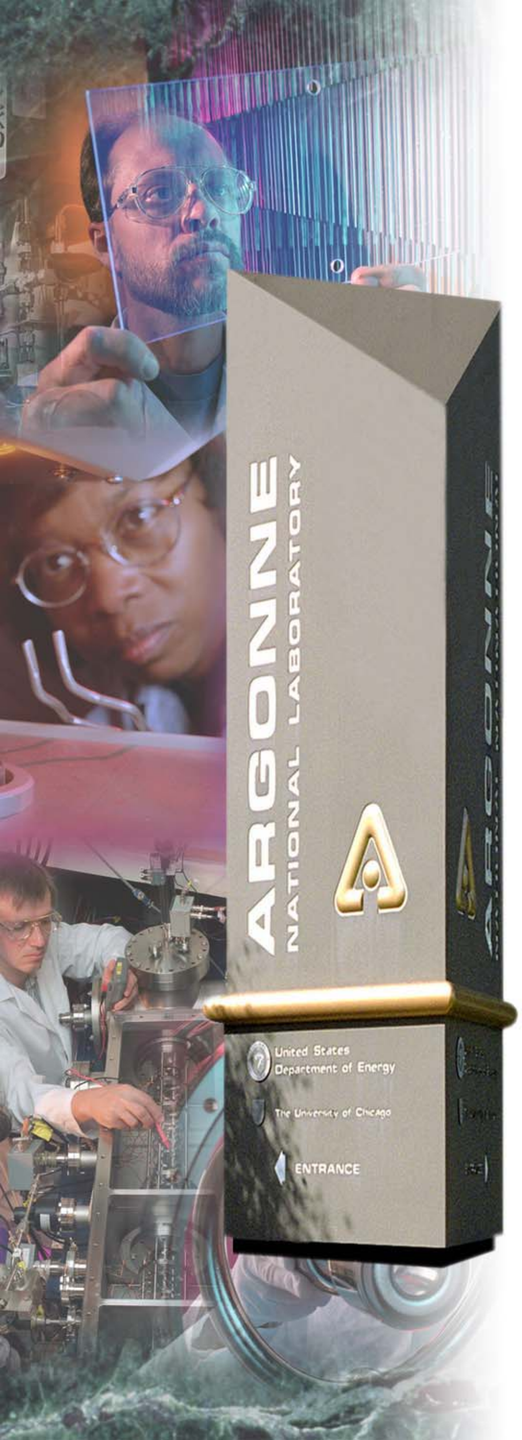


X-ray Beamlines: Operations and Research

Efim Gluskin

The University of Chicago Review
for of the Advanced Photon Source
at Argonne National Laboratory

September 17-19, 2003

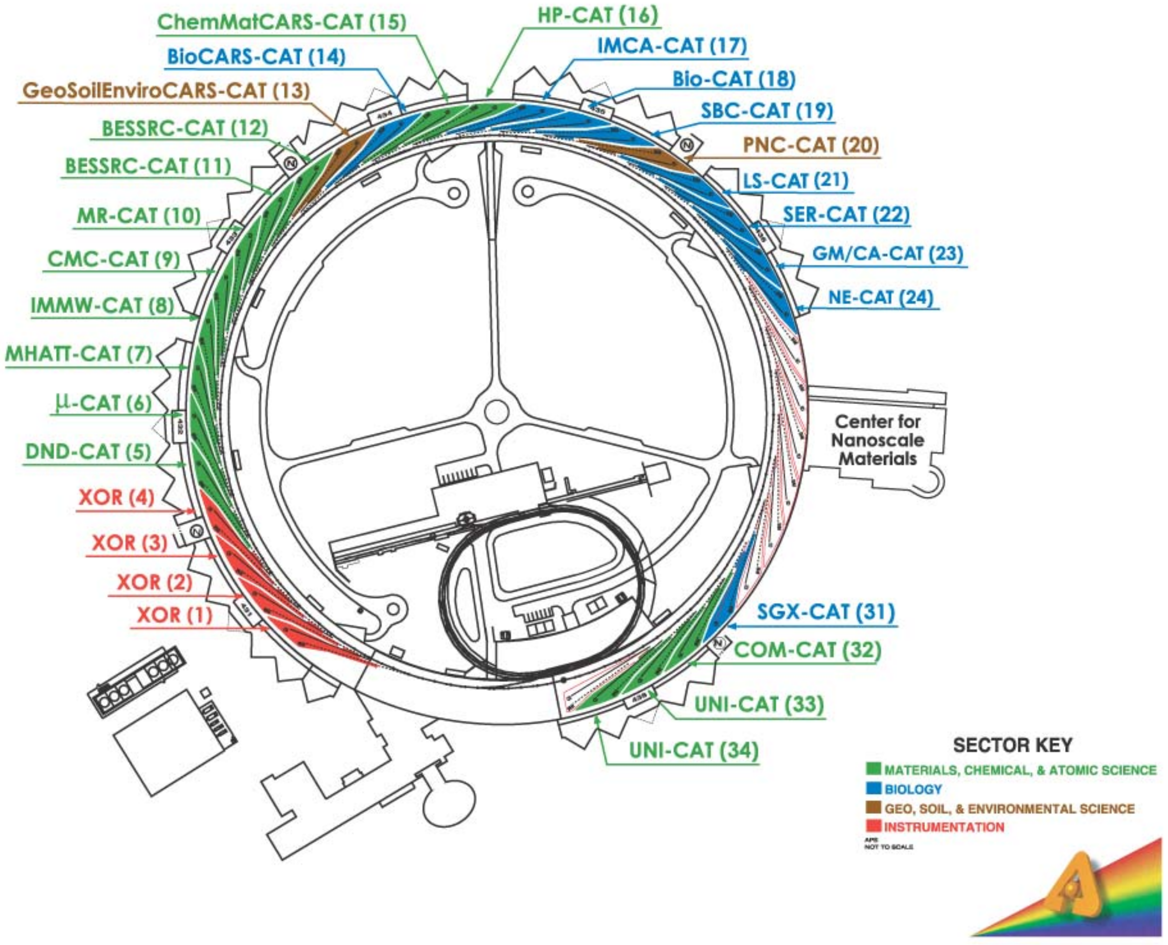
Office of Science
U.S. Department of Energy

A U.S. Department of Energy
Office of Science Laboratory
Operated by The University of Chicago

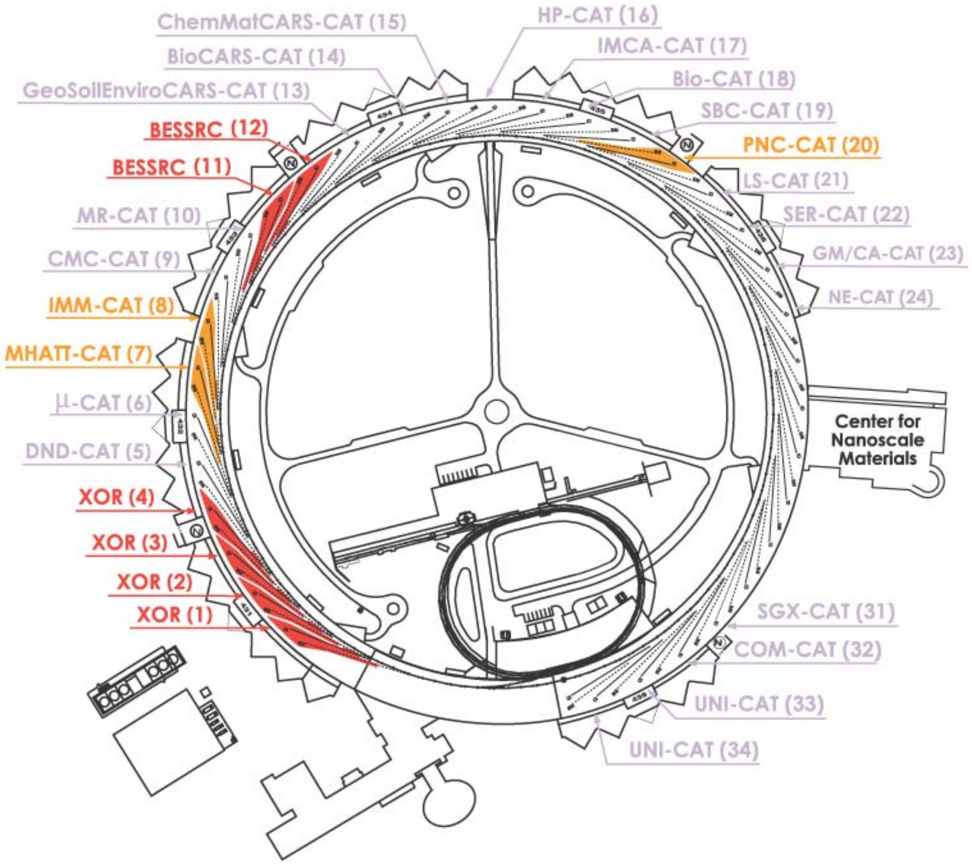
Outline

- Summary of XOR operations
- Research examples

APS Sectors/CATs by Discipline



XOR Sectors

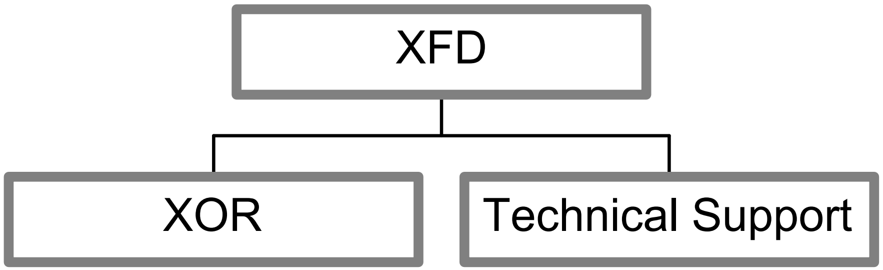


ChemMatCARS-CAT (15)
BioCARS-CAT (14)
GeoSoilEnviroCARS-CAT (13)
BESSRC (12)
BESSRC (11)
MR-CAT (10)
CMC-CAT (9)
IMM-CAT (8)
MHATT-CAT (7)
μ-CAT (6)
DND-CAT (5)
XOR (4)
XOR (3)
XOR (2)
XOR (1)
HP-CAT (16)
IMCA-CAT (17)
Bio-CAT (18)
SBC-CAT (19)
PNC-CAT (20)
LS-CAT (21)
SER-CAT (22)
GM/CA-CAT (23)
NE-CAT (24)
Center for Nanoscale Materials
SGX-CAT (31)
COM-CAT (32)
UNI-CAT (33)
UNI-CAT (34)

