Generation and Use

of

Synchrotron Light

H. Wiedemann

Stanford University

Worldwide Facilities

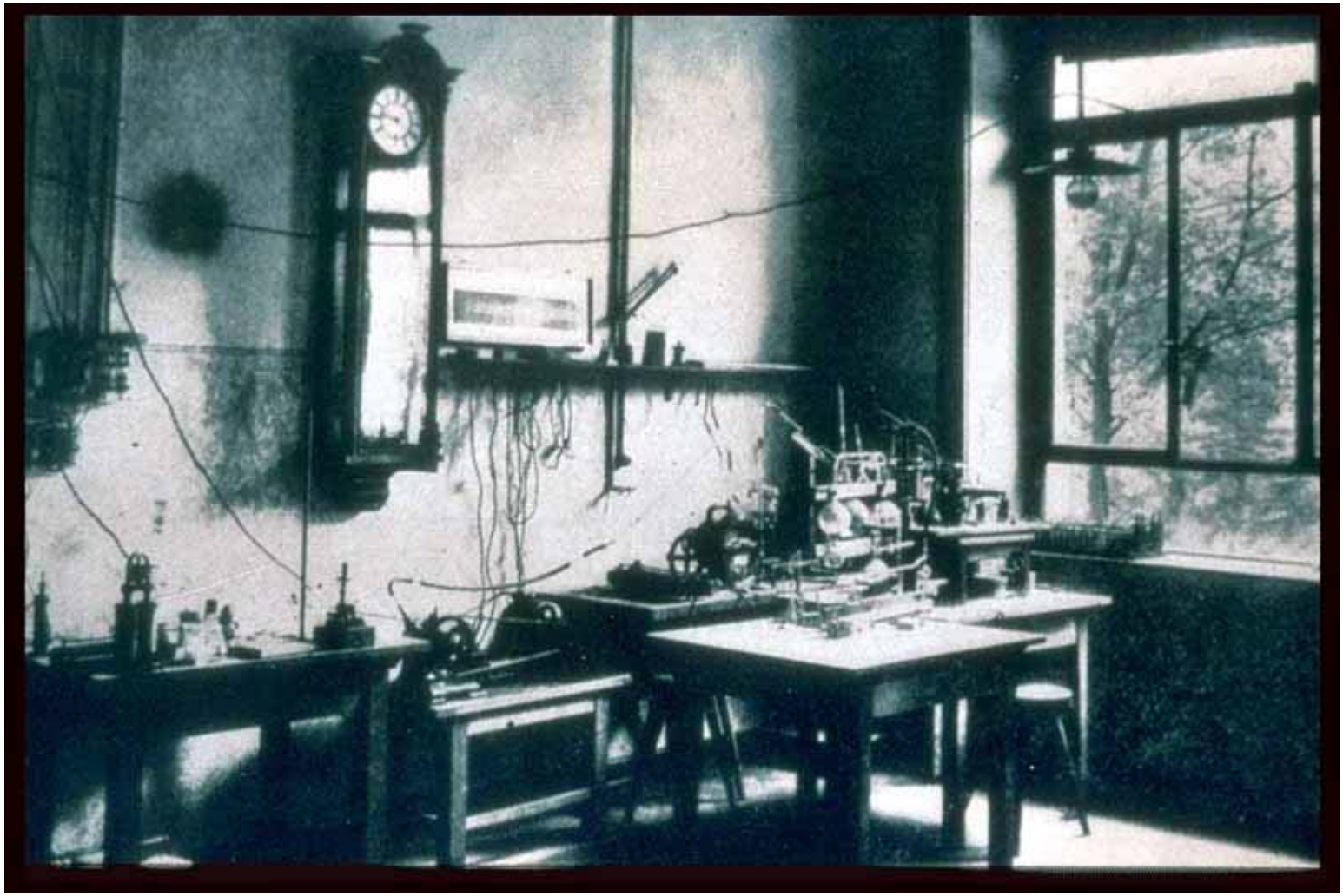
America:	13	[USA(11), Canada, Brazil]
Europe:	21	[Germany(6), France(2), England(2), Italy(2), Sweden(3), Russia(3), Denmark, Spain, Switzerland]
Asia:	20	[Japan(11), China(3), India(2), Taiwan, Korea, Singapore, Thailand, Armenia*, Jordan*]
Australia:	1	
Africa:		(thinking about it)

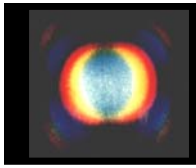
* proposal

what is
Synchrotron Light
and
how do we generate it?

Applications of Synchrotron Light

Roentgen's Laboratory, 110 years ago



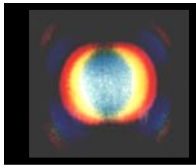


Applications of Synchrotron Light

to generate synchrotron light,
we need:

electron accelerator

transverse electric/magnetic field



Applications of Synchrotron Light

Properties of Synchrotron Radiation:

broad spectral band width: IR to hard X-ray

high collimation

polarization

high flux density

What can Synchrotron Radiation do for You?

- Synchrotron Radiation is rarely used to produce an end product
- Synchrotron Radiation is a diagnostic tool
- it helps to get insight into materials and its properties
- knowing the origin of material properties allows the development of new materials
- detection of trace impurities/pollution

Experimental Methods:

X-Ray absorption spectroscopy, XAS,
fluorescence at extreme sensitivity
microscopy on nano-scale, STM, PEEM
diffraction, scattering, SAXD, WAXD
protein-crystallography
micromachining, LIGA
.....

