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U.S. DEPARTMENT OF ENERGY

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Bulk Probes

Peter J. Chupas

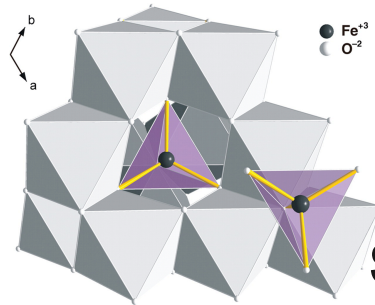
Ulrich Lienert

Bulk Probes

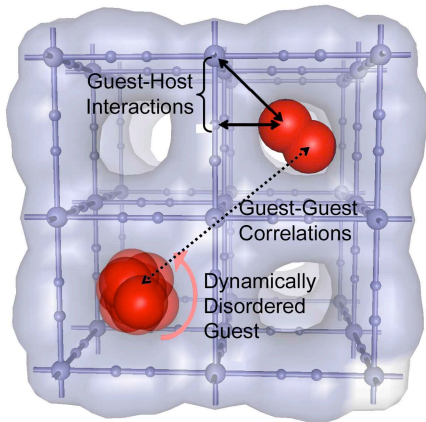
- *Pair Distribution Function (PDF)*
- *Small-Angle-Scattering (SAXS)*
- *Non-resonant Magnetic Scattering*
- *Aggregate Stress / Strain / Texture*
- *Laue Micro-diffraction Microscopy*
- *High Energy Diffraction Microscopy (HEDM)*

Bulk Probes: Spanning Length-scales in Real Materials

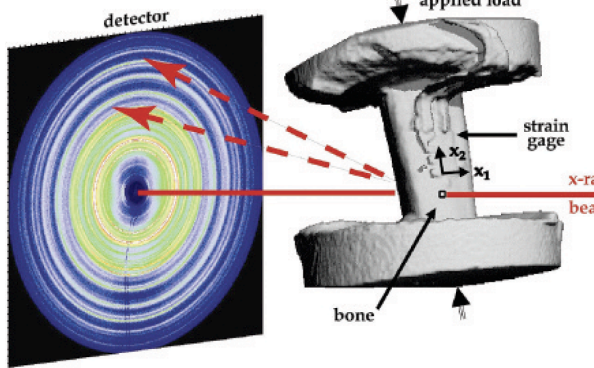
Nano-structure determination



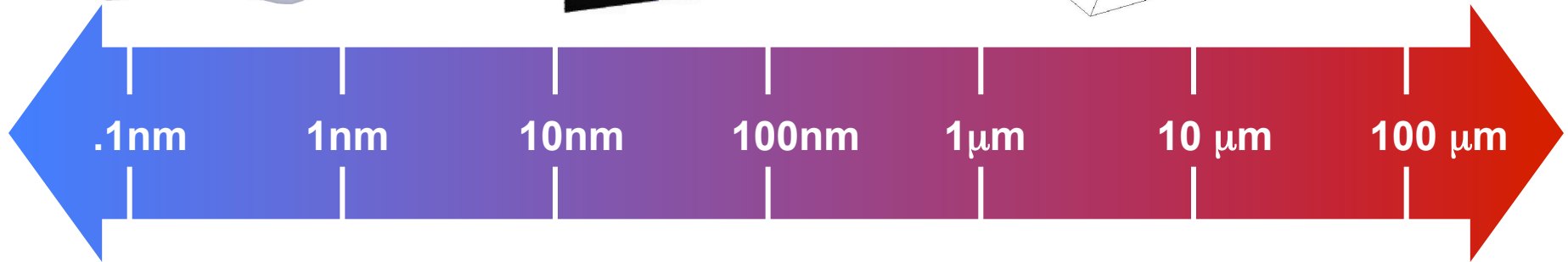
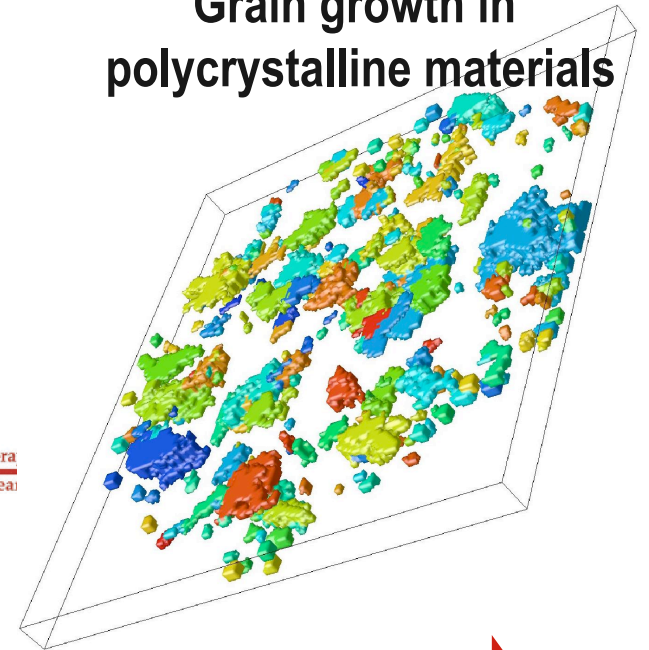
Hydrogen Storage



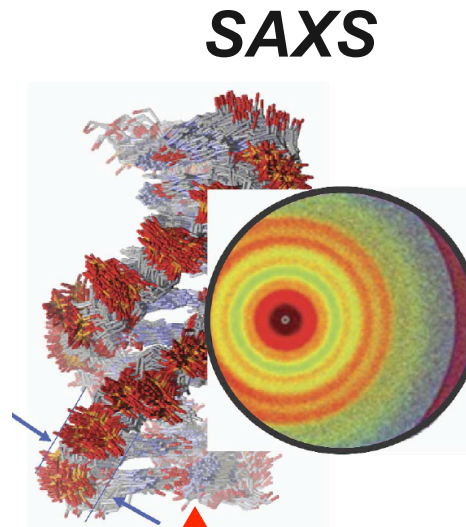
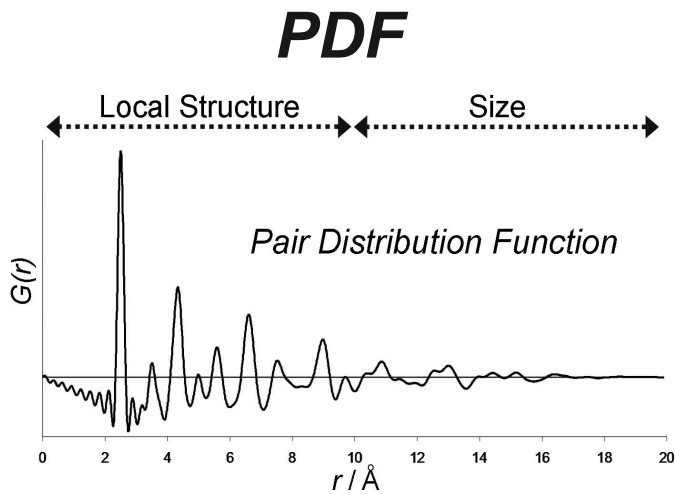
Stress/Strain bone



