof of concepts, tests of theories!

- In the 50s and 60s machines built for High Energy Physics: synchrotrons (***1947 First 'visual observation of synchrotron radiation***).
- Synchrotron radiation was considered a ***nuisance by particle physicists***: ***unwanted but unavoidable loss of energy***!
- 1961 US National Bureau of Standards (now NIST) modified their electron synchrotron : ***access to the synchrotron radiation users***.
- Synchrotron radiation scientists became ***parasites*** of nuclear physics experiments. (***1961 Frascati – CNEN Electro synchrotron – (0.4-1.1) GeV***)
- 1968 ***First storage ring dedicated*** to synchrotron radiation research: ***Tantalus*** (University of Wisconsin) only ***bending magnets.***

F.R. Elder, A.M. Gurewitsch, R.V. Langmuir, and H.C. Pollock, *Radiation from Electrons in a Synchrotron*, *Phys. Rev.* *71*, 829 (*1947*)
G. C. Baldwin and D.W. Kerst, *Origin of Synchrotron Radiation*, Physics Today, **28**, 9 (*1975*)

Synchrotrons and Storage Rings





Synchrotron

Particle beam on fixed target

$E_{CM} = (mE)^{1/2}$

Storage rings

Colliding particle beams

$E_{CM} = 2E$

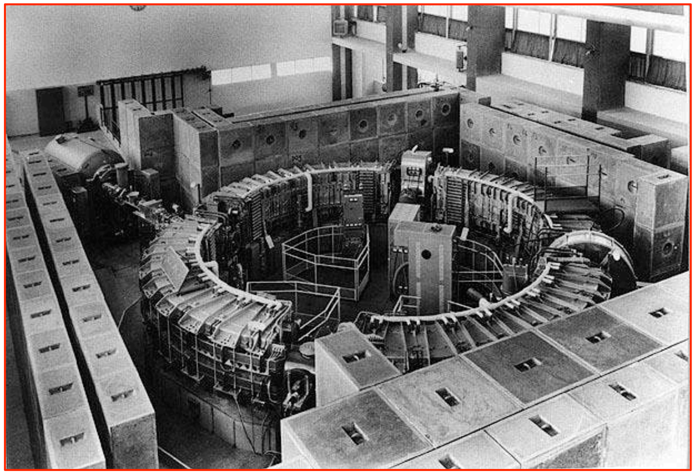
Colliding beams more efficient

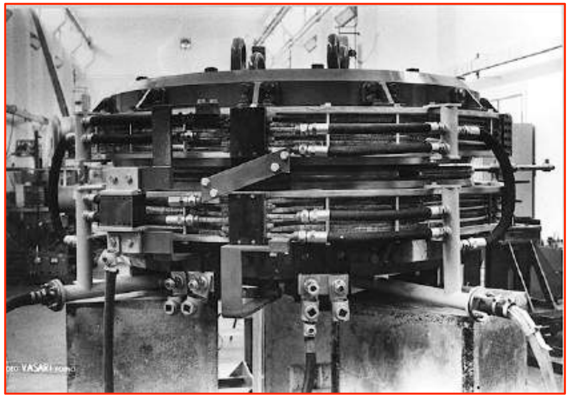
E= particle energy >> mc^2; E_{CM}= centre-of-mass energy

Synchrotron radiation: short history

Frascati: Electro-Synchrotron, ADA and ADONE

Frascati - CNEN (Comitato Nazionale Energia Nucleare) Laboratory Elettro-Sincrotrone – (0.4-1.1) GeV, C= 28 m (1959-1975)





LNF ADA (Anello Di Accumulazione) – first electron-positron storage ring (proposed by B. Touschek) 0.25 GeV, C= 5 m (1961-1964)

LNF ADONE (big ADA) electron-positron storage ring 1.5 GeV per beam, C = 105 m (1969-1993)

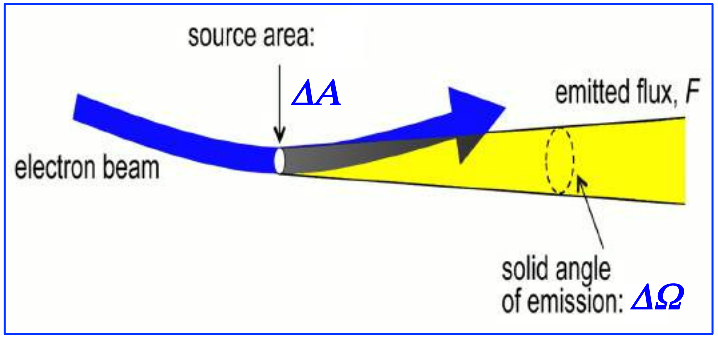
1976-1993 LNF ADONE 1.5 GeV parasitic/dedicated use for SR experiments after its use for HE experiments.

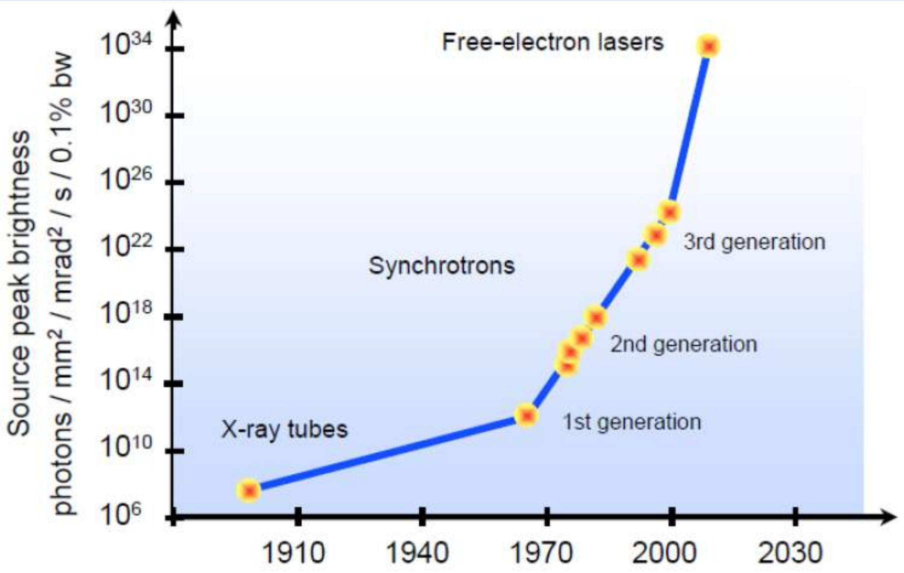
Increasing brightness

Brightness (flux density in phase space) is an invariant and depends on the ***size of the source (ΔA)*** (electron beam) and on ***the angular divergence of the radiation ($\Delta\Omega$)***, given by the convolution of the angular distribution of synchrotron radiation with the angular divergence of the electron beam.

Brightness more important than flux (photons/s).

Brightness = photon flux/ [(ΔA) ($\Delta\Omega$)]





In a storage ring the ***product of the electron beam transverse size*** and ***angular divergence*** is a constant along the ring and is called ***emittance (vertical*** and ***horizontal emittance).***

Increase of a factor 1000 every 10 years!!!

Brightness is the main figure of merit of synchrotron radiation sources and its huge increase, was obtained designing low emittance machines, minimizing the source size and the beam divergence.

$$\text{Spectral Brightness} = \frac{photons}{second \cdot mrad^2 \cdot mm^2 \cdot 0.1\%BW}$$

Synchrotron radiation: short history

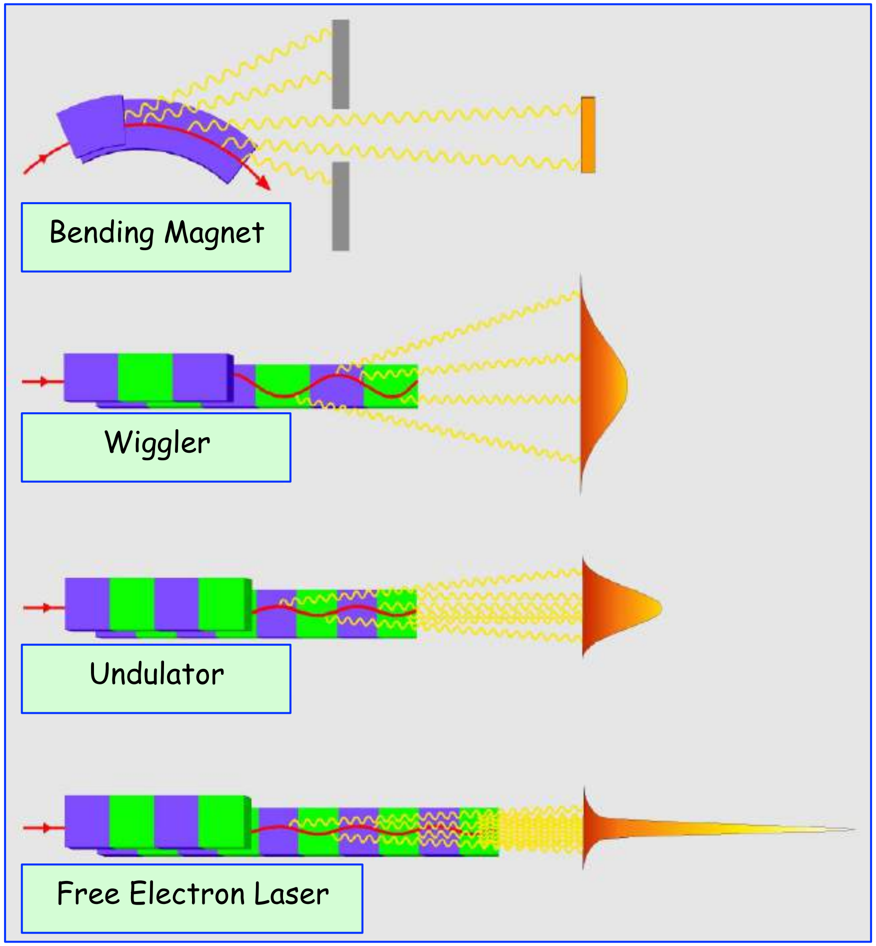
Third generation: optimized sources

Synchrotron light is now a unique tool for science!

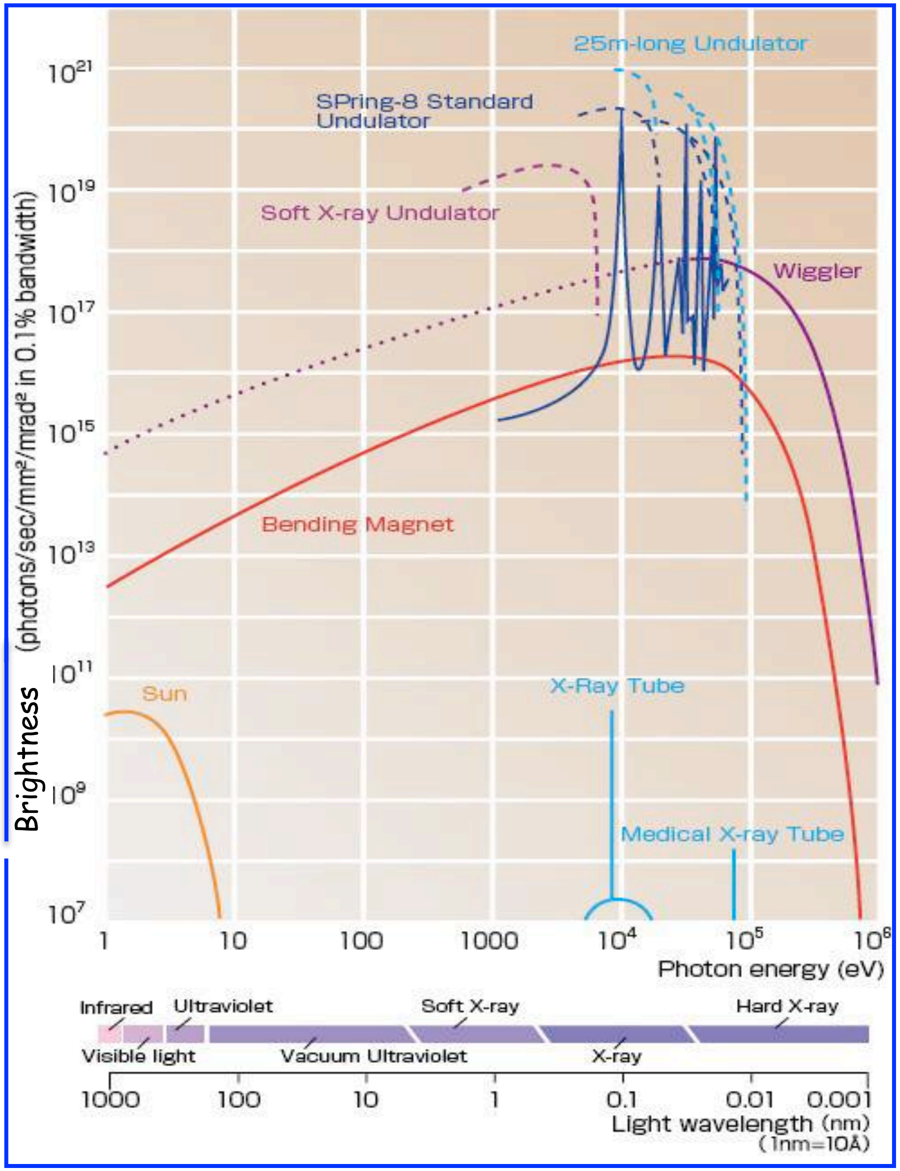
ESRF, Grenoble - France 6 GeV, C = 844 m opened to users in 1994

- *Sources designed specifically for high brightness or low emittance.*
- *Emphasis on research with insertion devices like undulators!*
- *High-energy machines able to generate hard x-rays*
- *Larger facilities to support rapidly growing user community, many beamlines high number of users.*

Comparing the achievable brightness



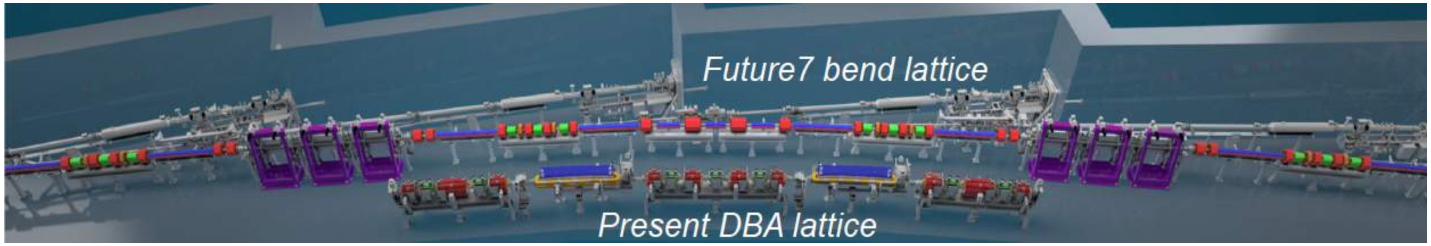
Courtesy DESY



Courtesy SPring-8

Synchrotron radiation: history
Present and future: Ultimate Storage Rings

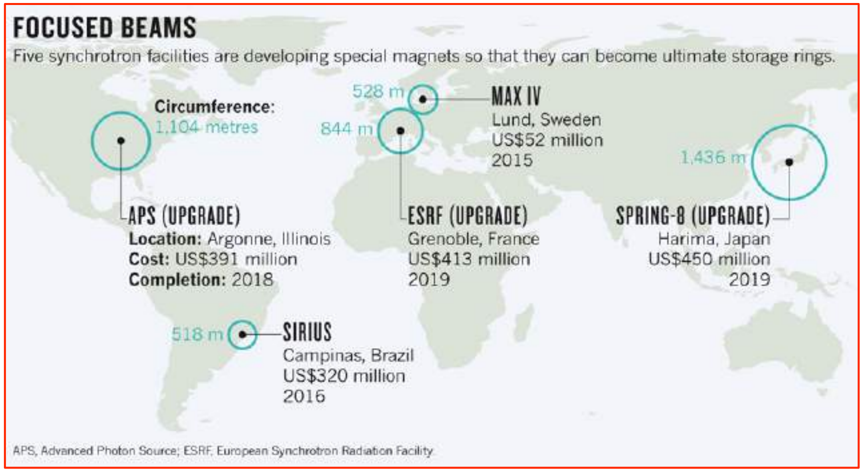
*Brightness and transverse coherence increase in the X-ray range with implementation of **low emittance lattices** (multi-bend achromat schemes).*



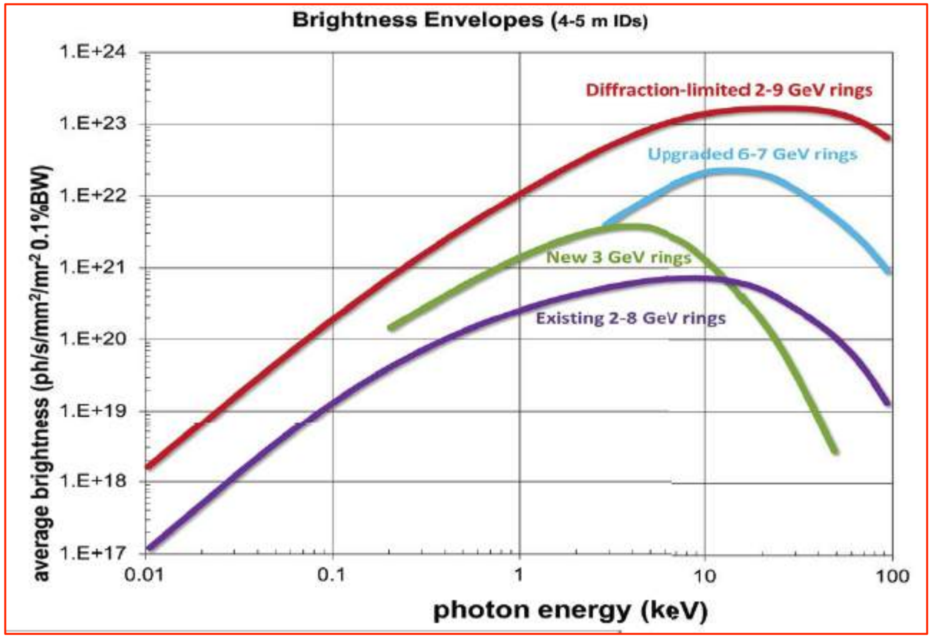
$$\varepsilon_{x_0,min} \propto \frac{E^2}{N_B^3}$$

J.Jacob, Status of the ESRF operation & upgrade, 2013

ESRF ε_x= 4 nm; ε_x= 5 pm in the future ε_x= 0.16 nm



E.S. Reich, Ultimate upgrade for US synchrotron, Nature, 2013



H. Owen – Univ. of Manchester (UK)

3rd Generation SR Light Sources

ESRF - France

DIAMOND - UK

ALBA - Spain

Under construction – Ultimate SR facilities

Lund - Sweden

Sirius - Brazil

Shanghai -China

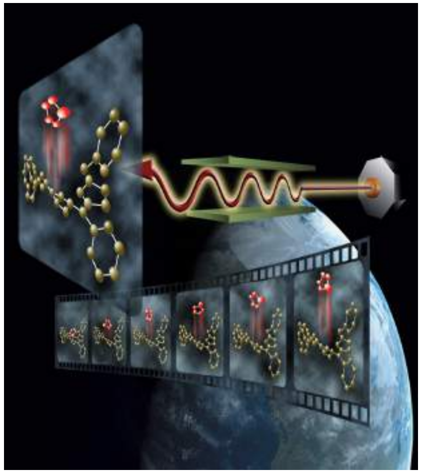
Fourth generation:
X-ray free electron lasers
XFELs

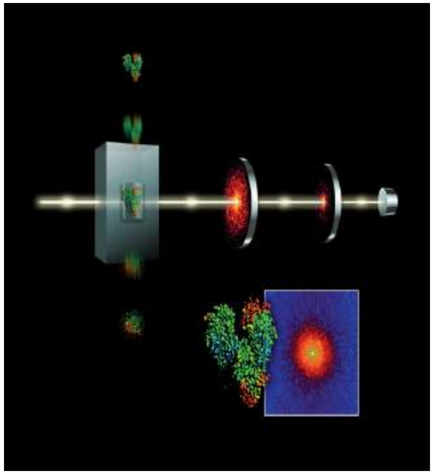
Electrons in a FEL are not bound to atoms or molecules. The "free" electrons traverse a series of alternating magnets, called "undulators," and radiate light at wavelengths depending on electrons energy, undulator period and magnetic field.