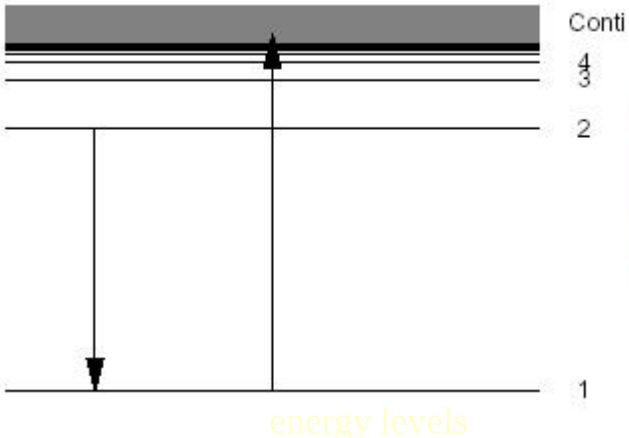
observation on atomic/molecular scale

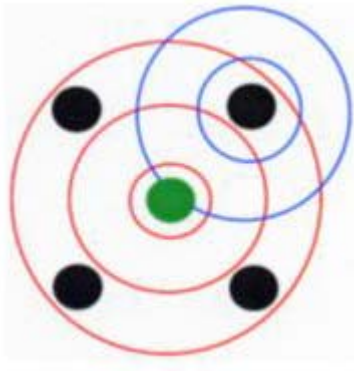
just a few examples

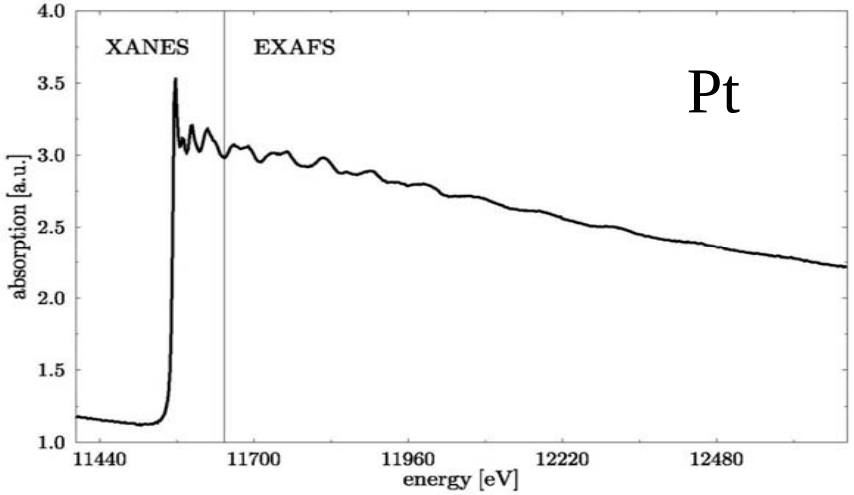
X – Ray Absorption Spectroscopy
XAS

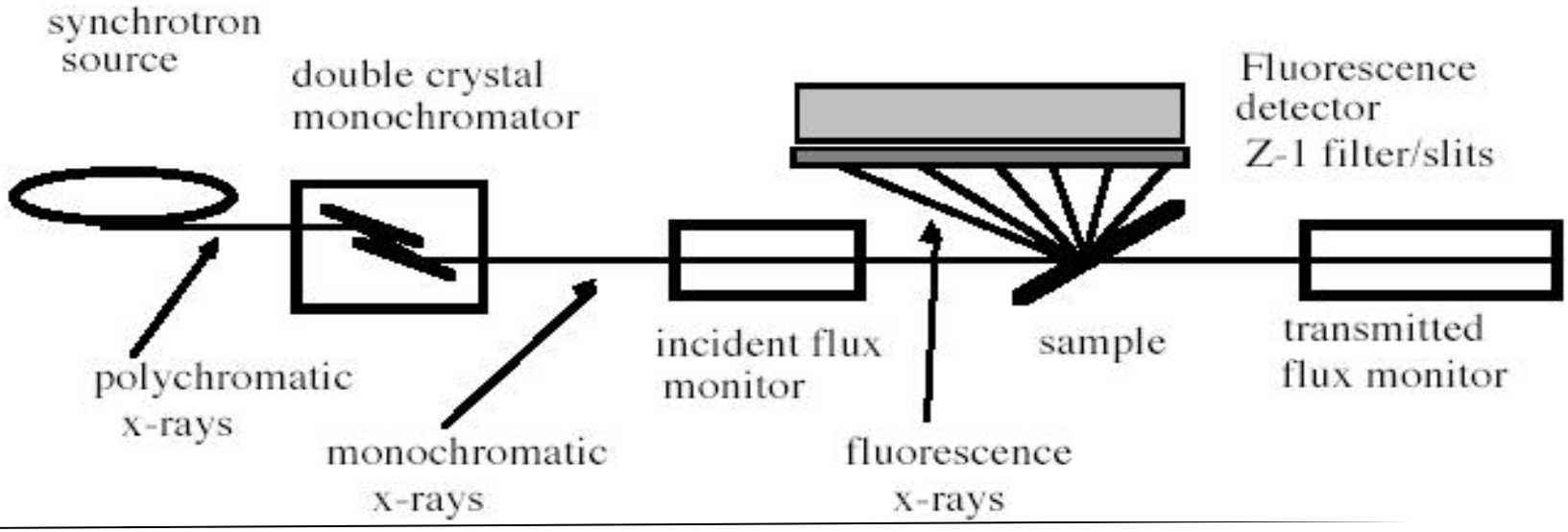
Applications of Synchrotron Light

X-ray absorption spectroscopy







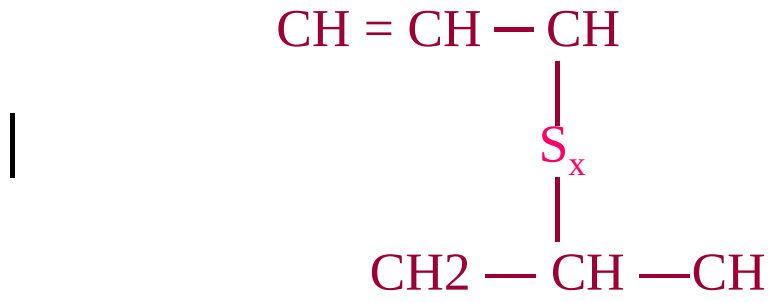


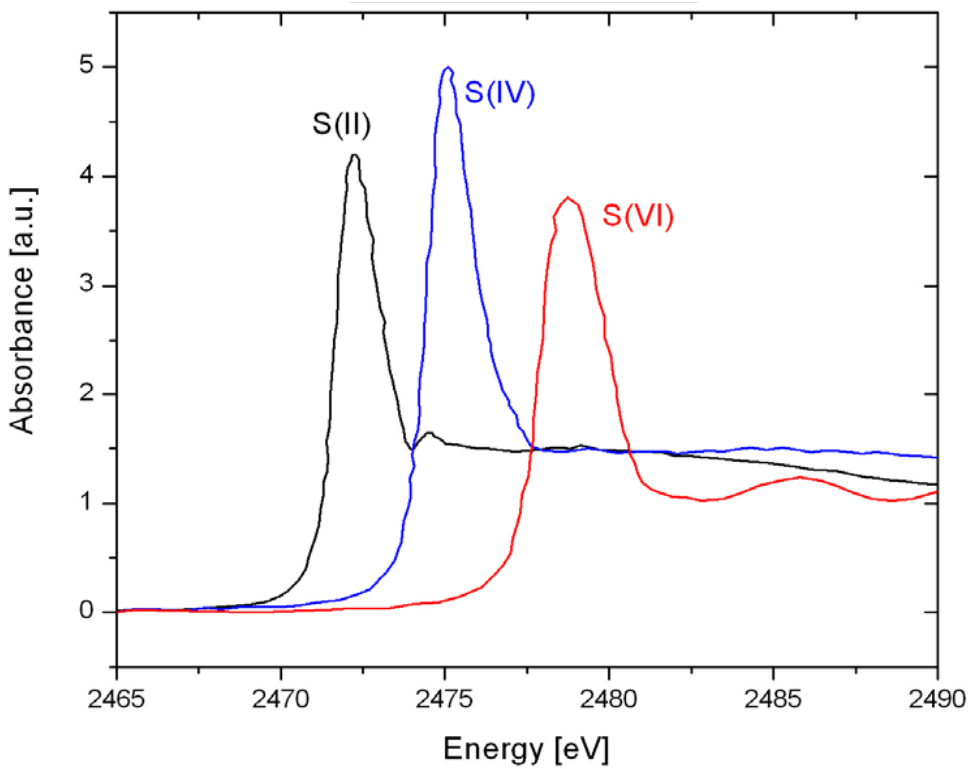
Structural Analysis with XAS:

Sulfur cross-linking in rubber (NR)

Problem:

The length of sulfur crosslinks determines the mechanical properties of rubber

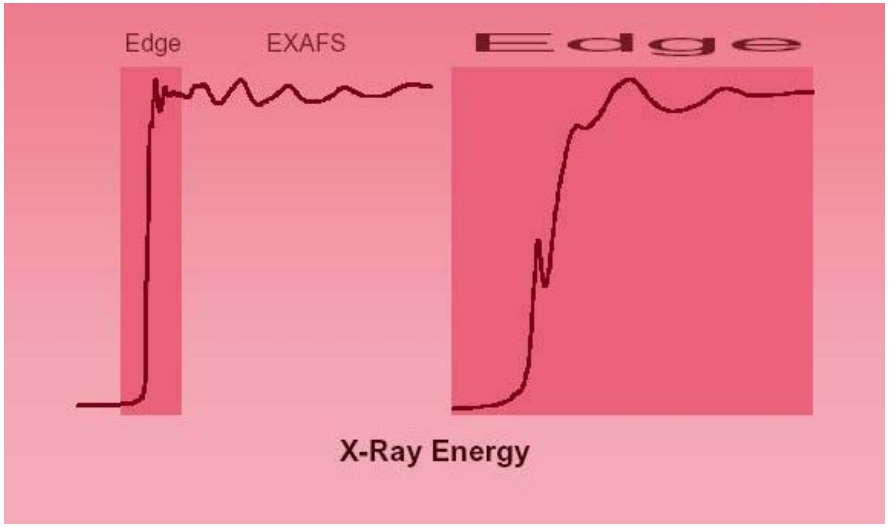




J. Hormes

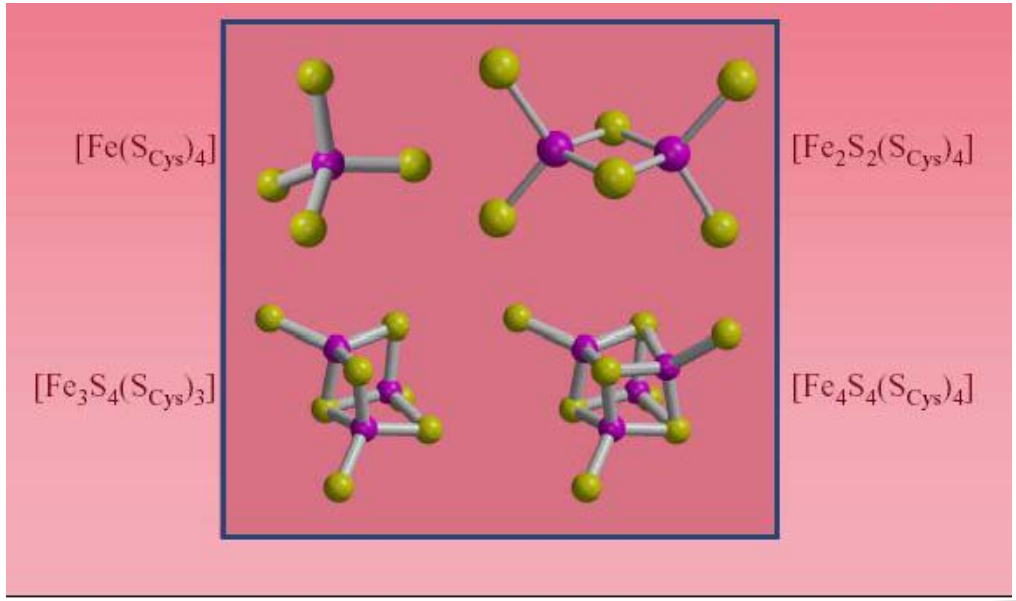
Applications of Synchrotron Light

Applications of Synchrotron Light



How many of what type of ligands are at what distance from metal?

Observable		Information
Frequency	→	Distance
Phase Shift	→	Type of Atom
Amplitude	→	# of Atoms



Applications of Synchrotron Light

Archeology

Figure 1: (**a**) Warrior # T18G21-08, a kneeling archer. The pigment samples in this study have been taken from this terracotta warrior. (**b**) Close-up picture of the purple paint on the terracotta warrior. (**c**) Images of the purple paint samples used in this study.

What is it?

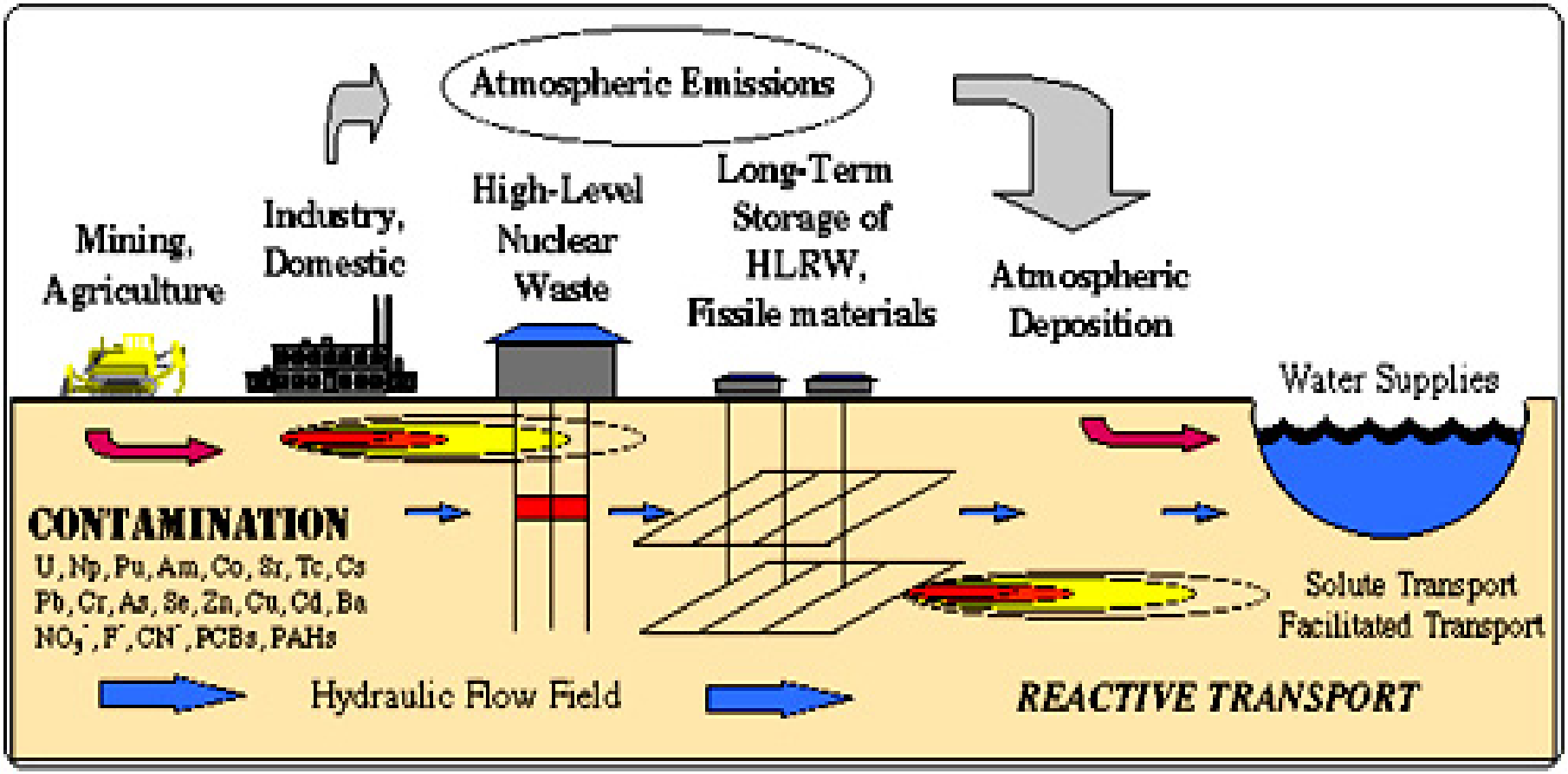
Egyptian Blue
($CaCuSi_2O_{10}$)

or

Chinese
Blue ($BaCuSi_2O_{10}$)
Purple ($BaCuSi_2O_6$)

