Argonne NATIONAL LABORATORY

... for a brighter future

U.S. Department of Energy

UChicago ▶ Argonne LLC

Office of Science U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by UChicago Argonne, LLC

# APS Renewal Instrumentation Open Forum January 9th, 2009

# Proposed Upgrades to X-ray Spectroscopy Capabilities

## Inelastic Scattering
## Nuclear Resonant Scattering


Steve Heald

Thomas Gog

Ercan Alp

## *X-ray spectroscopy methods well suited to the themes of the upgrade*

- Long history of application to real systems under real conditions
  - *Disordered materials, dilute species*
  - *Catalysts, batteries, biological systems, environmental science…*
- Brilliance of APS allows sub-micron resolution in conjunction with imaging
  - *Most existing and proposed microprobes have XAS capabilities*
- Fill patterns at the APS are especially favorable for time dependent studies
  - *Currently time dependent XAS carried out on time scales of sub-nsec (pump-probe) to seconds (quick XAFS)*
  - *Even better resolution with crab cavities*

## *Many proposals have a spectroscopy component*

- Micro/Nano XAFS
  - Bio-nanoprobe; 2-ID (2 out of three stations); 13-ID; 16-ID; 18-ID; 20-ID
- Time-resolved XAFS
  - 7-ID; 11-ID-D; 20-ID-C
- XMCD
  - 4-ID-C; 4-ID-D; New ID line
- Bulk XAFS (BM lines)
  - 9-BM; 5-BM-D

# *Microprobes*

- Zone plate based
  - *2-ID: Independent RT and Cryo-stations to 50 nm*
  - *Bio-nanoprobe: Cryo-station to 20 nm*

- K-B mirror based
  - *13-ID: dedicated canted-undulator microprobe*
  - *20-ID: dedicated canted-undulator microprobe*
  - *16-ID: sub-micron beam for H-P research*
  - *18-ID: new undulator for 2x flux – other upgrades funded separately*

## *Time resolved spectroscopy*

**These should all be coordinated with the potential crab cavity upgrade**

- 7-ID: Major rebuild of beamline including better focusing and high resolution emission spectroscopy
- 11-ID-D: Upgrade laser, optics, ID and detectors for both spectroscopy and WAXS
- 20-ID-C: Detector and focusing upgrade as part of canted undulator expansion

# XMCD

## 3 related proposals

- Sector 4 proposing major upgrades to both branch lines to build upon a successful program
  - *4-ID-C: new optics, detectors, endstations, and PEEM for soft x-ray line*
  - *4-ID-D: new ID, microbeams, detectors, and endstations for hard x -ray line*

- New sector for optimized soft x-ray XMCD
  - *Less constraints and dual undulators allows even more improvement over 4-ID-C*

## *Bulk XAFS*

- Two proposals to improve XAS capabilities for BM lines
  - *9-BM: upgrade mono and instrument 9-BM-C with focus on catalyst research (partially funded through LDRD)*
  - *5-BM-D: improve operational efficiency with new mono – part of a larger upgrade of sector*

- At workshop it was suggested that dispersive XAFS line should be considered
  - *On ID sub-millisecond time resolution*
  - *Small beams good for in-situ cells*
  - *Highly stable setup*
  - *Is there enough demand for these special characteristics?*

Upgrade presents an opportunity to enhance the user experience for all of the bulk XAFS lines through a coordinated effort to improve sample environments, detectors and software

## *Enabling technologies for spectroscopy*

### Detectors, Detectors, Detectors

- All of the spectroscopy efforts would benefit from better detectors
- Large numbers of Si drift detectors would satisfy many efforts
  - *Miniaturization and more efficient packing of existing detectors*
  - *Improvement of PAD detectors to include spectroscopic capabilities*

Upgrade funding gives the opportunity to partner with other labs or industry in developing the next generation of detectors

# *Inelastic Scattering Instrumentation*

Associated Beamline Proposals:

| | |
|---|---|
| 3-ID | μeV-resolution X-ray Spectroscopy |
| 9-ID | Medium-Energy-Resolution Inelastic Scattering |
| 16-ID | Directing and Controlling Matter under Extreme Pressures and Temperatures |
| 20-ID | LERIX |
| 30-ID | Midterm Upgrade Proposal |
| HPSynC | Frontier Science by Adding Pressure as a new Dimension |

## *IXS Science*

Inelastic Hard X-ray Scattering

**Discovery, Development, Understanding, Control of**

**Real Materials**

**Materials Science** (Complex Transition Metal Oxides,
High-Tc Superconductivity, Giant Magnetoresistance, …)
**Fundamental Physics research** (Earth, Atmospheric, Environment, …)
**Biophysics** (Structure, Dynamics, Functionality of Metalloproteins, Membranes, …)
**Geophysics** (Earth and Planetary Matter under ultra-high pressure, …)

## *Inelastic Scattering*

**electronic excitations** (RIXS, NIXS, x-ray Raman, x-ray emission spectroscopy)

medium energy resolution 20 meV … 1 eV

9-ID RIXS, 20-ID “LERIX”, 30-ID “MERIXS”