Synchrotron radiation: history

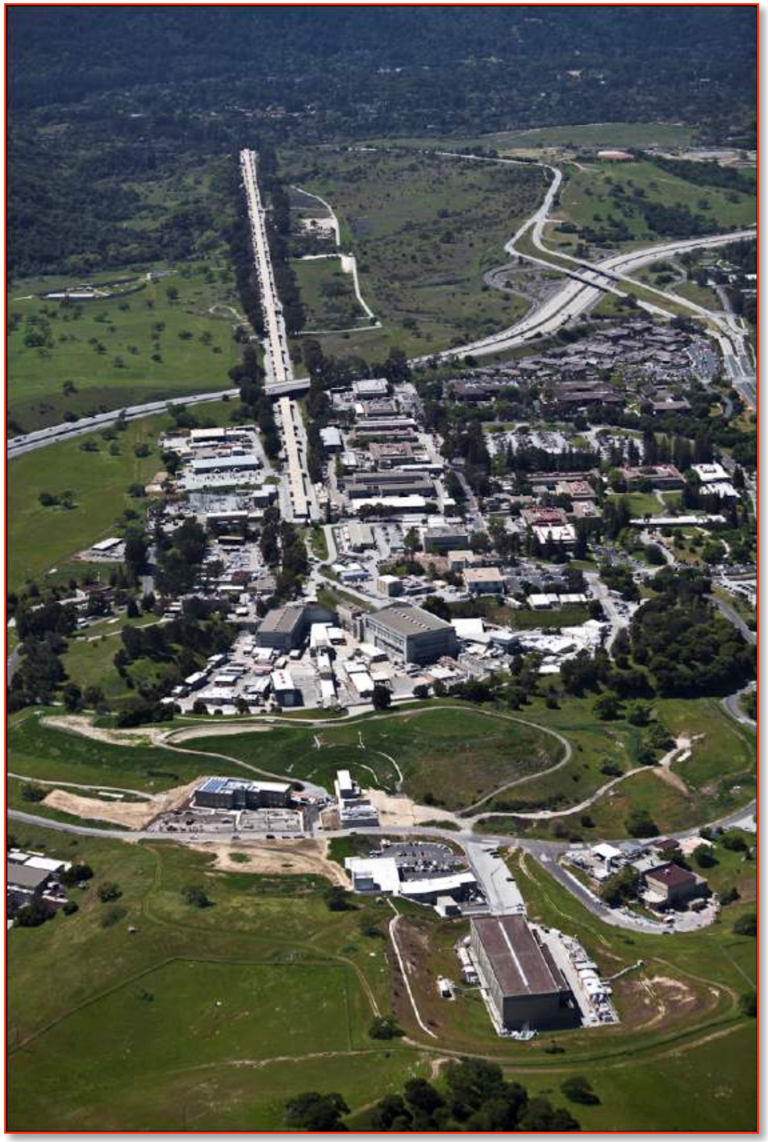
Fourth generation: LINAC based sources and Free Electron Lasers

- *Extremely bright and coherent sources*
- *Ultrafast pulses*
- *Already working in IR to UV and X-ray (LCLS April 2009) ranges*
- *European XFEL started activity in 2017*
- *Filming chemical reactions as they occur*
- *Protein crystallography no longer needed –image molecules directly*

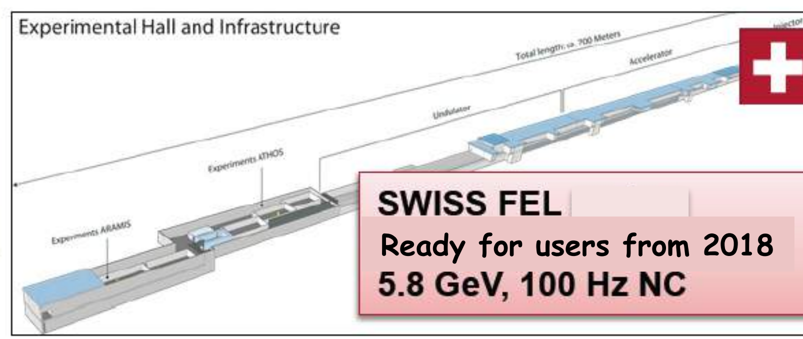




LCLS http://www-public.slac.stanford.edu/lcls/aboutlcls.aspx



XFELs present and future



NC: normal conducting acceleration, SC: super conducting acceleration

FUTURE

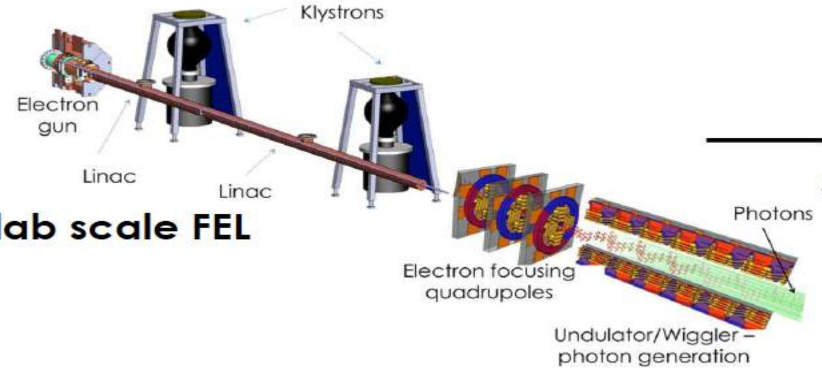
Fifth Generation Light Sources

… re-invent XFELs to fit in campus laboratory.

The X-ray FEL is enormous. Challenge is to fit this…

1 km

Into this…



The university lab scale FEL

5 m

J. Rosenzweig, UCLA - Fifth Generation Light Sources X-ray FELs Based on New Accelerator and Undulators - 2014

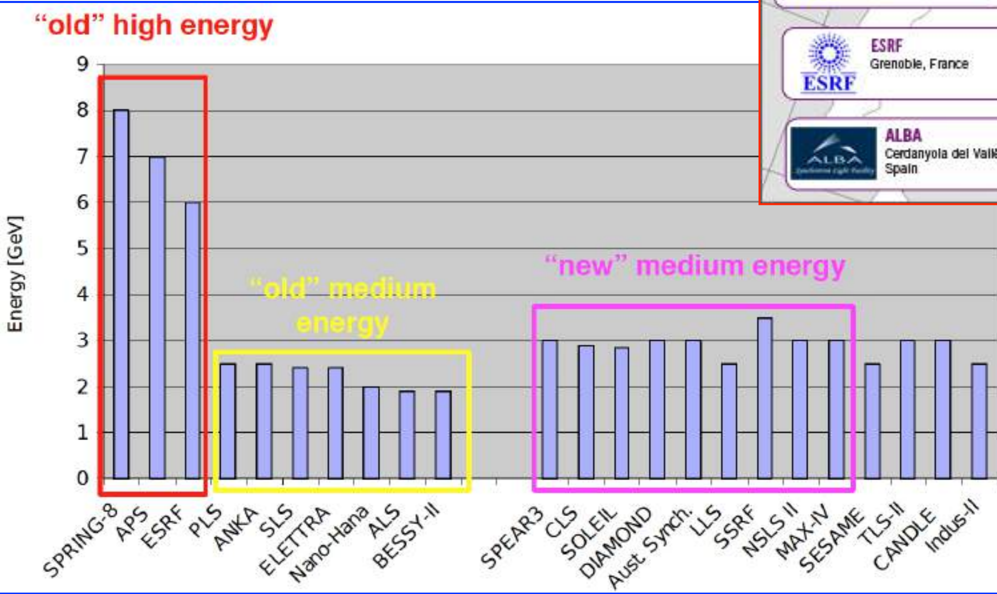
Ongoing research on laser/plasma/wakefield accelerators, high frequency, high repetition rate linacs and electron beam injectors can lead in the future to very compact, university scale, X-ray FELs.

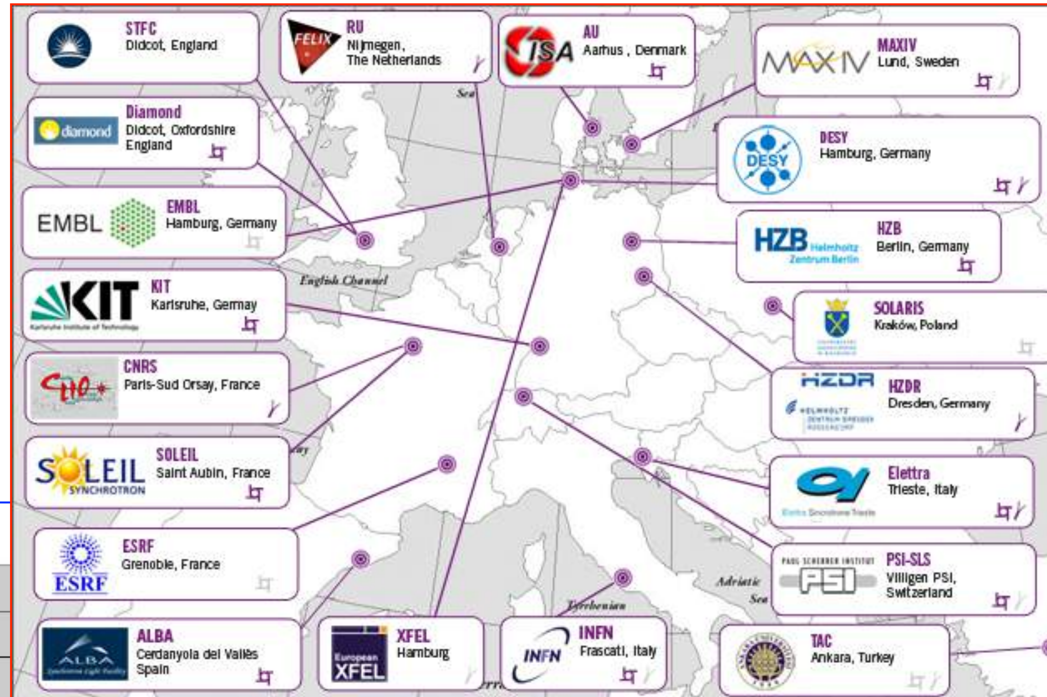
C. Pellegrini, UCLA - 5th Generation light sources - 2011

SR facilities

Synchrotron radiation facilities

18 in America
25 in Asia
25 in Europe
1 in Oceania
including facilities under design and FELS





EUROPE

Info on European Synchrotron Radiation Facilities: www.wayforlight.eu
About 67 operational Synchrotron Radiation Facilities Around the World information on: www.lightsources.org

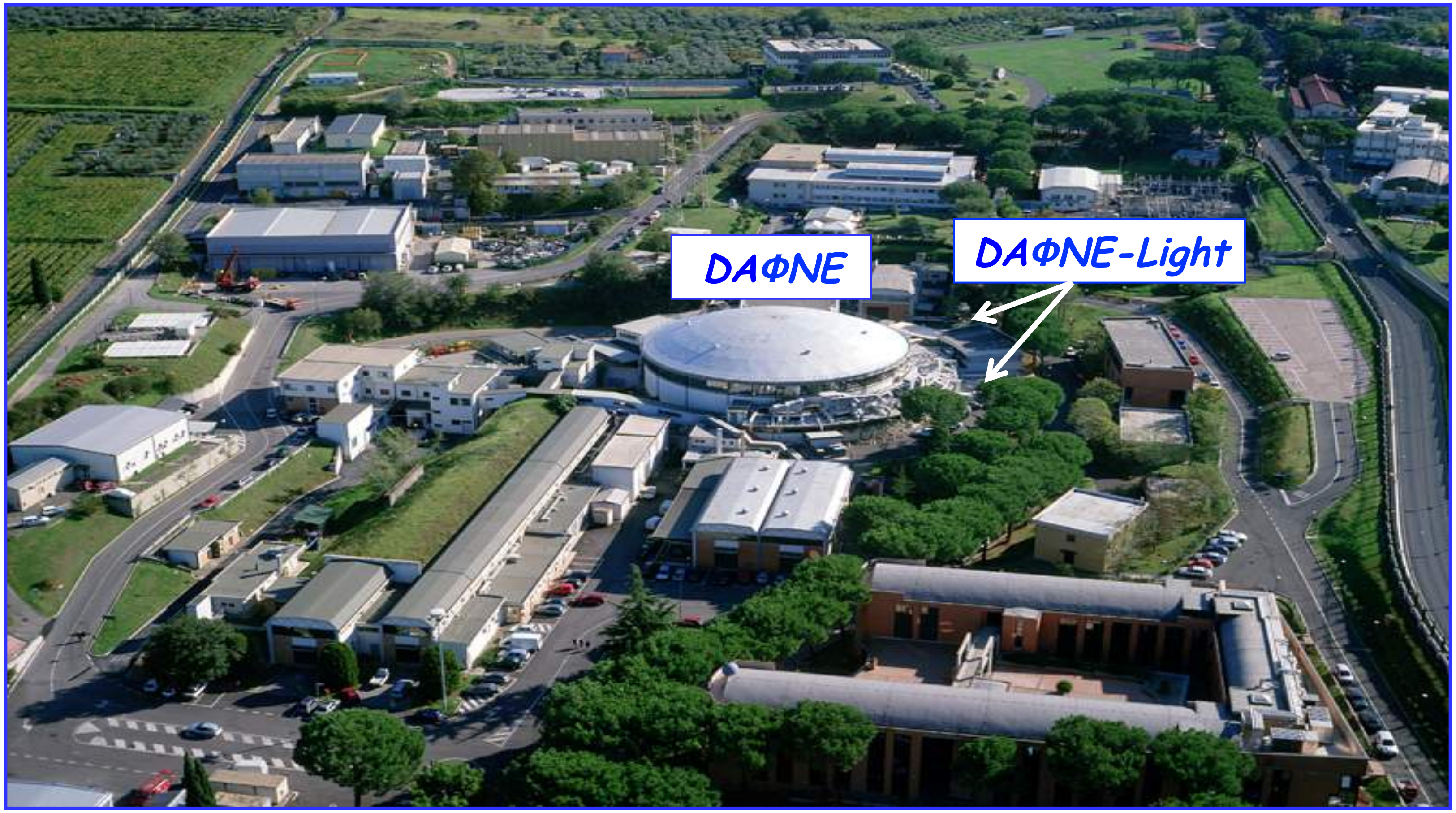
Synchrotron radiation @ INFN-Frascati National Laboratory

DAΦNE Light

Synchrotron Radiation Facility

DAΦNE-Light

INFN-LNF Synchrotron Radiation Facility



Beamlines @ DAΦNE

1) SINBAD - IR beamline (1.24 meV - 1.24 eV)

2) DXR1- Soft x-ray beamline (900-3000 eV)

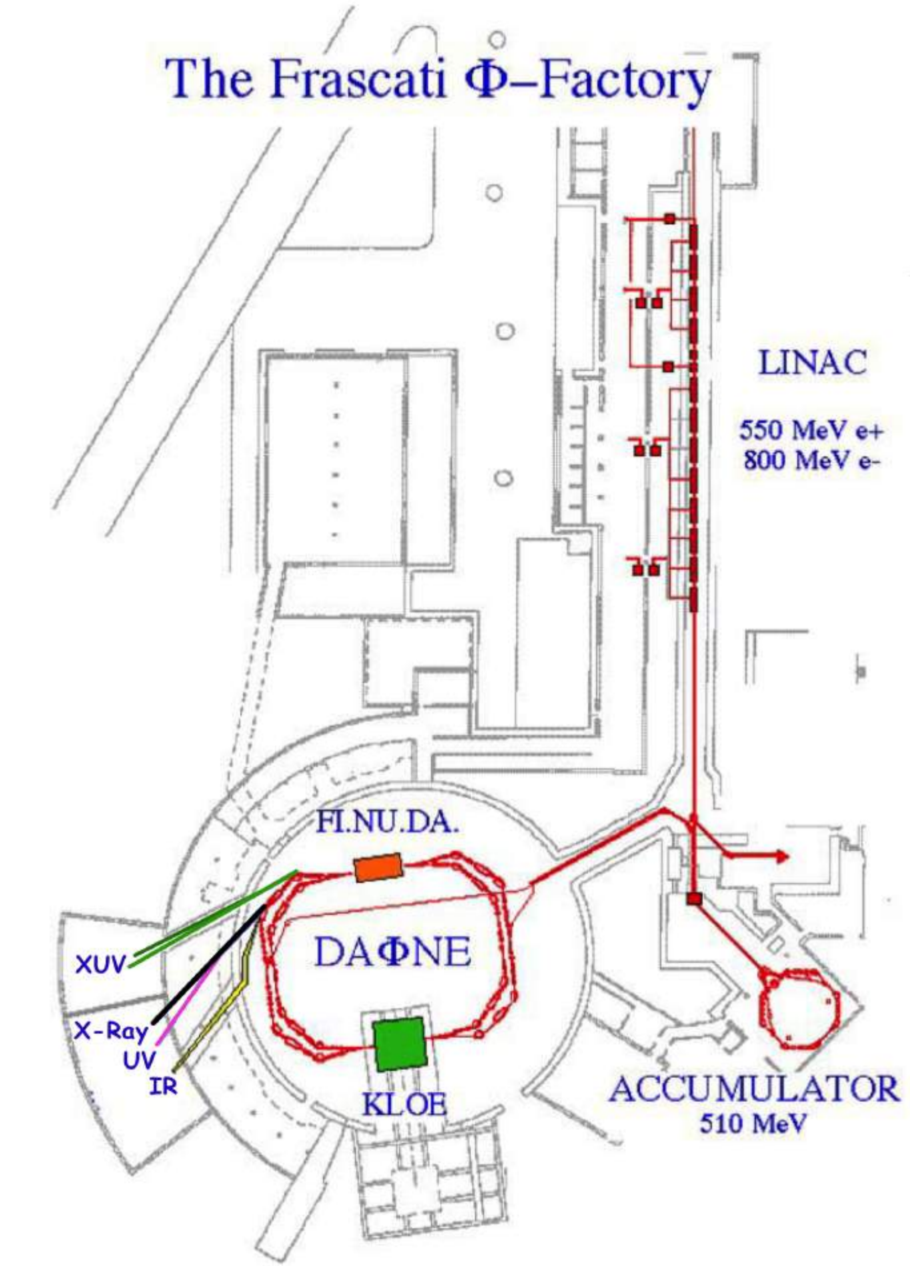
Open to Italian and EU users

3) DXR2 – UV-VIS beamline (2-10eV) new setup.

XUV beamlines

4) Low Energy Beamline (35-200 eV) under commissioning;

5) High Energy Beamline (60-1000eV) under commissioning.



Available techniques

- FTIR spectroscopy, IR microscopy and IR imaging

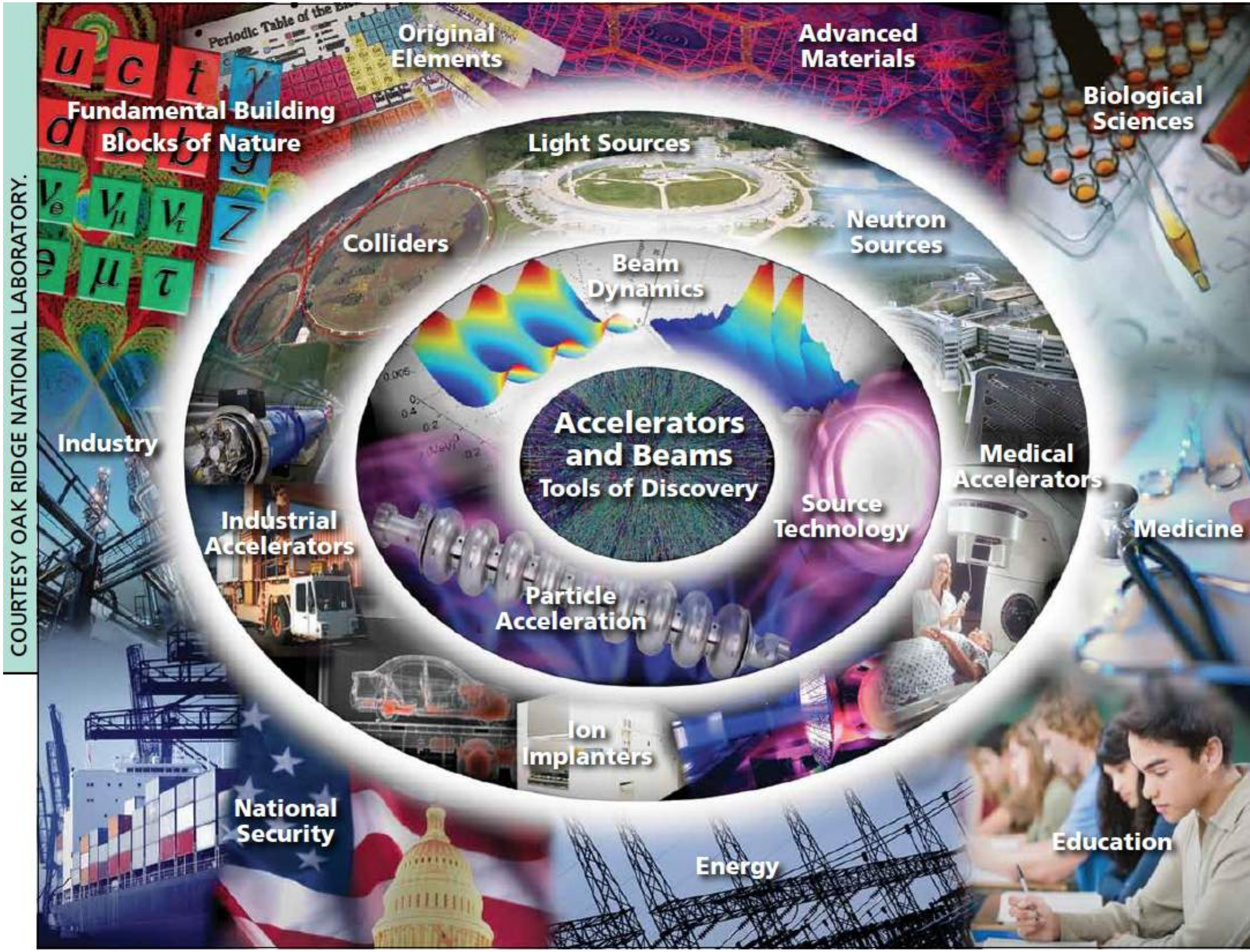
- UV-Vis absorption spectroscopy

- Photochemistry: UV irradiation and FTIR micro-spectroscopy and imaging.

