



*Canadian Light Source* *Centre canadien de rayonnement synchrotron*

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# The Canadian Light Source

Tom Ellis

CLS Director of Research

[thomas.ellis@lightsource.ca](mailto:thomas.ellis@lightsource.ca)

Surface Canada May 2013

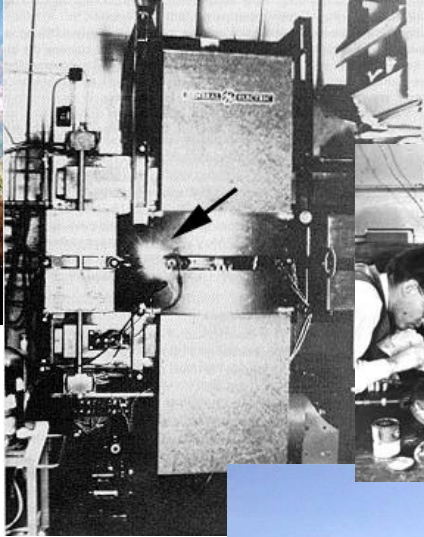
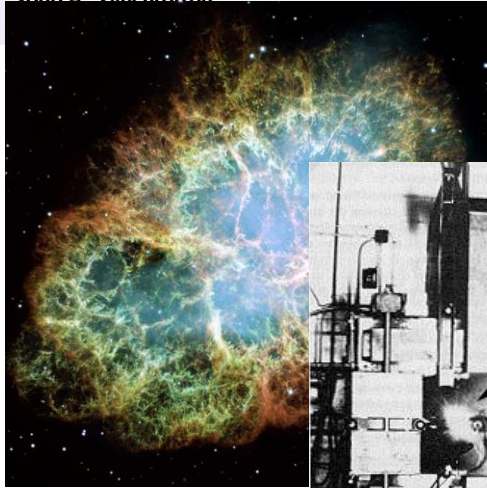






# A Brief History of Synchrotron Radiation Sources

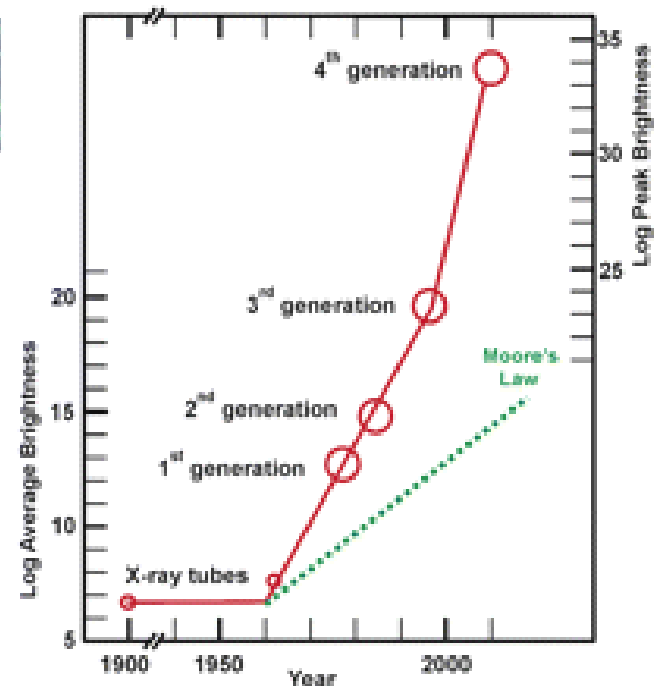
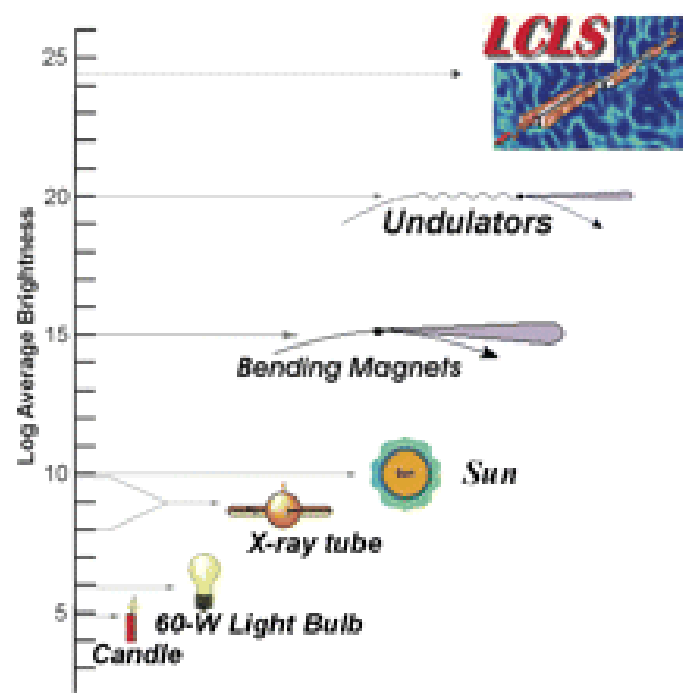
Canadian Centre canadien  
Light de rayonnement  
Source synchrotron





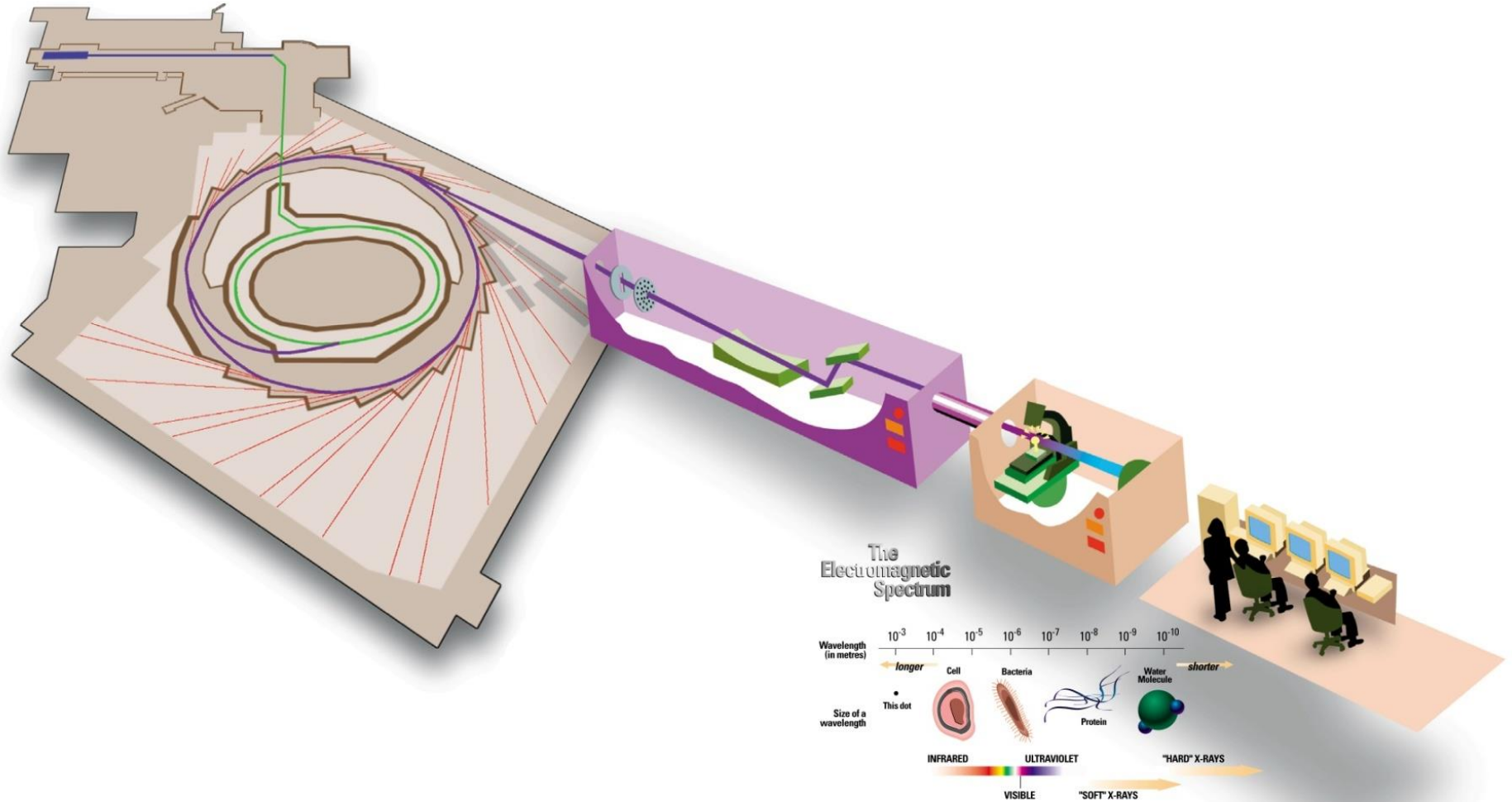
Canadian Centre canadien  
Light de rayonnement  
Source synchrotron

# X-ray Brightness



Parise and Brown (2006)

# How a synchrotron works









# CLS Timeline

- **September 27, 1999** – Groundbreaking ceremony
- **February 26, 2001** – Building dedication ceremony
- **September 18, 2002** – Booster ring commissioning complete
- **December 9, 2003** – First synchrotron light detected
- **October 22, 2004** – Official opening
- **May 27, 2005** – First CLS user
- **June 30, 2005** – Official completion of the CFI project



# Capital Investment to Date



- Original Construction (7 beamlines) \$141M
- Phase II (7 beamlines) 52M
- Phase III (7 beamlines & upgrade) 68M
- Isotopes Project 12M



# CLS Features

- Canada's national synchrotron facility
- One of the world's first ~3 GeV synchrotrons
  - Superconducting RF cavity
  - Canted insertion devices
  - Hard X-rays from superconducting wigglers
- Full spectrum of photon energies for spectroscopy (THz to hard X-rays)
- Other highlights: STXM, medical imaging, soft X-ray REIXS, soil science and mining applications