Applications of Synchrotron Light

Environmental Science

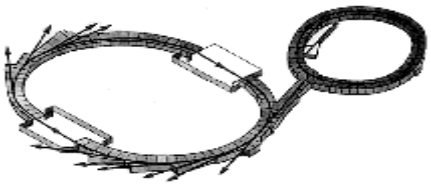


Environment and Health

- **heavy metals**: some necessary for good health, some detrimental, need to know species and oxidation state need to know in very **dilute samples** (XAS)
- contaminants in industrial dump sites (Cr^{+3}, Cr^{+6}.....)
- sewage treatment
- **phyto-remediation**: absorption of heavy metals by plants, e.g. Cd and Zn. (XAS, XANES)
- **migration** of pollutants (XAS, EXAFS)
- **waste incineration** – ash production/management (XAS)
- **waste filler in cement**, leaching out of metals (XAS)
- trace metals in food like **mercury in fish** (XAS)
- hair and skin products (reaction of skin/hair to product)

Applications of Synchrotron Light

Molecular Environmental Science and Synchrotron Radiation



Who cares about the distances between atoms?

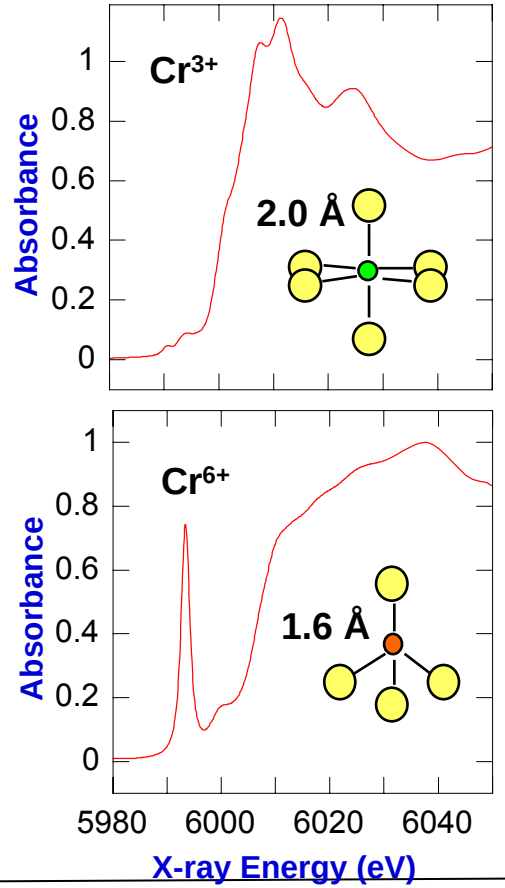
Just about everyone should, including your next door neighbor, because such distances define molecular structure which in turn defines function or properties in natural materials, including those occurring in the environment and in living cells. The molecular form or speciation of environmental contaminants, such as chromium, arsenic, lead, uranium, or plutonium, determines their toxicity and availability to organisms.

Molecular Environmental Science

- **Main objective is to provide information on the types, spatial distribution, and reactivity of contaminant species.**

Synchrotron Light Sources

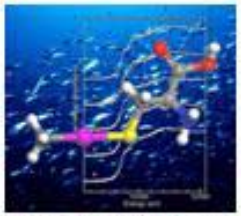
Now play a very important role in environmental science because of the extremely intense x-rays from these sources are needed to characterize the chemical speciation and physical distributions of environmental contaminants at very low concentration levels in highly complex materials.



Applications of Synchrotron Light

Stanford Synchrotron Radiation Laboratory

A national user facility for academia, industry, and national laboratories



view large image

Friday, 29 August 2003

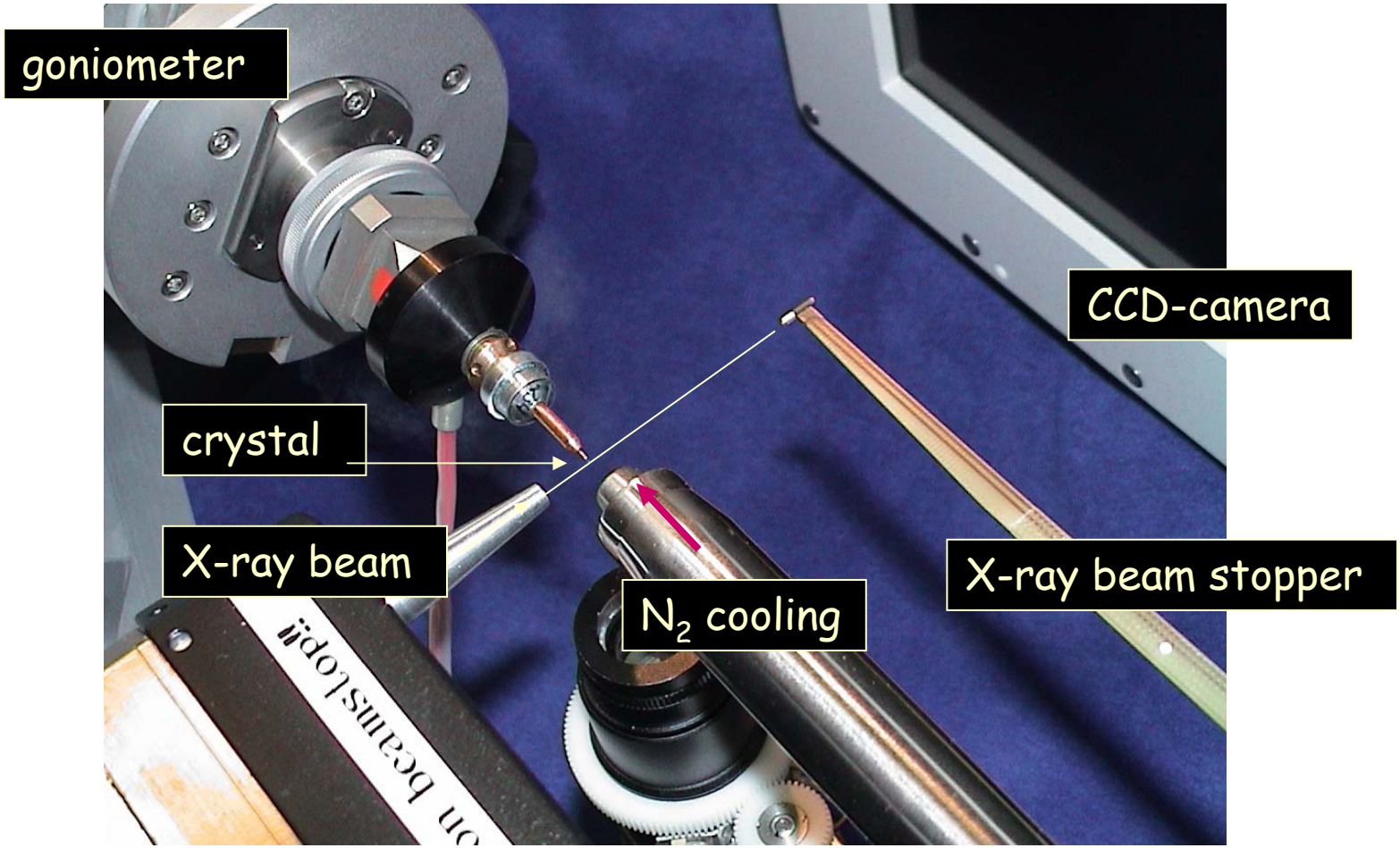
X-ray Absorption Spectroscopy Catches the Chemical Form of Mercury in Fish - SSRL Scientists Reveal New Findings in Science Article

The presence of "methyl mercury" in fish is well-known, but until now the detailed chemical identity of the mercury has remained a mystery. In an x-ray absorption spectroscopy study published in the August 29 issue of Science (Science 301, 2003: 1203; Science now: Murky Picture on Fish Mercury), SSRL scientists report that the chemical form of mercury involves a sulfur atom (most likely in a so-called aliphatic form). The study presents significant new knowledge - because the toxic properties of mercury (or any element) are critically dependent upon its chemical form - and represents an important milestone in developing an understanding of how harmful mercury in fish might actually be. The study was carried out by SSRL staff scientists Ingrid Pickering and Graham George and postdoctoral fellow Hugh Harris using SSRL's structural molecular biology beam line 9-3. The very high flux, excellent beam stability and state-of-the-art detector technology allowed the team to measure samples of fish containing micromolar levels of mercury, much lower than had previously been possible.