- Soft x-ray spectroscopy: XANES (X-ray Absorption Near Edge Structure) light elements from Na to S

- SEY (secondary electron yield) and XPS (X-ray photoelectron spectroscopy) – by electron and photon bombardment

From accelerators to applications



E. Malamud *Ed.*, ***Accelerators and Beams tools of discovery and innovation*** (http://www.aps.org/units/dpb/news/edition4th.cfm) ***2013***

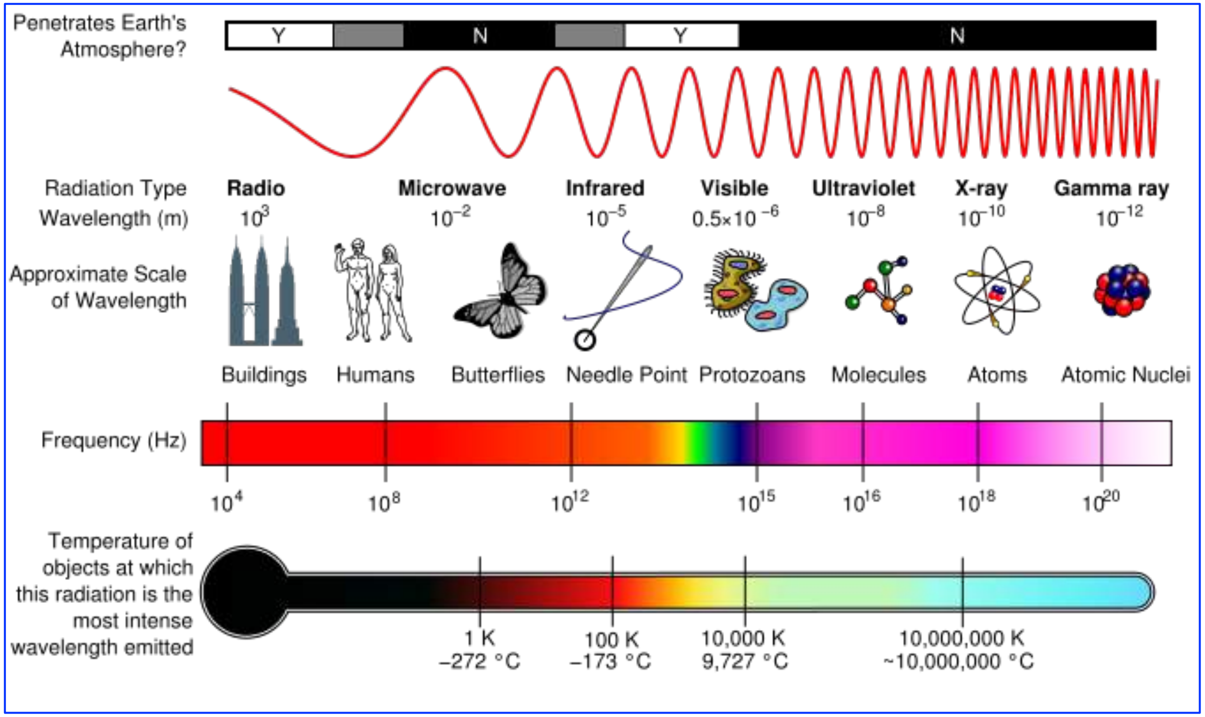
X-Ray Interaction with Matter

Light waves across the electromagnetic spectrum behave in similar ways.

When light waves encounter objects, they are either **transmitted**, **reflected**, **absorbed**, **refracted**, **polarized**, **diffracted**, or **scattered** **depending on the composition of the object and the wavelength of the light**.

Interaction of radiation with matter

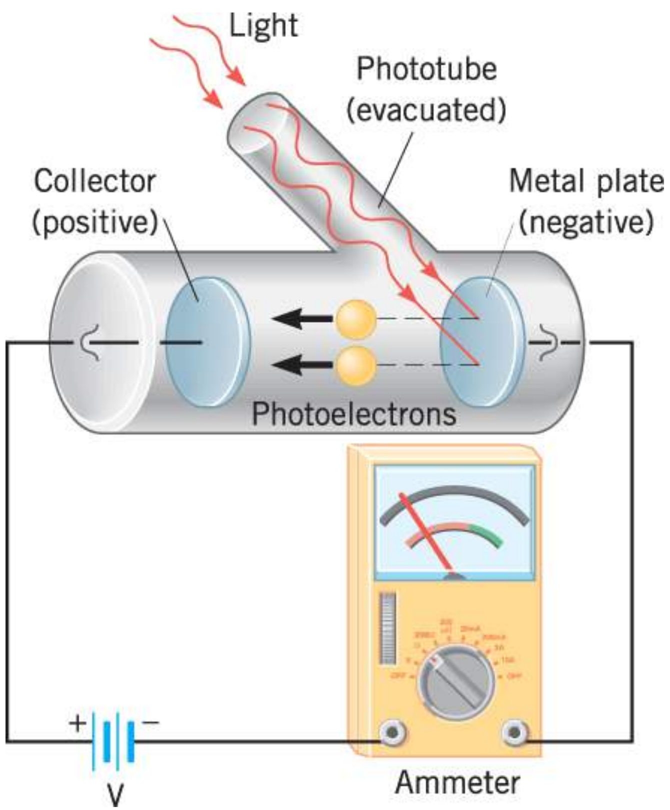
The different kind of radiation (IR, VIS, UV, X) have very **different interaction with matter**.
The **human body is quite transparent to low frequency radio waves**. You can listen to your radio at home since the radio waves pass freely through the walls of your house.



Moving upward through microwaves and infrared to visible light, UV and X-rays radiation is absorbed more and more strongly.

Photoelectric effect

Electromagnetic radiation can be used to **knock out electrons, freeing them from the surface of a solid**. This process is called the ***photoelectric effect*** (or ***photoelectric emission*** or ***photoemission***), a material that can exhibit this phenomena is said to be ***photoemissive***, and the ejected electrons are called ***photoelectrons***; but there is nothing that would distinguish them from other electrons. All electrons are identical to one another in mass, charge, spin, and magnetic moment.



The photoelectric effect does not occur when the frequency of the incident light is less than the ***threshold frequency***. ***Different materials have different threshold frequencies***.

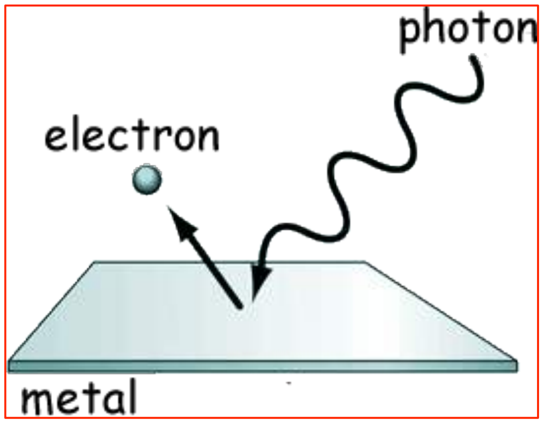
Photoelectric effect

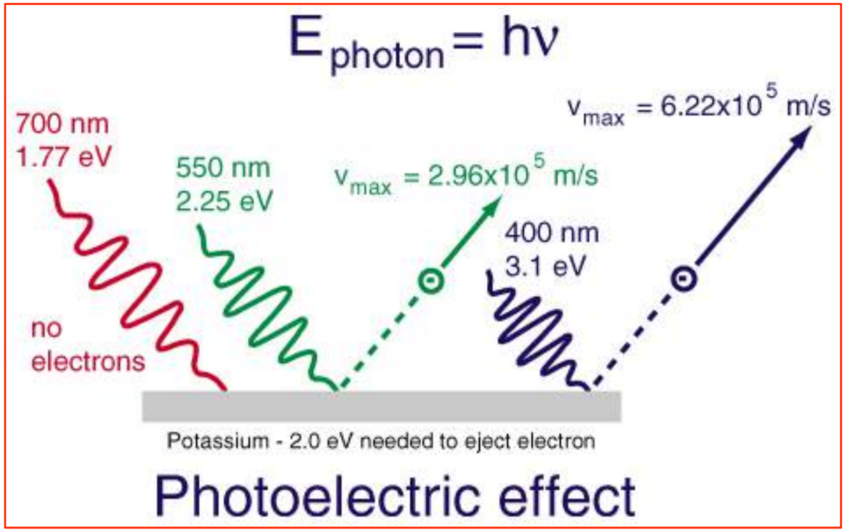
Classical physics cannot explain why…

- no photoelectrons are emitted when the incident light has a frequency below the threshold,
- the maximum kinetic energy of the photoelectrons increases with the frequency of the incident light.

Modern physics states that…

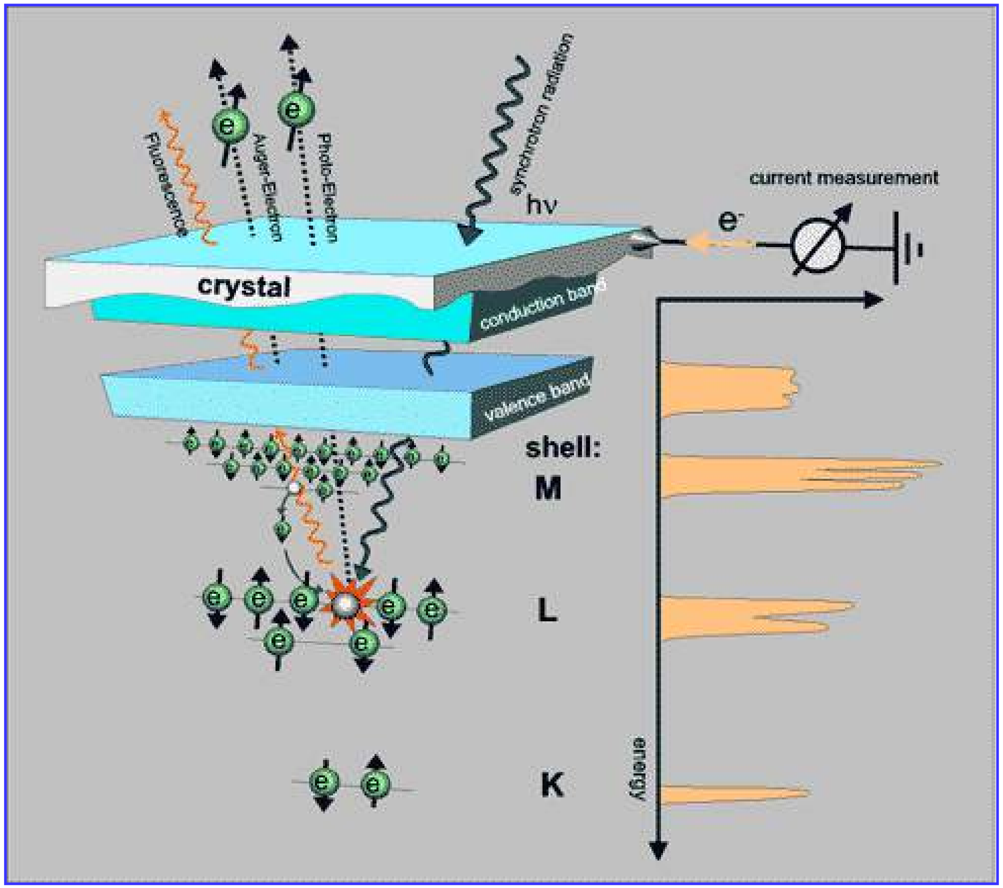
- electromagnetic radiation is composed of discrete entities called photons
- the energy of a photon is proportional to its frequency
- the work function of a material is the energy needed per photon to extract an electron from its surface

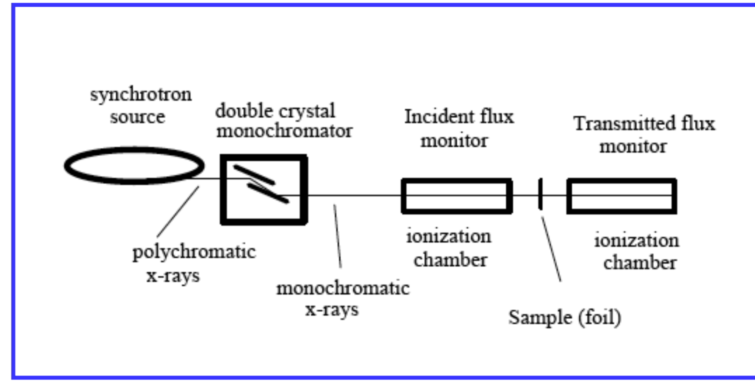


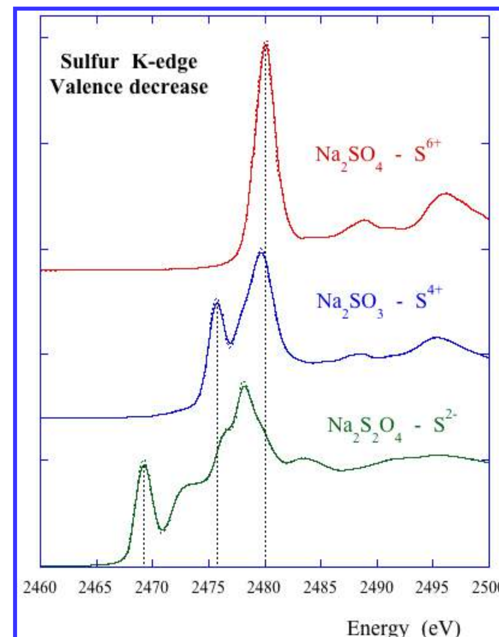


In 1905, *Albert Einstein realized that light was behaving as if it was composed of tiny particles* (initially called quanta and later called photons) and that **the energy of each particle was proportional to the frequency of the electromagnetic radiation** (**Nobel Prize in Physics in 1921**).

Photoelectric effect and X-rays



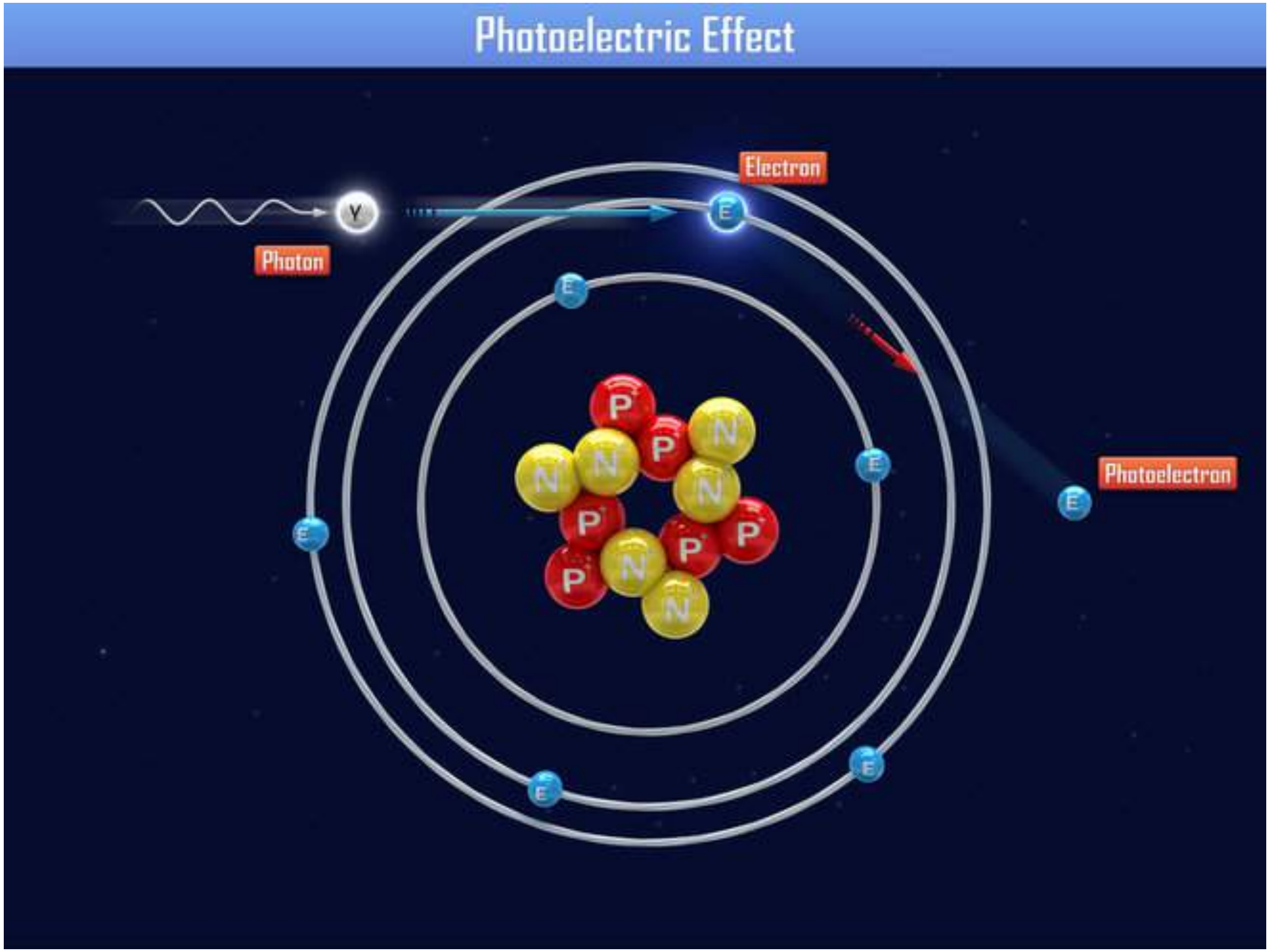




X-ray absorption spectra at the Sulfur K edge - XANES

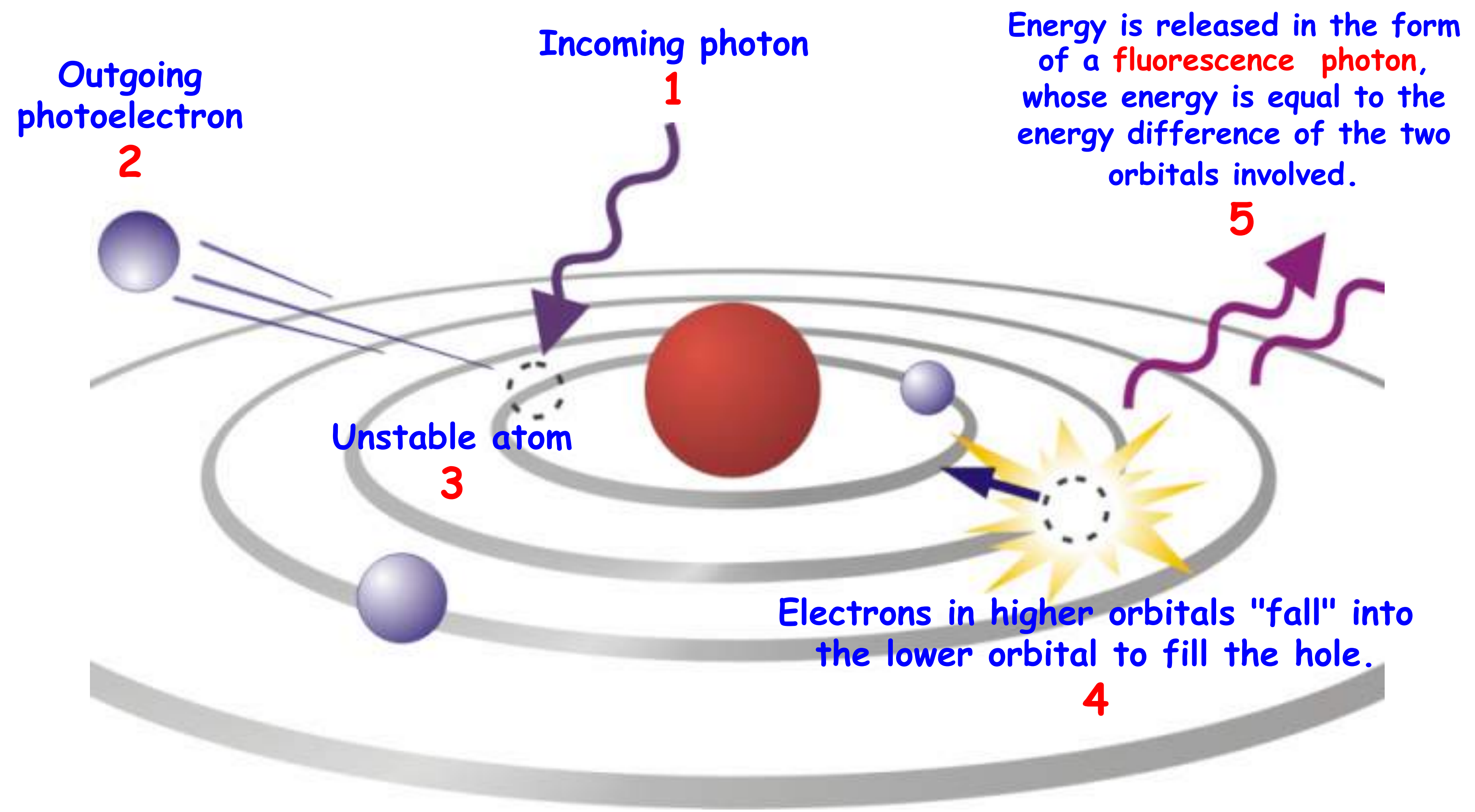
X-ray Absorption and Fluorescence Decay

Photoelectric effect



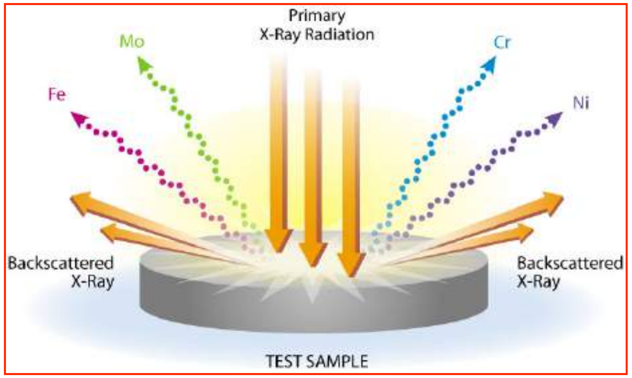
A photon (Y) strikes an electron (E), it knocks, it loose and creates a photoelectron.
Credit: general-fmv

Absorption and fluorescence decay

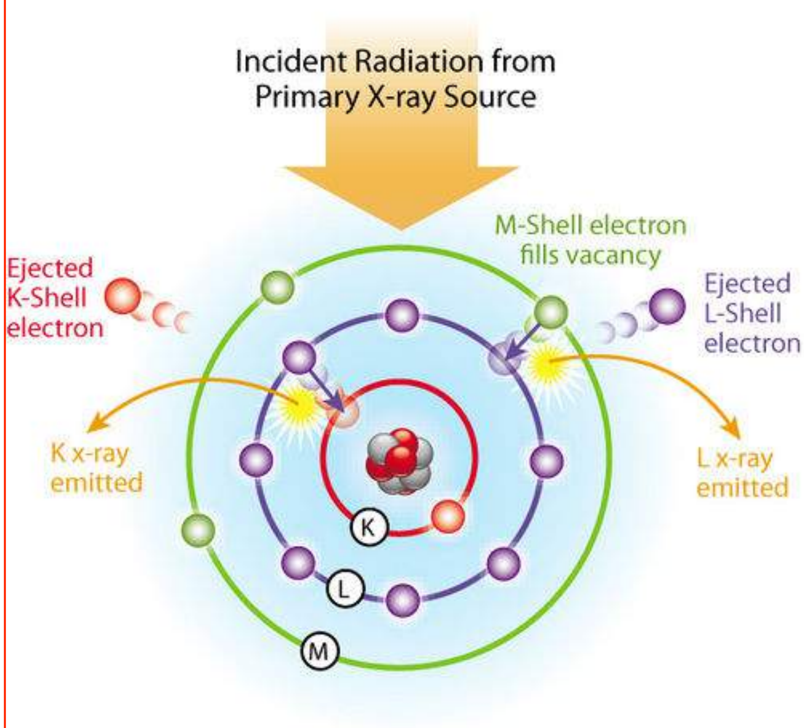


The material emits radiation, which has energy characteristic of the atoms present.