Western  
The University of Western Ontario

Boehringer  
Ingelheim

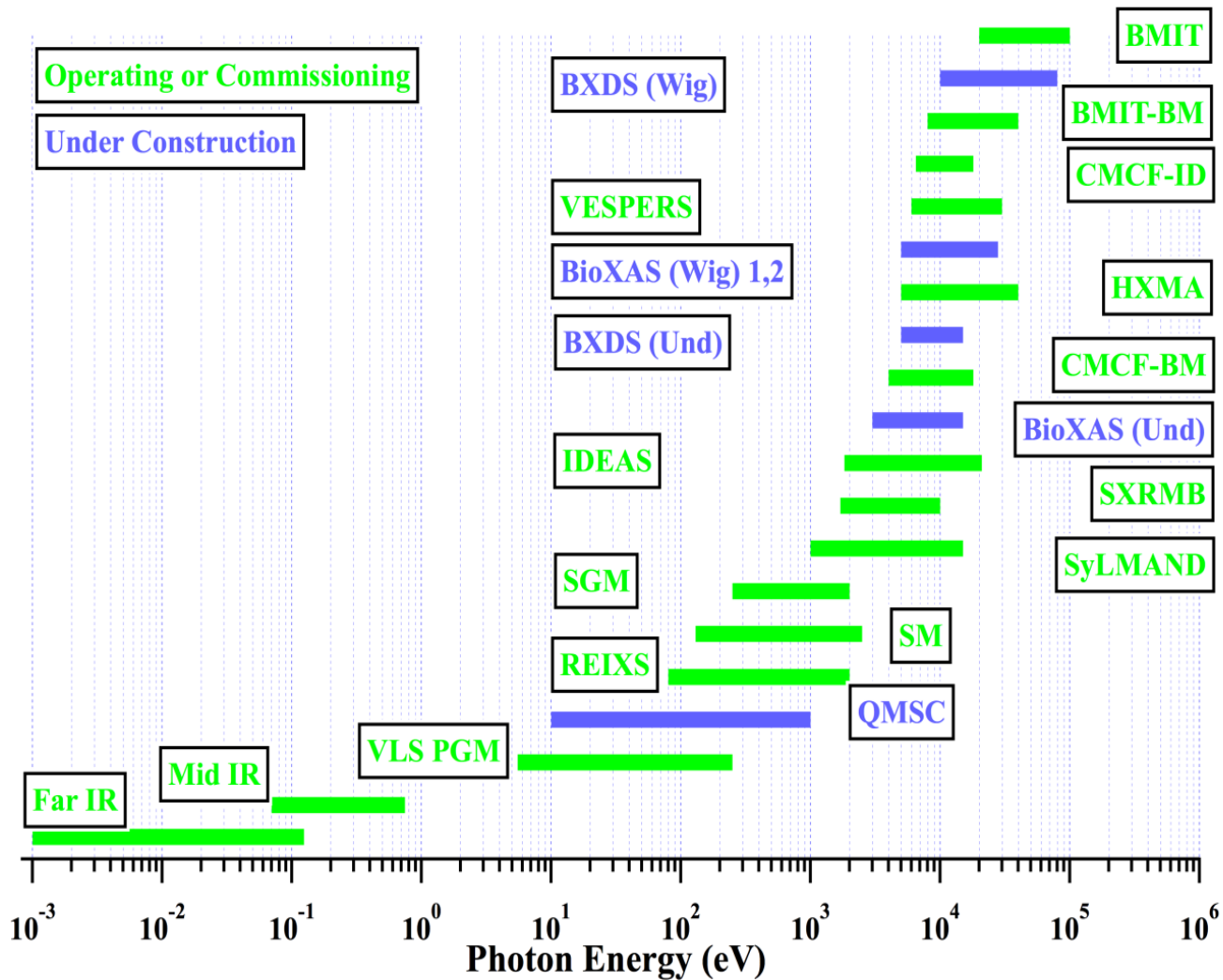
SaskPower





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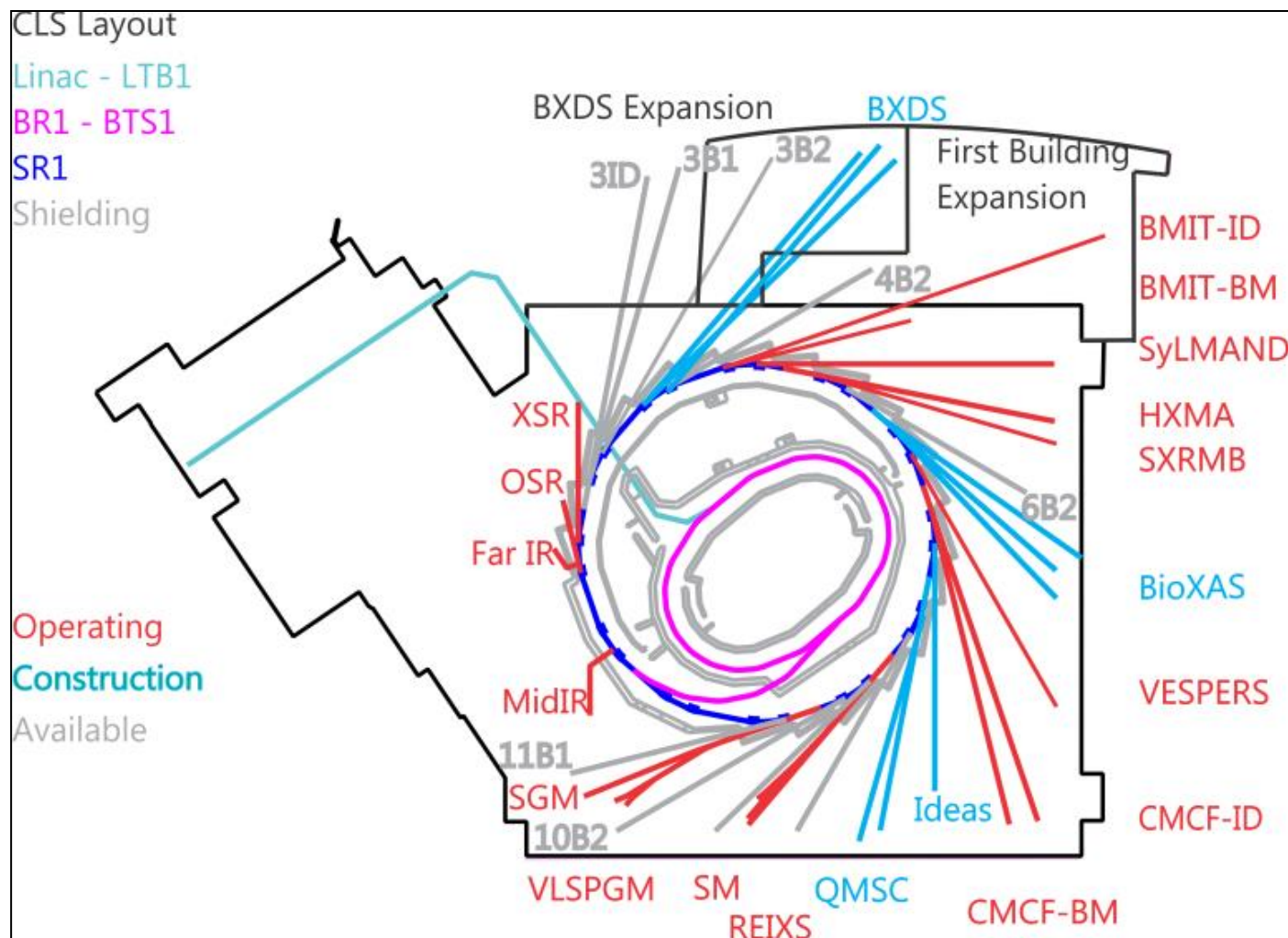
# Energy Range





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# CLS Layout

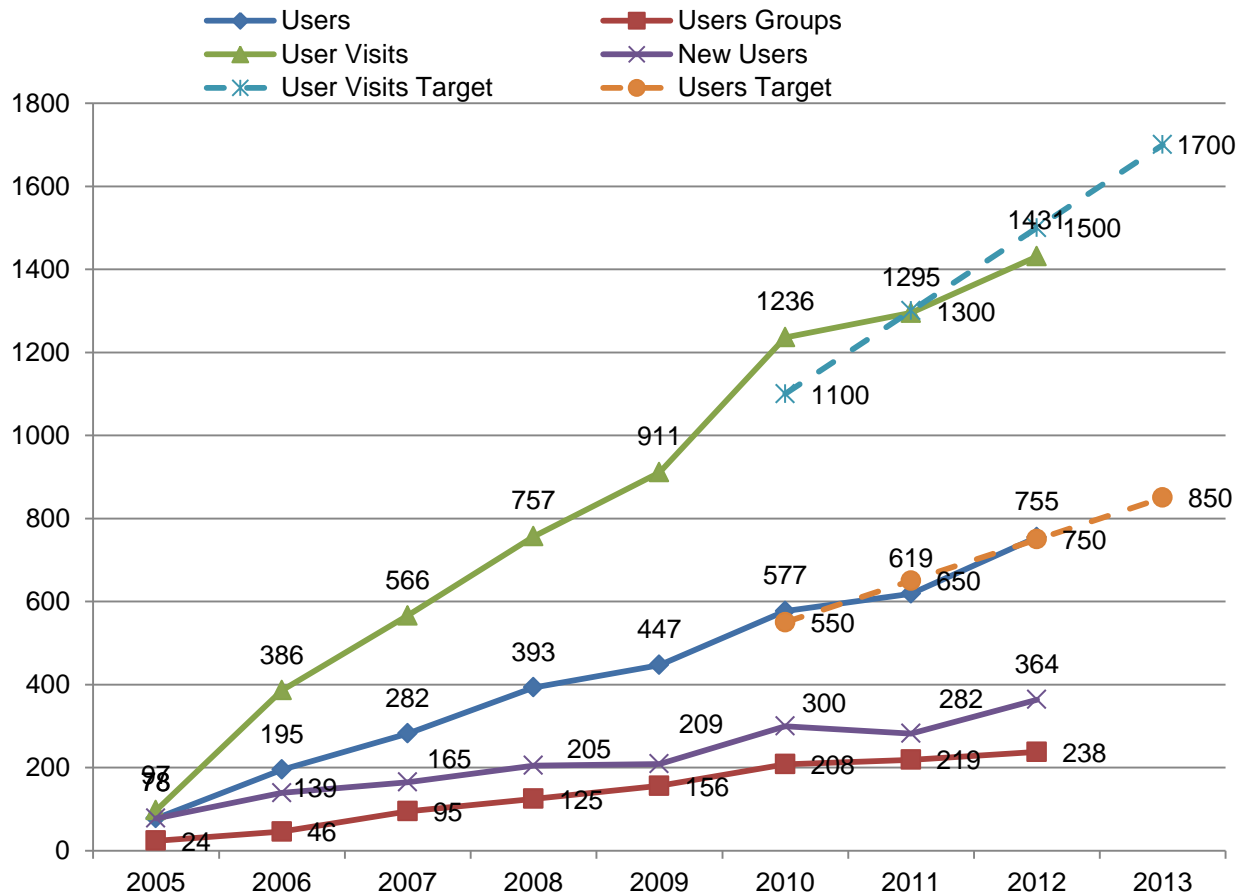






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Light de rayonnement  
Source synchrotron

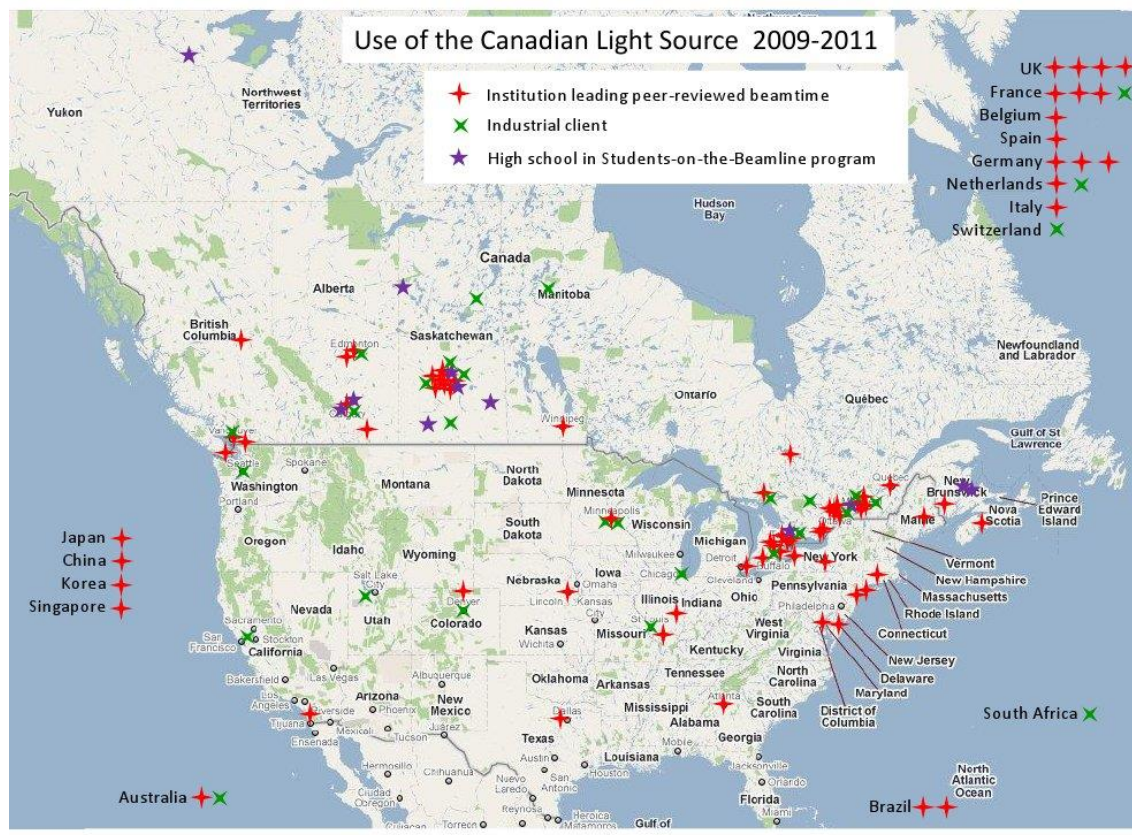
# Users and User Visits





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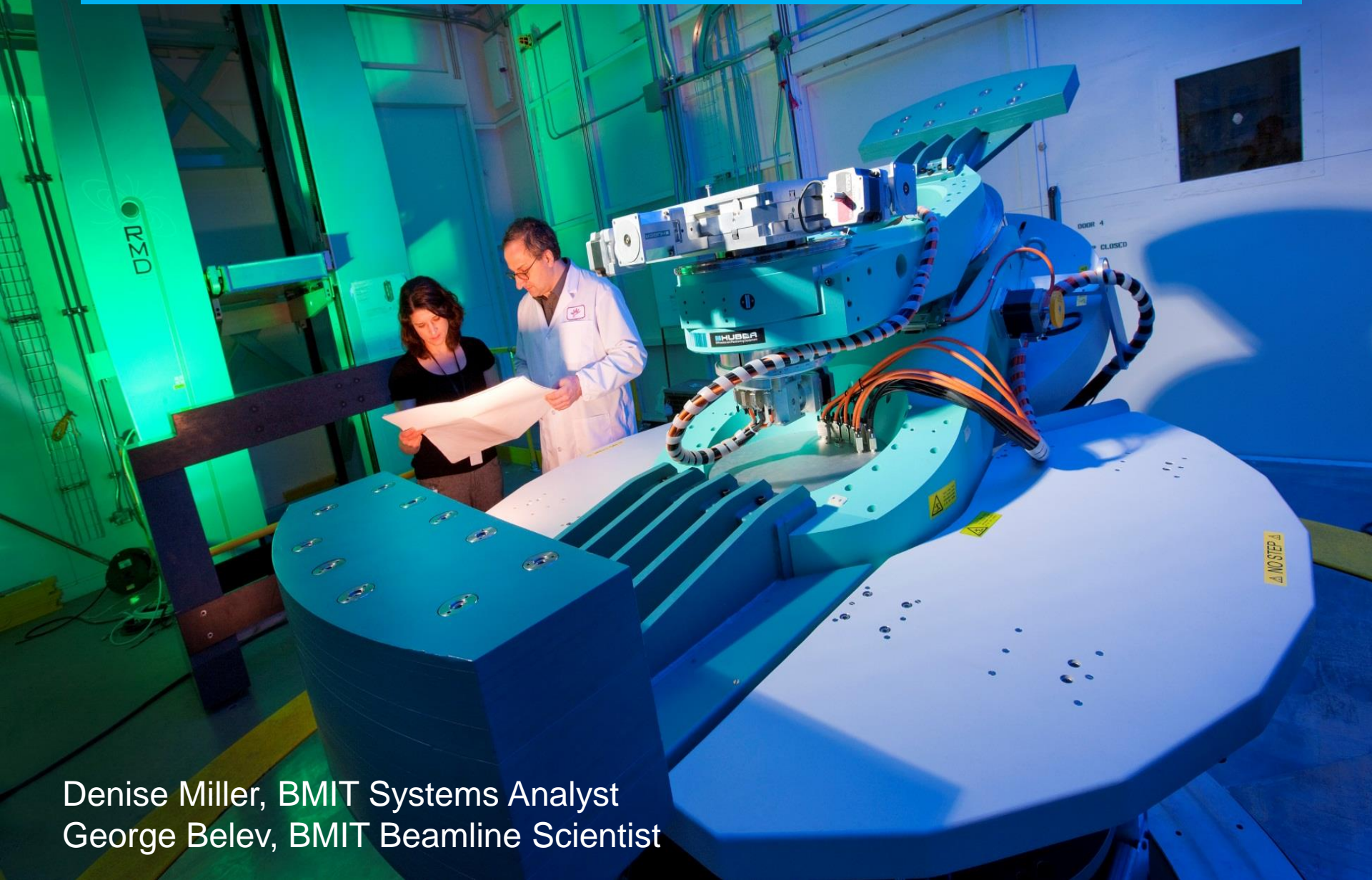
# The nature of our user base



Geographic distribution of shifts	2008	2009	2010
Canada – SK	560 (46%)	590 (35%)	716 (30%)
Canada – other provinces	554 (45%)	828 (49%)	1233 (52%)
International	114 (9%)	275 (16%)	406 (17%)



# Unique in the world: the large animal positioning system on the Biomedical Imaging and Therapy (BMIT) Beamline



Denise Miller, BMIT Systems Analyst  
George Belev, BMIT Beamline Scientist



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# Call for Proposals

**A call for proposals is issued twice per year for experimental beam time**

Call Open	Proposal Deadline	Review Date (Safety, Technical, Peer Review)	Peer Review Meeting (week of)	Results Announced (week of)	Scheduling Period Begins	Scheduling Period Ends	Cycle
Jan.30/13	Feb.27/13	Apr. 8/13	Apr.29/13	May 13/13	July 1/13	Dec. 31/13	18
Aug.1/12	Sept.5/12	Oct.15/12	Nov. 5/12	Nov.19/12	Jan. 1/13	June 30/13	17

## **Submit a Proposal:**

**Step 1:** Contact the appropriate [CLSI beamline scientist](#) to discuss your research.

**Step 2:** Logon to <https://user.lightsource.ca>.

If you have not previously [registered](#) you will be required to register and you will receive a username prior to submitting a proposal.