IXS

**vibrational excitations**

high energy resolution 0.5 meV … 5 meV

3-ID, 30-ID “HERIX”

## *IXS Renewal Pursuits, Level 1*
## *Infra-Structure Improvements to existing Facilities*

- **X-ray Source, optimize incident photon flux**
  - Longer straight sections with double, triple undulators
  - Specialized insertion devices
  - Increased ring current

- **Incident beam (micro-) focusing**
  - **Accommodate small, inhomogeneous samples**
  - **Accommodate "extreme" sample environments**
    **(**diamond-anvil cells, cryostats, magnets, …)
  - **Realize energy resolution made possible with strip detectors**

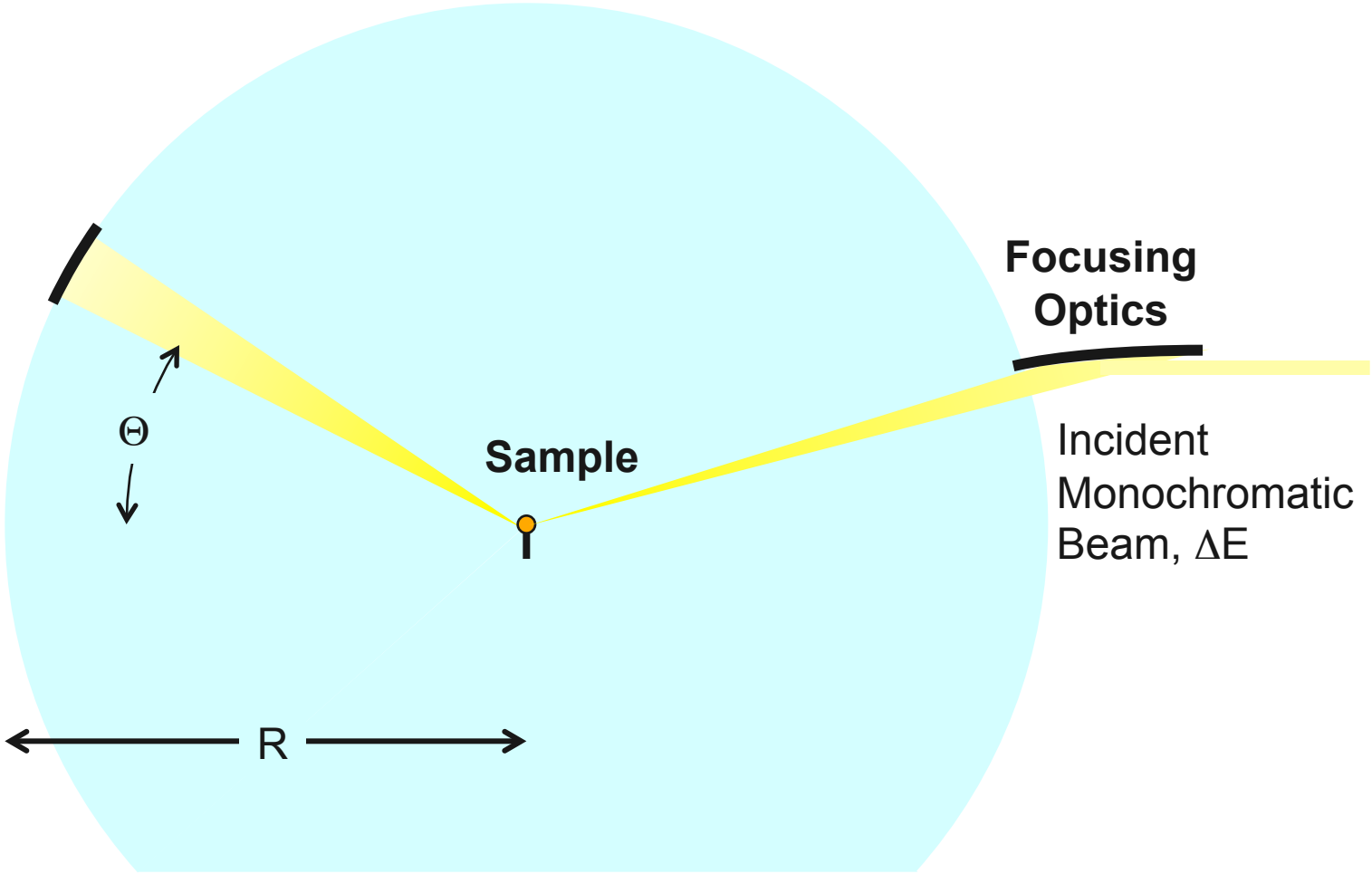
- **"Extreme" sample environments**
  - high pressure, very low and very high temperatures, high magnetic fields, …
  - Micro-positioning