Decay Process: X-ray Fluorescence

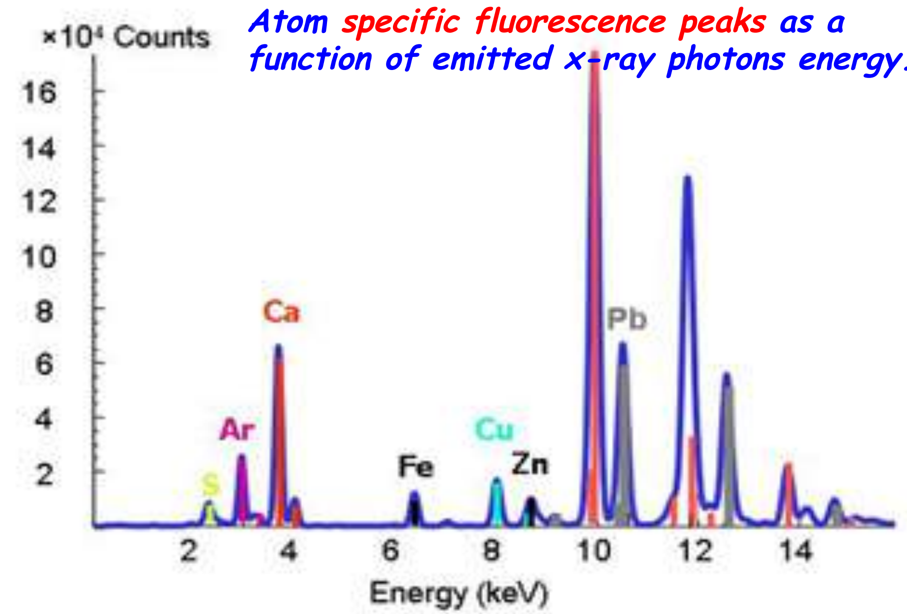


X-ray fluorescence spectrometry (***XRF***) can be used to accurately ***measure the atomic composition of a material***. In an atom, the **electrons orbit around the nucleus in characteristic patterns referred to as "shells."** ***X-rays have enough energy to knock electrons out of a shell***. If an electron is extracted from an inner shell, an electron from an outer shell will move to replace it. When the electron moves from the outer shell to the inner shell, it ***releases energy in the form of a photon (light)***. The energy of the ***"fluorescent" photon*** released is **distinct for each atomic element creating** a measurable "**fingerprint**" for that element. XRF produces ***a non-destructive chemical analyses of any kind of sample***.

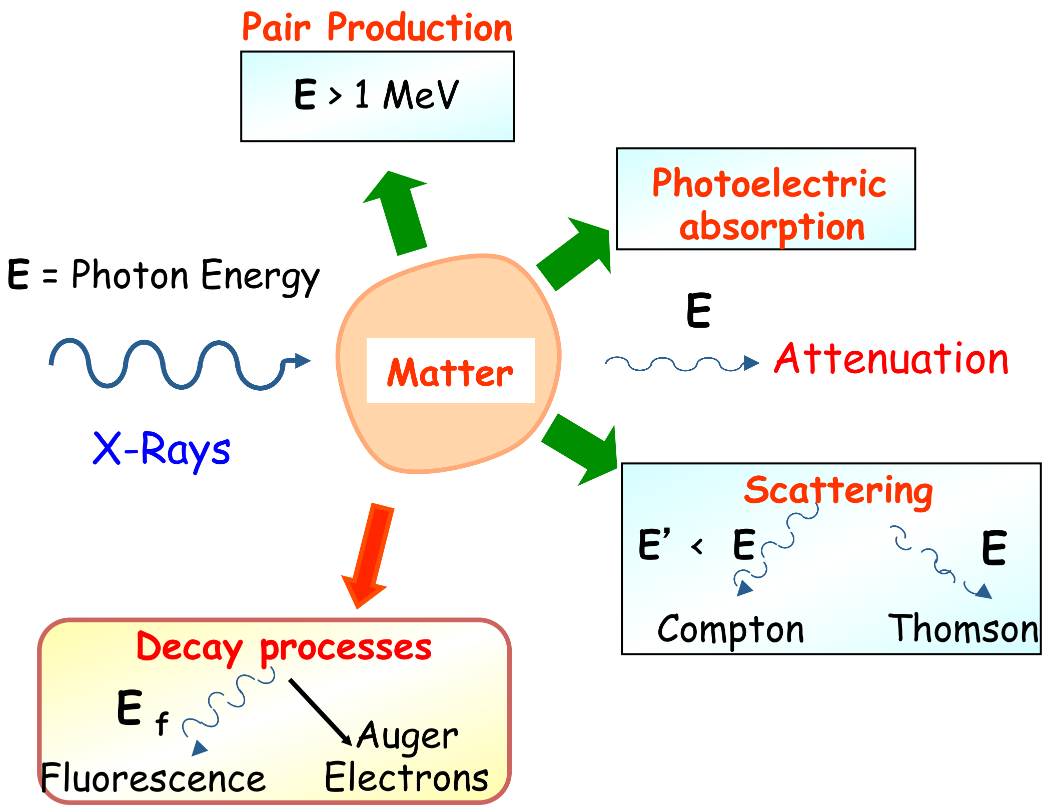


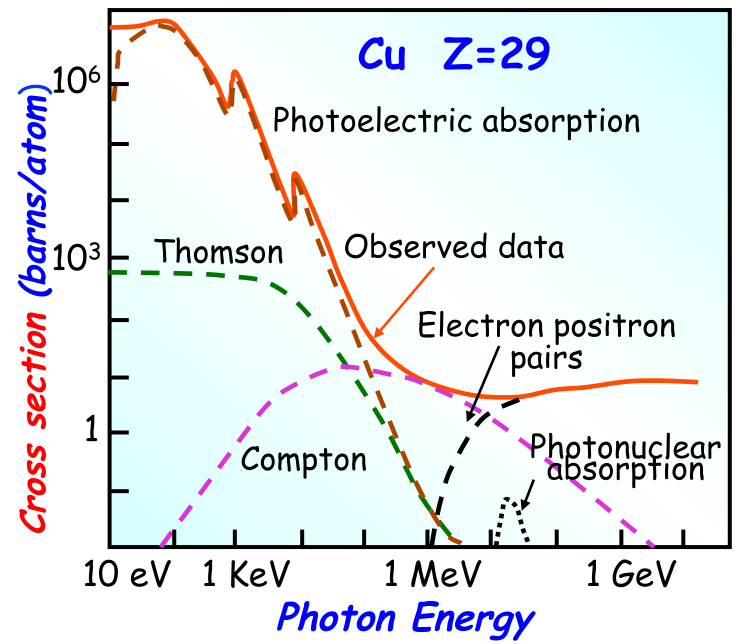
Moseley's Law : $E \approx Z^2$

Atom specific fluorescence peaks as a function of emitted x-ray photons energy.



Interaction of X-rays with matter

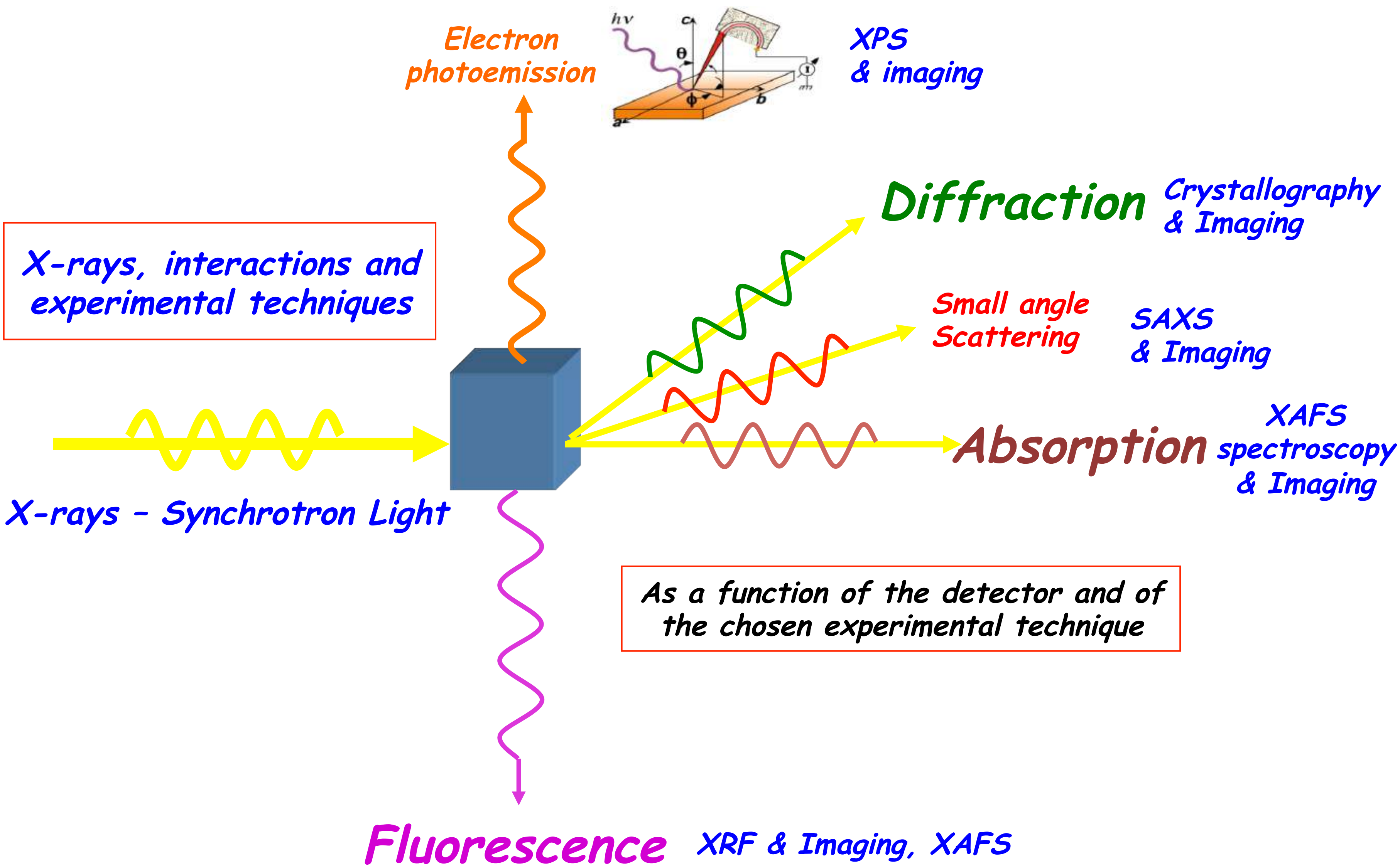




There are different types of **interaction of X-rays with** matter but taking into account the energy range of interest the **ones that will be taken into account** are ***absorption*** and ***elastic*** or ***Thomson scattering***.

The PROBABILITY for any given reaction to occur is in PROPORTION to its CROSS SECTION.

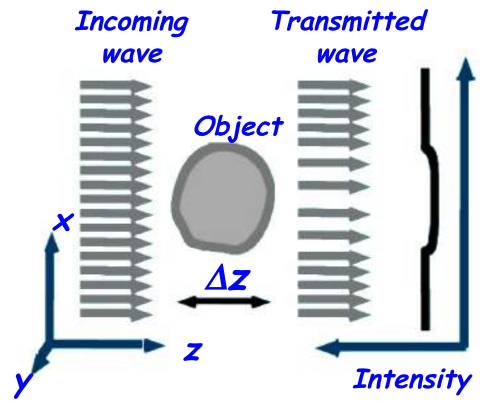
X-rays interactions with matter and experimental techniques

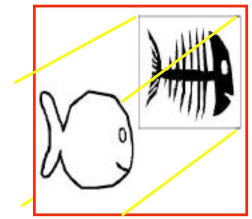


Some X-ray techniques

Imaging

Conventional radiology relies on X ray absorption

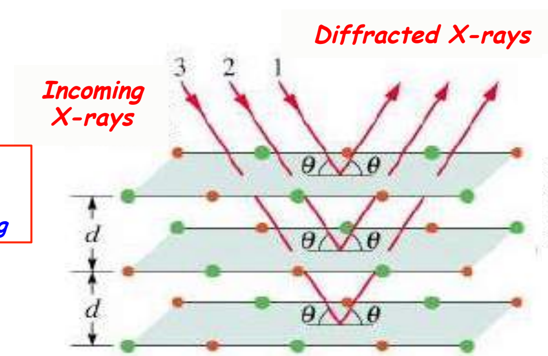


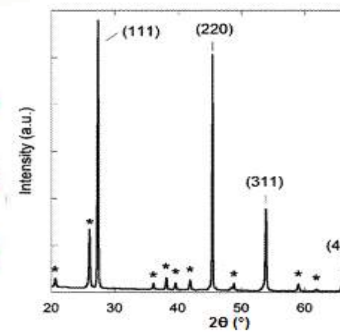


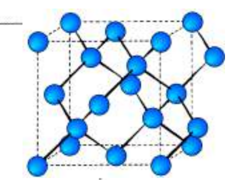
Diffraction

Elastic scattering : Thomson (elastic) if $E<E_{binding}$

$$n\lambda = 2d\sin\theta \quad \text{Bragg's Law}$$





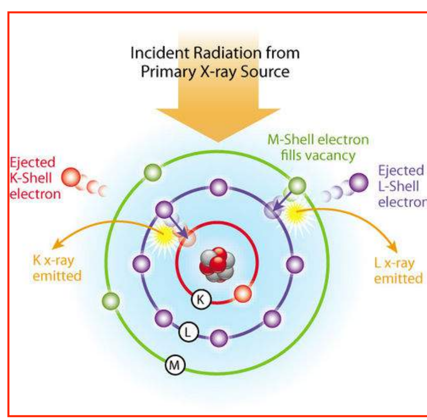


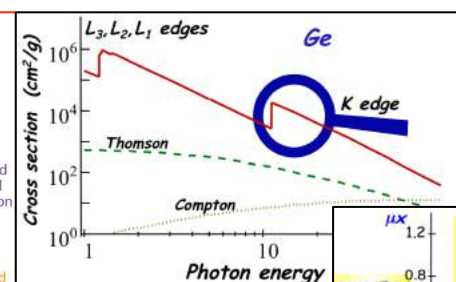
Ge crystal structure

Absorption Spectroscopy

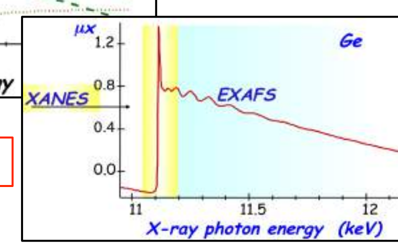
X ray Absorption Fine Structure (XAFS)

Transmission and fluorescence mode

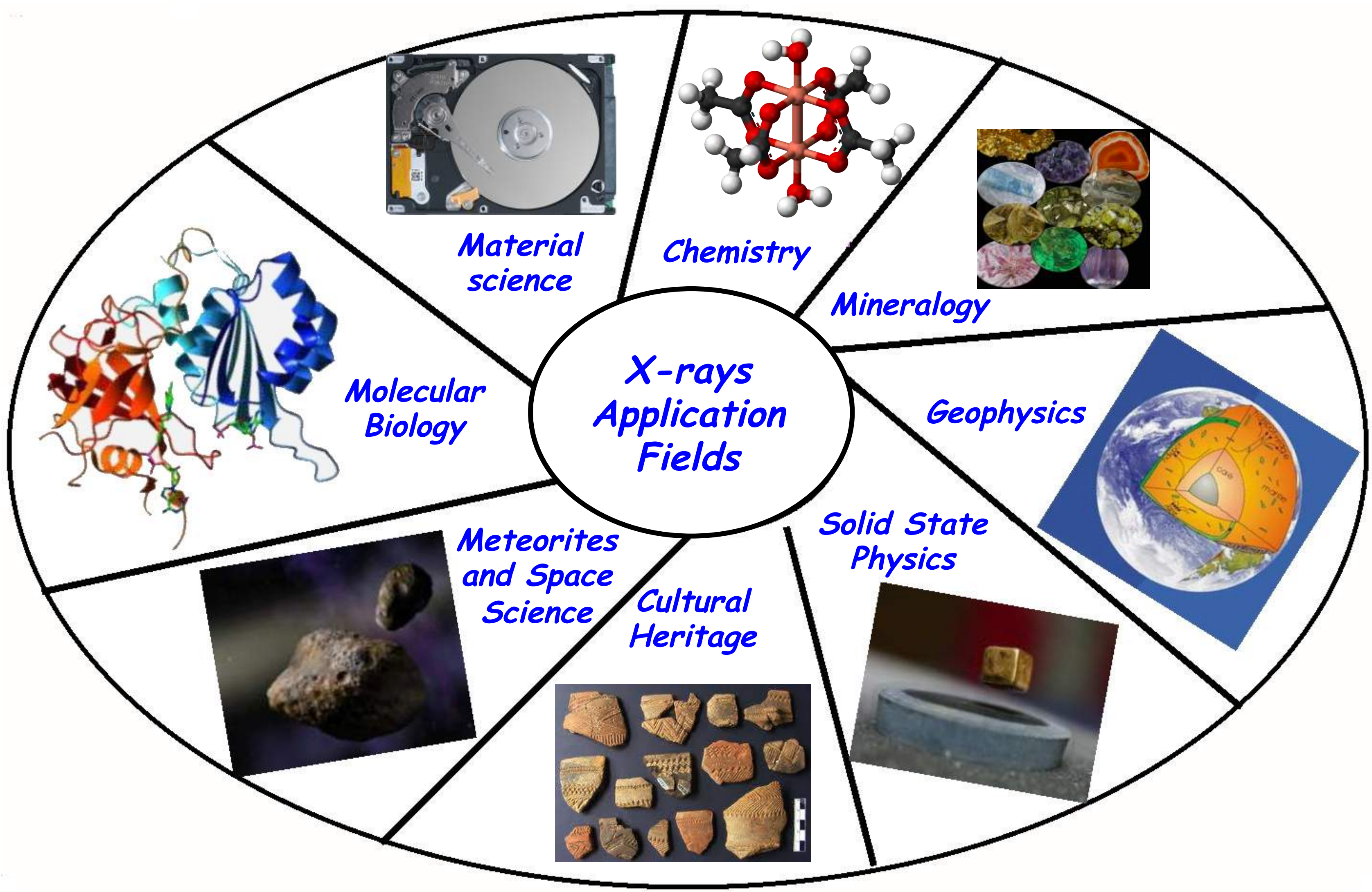




XAFS=XANES+EXAFS



Synchrotron radiation applications using X-rays



Some applications using X-rays (synchrotron light)