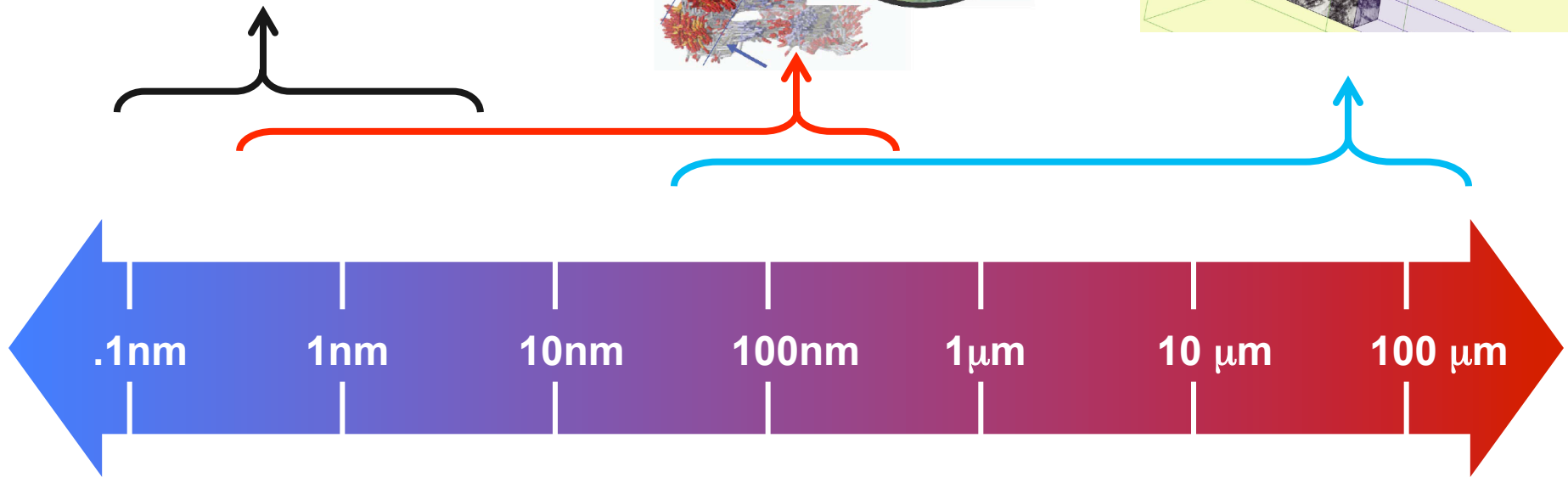
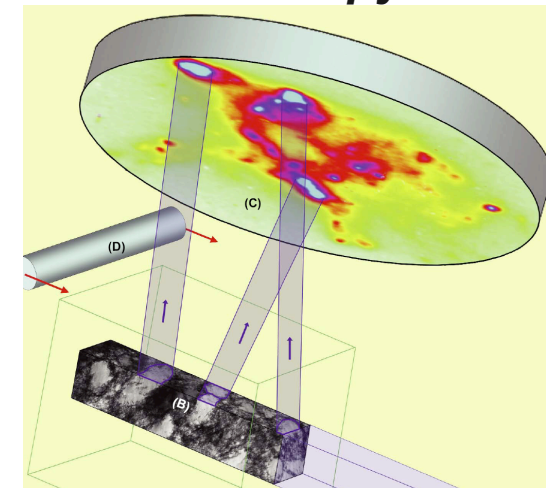
Grain growth in polycrystalline materials