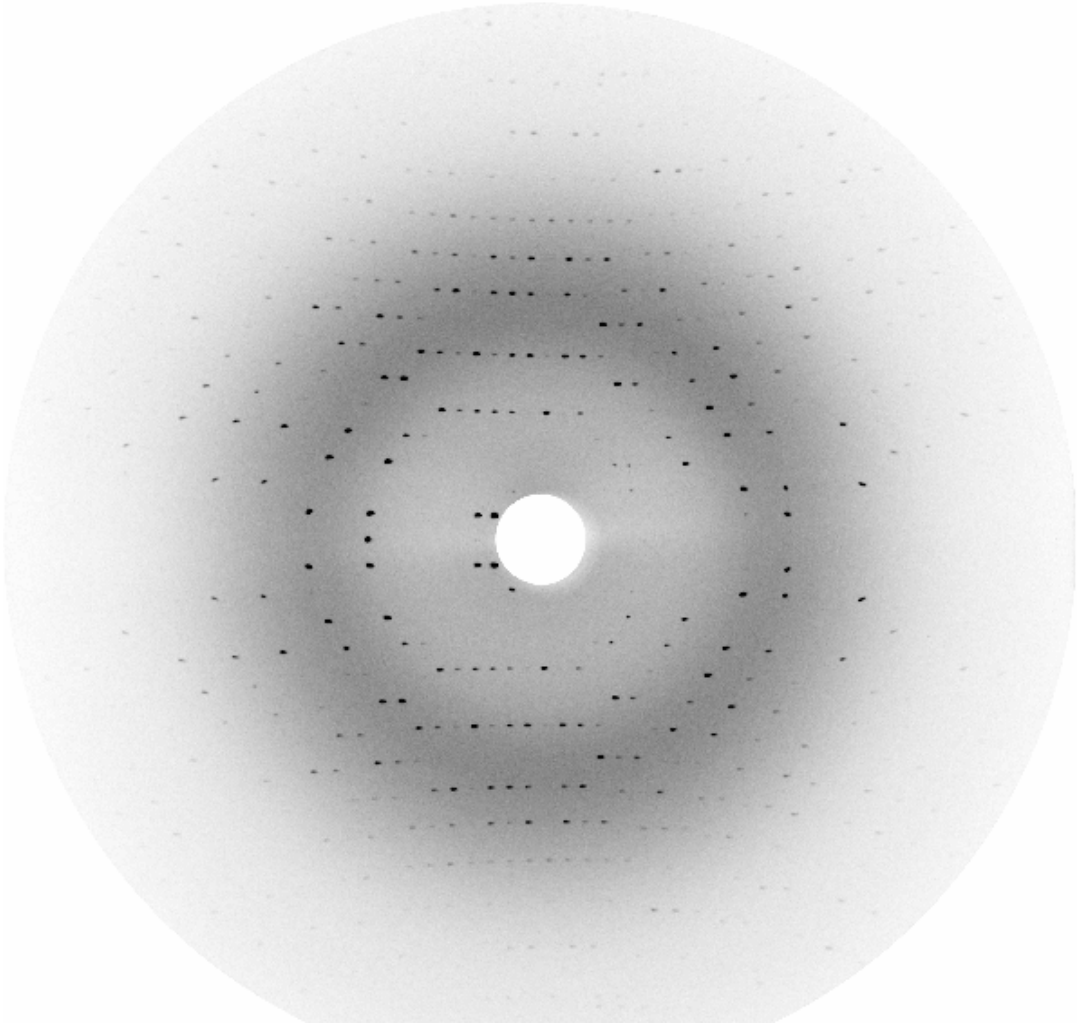
Protein Crystallography

- atomic **structure of biological macromolecules**
- functions of **enzymes, virus infections**
- crystallographic scattering technique applied to proteins (Diff. & Scat.)
- **drug development**, inhibitors, structure-function relationships
- structure of a bacterial protein that contributes to the ability of bacteria to become resistant to life-saving antibiotica

Protein Crystallography



Applications of Synchrotron Light



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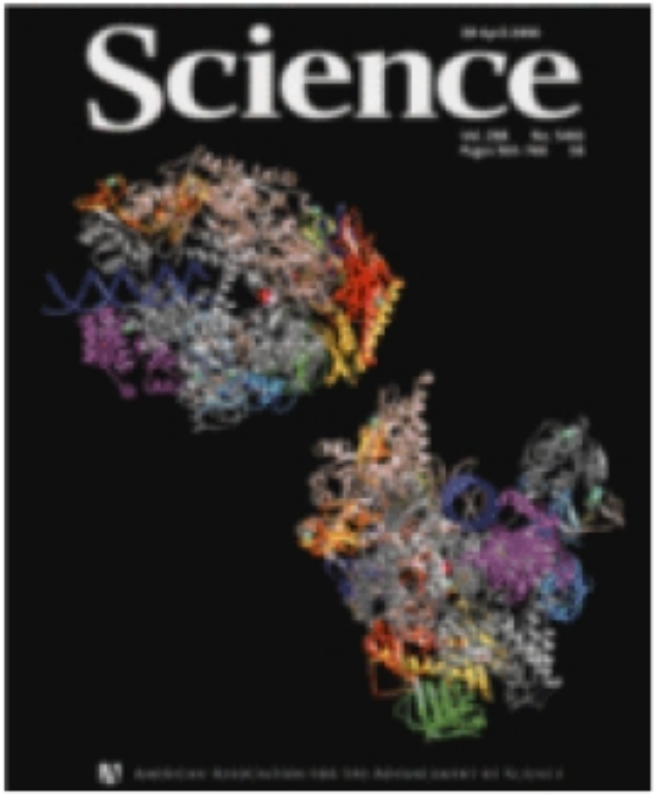
Applications of Synchrotron Light



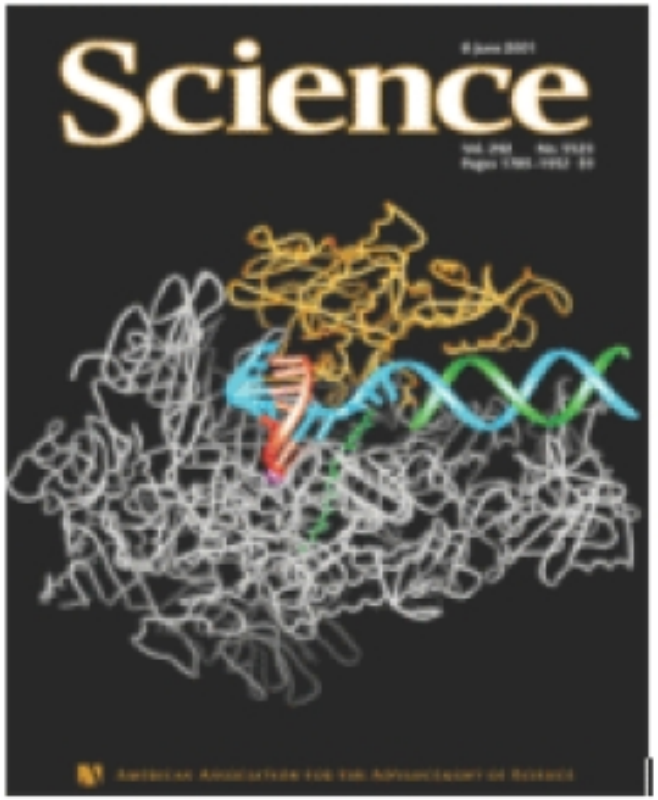
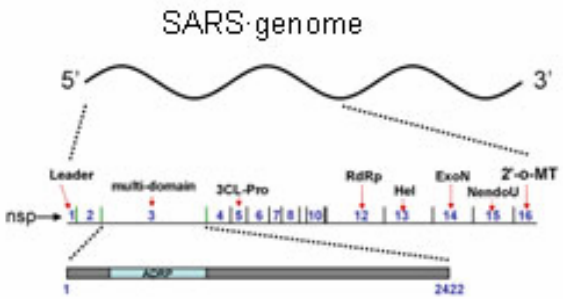
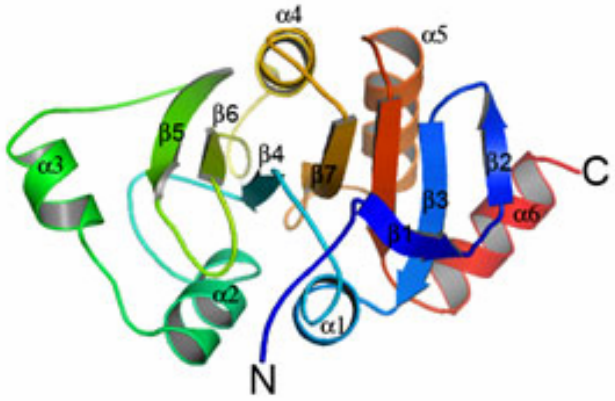


Fig. 1. The structure of RNA polymerase II (left) and RNA polymerase II in the act of transcription (right) featured on covers of *Science* Magazine. During transcription, a template strand of DNA (shown in blue on the right) is unwinding just before the active center. Newly formed RNA is shown in red.

Applications of Synchrotron Light

R. Kornberg, Stanford University, Chemistry
Nobel Prize in Chemistry 2006





SARS

Just starting to characterize
the structure and function of all the proteins
built or used by SARS

The Scripps Research Institute in California

Saikatendu KS, Joseph JS, Subramanian V, Clayton T, Griffith M, Moy K, Velasquez J, Neuman BW, Buchmeier MJ, Stevens RC, Kuhn P (2005). Structural basis of severe acute respiratory syndrome coronavirus ADP-ribose-1''-phosphate dephosphorylation by a conserved domain of nsP3. Structure. 13, 1665-1675.

Applications of Synchrotron Light

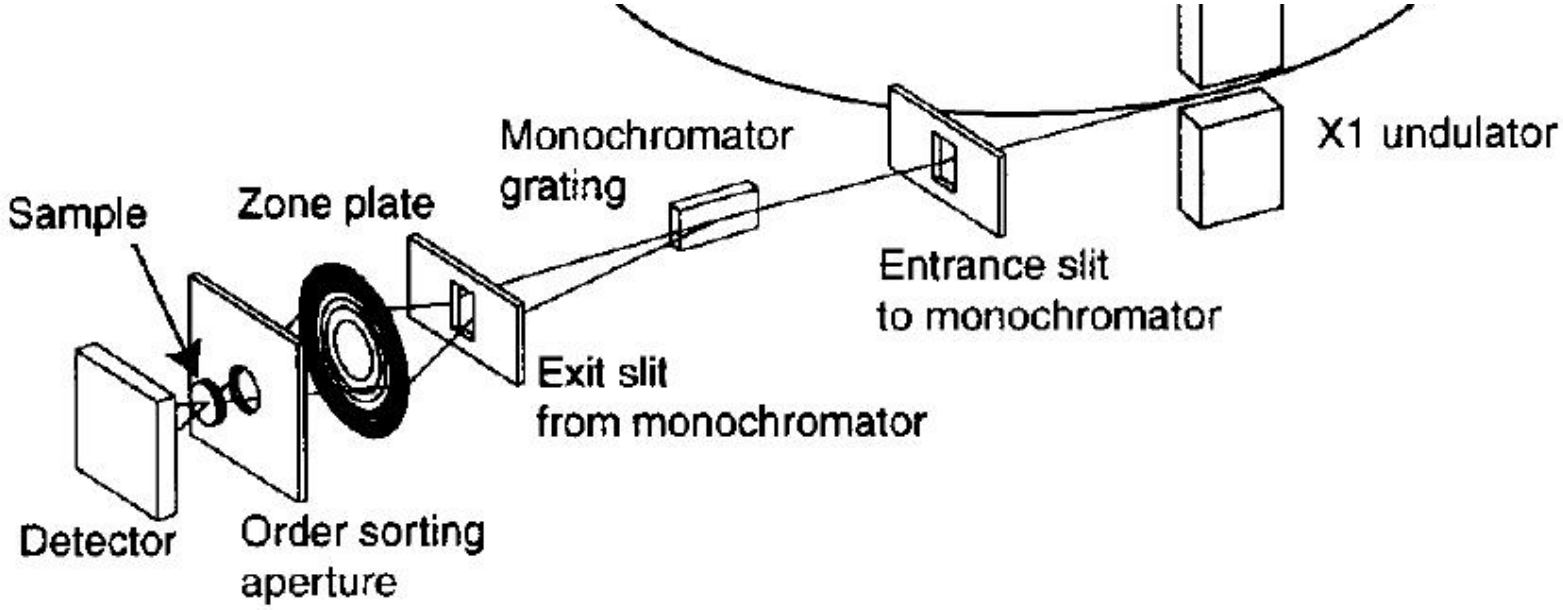
Myoglobin.mov

Scanning Transmission X-ray Microscope

materials of commercial significance
are usually
multi-component systems elastomers and fillers