X-rays and Cultural Heritage

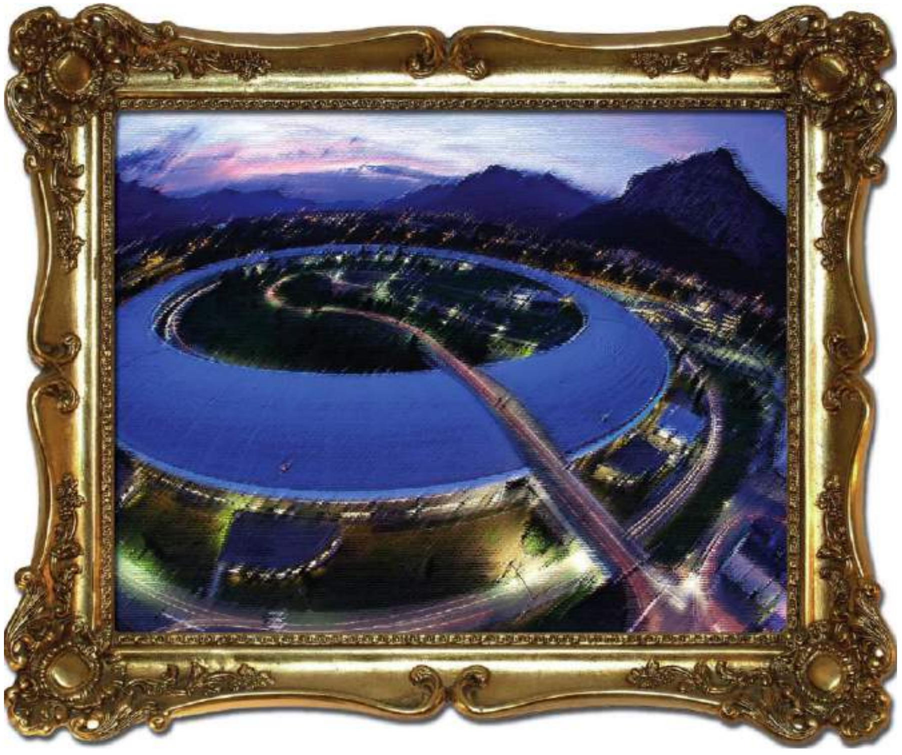
X-rays and Paleontology

X-rays and Food

X-rays and Biology

X-rays and Extreme Conditions

Applications in the field of cultural heritage

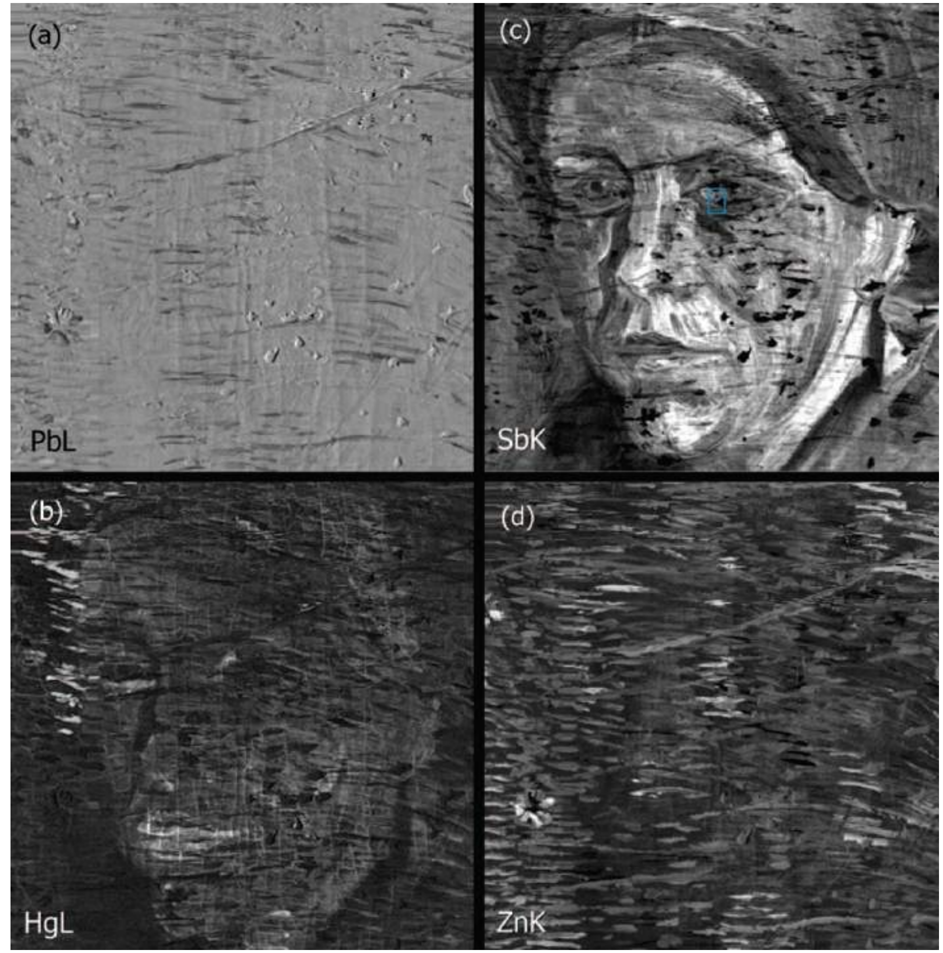


Visualization of a Lost Painting of van Gogh using XRF

***Vincent van Gogh*, Patch of Grass**, Paris 1887, Kroller-Muller Museum, Otterlo, The Netherlands, (KM 105.264; F583/JH1263).

Conventional X-Ray Radiation (XRR)transmission radiography

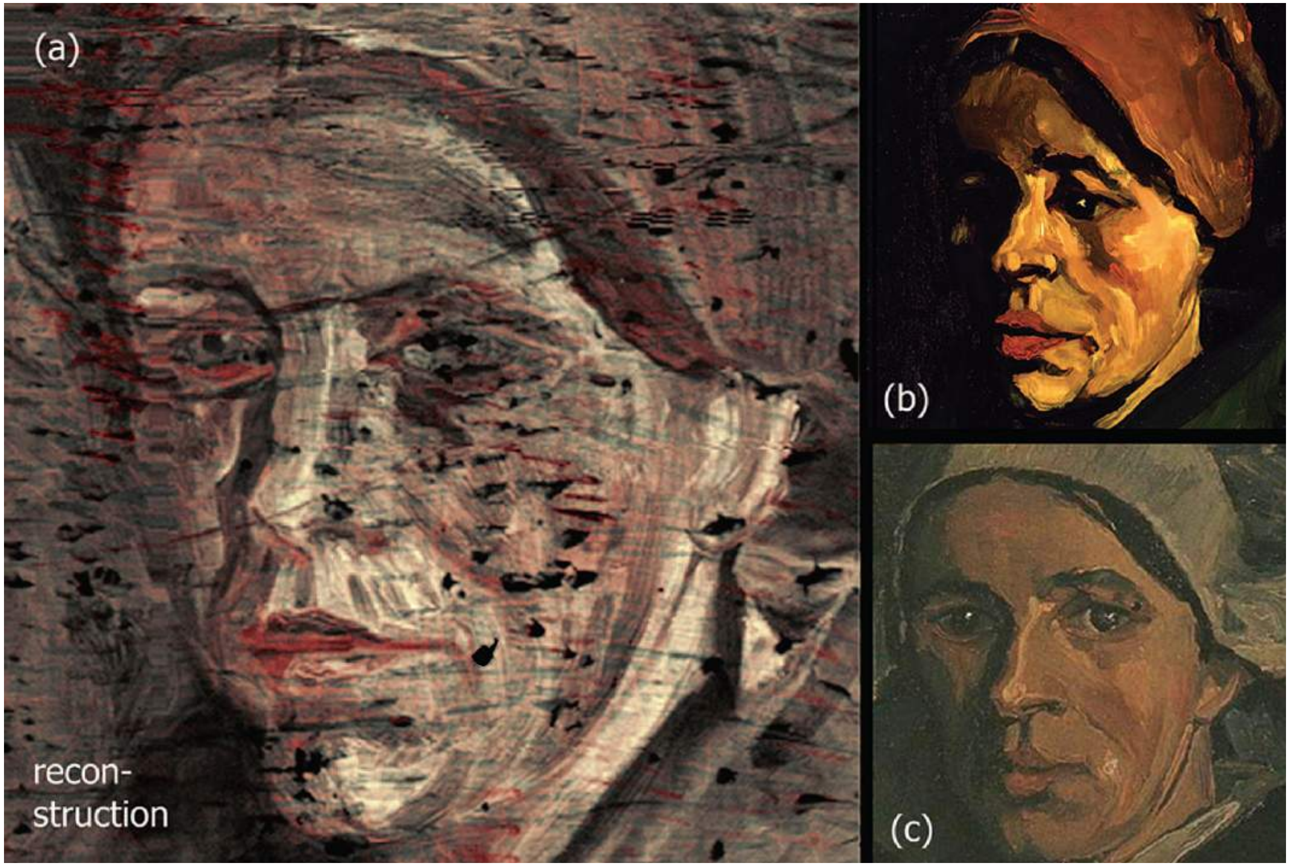
Infrared reflectography (IRR)



Synchrotron Radiation – XRF (black, low intensity and white, high intensity). Hg L shows the distribution of vermillion, Sb K of Naples yellow and Zn K of zinc white.

J. Dik et al., *Visualization of a Lost Painting by Vincent van Gogh Using Synchrotron Radiation Based X-ray Fluorescence Elemental Mapping*, **Anal. Chem. 2008**, **80**, 6436

Visualization of a Lost Painting of van Gogh



a) ***Tritonal color reconstruction*** of Sb (yellowish white) and Hg (red) (b) **Detail from Vincent van Gogh, Head of a Woman**, Nuenen 1884-85,Kro ller-Muller Museum, Otterlo (KM 105.591;F154/JH608). (c) **Detail from Vincent van Gogh, Head of a Woman**, Nuenen 1884-85, Van Gogh Museum, Amsterdam (F156/ JH569).

Vincent van Gogh (1853-1890), is best known for his vivid colors and his short but highly productive career. His productivity is even higher than generally realized, as many of his known paintings cover a previous composition. Van Gogh would often reuse the canvas of an abandoned painting and paint a new or modified composition on top. These hidden paintings offer a unique and intimate insight into the genesis of his works.

J. Dik et al., *Visualization of a Lost Painting by Vincent van Gogh Using Synchrotron Radiation Based X-ray Fluorescence Elemental Mapping*, **Anal. Chem. 2008**, **80**, 6436

Blackening of Pompeian Cinnabar paintings studied using X-rays

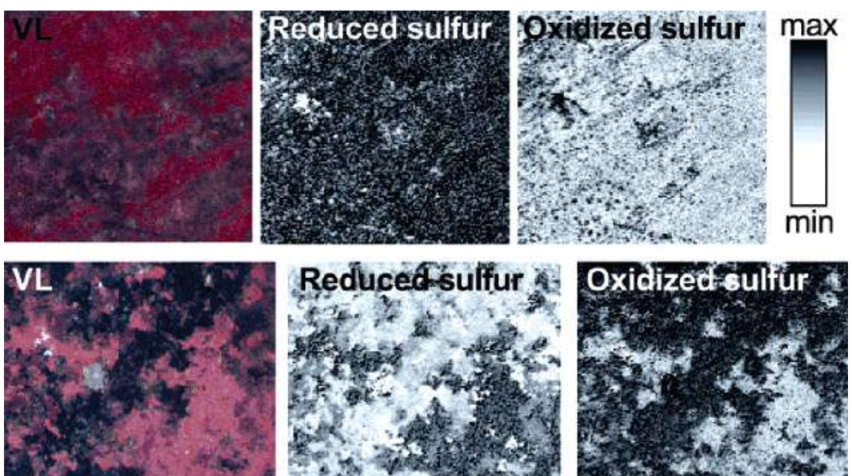
A wall painted red in the remains of Pompei: turning black

Painting alterations

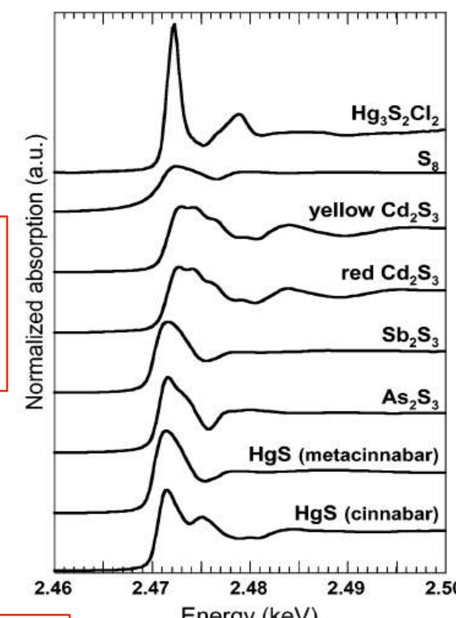
Scientists have ***wondered for many years why the red in Pompeii's walls***, a dye that is made from cinnabar (HgS), **turns black**.

The ESRF scientists found that the ***chemical composition in the affected samples*** was different from that of cinnabar, which indicated that **some important chemical reactions had taken place.**

On the one hand, ***cinnabar had reacted with chlorine***, which led to the formation of grey chlorine mercury compounds. The chlorine came from the sea and possibly punic wax (the wax that was used in the frescoes). ***Reduced and oxidized sulfur distributions reveal that the sulfated black coating consists of a 5-μm-thick layer covering intact cinnabar***.



X-ray absorption spectra of Sulfur-XANES reflecting the sulfur oxidation-state diversity.



M. Cotte, et al., Anal. Chem., 78, 7484-7492 (2006).

Archimedes Palimpsest

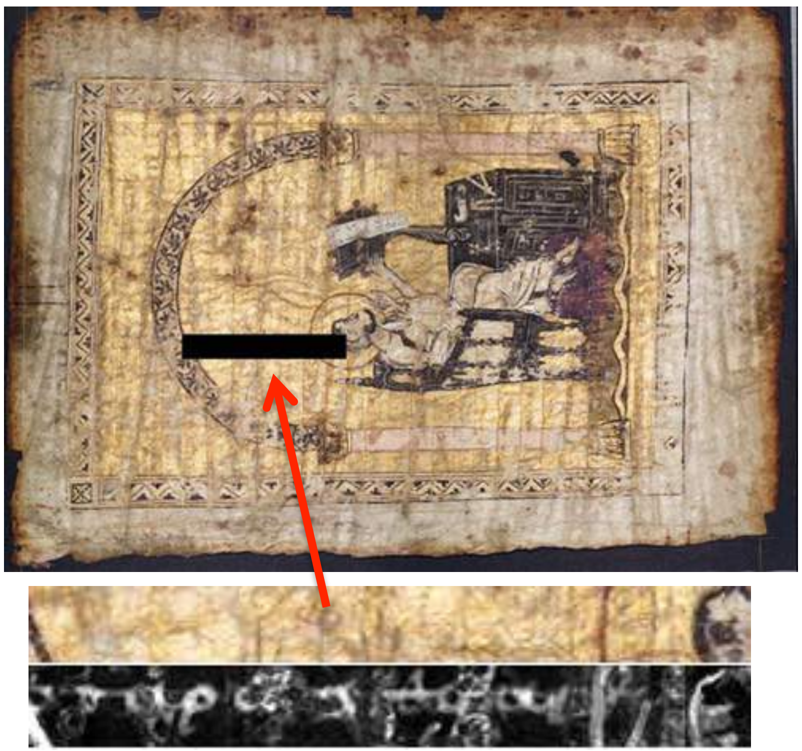
The Archimedes Palimpsest is a 1000-year-old manuscript on Parchment that contains seven treatises of the Greek mathematician, Archimedes.
Eight hundred years ago, the writings were erased and overwritten (parchments were very precious) becoming a Christian prayer book.
In 1998, the manuscript was purchased by a private collector (2.200.000 $).
Over the last ten years, a team of researchers has devoted a significant effort to recover the erased writing of Archimedes.

The book has suffered very severely from mold

http://archimedespalimpsest.org/

Archimedes Palimpsest

A synchrotron X-ray beam at the SSRL facility (Stanford USA) illuminated an erased work, written in **iron gall ink on parchment and obscured by gold paint.**



https://news.stanford.edu/news/2005/may25/archimedes-052505.html

U. Bergman, K. Knox, Pseudo-color enhanced X-ray fluorescence imaging of the Archimedes Palimpsest, SPIE-IS&T 7247 (2009) 724702

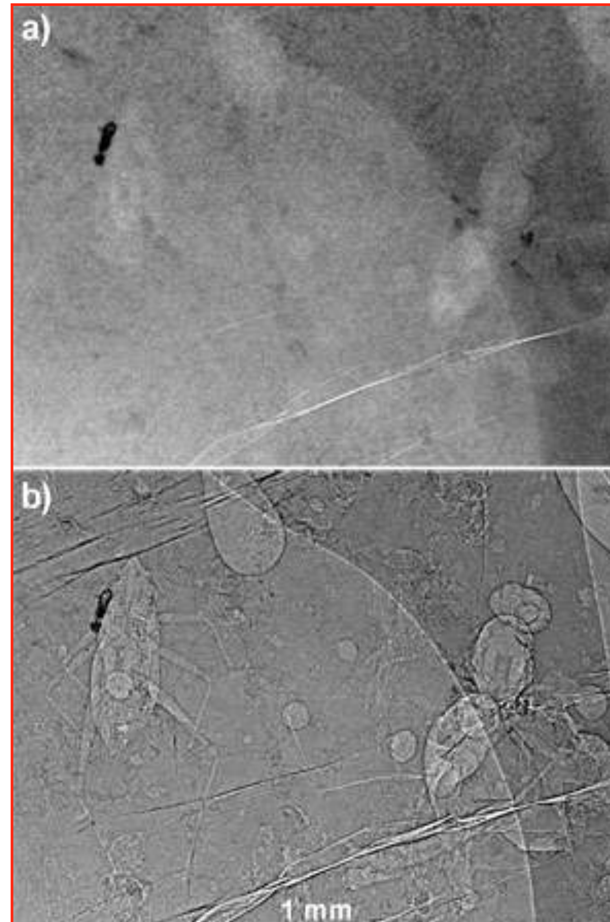
X-rays and Paleontology

Imaging and paleontology

Amber has always been a rich source of fossil evidence. X-rays now make it possible for paleontologists to ***study opaque amber, previously inaccessible using classical microscopy techniques***. Scientists from the University of Rennes (France) and the ESRF found ***356 animal inclusions, dating from 100 million years ago***, in two kilograms of opaque amber from ***mid-Cretaceous*** sites of Charentes (France).

Pieces of opaque amber-
Image credits: V. Girard/D. Néraudeau, UMR CNRS 6118 Rennes

Fossil inclusions in opaque amber



Radiography of an amber block with inclusions viewed in absorption mode.

Propagation phase contrast X-ray synchrotron imaging (990 mm prop. distance)

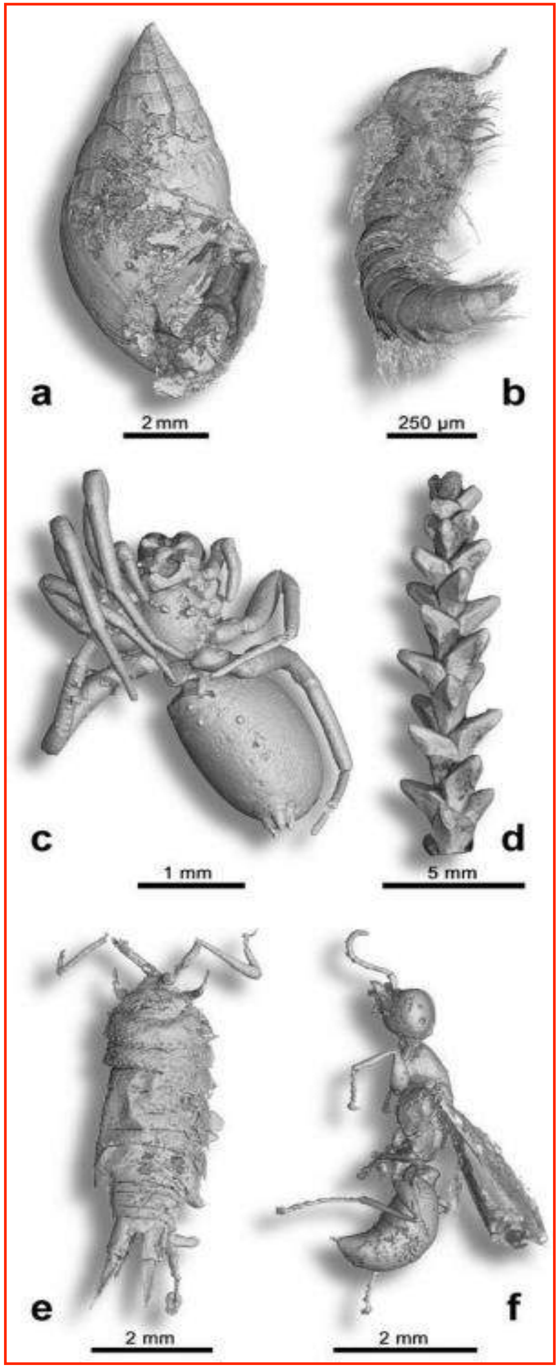
M. Lak, D. Neraudeau, A. Nel, P. Cloetens, V. Perrichot and P. Tafforeau, *Phase Contrast X-ray Synchrotron Imaging: Opening Access to Fossil Inclusions in Opaque Amber*, ***Microscopy and Microanalysis, (2008), 14:251-259***

Imaging and paleontology

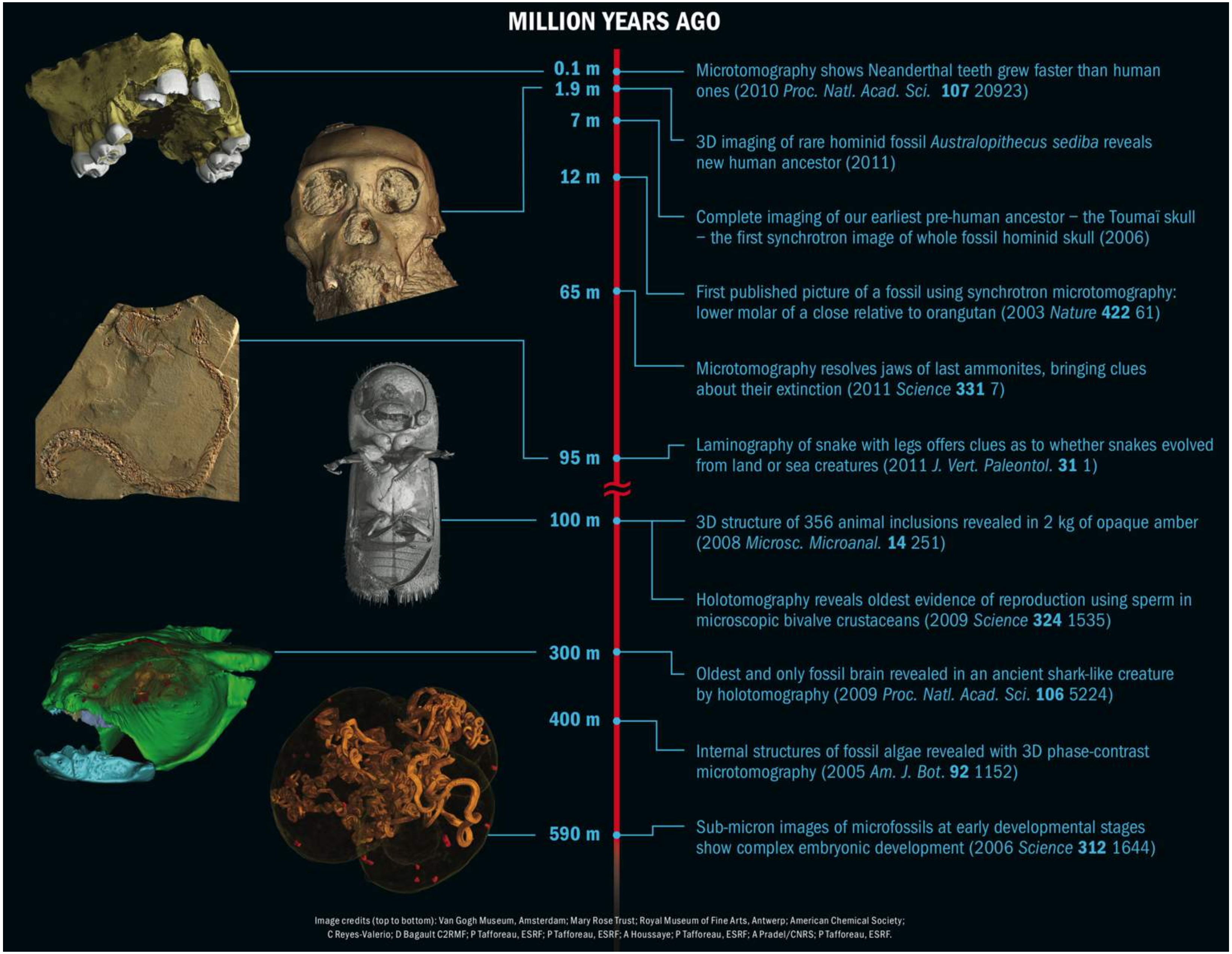
Synchrotron X-ray micro tomography was used to determine the 3D reconstruction and allowed the paleontologists to study the organisms in detail and to describe them.