Bulk Probes: Techniques Spanning Length-scales

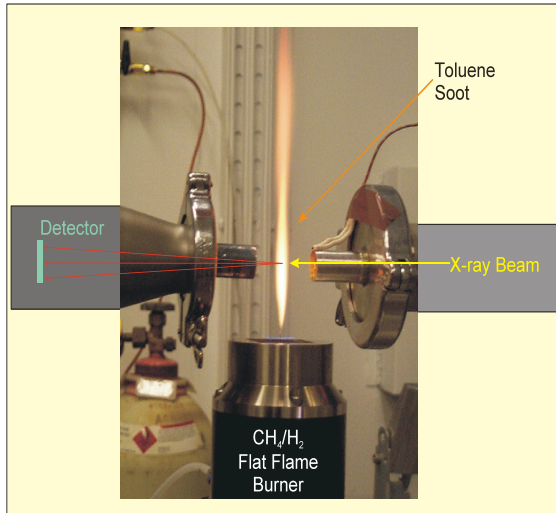


Laue Microdiffraction Microscopy

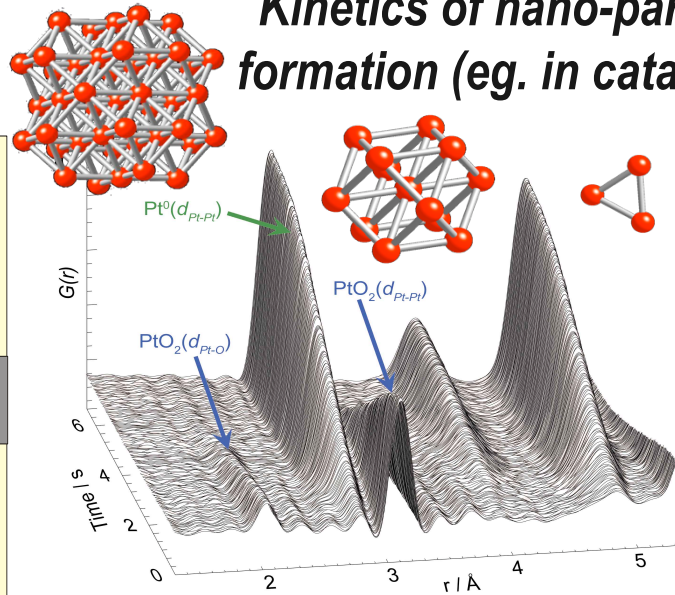


Bulk Probes: Temporal Hierarchy

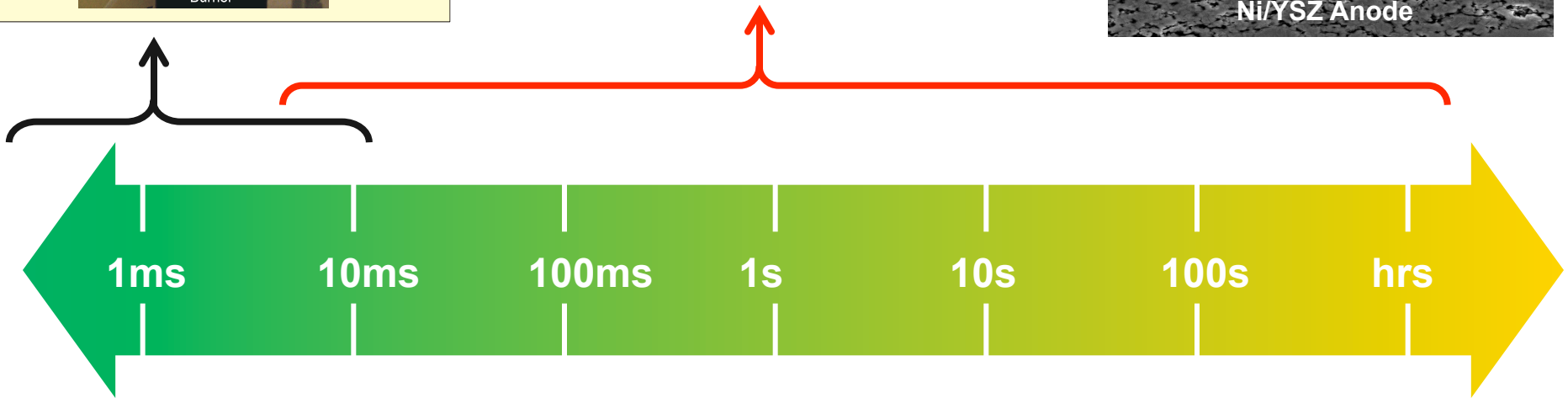
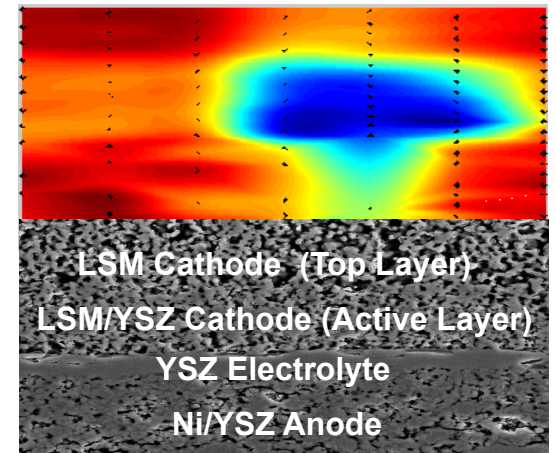
Soot formation



Kinetics of nano-particle formation (eg. in catalysis)



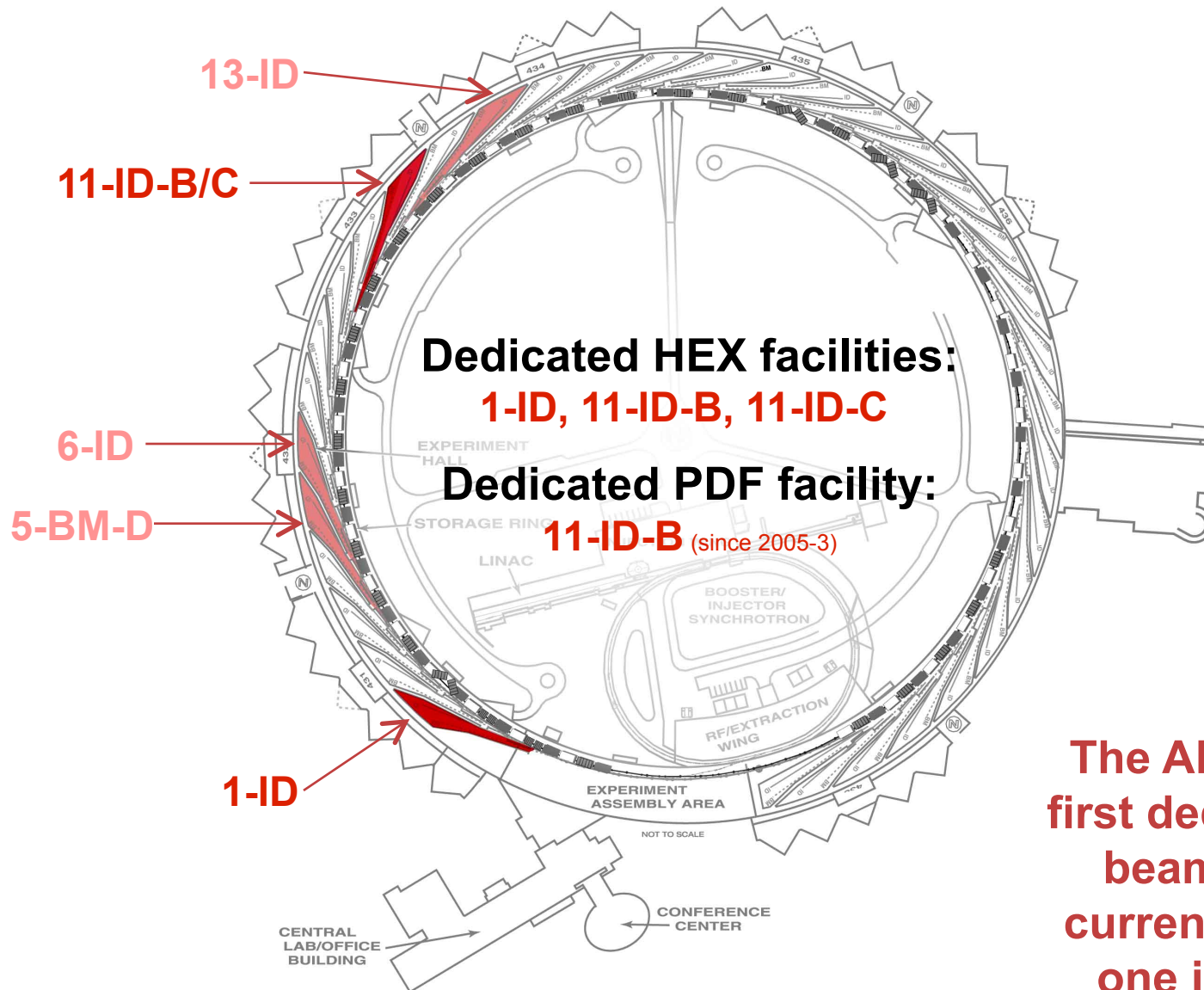
Deviatoric Strain In SOFCs



A Holistic Approach Enables New Science

- **Software** (seamless data reduction and analysis)
- **Area Detectors** (matched to the experimental needs)
- **Sample environment** (optimized for an instrument)
- **Dedicated instruments** (minimize set-up time)
- **Optimized insertion devices** (eg. superconducting undulators)
- **Rationally combining techniques** (eg. PDF & SAXS)
- **Expanding modes of access** (Mail-in / Rapid-Access / Long Term Access)