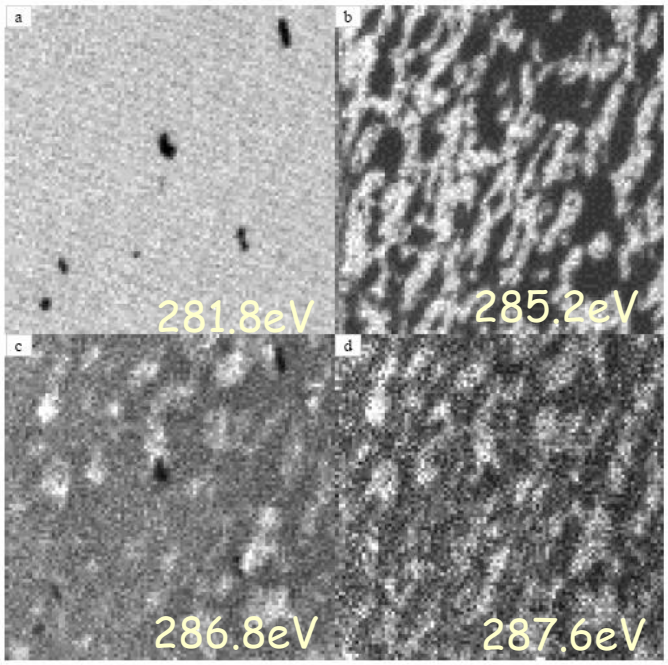
how does morphology from blending affect material properties?

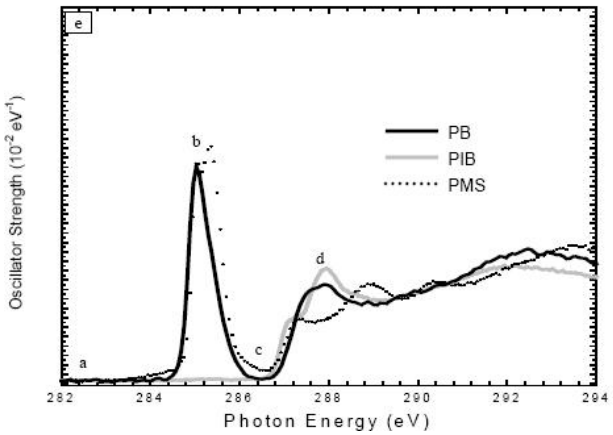
suitable approach with synchrotron based STXM
with chemical specificity of NEXAFS (Near Edge X-ray Absorption Fine Structure)

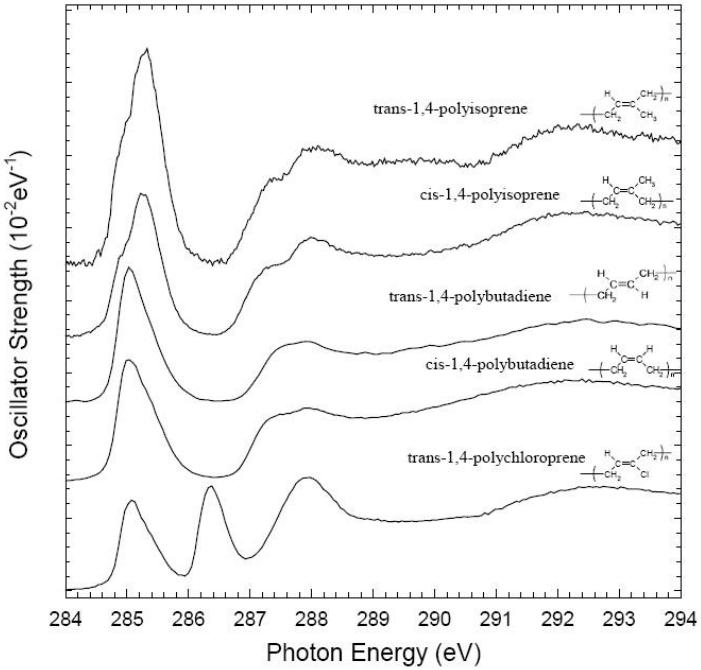


Applications of Synchrotron Light

micrograph of sample at four different energies







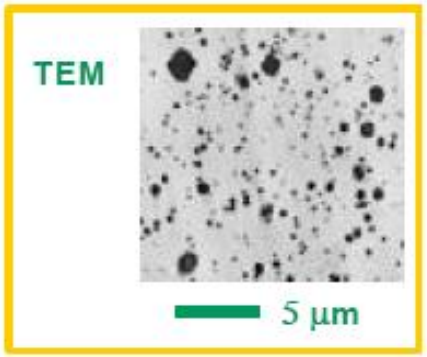
reference spectra

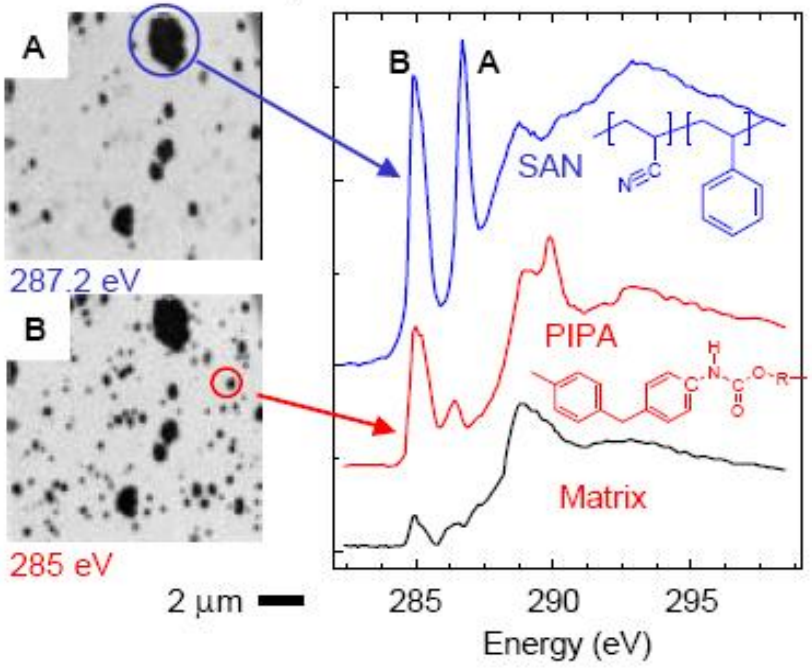
D.A. Winesett et. al.

Chemical Sensitivity of Polymer NEXAFS

Chemical Identification of Polymer Fillers

Sample #355: Polyurethane polymer containing styrene acrylonitrile and PIPA (aromatic polyurethane) filler particles





- NEXAFS features provide a "chemical contrast mechanism" to distinguish fillers
- Spectra extracted from polymer fillers can be used for chemical identification

Sample: E.G. Rightor, Experiment: A.P. Smith, H. Ade, SUNY-SB STXM

Stephen Urquhart (Univ. Saskatchewan) 18 Jan. 2000

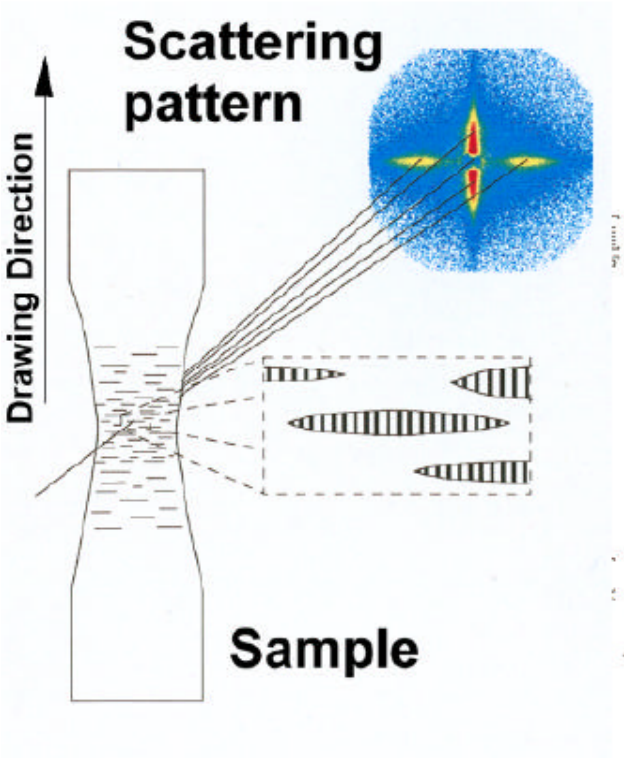
Research on Rubber/Polymers/Fibers/Plastic

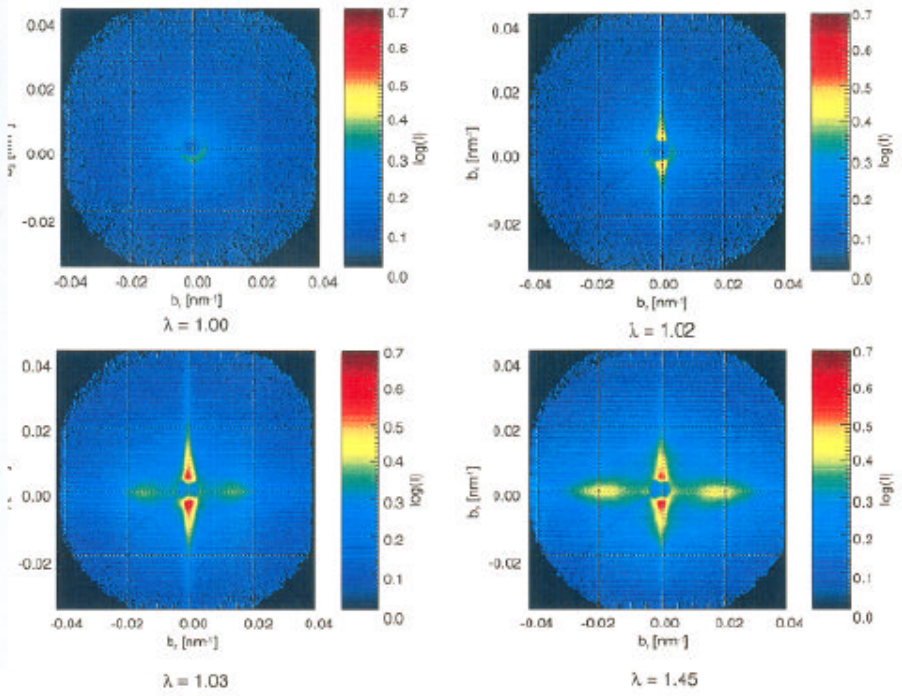
correlate **vulcanization with mechanical properties** (XAS)

