



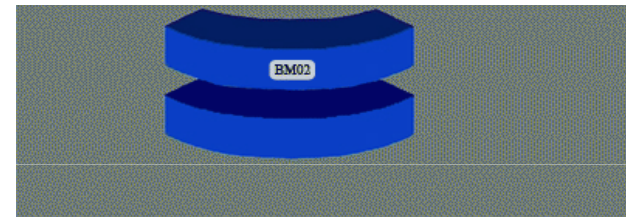
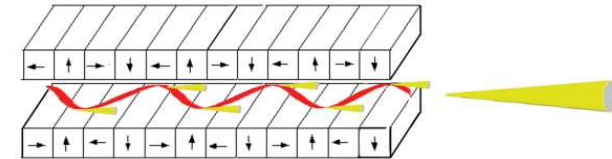
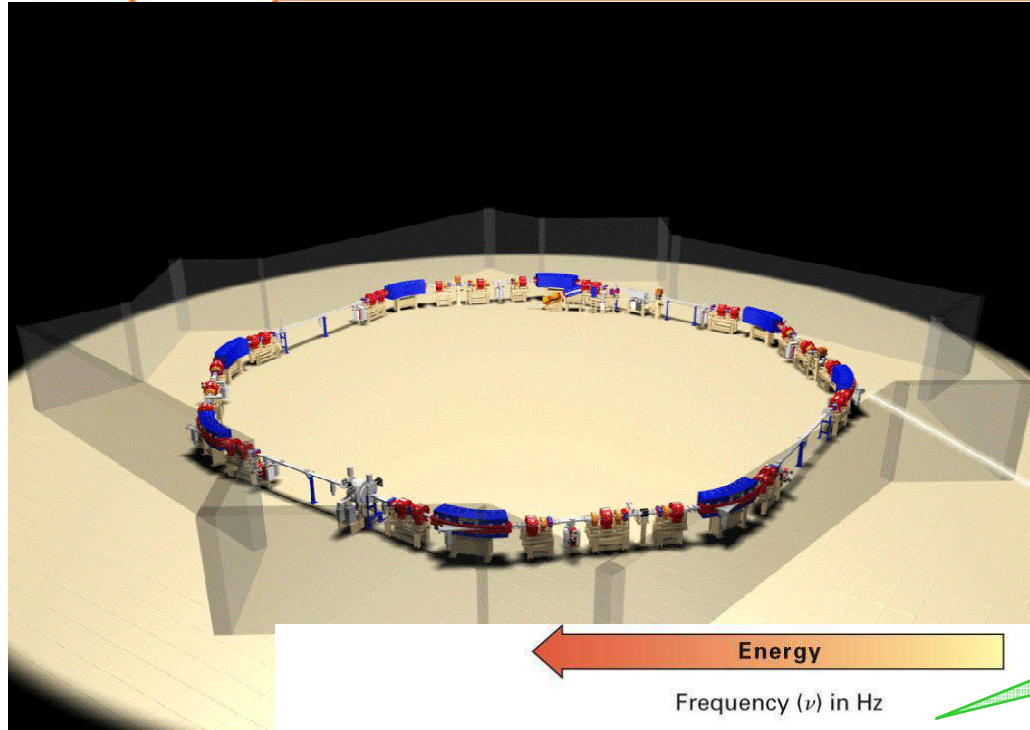
การประยุกต์แสงซินโครตรอน ในการวิจัยและพัฒนาด้านนาโนเทคโนโลยี

Pinit Kidkhunthod (Ph.D. Physics)

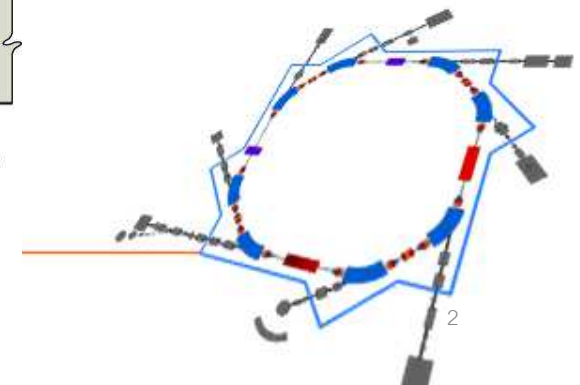
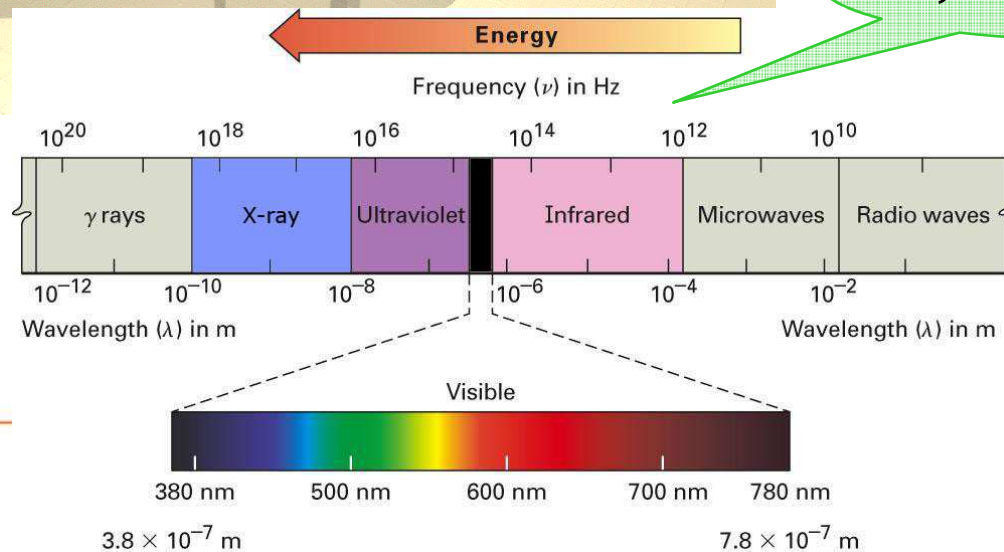
Beamline scientist at BL5.2 XAS
Synchrotron Light Research Institute
Nakhon Ratchasima, THAILAND
email : pinit@slri.or.th



How to produce Synchrotron Light?



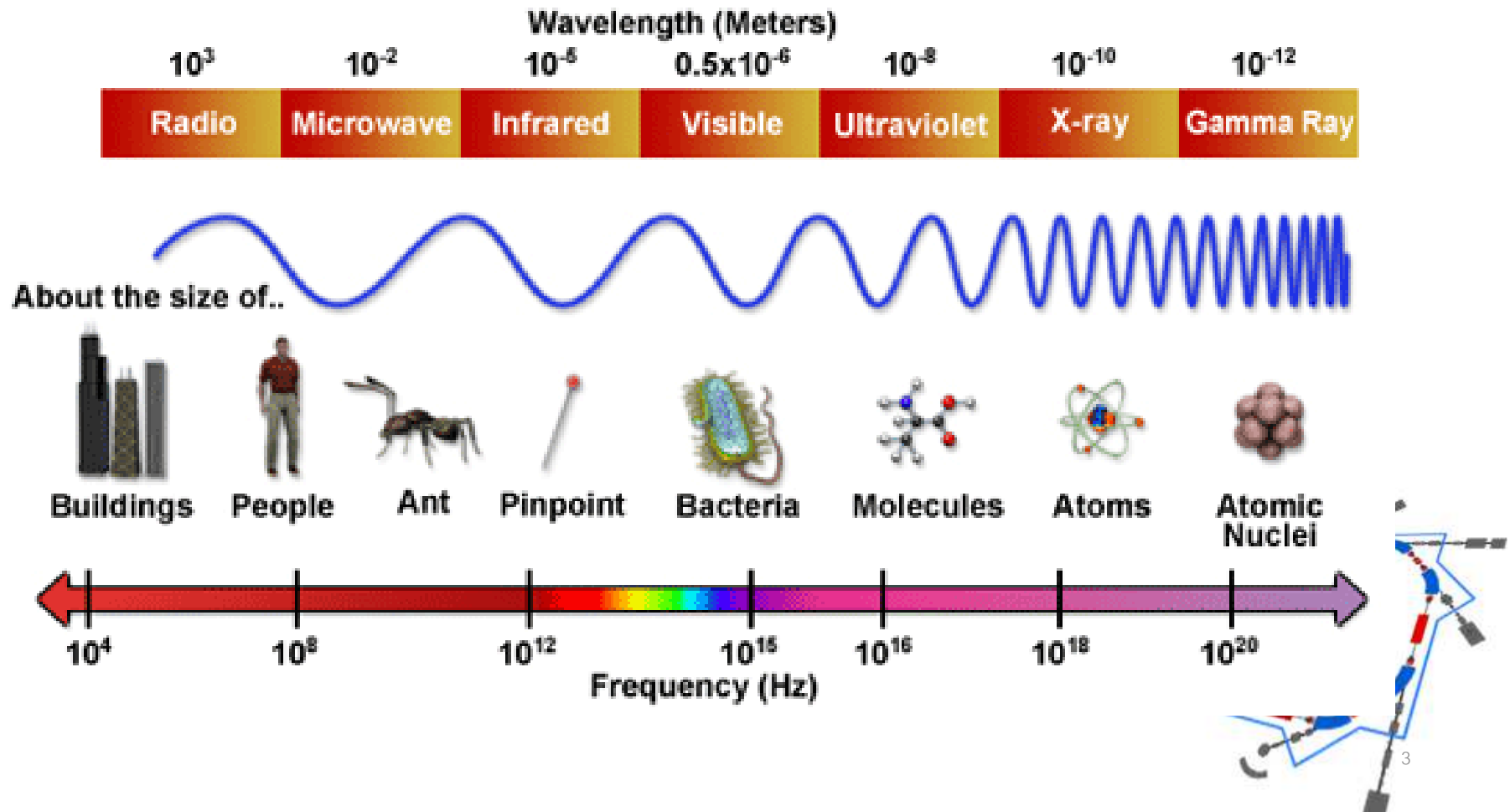
Synchrotron Light





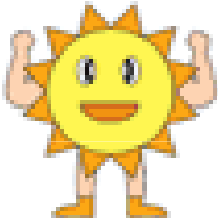


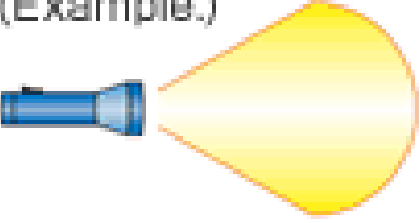
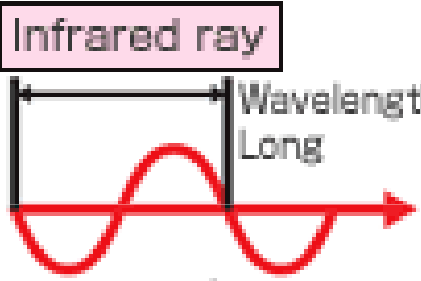
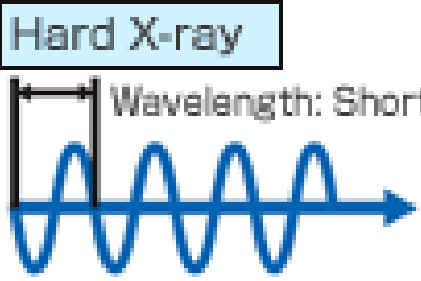
Electromagnetic Spectrum