Cretaceous beetle

Examples of virtual 3D extraction of organisms embedded in opaque amber: a) Gastropod Ellobiidae; b) Myriapod Polyxenidae; c) Arachnid; d) Conifer branch (Glenrosa); e) Isopod crustacean Ligia; f) Insect hymenopteran Falciformicidae.



M. Lak, D. Neraudeau, A. Nel, P. Cloetens, V. Perrichot and P. Tafforeau, Phase Contrast X-ray Synchrotron Imaging: Opening Access to Fossil Inclusions in Opaque Amber, Microscopy and Microanalysis, (2008)



Marine Cotte *- Synchrotron culture : Focus on: paleontology and cultural heritage* ***– ESRF News –June 2011***

X-rays and food

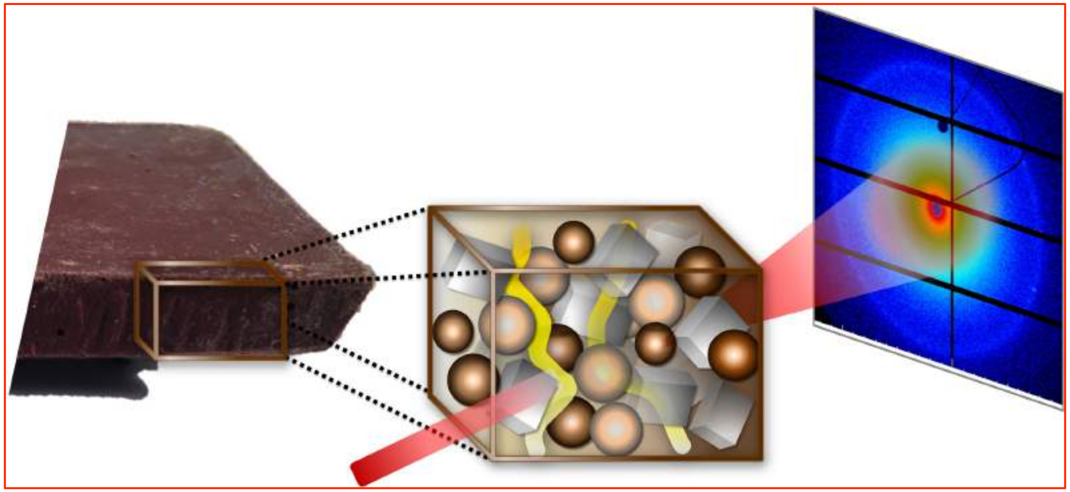
Structure of Chocolate

Most chocolate eaters will have had the surprising experience that a newly opened bar of chocolate (pure or milk) has a greyish-white layer instead of the familiar chocolate color.

A basic chocolate recipe consists of roughly **one-third cocoa butter**, a **fat that crystalizes easily**. **How the butter crystalizes determines the quality of the chocolate.**

http://www.esrf.eu/UsersAndScience/Publications/Highlights/2004/SCM/SCM8

Even though fat bloom does not actually constitute any deterioration in the quality of the product, the visual alteration associated with it can lead to a large number of consumer complaints.

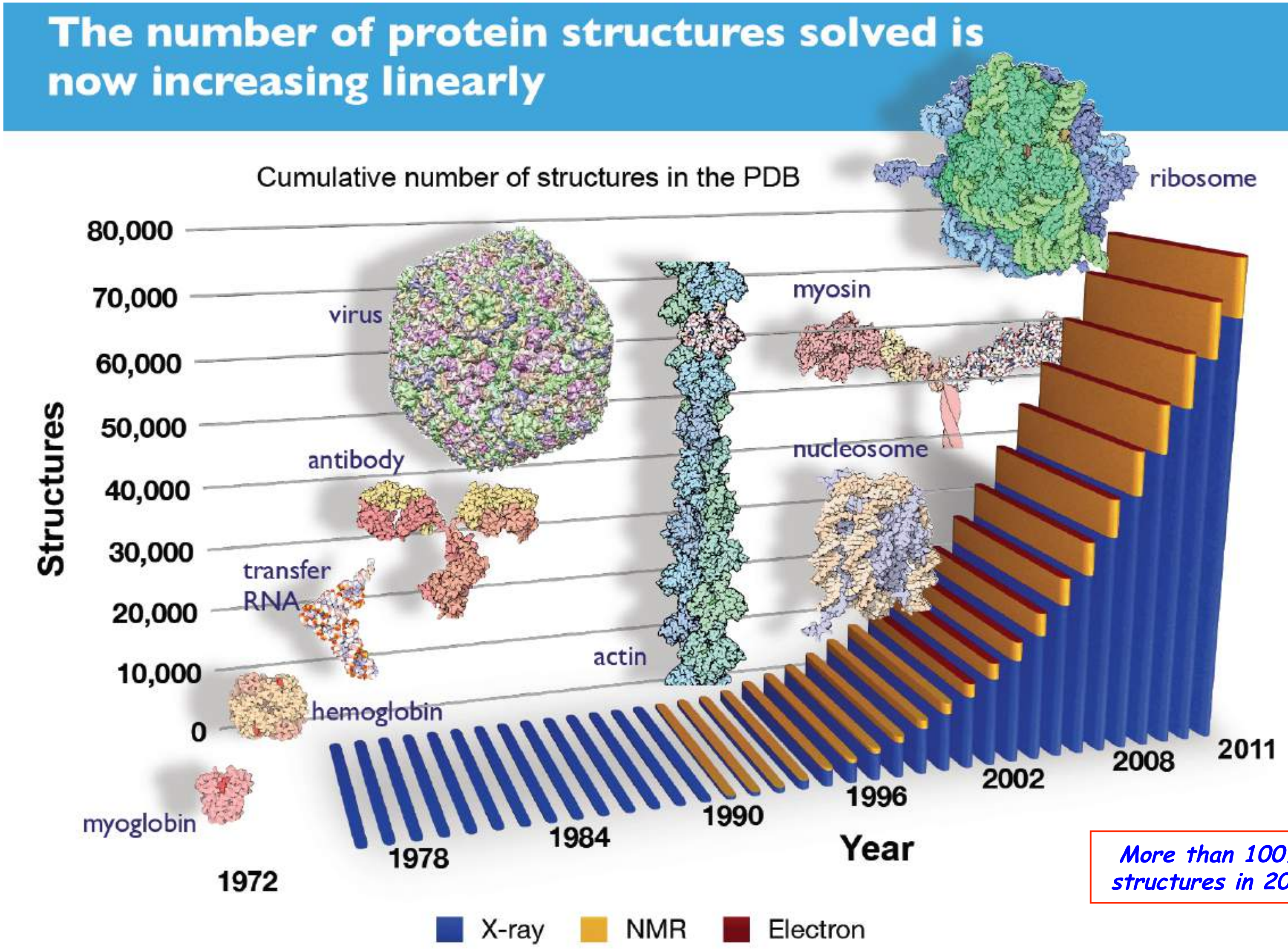


Cocoa butter crystallizes in six different crystal forms. The amount of fluid also depends on the form of the crystals.
Manufacturers can also limit fat bloom by controlling crystallization.

***Svenja K. Reinke et al.**, Tracking Structural Changes in Lipid-based Multicomponent Food Materials due to Oil Migration by Microfocus Small-Angle X-ray Scattering*, **ACS Appl. Mater. Interfaces 2015, 7, 9929**

Bio-crystallography

Bio-crystallography



More than 100.000 structures in 2014 !!!

H. Chapman – Lecture on Imaging Molecules with X-ray Free-Electron Lasers - 2012

Peso molecolare:
1kDa = 1000 g mol^{-1}

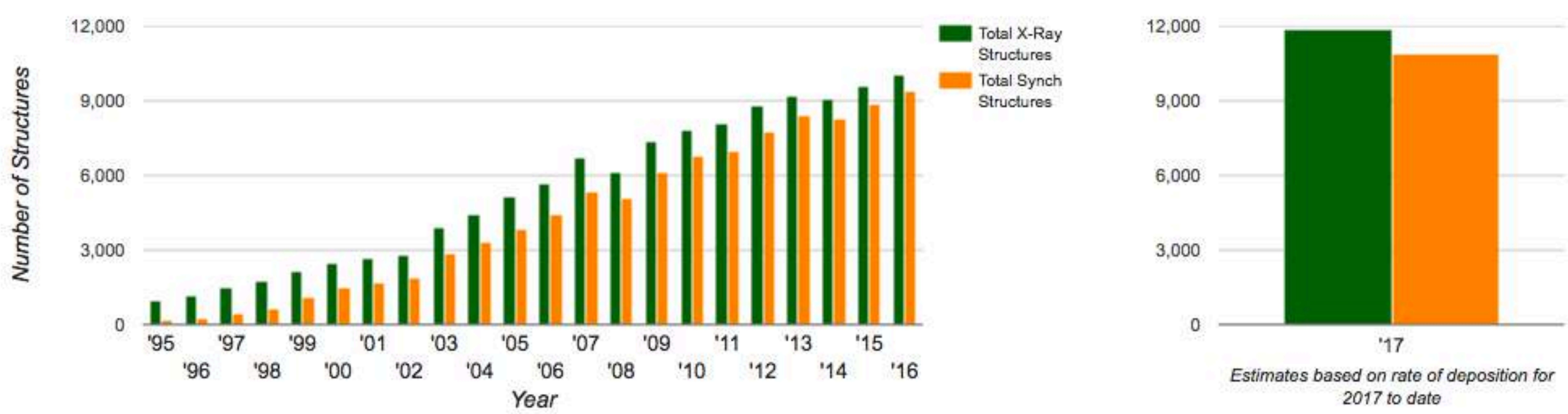
Myoglobin
16 kDa

50 YEARS

Ribosome 2.5 MDa

M. Bolognesi, Univ. Milano, Biologia strutturale, Conf. Luci di sincrotrone, CNR, 2014

X-ray bio-crystallography and synchrotron radiation

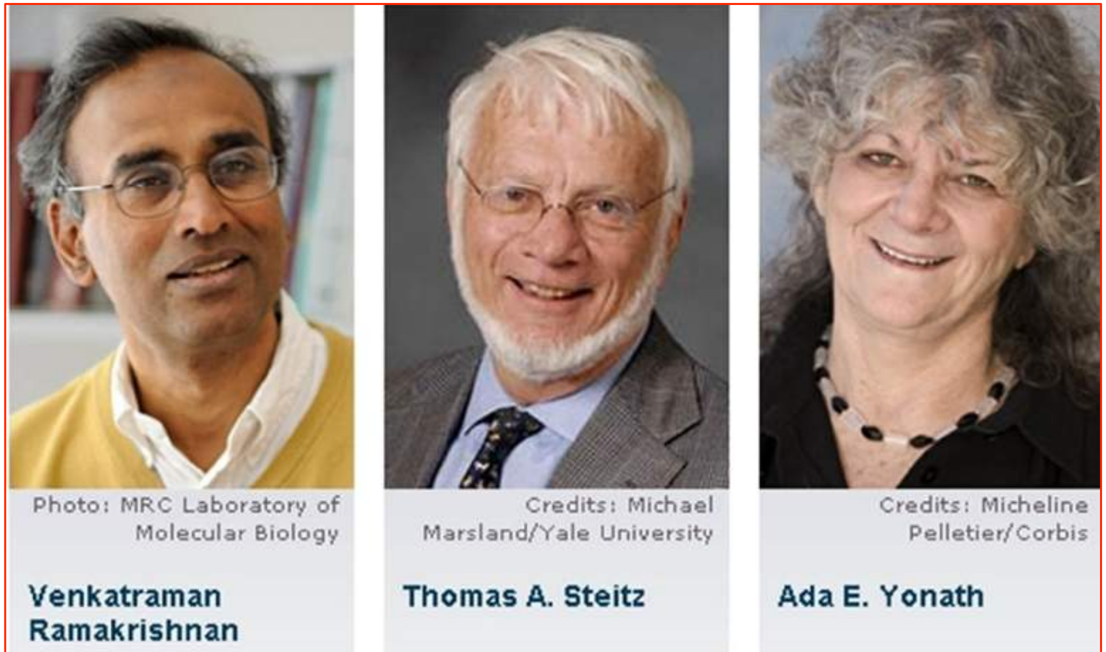


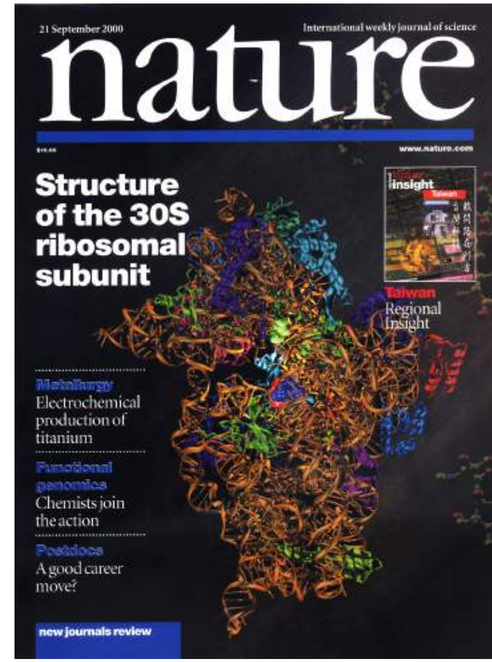
2016 http://biosync.sbkb.org/index.jsp

Very recent Noble Prizes in Chemistry

2009 "for studies of the structure and function of the ribosome"

Biocrystallography vs. Structural Biology





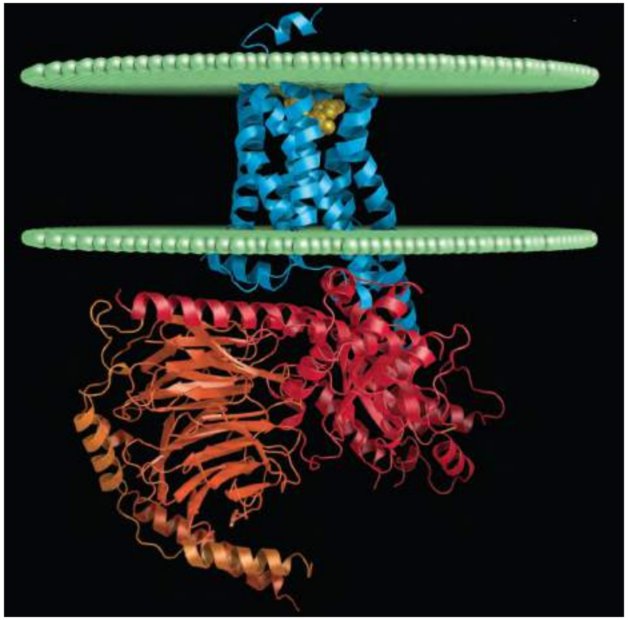
Using Synchrotron radiation Research

An understanding of the ribosome's innermost workings is important for a scientific understanding of life. This knowledge can be put to a practical and immediate use; **many of todays antibiotics cure various diseases by blocking the function of bacterial ribosomes**. Without functional ribosomes, bacteria cannot survive. This is why **ribosomes are such an important target for new and more efficient antibiotics**.

Very recent Noble Prizes in Chemistry

2012 "for studies of G-protein-coupled receptors"

Biocrystallography vs. Structural Biology



Using Synchrotron radiation Research

G-Protein Coupled Receptor (blue) sits within lipid bilayer (green) to respond to hormone (yellow)- **Image by Wayne Decatur** - http://www.hhmi.org/bulletin/winter2013/features/index.html

G protein coupled receptors (GPCRs) represent ***the largest family of membrane proteins*** (about 800 different proteins) ***controlling body functions***, ***drug transit across membranes*** and representing the richest source of targets for the pharmaceutical industry.

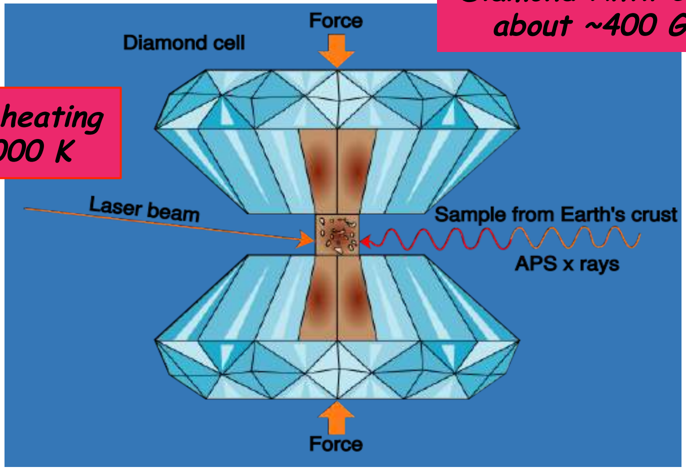
X-rays and extreme conditions

X-rays and extreme conditions (P,T): new opportunities

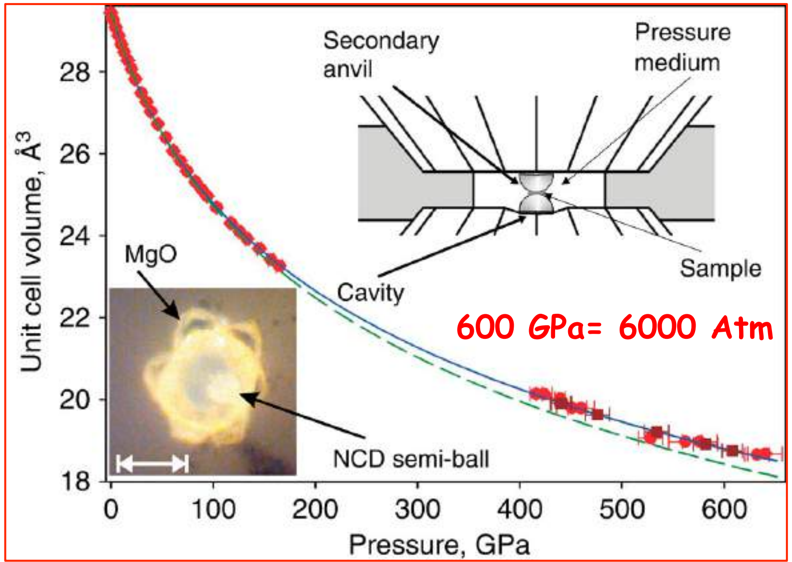
P. W. Bridgman (Nobel Prize in Physics in 1946 – discoveries made in the field of high pressure physics)- ***"Compression offers a route to breaking down the electronic structure of the atoms themselves and to the possibility of creating entirely different bulk properties".***

