Schweizerische Gesellschaft für Kristallographie
Société Suisse de Cristallographie
Società Svizzera di Cristallografia
Swiss Society for Crystallography

$$f(SGK) = \int_{\infty}^{\infty} e^{-iSGKtCH}\,\mathrm{d}CH$$

From the early years to large scale facilities

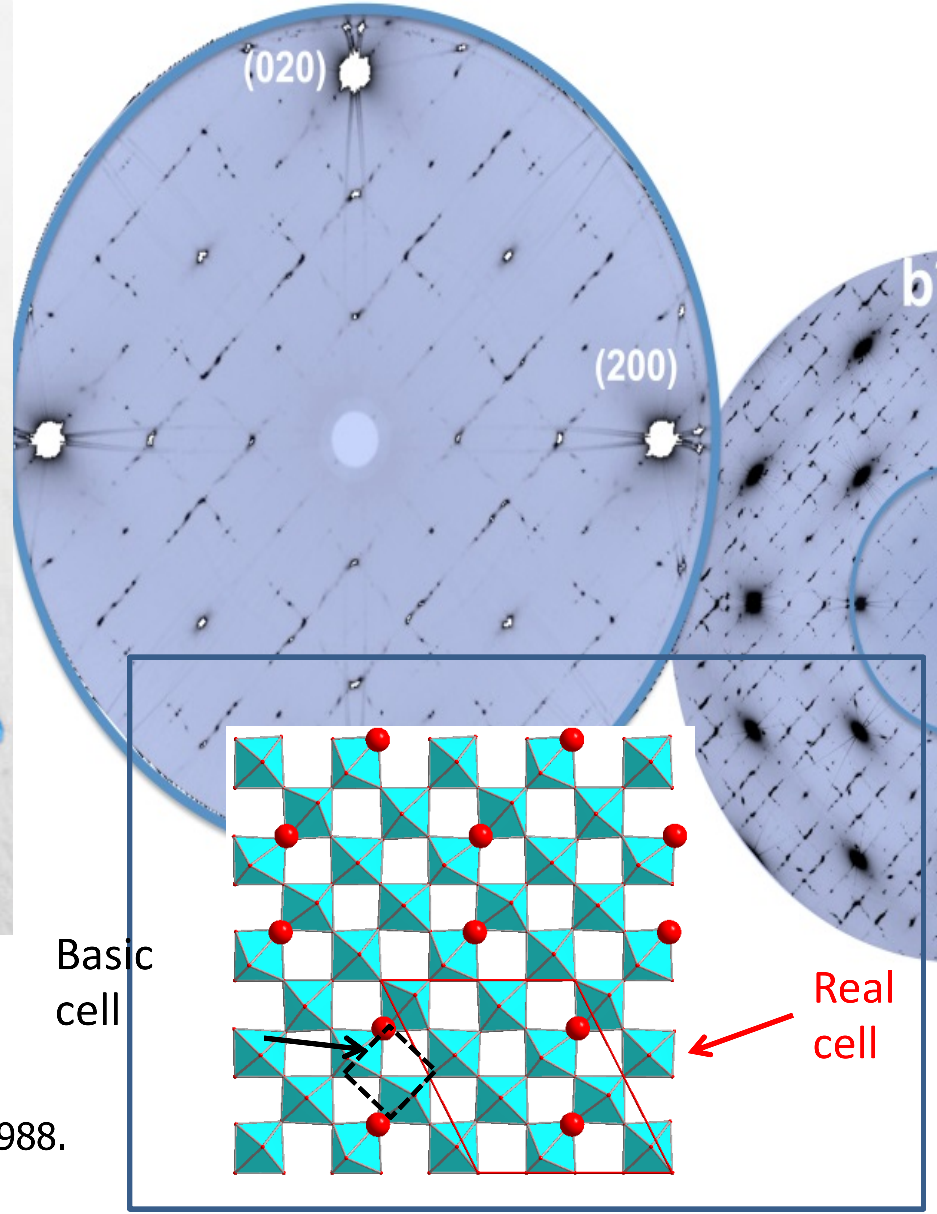
- **Early days of crystallography**
- **Today**
  focus on the large scale facilities
  SLS – SINQ – SNBL@ESRF
- **Year of Crystallography**
- **outlook**

Jürg Schefer
Laboratory for Neutron Scattering and Imaging
LNS, Paul Scherrer Institut

Jurg.Schefer@psi.ch

1912

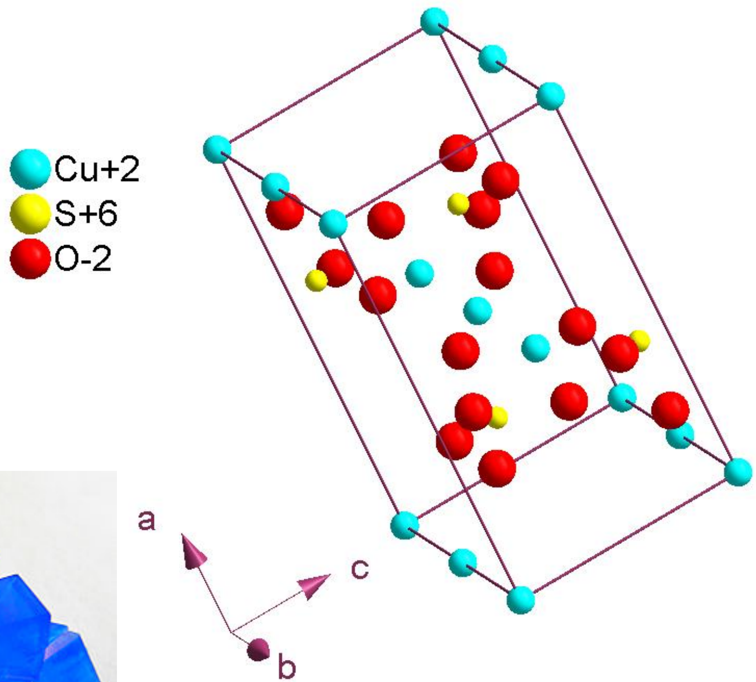
2011

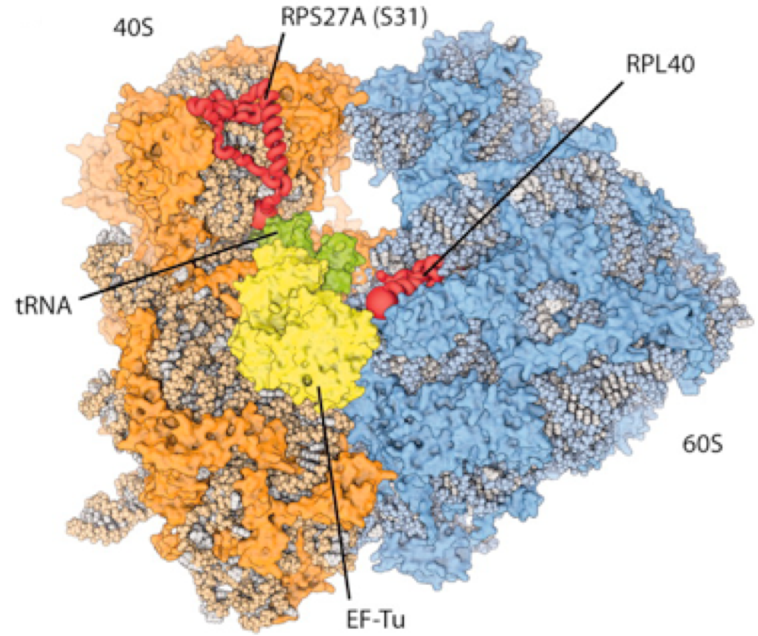


Proceedings of the Bavarian Academy of Sciences
(Proc. Bayer. Akad. Wiss.) (1912) 303-322;
Reprinted in: Annalen der Physik **41** (1913) 971-988.

1912

2011



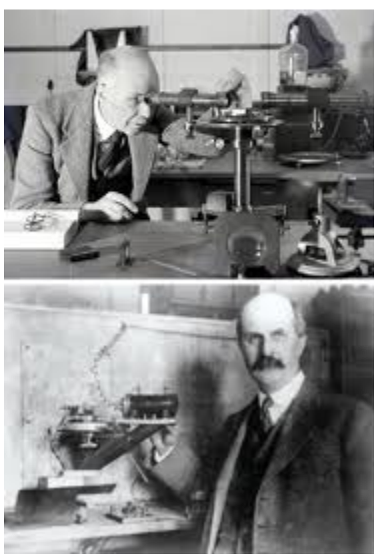


Eukaryothic Ribosomes
Klinge, Voigts-Hoffmann, Leibundgut, Arpagaus, Ban
Science 2011
Among most complex machineries of the cells
Responsible for the production of proteins

$CuSO_4$
M. Wildner, G. Giester
Orthorhomic Pnma (No.62)
a=8.4 b=6.7 c=4.8 A

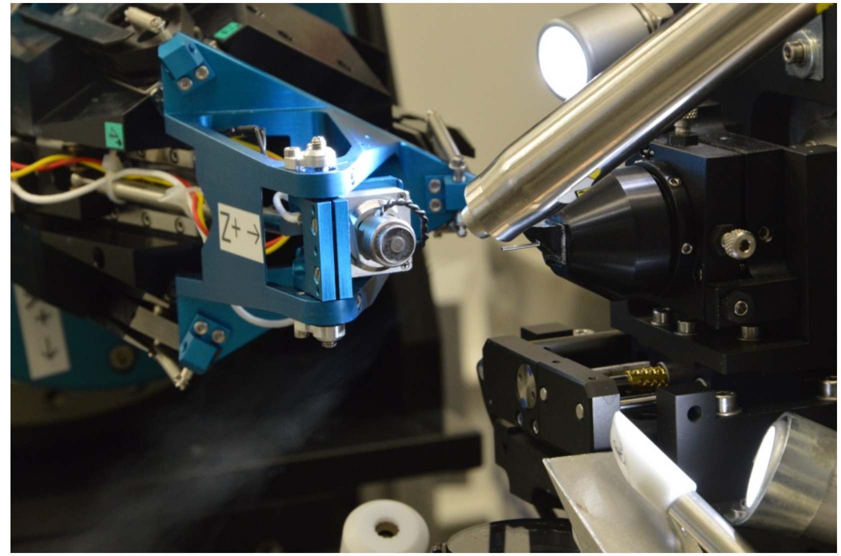
~1912

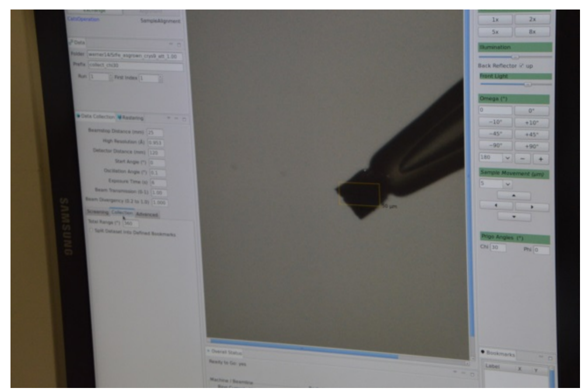
WH. Bragg
(father)



Pictures: Royal Institution/Science Muesum London/Schefer

2014





PXIII@SLS-PSI, “big” crystal of 50µm, 0.05 s/slide

Feb. 2013, SrFeOx, search for charge ordering

PILATUS2M Detector, Dectrics-Baden

~1912

2014

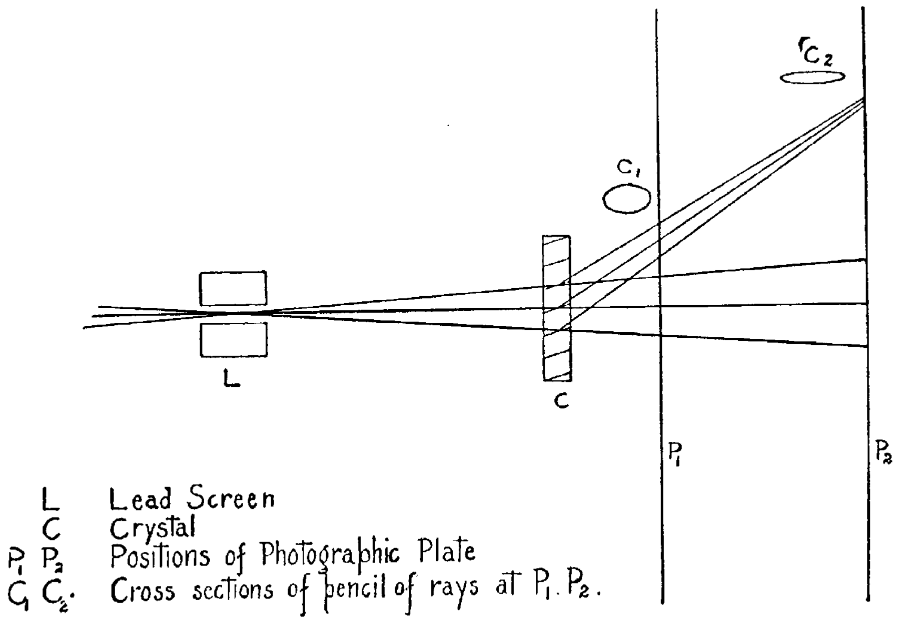
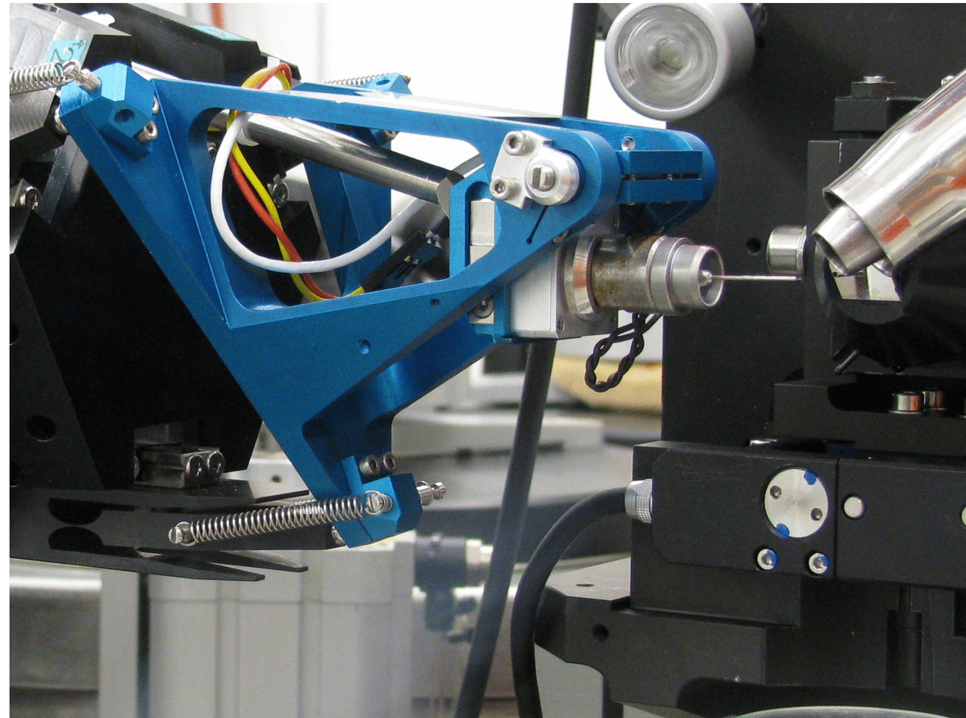


Fig. 2.