Electromagnetic Spectrum





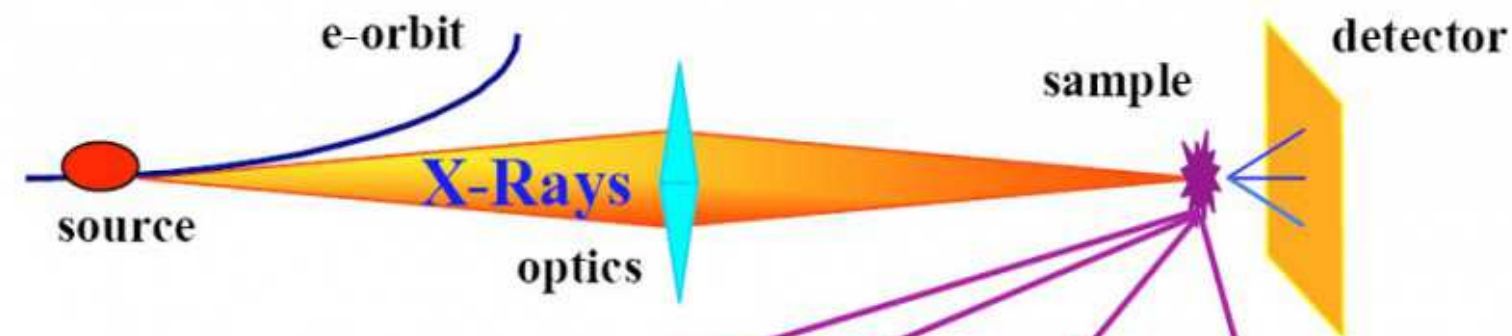
Why do we need Synchrotron Light?

Very bright	Narrowly focused and does not spread (High directionality)	Includes a broad wavelength range from infrared to hard X-rays
<p>Synchrotron radiation</p>  <p>10 billions times sunlight</p>  <p>Sunlight</p>	<p>Synchrotron radiation</p>  <p>Narrowly focused</p> <p>Flashlight's beam (Example.)</p>  <p>The beam spreads</p>	<p>Infrared ray</p>  <p>Wavelength: Long</p> <p>Hard X-ray</p>  <p>Wavelength: Short</p>
<p>Rapid analysis Trace element analysis</p>	<p>Mapping with nano engineering</p>	<p>Chemical state analysis Local structure analysis</p>

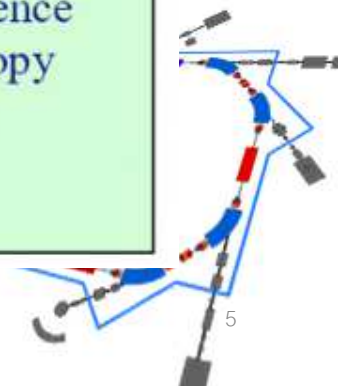




Synchrotron-based Techniques



Energy	Momentum	Position	Dynamics
Spectroscopy EXAFS XANES Fluorescence Spectromicroscopy	Scattering MAD SAD SAXS XMS Interferometry	Imaging Microscopy Tomography Topography Phasing Lithography	Time-resolving Diffraction Luminescence Spectroscopy Scattering Imaging





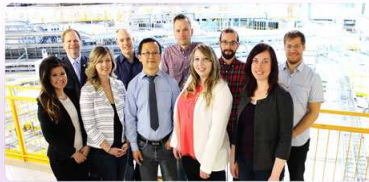
SR-Applications in Industrial Works :

INDUSTRIAL SCIENCE SOLUTIONS



Non-destructive synchrotron-based research provides unsurpassed information for industry

CLS's mandate includes vigorous industrial participation with innovative commercial research access, clear intellectual property policies, and scientists dedicated to working with industry.



- Unique-in-Canada analytical services
- A multi-disciplinary research centre – synergies between scientists from different disciplines
- The only synchrotron facility with a dedicated industrial support group
- Over 280 industrial science fee-for-service and collaborative projects to date
- 10% Industry use (highest rate in the world!)

Mining and Environment



Synchrotron techniques provide valuable information in many areas of the mining process, from metals recovery to tailings management, by identifying metallic phases and amorphous compounds in discharges and mine tailings.



"The data we're getting from our synchrotron work demonstrates that the CLS is a key resource for our technical operations."



"CLS helped us gain technical certainty in our quest for environmental sustainability."

Energy Storage



Development of Battery and Fuel Cell Materials

Synchrotron techniques are increasingly being recognized as a crucial tool for the development of new battery and fuel cell materials. Advanced X-ray techniques allow us to probe the structure and electronic properties of electrode materials, electrolytes, catalysts, separators, and additives.

"The group at the Canadian Light Source is the premier group in the world for this kind of work."



Oil and Gas



CLS can assist in isolating heavy metals in industrial sites, measuring the porosity and permeability of oil bearing rock, determining mineral reactions from CO2 injection and characterizing polymers and new industrial materials for use in extraction and refining processes. Specific to the oil sands, opportunities exist in the analysis of the nature of slurries and soils to assist in both extraction and remediation.



"We could only postulate how the contaminants were associated with adjacent elements. Now we can answer that question."

Aerospace



Synchrotron research is a key tool for aerospace industrial research, and is applied to a wide range of problems from the study of surface coating failures to 3D non-destructive testing of new materials.



"The CLS was able to offer conclusive evidence."



"We came to the CLS with problems... and now they're solving them with us."

Pharmaceuticals

Single crystal X-ray crystallography is a primary means of determining the molecular conformations of proteins and other biomolecules. It

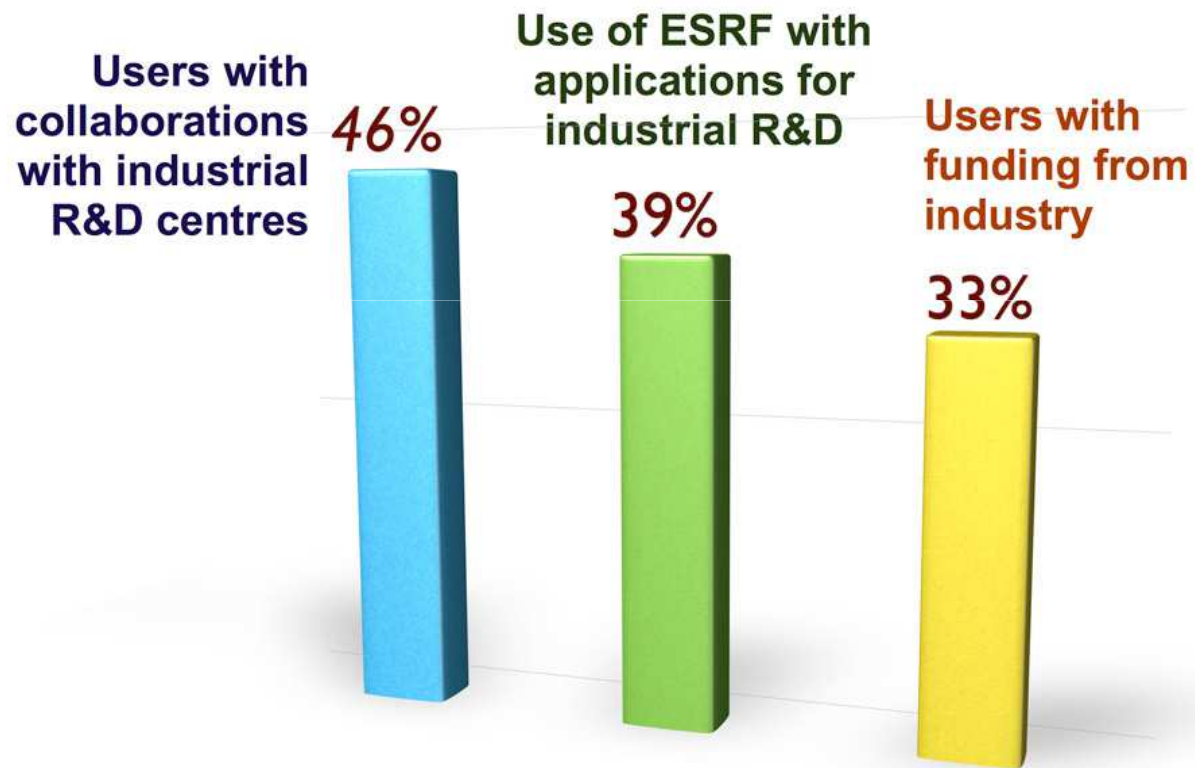


is critical for the understanding of medically-important targets and relied upon for pharmaceutical discovery and optimization.