New BES Policy for the Support of CATs

- **BES aims to transfer resources and responsibility for operations to facility *and to increase inadequate support.***
- **As APS takes responsibility, more time becomes available for competition.**
- **APS operates sectors 1,2,3,4,11,12 (fully) and *7,8,20* (partially).**
- **New sectors begin as collaborative development teams (CDTs): Nano (26), IXS (30) and Powder Diffraction (11-BM).**
- **Facility explicitly recognizes “partner users.”**

XOR Beamlines and Staffing



X-ray Beamline Operations/Construction and Research

51 Staff

17 operating beamlines

10 Postdocs

3 beamlines under construction

6 Technicians

Technical Support: Engineering, Optics Fabrication, IDs

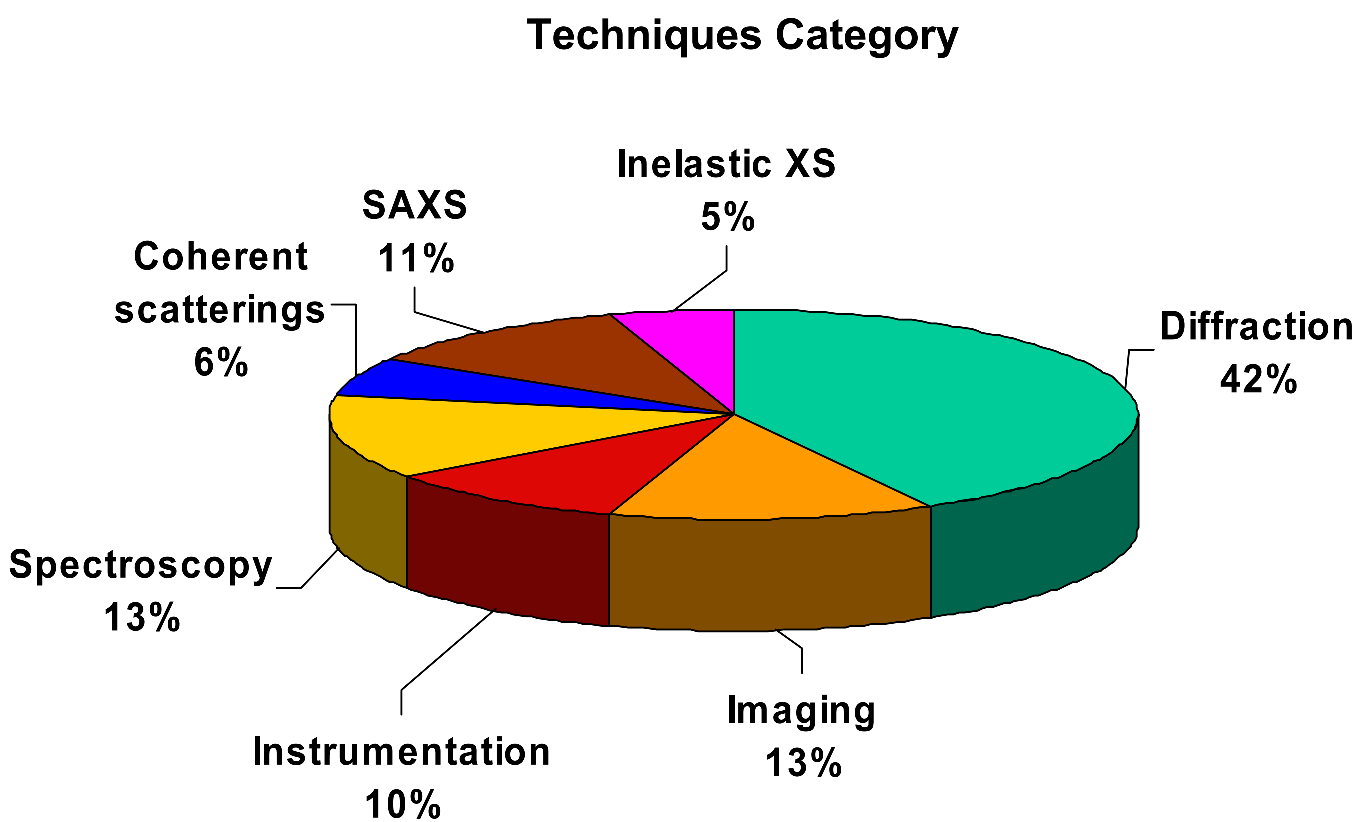
XOR: Beamlines and Staffing

X-RAY OPERATIONS AND RESEARCH

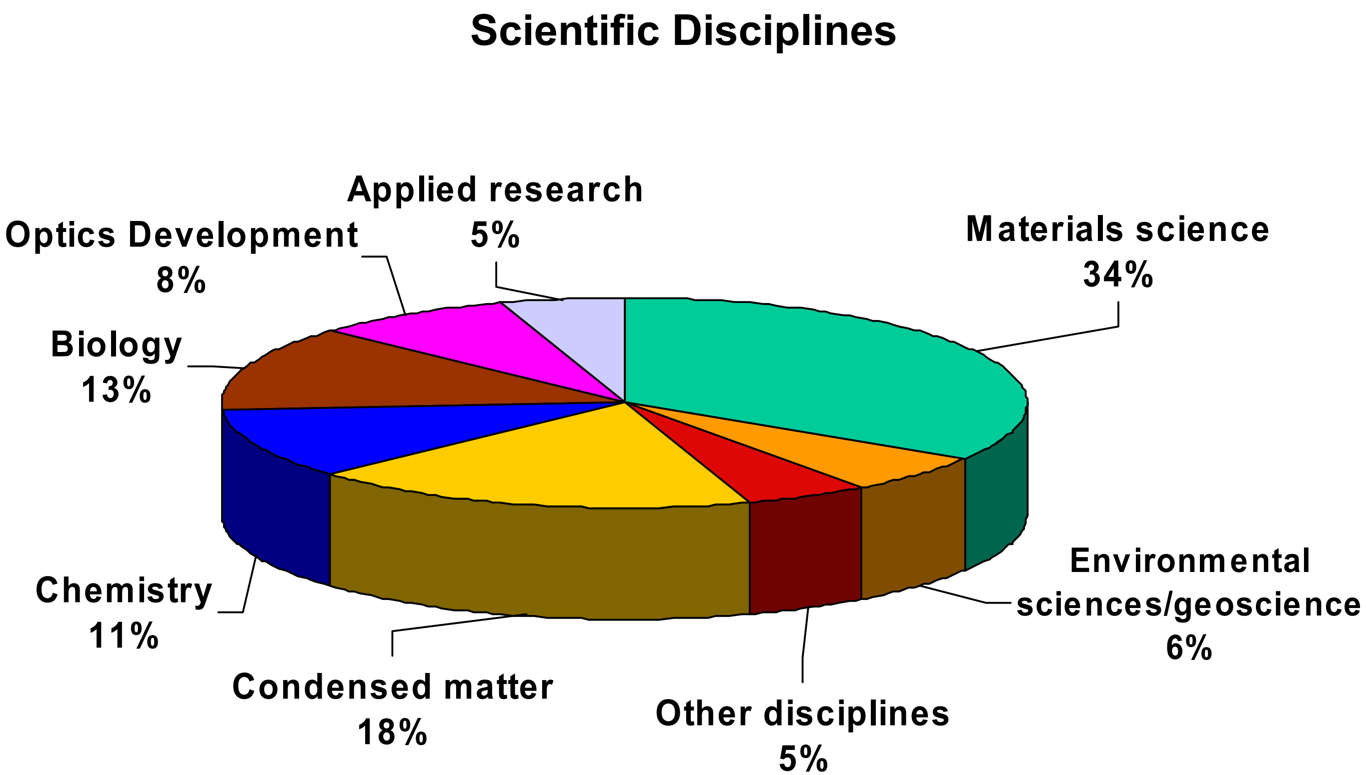
- **HIGH-RESOLUTION X-RAY SCATTERING**
  - 1 operating beamline
  - 1 beamline under construction
  - 5 staff, 1 postdoc, 1 technician
- **POLARIZATION STUDIES***
  - 4 operating beamlines
  - 11 staff, 2 postdocs, 1 technician
- **X-RAY PHYSICS**
  - 2 operating beamlines
  - 1/2 beamline under construction
  - 8 staff, 1 postdoc, 2 technicians
- **X-RAY MICROSCOPY**
  - 3 operating beamlines
  - 1 beamline under construction
  - 10 staff, 3 postdocs, 2 technicians
- **TIME-RESOLVED X-RAY RESEARCH***
  - 3 operating beamlines
  - 8 staff, 1 postdoc
- **BESSRC**
  - 4 operating beamlines
  - 1/2 beamline under construction
  - 9 staff, 2 postdocs