Diamond Anvil Cell (DAC) about ~400 GPa, RT

Laser heating ~ 7000 K



100GPa=1Mbar



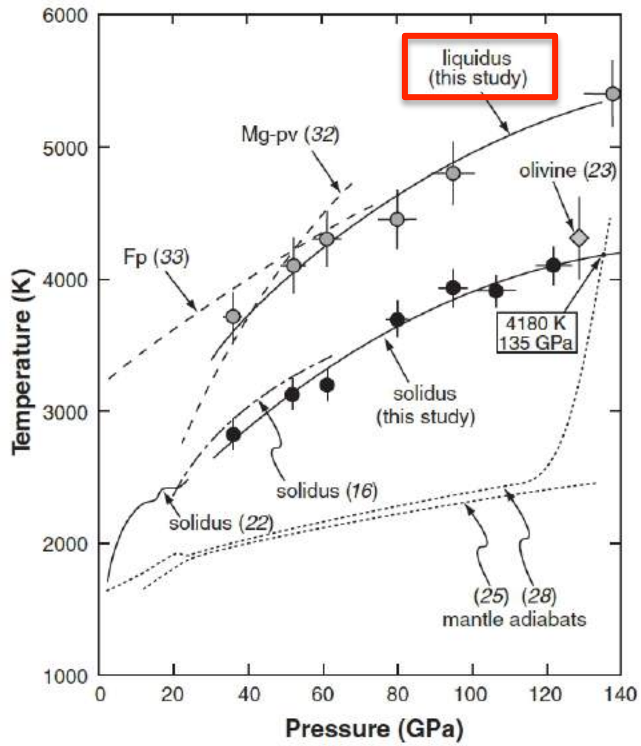
New system to increase Pressure

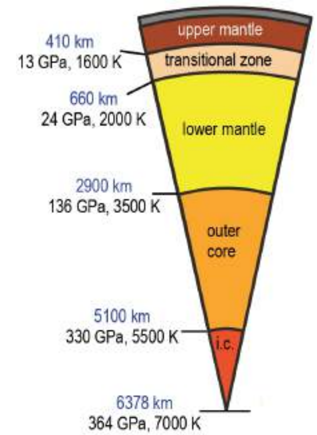
L. Dubrovinsky et al. *Implementation of micro-ball nano-diamond anvils for high-pressure studies above 6 Mbar* - **Nature Comm. (2012)**

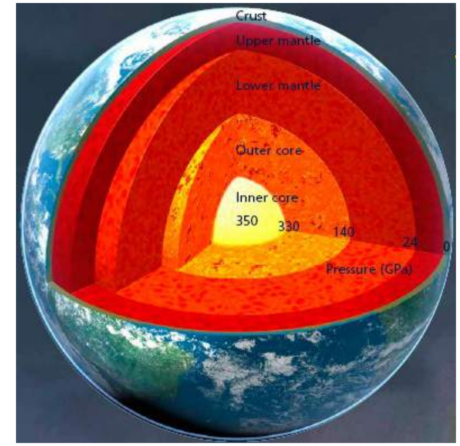
Application fields:
- **Earth and Planetary Science**
- **Condensed Matter Physics**
- **Chemistry and Materials Science**
- **Biology and Soft Matter**

- **Novel transformations: solids, liquids, glasses**
- **Structures: unexpected complexity**
- **Molecules break down, but new ones form**
- **Novel electronic and magnetic phenomena**
- **New chemical reactions: low to high pressure**
- **New recoverable materials**

Extreme conditions (P,T)



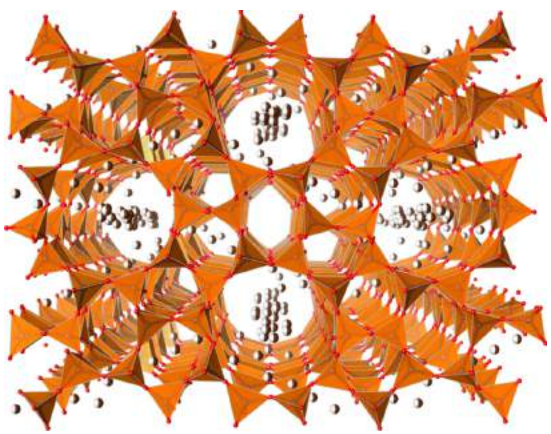




Melting of Peridotite (Olivine and iron-magnesium silicates)

First direct evidence (ESRF ID27) that the ***layer located at the bottom of the Earth's mantle*** (2900 km depth) ***contains partially molten minerals.*** This result supports the existence of a deep magmatic ocean. (**P: 36 –140 GPa T:2500-5000 K**)

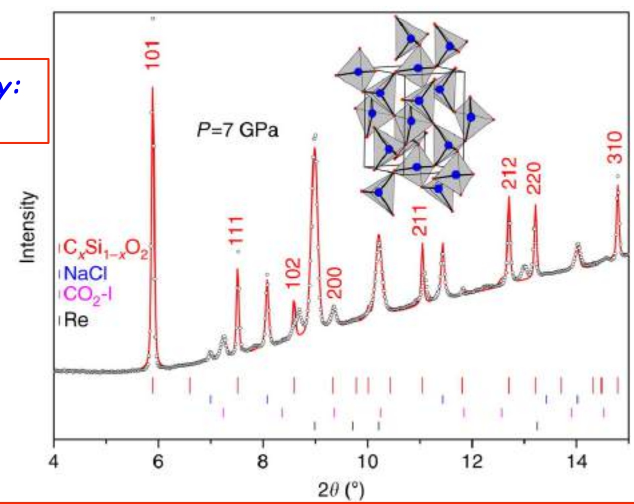
G. Fiquet, et al. *Melting of Peridotite to 140 GPa* **Science (2010)**



New materials: conducting polymers

D. Scelta et al. *High Pressure Polymerization in a Confined Space: Conjugated Chain/Zeolite Nanocomposites.* ***Chem. Mater. (2014)***

New oxide chemisty:
$C_{0.6(1)}Si_{0.4(1)}O_2$



M. Santoro et al. *Carbon enters silica forming a cristobalite-type CO_2–SiO_2 solid solution,* ***Nature Comm. (2014)***

Conclusions

- Synchrotron radiation has surely revolutionized X-ray applications.
- Most of the SR facilities in the world have beamlines dedicated to different X-ray applications.
- Synchrotron radiation X-ray applications still have a very bright future.

Thank you for your attention

Supplementary material- f.y.k.

Reminder

- *Speed of light* — $c = 2.99792458 \times 10^8$ m/s
- *Electron charge* — $e = 1.6021 \times 10^{-19}$ Coulombs
- *Electron volts* — $1\ eV = 1.6021 \times 10^{-19}$ Joule
- *Energy and rest mass* — $1 eV/c^2 = 1.78 \times 10^{-36}$ kg

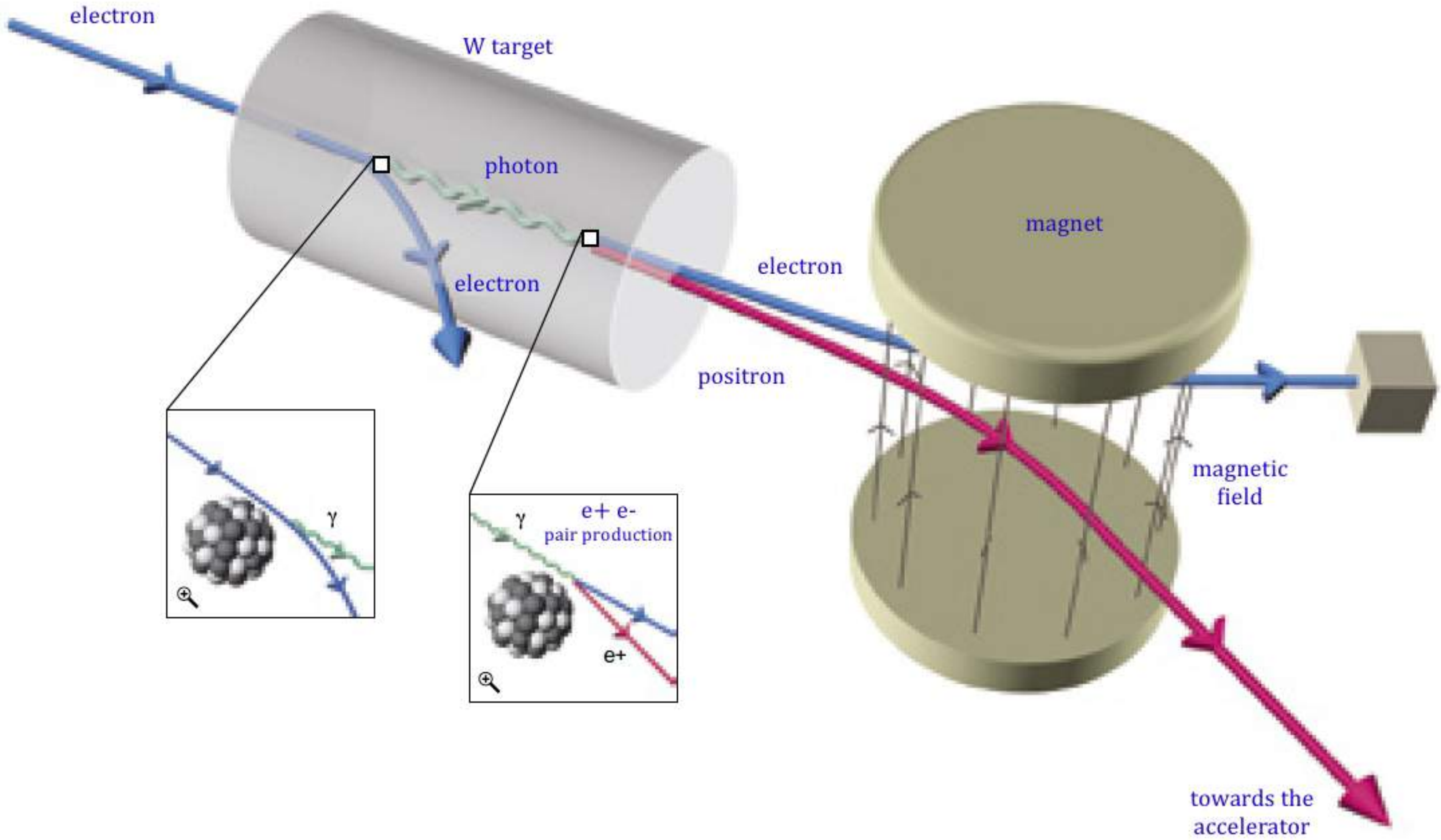
  Electron — $m_0 = 511.0\ keV/c^2 = 9.109 \times 10^{-31}$ kg

  Proton — $m_0 = 938.3\ MeV/c^2 = 1.673 \times 10^{-27}$ kg
- *Relativistic energy, E* — $E = mc^2 = m_0 \gamma c^2$
- *Lorentz factor,* γ — $\gamma = 1/[(1-v^2/c^2)^{1/2}] = 1/[(1-\beta^2)^{1/2}]$

  $\beta = v/c$
- *Relativistic momentum, p* — $p = mv = m_0 \gamma \beta c$
- *E-p relationship* — $E^2/c^2 = p^2 + m_0 c^2$

  for ultra-relativistic particles — $\beta \approx 1$, $E = pc$
- *Kinetic energy* — $T = E - m_0 c^2 = m_0 c^2 (\gamma - 1)$

Anti-matter positron production



M. Calvetti, *Antiparticelle accelerate*, ***Asimmetrie*** 7, 16-21 (***2008***)

X-rays discovery


EINE NEUE ART

VON

STRAHLEN.

VON

DR. W. RÖNTGEN,

O. O. PROFESSOR AN DER K. UNIVERSITÄT WÜRZBURG.

WÜRZBURG.

VERLAG UND DRUCK DER STAHEL'SCHEN K. HOF- UND UNIVERSITÄTS-BUCH- UND KUNSTHANDLUNG.

Ende 1895.

60 ₰.

On a New Kind of Rays

While ***Wilhelm Roentgen*** was working on the effects of ***cathode rays*** during ***1895***, he discovered X-rays. His experiments involved the passing of electric current through gases at extremely low pressure. On **November 8, 1895** he observed that certain rays were emitted during the passing of the current through discharge tube. His experiment that involved working in a totally dark room with a well covered discharge tube resulted in the **emission of rays which illuminated a barium platinocyanide screen**. ***The screen became fluorescent even though it was placed two meters away from discharge tube***.

Wilhelm Conrad Roentgen

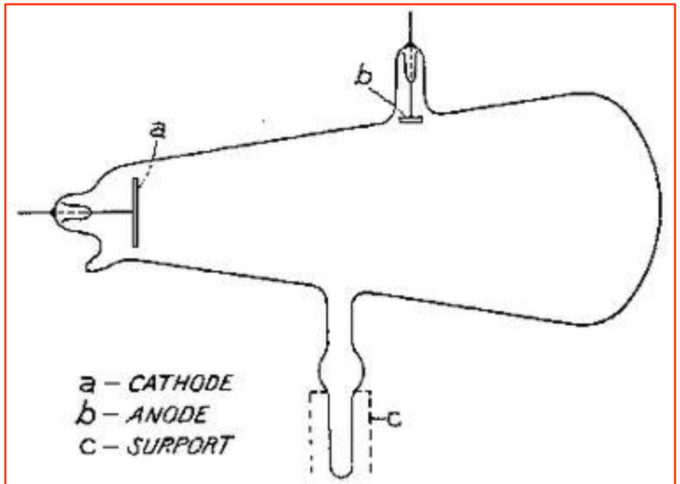


Fig. 1. Earliest type of roentgen tube.

Gas tube: electrons are freed from a **cold cathode by positive ion bombardment, thus necessitating a certain gas pressure.**

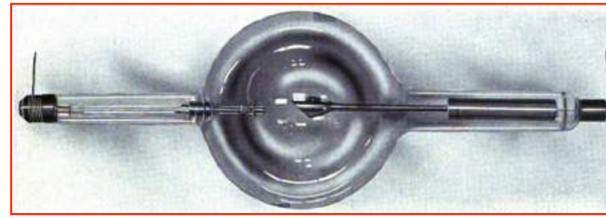
He continued his experiments using **photographic plates** and generated the very ***first*** "***roentgenogram***" by developing the image of ***his wife's hand*** and analyzed the variable transparency as showed by her bones, flesh and her wedding ring.

X-ray tubes

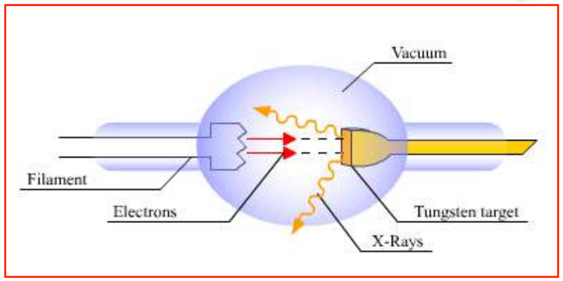
X-rays: conventional sources

From **gas tubes** (cold cathode) to **high vacuum tubes** (hot cathode)

Crookes tube

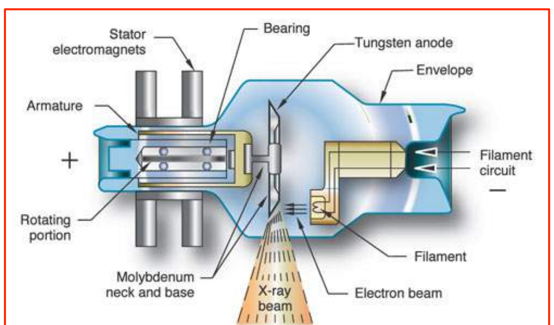


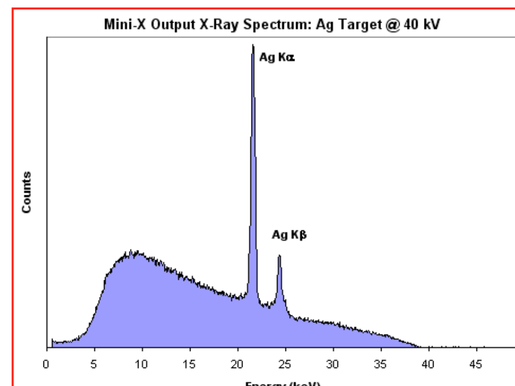
Coolidge tube



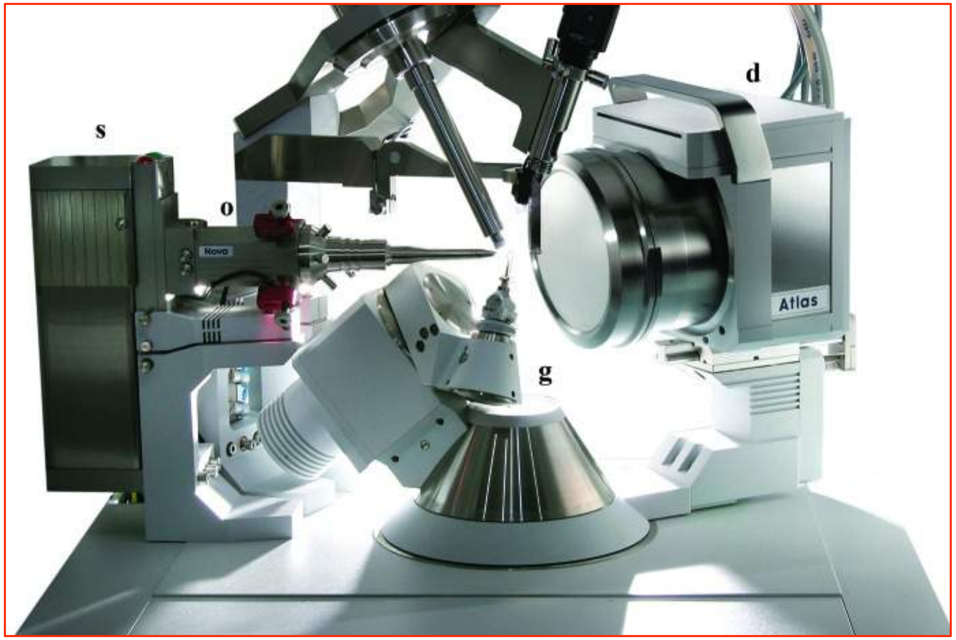
The ***Coolidge tube (1913)***, also called ***hot cathode tube***, is the most widely used. Electrons are produced by thermionic effect from a tungsten filament heated by an electric current. The filament is the cathode of the tube. The high voltage potential is between the cathode and the anode, the electrons are accelerated, and hit the anode.

The ***rotating anode tube is an improvement of the Coolidge tube*** anode surface (water cooled) is always moving, so heat is spread over a much larger surface area giving a 10-fold increase in the operating power.





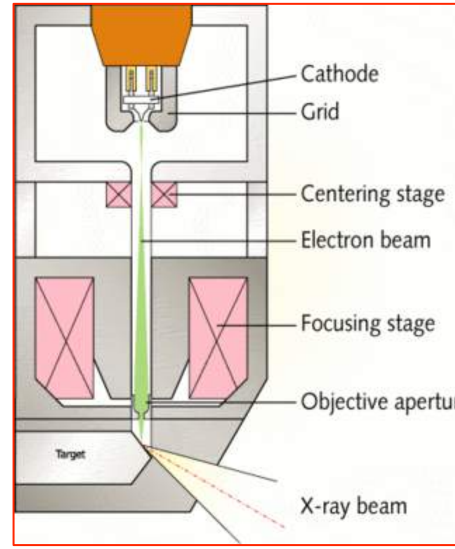
X-rays: conventional sources



EVOLUTION

A compact X-ray in-house laboratory system consisting of ***a microfocus sealed-tube source*** (s), focusing multi-layer optics (o), a four-circle goniometer (g) and a CCD detector (d).