ESRF Case for Industrial Works



www.esrf.eu





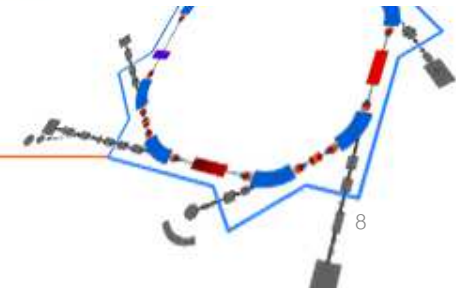
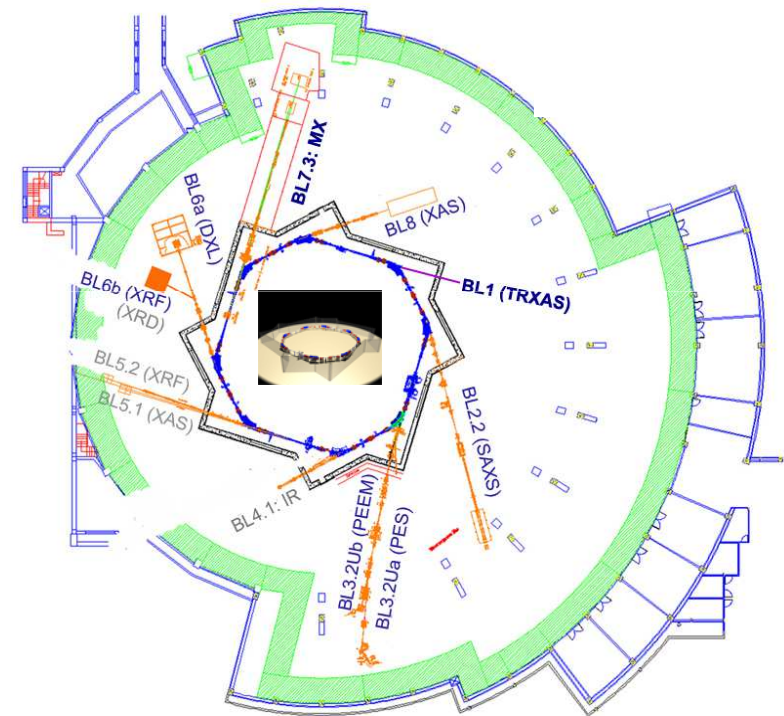
Beamlines at SLRI, THAILAND

In Operation :

- BL1.3 : SAXS : Small Angle X-ray Scattering
- BL2.2: TR-XAS : Timed-resolved XAS
- BL3.2a: PES : Photoelectron Emission Spectroscopy
- BL3.2b: PEEM : Photoemission Electron Microscopy
- BL5.2: SUT-NANOTEC-SLRI XAS beamline
- BL6a: DXL : Deep X-ray Lithography
- BL6b: micro-XRF : X-ray Fluorescence
- BL7.2: MX (Protein Crystallography)
- BL8: XAS : X-ray Absorption Spectroscopy

In Commissioning

- BL4.1: IR Spectroscopy & Imaging

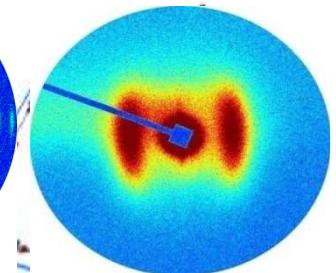
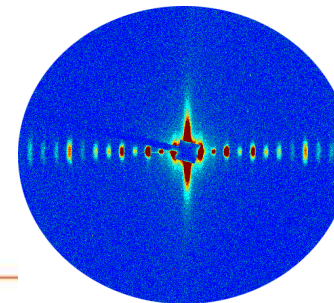
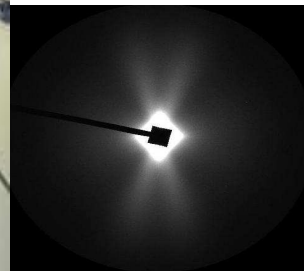
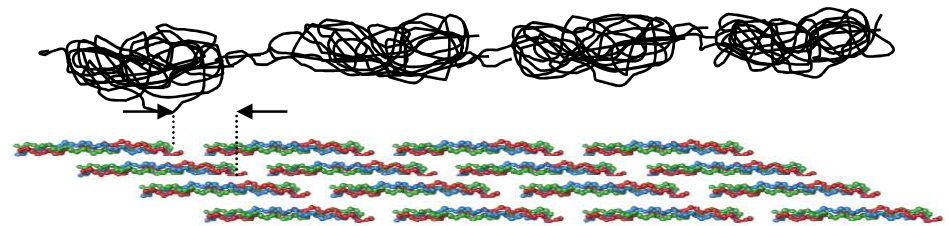




BL1.3 : SAXS : Small Angle X-ray Scattering



- ❖ ขนาดและรูปร่างของอนุภาคที่มีขนาดในช่วงประมาณ -100 นาโนเมตร
- ❖ โครงสร้างระดับนาโน และการจัดเรียงตัวของโมเลกุลในวัสดุ เช่น *precipitation in metal*, พอลิเมอร์หรือเส้นใย

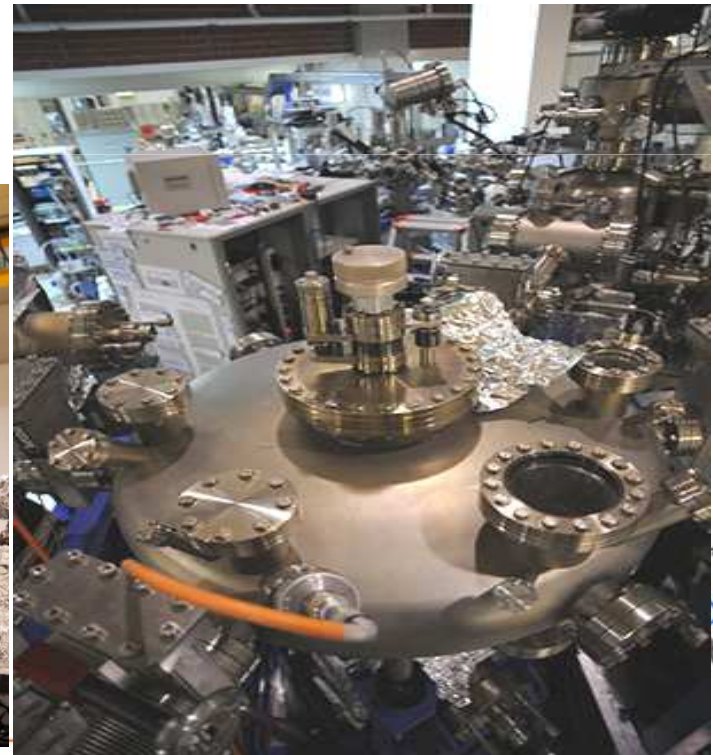
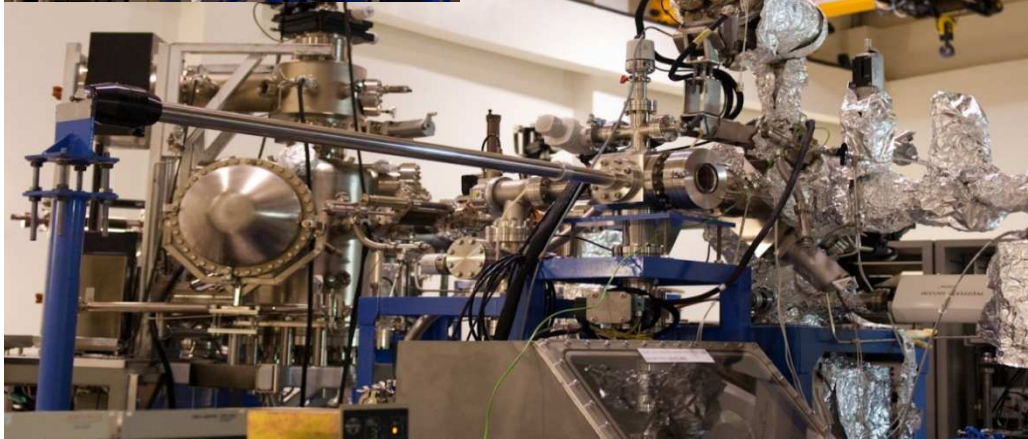




BL3.2a: PES: Photoelectron Emission Spectroscopy



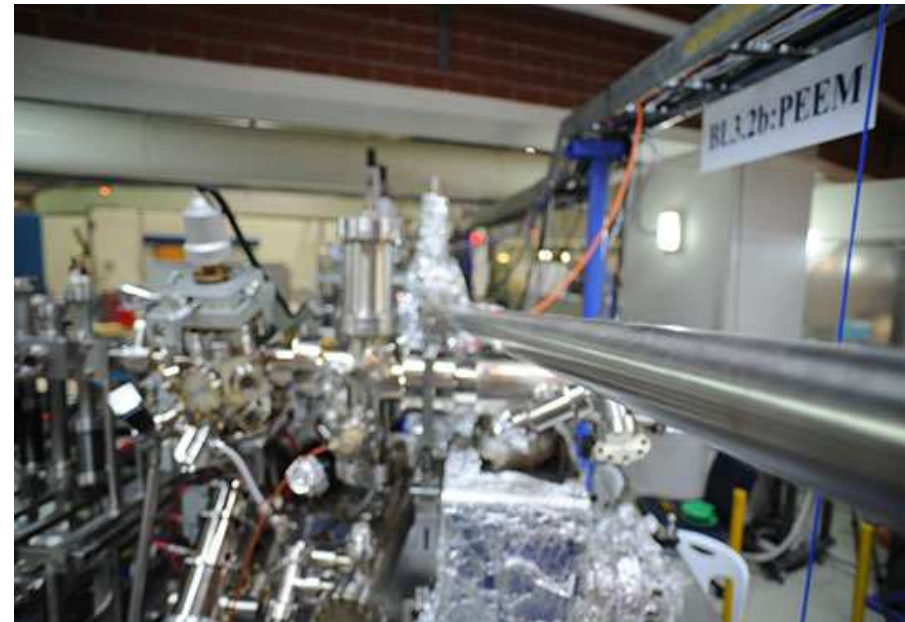
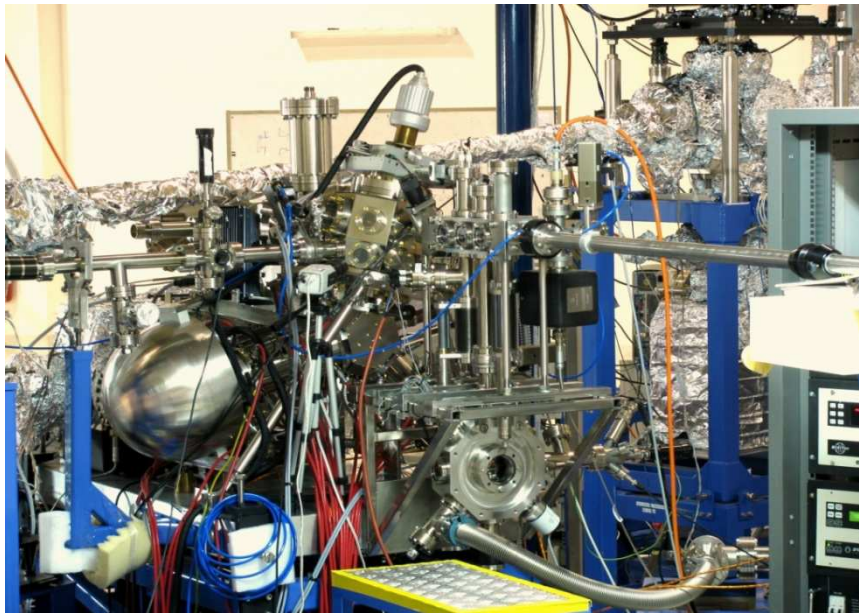
สำหรับการศึกษารวิจัยสมบัติของวัสดุ โดยเฉพาะวัสดุที่มีขนาดระดับนาโนเมตร เพื่อศึกษาคุณลักษณะของพื้นผิว และรอยต่อของชั้นผิววัสดุ ธาตุบนพื้นผิวของสารตัวอย่าง และยังใช้ในการวัดสมบัติความเป็นแม่เหล็กของสารตัวอย่างอีกด้วย