- understand **cross linking** and **crystallization**
  in rubber, silk, polymers (**SAXS, WAXS**)
- **fiber processing**, structure/property/process relationship
  in situ studies with SAXS,WAXS during fiber extrusion
  properties of final fiber is strongly influenced by manufacturing)
- **molding** of plastics **and mechanical properties** (STXM)
- **fracture mechanisms in fibers** (STXM, in situ SAXS, WAXS)
- **rubber/plastic blend** process/property relationship (STXM, SAXS,WAXS)
  microscopy with <100 nm resolution and chemical analysis
- **goal: stronger, lighter, cheaper**

Applications of Synchrotron Light

Craze formation in Polycarbonate





Draw ratio $\lambda = l/l_o$

T=130°C, $d\lambda/dt = 7.2 \cdot 10^{-5}$

In-situ Synchrotron Study of Fiber Processing

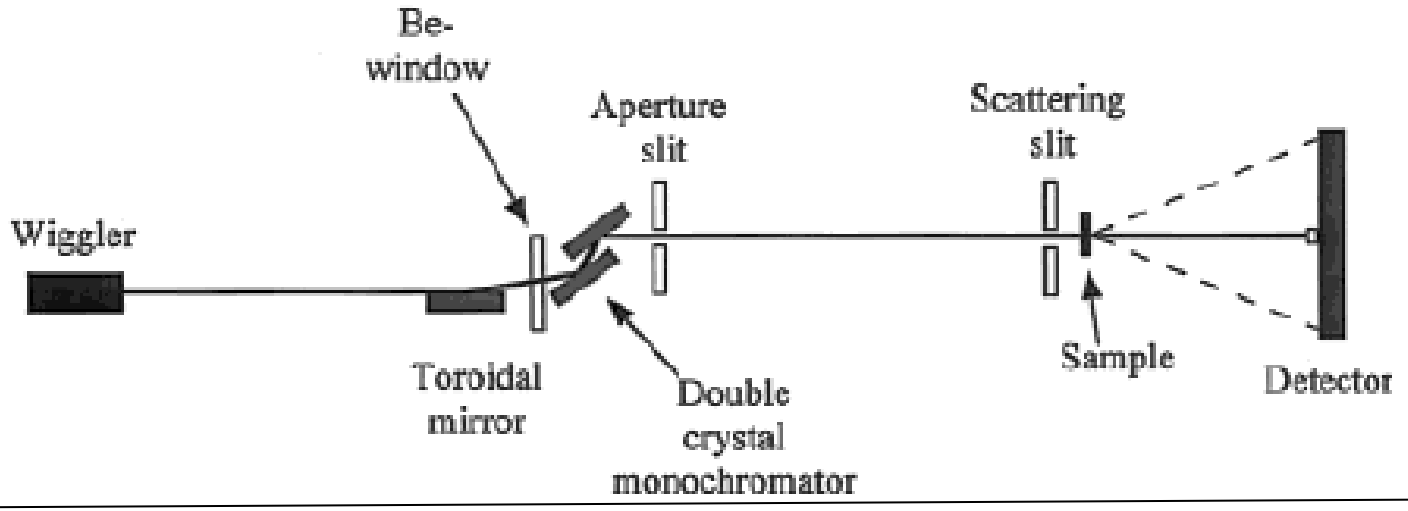
Objective: identify spinning conditions to produce strong, dyable fibers in high speed spinning and to engineer specific fiber morphology and properties for lower spinning speeds

(1) Identify evolution of intermediate structures during fiber spinning and relate with properties of resulting fiber

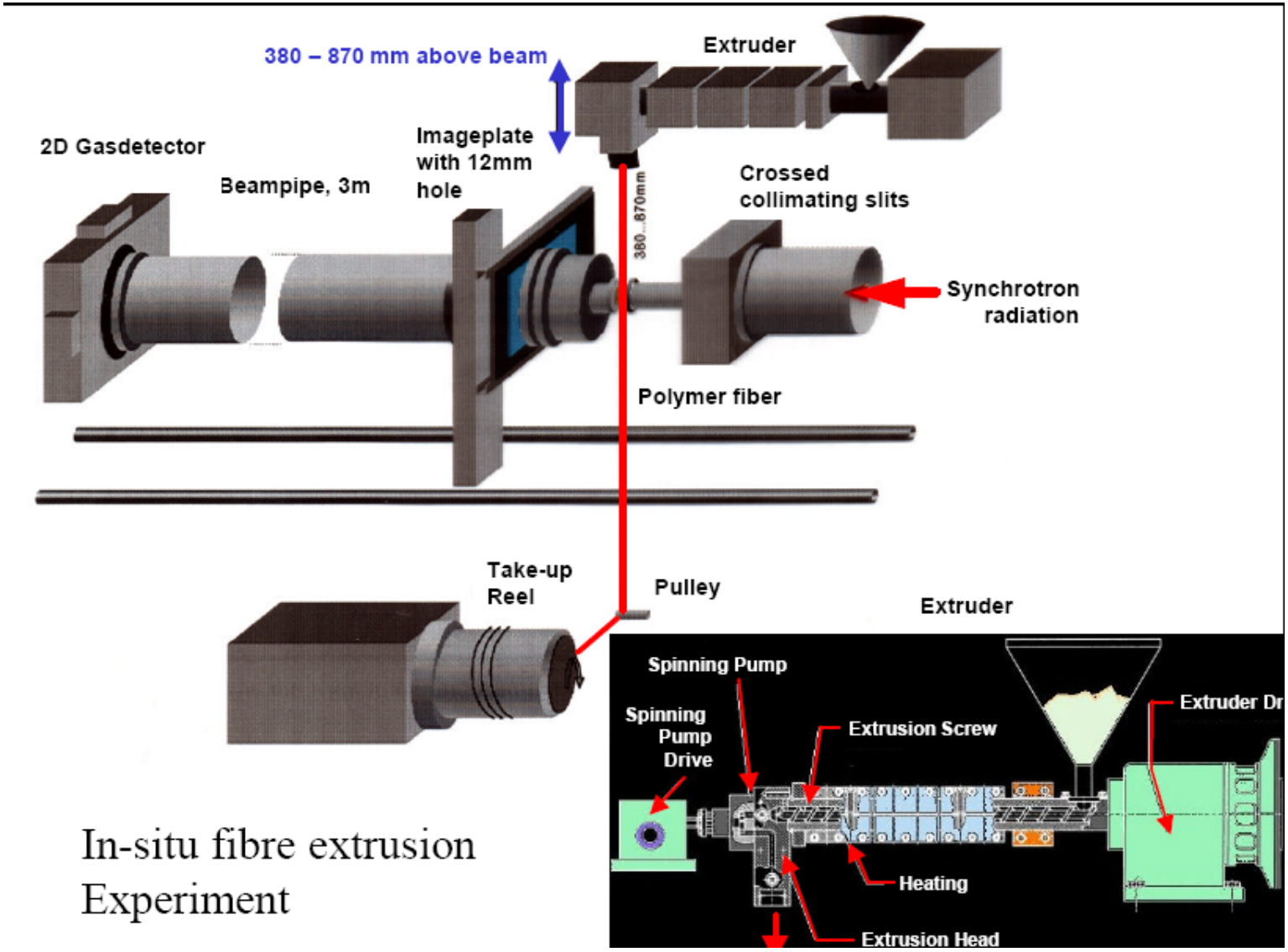
(2) Understand crystalinity and orientation with objective of engineering fibers by modifying spinning conditions