* Operation also is supported by CAT Staff

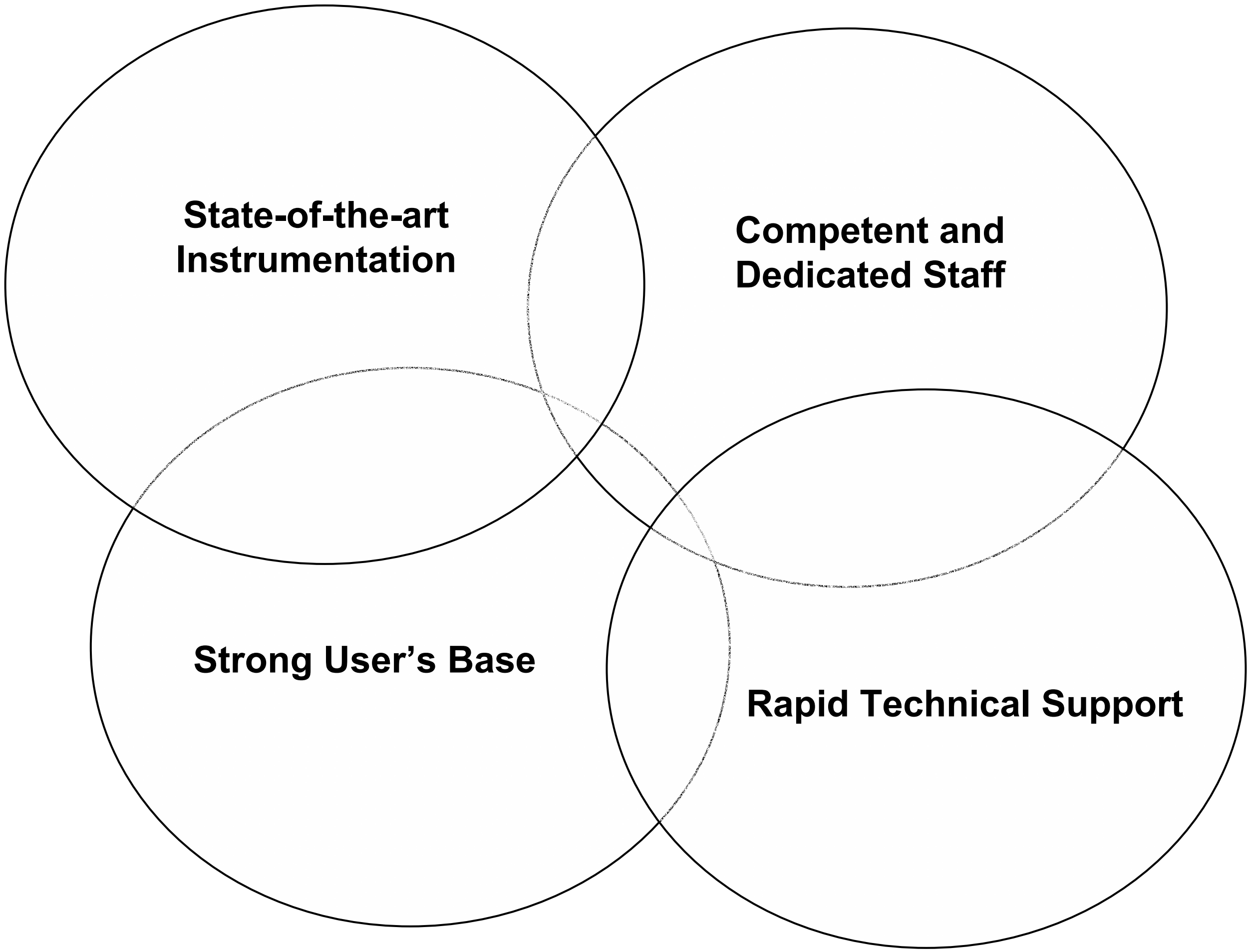
XOR Beam Time Usage



XOR Beam Time Usage



Basis for Scientific Excellence and High Productivity

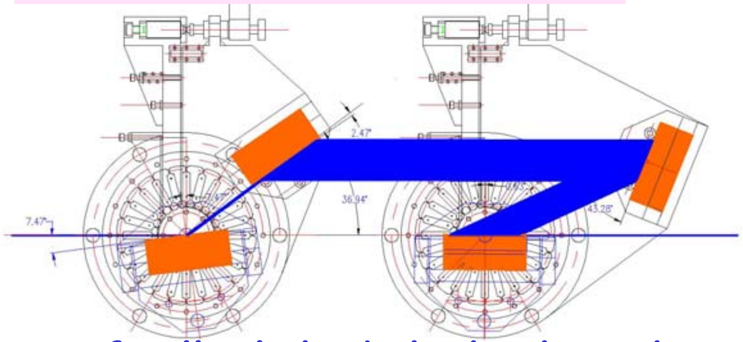


Posters

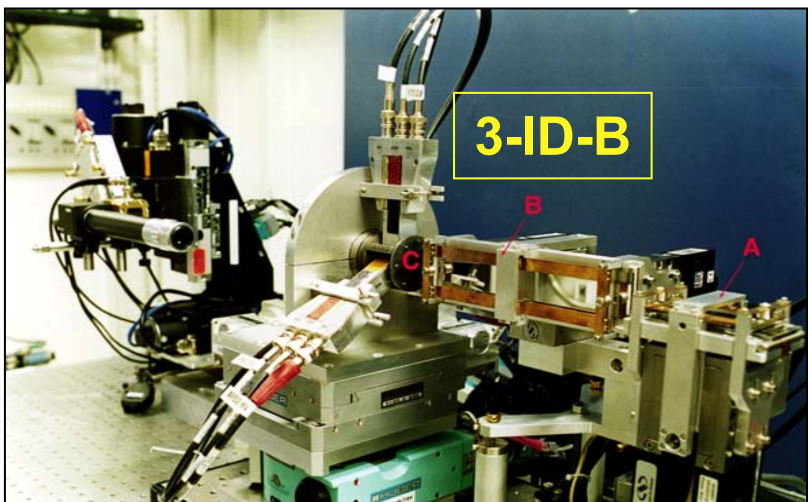
1. Surface nucleation of magnetic twist in artificial Fe/Gd Multilayers
2. Magnetic Domain Imaging in Nanoscale Structures Using X-ray Photoemission Electron Microscopy
3. Nanoscale Modulations in YBCO High T_c Superconductors
4. Dedicated High-Resolution Powder Diffraction Beamline at the Advanced Photon Source
5. Actinide Chemistry at the APS
6. In-Situ Synchrotron X-ray Studies of Metal-Organic Chemical Vapor Deposited PbTiO3 Thin Films
7. Direct Observation of Orthoclase Mineral Dissolution: Mechanisms and Kinetics
8. Probing Molecular Structure of Photoexcited States with 100 ps Time Resolution
9. High-energy SAXS/WAXS: A Versatile Tool for Materials Analysis
10. Phase Enhanced Live-Imaging of Small Insects
11. High Pressure Studies with Nuclear Resonant Inelastic Scattering Studies
12. High Resolution Inelastic X-Ray Scattering in Liquids and Solids
13. High Throughput X-ray Microtomography
14. X-Ray Diffraction Microscopy for Structural Characterization Down to Nanometer Length Scales
15. Biological X-ray Fluorescence Microscopy
16. From Flat Substrate to Elliptical K-B Mirror by Profile Coating
17. Development of a Linear Stitching Interferometric System for Evaluation of Very Large X-ray Synchrotron Radiation Substrates and Mirrors
18. Dual Canted Undulators at the Advanced Photon Source
19. Design and Development of a Robot-Based Automation System for Cryogenic Crystal Sample Mounting at the Advanced Photon Source
20. New High-Heat-Load Front End for Multiple In-Line Undulators at the Advanced Photon Source
21. *In-Situ* High-Energy X-Ray Powder Diffraction Studies in Heterogenous Catalysis: Coupling the Study of Long Range and Local Structural Changes
22. Phase-Contrast Micoscopy

Properties of Core Materials at High Pressure-High Temperature Using Nuclear Resonant Inelastic X-ray Spectroscopy