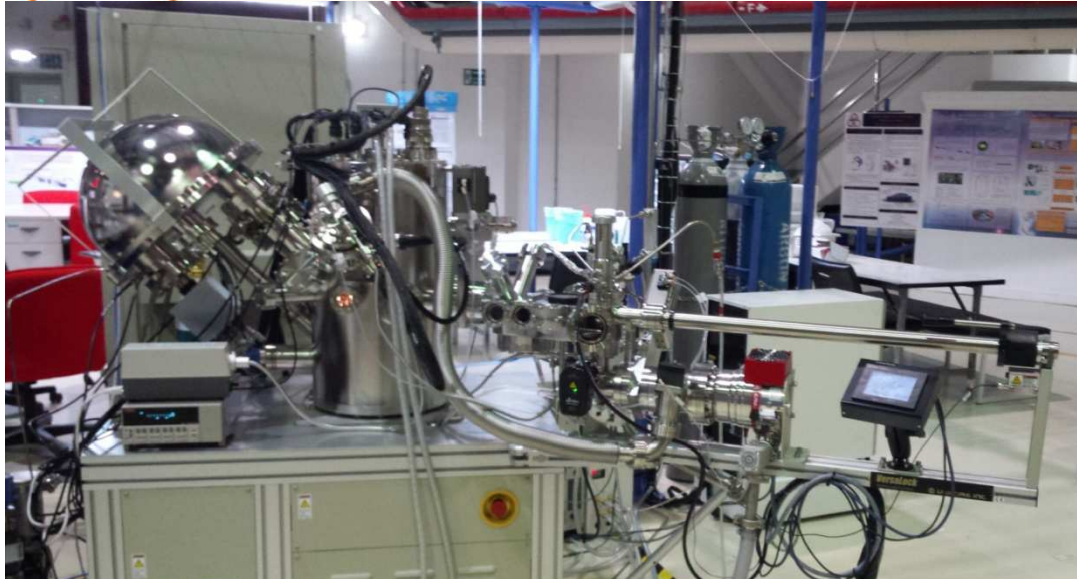


BL3.2b: PEEM: Photoelectron Emission Microscopy

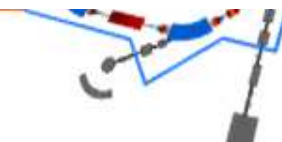
เป็นเทคนิควิเคราะห์ที่มีประสิทธิภาพสูง และมีประโยชน์สำหรับงานศึกษาด้านพื้นผิว และการปลูกฟิล์มบาง เนื่องจากสามารถเลือกถ่ายภาพบริเวณที่สนใจบนผิวของตัวอย่างได้โดยมีความละเอียดในระดับนาโนเมตร นอกจากนี้ยังสามารถเลือกวิเคราะห์องค์ประกอบทางเคมี และโครงสร้างทางผลึกของวัตถุนผิวของสารตัวอย่าง



PHI 5000 VersaProbe II Scanning XPS Microprobe

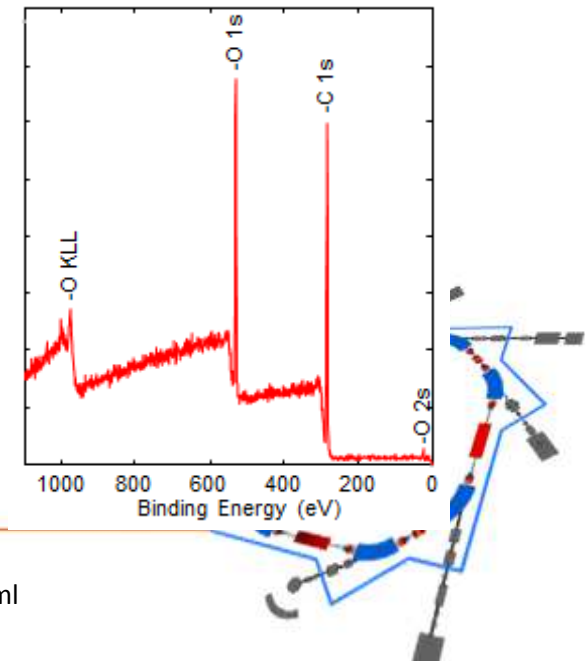
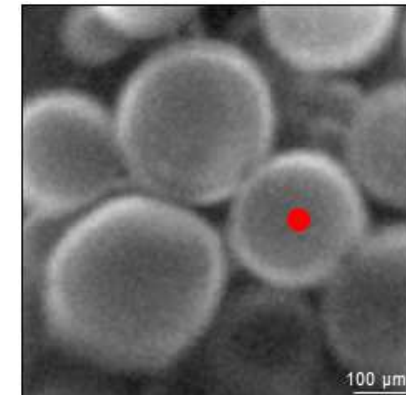
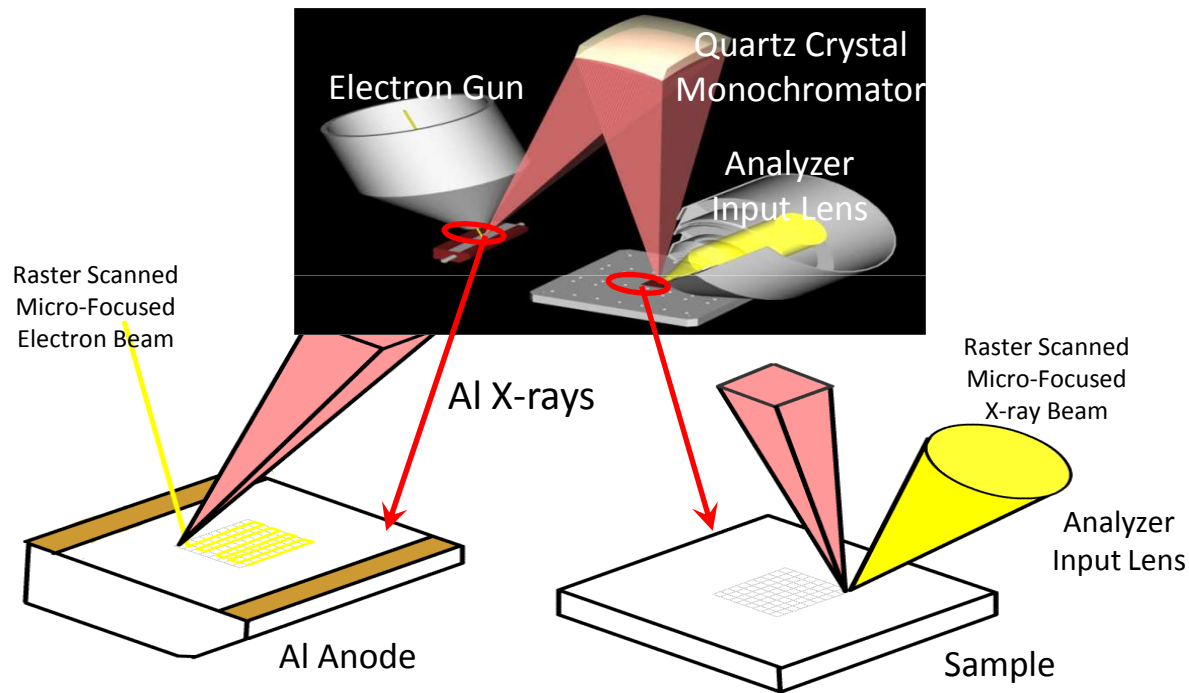


- Scanning monochromatic X-ray source (Al $K\alpha$) with a focused beam $\leq 10 \mu\text{m}$
- Argon ion gun for sample cleaning and depth profiling
- Dual beam charge compensation system
- Sample can be heated up to $800 \text{ }^\circ\text{C}$ in static mode and $500 \text{ }^\circ\text{C}$ in dynamic mode.



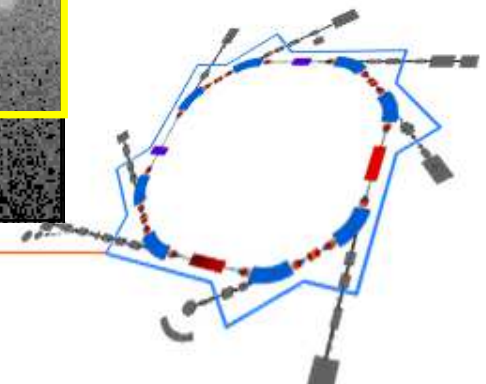
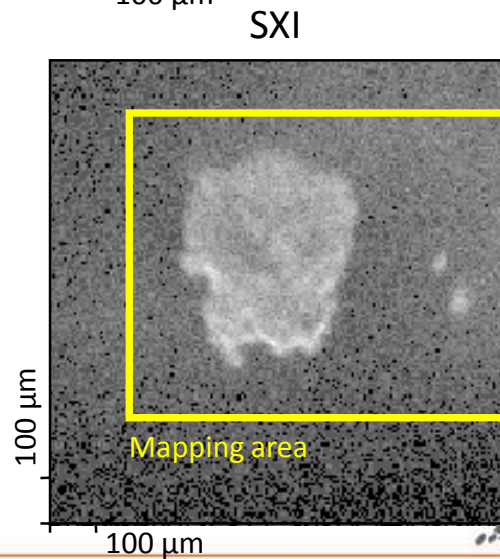
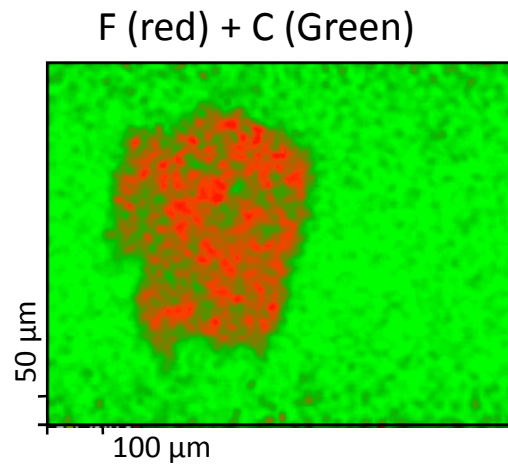
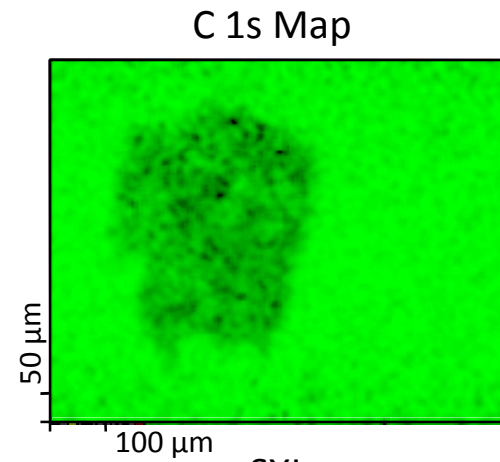
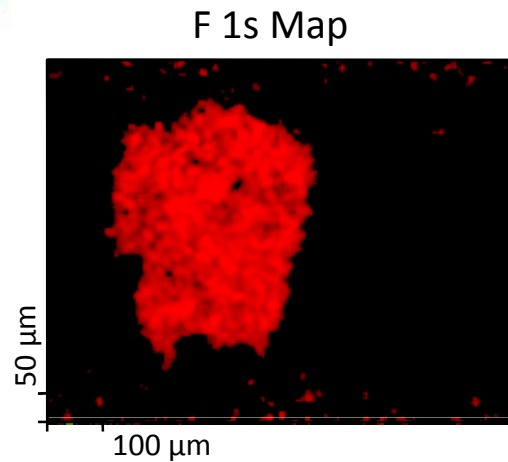
Scanning monochromatic X-ray source (Al K α) with a focused beam $\leq 10 \mu\text{m}$