# Peer Review

## Evaluation Criteria

Each proposal is reviewed and scored by at least three external reviewers and at least one member of the Peer Review Committee. Reviewers are asked to provide an integer score in each of the three Evaluation Criteria, as described below

### **Quality of scientific research in the context of the field**

Does the proposal describe what is to be studied and the importance of it? What hypothesis would be tested, how will the results impact the field, and what is the likelihood of success?

### **Suitability of CLS resources being allocated relative to the proposed research**

Is this a good use of CLS resources? Does the experiment require the resources being requested? Reviewers are also asked to comment on the appropriateness of the number of shifts that have been requested.

### **Quality and capability of the researchers based on their track record**

Does the research team have recent synchrotron and/or other relevant experience? If they are a past user they should have clearly demonstrated their track record and productivity in the proposal.



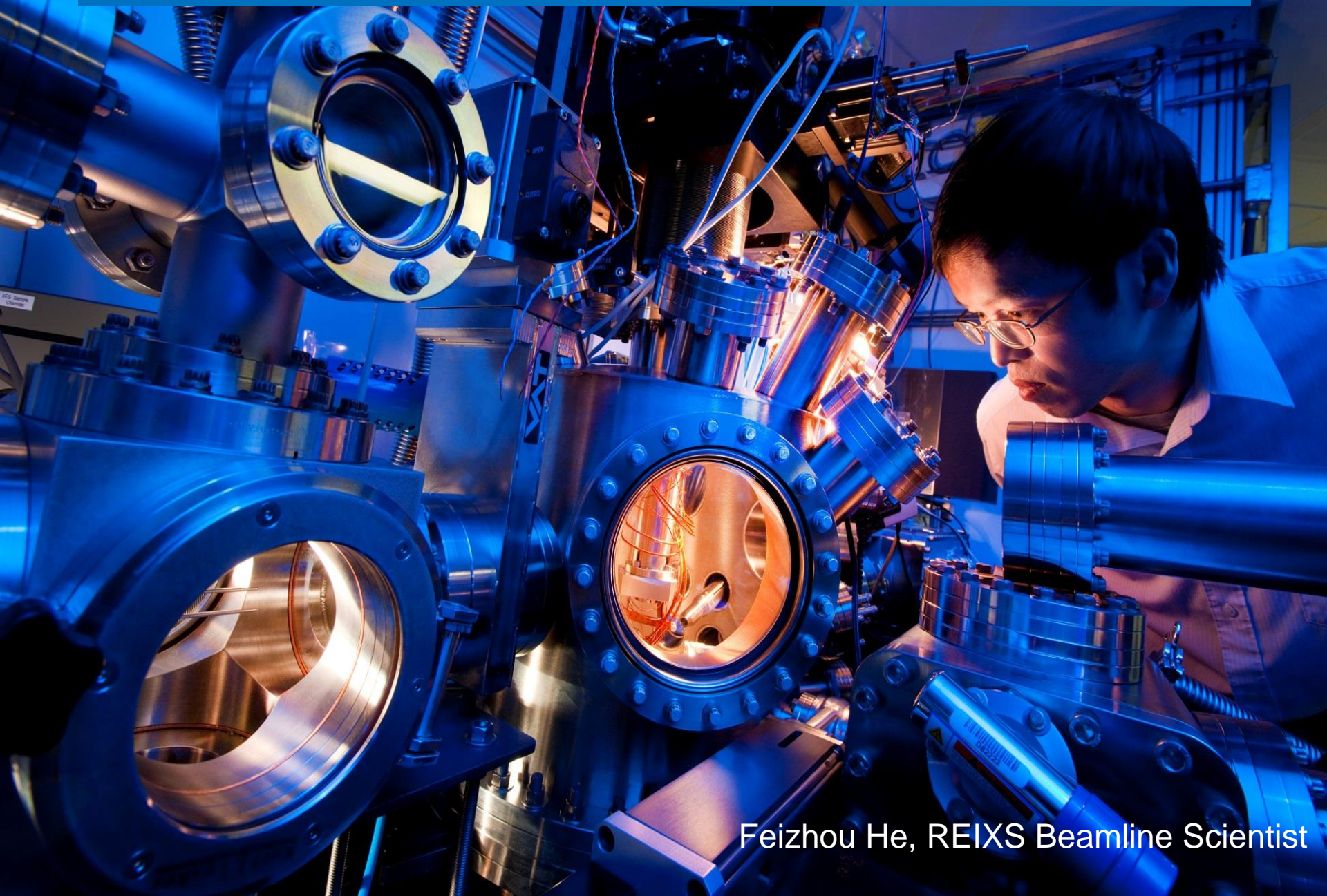
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# Peer Review Access

	2009	2010	2011
Number of shifts requested	1768	2675	3456
Number of shifts allocated	1252	1816	2203
Oversubscription	41%	47%	57%



A global leader in synchrotron science and its applications



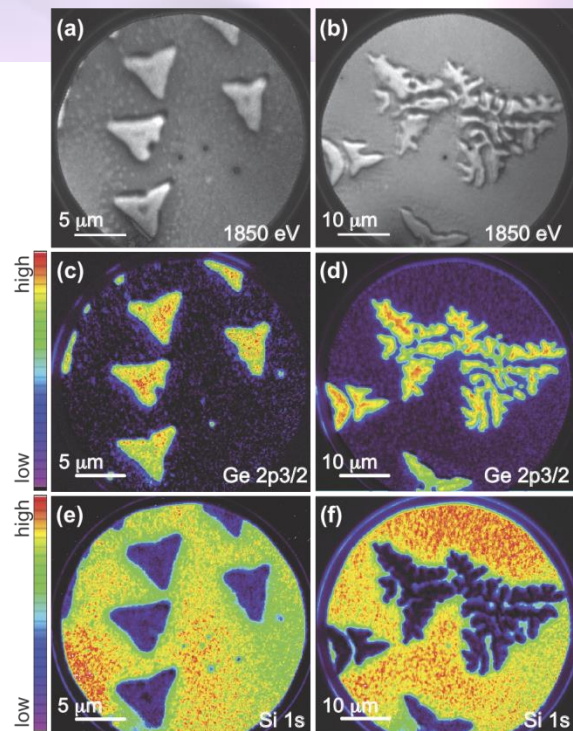
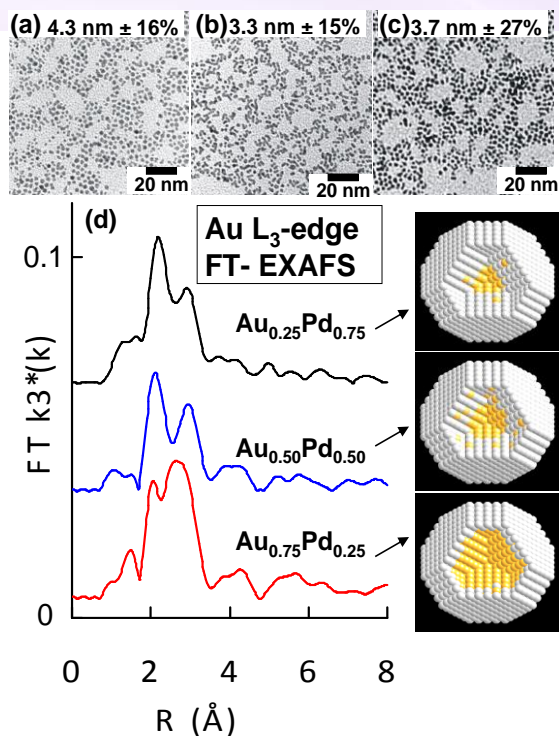
Feizhou He, REIXS Beamline Scientist





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# Nanoscience



The local structure of Au-Pd nanoparticles capped with mixed amine and bromide ligands: An EXAFS Study

F. Liu and P. Zhang

Department of Chemistry, Dalhousie University

Si/Ge Intermixing in Very Large Ge Islands on Si(111)

J. M. MacLeod (1, 2), J. A. Lipton-Duffin (1, 2),  
F. Rosei (1)

(1) INRS-EMT, Université du Québec, 1650 Boul. Lionel Boulet  
J3X 1S2 Varennes (QC), Canada

(2) Current address: Dipartimento di Fisica, Università degli  
Studi di Trieste, Trieste (TS), Italy



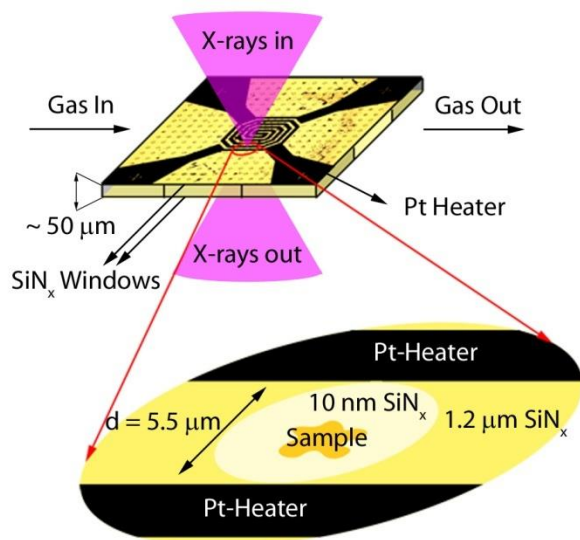
# Nanoscale Chemical Imaging of the Reduction Behaviour of a Single Catalyst Particle

E. de Smit (1), I. Swart (1), J. F. Creemer (2), C. Karunakaran (3), D. Bertwistle (3), H. W. Zandbergen (2), B.M. Weckhuysen (1) and F. M. F. de Groot (1)

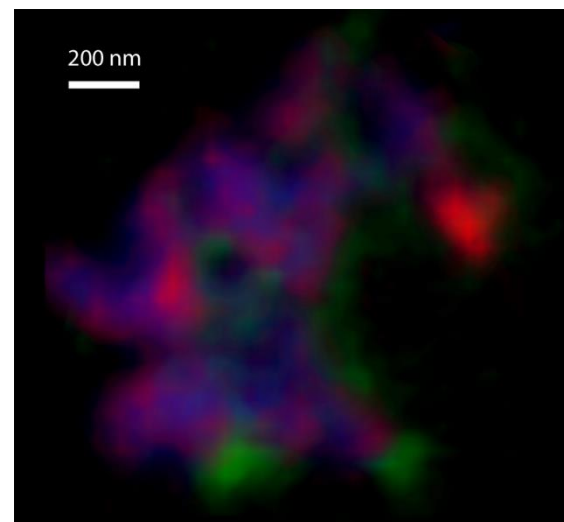
(1) *Utrecht University, The Netherlands*

(2) *Delft University of Technology, The Netherlands*

(3) *Canadian Light Source, Inc.*



**Figure 1:** Description and relevant dimensions of the nanoreactor chamber.



**Figure 2:** Chemical species contour map showing the distribution of Fe-species of different valence after treatment at 250°C in H<sub>2</sub>. Blue: mixed Fe<sup>2+/3+</sup> (Fe<sub>3</sub>O<sub>4</sub>), green: pure Fe<sup>2+</sup>, red: Fe<sup>0</sup>.



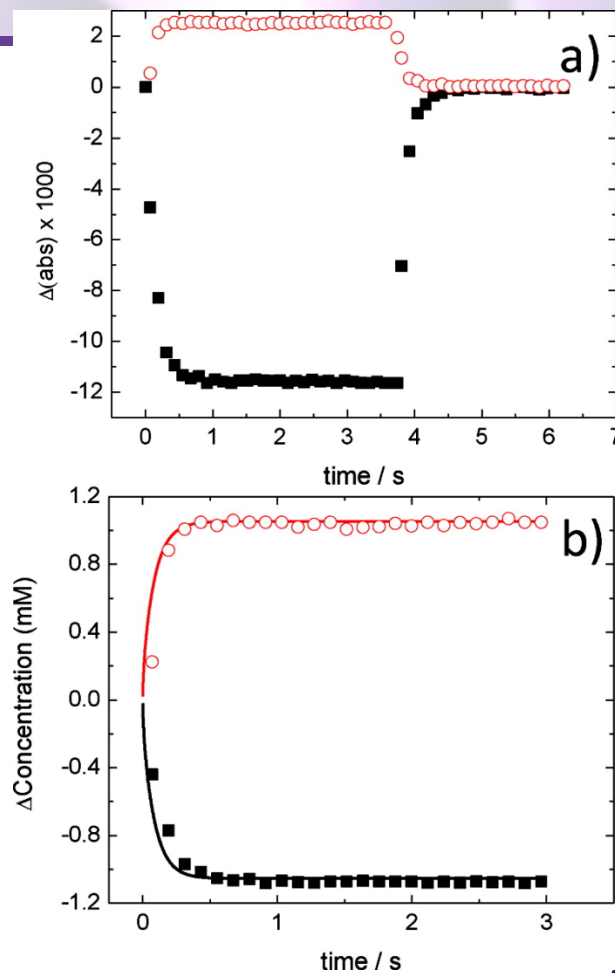
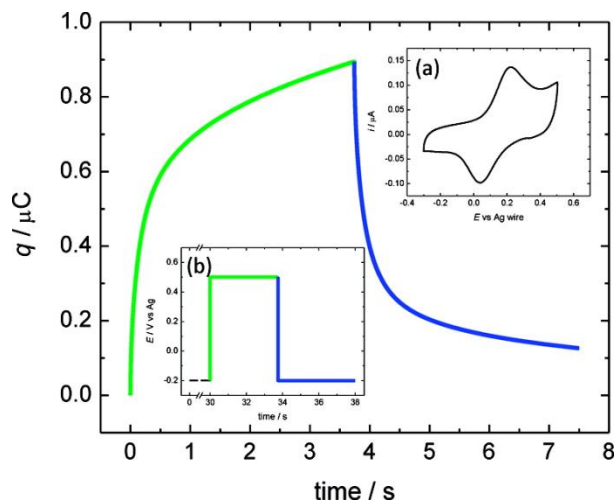
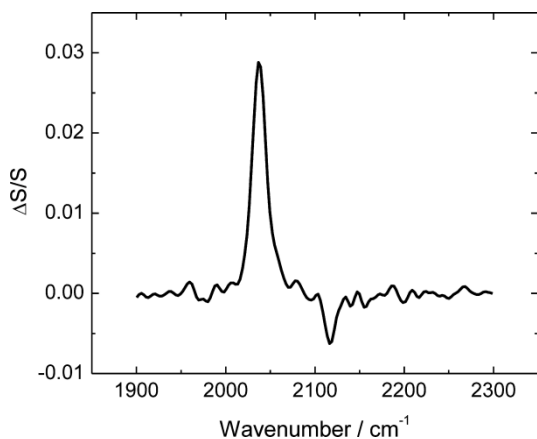
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Flow Through Holes