## *IXS Renewal Pursuits, Level 1*
## *Enabling Technologies*

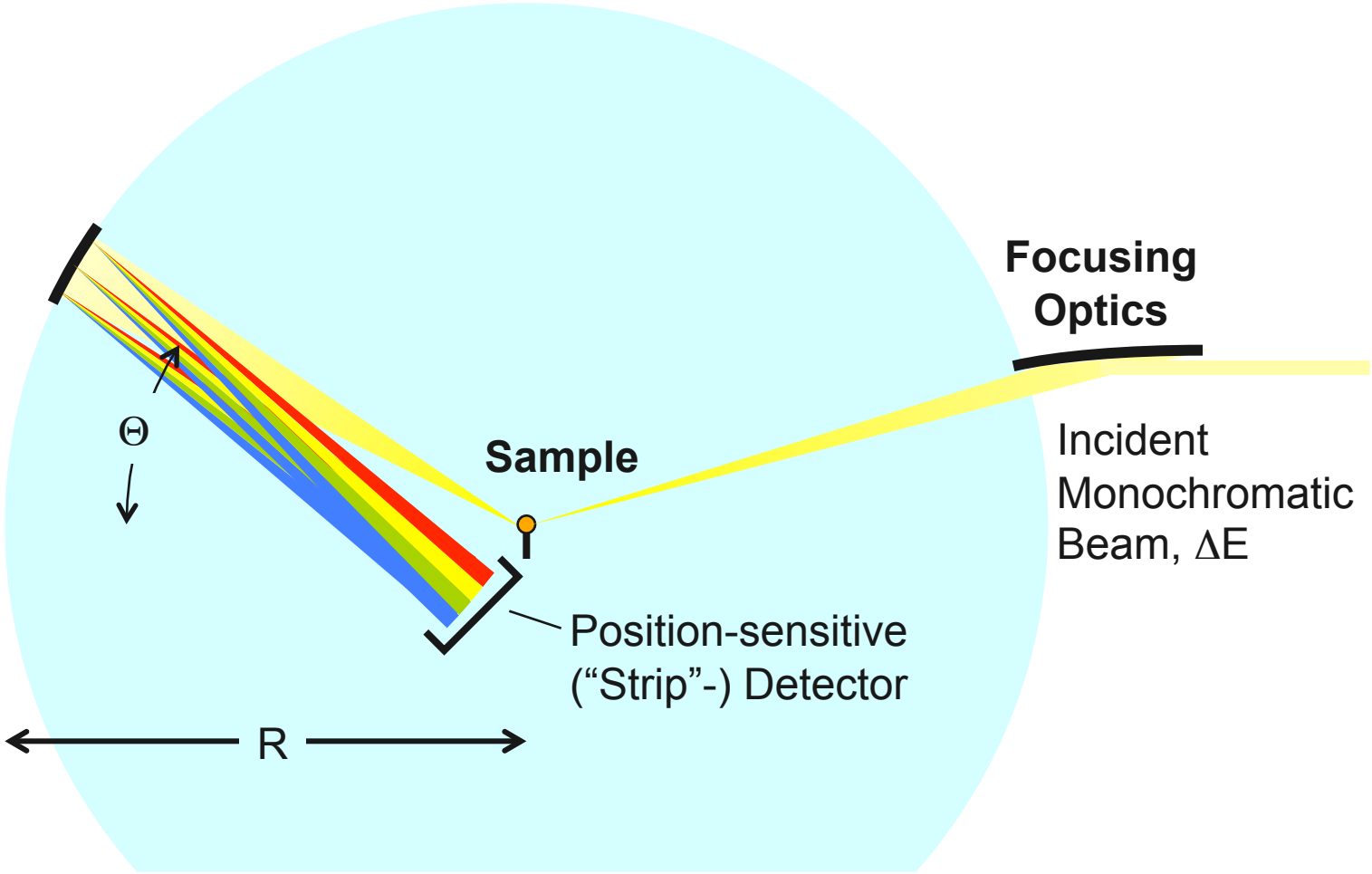
- **Novel Detectors**
  - Strip- and Pixel- detectors, Si, Ge
  - Integrated Arrays of these Detectors

- **Crystal Analyzer, Optics Development and Fabrication**
  - Spherical analyzers, various materials, reflections
  - Specialty crystals
  - Advanced monochromators
  - Advanced focusing mirrors