Proceedings of the Cambridge Philosophical Society XVIII (1913)

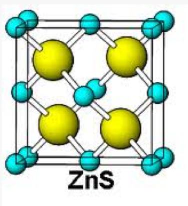


http://www.psi.ch/sls/pxiii/endstations

Left: PILATUS detector, right: monochromatic beam

~1912

Laue, Friedrich, Knipping
Sitzungsberichte Bayrische Akademie der Wissenschaften, Juni 2012



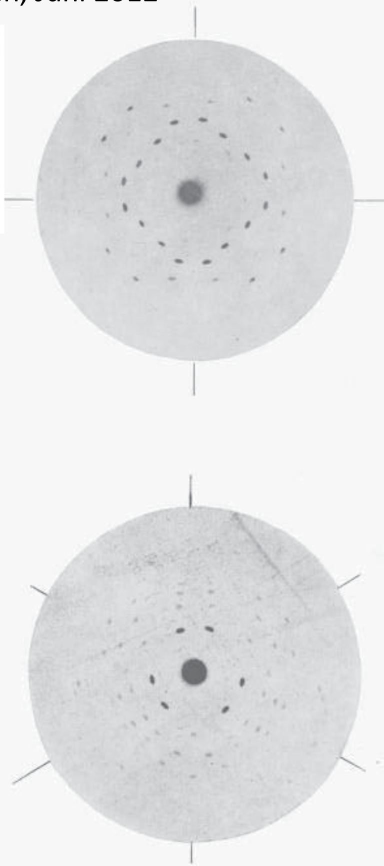
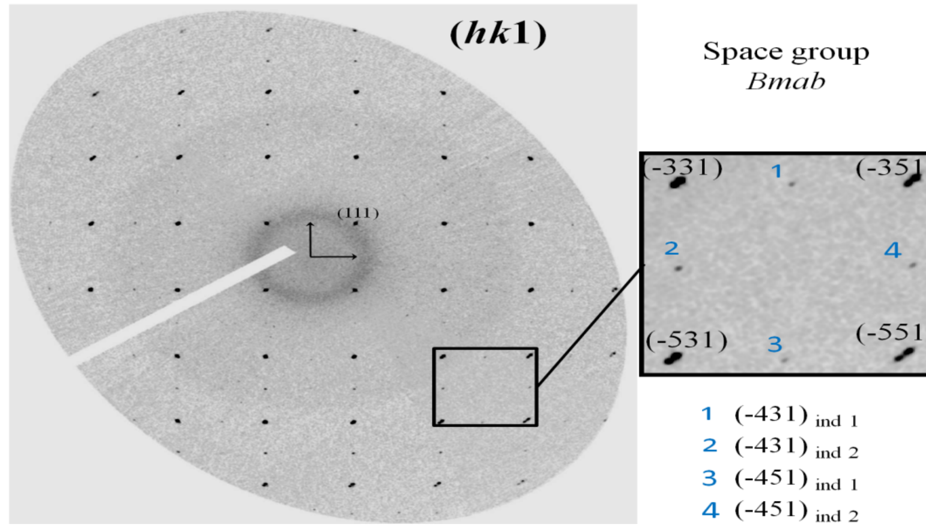
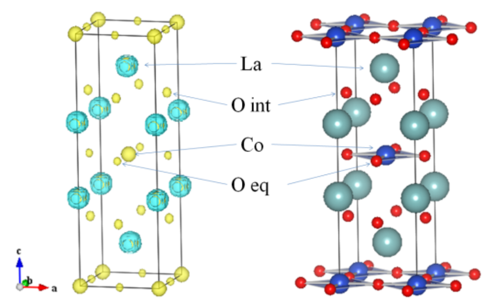


Fig. 4-4(3) and (4). Zincblende Laue photographs along four-fold and three-fold axes. (Laue, Friedrich & Knipping, *Sitz.ber. Bayer. Akademie d. Wiss.* 8. Juni 1912).

2014





3D electron density (Fourier obs)

Structural model *I4/mmm* unit-cell

La_2CuO_4, SNBL, reconstructed hk1 plane
ID11@ERSF
L. Le Dreau, W. Paulus et al.
Our goal: oxygen diffusion, needs disorder of octahedra

WH Bragg and WL Bragg

1862–1942 1890–1971

William and Lawrence Bragg. This portrait to the right was taken just before Sir Lawrence's broadcast on X-ray metals, delivered from London on February 23rd, 1942. This was the only occasion on which two Nobel laureates, father and son, took part jointly in a broadcast.

Bragg, W.H., X rays and Crystals, 24th October 1912. Letter to Nature **90** (1912) 219.
Bragg, W.L. The Diffraction of Short Electromagnetic Waves by a Crystal. Proceedings of the Cambridge Philosophical Society, **17** (1913) 43-57.
Bragg, W.L. Societies and Academies: Cambridge. Nature **90** (1912) 402.
Bragg, W.L. The specular reflection of X rays. Nature **90** (1912) 410.

Wilhem Conrad Röntgen (1845-1923)
1901: first Nobel Prize in Physics
discovery of the X-rays

Source: Röntgen: www.uni-giessen.de/uni/broschuere/geschichte.html
Laue: allg. Deutscher Nachrichtendienst, photographer unknown


№ 5.

DEUTSCHE
MEDICINISCHE WOCHENSCHRIFT.

Zweiundzwanzigster Jahrgang.

INHALT

Hand with Rings

one of the first X-Ray pictures

Wilhem Conrad Röntgen (1845-1923)

left hand of his wife Anna Bertha Ludwig

Print presented to
Prof. Ludwig Zehnder Physik Institut,
University of Freiburg/Germany
January 1896

.. Not to forget

Max von Laue (1879-1960)
1914: Prize in Physics
discovery of the diffraction
of X-rays by crystals

Source: Röntgen: www.uni-giessen.de/uni/broschuere/geschichte.html
Laue: allg. Deutscher Nachrichtendienst, photographer unknown

Resolution adopted by the General Assembly
(A/66/L.51 and Add.1) **66/284.**
International Year of Crystallography

The General Assembly, …

Stressing that **education about and the application of crystallography** are critical in addressing challenges such as

diseases and environmental problems, by providing protein and small molecule structures suited for drug design essential for medicine and public health, as well as solutions for plant and soil contamination,

Considering that the impact of crystallography is present everywhere in our daily lives, in modern drug development, **nanotechnology** and **biotechnology**, and underpins the development of all **new materials**, from .. to … components,

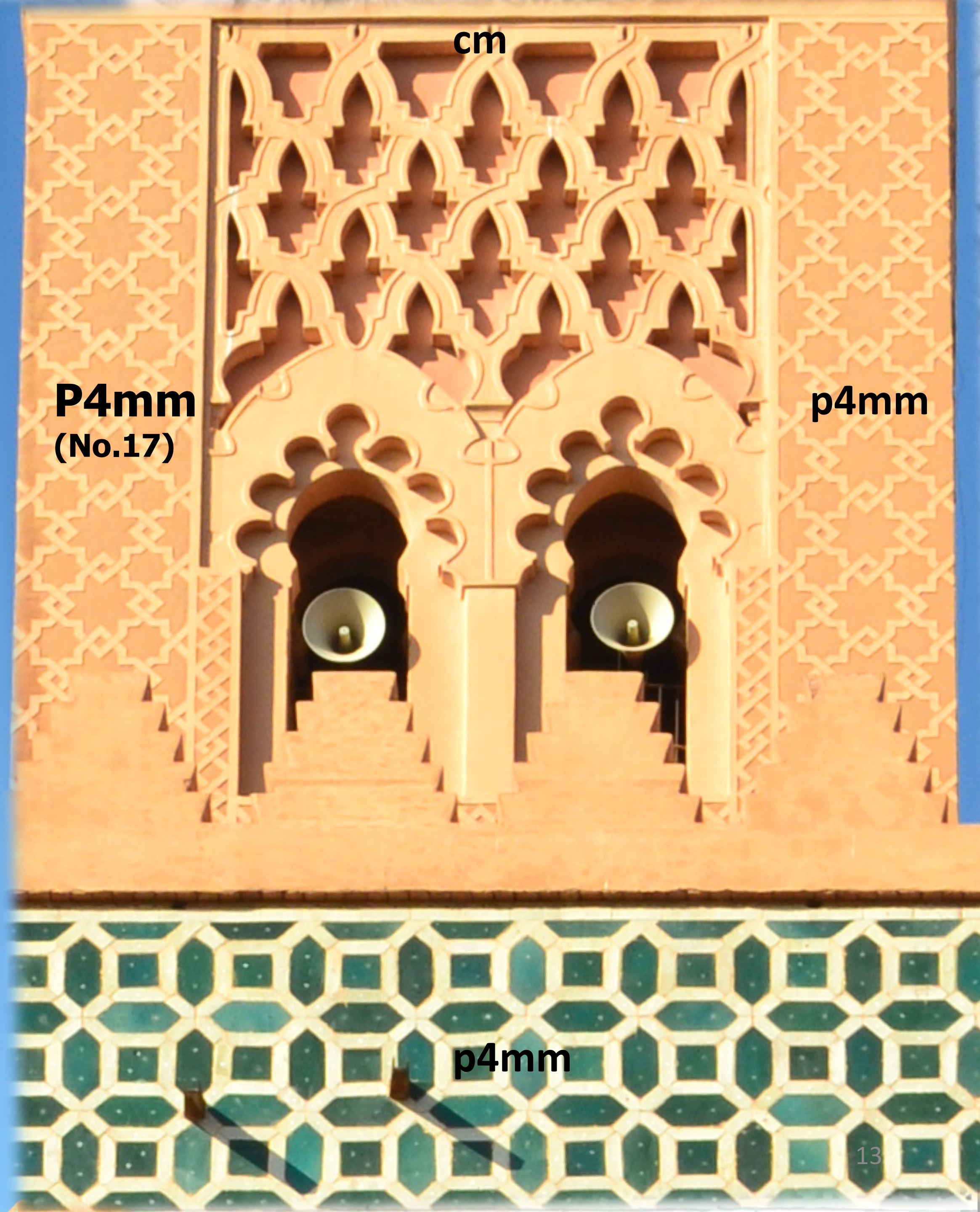
Considering also the significance of the scientific achievements of crystallography, as illustrated by **twenty-three Nobel Prizes awarded in the area,** and that crystallography is still fertile ground for new and promising fundamental research,

Considering further that 2014 marks the centenary of the beginning of modern crystallography and its identification as the **most powerful tool for structure determination of matter,**

…

Almohades

Source:
A. Thalal



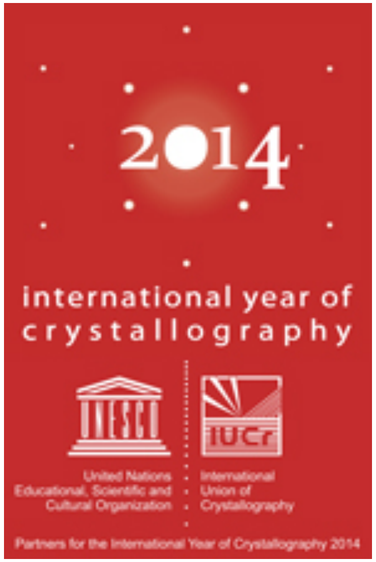
Involvement of the Moroccan Crystallographic Association in the Project of the IYCr

1st North African Conference of Crystallography Casablanca (NACC1) November 2010

Former IUCr president Prof Sine Larsen:

IUCr intend to apply for the declaration by UN of 2013 International Year of Crystallography.

She proposed MCA to spearhead the process leading to the UN via the Moroccan Representation to UNESCO



Opening Ceremony
Paris, Jan. 20, 2014

Video message of Ban Ki-moon,
Secretary-General of the United Nations

Irina Bokova, Director-General of UNESCO

Source: IUCr/UNESCO

Goals of the year of Crystallography as summarized by IUCr:

- increase public awareness of the science of crystallography and how it underpins most technological developments in our modern society
- inspire young people through public exhibitions, conferences and hands-on demonstrations in schools
- illustrate the universality of science
- intensify the program Crystallography in Africa and create similar programs in Asia and Latin America
- foster international collaboration between scientists worldwide, especially North–South contributions
- promote education and research in crystallography and its links to other sciences
- involve the large synchrotron and neutron radiation facilities worldwide in the celebrations of IYCr2014, including the SESAME project set up under UNESCO auspices


⟨S|P|S⟩ swiss physical society

Swiss Physical Society
Schweizerische Physikalische Gesellschaft
Société Suisse de Physique

Suchen & Finden | suchen

Sprache DE EN FR | Home | SPG | Veranstaltungen | SPG Preise | Artikel | Archiv | Links | Stellenangebote

Fortschritte in der Physik
- Crystallography at SLS, SINQ and SNBL (40)
- IceCube pushes neutrinos to the forefront of astronomy (38)
- Molecular Lego: Bottom-up Fabrication of Atomically Precise Graphene Nanostructures (37)
- Modern Techniques in Radiation Oncology (36)
- A snowflake in a million degree plasma (35)
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- Outreach: Can Physics Cross Boundaries ? (33)
- New probes for condensed matter research at the Paul Scherrer Institute (32)
- Past and Present Challenges in Nuclear Astrophysics (31)
- New heavy boson discovered by the LHC experiments (30)
- Understanding exchange bias in thin films (29)
- SATW Forum "Advanced Optoceramics" (28)
- Brownian Motion beyond Einstein (27)
- Improving energy confinement in fusion plasmas by plasma shaping and current profile tailoring

Crystallography at SLS, SINQ and SNBL

J. Friso van der Veen, Swiss Light Source, PSI Villigen and ETH Zürich,
Jürg Schefer, Laboratory for Neutron Scattering, PSI Villigen

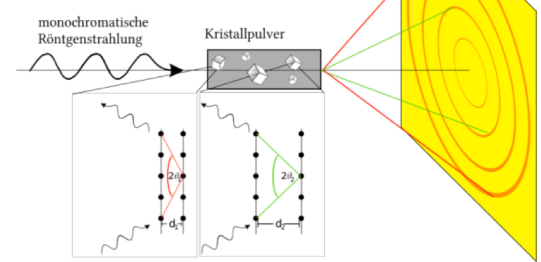
Each year, the United Nations' Org attention to a field of great import Scientific topics were repeatedly i biological diversity, 2011 chemistr 2014, crystallography. The openin International Year of Crystallograp January 20 and 21, 2014 (IYCr20 headquarters in Paris. The Interna Crystallography takes the lead in IYCr2014. The activities in this ye celebration in 2012 of the 100th ar discovery of Bragg's Law and pay Nobel prizes awarded to crystallog we provide a selection of recent re large facilities of Paul Scherrer Ins Swiss Light Source) and at the Sw beamline of the European Synchro Grenoble, France.