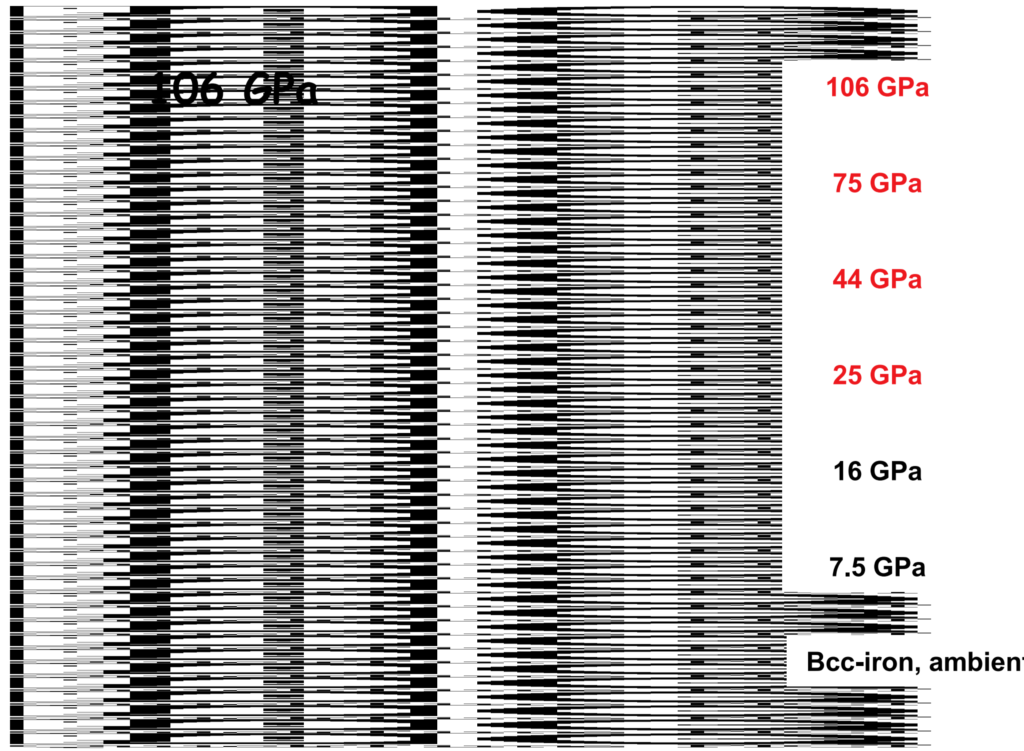
E= 14.4 keV, ΔE= 1.0 meV



Artifically-linked, high-throughput high-resolution monochromator



$Fe_{92.5}Ni_{7.5}$

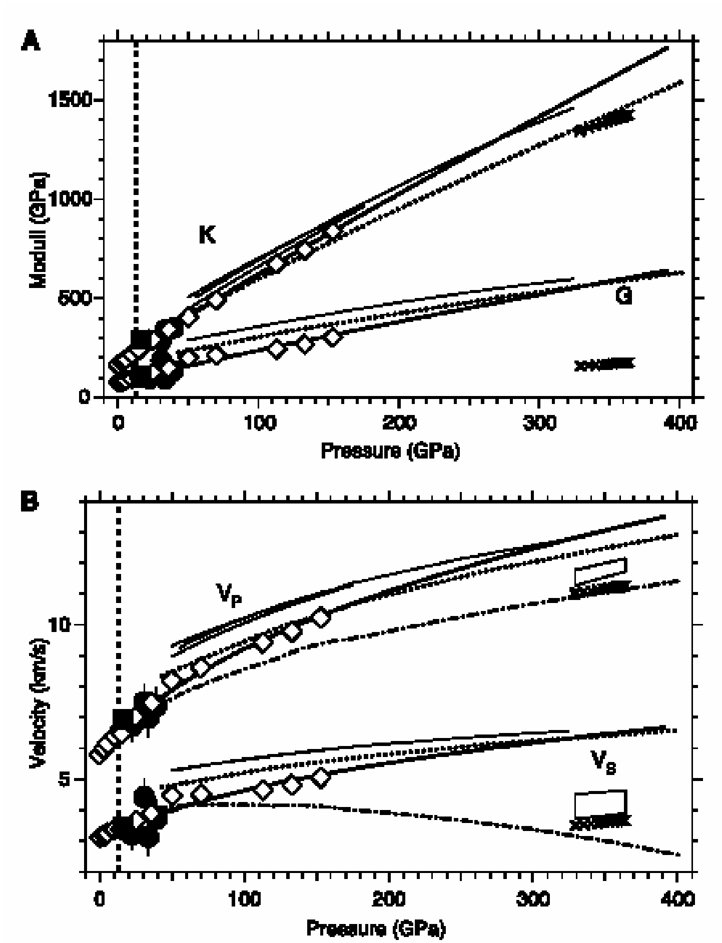


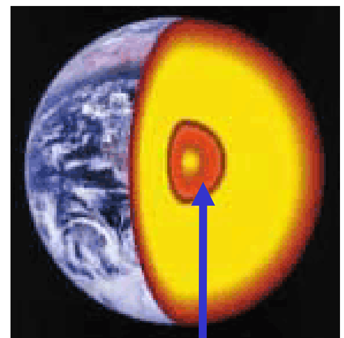
H.K. Mao, V. Sturzhkin (Carnegie Institute of Washington)
W. Sturhahn, J, Zhao, E. Alp (APS)

Properties of Core Materials at High Pressure-High Temperature Using Nuclear Resonant Inelastic X-ray Spectroscopy

ΔE = 1 meV, T > 2000 K, P > 1.5 Mbar, ϕ ~ 5μm

Unique combination where 1 meV energy resolution, microfocusing, high pressure and high temperature are combined for geophysics interests.