# Electrochemical external reflection IR spectroscopy

CE WE

TOP VIEW

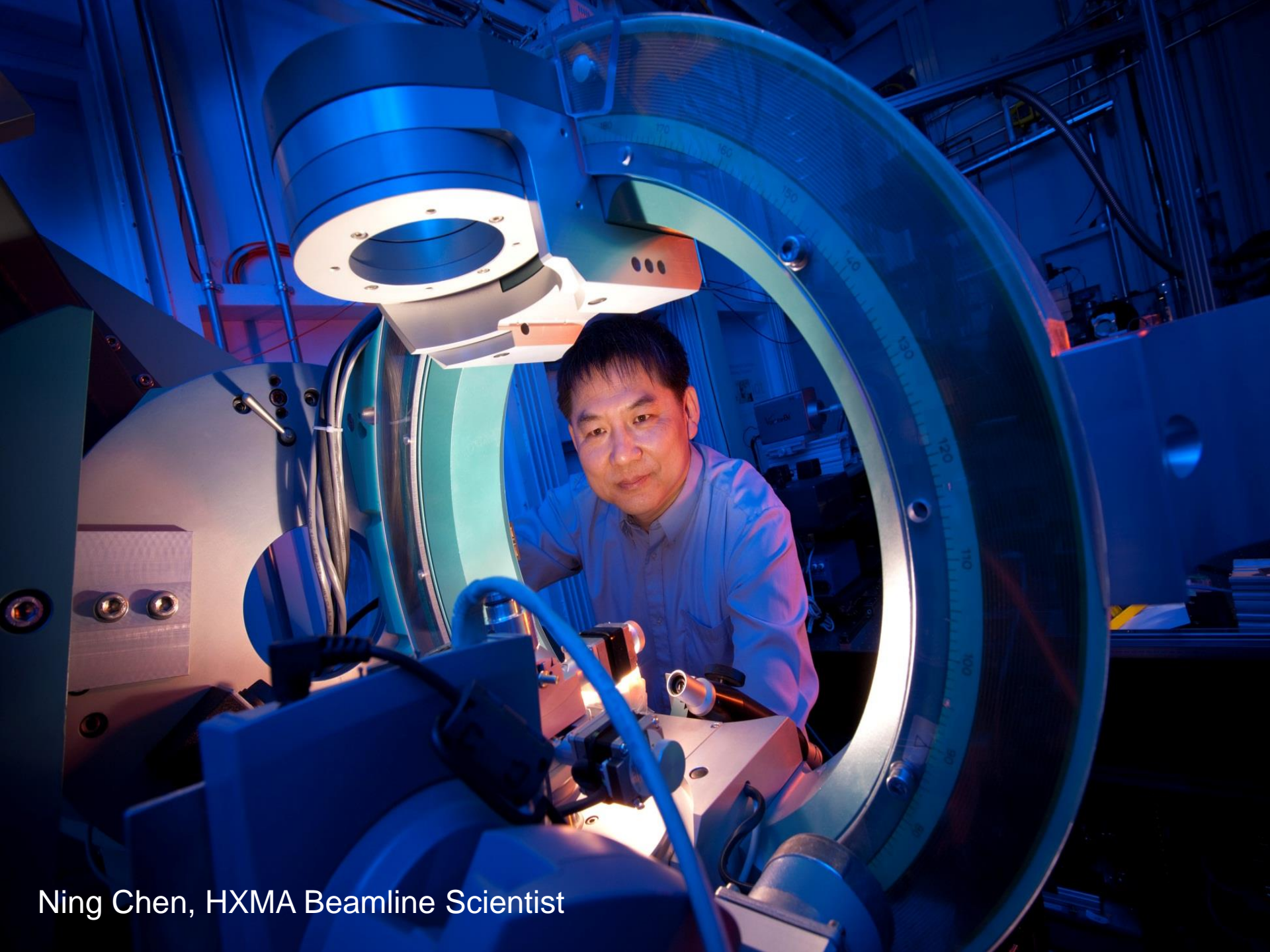
RE



Rosendahl et al., Anal. Chem. (2011)

Ian Burgess, Chemistry, University of Saskatchewan





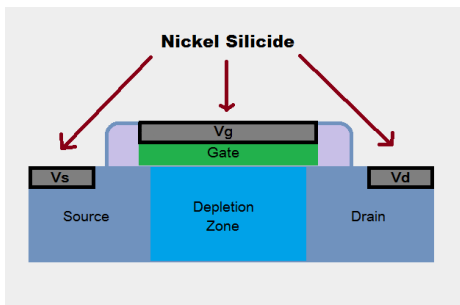
Ning Chen, HXMA Beamline Scientist



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Light Source de rayonnement  
synchrotron

# GI-XAFS Thin Film Studies at CLS HXMA

## Material applications

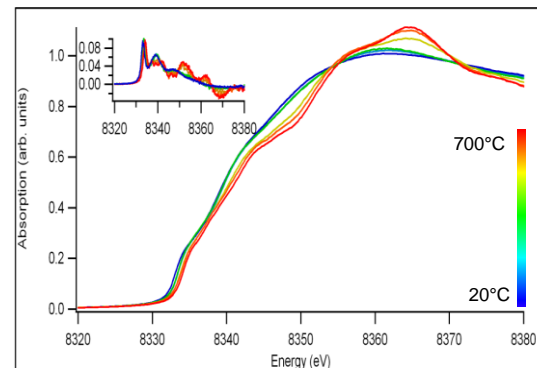
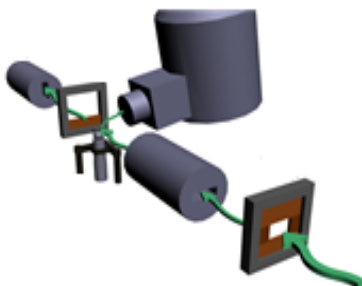


Ultrathin epitaxial Ni-Si film on  
Si(001):

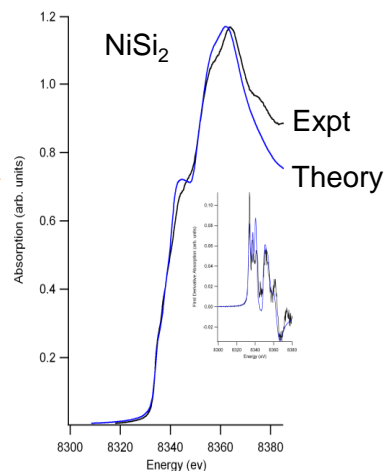
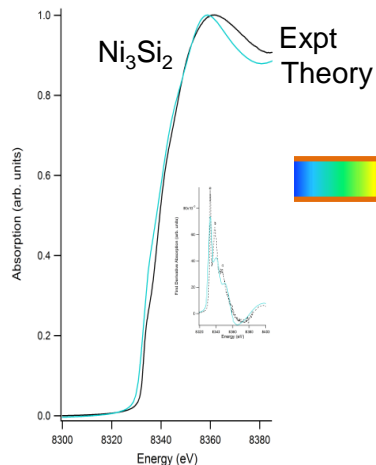
Z. Arthur<sup>1</sup>, K. Miller<sup>1</sup>, N. Chen<sup>2</sup>, C. Lavoie<sup>3</sup>, Y. Joly<sup>4</sup>, D.T. Jiang<sup>1</sup>

<sup>1</sup>U of Guelph, Canada; <sup>2</sup>CLS, Canada; <sup>3</sup>IBM T.J. Watson Research Ctr., USA; <sup>4</sup>CNRS Grenoble, France

CLS HXMA BL  
GI-XANES schematic

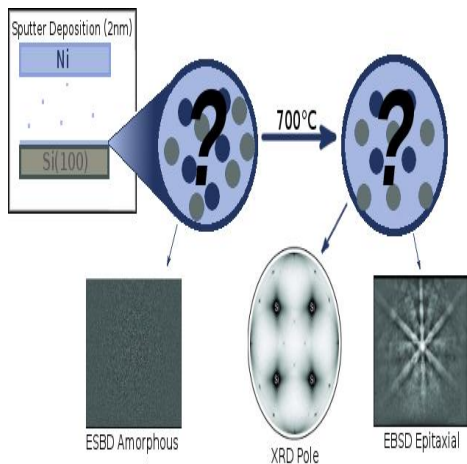


FDMNES Simulation



More detail in presentation: MDPEZ3

- Epitaxial phase is NiSi<sub>2</sub>
- As-deposited is mostly Ni<sub>3</sub>Si<sub>2</sub>
- Surface/interface effects dominate the phase formation sequence – seems NiSi phase is bypassed, different from thicker film cases.



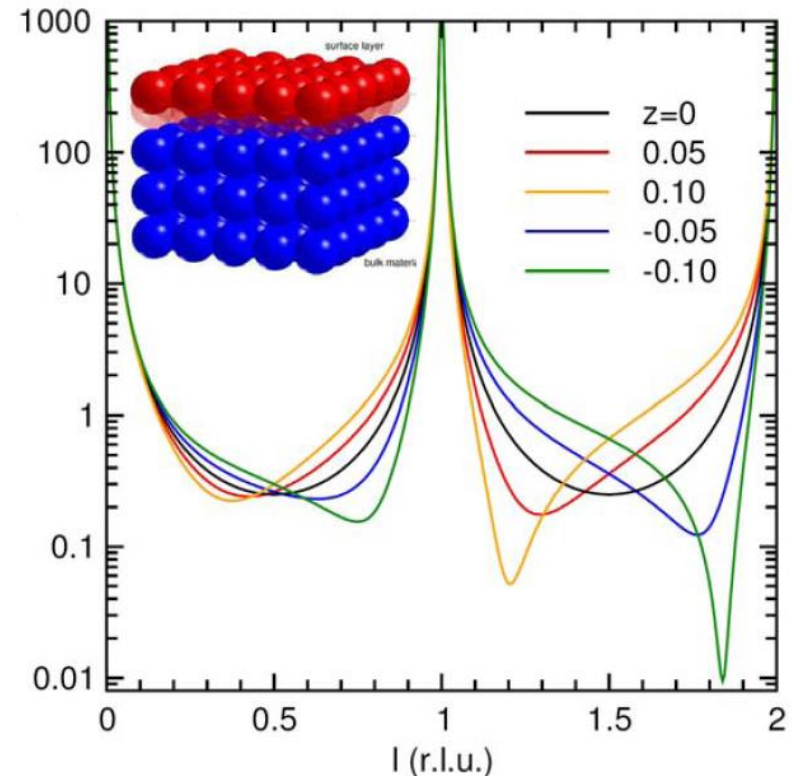
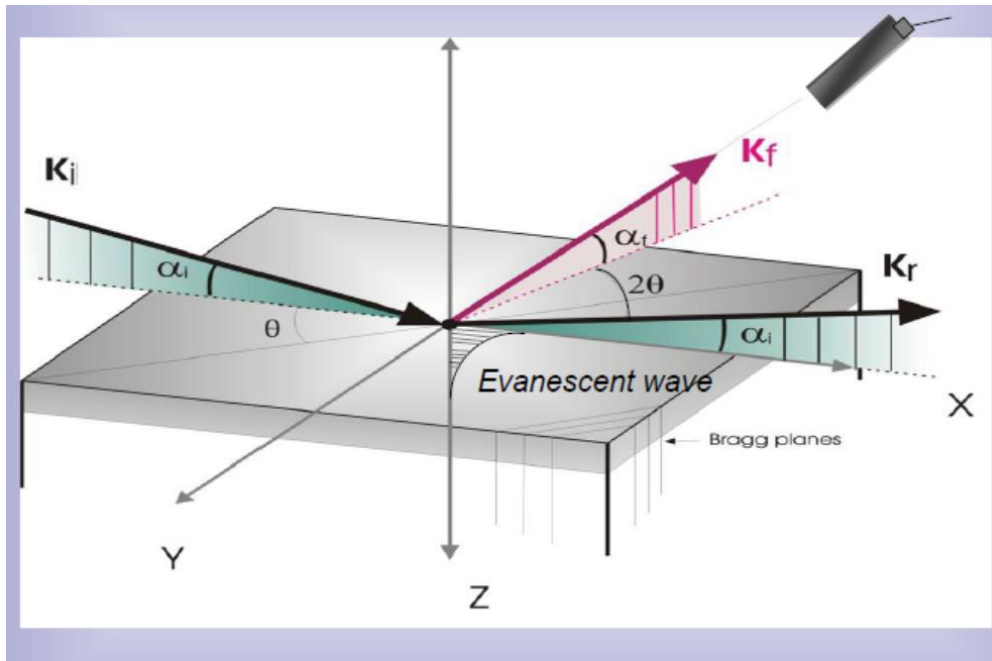


# Surface X-ray Scattering

X-ray scattering from an infinite crystal  $\rightarrow$  Bragg peaks.

Presence of crystalline surfaces  $\rightarrow$  truncation rods (normal to the surface).

Crystal Truncation Rod (CTR) measurements allow detailed determination of atomic structure at the surface.