1 Swiss activities for the UN year of crysta

www.sgk-sscr.ch/iycr2014-2/

www.post.ch/philshop

Debye-Scherrer Verfahren



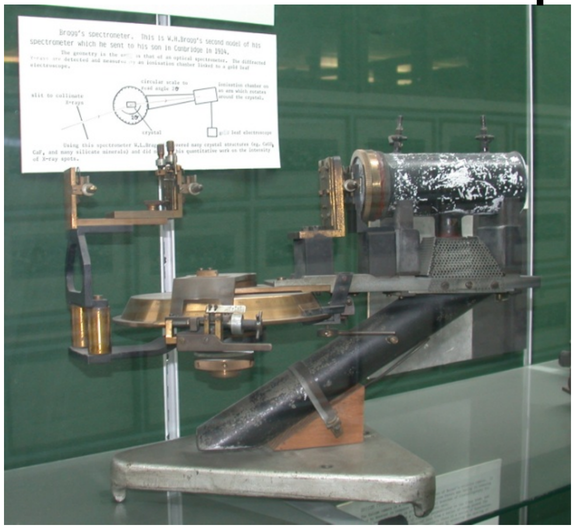
Jürg Schefer
Laboratory for Neutron Scattering
Paul Scherrer Institut
Jurg.Schefer@psi.ch

Some examples

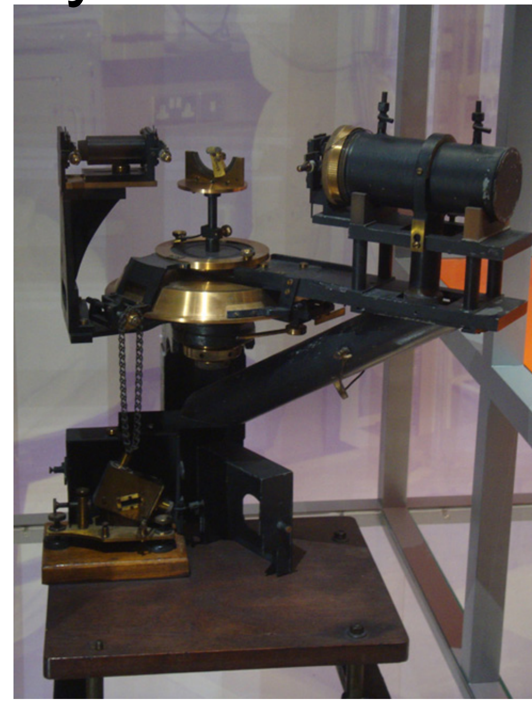
- **100 years ago: Instruments and computing**
- **Crystallography in Switzerland**
- **Focus on the large Scale Facilities**

- SLS – SINQ – SNBL@ESRF

WHB's ionization spectrometers built by C.H. Jenkinson



Cavendish Lab, Cambridge



Royal Institution

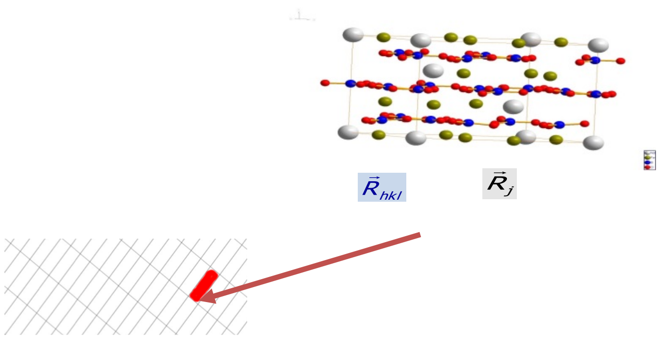
Science Museum, London

Fourier Transformations
Connects the measurement to the world

Real space r (real world)
Reciprocal space q (measurement)

$$f(r) = \int_{-\infty}^{+\infty} F(q)\cdot e^{2\pi iq}dq$$

$$F(q) = \int_{-\infty}^{+\infty} f(r)\cdot e^{2\pi iq}dr$$

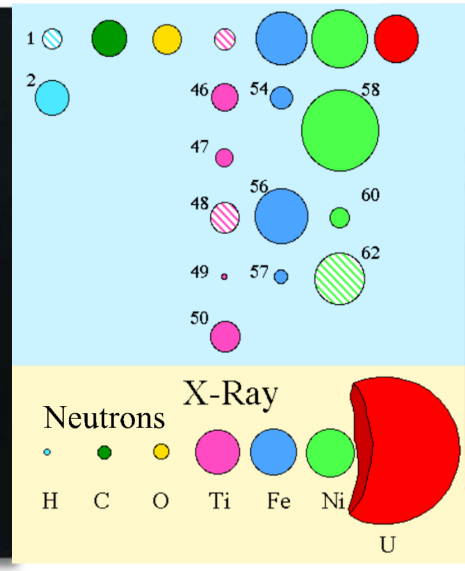


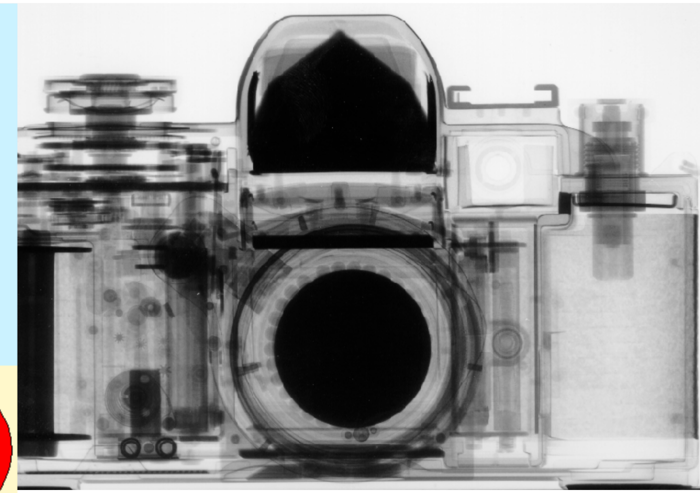
Prototype of the famous Beevers-Lipson strips, rescued by Mike Glazer on moving from the Austin Wing of the Cavendish Laboratory in 1974.

Real space: Lattice (translation), unit-cell (basic unit for atoms)
ki-kf: Momentum transfer to probing neutrons, X-rays, …

$$\sum_{hkl}^{lattice}\sum_{j}^{atoms} b_j\cdot e^{i(\vec{k}_i-\vec{k}_f)\cdot(\vec{R}_{hkl}+\vec{r}_j)} = \sum_{hkl,j} \quad e^{i(\vec{k}_i-\vec{k}_f)\cdot\vec{R}_{hkl}} \quad b_j e^{i(\vec{k}_i-\vec{k}_f)\cdot\vec{r}_j}$$

Neutrons or X-Rays ?





Metalbody/
Glass with B

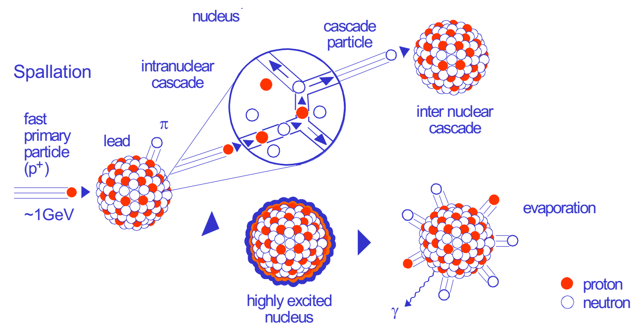
Water/Bones

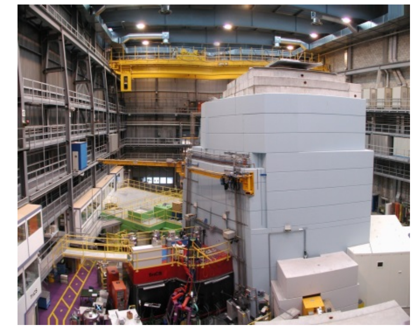
Absorption (Bor) = 767 (10^{-24}cm^2)
Absorption (Al) = 0.231

Materials with different
Electron density
Z_H=1, Z_C=12

Neutron Scattering at SINQ

- A strong neutron source

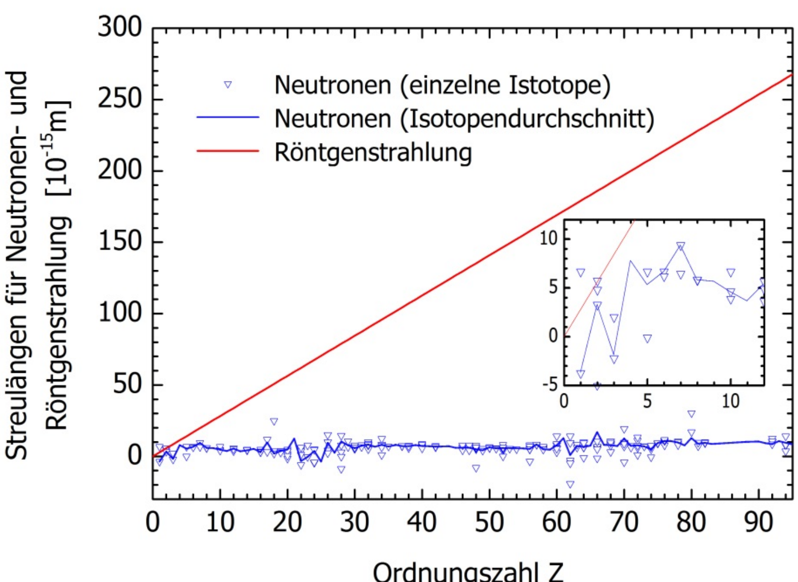




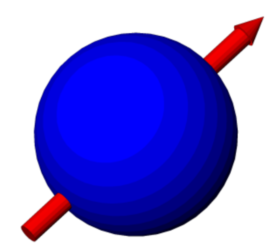
- A scattering process
  (nuclear or magnetic)

$$\left(\frac{\partial^2\sigma}{\partial\Omega\partial\omega}\right)_{\sigma_i\lambda_i\to\sigma_f\lambda_f} = \frac{k_f}{k_i}\left(\frac{m}{2\pi\hbar^2}\right)^2 \cdot \sum_{\lambda_i} p_{\lambda_i} \sum_{\lambda_f} \left\langle k_f\lambda_f \middle| V(\vec{r},t) \middle| k_i\lambda_i \right\rangle \cdot \delta(\hbar\omega + E_{\lambda_i} - E_{\lambda_f})$$

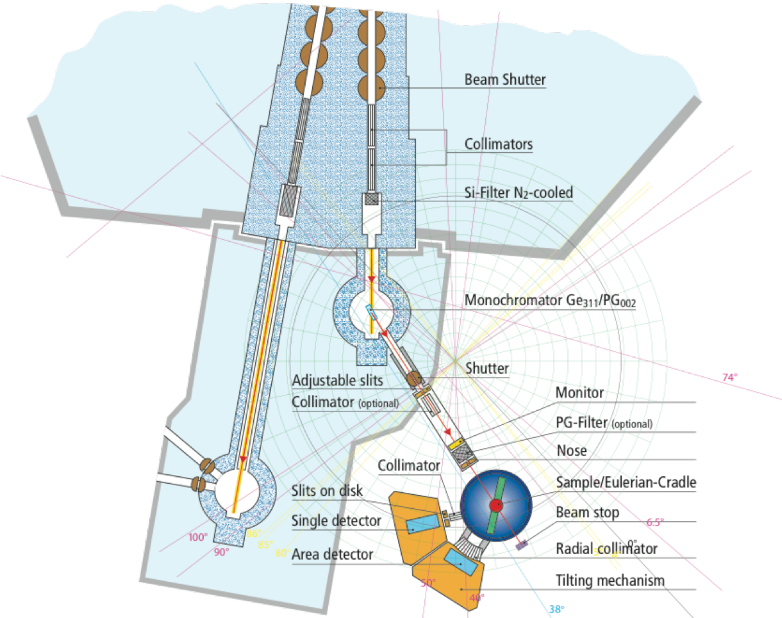
- The interaction potential

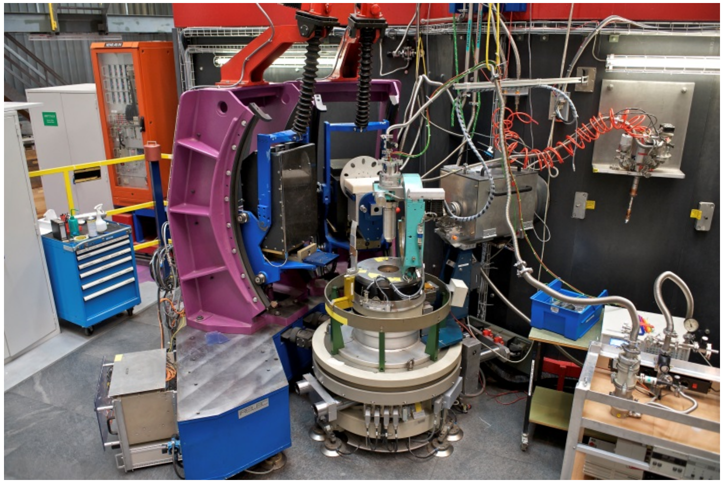


$\mu_{\text{Neutron}} = 1.04 \cdot 10^{-3}\ \mu_{\text{Bohr}}$



Single Crystal Diffractometer TriCS





Instrument Responsible: O. Zaharko

Speciality 1: phase transitions

As a function of temperature, magnetic/ electric field, pressure

Speciality 2: superstructures and complex magnetic structures

Multiferroic $CuCrO_2$ M. Frontzek et al. J Phys Cond Mat **24** (2012)16004

low temperature magnetic structure is unambiguously helical:

Speciality 3: small crystals

for crystal mass of 10-20 (200-300) mg

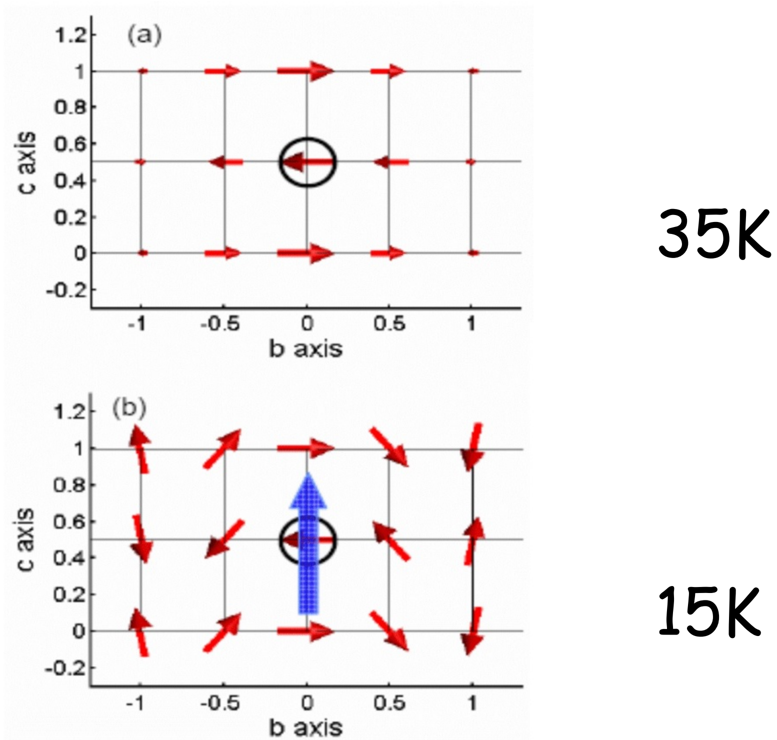
magnetic structure with 1 (0.1) μ_B can be determined

Yet, not with bulky sample environment

(dilution, magnet due to large background)

Coexistence of Magnetism and Ferroelectric ordering an urgent question for new materials

C. Ederer et al., Nature **3**, 849 (2003)
N. Hur et al., Nature **429**, 392 (2004)
Th Lottermoser et al., Nature **430**, 541 (2004)



$\mu_{Neutron} = 1.04 \cdot 10^{-3}\ \mu_{Bohr}$

Example: TbMnO3

Breaking inversion symmetry
Allows an electric polarization along c

Idea:
Electric field or Magnetic field
Can switch each-other

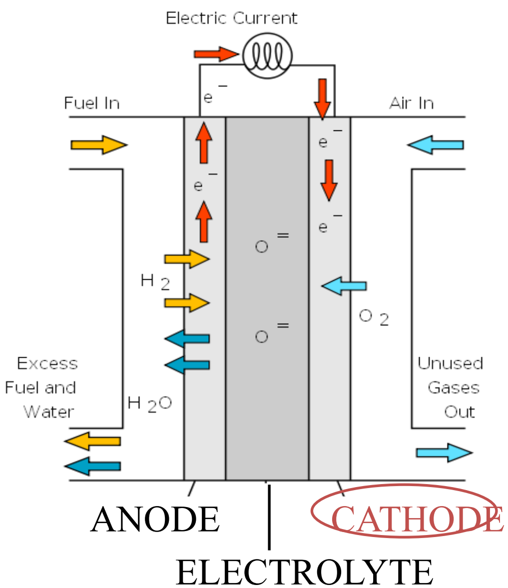
Magnetic structure of $TbMnO_3$ at 35K (a) and at 15K (b) determined on TriCS/SINQ.