(3) Develop computer modeling techniques to predict structure and morphology

SAXD/WAXD beam line

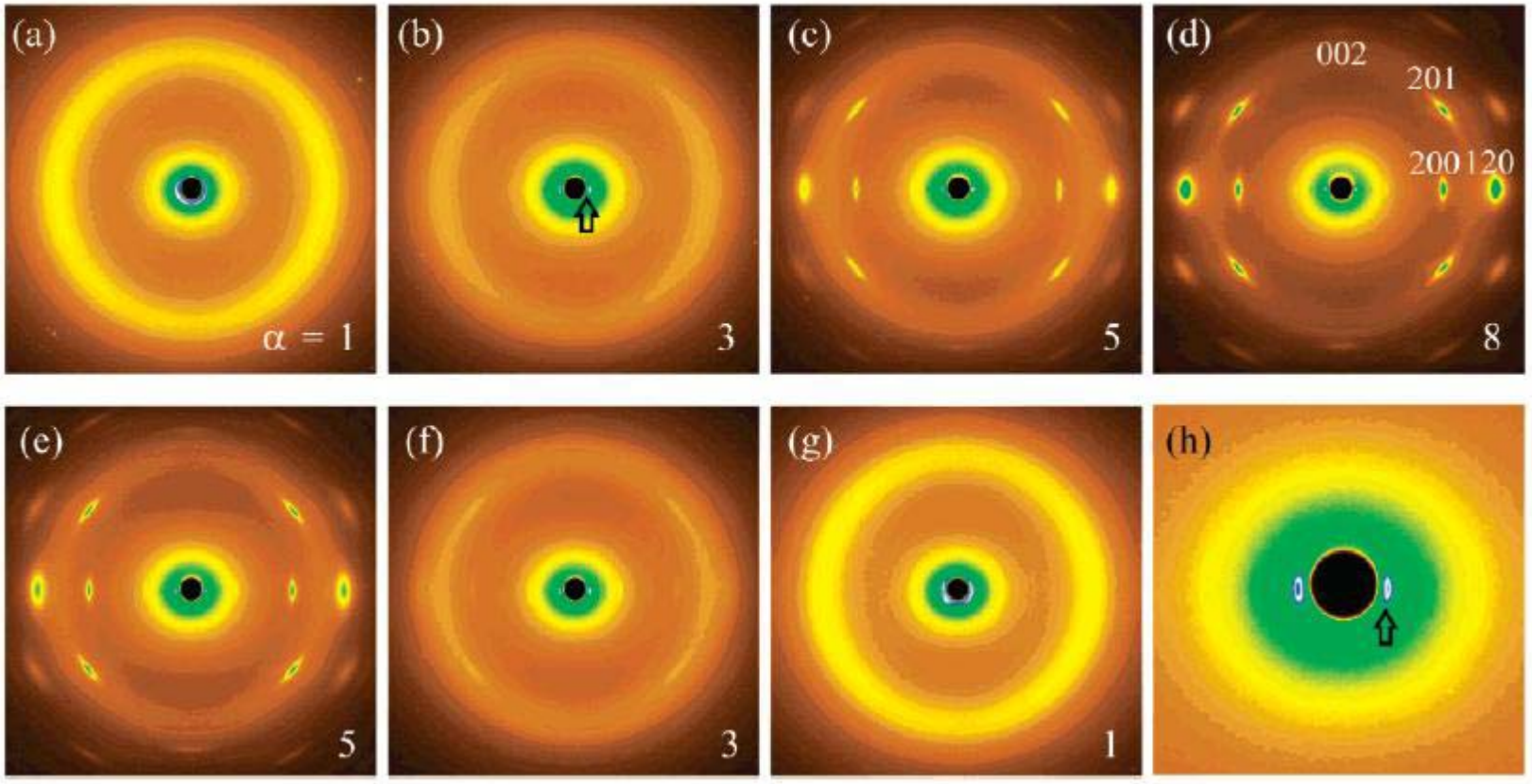


Applications of Synchrotron Light



In-situ fibre extrusion Experiment

Diffraction



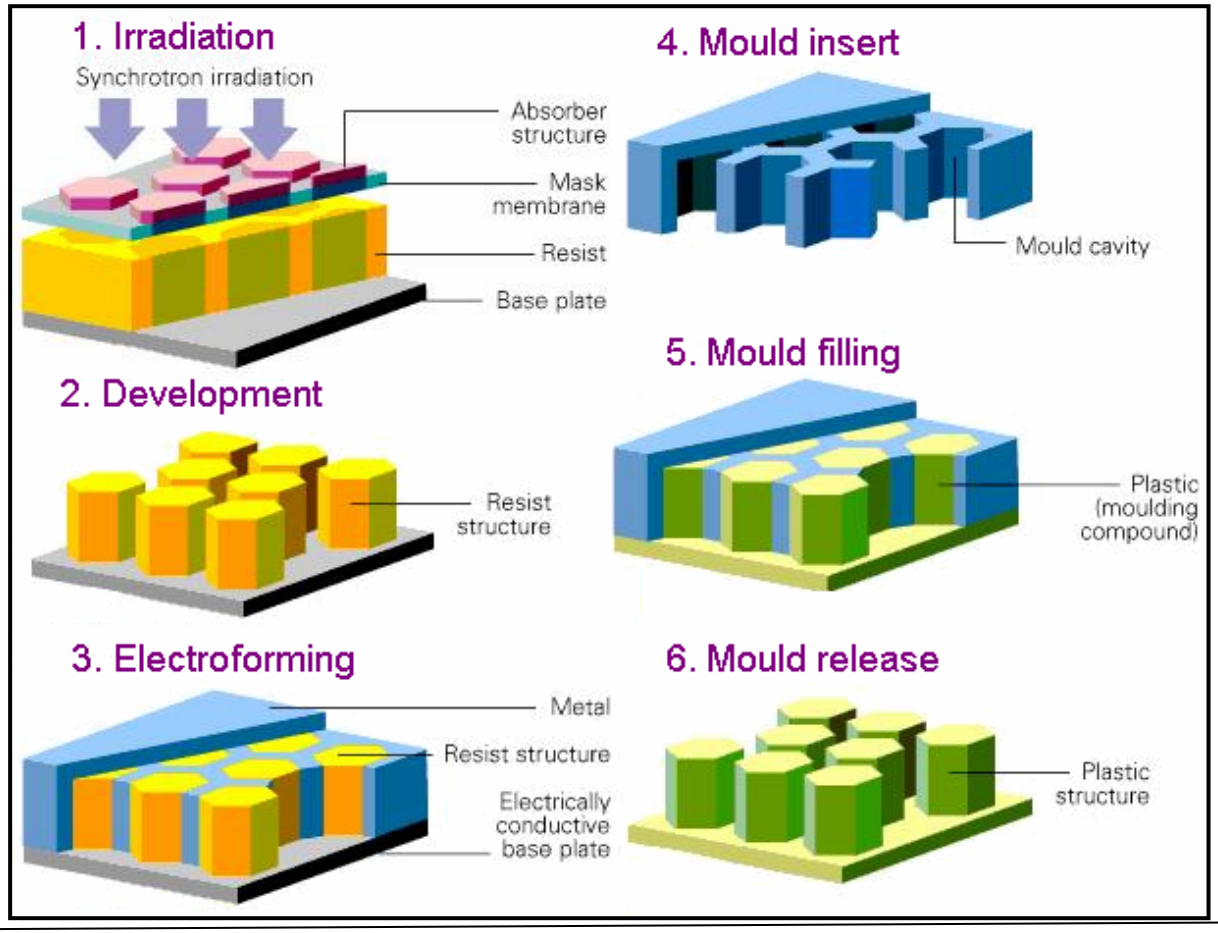
Tosaka, et. al. Macromolecules 2004, 37, 3299

Deep X-ray Lithography

- high aspect ratio mechanical systems
- new enabling technology
- used to mass produce devices
-
  - **humidity sensors**
  - **breath analyzers, sensor in air conditioner**
    pneumatic Braille display for the blind
  - **micro-switches** in electronic/electrical appliances
  - **lab-on-a-chip**
  - .....

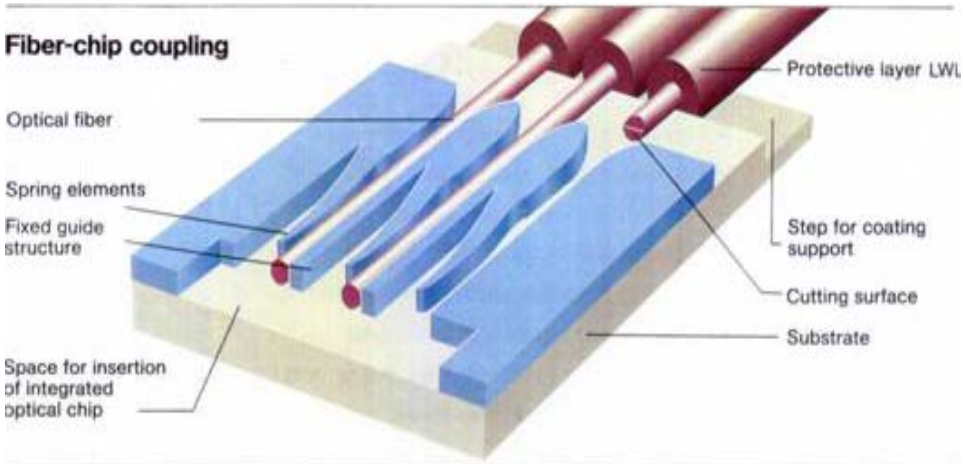
Applications of Synchrotron Light

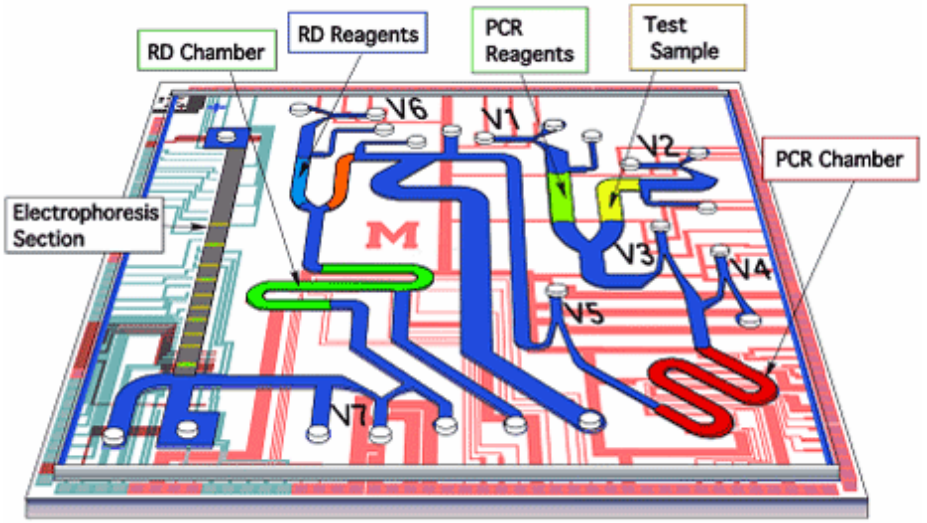
LIGA is the German acronym for X-ray lithography (X-ray Lithographie), Electroforming (Galvanoformung), and Molding (Abformung).