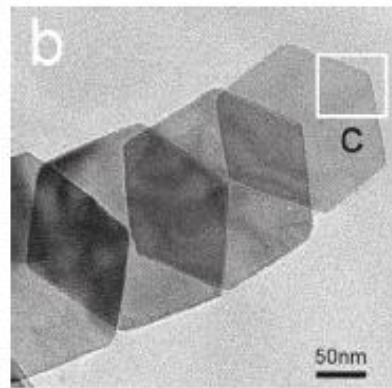
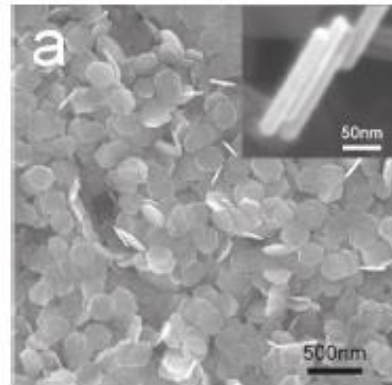


# Applying Surface X-ray diffraction technique

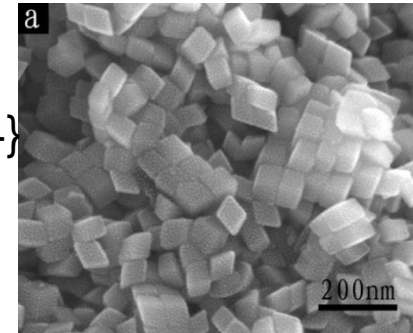
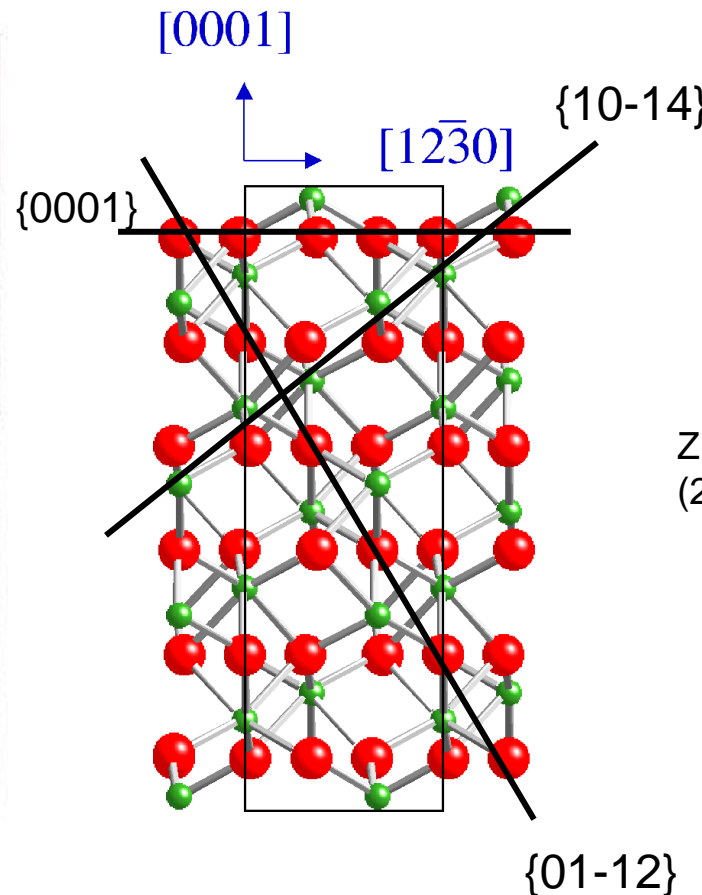
to uniform shape, mono-dispersive single crystalline nanoparticle →

Correlation of enhanced performance of nanoparticle to its surface structure

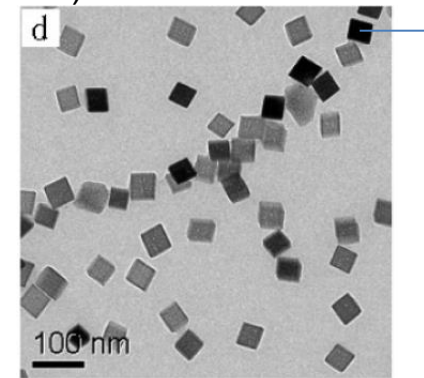
## Shape control of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles



$\alpha\text{-Fe}_2\text{O}_3$  nanoplate,  
L. Chen et al, Inorg. Chem.  
2010, 49, 8411–8420



$\alpha\text{-Fe}_2\text{O}_3$   
nanorhomboheda  
Z. Pu et al, Nanotechnology 17  
(2006) 799–804

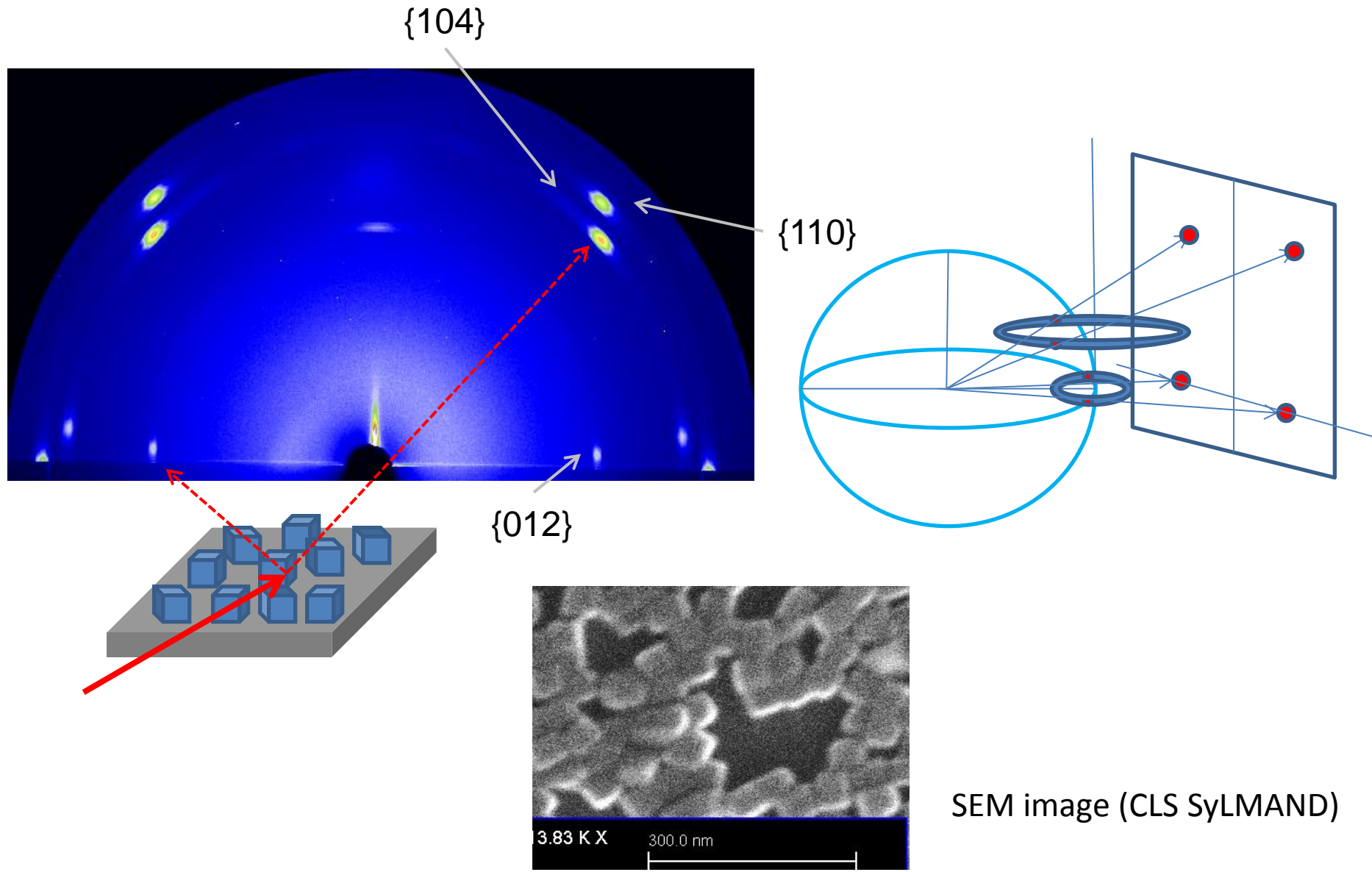


$\alpha\text{-Fe}_2\text{O}_3$  nanocube  
Y. Zheng et al, J. Phys. Chem. B  
2006, 110, 3093

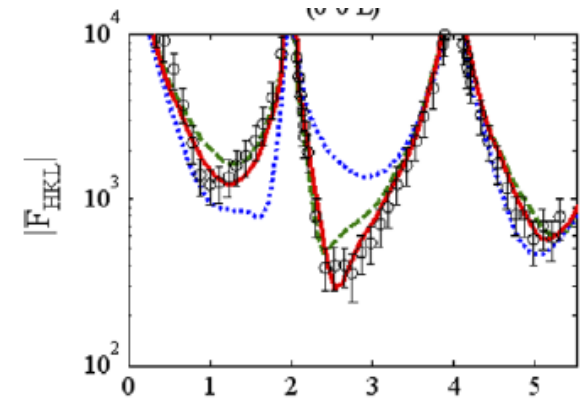
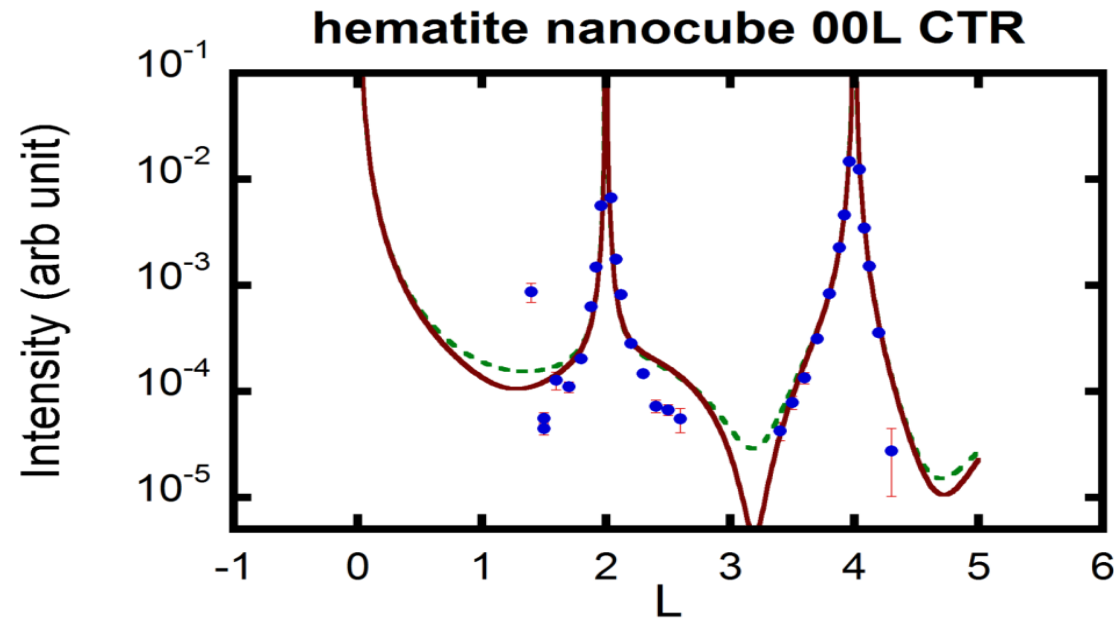


# Grazing incidence X-ray diffraction (GIXRD)

## from dip-coated hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) nano-cubes

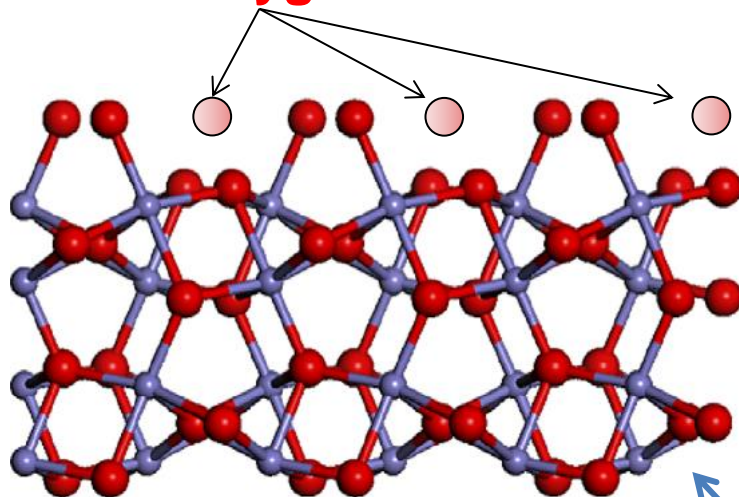


# Crystal truncation rod (CTR) from $\alpha\text{-Fe}_2\text{O}_3(012)$ nanocube



CTR from single crystal  
K.S. Tanwar et al. / Surface Science  
601 (2007) L59–L64

**Extra oxygen on NC surface**



*Hydroxylated stoichiometric termination (Model C)*

Single crystal model

**Existence of extra oxygen on hydroxylated nanocube surface agrees well with observations that nanoparticle can be easily hydroxylated**