While inversion symmetry is preserved in the longitudinally modulated structure at 35K, it is broken by the spiral structure at 15K, allowing an electric polarization to develop (blue vertical arrow).

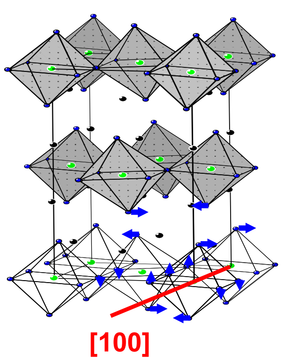
M. Kenzelmann et al.
Phys. Rev. Lett. 95, 087206 (2005)

Towards room temperature oxygen conductors

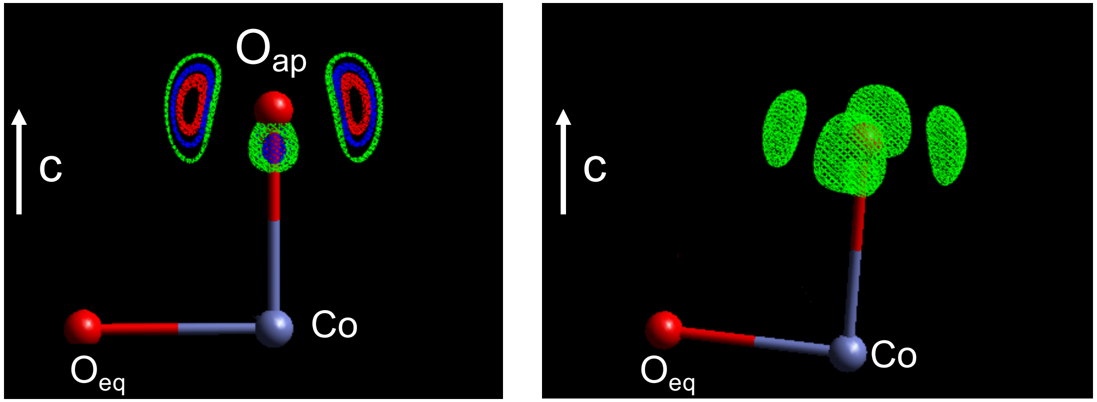
TriCS@SINQ, ID11@ESRF



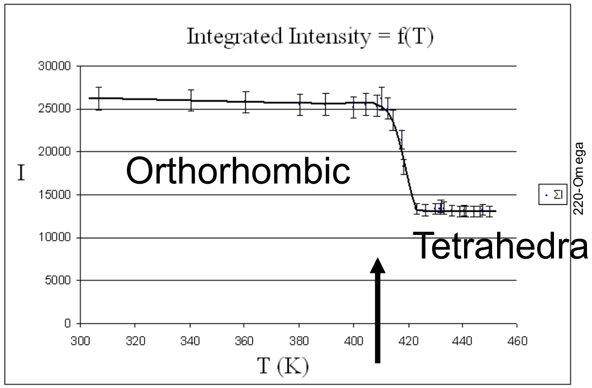
$La_2CoO_{4.14}$



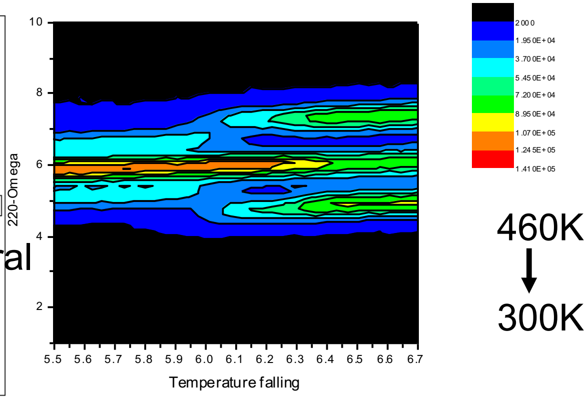
Fourier difference map around apical Oxygen atoms



Single crystal data collected at TriCS@SINQ@PSI



410K



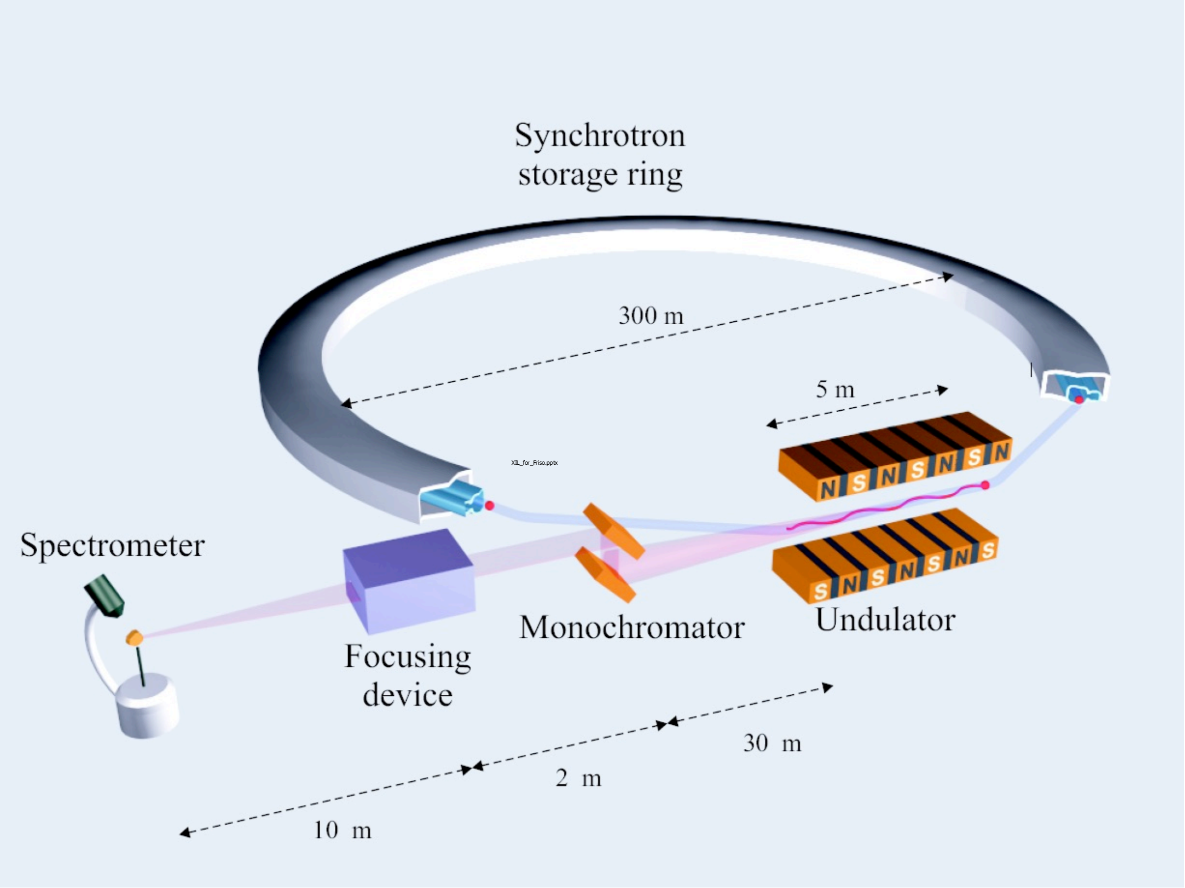
460K → 300K

Splitting of the twins at 410K

Le Dreau et al., Thesis 2011, Rennes

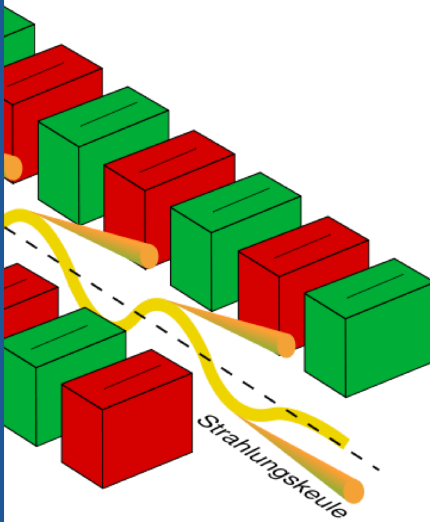
Synchrotron radiation facility

A very brilliant X-ray source: 10^{12} X-ray tube !



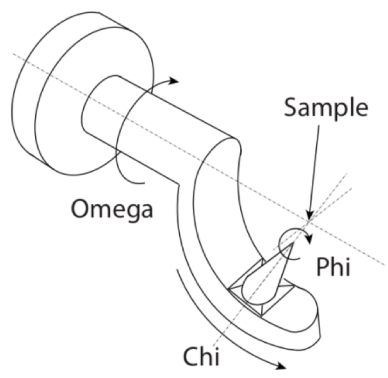
From: Elements of Modern X-Ray Physics, J. Als-Nielsen and Des McMorrow

SLS: a brillant Synchrotron Source

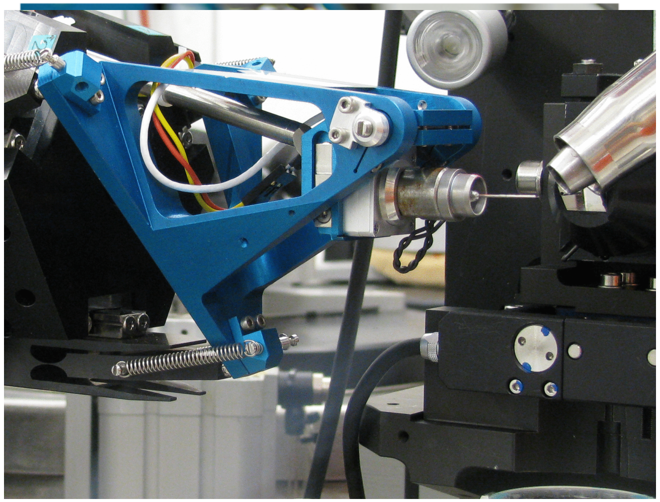


Multi-axis goniometer (PRIGo) at PXIII

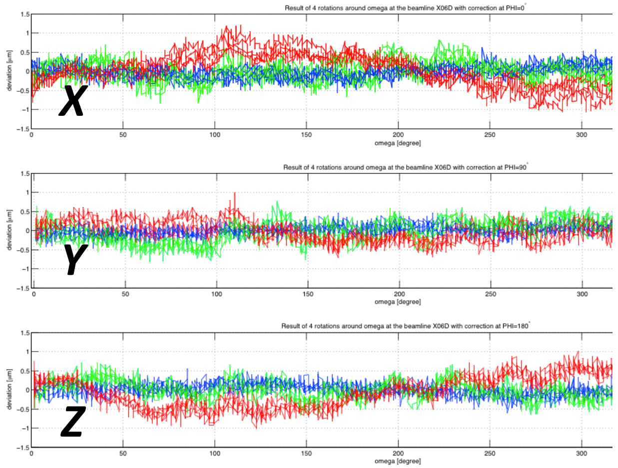
a)



b)



SOF < 1 µm



***Omega:* 0 – 360 degree**

Advantages of multi-axis goniometer

- Select best part of crystal
- Misalign cell-axis to avoid blind zone
- Align long cell-axis to avoid spots overlap
- Align even-fold symmetry axis to record Bijvoet pairs on same diffraction image
- Multi-orientation data sets to obtain true redundancy and better absorption correction (At longer wavelength)

S-SAD phasing example at PXIII

Single-wavelength **a**nomalous **d**iffraction (SAD) utilizing the weak signal of inherently present S atoms

anomalous scattering length of S atoms (f"" in electrons)

Novartis protein X

$P2_12_12_1$

Data collection with PRIGo and PILATUS at PXIII

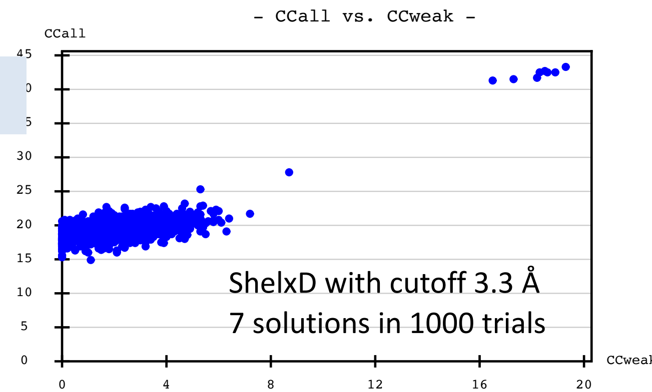
Low-dose, high-redundancy and multi-orientation

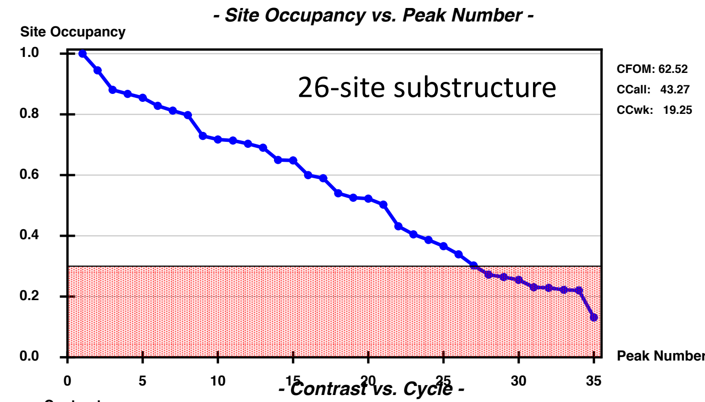
6 keV (2.066 Å), 0.1° / 0.1 sec

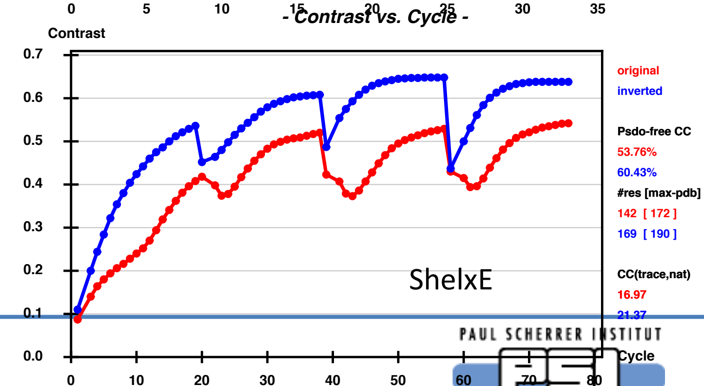
Diffraction resolution: 2.5 Å

Total 6 × 720° data collected with different Chi angle at two different positions of one crystal

	Chi	*Chi*	*Chi*
Position 1	0	25	8
Position 2	0	10	20







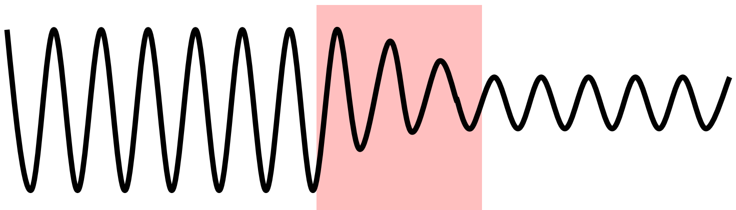
PAUL SCHERRER INSTITUT

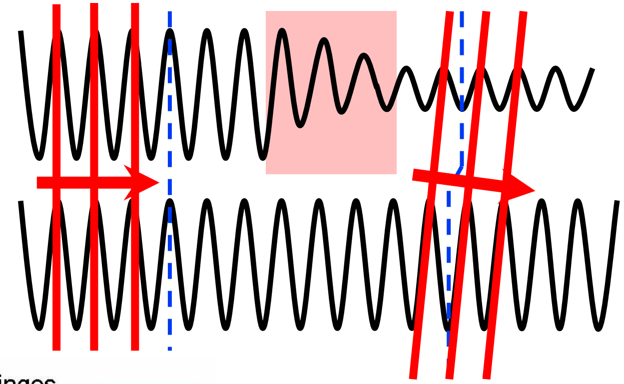
Going a little by away from standard crystallography