PDF – Current Status



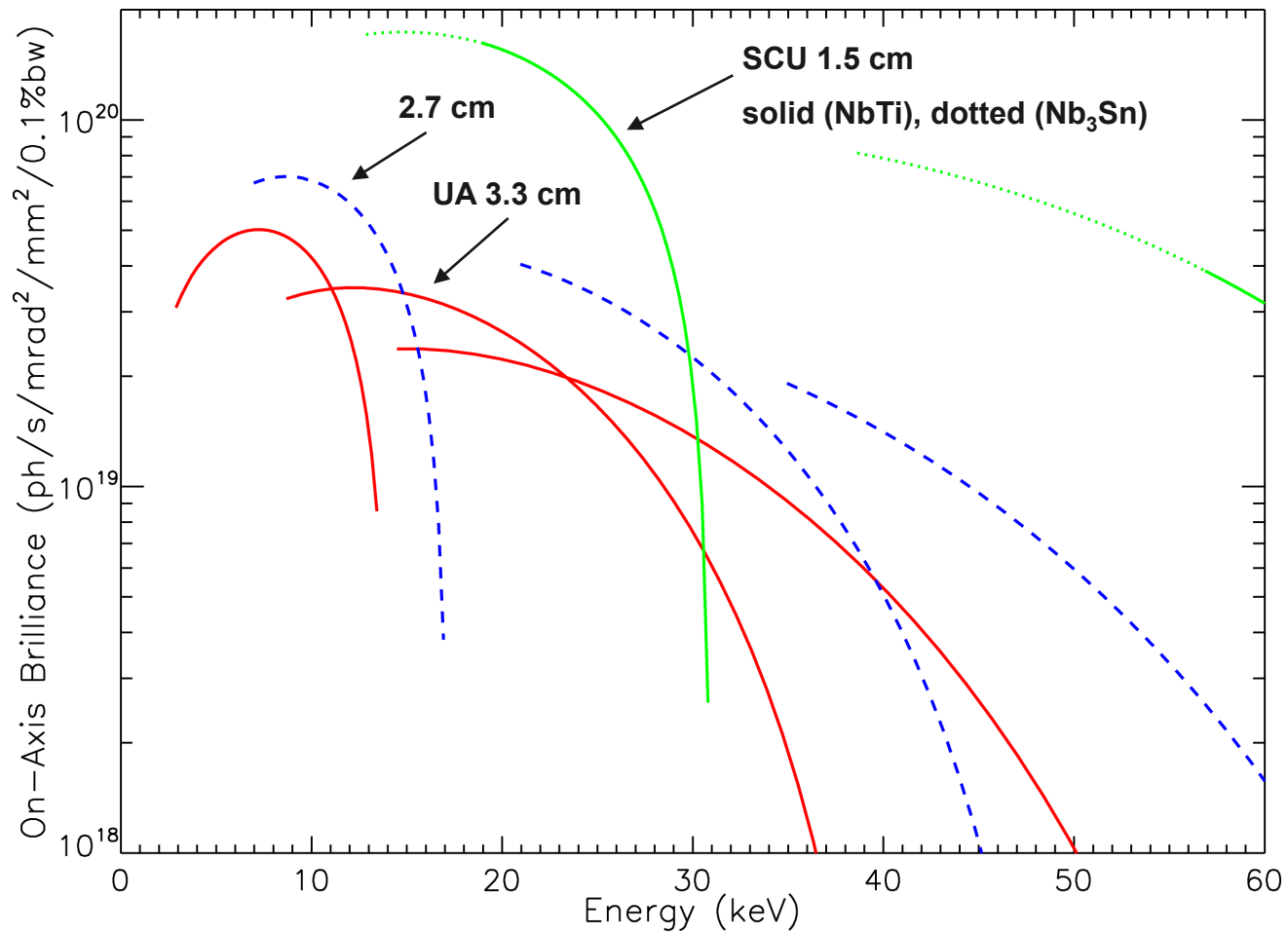
The APS has the first dedicated PDF beamline, and currently the only one in the U.S.

PDF- Critical Beamlines

- **Dedicated PDF (High throughput PDF / in-situ chemical studies)**
 - Optimize 60 keV
 - Optional Focusing to 10 microns
 - Wide Q range 0.01 – 40.0 Å⁻¹
 - Optional SAXS / moderate resolution powder diffraction
- **Sub-microfocus HE beamline (High pressure / extreme conditions / spatially resolved studies)**
 - Spatially resolved measurements
 - High-pressure DAC
 - 200 nm focus

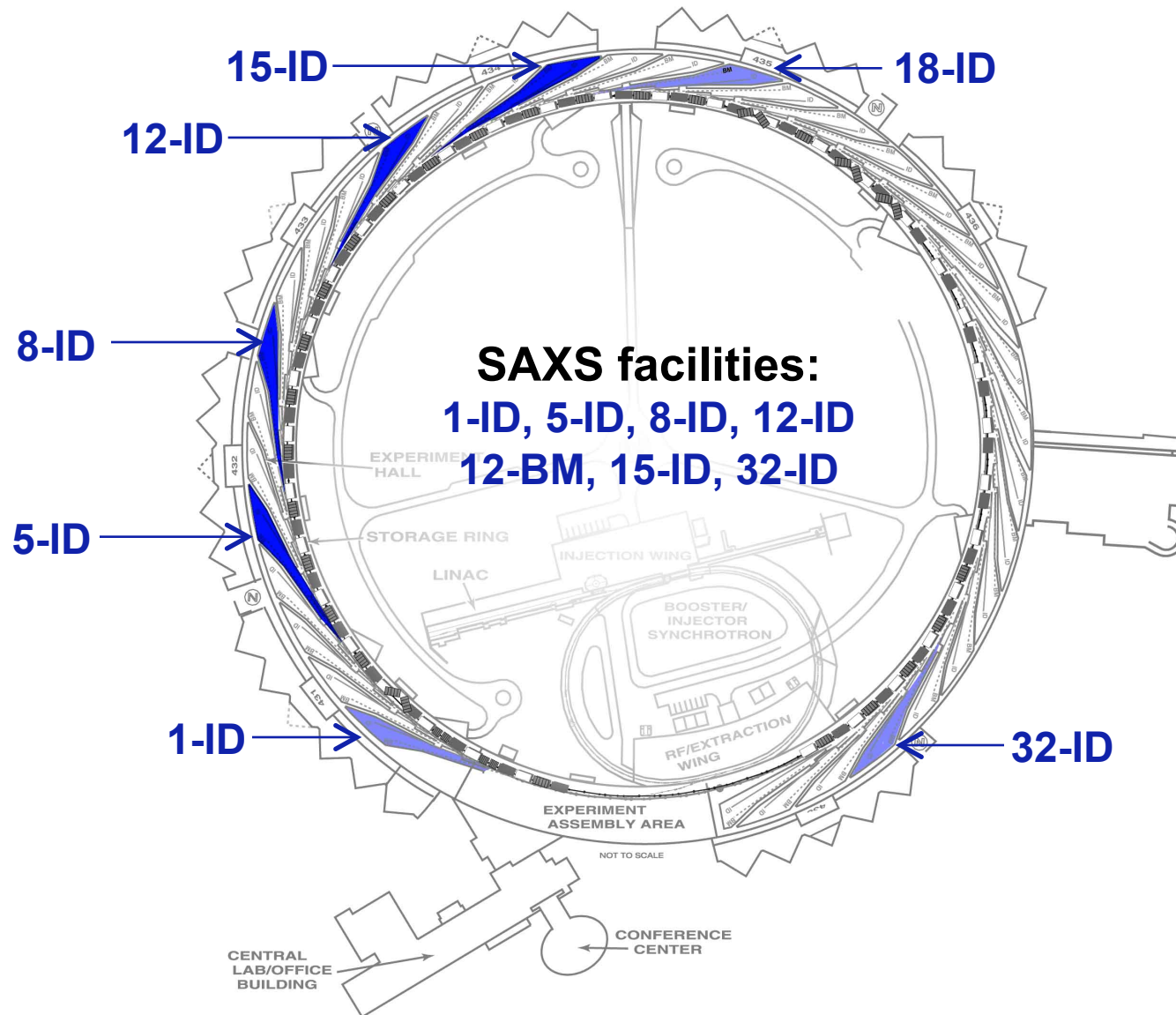
All require high energy X-rays and thus require APS