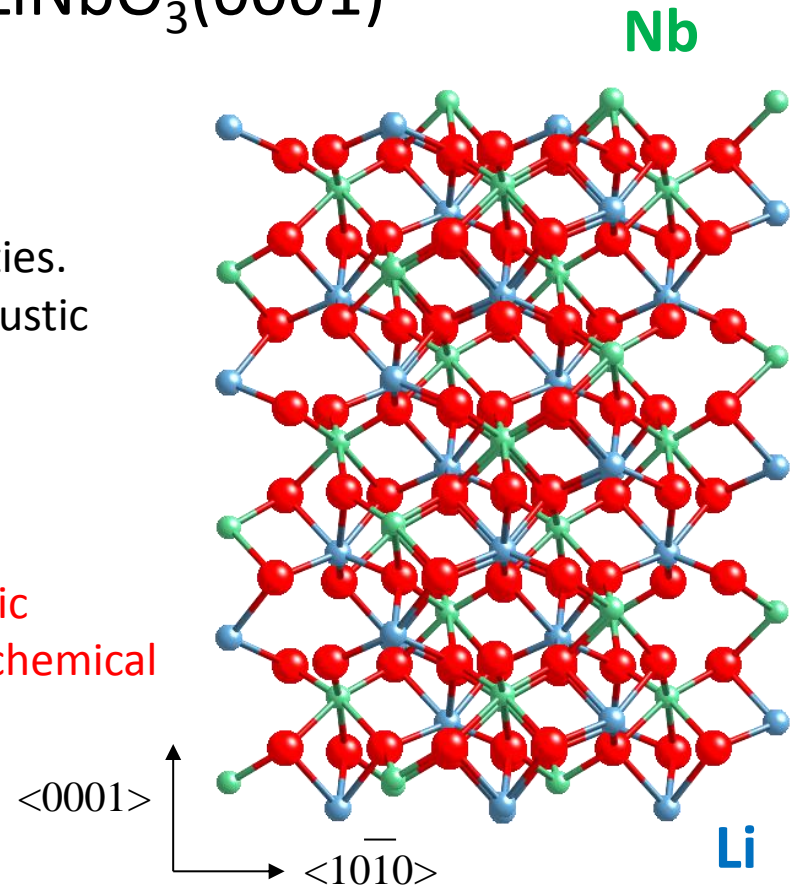


# Surface X-ray Scattering Study of Polarization Dependent Surface Structure and Stoichiometry of $\text{LiNbO}_3(0001)$

## Lithium Niobate ( $\text{LiNbO}_3$ )

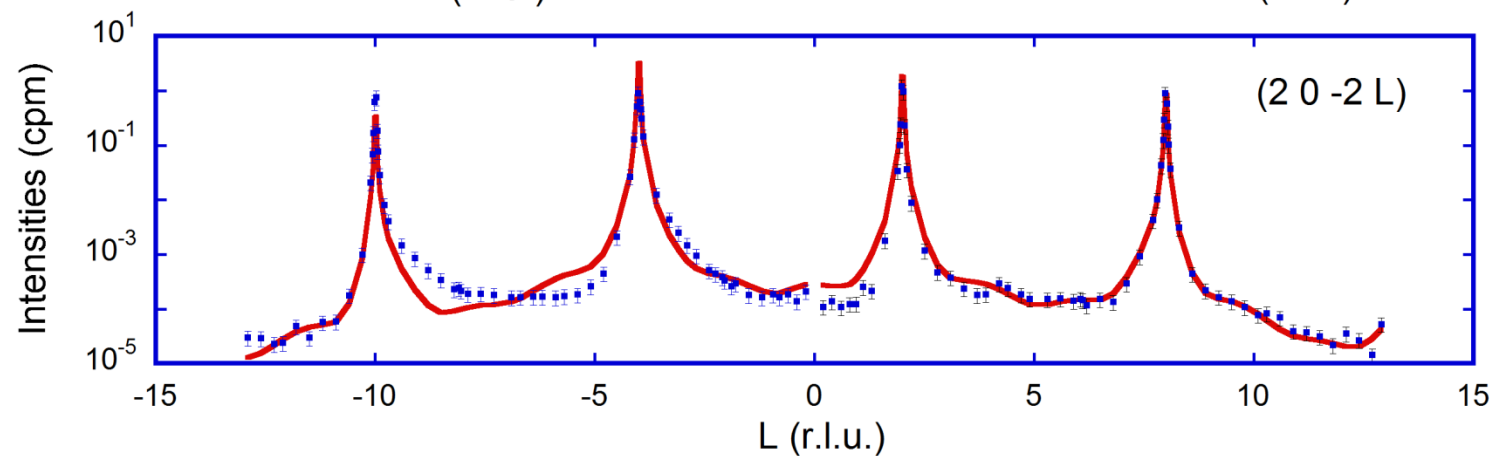
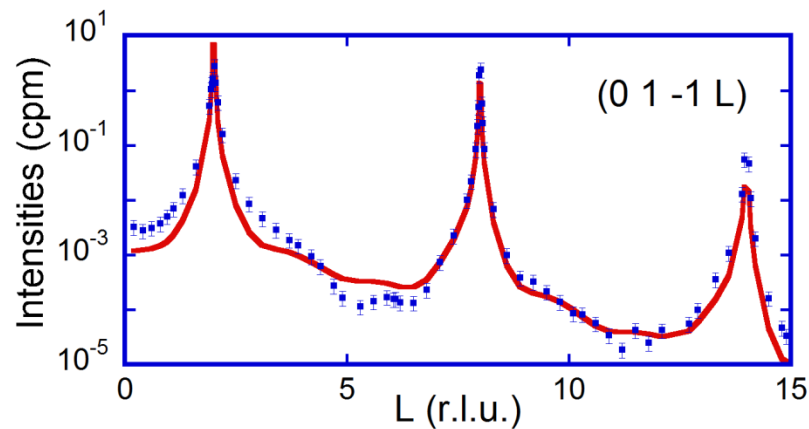
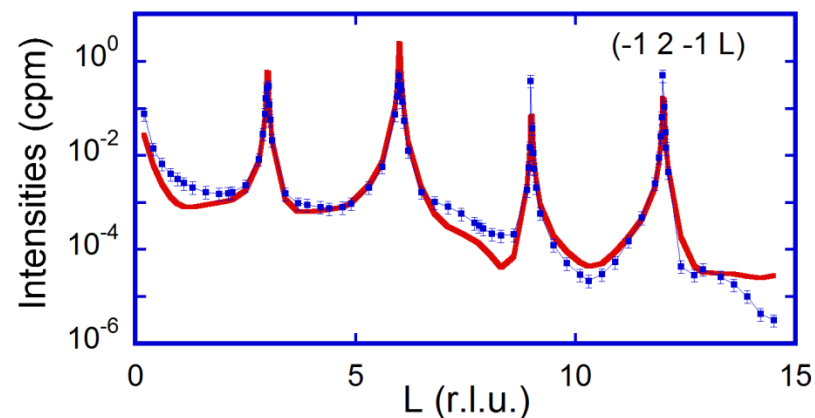
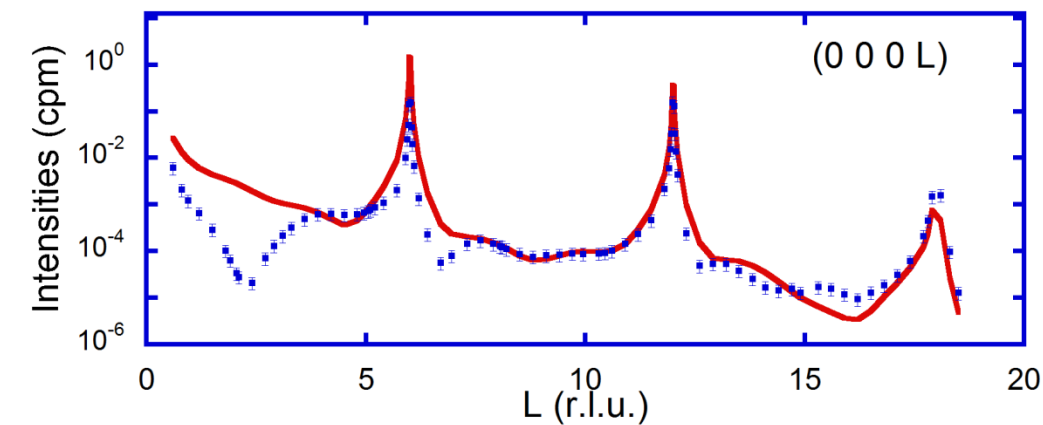
- Ferroelectric material with pyroelectric, piezoelectric and nonlinear optical properties.
- Widely used for electro-optic, surface acoustic wave generation, and second harmonic generation.

Oppositely poled LNO(0001) surfaces have different stoichiometries, atomic/electronic structures, photo-catalytic activities, and chemical reactions.

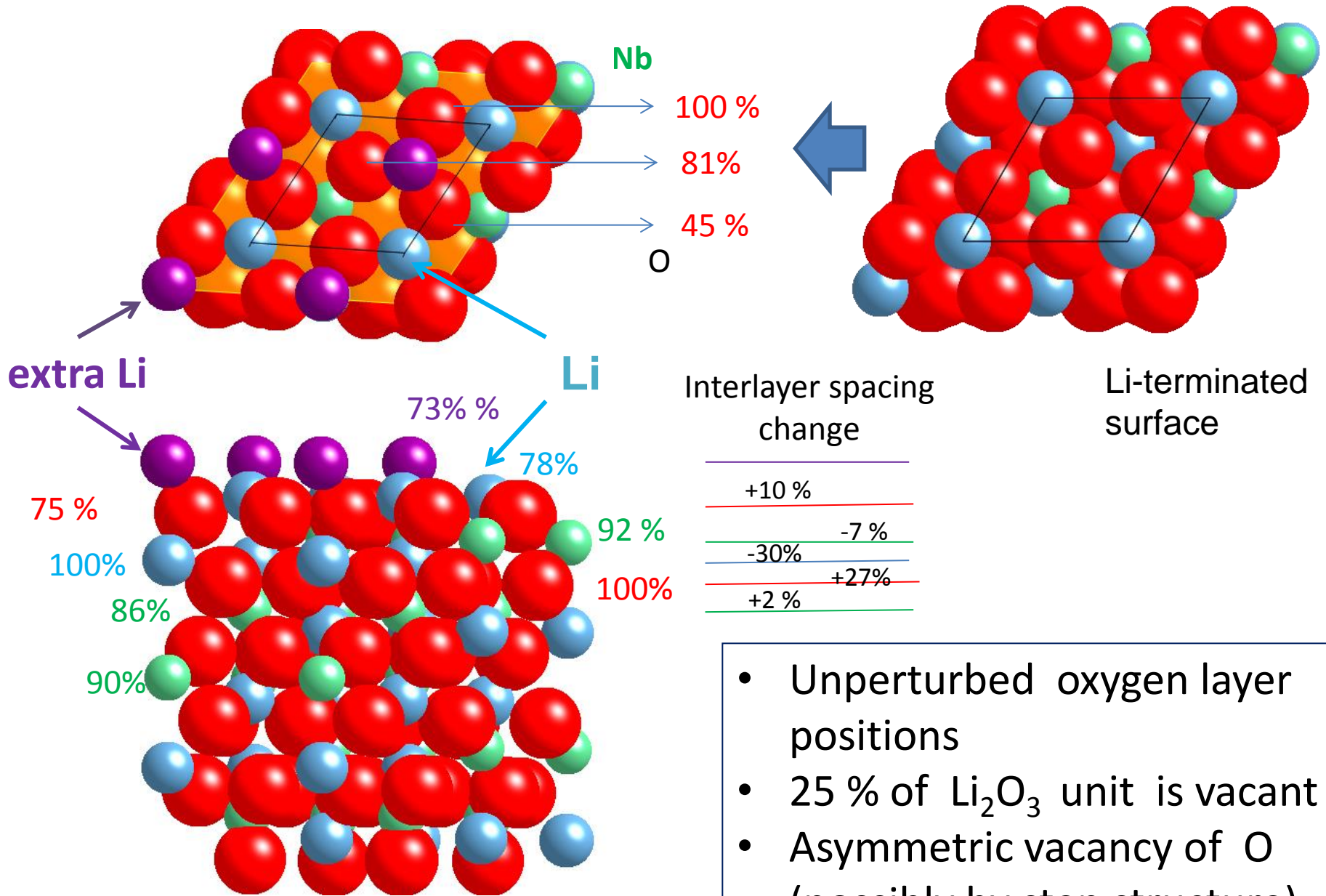


Surface structure characterization is essential for further study of the polarization dependent properties.

**Model fit of CTR data  
from positively  
polarized surface**



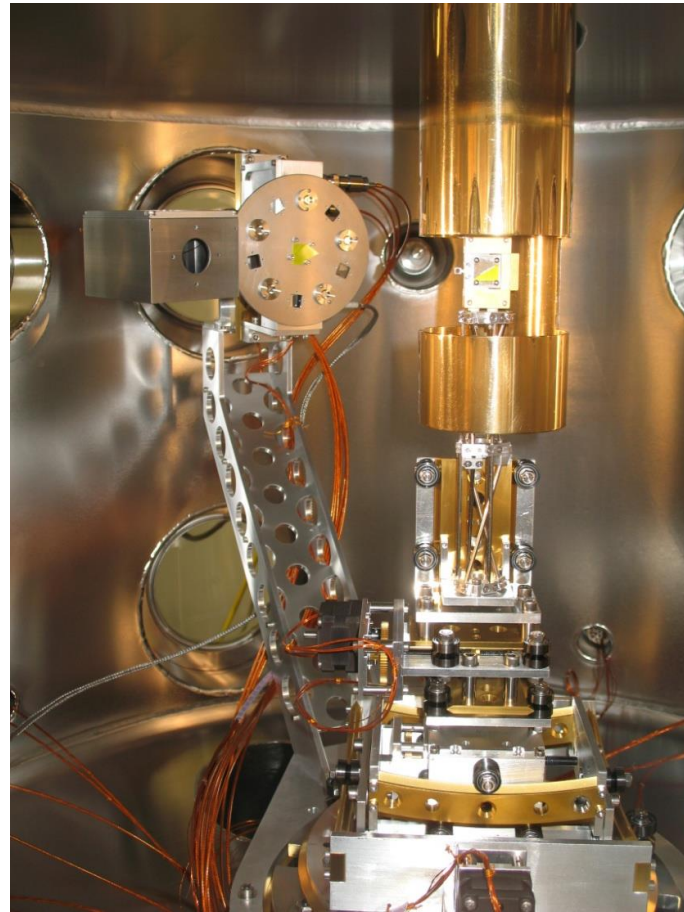
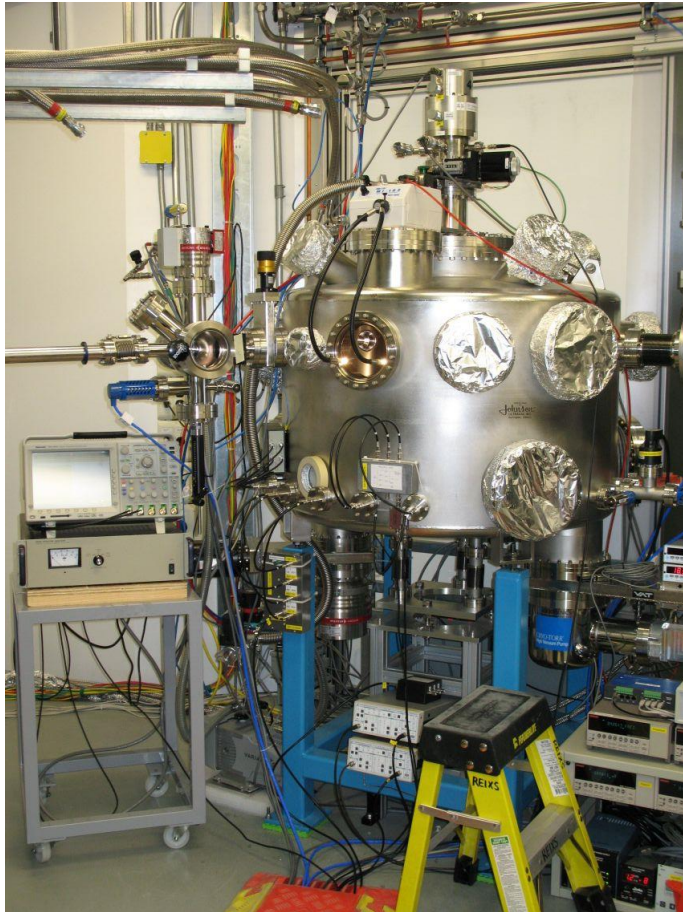
# Best fit model of positively polarized surface