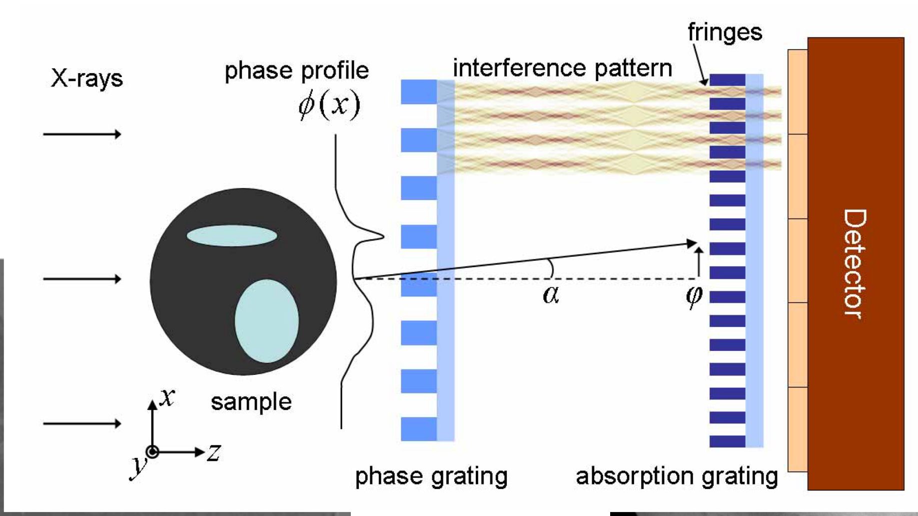
Using nanostructures for X-ray imaging

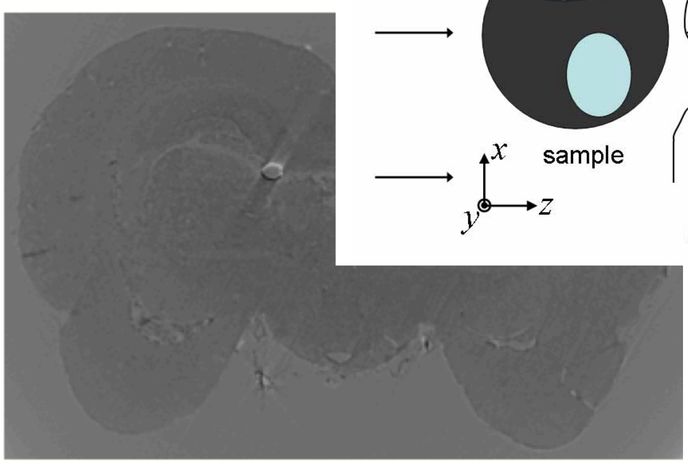
Better X-ray imaging

M. Stampanoni et al.

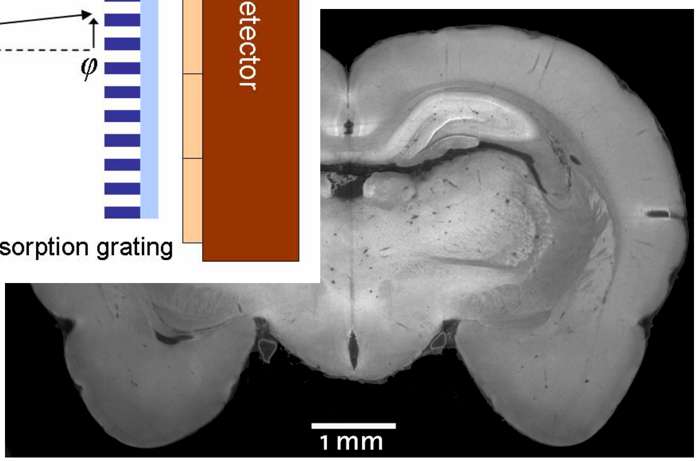






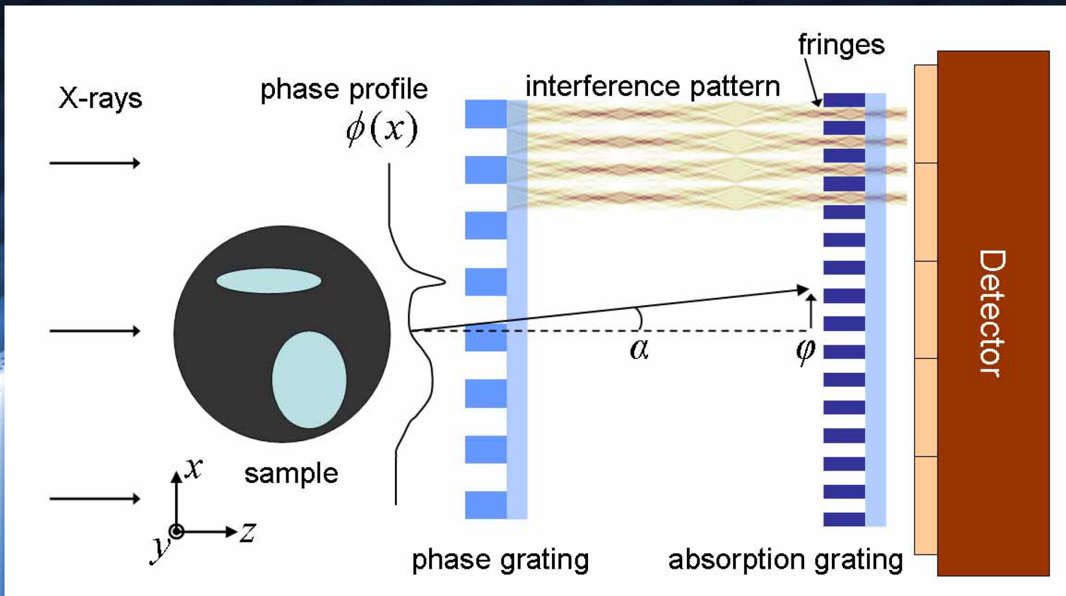


Absorption contrast



Phase contrast

Sensing the refraction angle with grating interferometry

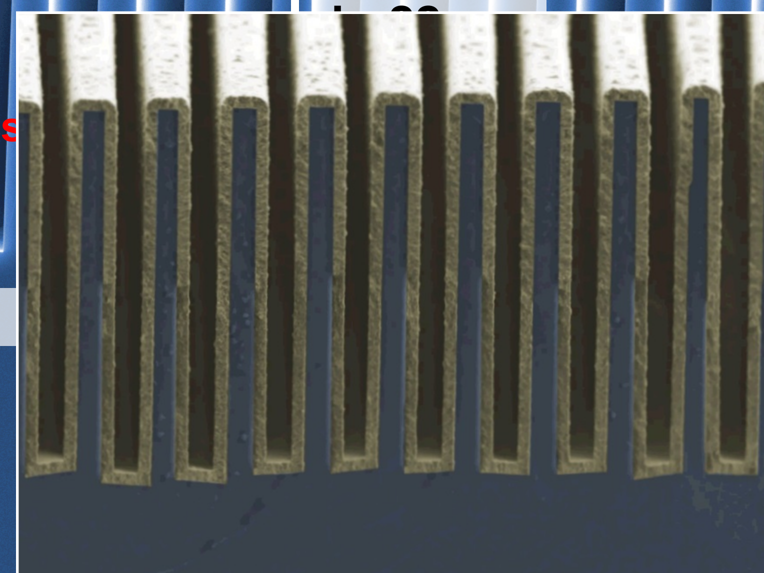


Absorption contrast

$$-\log\left(\frac{I_s}{I_b}\right)$$

Differential phase contrast

$$\Phi_s - \Phi_b$$



nature COMMUNICATIONS

Swiss Norwegian Beamline @ ESRF Grenoble

ARTICLE

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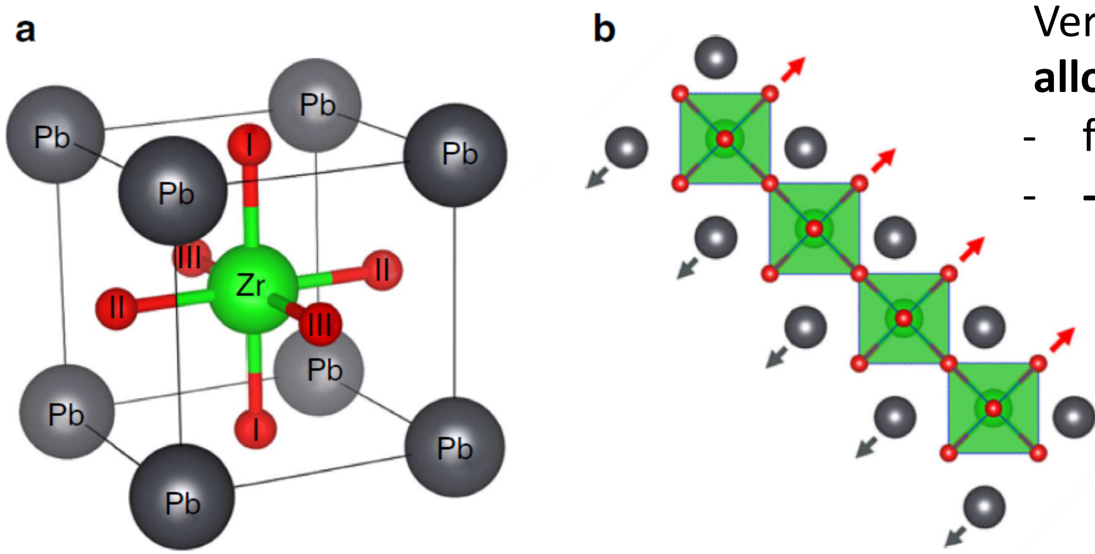
The origin of antiferroelectricity in $PbZrO_3$

A.K. Tagantsev[1,2], K. Vaideeswaran[1], S.B. Vakhrushev[2,3], A.V. Filimonov[3], R.G. Burkovsky[3,4], A. Shaganov[3], D. Andronikova[3], A.I. Rudskoy[3], A.Q.R. Baron[5], H. Uchiyama[5], D. Chernyshov[6], A. Bosak[4], Z. Ujma[7], K. Roleder[7], A. Majchrowski[8], J.-H. Ko[9] & N. Setter[1]

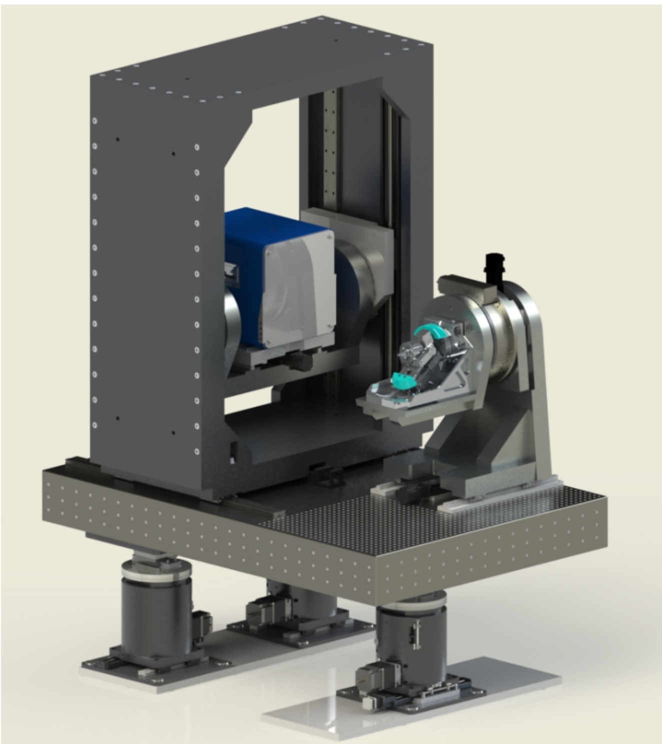


Very common: leaed zirconate titate **alloy of**

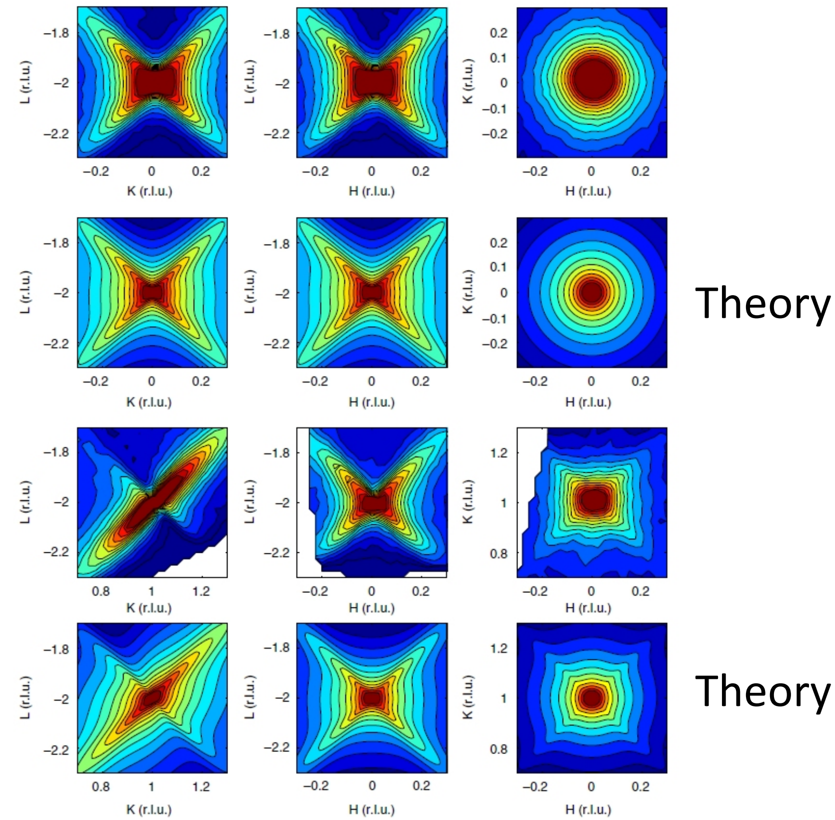
- **ferroelectric lead titanate**
- **- antiferroelectric lead zirconate**