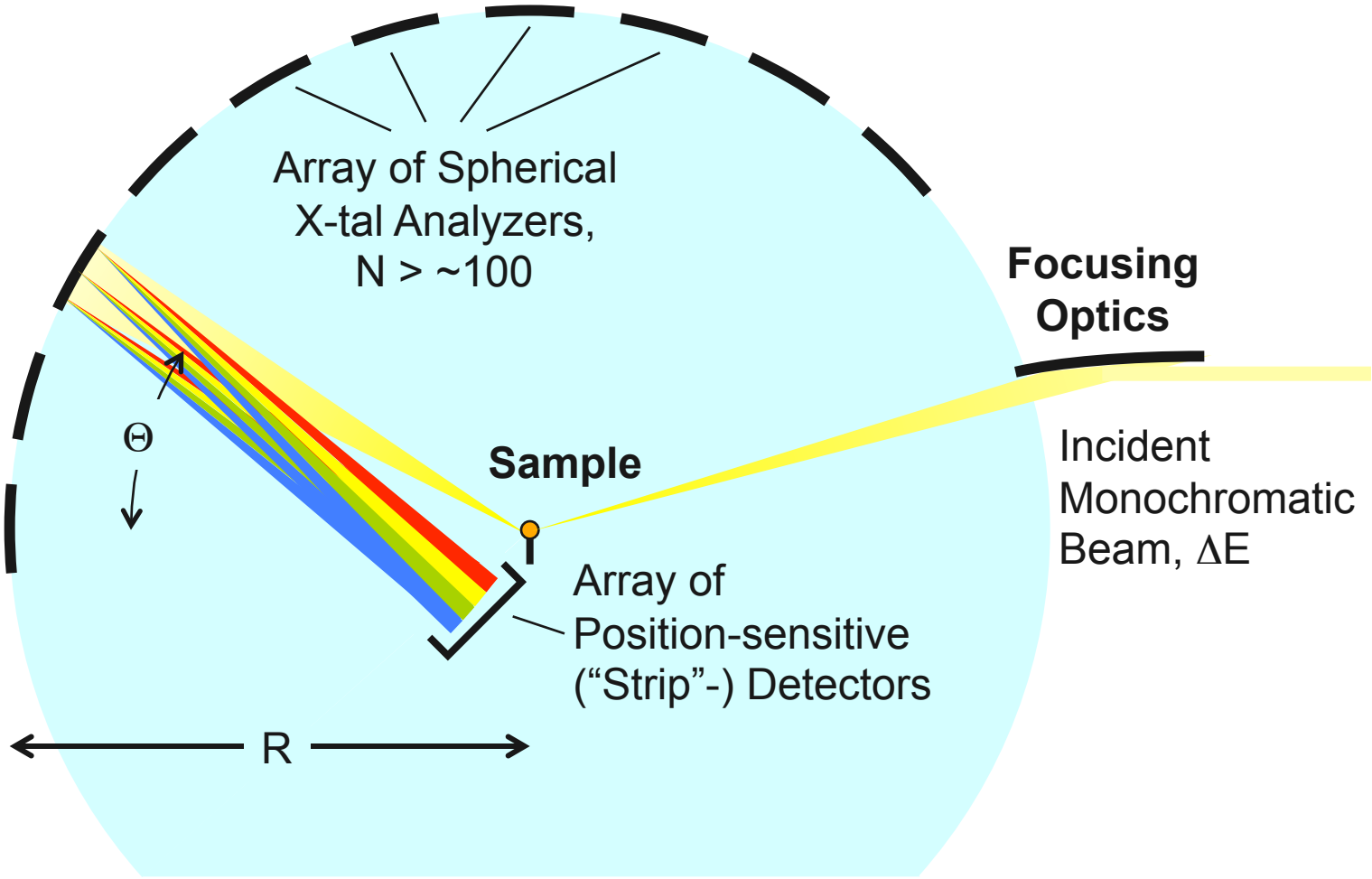
## *IXS Renewal Pursuits, Level 2*
## *New Instruments, based mostly on established technologies*