High Energies- Optimized Insertion Devices



(courtesy L. Moog)

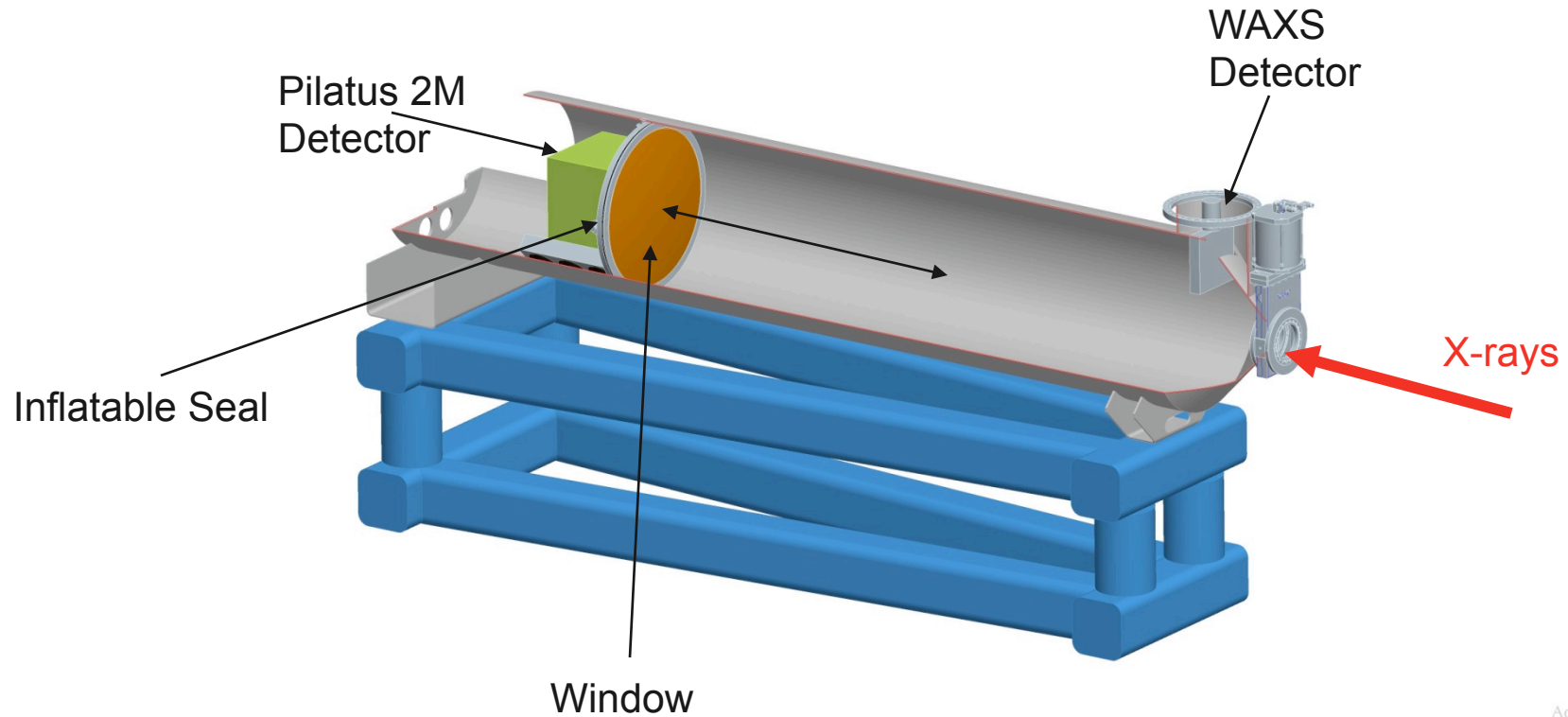
SAXS – Current Status



SAXS – Critical Beamlines

- **Anomalous and time resolved SAXS/WAXS/USAXS beamline**
 - Technically complicated experiments which take full advantage of capabilities
 - Pink beam – high flux for fast experiments
 - Wide Q range 0.001 – 5.0 / Wide energy range 4.5 – 36 KeV
 - Combined experiments – XANES, MS, DSC
- **Dedicated SAXS/WAXS/GISAXS beamline**
 - Easily adjustable Q range (0.006 – 2.0)
 - Rapid access/ Regular access (set aside time)
- **High-Energy SAXS / WAXS**
 - Wide Q range
 - Bulk / strongly absorbing samples