Possibilities Today at SNBL@ESRF

Swiss-Norwegian Beamline



Pilatus 2M DECTRIS®
Baden-Dättwil



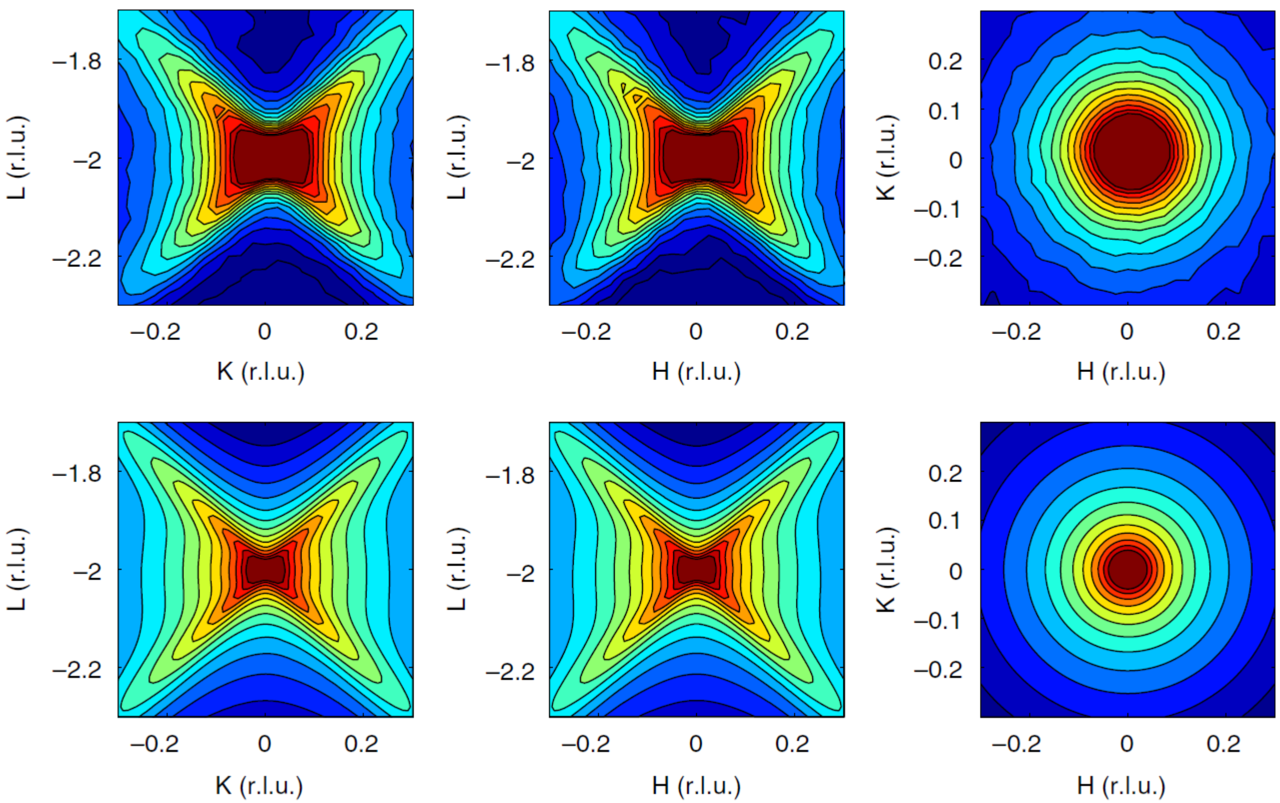
The origin of antiferroelectricity in $PbZrO_3$

A.K. Tagantsev et. Al, Nature Comm. **4** (2013) 2229

PILATUS detector: Looking between the Bragg spots

flexoelectric coupling

$$F = \frac{\alpha}{2}P^2 + \frac{c_{ijkl}}{2}u_{ij}u_{kl} + \frac{g_{ijkl}}{2}\frac{\partial P_i}{\partial x_j}\frac{\partial P_k}{\partial x_l} - \frac{f_{ijkl}}{2}\left(P_k\frac{\partial u_{ij}}{\partial x_l} - u_{ij}\frac{\partial P_k}{\partial x_l}\right)$$



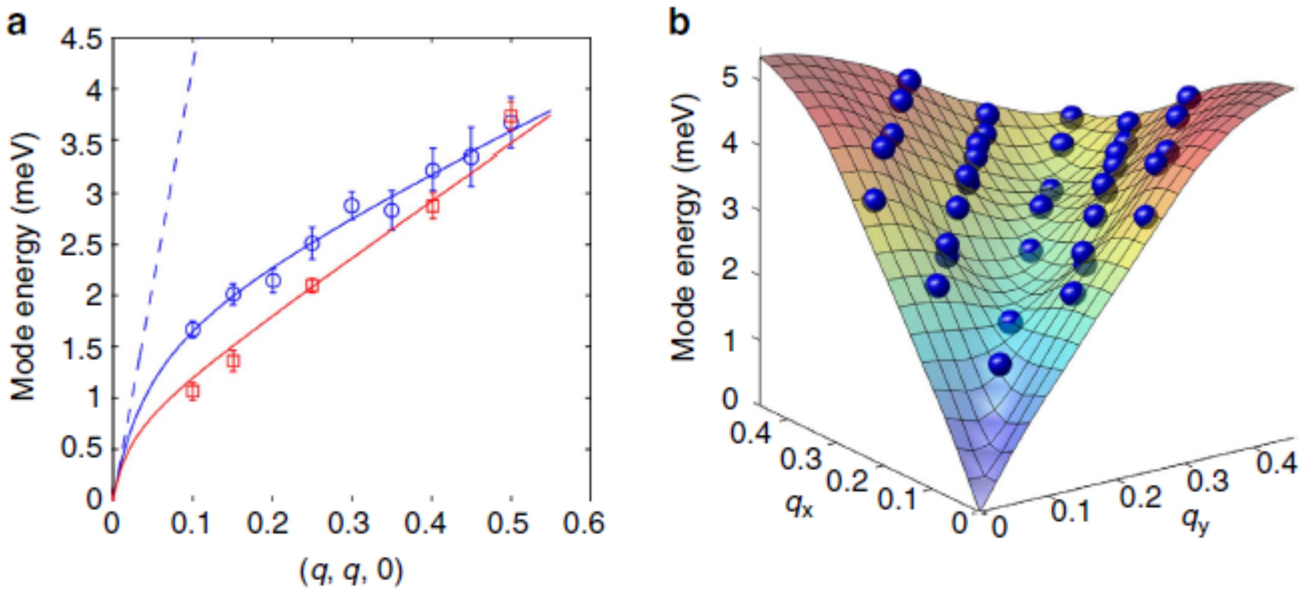
Data from SNBL
ESRF@Grenoble
Cuts at (0 0 -2)
in 3 directions

Calculation with
flexoelectric coupling

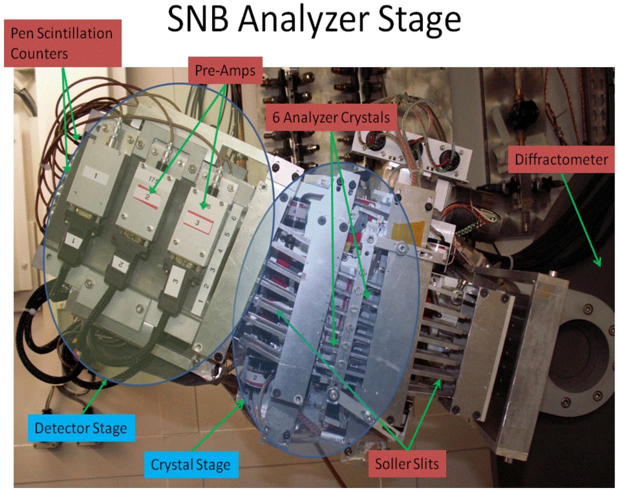
$PbZrO_3$: Tagantsev et. al, Nature Comm 2013

PAUL SCHERRER INSTITUT

Dispersion of the TA phonons in lead zirconate



SNB Analyzer Stage



flexoelectric coupling

$PbZrO_3$: Tagantsev et. al, Nature Comm 2013

Upcoming Sources

ESS – Lund, Sweden

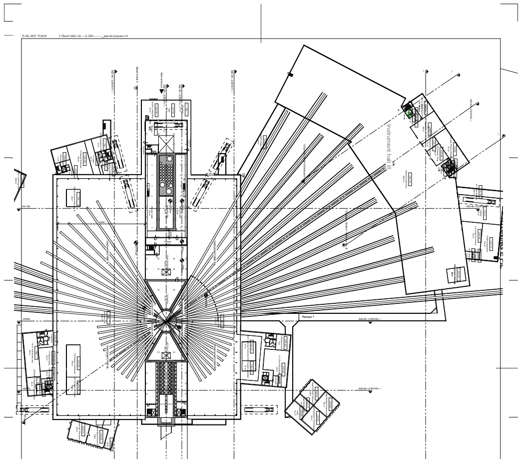
Neutron Spallation Source

SwissFEL – PSI Villigen

Free Electron Laser

ESS Lund , Sweden
next to MAX IV

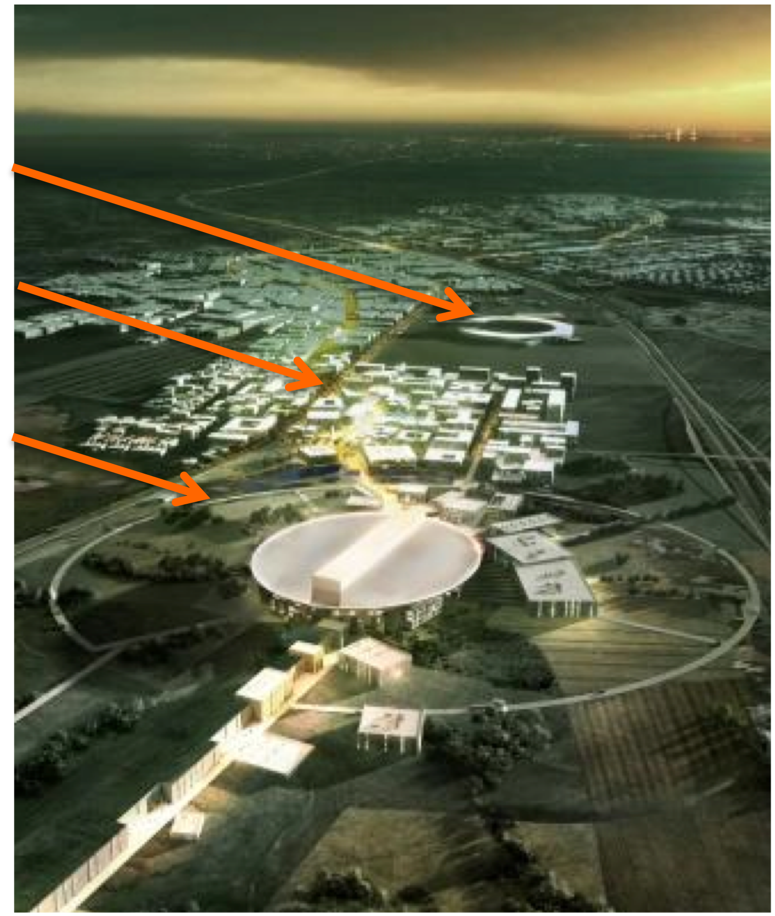
EUROPEAN SPALLATION SOURCE



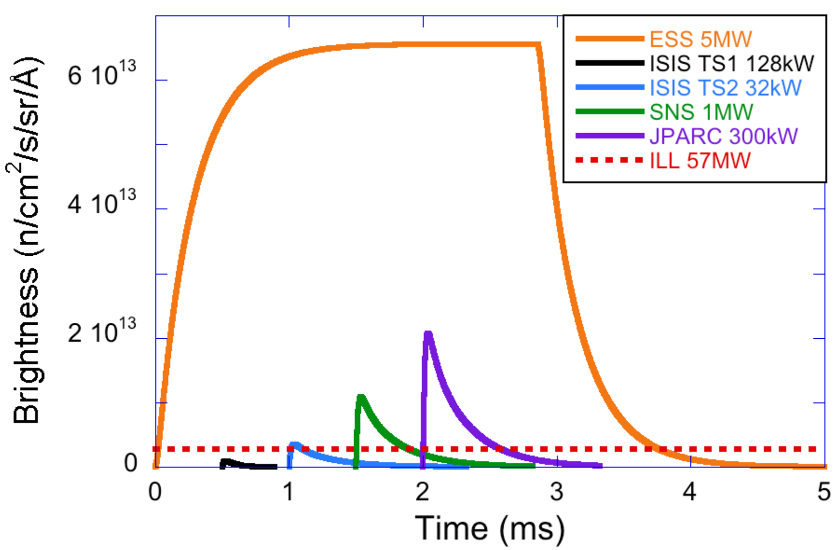
MAX IV synchrotron

Science Village Scandinavia

ESS



Single-Pulse Brightness at 5 Å

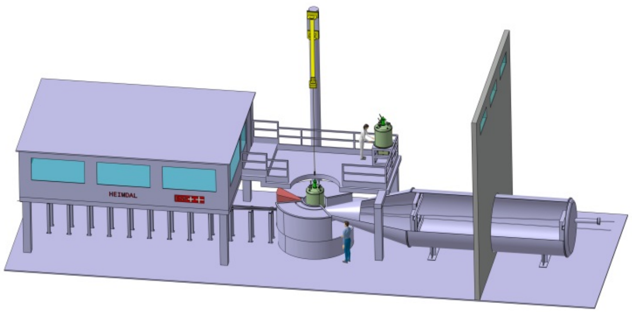


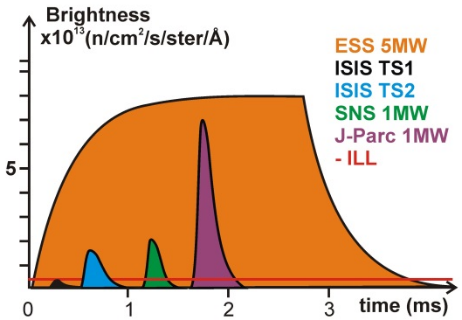
HEIMDAL – ESS Powder Diffractometer

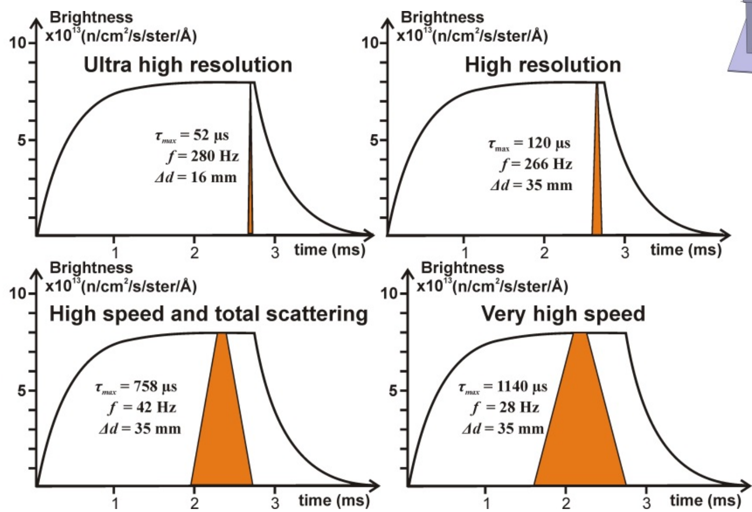
Combining NPD with SANS

ESS

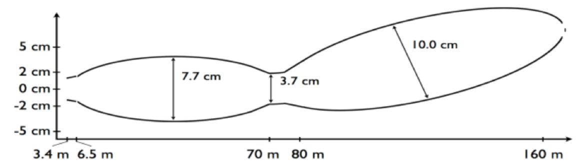
- Light elements and energy related materials
- Composites, scaffolds or matrix embedded systems
- Phase transition and nucleation
- Materials with magnetic properties







Thermal guide: m=3-5



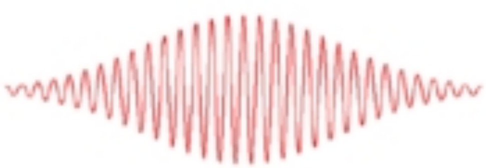
Cold guide:

M=2, relatively simple

resolution (λ_{mean} =1.5 Å)
Δd/d@90° 0.1% to 2%
Δd/d@170° 0.015% to 1.5%
time averaged flux of ~$3 \cdot 10^8$-$2 \cdot 10^9$ n/s/cm^2

SwissFEL

Optical laser
Good time resolution (short pulse)
Poor spatial resolution (long wavelength)

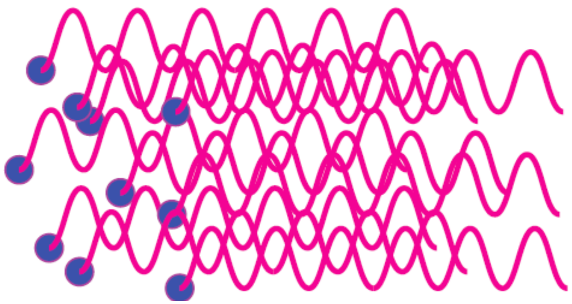


Synchrotrons
Low time resolution (long pulse)
Good spatial resolution (short wavelength)

FEL
Good time resolution (short pulse)
Good spatial (short wavelength)