## *IXS Renewal Pursuits, Level 2*
## *New Instruments, based mostly on established technologies*

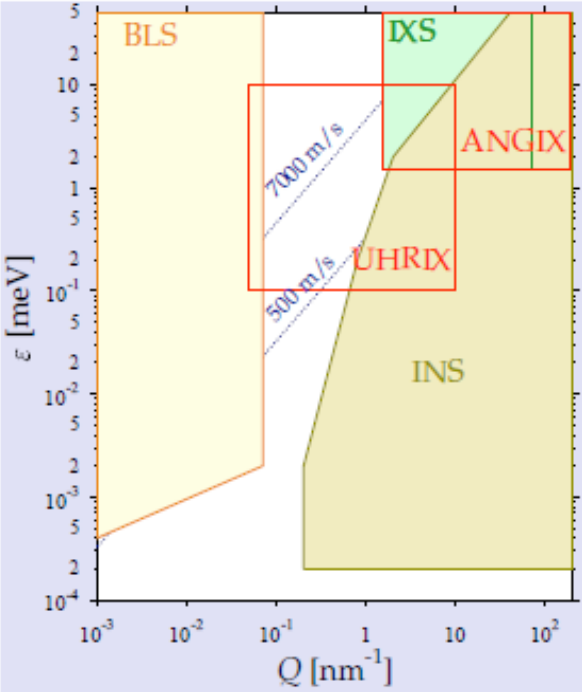


## *IXS Renewal Pursuits, Level 2*
## *New Instruments, based mostly on established technologies*



## *IXS Renewal Pursuits, Level 3*
## *New Instruments, based mostly on novel concepts, optics*

*"UltraHighResolutionIX"*
*Y. Shvyd'ko*



## *Conclusions*

## *IXS Renewal Pursuits on three Levels*

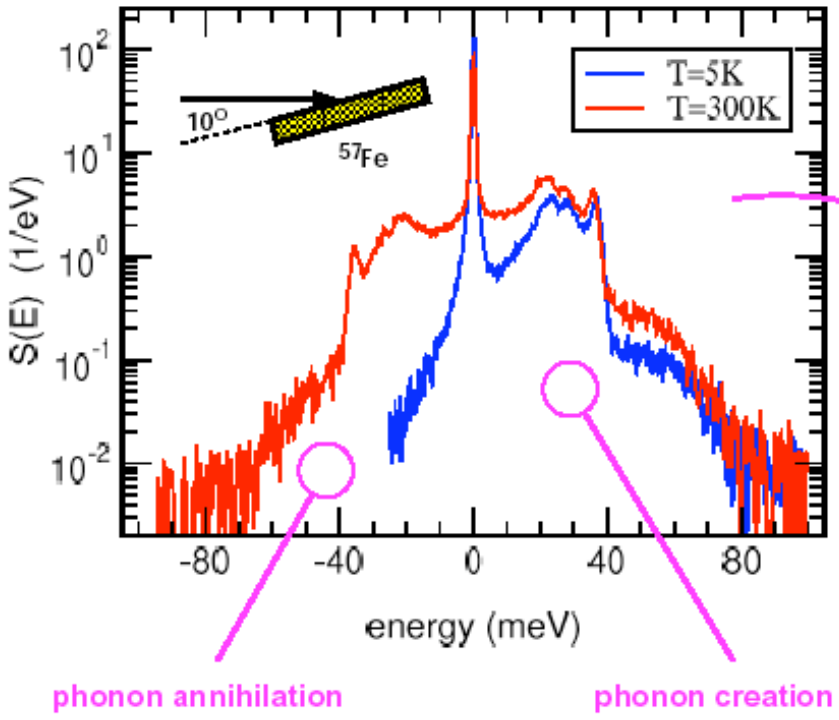
- Infra-Structure Improvements to existing Facilities
- One or Two new Instruments based on exsting technologies (elctrons, phonons)
- One new “research” Instruments based on novel technologies (UHRIX, ultra-high resolution)

# NUCLEAR RESONANT SCATTERING

***micro-eV resolution***

***inelastic x-ray spectroscopy***

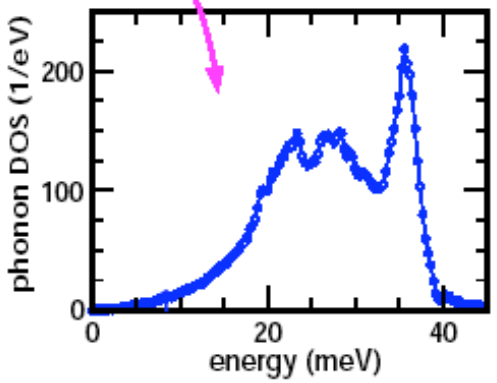
NRIXS on polycrystalline Fe (bcc):



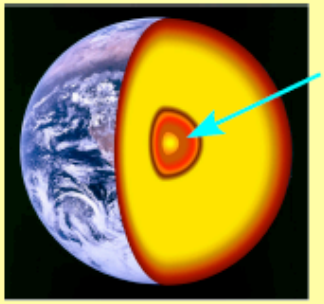

☆ the partial phonon DOS is extracted from the spectrum

*M.Hu et al., Nucl.Intrum.Methods A 428 (1999)*

*W.Sturhahn, Hyperfine Int. 125 (2000)*



# Extreme conditions: High pressure & temperature



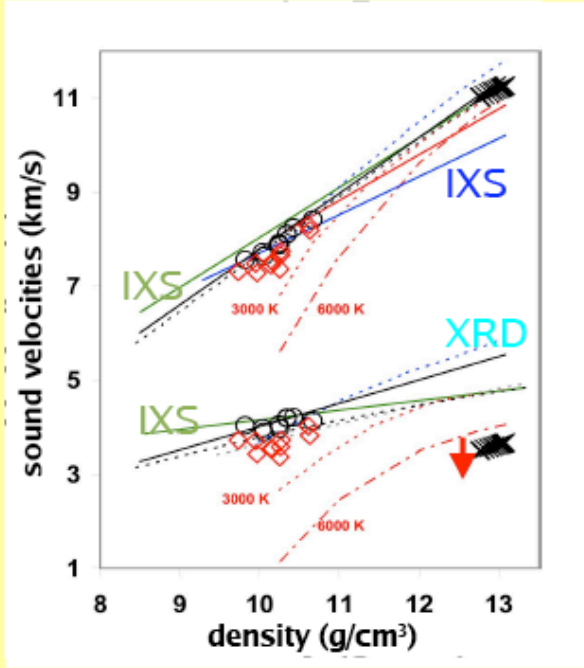
P > 1Mbar
T > 2000K

*J.-F. Lin, W. Sturhahn, J. Zhao, G. Shen, H.-k. Mao, R.J. Hemley, Science **308** (2005) 1892*
*"Sound Velocities of Hot Dense Iron: Birch's Law Revisited"*