Pictures of ***Agilent Technologies.***



Approximate X-ray beam brilliance for the main types of in-house sources with optics

System	Power (W)	Actual spot on anode (μm)	Apparent spot on anode (μm)	Brilliance (photons s^{-1} mm^{-2} $mrad^{-1}$)
Standard sealed tube	2000	10000 × 1000	1000 × 1000	0.1×10^9
Standard rotating-anode generator	3000	3000 × 300	300 × 300	0.6×10^9
Microfocus sealed tube	50	150 × 30	30 × 30	2.0×10^9
Microfocus rotating-anode generator	1200	700 × 70	70 × 70	6.0×10^9
State-of-the-art microfocus rotating-anode generator	2500	800 × 80	80 × 80	12×10^9
Excillum JXS-D1-200	200	20 × 20	20 × 20	26×10^9

*T. **Skarzynski**, Collecting data in the home laboratory: evolution of X-ray sources, detectors and working practices, **Acta Cryst**.* **D69 (2013) 1283–1288**

Synchrotron radiation

Synchrotron radiation: physics

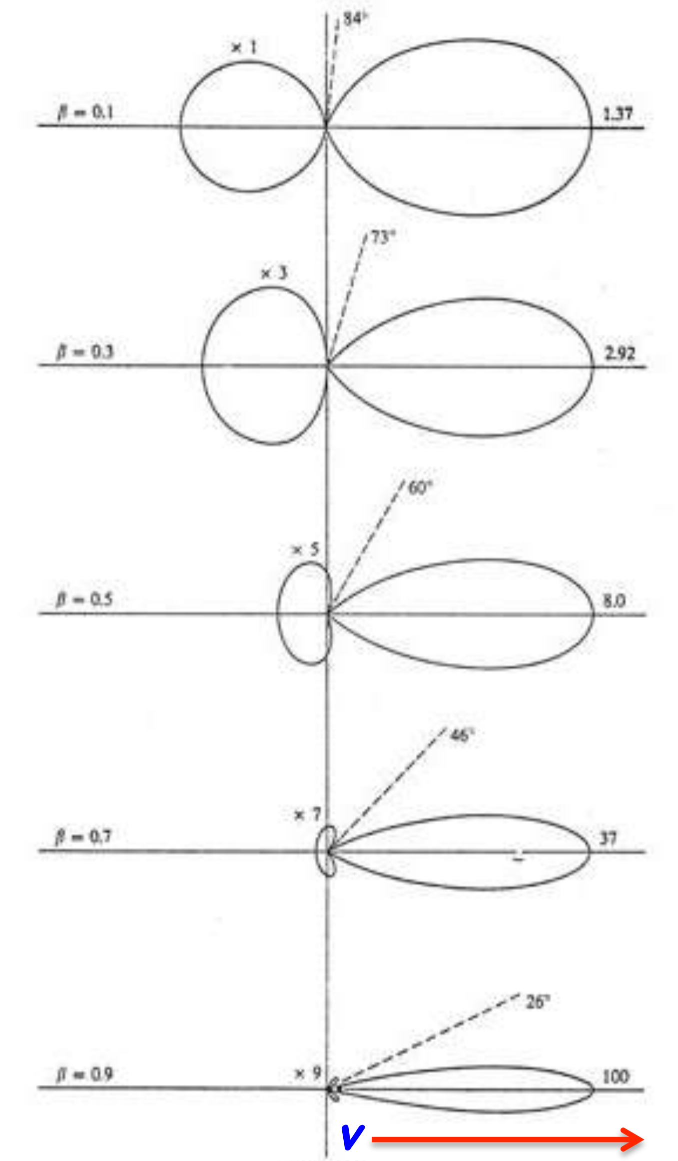


FIG. 7-7

v << c or $\beta = v/c << 1$

As β approaches 1:

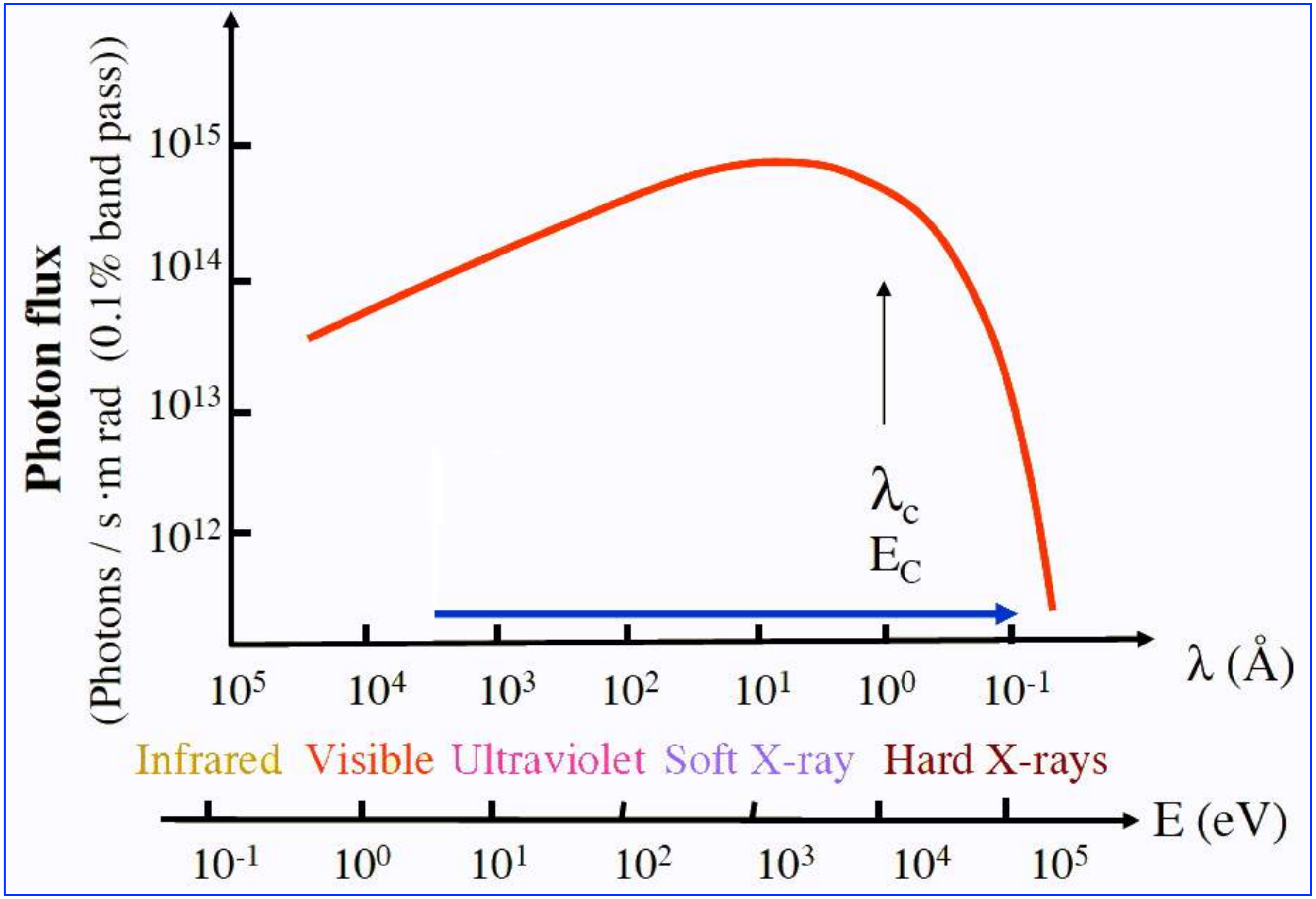
1) The shape of the radiation pattern changes: it is more in the forward direction!
2) the node at θ' = 90° in the frame of the radiating particle transforms to:

$$\tan\theta_{lab} = \frac{\sin\theta'}{\gamma\left(\cos\theta' + \beta\right)} = \frac{1}{\gamma\beta} \approx \frac{1}{\gamma}$$

$$\gamma^{-1} \approx mc^2/E$$

v ≈ c or $\beta = v/c \approx 1$

Spectral distribution: universal synchrotron radiation function



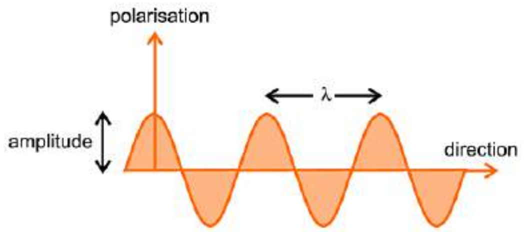
E_c and λ_c respectively critical energy and critical wavelength

$$E_c[keV] = 2.218\frac{E[GeV]^3}{\rho[m]} = 0.665 \cdot E[GeV]^2 \cdot B[T]$$

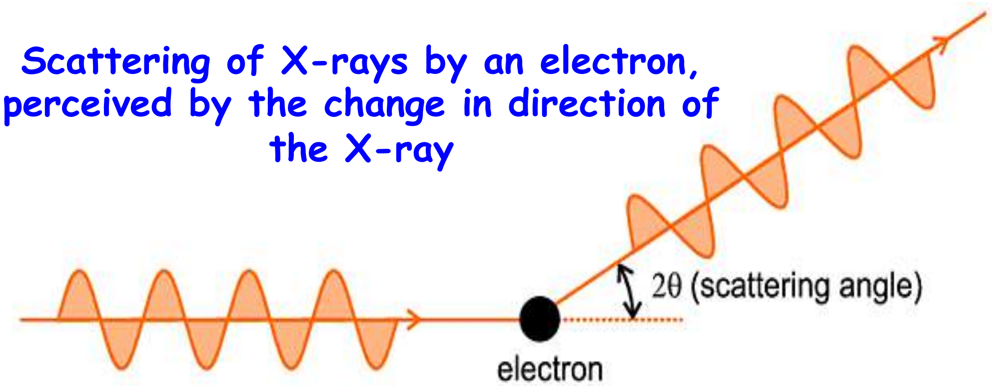
Elastic Scattering and Diffraction

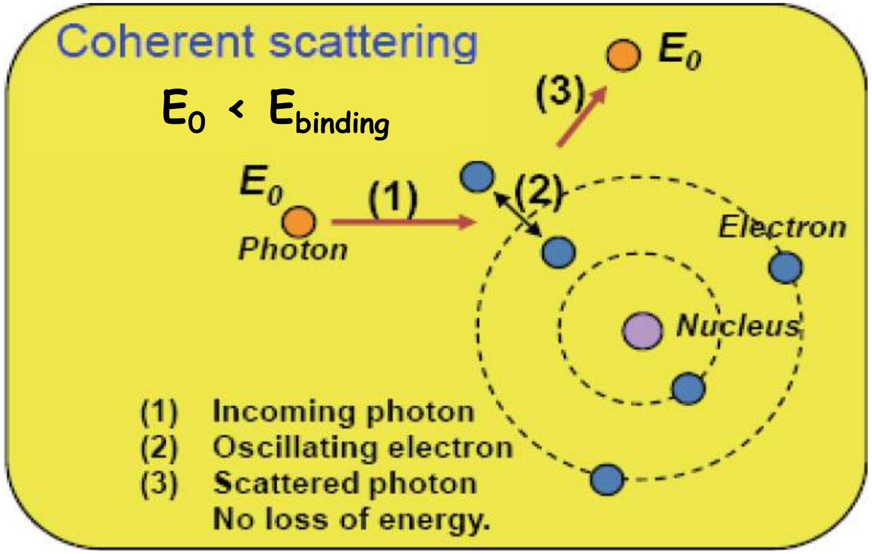
Elastic or coherent Scattering

Incoming X-rays

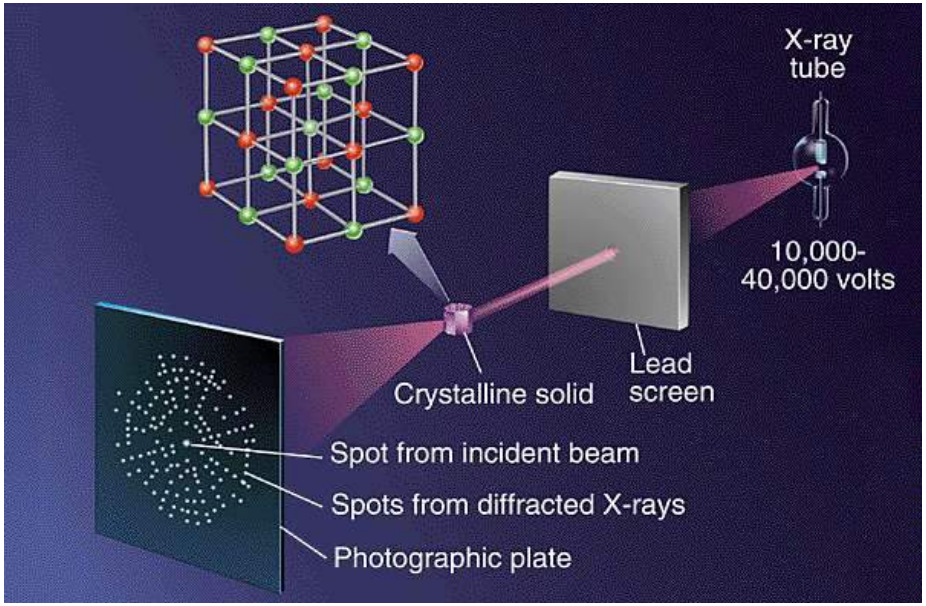


Scattering of X-rays by an electron, perceived by the change in direction of the X-ray





Scattering of X-rays by the electrons of the atoms.

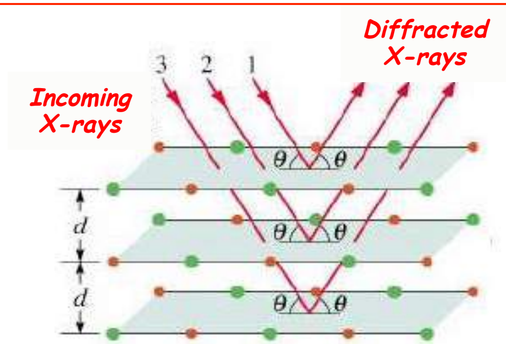


Scattering and diffraction of X-rays by the electrons of the atoms of a crystalline solid.

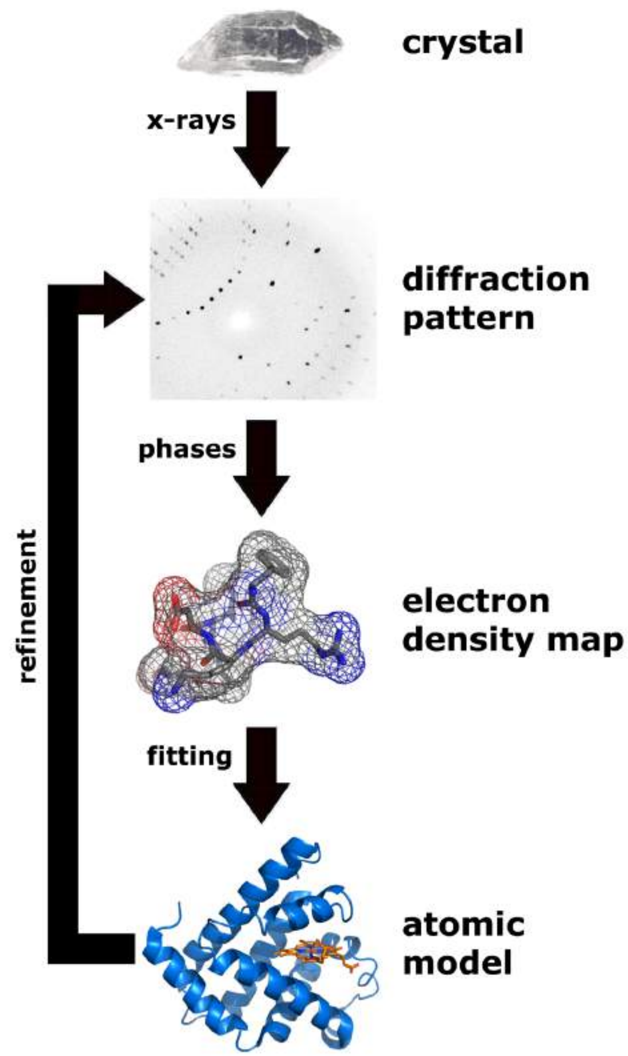
Elastic scattering and diffraction

X-ray diffraction is an important tool used to identify ***phases by comparison with data from known structures***, ***quantify changes in the cell parameters***, ***orientation***, ***crystallite size and other structural parameters***. It is also used to determine the (crystallographic) structure (i.e. cell parameters, space group and atomic coordinates) of novel or unknown crystalline materials.

The ***interference pattern of X-rays scattered by crystals*** (XRD or X Ray Diffraction pattern) can be ***used study the atomic structure of interest***. ***Bragg's law explains the relation between***: ***d***, the ***distance between atomic layers*** in a crystal, λ is ***the wavelength of the incident X-ray beam*** and θ ***the angle of incidence*** at which the faces of ***crystals appear to reflect X-ray beams.***

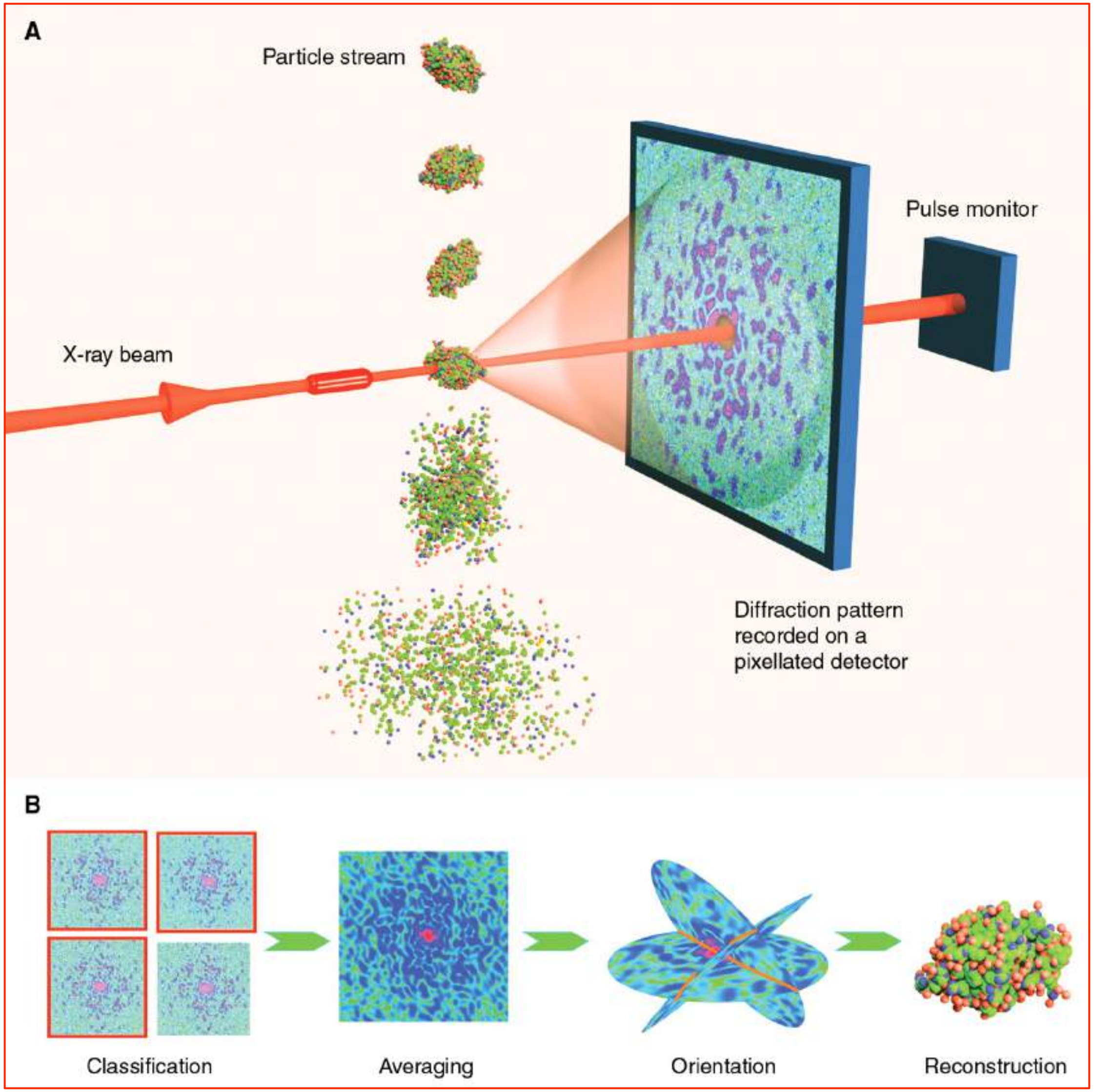


Bragg's Law: $n\lambda = 2d \sin\theta$



Structure determination by X-ray crystallography - Thomas Splettstoesser (www.scistyle.com)
https://commons.wikimedia.org/w/index.php?curid=1248574

Scattering and imaging



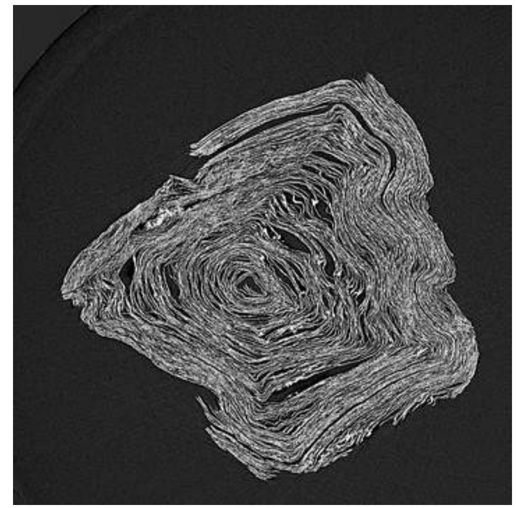
K. J. Gaffney and H. N. Chapman - Imaging Atomic Structure and Dynamics with Ultrafast X-ray Scattering
Science DOI: 10.1126/science.1135923

Revealing letters in rolled Herculaneum papyri using X-ray phase-contrast tomography

Close up photography of ***Herculaneum Papyrus*** scroll PHerc.Paris.4. The photographied zone is 5cm. (*Credit: E. Brun*)

Hundreds of papyrus rolls, buried by the eruption of Mount Vesuvius in 79 AD and belonging to the only library passed on from Antiquity, were discovered 260 years ago at Herculaneum.

These carbonized papyri are extremely fragile and are inevitably damaged or destroyed in the process of trying to open them to read their contents.



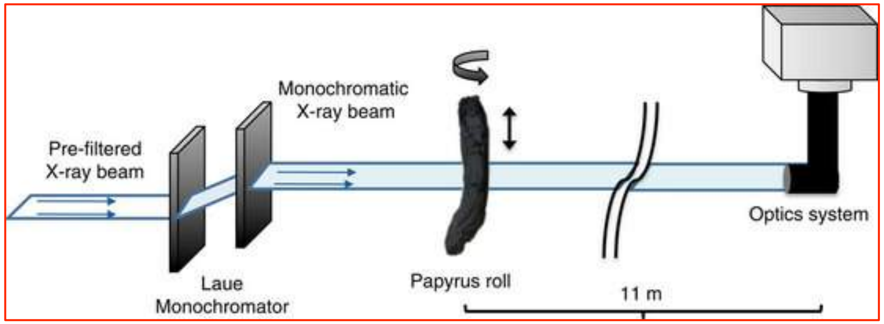
A section of papyrus. Letter sequences are found in a fragment of a hidden layer. (*Credit: CNRS-IRHT UPR 841 / **ESRF** / CNR-IMM Unité de Naples*)

V. Mocella *et al.*, **Nature Communications** - DOI: 10.1038/ncomms6895- ***January 2015***

https://www.youtube.com/watch?v=d3aWBgNYOCU

Revealing letters in rolled Herculaneum papyri.

In recent years, new imaging techniques have been developed to read the texts without unwrapping the rolls. ***Until now, specialists have been unable to view the carbon-based ink of these papyri***, even when they could penetrate the different layers of their spiral structure.
For the first time ***X-ray phase-contrast tomography*** (*beamline ID17 of the ESRF, Grenoble, France*) can ***reveal various letters hidden inside the precious papyri without unrolling them***.
This attempt opens up new opportunities to read many Herculaneum papyri, which are still rolled up, thus enhancing our knowledge of ancient Greek literature and philosophy.



The ***papyrus alphabet from PHerc.Paris.4. as revealed by the XPCT experiment***, primarily from the ***innermost region of the papyrus***, where the individual coils are more distinguishable, are reported on lines 1 and 2; on ***line 3 the infrared images of the same letters from the unrolled (opened) papyrus PHerc. 1471***, which was used as a reference for the writing style of the scroll PHerc.Paris. 4.

V. Mocella *et al.*, **Nature Communications** - DOI: 10.1038/ncomms6895- ***January 2015***