SAXS – Optimized Detectors

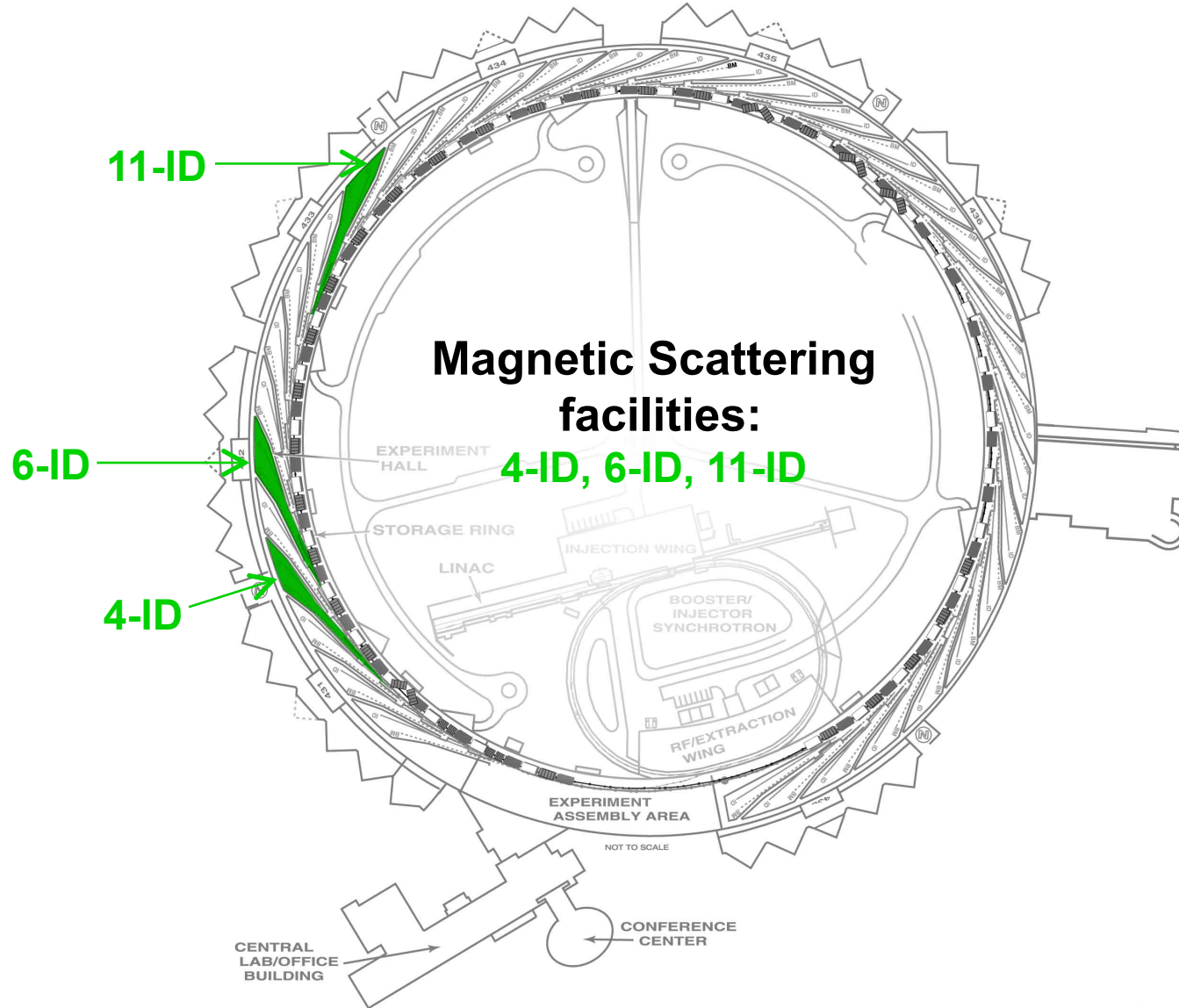


Active Part:SAX-DETI

Area detectors are a critical enabler

X.XX+0.01
X.XXX+0.001
ANG.+0.5

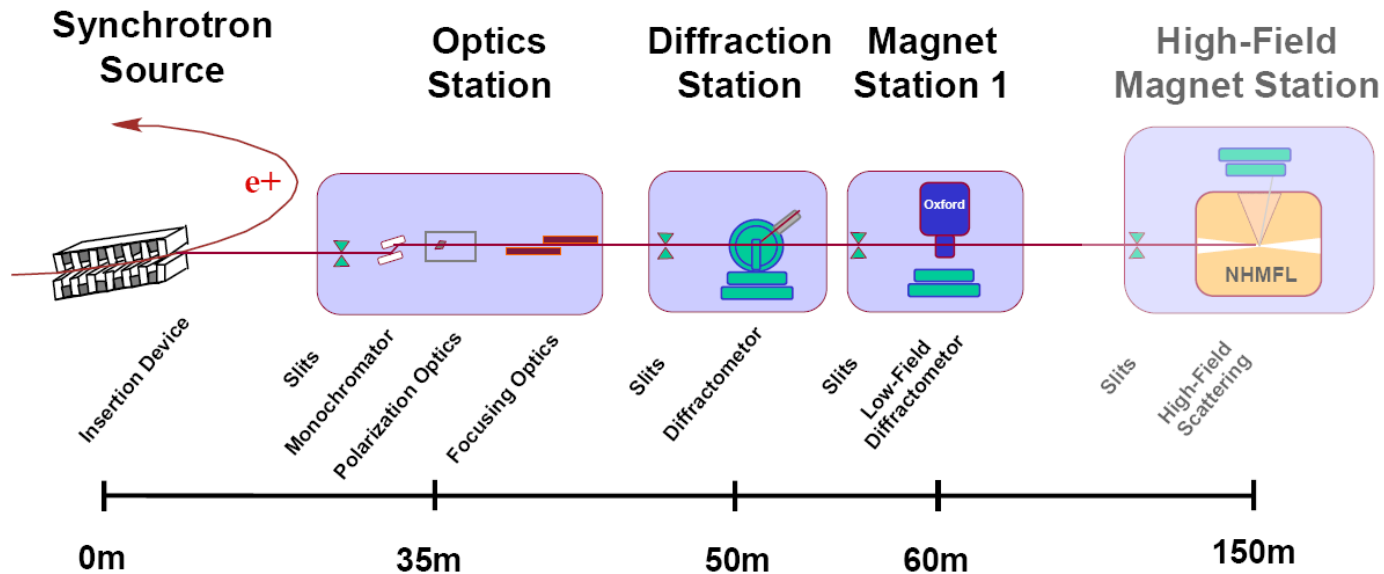
Magnetic Scattering (non-resonant) – Current Status



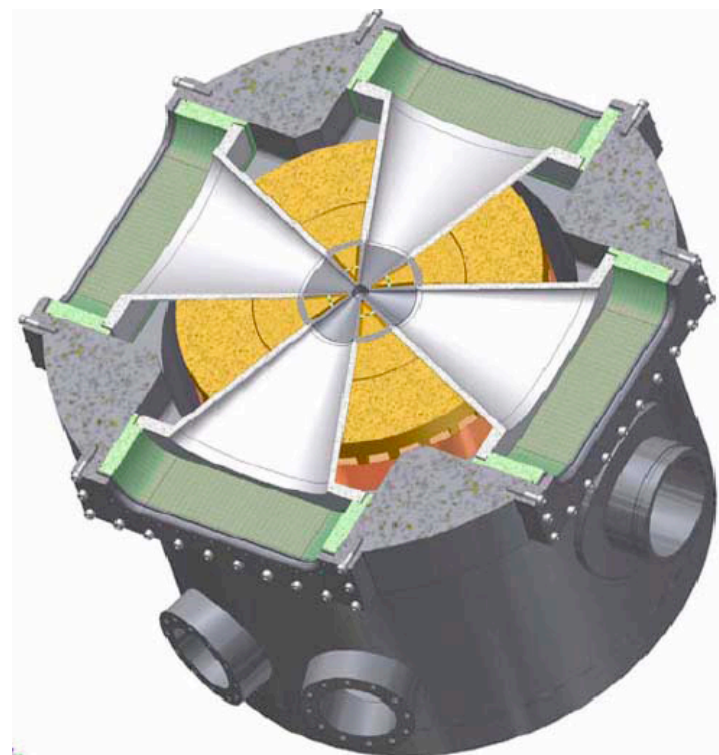
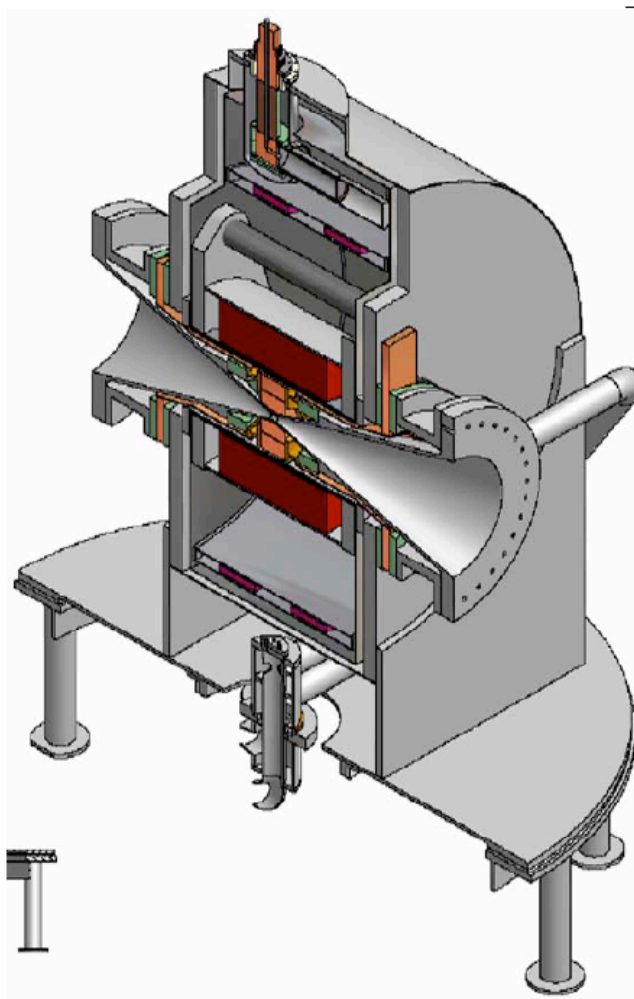
Non-resonant Magnetic Scattering- Critical Beamlines