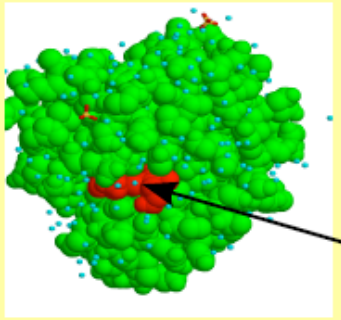
- explicit temperature dependence
- v ∝ ρ rule (Birch's Law) cannot be used for extrapolations

- *APS publications*


*H.-k. Mao et al., Science **292** (2001) 914*
*V.V. Struzhkin et al., Phys. Rev. Lett. **87** (2001) 255501*
*J.-F. Lin et al., Geophys. Res. Lett. **30** (2003) 2112*
*W. Mao et al., Geophys. Res. Lett. **31** (2004) L15618*
*J.-F. Lin et al., Earth and Planet. Sci. Lett. **226** (2004) 33*
*H. Kobayashi et al., Phys. Rev. Lett. **93** (2004) 195503*
*G. Shen et al., Phys. Chem. Minerals **31** (2004) 353*
*J. Zhao et al., High Press. Res. Phys. **24** (2004) 447*
*J.-F. Lin et al., Geophys. Res. Lett. **31** (2004) L14611*
*J.S. Tse et al., Nature Materials **4** (2005) 917*
*J.-F. Lin et al., Science **308** (2005) 1892*
*W. Sturhahn et al., Geophys. Res. Lett. **32** (2005) L12307*
*J.M. Jackson et al., American Mineralogist **90** (2005) 199*
*W. Mao et al., Science **312** (2006) 564*
*H. Giefers et al., Phys. Rev. B **73** (2006) 094303*
*J.-F. Lin et al., Phys. Rev. B **73** (2006) 113107*
*J. Li et al., Phys. Chem. Minerals **33** (2006) 575*
*J.-F. Lin et al., Geophys. Res. Lett. **33** (2006) L22304*
*H. Giefers et al., Phys. Rev. Lett. **98** (2007) 245502*

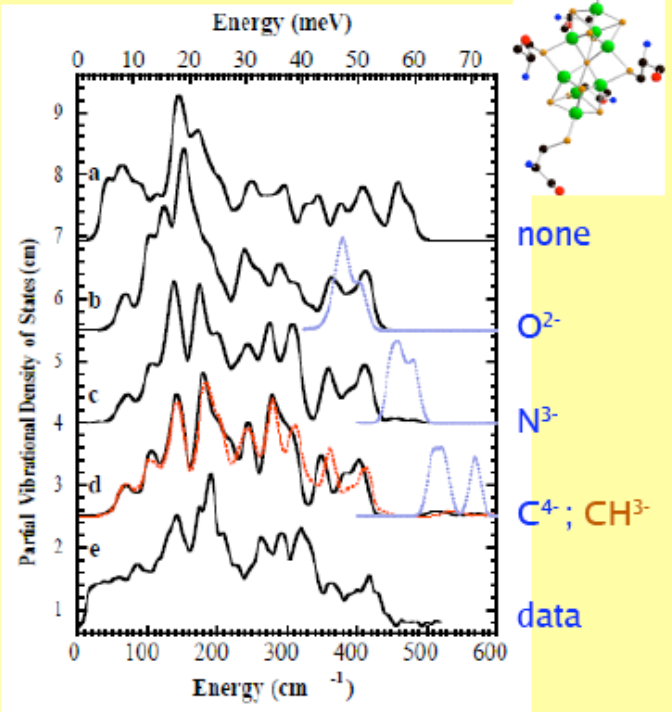
# Enzymes, proteins, and other large molecules



$^{57}$Fe in myoglobin

*Y. Xiao, K. Fisher, M.C. Smith, W. Newton, D.A. Case, S.J. George, H. Wang, W. Sturhahn, E.E. Alp, J. Zhao, Y. Yoda, S.P. Cramer, J. Am. Chem. Soc. 128 (2006) 7608*
*How Nitrogenase Shakes - Initial Information about P-Cluster and FeMo-cofactor Normal Modes from Nuclear Resonance Vibrational Spectroscopy (NRVS)*

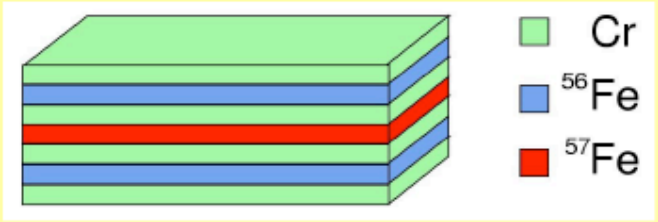
- only the nuclear resonant $^{57}$Fe atoms produce the signal.
- other nearby light atoms modify the Fe vibrations.