Secondary Electron Images of PMMA micro spheres



BL 5.2: SUT-NANOTEC-SLRI

- XPS maps provided spatial distribution information and identified areas for additional micro-area spectroscopy

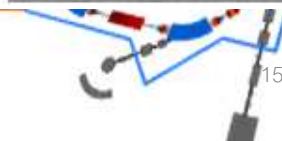




XAS : X-ray Absorption Spectroscopy

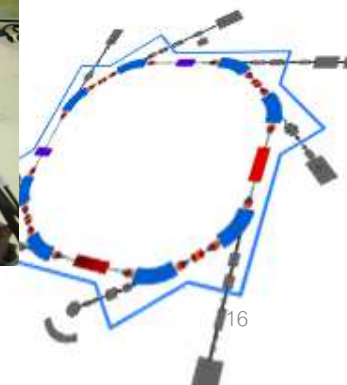
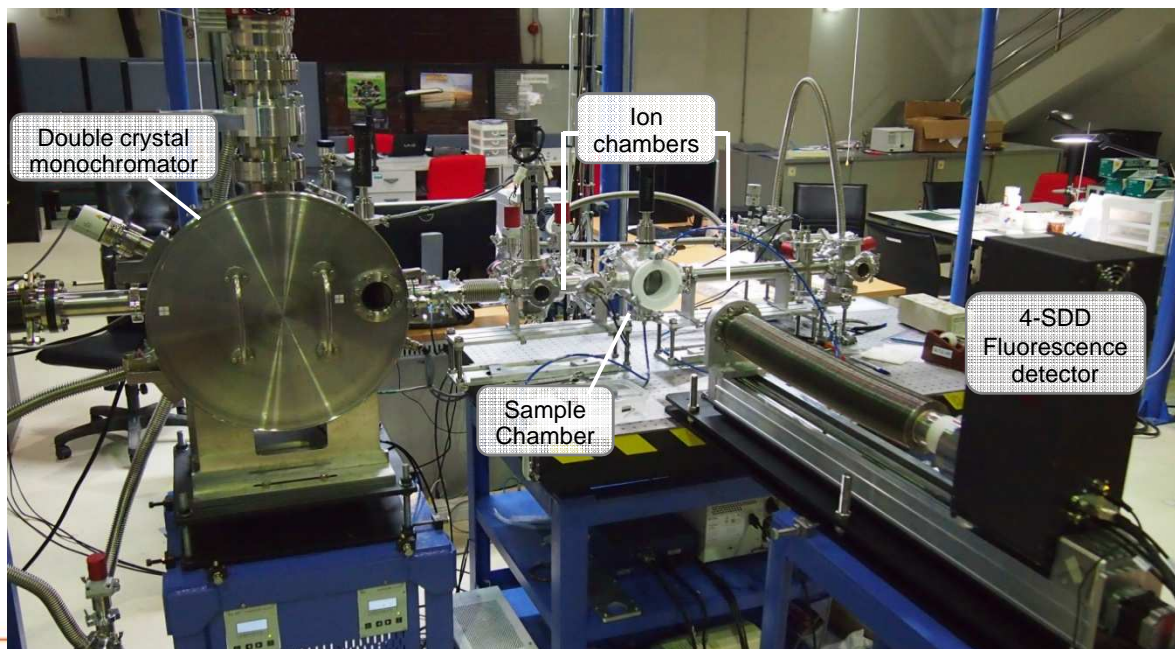
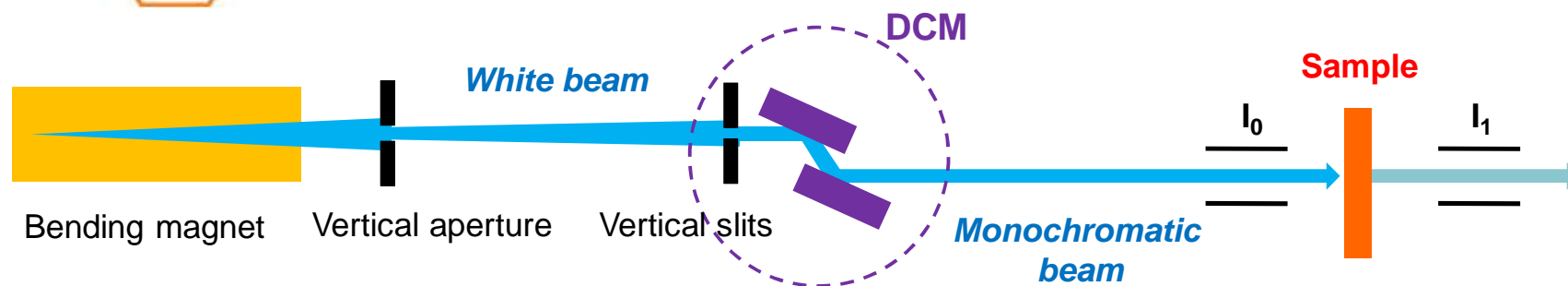
สำหรับศึกษาโครงสร้างระดับอะตอม เช่น การจัดเรียงตัวของอะตอม และการระบุสถานะออกซิเดชัน (oxidation state) ของอะตอม สามารถประยุกต์ใช้ในงานวิจัยได้หลากหลายสาขา เช่น

อุตสาหกรรม : ผลิตภัณฑ์เซรามิก เหล็ก ยาง ซีเมนต์และโพลีเมอร์





Conventional XAS beamline : BL 5.2

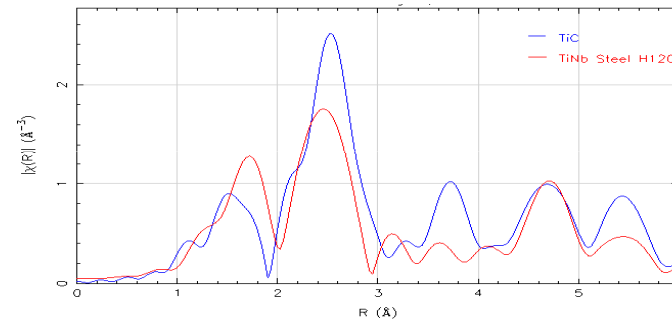
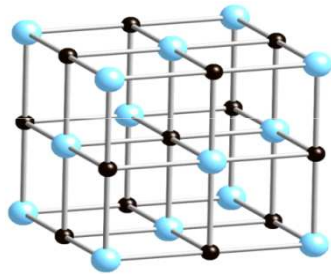




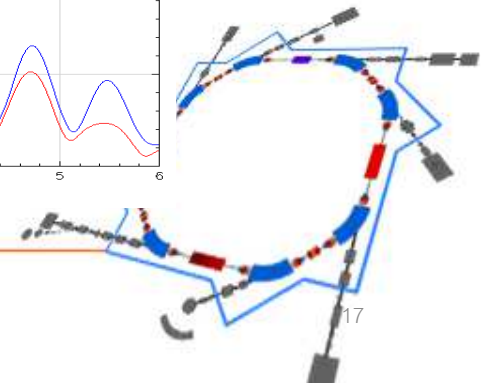
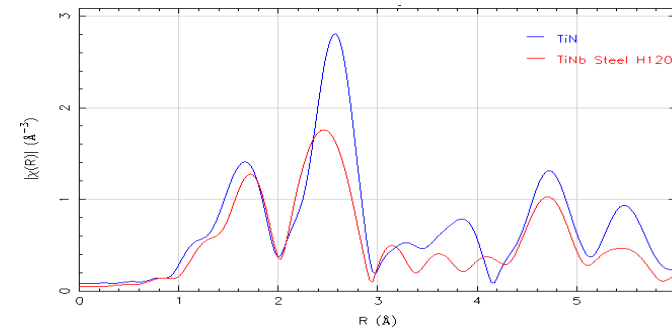
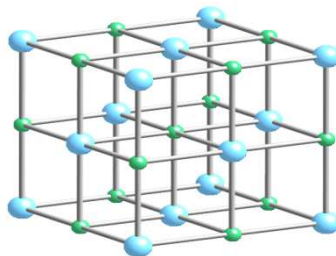
Examples in Metallurgy

Particle-size estimation of Metal precipitation in high-strength low-alloy steel by XAS