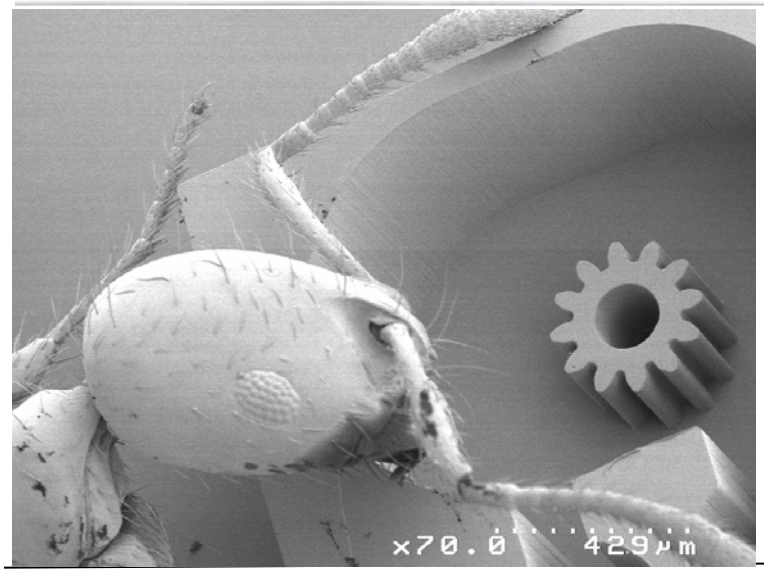


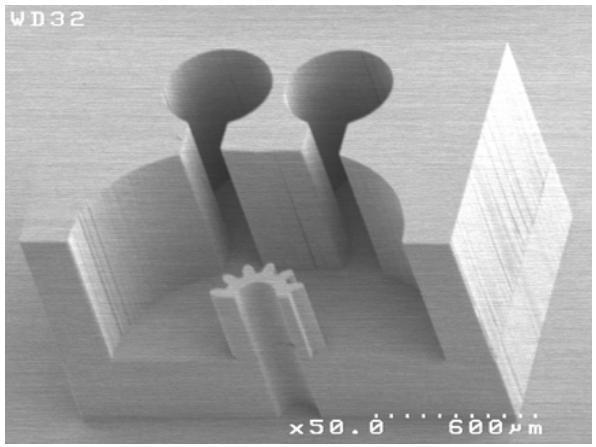
Applications of Synchrotron Light

Micro Machining - LIGA







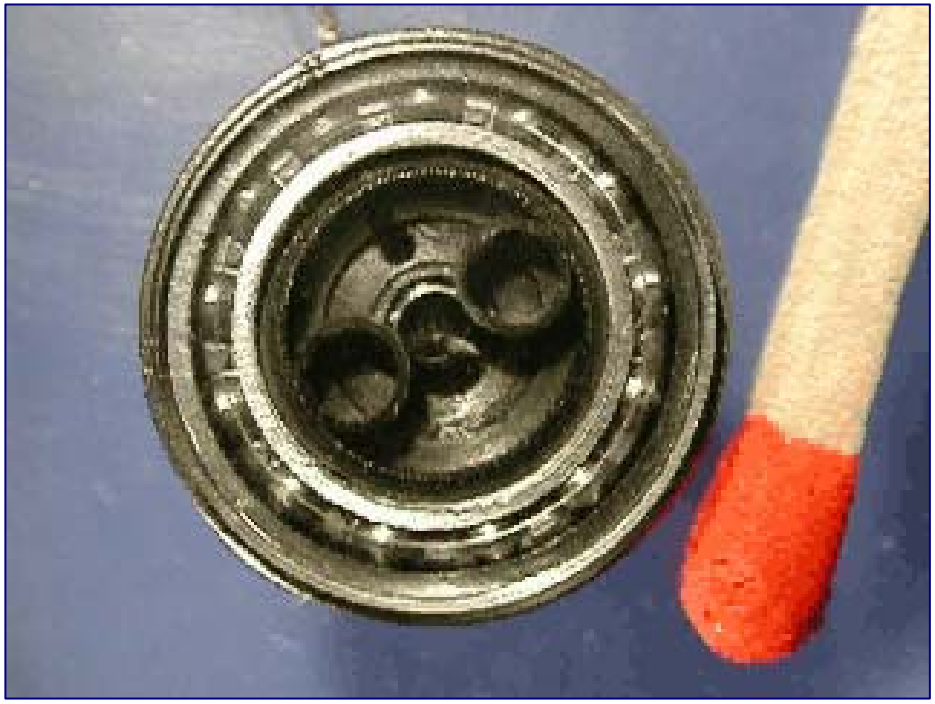


Applications of Synchrotron Light

Size comparison with a bee

Micro Harmonic Drive®

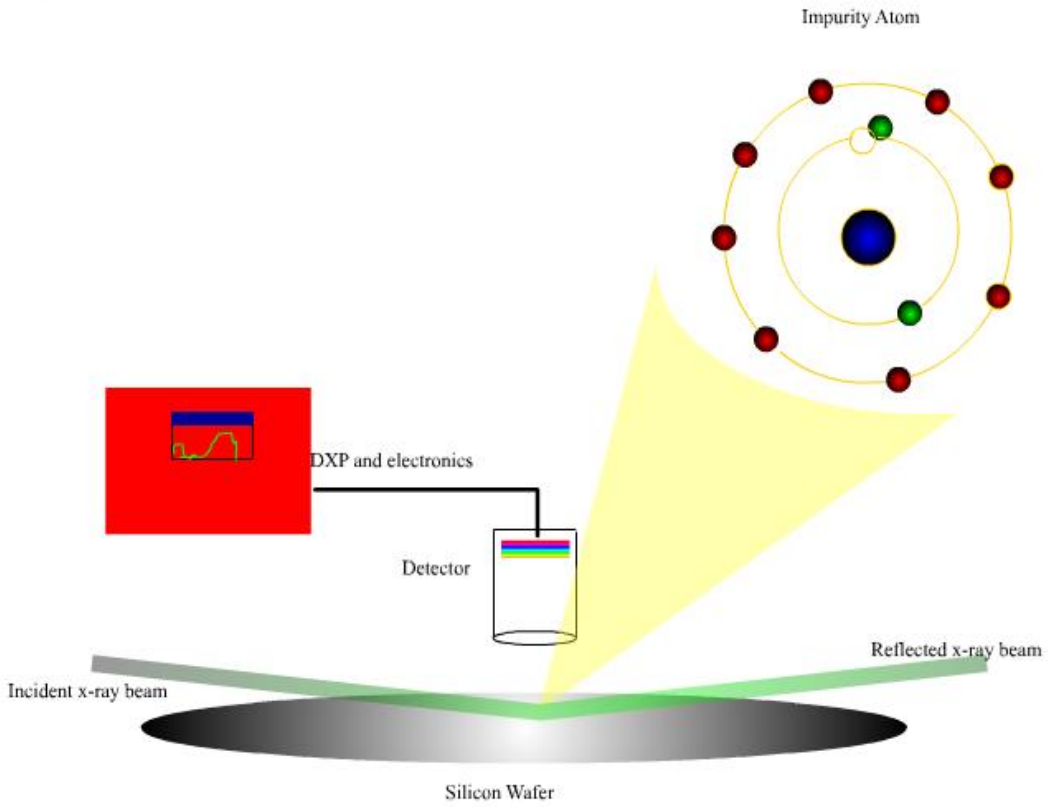
Size comparison with a safety match



Applications of Synchrotron Light

Clean Semiconductor Wavers

how clean can we make them ?



Industrial Collaborators

- Advanced Micro Devices
- Applied Materials
- ARACOR
- Balazs Analytical Lab
- Charles Evans and Associates
- Digital Equipment Corp
- Dow Chemical
- Hewlett-Packard (IMEC, Toshiba)
- IBM
- Intel
- Komatsu America
- Lucent
- Motorola
- National Semiconductor
- Texas Instruments
- Wacker (Atominstitut)

Applications of Synchrotron Light

1. Food
2. Automobile
3. Microchips
4. Polymers/Fibers/Plastics/Rubber
5. Health
6. Environment
7. Materials
8. Biotechnology
9. Micromachining (Lithography)

1. Food

- **trace metal in food** (e.g. Mercury in fish, pollution)
- agricultural biotechnology, plant proteins under stress (heat, cold, draught, salty soil etc.)
- bioengineering,
- breeding of plants for digestible animal food
- develop **disease resistant plants**
- improve taste (e.g. chocolate polymer processing) chocolate melts into 6 crystalline forms, only one tastes good
- food packaging
- food storage, processing

2. Automobile

- **Catalyst**, study molecular structure and change during process
- **Corrosion**
- strong, durable, light **plastics** (polymer research)
- hydrogen adsorption in nano-channel polymers (**H_2-storage**)
- XAS research on rubber, what determines mechanical property? etc
- bio-fuel (e.g. study melting process of solids, melting of rice?)
- MEMS (micro-electro-mechanical systems),
  enabling technology based on micromachining
- micromachining (sensors)
- pollution, detect trace contamination
- detergent additives in engine oils, where do they go ?
- Aerospace and automobile companies test metal, plastic and other components for safety standards and failures of performance.

3. Microchips

- Evaluate **trace metals on future Si wafers** with TXRF

  Stanford Collaboration (AMD, Applied Materials, Atominstitute, Balazs Analytical Labs, Charles Evans, DEC, Dow Chemical, Hewlett Packard, IBM, Intel, Komatsu, Lucent, MIT, Motorola, National Instruments, Rigaku, Sandia National Laboratory, Stanford University, Texas Instruments, Wacker, and Waseda University.)
- material science (materials properties)
- magnetic storage devices, recording heads, thin film media
- magnetic domain microscopy based on magnetic dichroism
- microscopy on **nano structure defects**
- chemical analysis of nanostructures as quantum dots/whiskers
- **flat panel displays**
- SAXS for thin films of metals, semiconductors and polymers.
- micromachining of connectors, packaging

4. Polymers/Fibers/Plastics/Rubber

- research on polymers
- mechanical properties of polymers, stronger, cheaper, lighter
- **molding of plastic** determines properties
- in-situ study during **fiber processing**, plastic extrusion,
- real time evolution of structure/process relationship
- **fracture mechanism in fibers/plastic** is not known
- present remedy: overdesign, expensive, heavy
- proving patents by structural analysis of new polymorph
- packing for hot coffee/tea,
- why do bags split during filling?
- polymer in diapers (20 bn$ market). "super absorbent polymer" (Dow Chemical uses high tech polymers)

5. Health

- **heavy metals**: some necessary for good health, some detrimental, need to know species and oxidation state (XAS)
- need to know in very dilute samples (XAS)
- contaminants in industrial dump sites (Cr^{+3}, Cr^{+6}.....)
- sewage treatment

- **phyto-remediation**: absorption of heavy metals by plants, e.g. Cd and Zn. (XAS, XANES)

- migration of pollutants (XAS, EXAFS)
- **waste incineration** – ash production and management (XAS)
- waste as filler and fuel in cement, leaching out of metals (XAS)

- trace metals in food like mercury in fish (XAS)
- hair and skin products (reaction of skin and hair to product)

6. Environment

- **industrial & nuclear waste remediation**
- soil contamination from mines, industry etc. (Chromium from paint, tanning.
- **phyto-remediation** (some plants absorb heavy metals)
- bacterial oxidation of Mn(II) (global cycling of C,N,S, nutrients, and contaminants in environment, photosynthesis)
- Molecular Environmental Science (MES): study of interfacial, molecular- and nano-scale processes controlling contaminant and nutrient cycling in the biosphere

7. Materials

Mechanics and chemistry of materials study allows development of new materials:

- **Superconducting materials** (liquid-He temperature)
- Ceramic oxide materials – high T_c superconductors (liquid-N temperature)
- Semi-conductors / Electronics
- Metallurgical industry / Metals / Alloys
- New ceramics / Plastics / Polymers / Glasses
- Cosmetics
- Oils / Petro-chemistry
- Cement

8. Biotechnology

- **protein crystallography**, Crystal structures of proteins digestive enzymes, hormones, receptors and receptor/signaling proteins are critical to understand important physiological processes.
- protein distribution in plants, plant pathogens
- soil analysis, oxidation states of metals in soil
- **agricultural bio-technology**, e.g. digestibility of barley, animal food
- seed vigor and environment
- biological SAXS (small angle X-ray scattering) from atomic to protein to virus and up to sub-cellular structures

Applications of Synchrotron Light

Synchrotron Radiation

is a tool for

limitless possibilities in basic and applied sciences

need

interdisciplinary collaboration

of

scientists

biologist, chemist, physicist, engineers,
pharmacist, medical researcher, geologist, archeologist,

.....

with

creative imagination

Thank you