- *APS publications*

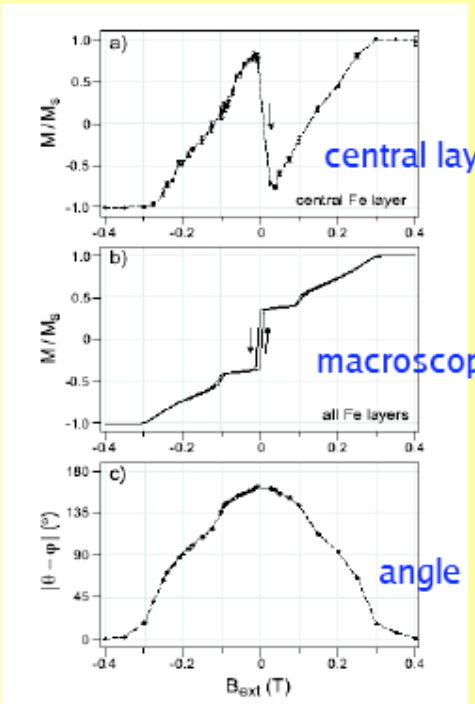

*C. Keppler et al., Eur. Biophys. J.* ***29*** *(2000) 146*
*J.T. Sage et al., Phys. Rev. Lett.* ***86*** *(2001) 4966*
*B.K. Rai et al., Phys. Rev. E* ***66*** *(2002) 051904*
*K. Achterhold et al., Phys. Rev. E* ***65*** *(2002) 051916*
*B.K. Rai et al., Biophys. Journ.* ***82*** *(2002) 2951*
*U. Bergmann et al., J. Am. Chem. Soc.* ***125*** *(2003) 4016*
*T.E. Budarz et al., J. Phys. Chem. B* ***107*** *(2003) 11170*
*B.K. Rai et al., J. Am. Chem. Soc.* ***125*** *(2003) 6927*
*B.M. Leu et al., J. Am. Chem. Soc.* ***126*** *(2004) 4211*
*M.C. Smith et al., Inorg. Chem.* ***44*** *(2005) 5562*
*W. Zeng et al., J. Am. Chem. Soc.* ***127*** *(2005) 11200*
*Y. Xiao et al., J. Am. Chem. Soc.* ***127*** *(2005) 14596*
*K. Adams et al., J. Phys. Chem. B* ***110*** *(2006) 530*
*Y. Xiao et al., J. Am. Chem. Soc.* ***128*** *(2006) 7608*
*V. Starovoitova et al., J. Phys. Chem. B* ***110*** *(2006) 13277*
*B.M. Leu et al., Biophys. J.* ***92*** *(2007) 3764*
*N.J. Silvernail et al., J. Am. Chem. Soc.* ***129*** *(2007) 2200*
*T. Petrenko et al., J. Am. Chem. Soc.* ***129*** *(2007) 11053*

# Nanostructures: Magnetism and Phonons




*C. L'abbé, J. Meersschaut, W. Sturhahn, J.S. Jiang, T.S. Toellner, E.E. Alp, S.D. Bader,*
*Phys. Rev. Lett.* ***93*** *(2004) 037201*
*"Nuclear Resonant Magnetometry and its Application to Fe/Cr Multilayers"*

- only the central Fe layer contains the nuclear resonant isotope $^{57}$Fe and produces the signal.




- *APS publications*


*T.S. Toellner et al., Phys. Rev. Lett.* ***74*** *(1995) 3475*
*R. Röhlsberger et al., J. Appl. Phys.* ***86*** *(1999) 584*
*R. Röhlsberger et al., Physica B* ***581*** *(1999) 236*
*W. Sturhahn et al., J. Magn. Magn. Mater.* ***198-199*** *(1999) 590*
*W. Keune et al., Hyperfine Interact.* ***123-124*** *(1999) 847*
*T. Ruckert et al., Hyperfine Interact.* ***126*** *(2000) 363*
*B. Roldan Cuenya et al., Phys. Rev. B* ***64*** *(2001) 235321*
*T. Ruckert et al., J. Magn. Magn. Mater.* ***240*** *(2002) 562*
*C. L'abbé et al., Phys. Rev. Lett.* ***93*** *(2004) 037201*
*W. Keune et al., J. Phys: Cond. Matt.* ***16*** *(2004) S396*
*M. Walterfang et al., Phys. Rev. B* ***71*** *(2005) 035309*
*B. Sahoo et al., J. Phys. Chem. B* ***66*** *(2005) 2263*
*B. Sahoo et al., Phase Transitions* ***79*** *(2006) 839*
*B. Roldan Cuenya et al., Phys. Rev. B* ***76*** *(2007) 195422*
*B. Roldan Cuenya et al., Phys. Rev. B* ***77*** *(2008) 165410*

# CURRENT STATUS AT THE APS

- HP-CAT, sector 16-ID, (2.5m, 3.3cm) undulator
  - SMS & NRIXS with $^{57}$Fe isotope
  - energy resolution 2 meV
  - micro-focusing
  - He-flow cryostats

- XOR, sector 3-ID, (5m, 2.7cm) undulator
  - SMS & NRIXS with resonances of $^{57}$Fe, $^{83}$Kr, $^{119}$Sn, $^{151}$Eu, $^{161}$Dy isotopes
  - energy resolution 1 meV or better
  - micro-focusing
  - Laser heating
  - integrated x-ray diffraction
  - He-bath/flow cryostats
  - magnetic fields conventional/superconducting
  - circularly polarized x rays