M-C



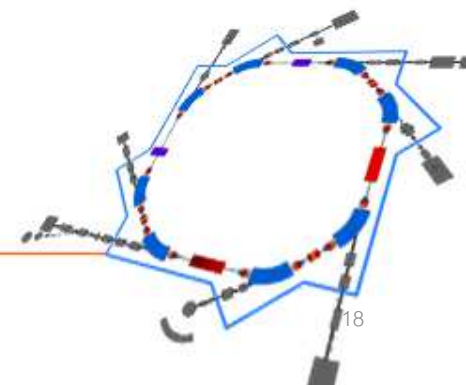
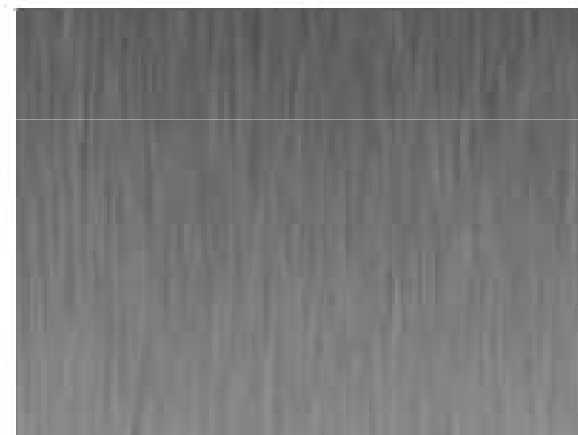
M-N





Examples in Metallurgy

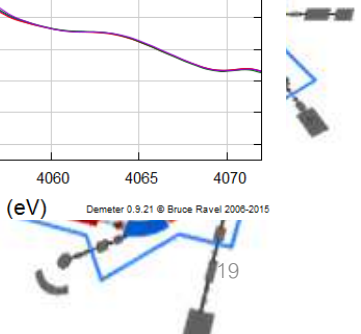
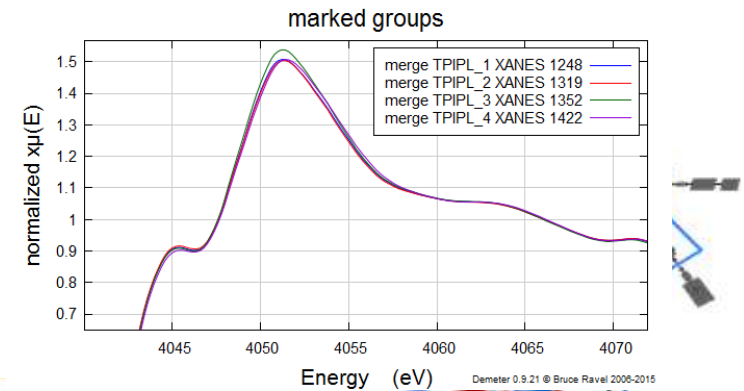
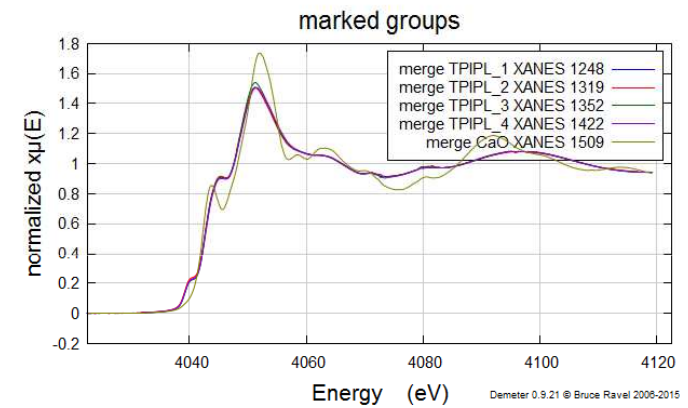
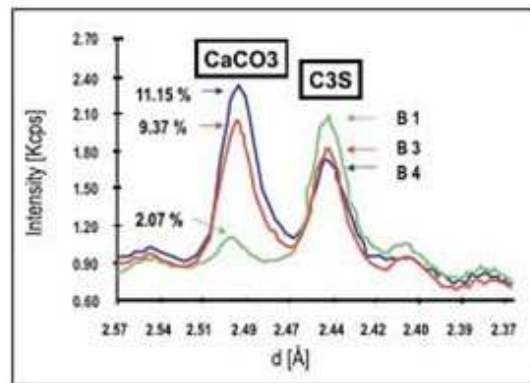
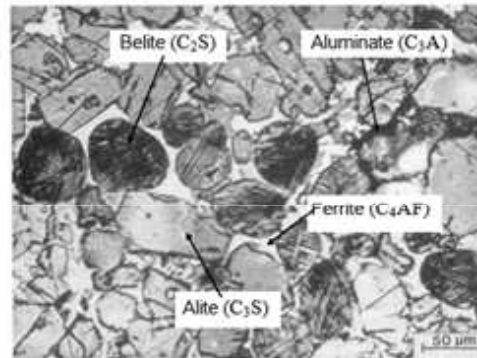
Surface Defect Probed by PES + PEEM + XAS





Examples in Cement

Phase Identification of Ca in Cement by XAS





XANES data on Magnetic Materials

Appl. Phys. A
DOI 10.1007/s00339-015-9416-5


Applied Physics A
Materials Science & Processing

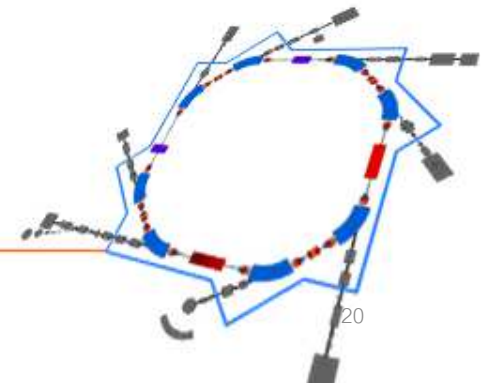


Room-temperature ferromagnetism in Fe-doped In_2O_3 nanoparticles

Kwanruthai Wongsaprom¹ · Somchai Sonsupap¹ · Santi Maensiri² · Pinit Kidkhunthod³

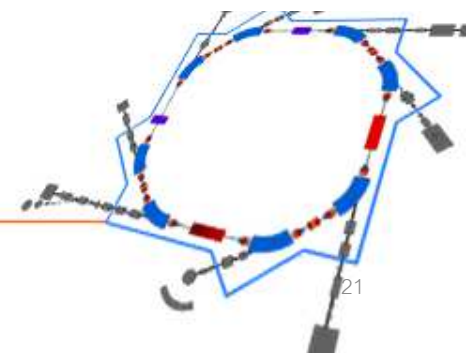
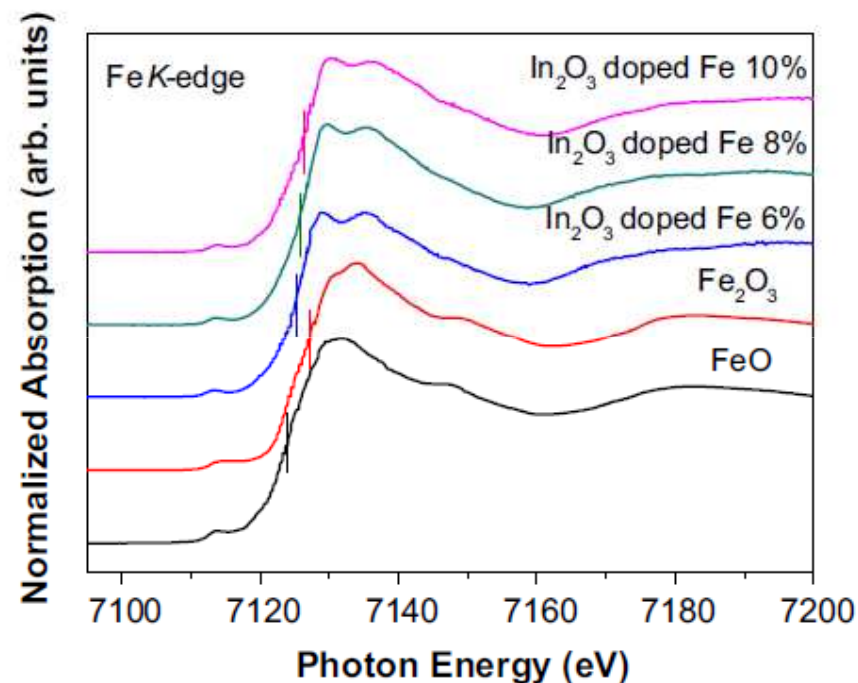
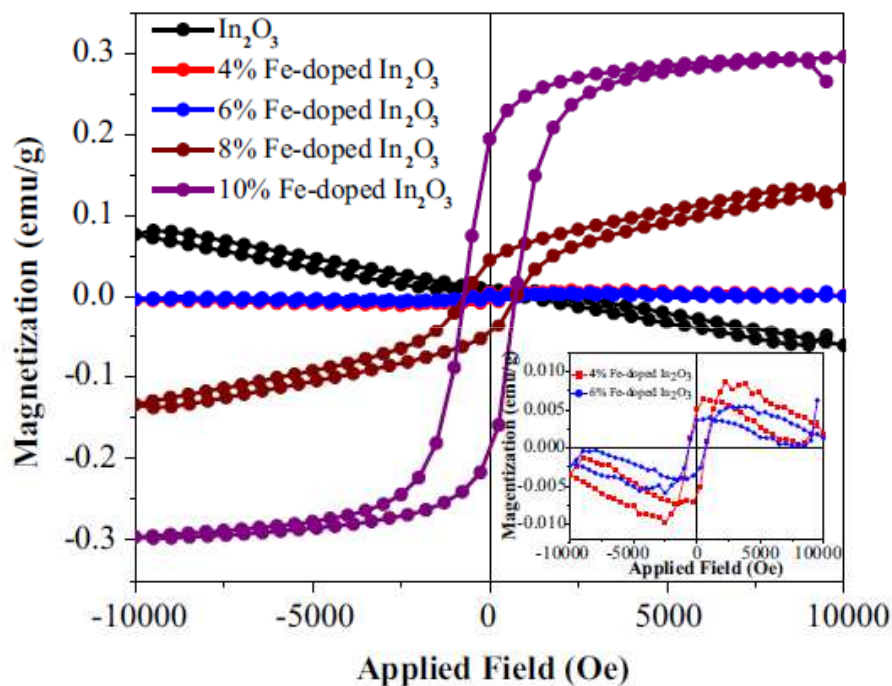
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 Springer





XANES data on Magnetic Materials





XANES data on Magnetic Materials

$$\langle \text{Fe oxidation state} \rangle = 3 \times \frac{\Delta E \text{ of sample}}{\Delta E \text{ of Fe}^{2+} \text{ and Fe}^{3+}} + 2 \\ \times \left(1 - \frac{\Delta E \text{ of sample}}{\Delta E \text{ of Fe}^{2+} \text{ and Fe}^{3+}} \right)$$

$$\% \text{ of Fe}^{2+} = \left(1 - \frac{\Delta E \text{ of sample}}{\Delta E \text{ of Fe}^{2+} \text{ and Fe}^{3+}} \right) \times 100 \%$$

$$\% \text{ of Fe}^{3+} = \left(\frac{\Delta E \text{ of sample}}{\Delta E \text{ of Fe}^{2+} \text{ and Fe}^{3+}} \right) \times 100 \%$$