■ Structural Studies (> 30 keV)

- Technically complicated experiments which take full advantage of capabilities
- Superconducting magnets to 15 T
- Novel hybrid magnet designs (to reach field in excess of 30 T)



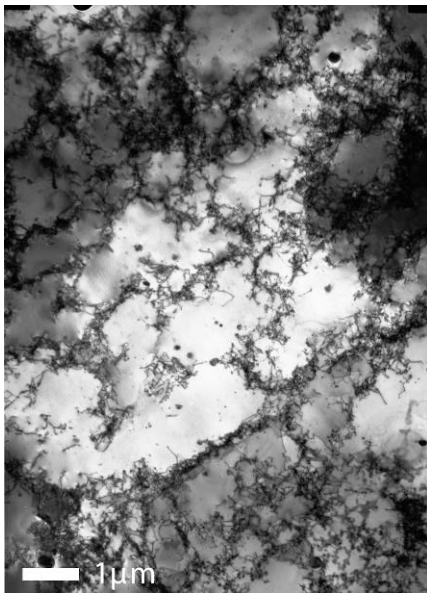
Non-resonant Magnetic Scattering- Partnerships



**Partnerships with NHMFL
for magnet designs**

Macro- & meso-scale polycrystalline materials

- Scientific case mainly covered by 'Engineering Applications and Applied research'
- Non-destructive



Hierarchically
Structured
Polycrystalline
Materials

Real space: 3D
Orientation: 3D
Strain: 6D
Time: 1D
+
processing

Aggregate Stress/Strain/Texture

- + Real materials, conditions, time
- Grain integrated information

High Energy Diffraction Microscopy

- + Fast volume mapping
- Spatial resolution detector limited

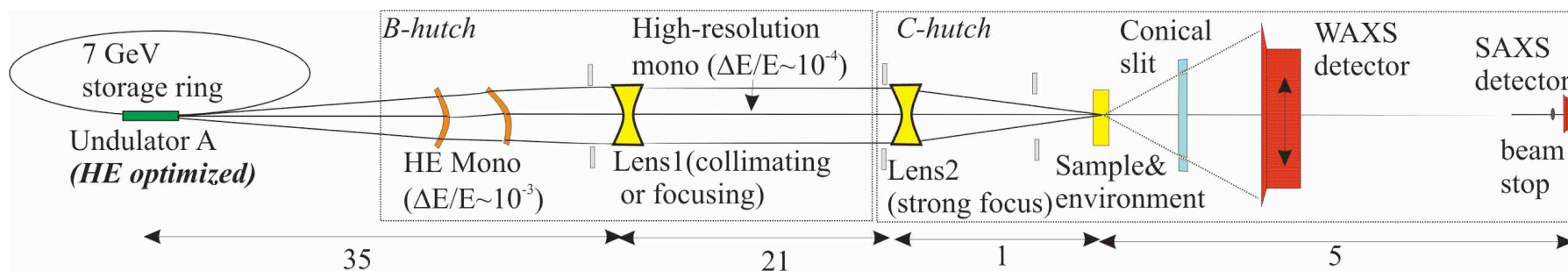
Laue Micro-Diffraction Microscopy

- + High spatial resolution
- Slow volume mapping

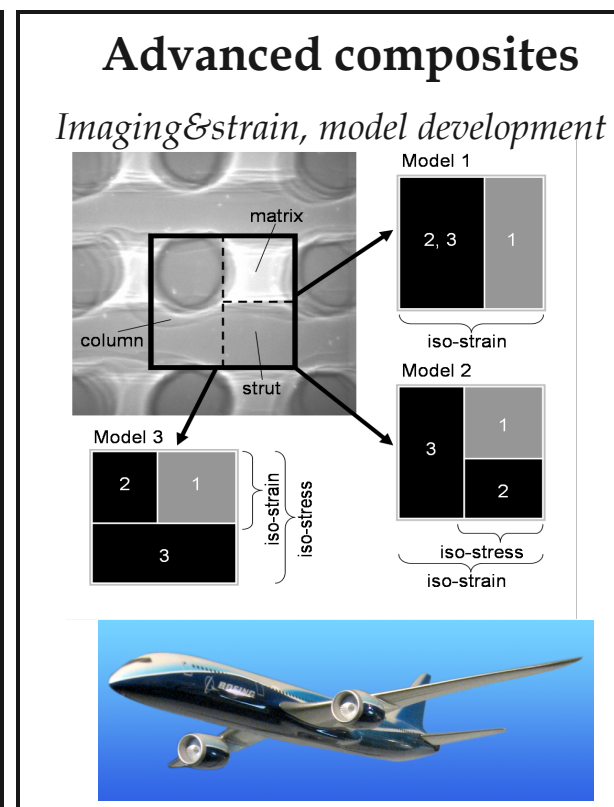
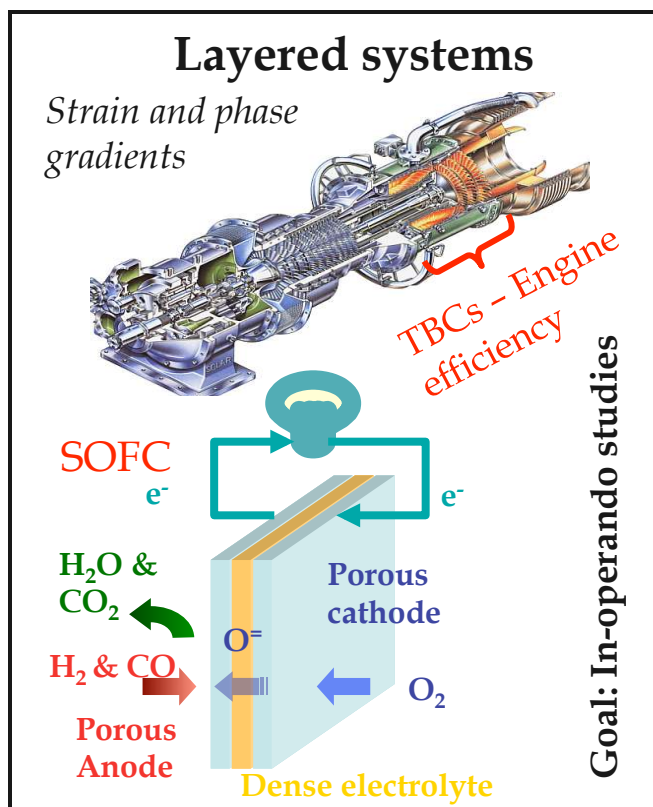
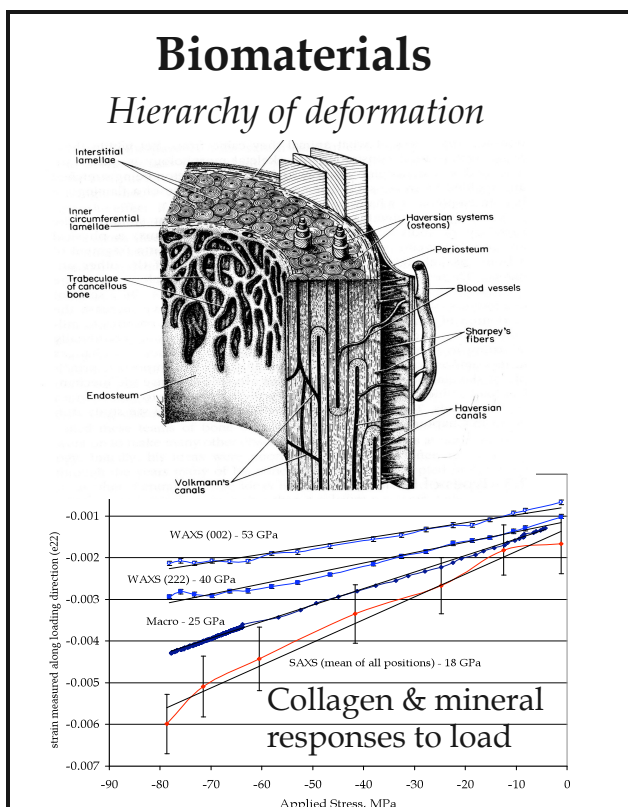
‘Engineering Applications and Applied Research’ recommendations