# UPGRADE OPTIONS for nuclear resonant scattering

➢ **increased brilliance and flux from the APS**
☆ higher current
☆ extended straight sections
☆ superconducting or in-vacuum undulators
☆ isotope-dependent source optimization

➢ **higher energy resolution monochromators: 0.1 meV**
☆ cryogenic monochromators with higher efficiency
☆ energy bandwidth of about 100 μeV

➢ **optics for new high energy isotopes**
☆ Sb, Ni

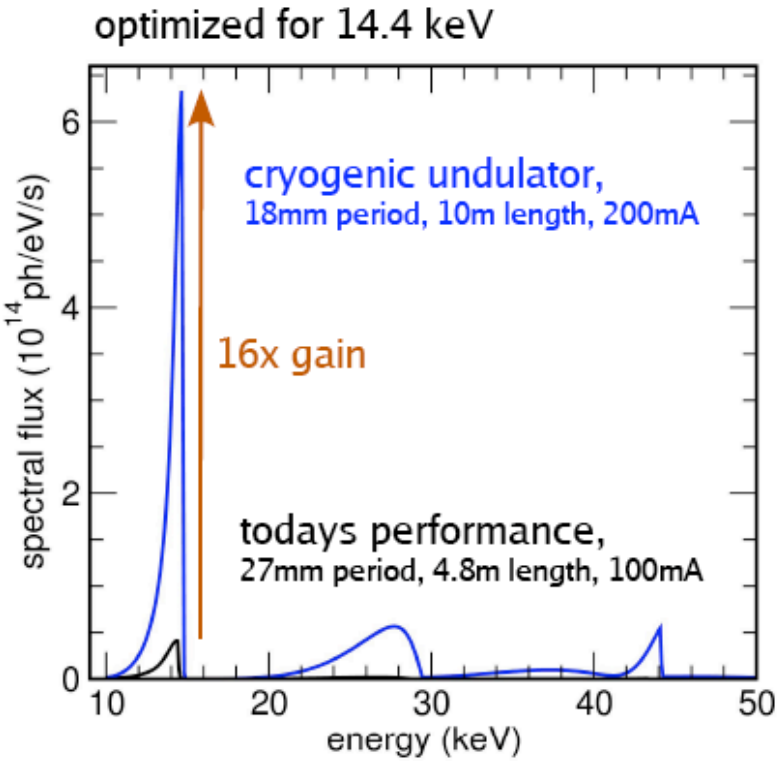
➢ **pixellated APD detectors**
☆ Improve throughput and collection efficiency, new imaging applications, "in-situ" material synthesis at high pressure and temperature with on-line characterizatin

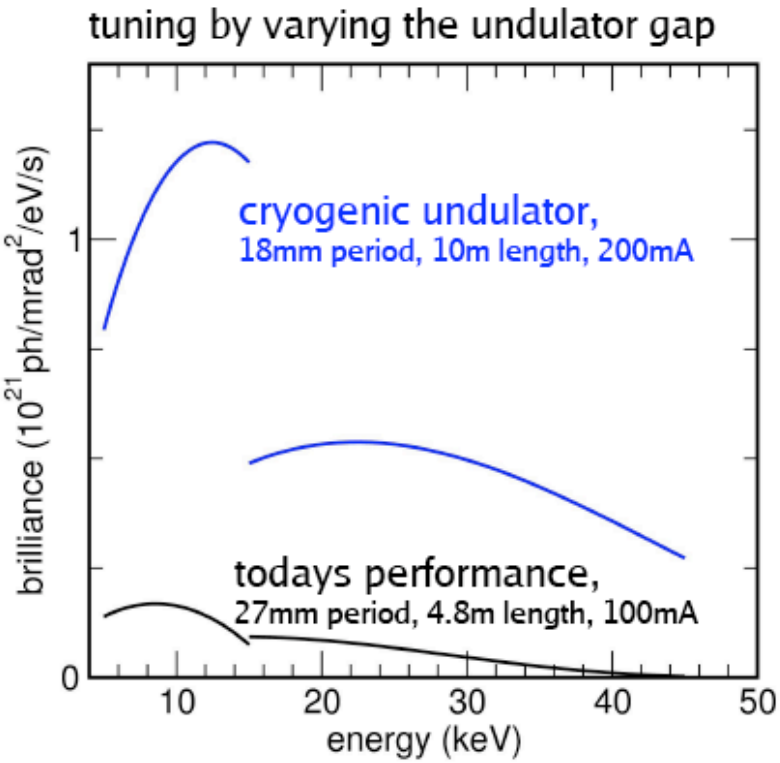
➢ **infrastructure improvements**
☆ expand the workspace available for experiments at 3-ID, special environments like high magnetic field, in-situ UHV chamber,

➢ **Software for on-line data analysis, and pre-experiment planning**
☆ Improved CONUSS and PHOENIX software with GUI and robust input files

➢ **bunch purity**
☆ why does it suck ?

# NEW UNDULATORS NEEDED



# BUNCH PURITY