In ₂ O ₃ sample	ΔE of sample (eV)	$\langle \text{Fe oxidation state} \rangle$	% of Fe ²⁺	% of Fe ³⁺
$x = 0.06$	3.35	2.63 (2)	37.27	62.73
$x = 0.08$	4.02	2.75 (1)	24.72	75.28
$x = 0.10$	4.58	2.86 (3)	14.23	85.77





Original Paper

Local structure determination of substitutional elements in $\text{Ca}_3\text{Co}_{4-x}\text{M}_x\text{O}_9$ ($\text{M} = \text{Fe}, \text{Cr}, \text{Ga}$) using X-ray absorption spectroscopy

Supree Pinitsoontorn^{1,2}, Natkrita Prasoetsopha³, Pornjuk Srepusharawoot^{1,2}, Atipong Bootchanont⁴, Pinit Kidkhunthod⁵, Teerasak Kamwana^{1,2}, Vittaya Amornkitbamrung^{1,2}, Ken Kurosaki⁶ and Shinsuke Yamanaka⁶

¹ Department of Physics, Faculty of Science, Khon Kaen University, Khon Kaen, 40002, Thailand

² Integrated Nanotechnology Research Center, Khon Kaen University, Khon Kaen 40002, Thailand

³ Materials Science and Nanotechnology Program, Faculty of Science, Khon Kaen University, Khon Kaen 40002, Thailand

⁴ School of Physics, Institute of Science, Suranaree University of Technology, Nakhon Ratchasima, Thailand

⁵ Synchrotron Light Research Institute (Public Organization), Nakhon Ratchasima, Thailand

⁶ Graduate School of Engineering, Osaka University, Suita 565-0871, Japan

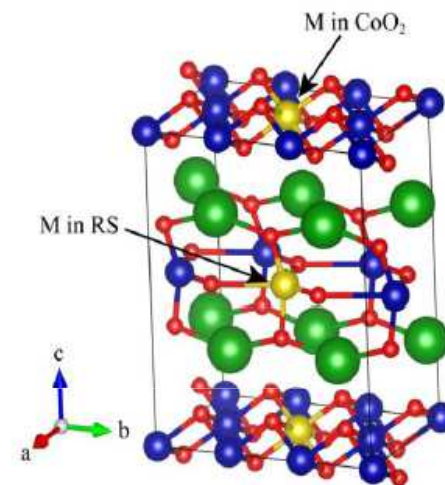
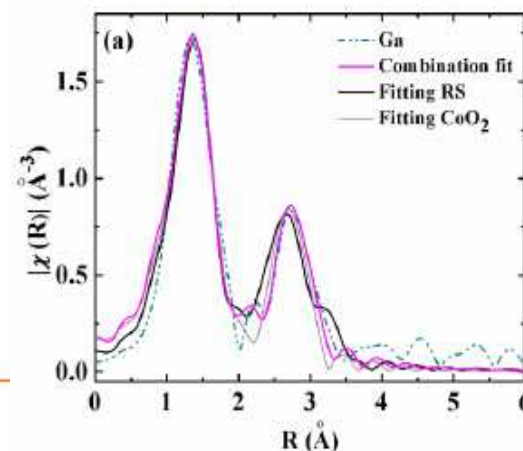
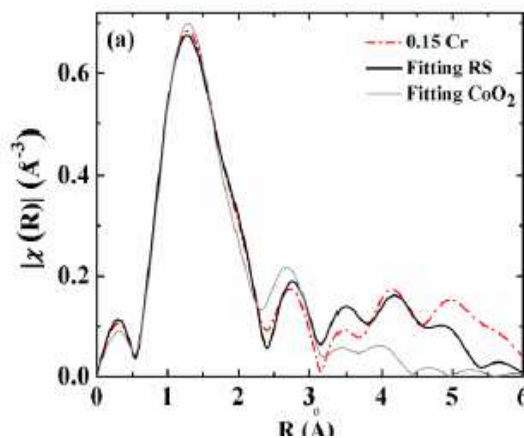
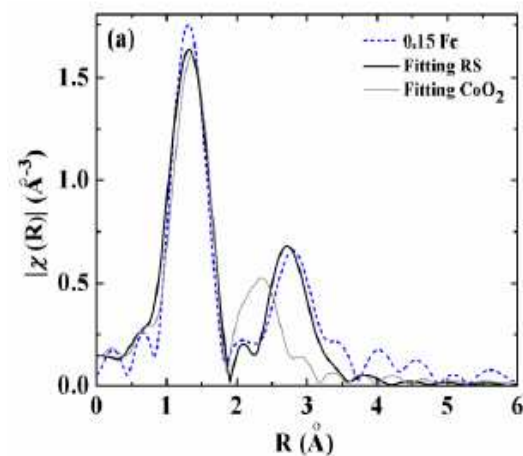


Figure 1 Structure model of $[\text{Ca}_2\text{CoO}_3]_4[\text{CoO}_2]_6$ when M is substituted in the RS layer or in the CoO_2 .





Materials Views

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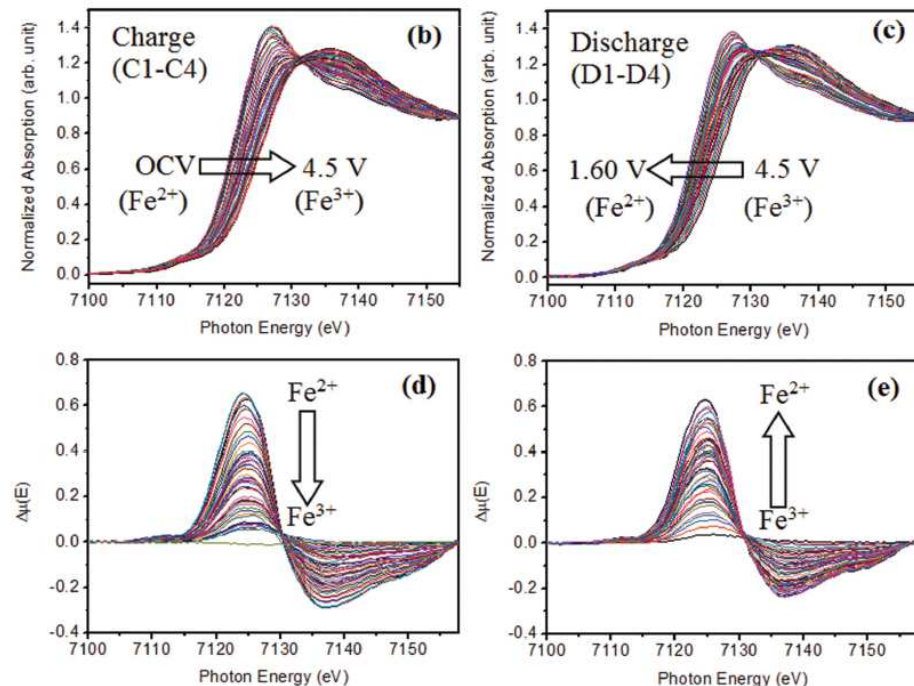
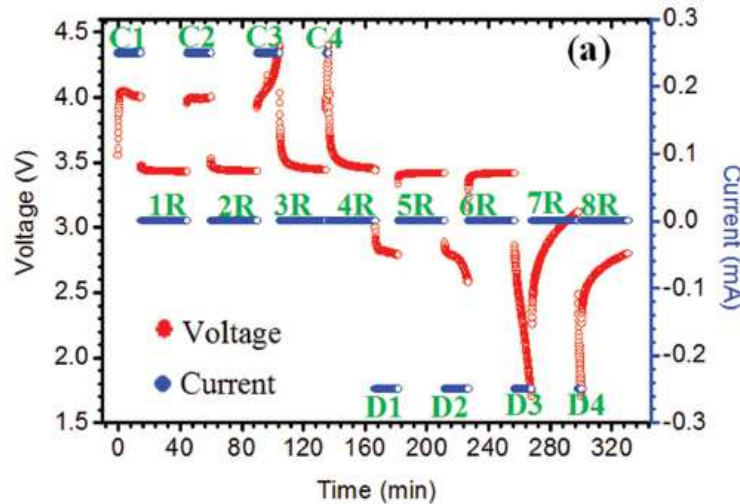
XANES data on Batteries

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XANES Investigation of Dynamic Phase Transition in Olivine Cathode for Li-Ion Batteries

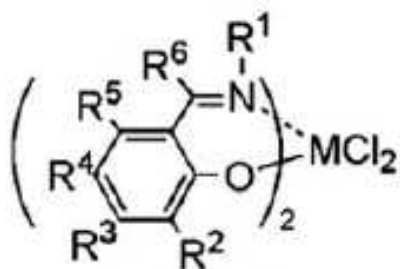
Sarawut Pongha, Boonyarit Seekoan, Wanwisa Limphirat, Pinit Kidkhunthod, Sutham Srilomsak,* Yet-Ming Chiang,* and Nonglak Meethong*





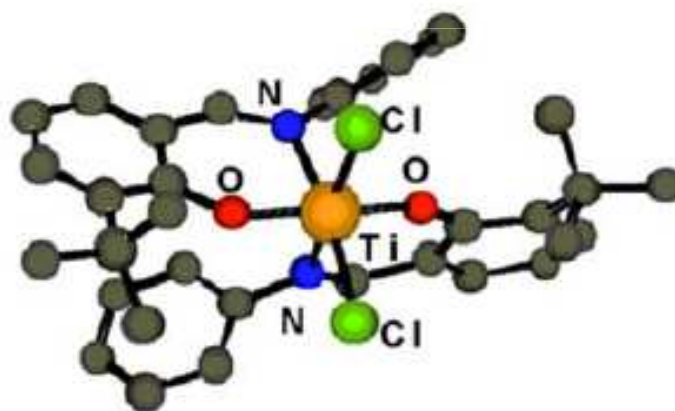
XAS : applications in polymerization

- Describing the ethylene and propylene polymerization behavior of bis(phenoxy-imine) Ti complexes (Ti-FI Catalysts), placing special emphasis on fluorinated Ti-FI Catalysts.



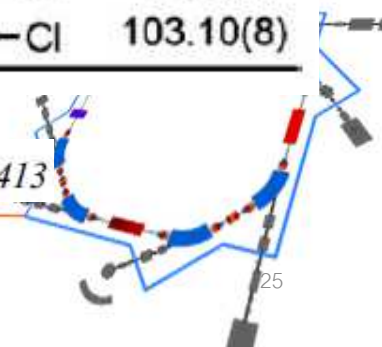
M: Ti, Zr, Hf, etc.