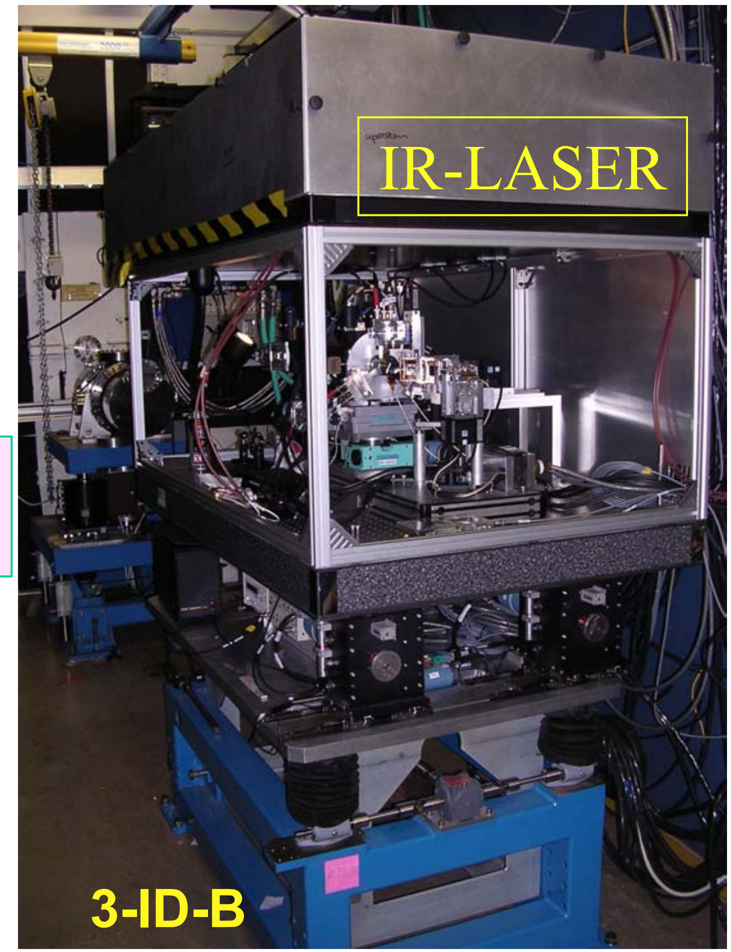




364 GPa
~ 6000 K

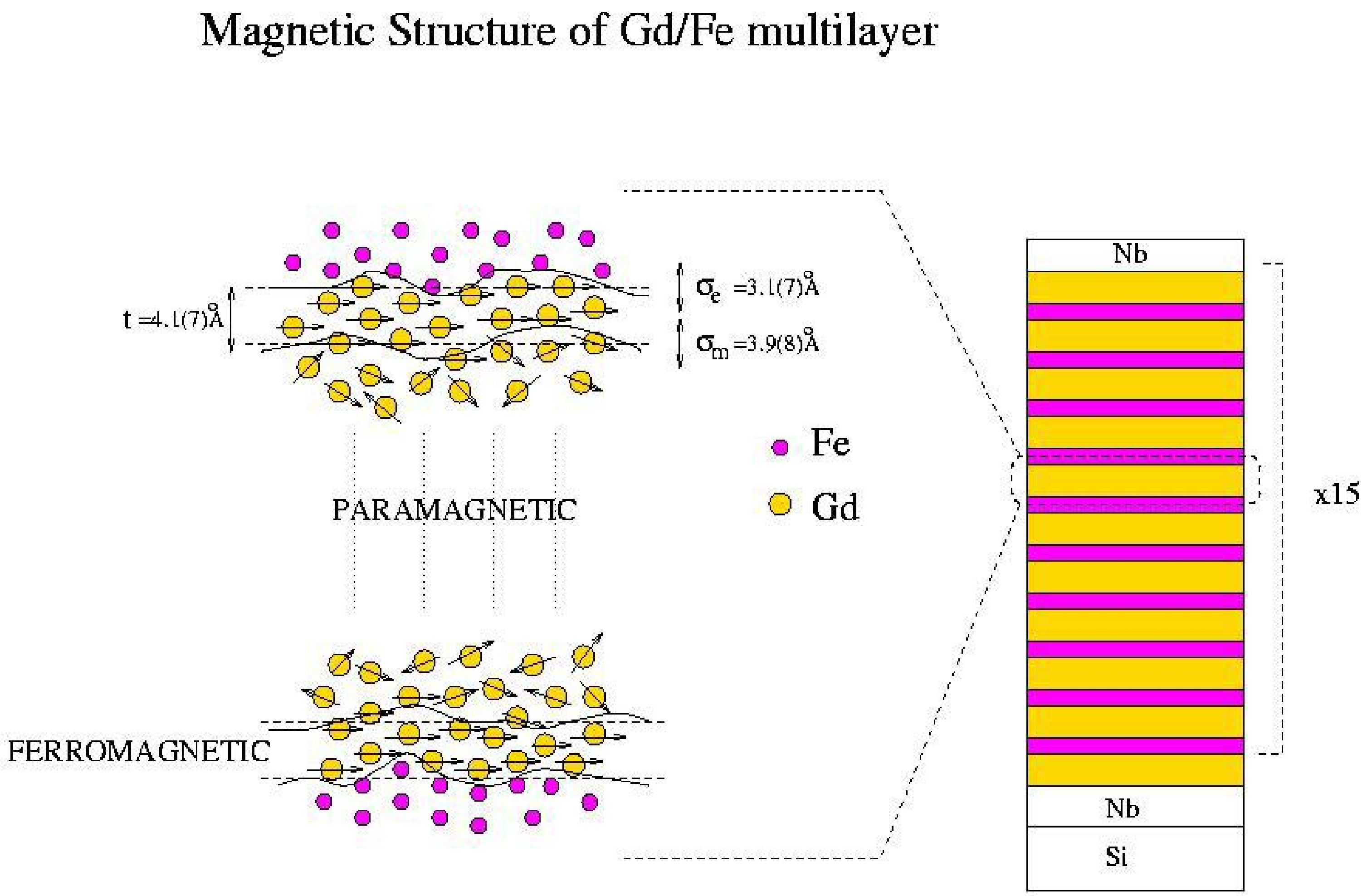
Mao et al., Science **292** (2001), 914

Struzkhin, PRL **87** (2001) 255501

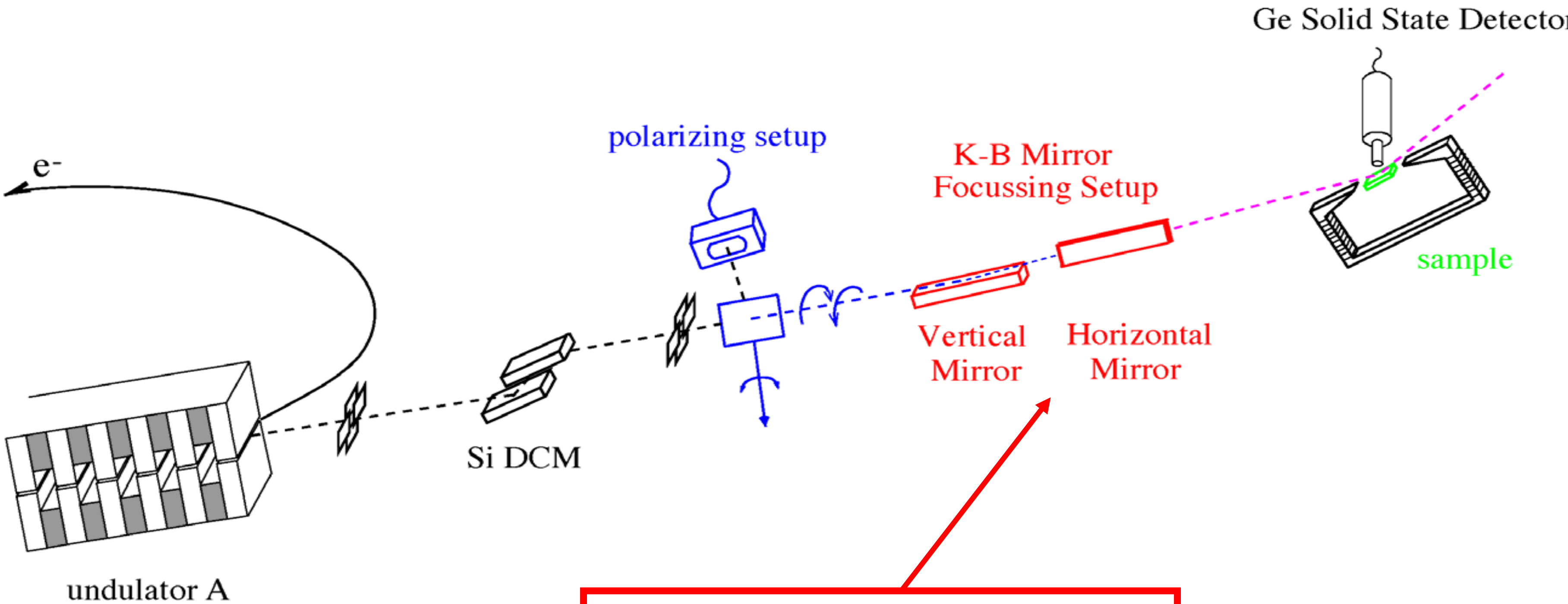


Enhanced Interfacial Magnetic Coupling of Gd/Fe Interface

D. Haskel et. al., Phys. Rev. Lett **87,** 207201 (2001)



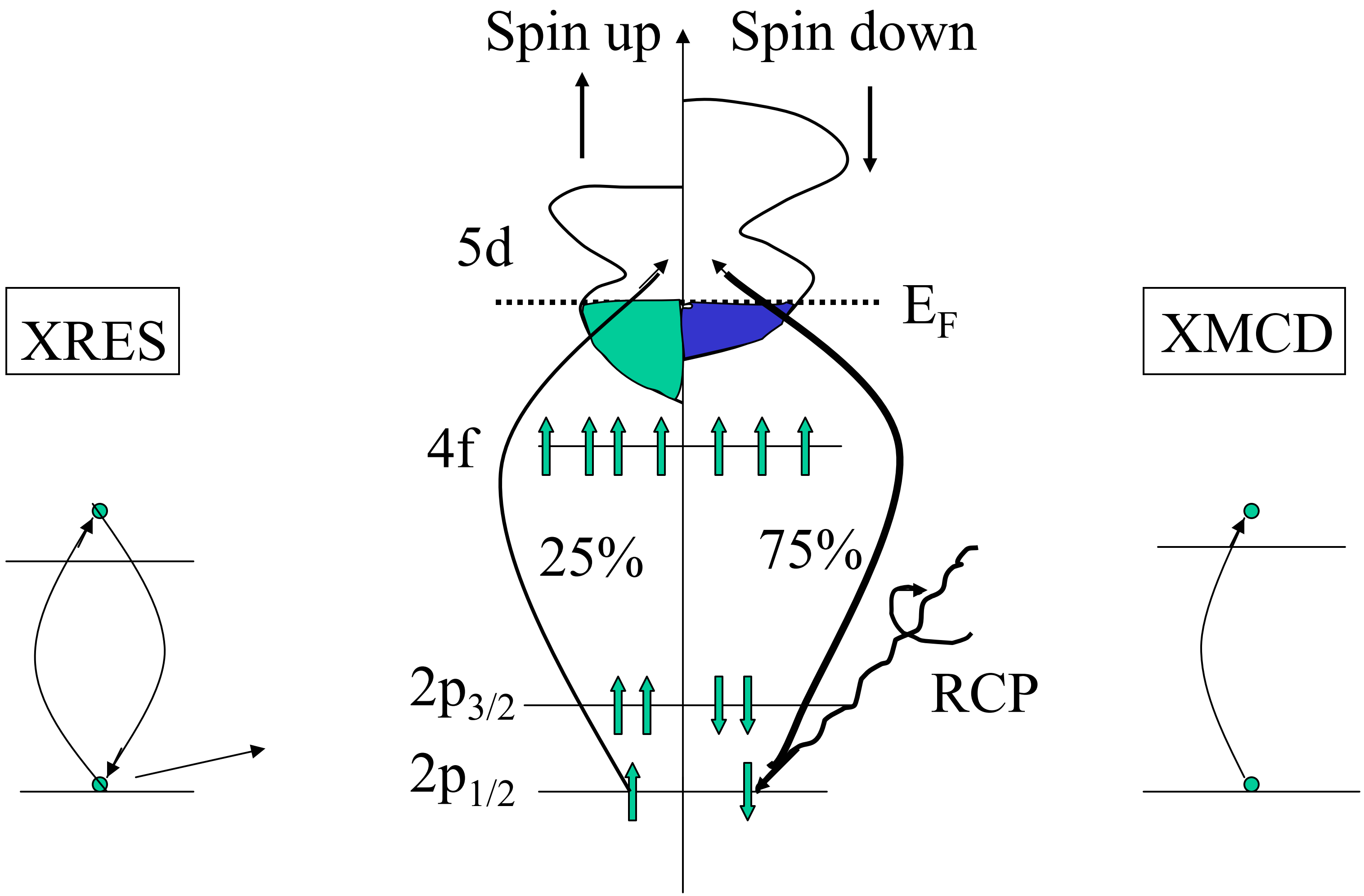
Experimental Setup



K-B Mirror Properties:

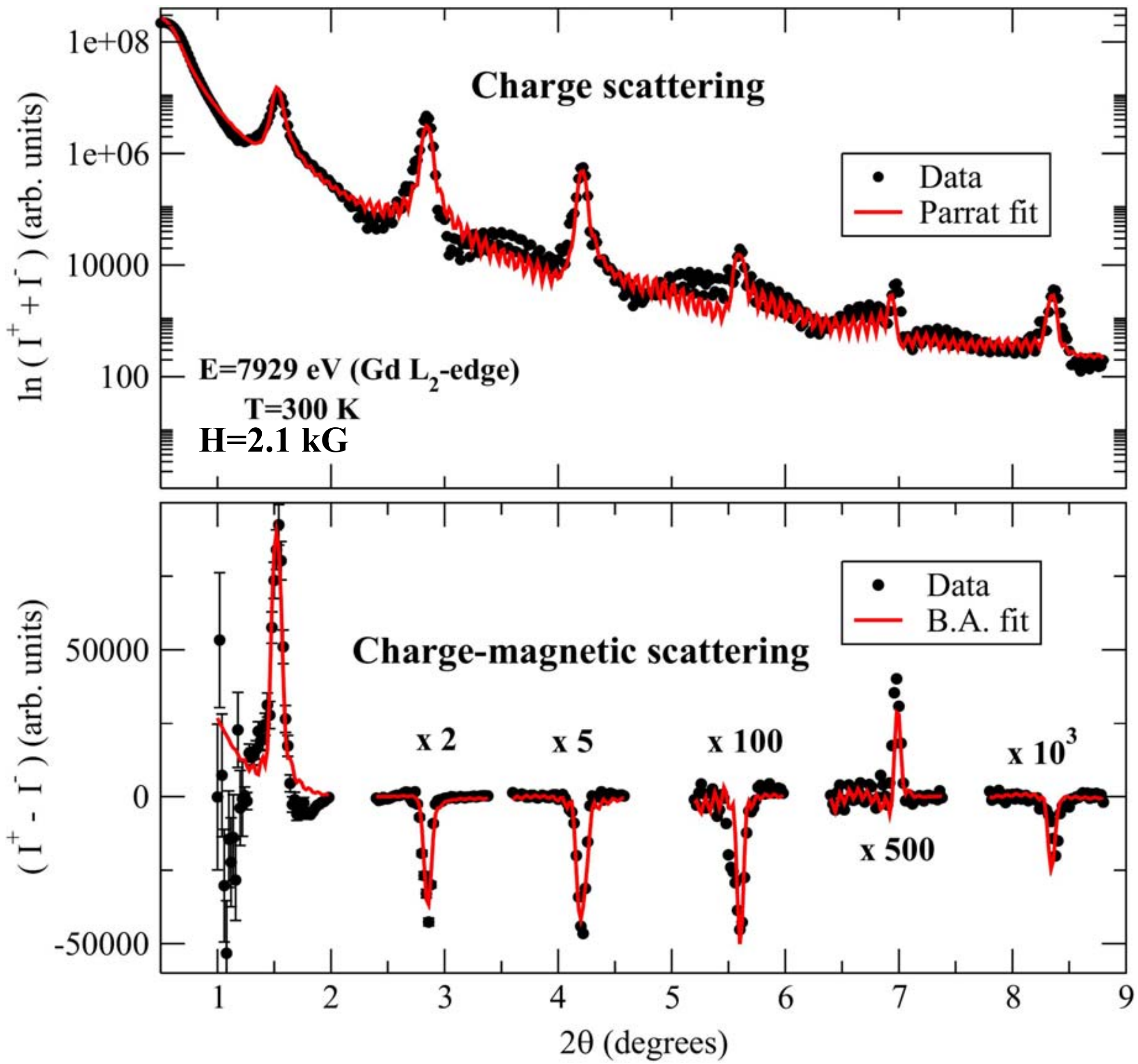
focal length	~30 cm
focal size	$1.0 \times 1.0\ \mu m^2$
flux	~ 10^{10} ph/s (100mA)
P_c	~ 0.99

Magnetic Sensitivity Using Circularly Polarized X-rays



Needs net magnetization ➡ Ferro/Ferri-magnets

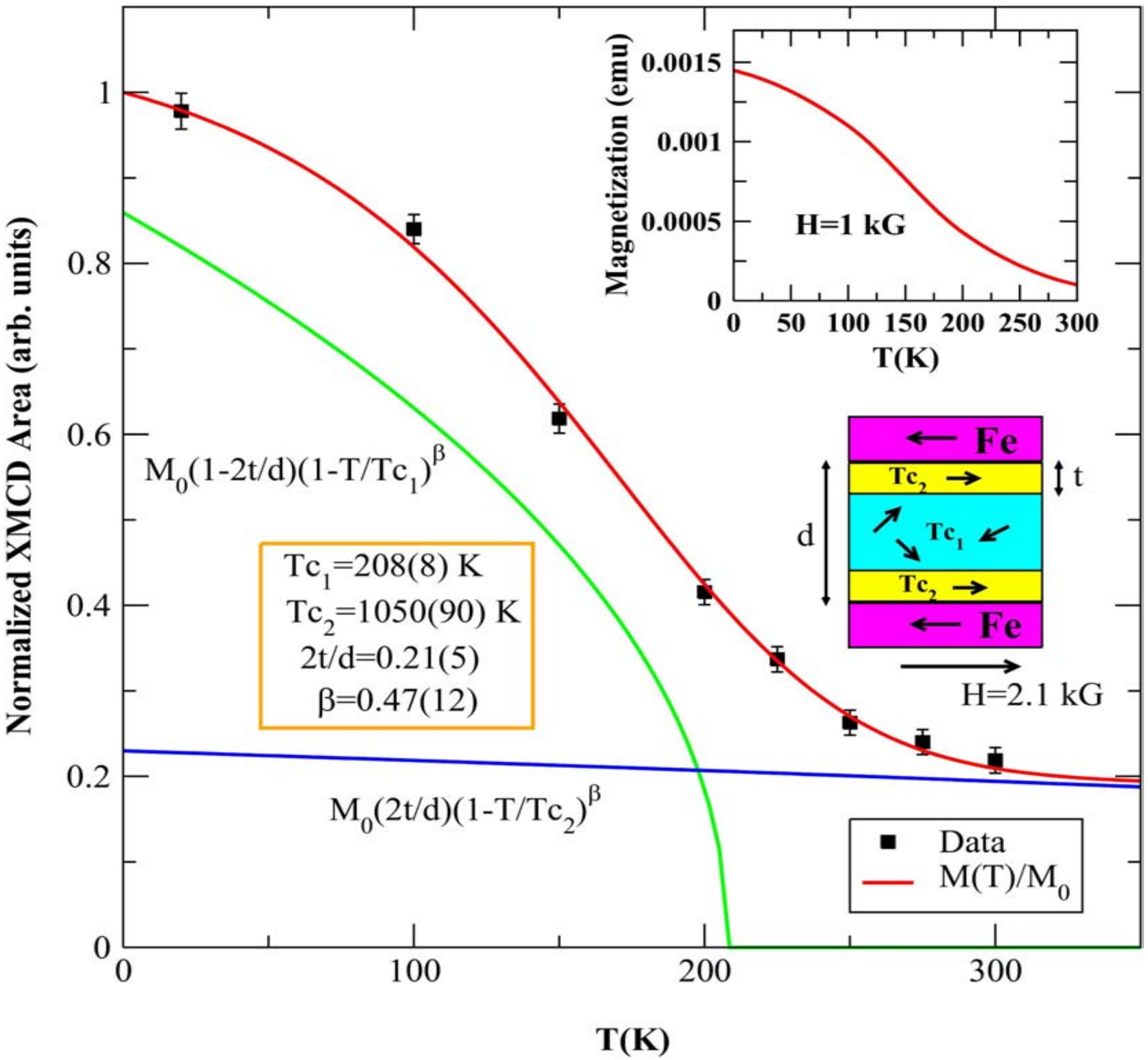
Fit Results to Charge and Charge-Magnetic Specular Scattering



$d\sigma/d\Omega \sim (e_f^{*}.e_i)^2 \, r_0^2 \, |Z+f_e|^2$

$d\sigma/d\Omega \sim \{i[(e_f^*.e_i)^* \, (e_f^* \times e_i).m] \, (Z+f_e)^*.f_m\} + c.c.$

XMCD at the Gd L2-Edge (7.929 keV)