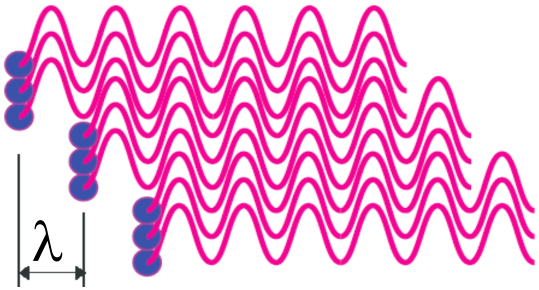
X-ray source with an extreme intensity

SLS



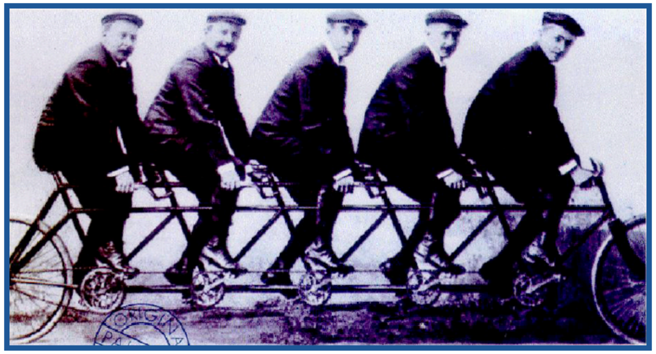
Incoherent X-rays:
→ Emission from individual, disordered, electrons

XFEL

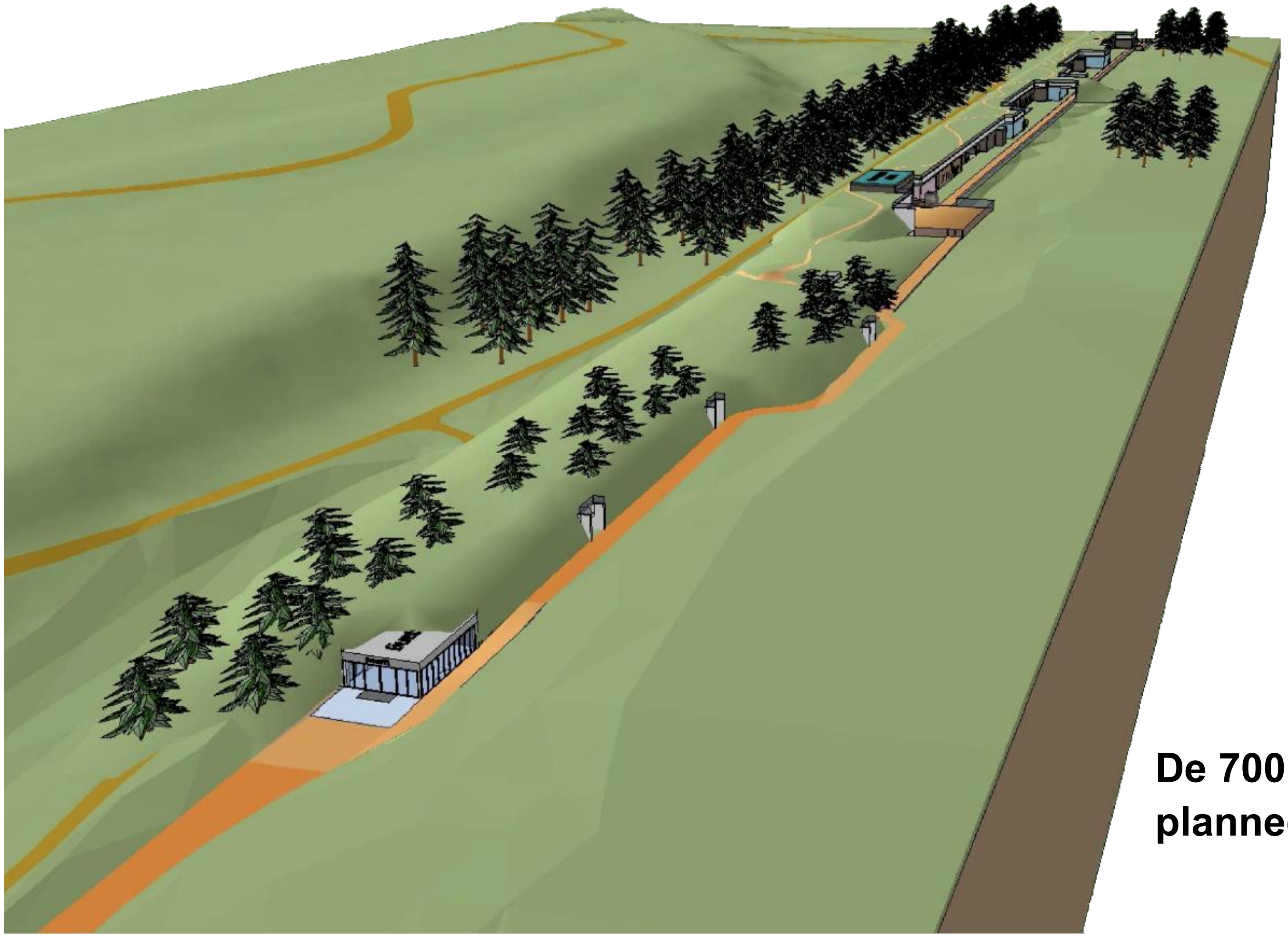


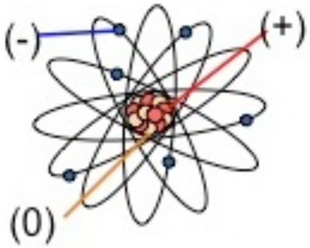
Coherent X-rays:
□ emitted by ordered electron bunches

XFEL
Atomic-resolution + time-resolution
to observe the "dance" of the atoms



New Large Facility at PSI SwissFEL



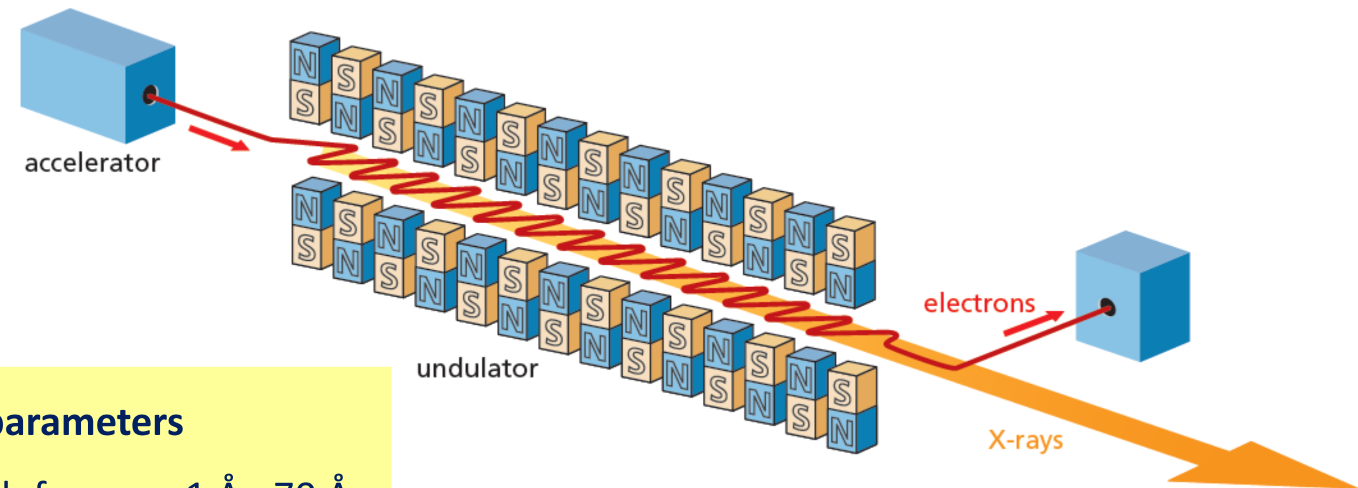


De 700 m. X-ray laser is planned start on 2016

The X-ray free electron laser

FEL principle

Electrons interact with periodic magnetic field of undulator magnet to build up an extremely short and intense X-ray pulse.



SwissFEL parameters	
Wavelength from	1 Å - 70 Å
Pulse duration	1 fs - 20 fs
e^- Energy	5.8 GeV
e^- Bunch charge	10-200 pC
Repetition rate	100 Hz

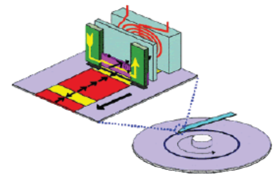
Peak brilliance: 10^{13} synchrotron

SwissFEL base lines

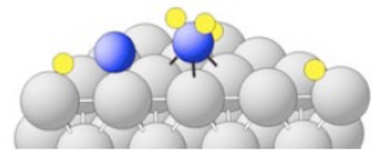
Basic FEL parameters	Aramis beamline	Athos beamline
Wavelength	1-7Å	0.7-7nm
Saturation length	45-21m	41.6-16.1m
Effective saturation power	~ 2.0GW	4.4-6.5GW
Bandwidth	0.03-0.14%	0.15-0.25%
Pulse length	13fs	11fs
Polarisation	planar	helical
Repetition Rate	100Hz	100Hz