# RSXS Endstation

- Resonant Soft X-ray Scattering (RSXS)
- X-ray Absorption Spectroscopy (XAS) by TEY and TFY
- Magnetic Circular Dichroism (MCD)
- X-ray Reflectivity



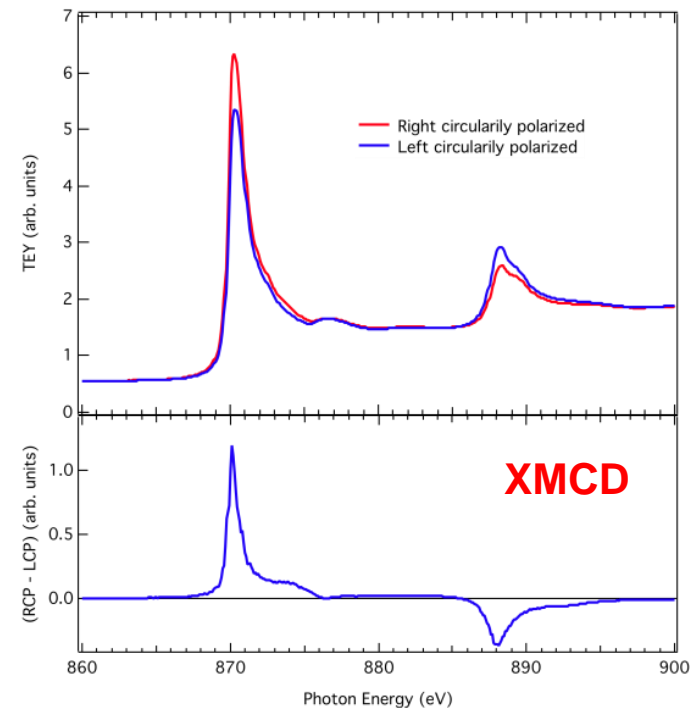
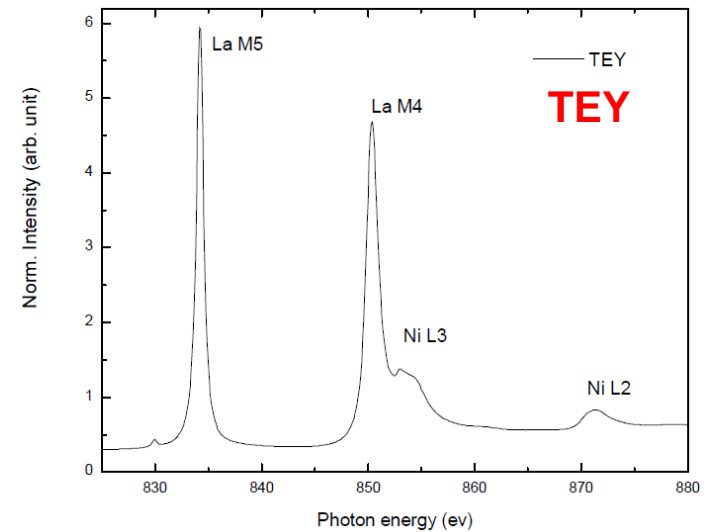
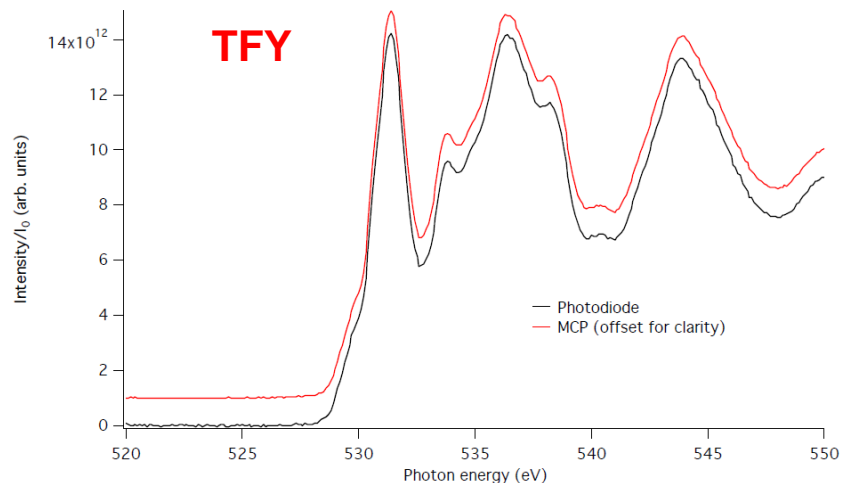
# Experimental Techniques

- **X-ray Absorption Spectroscopy**
  - TEY – sample current
  - TFY – Photodiode, Channeltron, Microchannel Plate (MCP)
- **XMCD – X-ray Magnetic Circular Dichroism**

High energy resolution

Polarized incoming beam

Measure TEY, specular reflection, fluorescence yield simultaneously



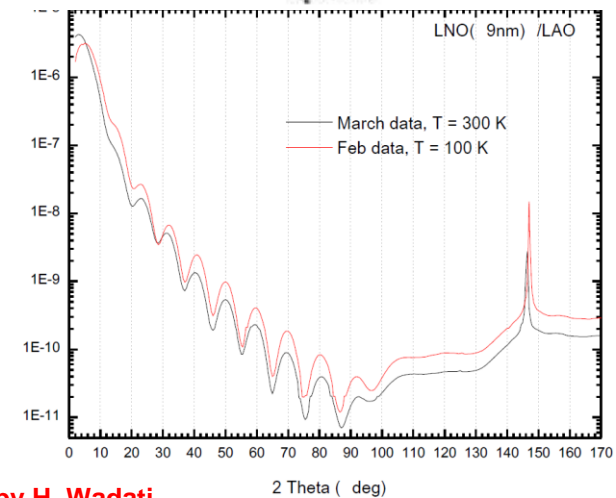
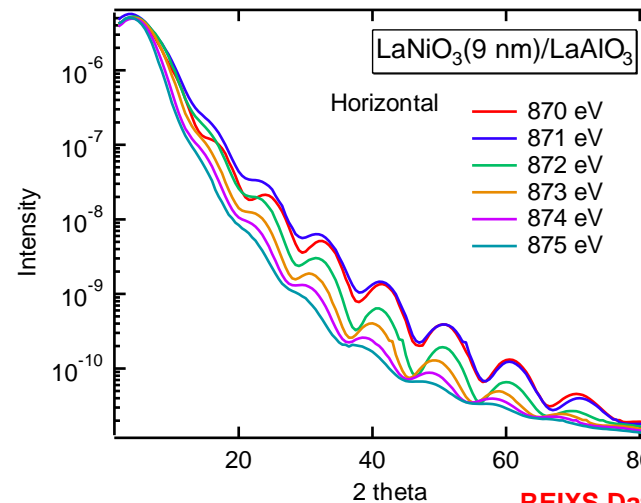
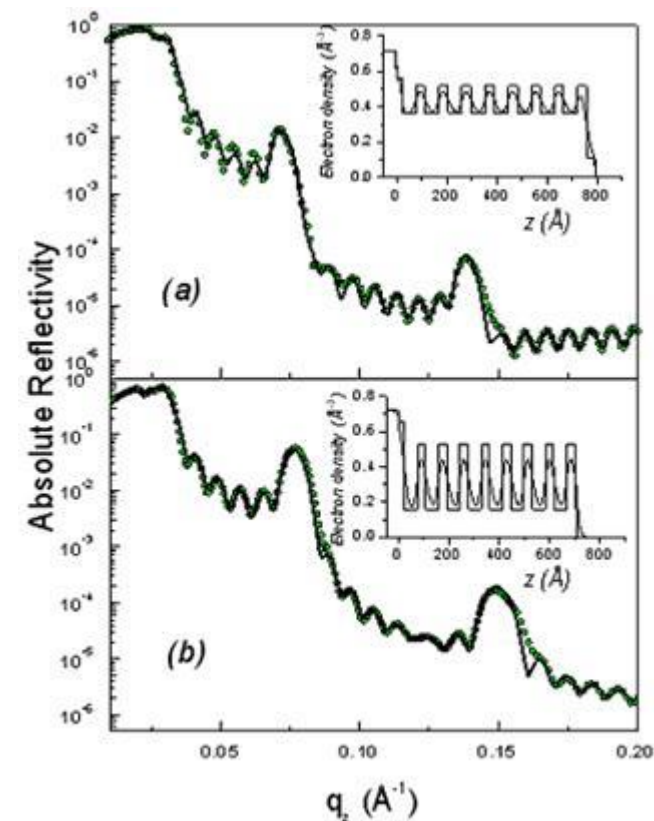
# Experimental Techniques

## ➤ X-ray Reflectivity

- Wide  $2\theta$  and  $\theta$  range (maximum  $2\theta$ :  $172^\circ$ )
- Energy dependence, temperature dependence, polarization dependence

Information on:

- Electron density profile in thin films, multilayers, interface
- Film thickness, multilayer periodicity
- Roughness of surface and interface



REIXS Data by H. Wadati



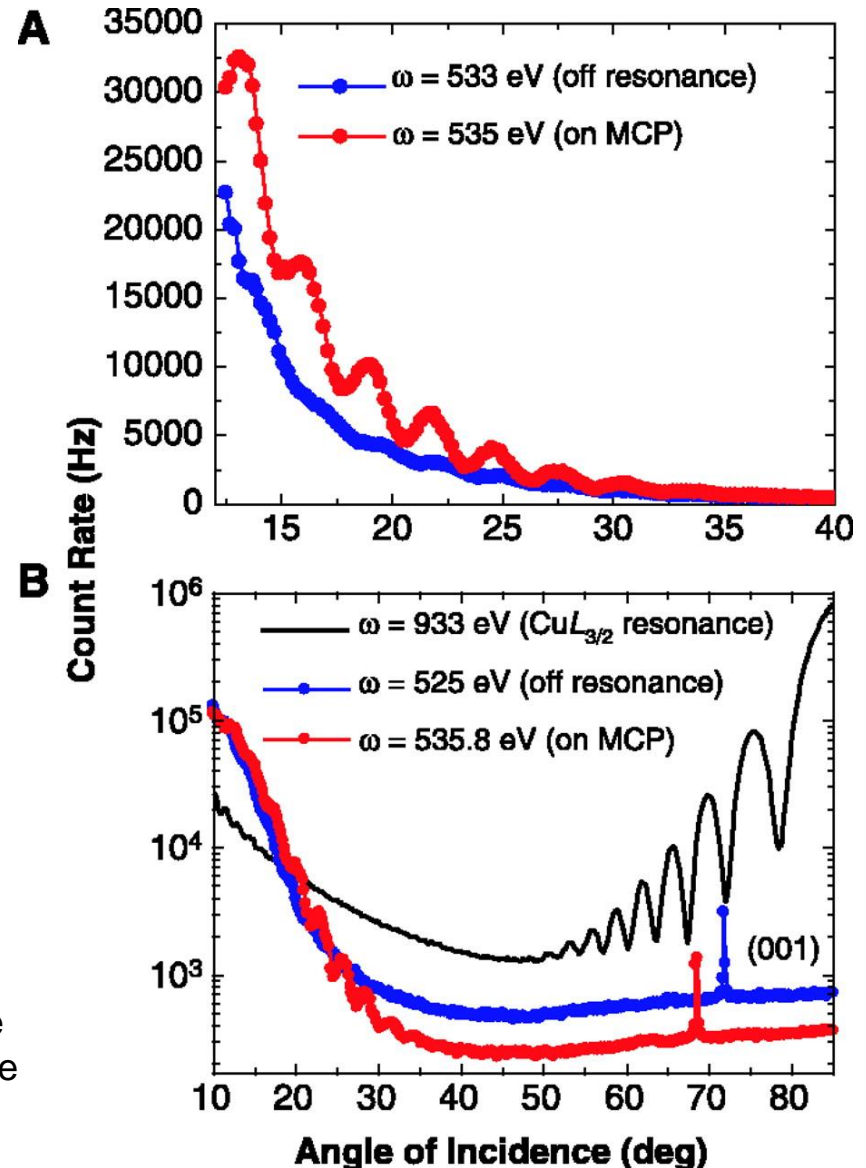
# Resonant Soft X-Ray Scattering

P. Abbamonte et al, Science 297, 581 (2002).

At resonance we have contrast for:

- Elements – each element has specific resonant energy → element selective
- Valence electron density
- Bond orientation; orbital ordering  
quadrupole moment orientation  
[linear pol. light]
- Spin density [circular pol. light and  $p$  or  $d$  core level]

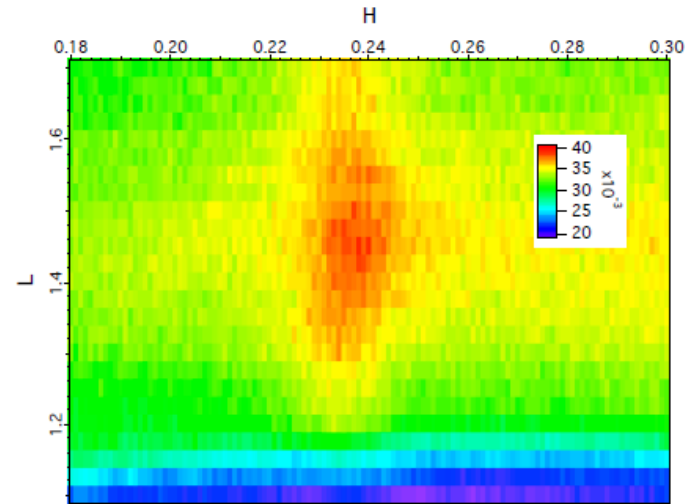
La<sub>2</sub>CuO<sub>4+δ</sub> film  
O  $K$  edge  
Cu  $L$  edge