Integrated intensity
Gd magnetization

Inset shows SQUID data

Agreement between bulk and microscopic XMCD

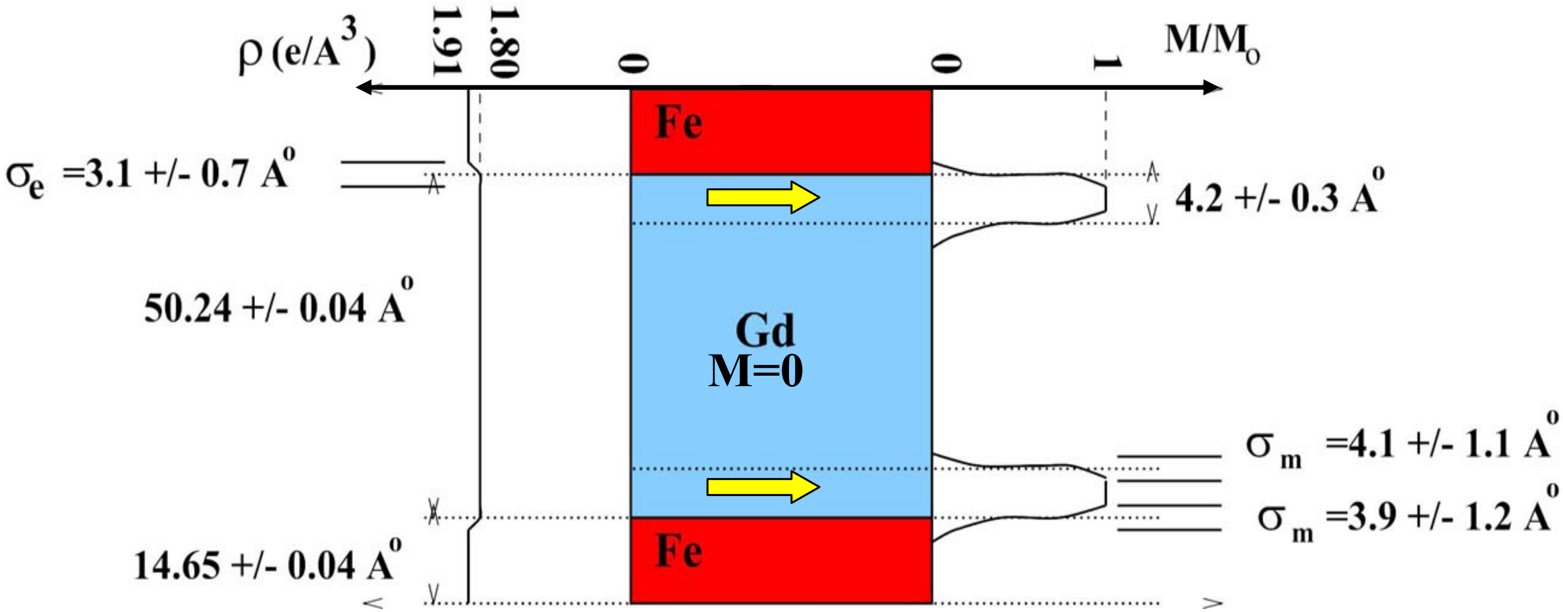
"Yellow" Gd sublayer is ordered above T_c

T_c = 1050 K

Thickness ***t*** of Gd sublayer

***t* ~ 4 Å**

Magnetic Structure of [Fe(15Å)Gd(50Å)]15 Multilayer at T=300K



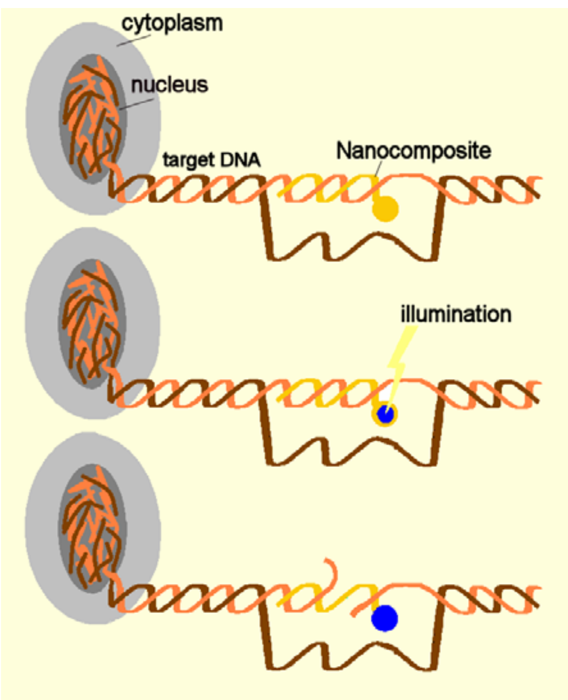
- Fit to charge-magnetic reflectivity in agreement with (4.2 ± 0.3 Å)with XMCD data
- Quantitative measure of spatial extent of Fe/Gd exchange interaction

Mapping TiO_2 Nanocomposites in Mammalian Cells

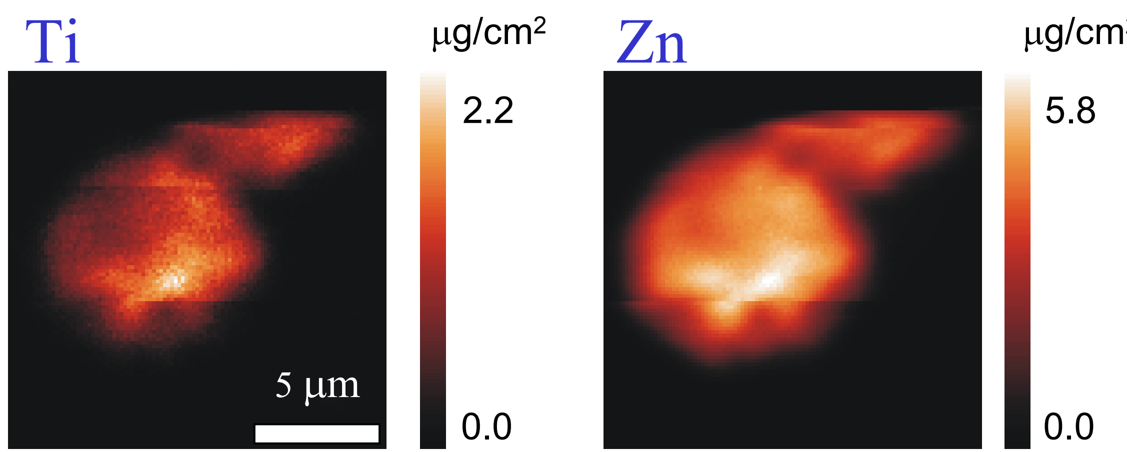
- Cell is transfected with TiO_2-DNA nanocomposites
- DNA targets specific chromosomal region
- TiO_2 photocleaves DNA strands upon illumination
- Potential use in intracellular manipulation,gene therapy

Map Ti distribution using x-ray induced K_α fluorescence to quantify success rate of TiO_2-DNA transfection.

Affinity of transfected DNA to ribosomal DNA causes nanocomposites to localize at the nucleolus.



Nuclei isolated from cells transfected with R18Ss- TiO_2 nanocomposite and a "free" R18Sas oligonucleotide

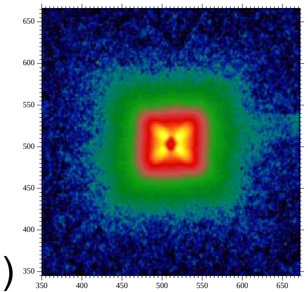


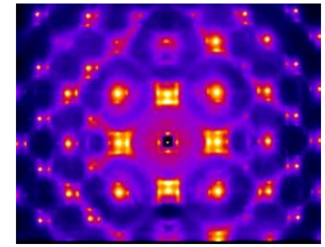
T. Paunesku et. al., *Nature Materials* **2**, 343 (2003)

1-ID

High-Energy X-ray Scattering

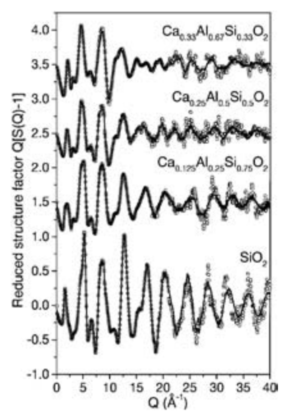
- Optics Development
- Stress/Strain/Texture
- Powder Diffraction
- Small-Angle Scattering
- Diffuse Scattering
- Amorphous Scattering (PDF)
- Trace Element Analysis
- Imaging
- Miscellaneous
  - *Archaelogy*

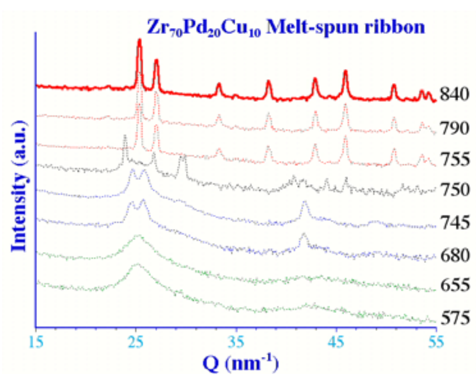


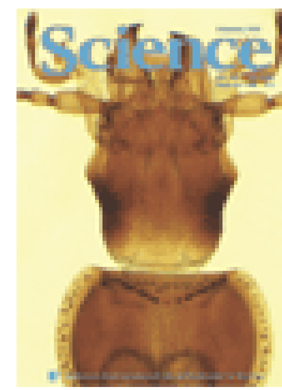


Optics & Imaging

- Interferometry
- Phase Contrast Imaging
- HHL Optics
- Si-Ge Graded Crystals







Beamline Optics

High-Energy Mono

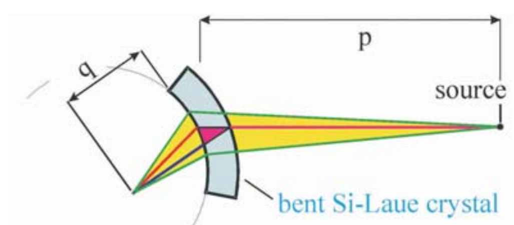
- 50-120 keV
- LN_2 Cooled

Low-Energy Mono

- Kohzu LN_2 Cooled
- 10-40 keV (Si (111))