SwissFEL science case

Magnetism

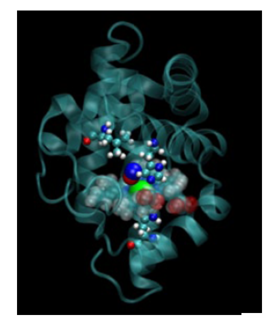


Catalysis and solution chemistry

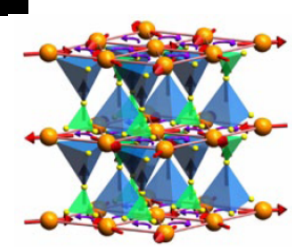


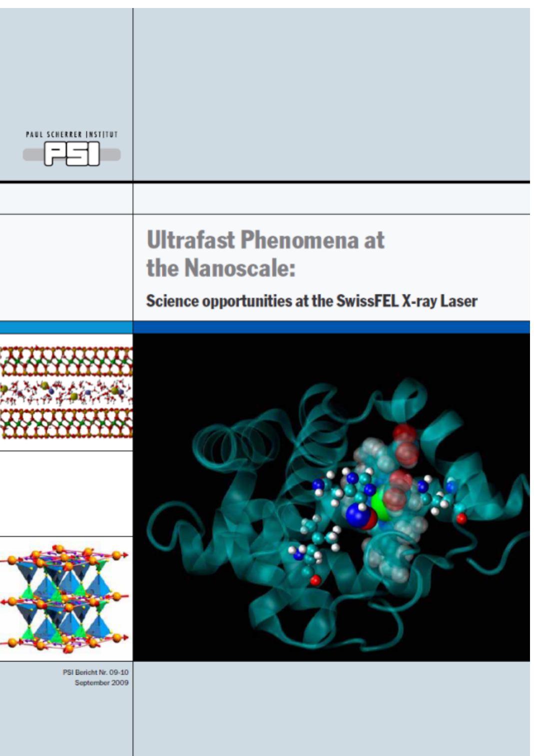
flash photography of matter

Biochemistry



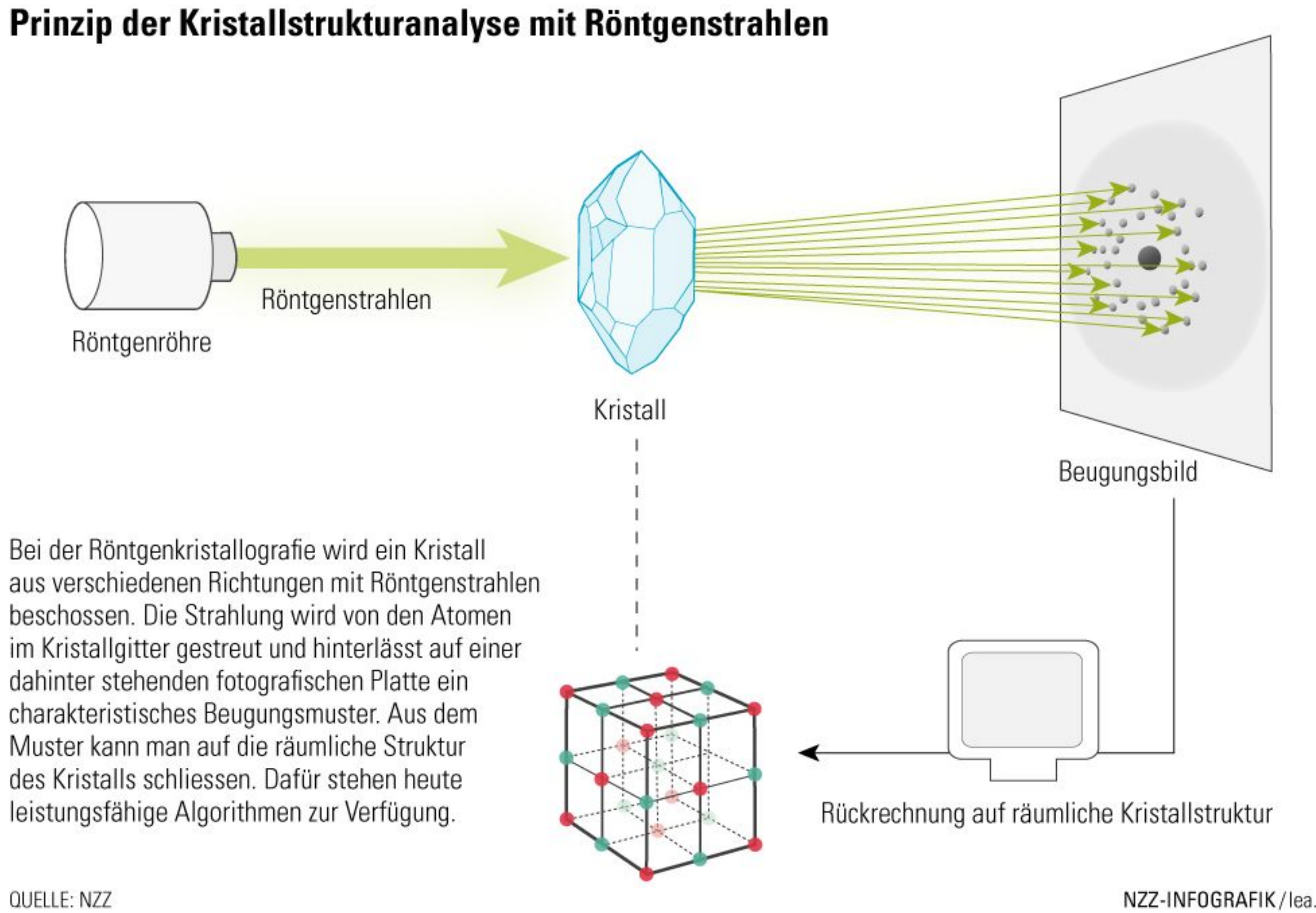
Correlated electron materials





PDF downloadable from
http://www.psi.ch/swissfel/swissfel

Measuring very short shots on intermediate states for example in catalysis



2014 – Year of Crystallography
One goal of this year

- Making crystallographic techniques available for everybody

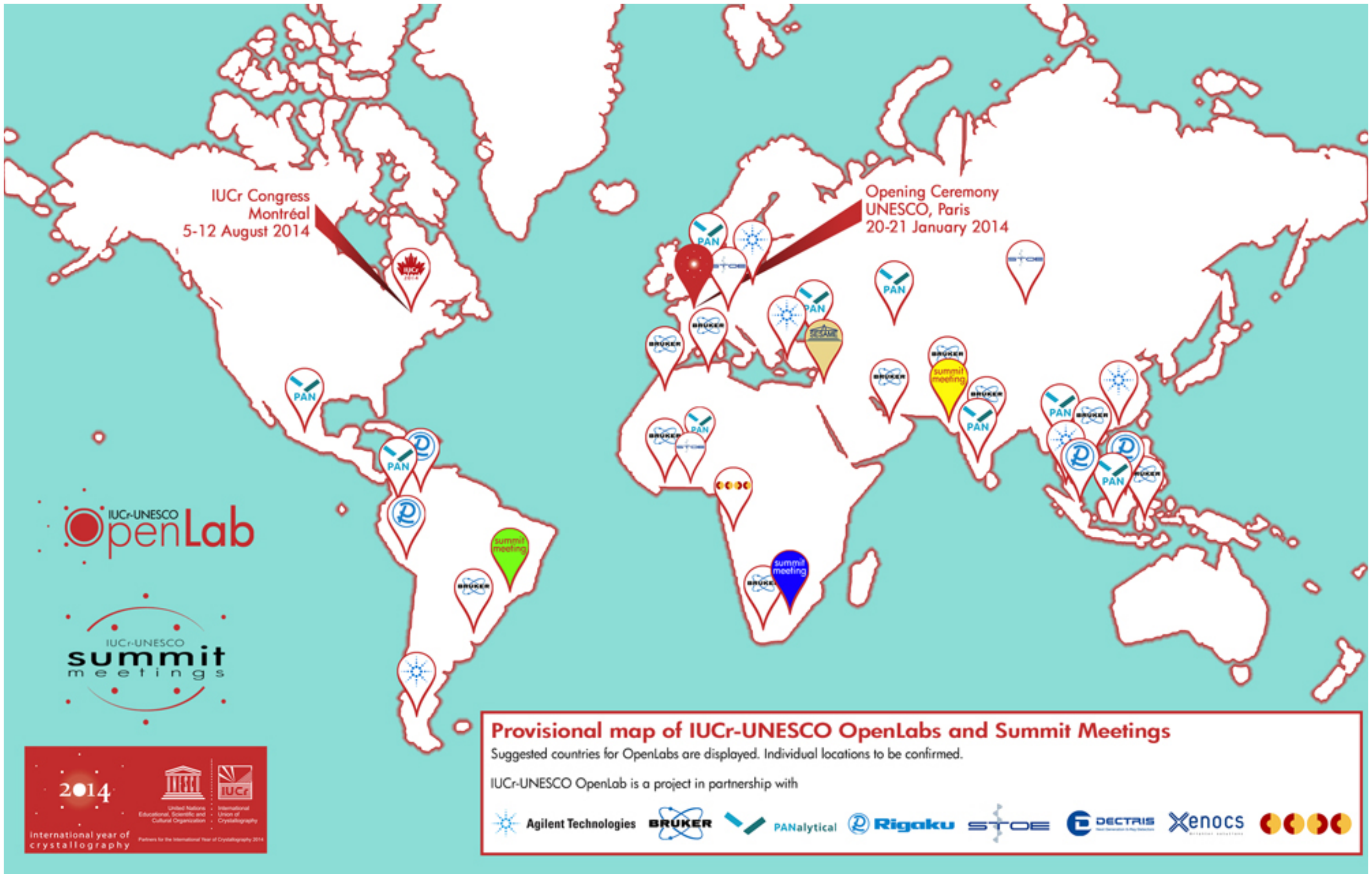
Two examples:

- open labs: UNESCO open factory
- Synchrotron in Jordan

Back to 2014 – Year of Crystallography

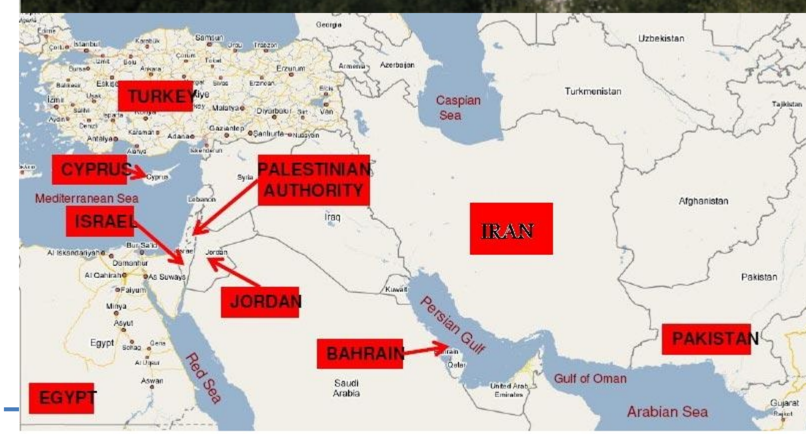
UNESCO OpenLabs

http://www.iycr2014.org/openlabs

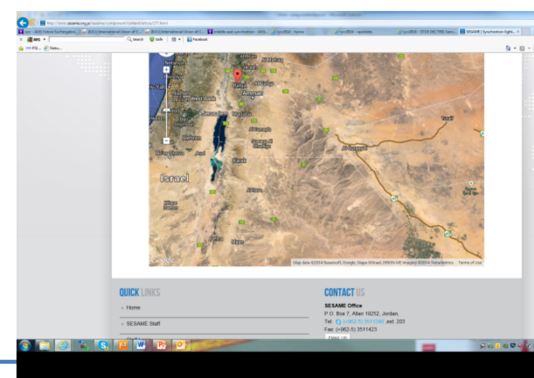


Synchrotron in Jordan: SESAME

www.sesame.org.jo , Al Balqa



SESAME Members



1912

2011

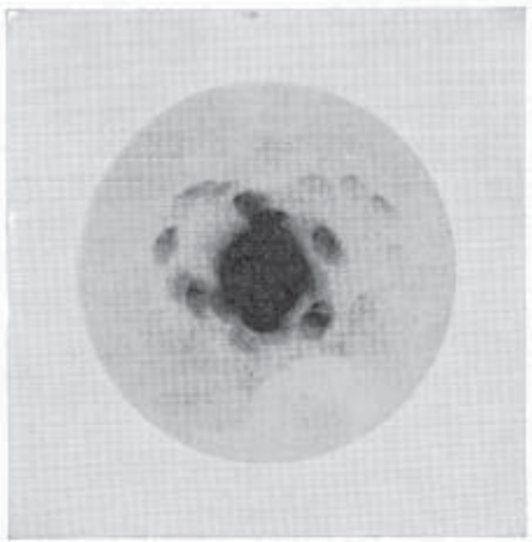
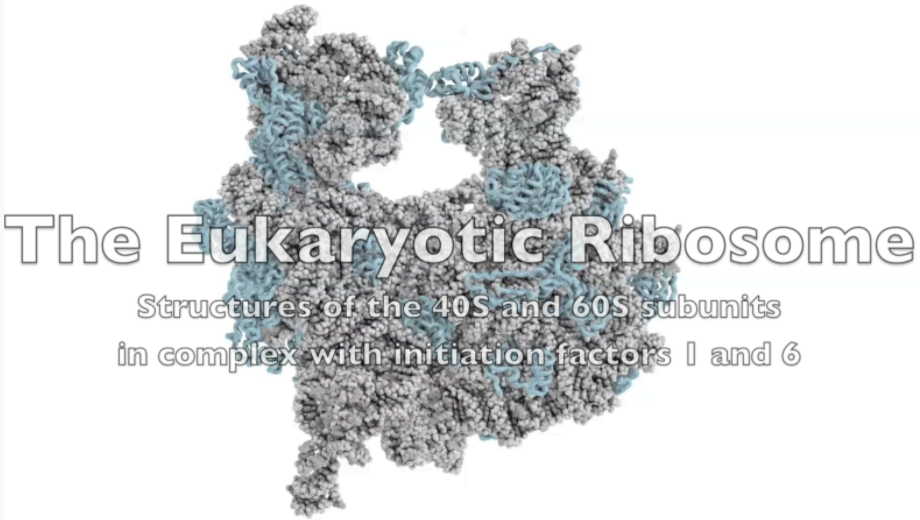


Fig. 4-4(1). Friedrich & Knipping's first successful diffraction photograph.



Ban et al.
http://www.mol.biol.ethz.ch/groups/ban_group/Ribosome

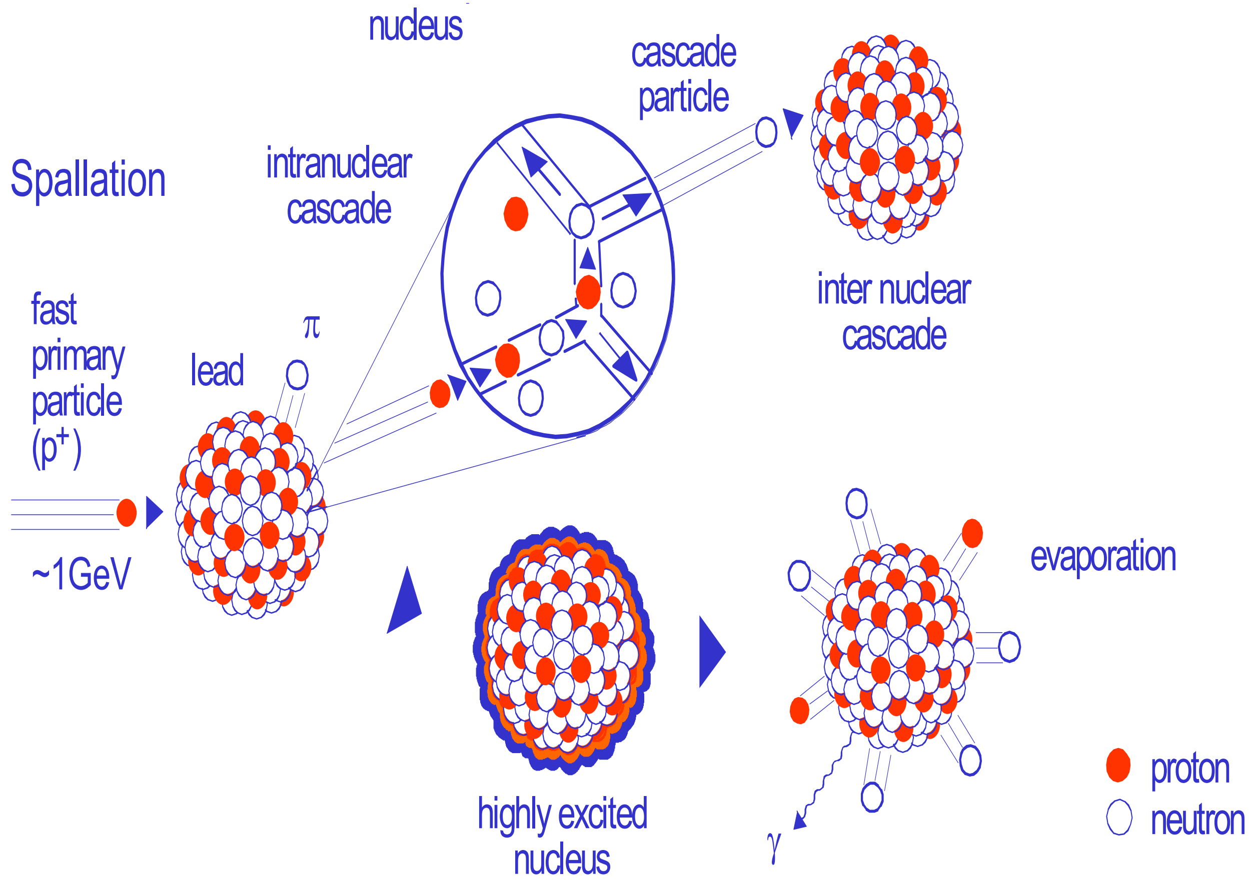
adds

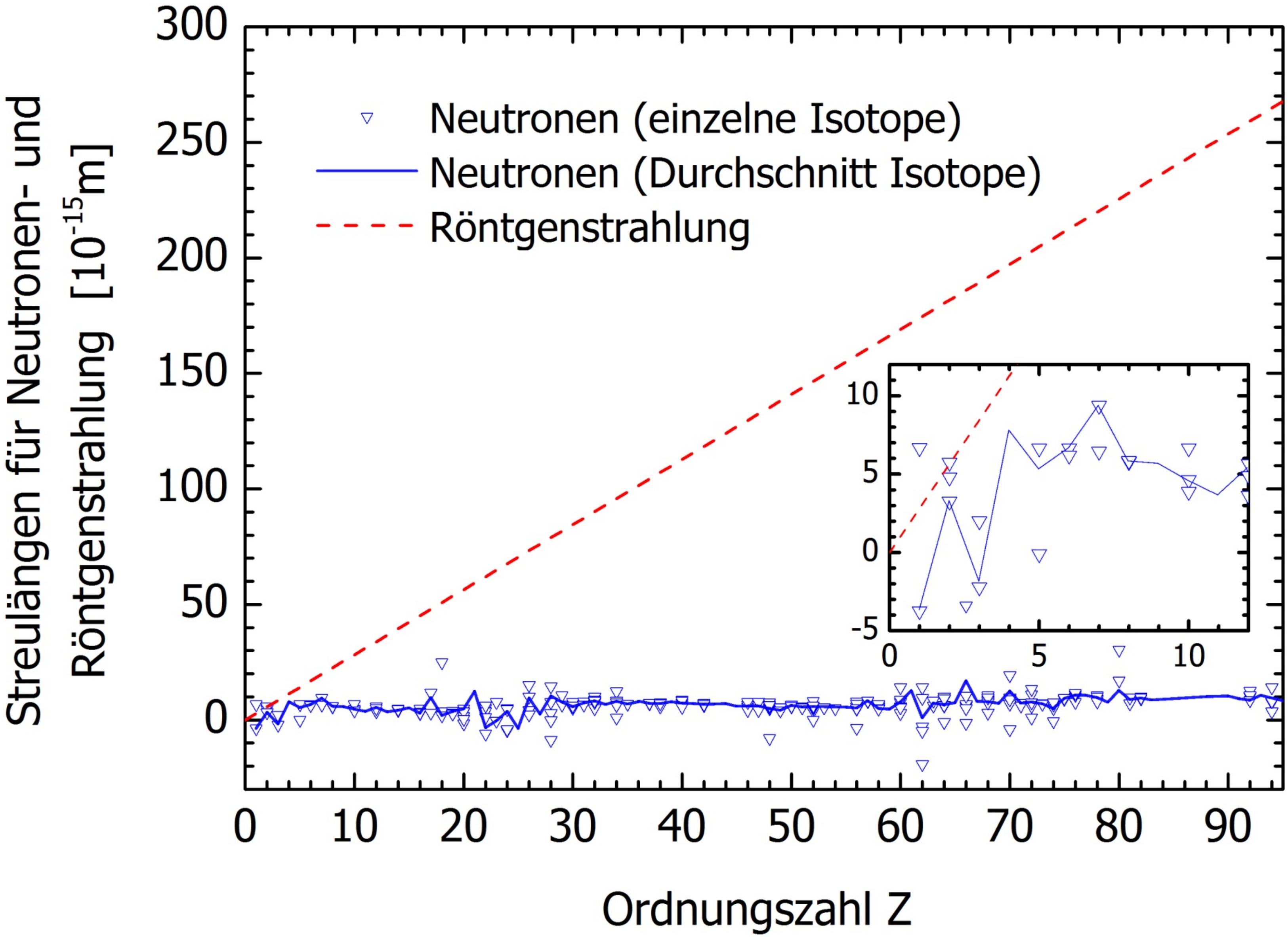
Art

crystallography is everywhere

ECM-2016: August 28 – Sept. 1
European Conference on Crystallography

Spallation Process: Heavy atoms (Pb, U, …) are “overheated” by high energy protons (> 500 MeV)




Neutronen (einzelne Isotope)
Neutronen (Durchschnitt Isotope)
Röntgenstrahlung
Streulängen für Neutronen- und Röntgenstrahlung [10^-15 m]
Ordnungszahl Z
300
250
200
150
100
50
0
10
20
30
40
50
60
70
80
90
5
-5