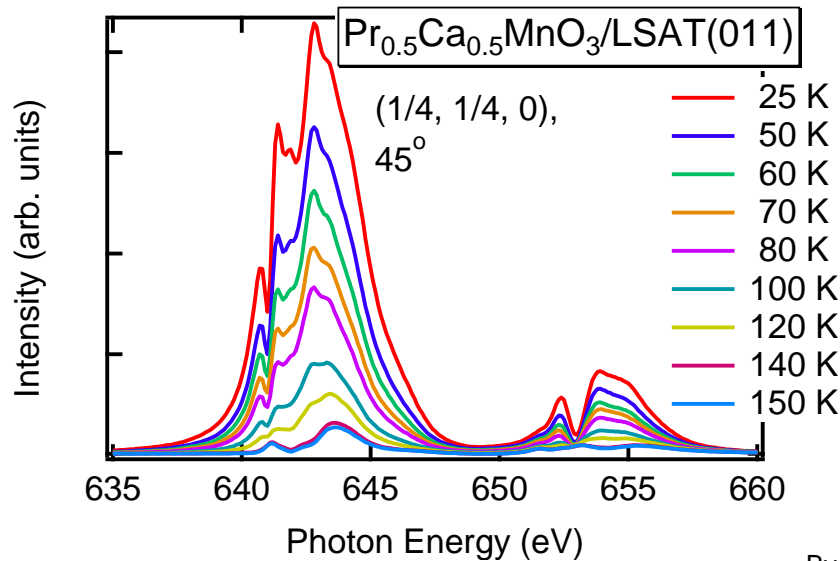
# Resonant Soft X-Ray Scattering

- REIXS RSXS
  - Temperature 18K – 400K
  - Energy 100 eV – 2500 eV
  - Arbitrary Polarization
  - FixQ energy scan
  - Reciprocal space mapping

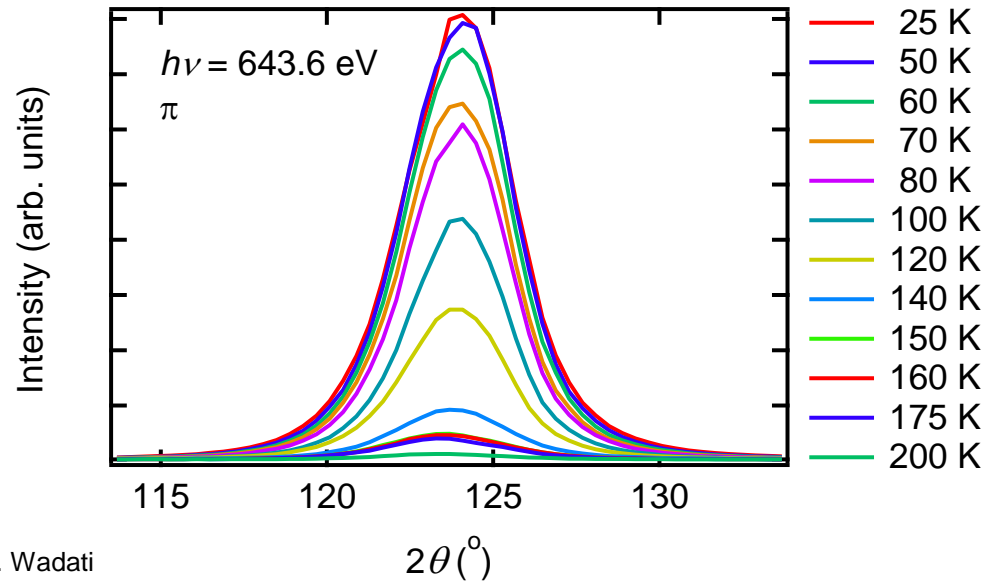
[H 0 L] mesh at 931.1 eV



By D. Hawthorn



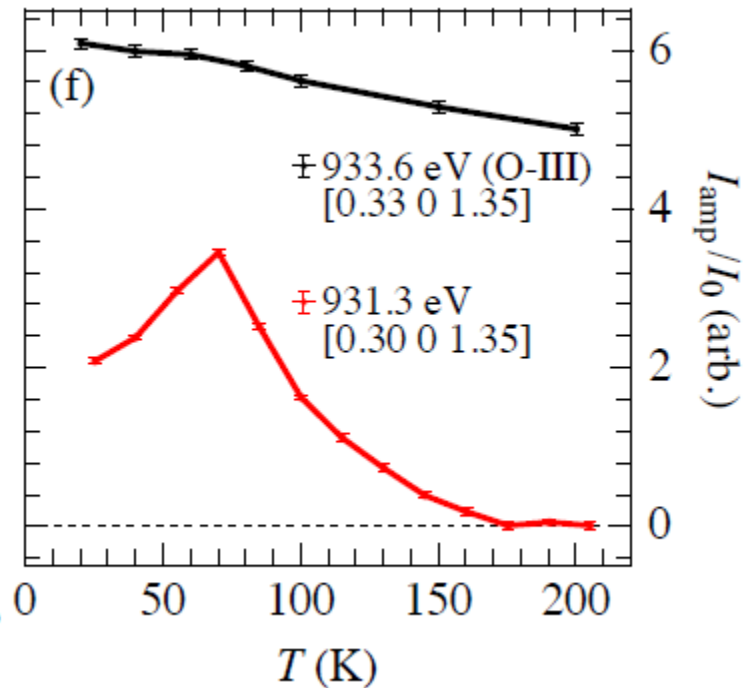
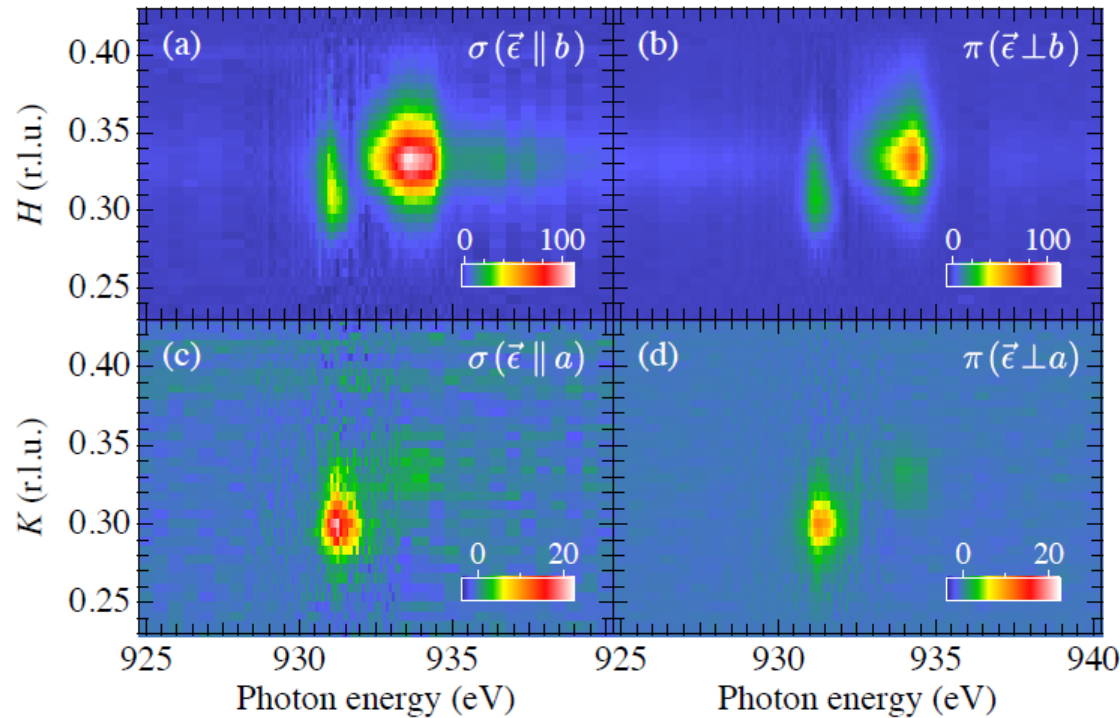
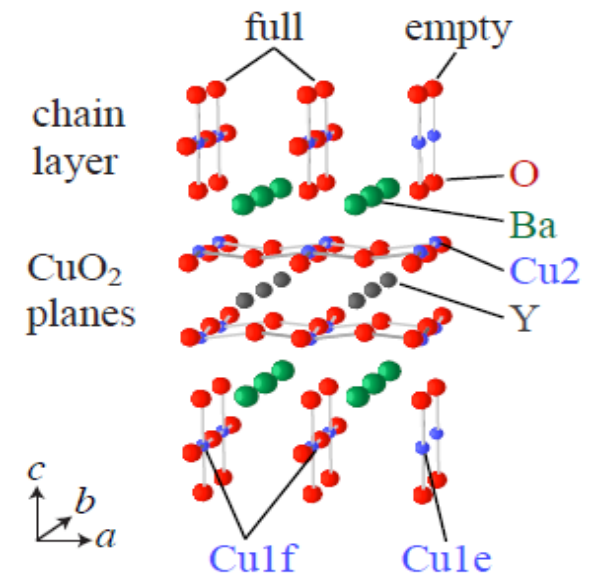
By H. Wadati





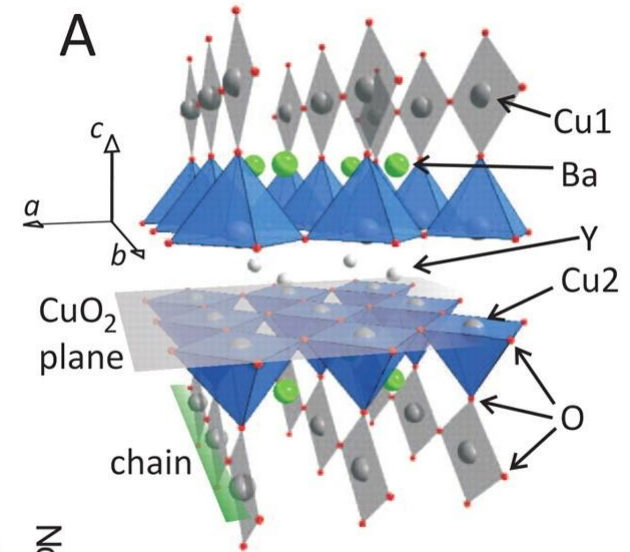
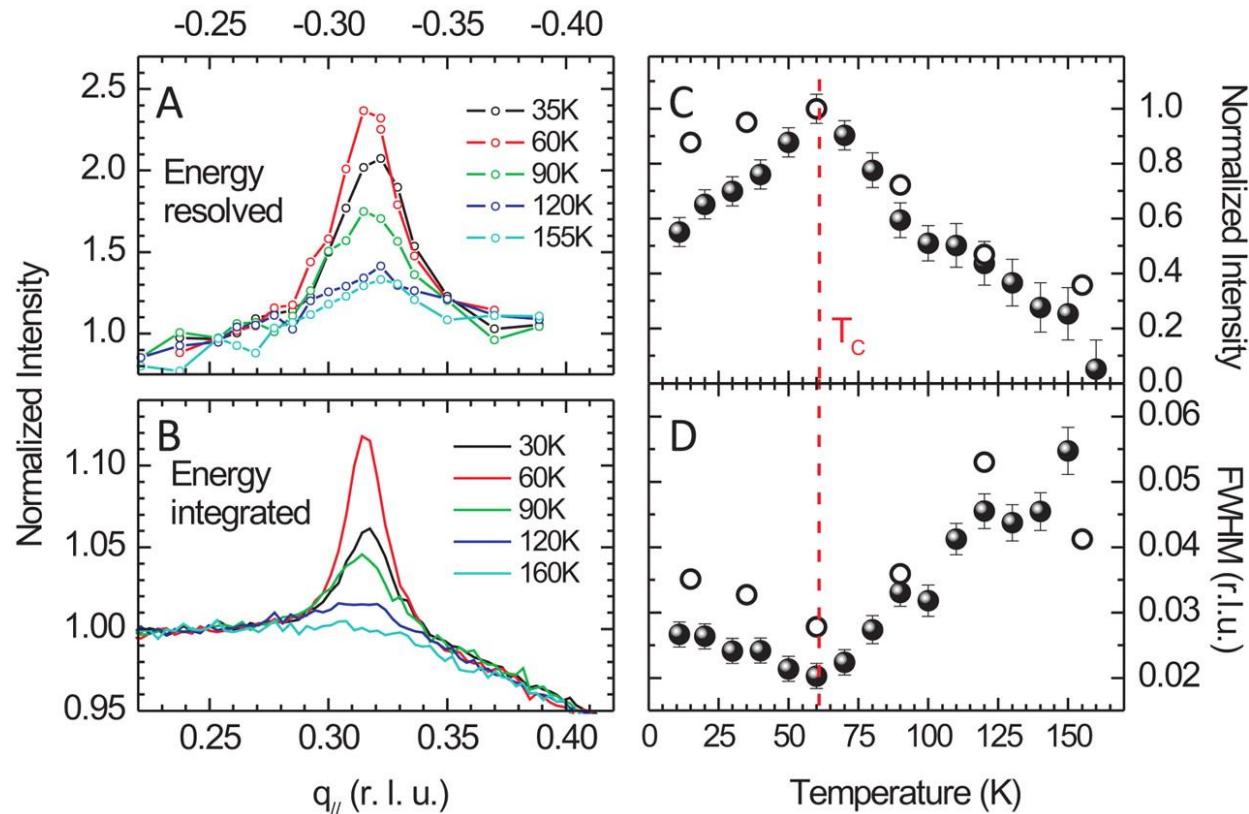
# Charge ordering in chain layer and plane layer in YBCO

A. Achkar et al, PRL (2012)



# Charge density wave in YBCO

G. Ghiringhelli et al, Science (2012).



# CLS Science Highlights



Download or request a copy:

[http://www.lightsource.ca/science/activity\\_reports.php](http://www.lightsource.ca/science/activity_reports.php)



# Research Report 2011

- Imaging Electronic Ripples and Doped Regions in Graphene
- Voltage Control of Surface Magnetization Domains in a Magnetoelectric Antiferromagnet
- X-ray Scattering Study of the Structural Phase Transition in  $\text{La}_{1.8}\text{Sr}_{0.2}\text{Fe}_{0.01}\text{O}_4$
- Temperature-dependent and in-situ Electrochemical XAFS Studies of  $\text{RuO}_2$ /Carbon Nanocomposites
- Nano-scale Chemical Imaging of a Single Sheet of Reduced Graphene Oxide
- Non-statistical Dopant Distributions of  $\text{Ln}^{3+}$ -doped  $\text{NaGdF}_4$  Nanoparticles
- Time-resolved FTIR Microscopy Studies of Electrochemical Reactions



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Thank you!