No Mirrors

White beam capable in 1-ID-B

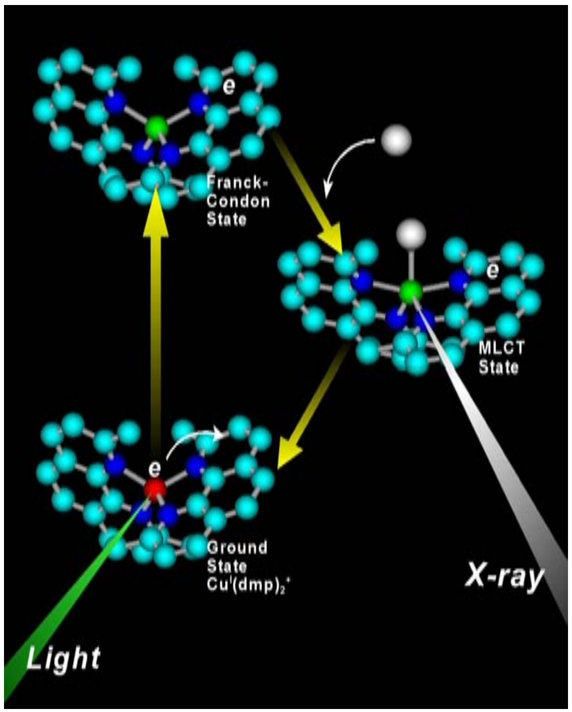


Probing Molecular Structures of Photoexcited States with 100-ps Time Resolution

Lin X. Chen*, George B. Shaw, David J. Gosztola, Tao Liu, Jan P. Hessler ***Chemistry Division, ANL***

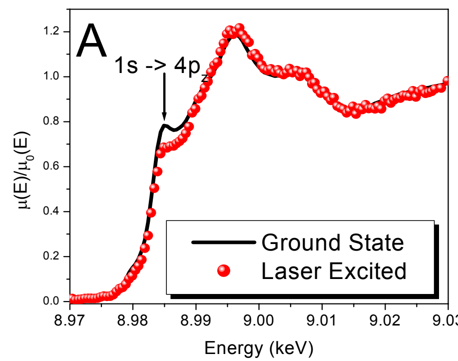
Guy Jennings, Klaus Attenkofer Experimental Facilities Division***, APS, ANL***

One of the important excited states in metal complexes is generated via photoinduced metal-to-ligand-charge-transfer (MLCT) where the electron density from the metal is shifted to ligands, changing the oxidation state of metal and molecular structure, which can be detected via pump-probe XAFS in solution.

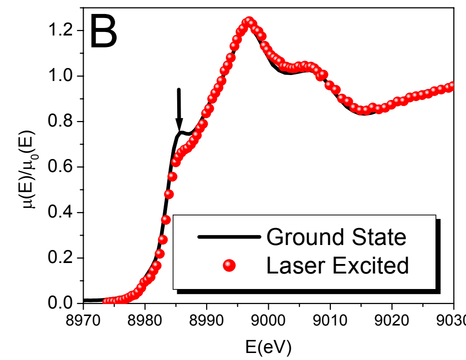


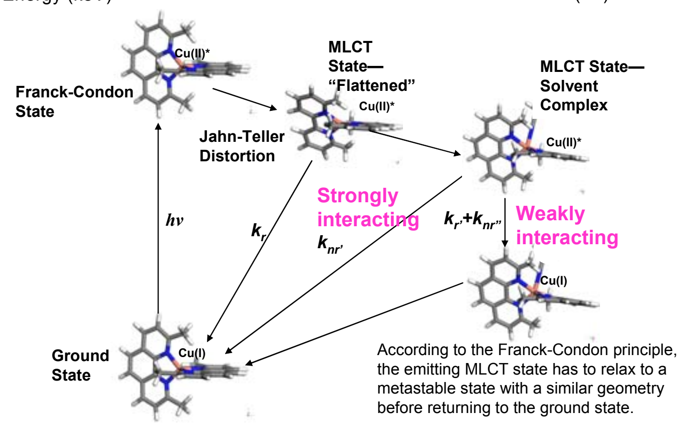
The MLCT excited state of [Cu(I)(dmp)^2]+ (dmp = 2,9-dimethyl-1,10-phenanthroline) plays important roles in photoinduced electron and energy transfer reactions.

In acetonitrile: 1.7 ns lifetime
Strongly interacting with solvent, "exciplex".



In toluene: 100 ns lifetime
Weakly interacting with solvent, luminescent.

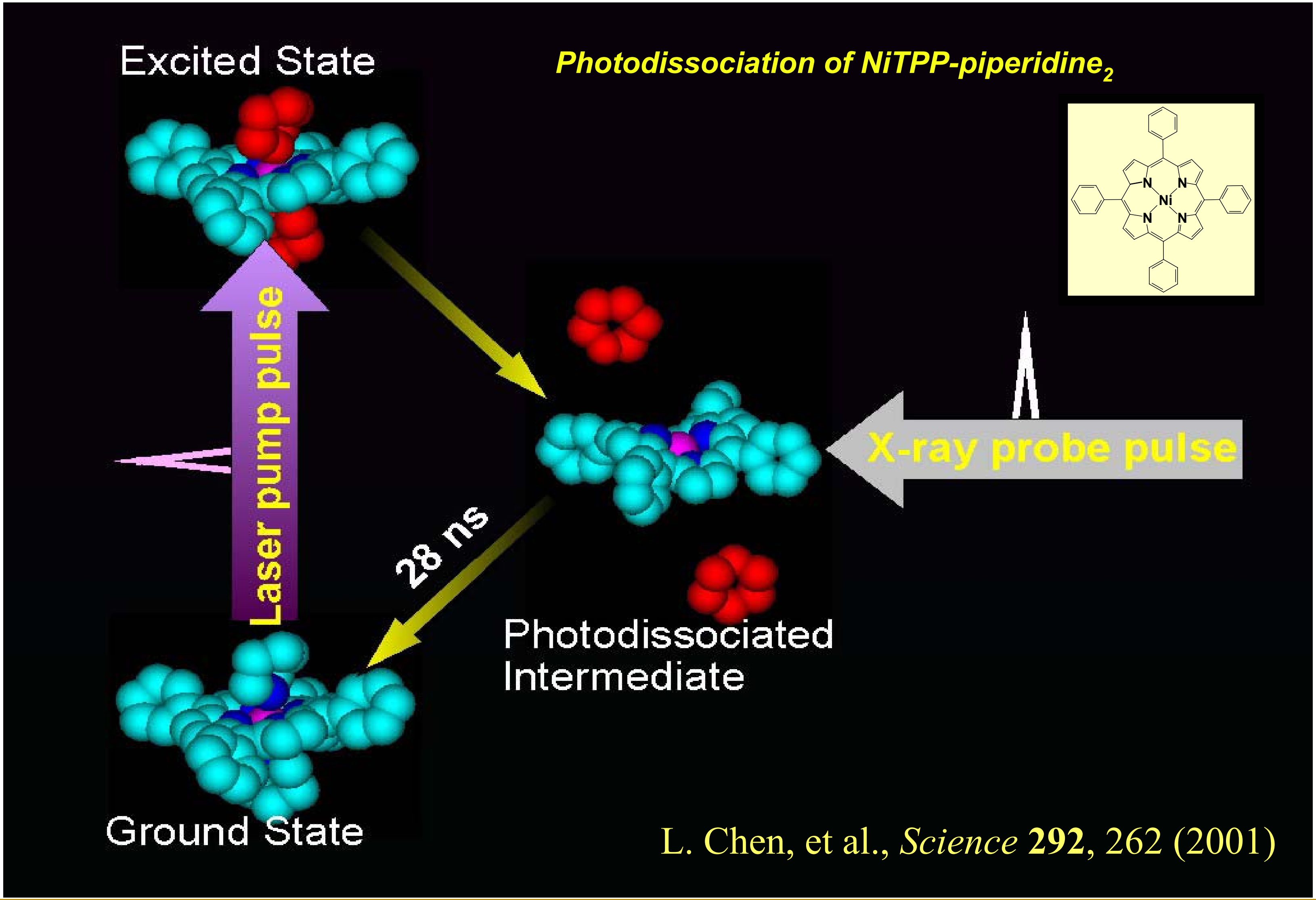




Revised mechanism for photoinduced MLCT excitation of $Cu(dmp)_2^+$ in solution based on pump-probe XAFS results.

Pump-probe XAFS measurements in time domain provide new insights into the fundamental aspects in the structural factors that may influence photoinduced electron and energy transfer processes.

L. X. Chen, G. B. Shaw, I. Novozhilova, T. Liu, G. Jennings, K. Attenkofer, G. J. Meyer, P. Coppens, J. Am. Chem. Soc. 125 7022-7034 (2003).
L. X. Chen, G. Jennings, T. Liu, D. J. Gozstola, J. P. Hessler, D. V. Scaltrito, G. J. Meyer, J. Am. Chem. Soc. 124, 10861-10867 (2002).


Excited State
Photodissociation of NiTPP-piperidine$_2$
N
N
Ni
N
N
Laser pump pulse
X-ray probe pulse
28 ns
Photodissociated
Intermediate
Ground State
L. Chen, et al., *Science* **292**, 262 (2001)

Summary - XOR Future

- Increase level of responsibilities for BES-funded beamlines.
- Move toward specialized beamlines.
- Reach 80/20 split for all BES-funded beamlines.
- Continue to build and preserve strong user community coupled with XOR.