FI Catalysts



Bond Distances (Å)	
Ti—O	1.852(4)
Ti—N	2.236(4)
Ti—Cl	2.305(2)
Bond Angles (°)	
O—Ti—O	171.6(2)
N—Ti—N	76.4(2)
Cl—Ti—Cl	103.10(8)

R. Furuyama et al. / *Journal of Organometallic Chemistry* 690 (2005) 4398–4413

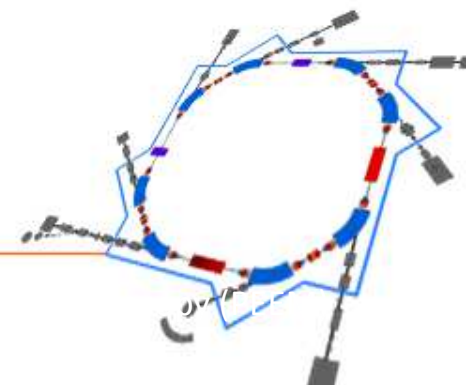
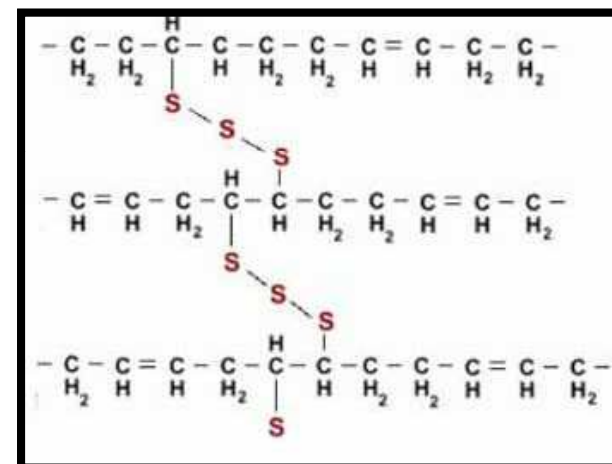
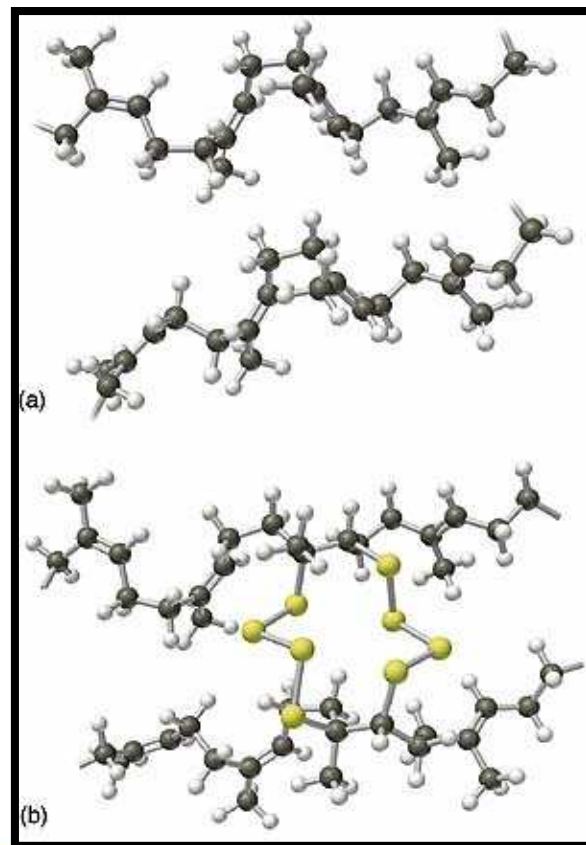




XAS : Cross-link polymers/Rubbers



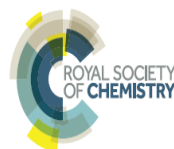
- Rubbers are harder and more flexible





XAS for Catalysis Science

Catalysis
Science &
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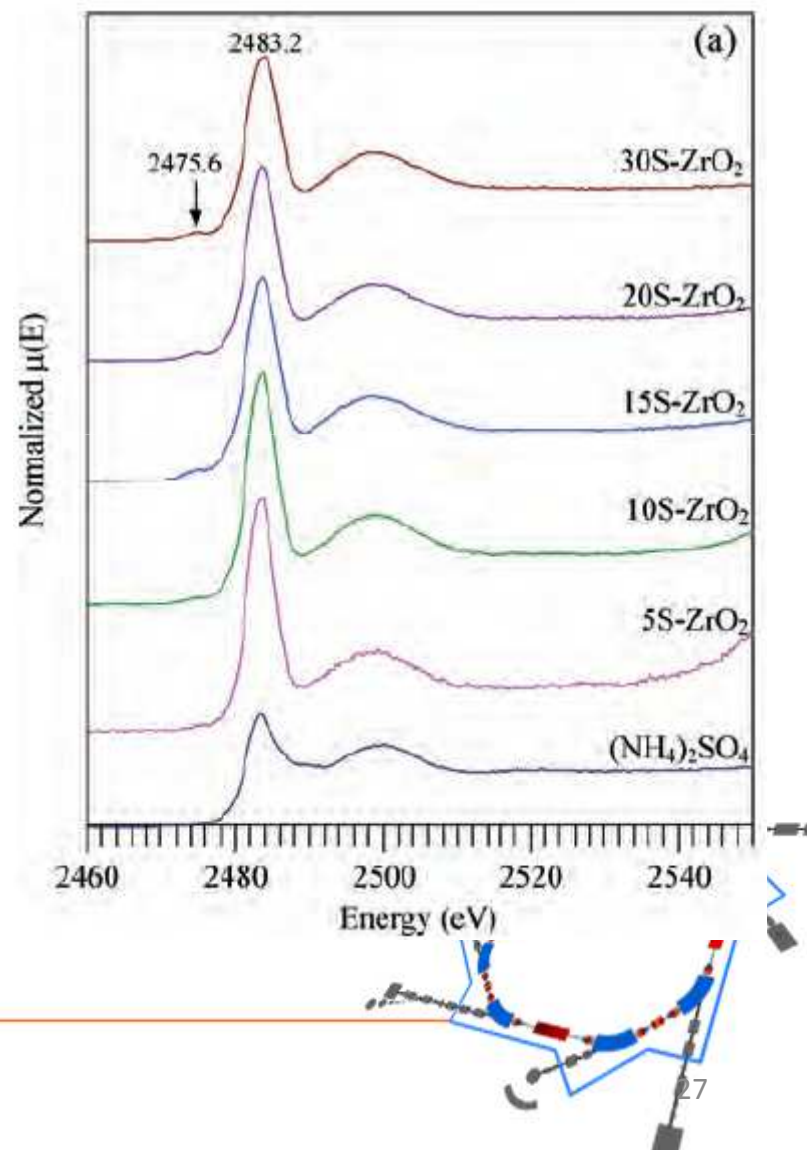


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Cite this: DOI: 10.1039/c4cy01568a

Direct synthesis of dimethyl ether from CO₂ hydrogenation over Cu-ZnO-ZrO₂/SO₄²⁻-ZrO₂ hybrid catalysts: effects of sulfur-to-zirconia ratios†

Thongthai Witoon,^{*abcd} Tinnavat Permsirivanich,^a Nawapon Kanjanasontorn,^a Chalairat Akkaraphataworn,^a Anusorn Seubsai,^a Kajornsak Faungnawakij,^e Chompunuch Warakulwit,^{bc} Metta Chareonpanich^{*abcd} and Jumras Limtrakul^{bcd}

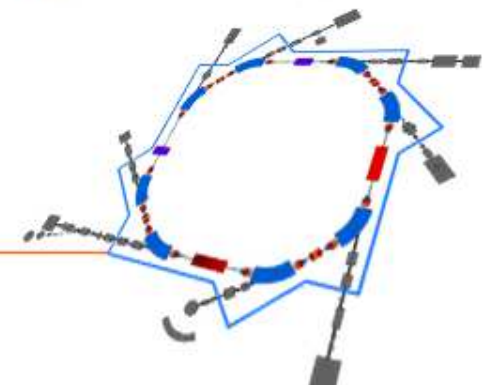
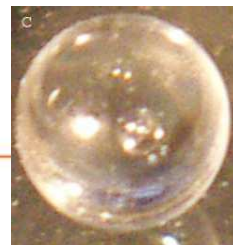
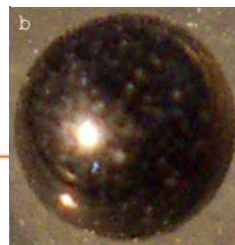
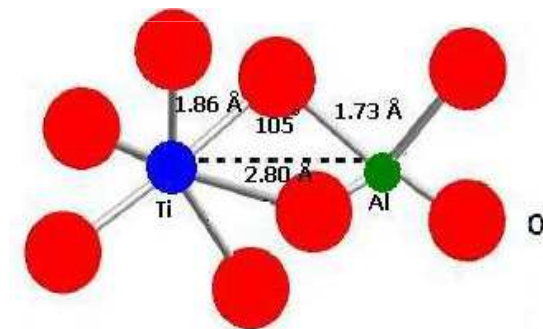
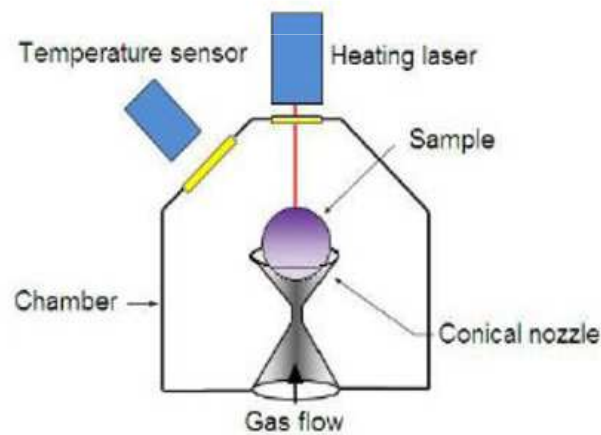


Structure of Ba-Ti-Al-O glasses produced by aerodynamic levitation and laser heating

Phys. Rev. B **90**, 094206 – Published 24 September 2014

Pinit Kidkhunthod, Lawrie B. Skinner, Adrian C. Barnes, Wantana Klysubun, and Henry E. Fischer

Aerodynamic Levitation and Laser Heating (ALLH)





Thank you for your attention



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