- Additional high-energy beamline for **aggregate** polycrystalline materials.
- Dedicated **HEDM** and μ -focused PDF/powder diffraction stations (1-ID Phase II upgrade).
- Independent **nano-** and **meso-scale Laue probes** on 34-ID.
- At least one **HE BM** (energy dispersive and monochromatic diffraction, tomography).
- Sample environments.
- Data acquisition and reduction software.
- Area detectors: faster, larger, smaller, efficient, ...

Aggregate polycrystalline materials – combined techniques

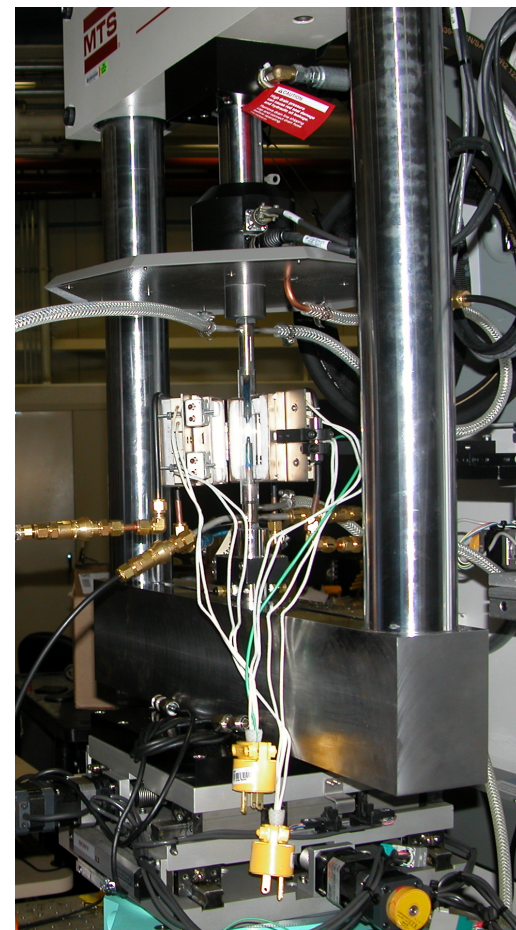


In-situ thermal, mechanical and thermo-mechanical studies



Aggregate polycrystalline materials - upgrade

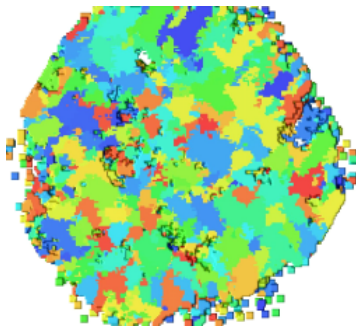
- Existing ID program
 - **Dedicated station/instruments**
 - Better integrate imaging with SAXS/WAXS
 - Area detectors (fast, efficient, more & smaller pixels)
 - Optimized HE insertion device
 - Sample environment →
 - Data acquisition & analysis software
- New BM white beam station
 - APS: High Energy BM spectrum
 - Complementary to highly over-subscribed high-energy ID instruments
 - Four instruments:
 - *1D Monochromatic WAXS*
 - *2D Monochromatic WAXS*
 - *Polychromatic diffraction*
 - *HE tomography (complementary to 2-BM)*



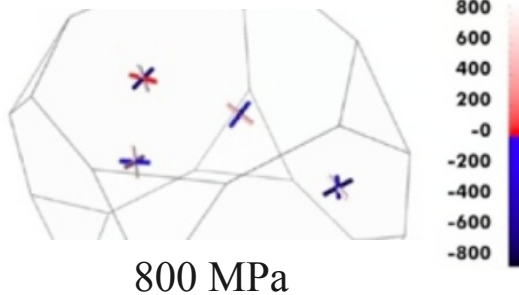
High Energy Diffraction Microscopy: status

- **High energies:** (40-80 keV)
 - bulk penetration throughout periodic system
 - sample environment
- q-space coverage
- **'tomographic' data acquisition:** time resolution

Near field

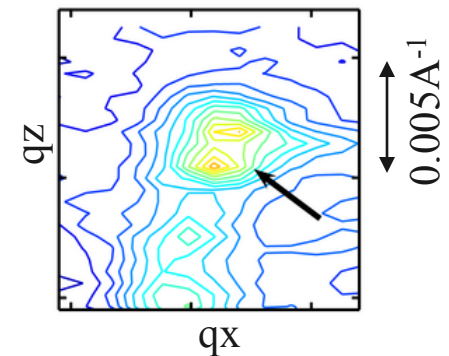


Far field, medium Δq



Far field, low Δq

Δ Strain: 0.09%



- Hierarchical imaging
- Real conditions (compared to EM)
- APS: high energies
- Collaboration with ESRF/Risoe project
- Large potential US user community (oversubscription limited)
- **1-ID: specialized optics but no dedicated station**
- sensitivity: 0.2 μm , resolution: 5-10 μm , 0.001 \AA^{-1}
- **sample environment, stability, robustness, data acquisition and reduction rate**

High Energy Diffraction Microscopy: upgrade

Medium term

- **Dedicated station/instruments**
- **Combine near- and far-field techniques**
- Tomography and DCT capability
- Area detectors (fast, efficient, more & smaller pixels)
- Optimized HE insertion devices
- Resolution -> 1 μm
- Sample environment
- **Data acquisition & on-line reduction (reconstruction) software, HPC**
- Partnering to leverage software development (ANL/MCS)

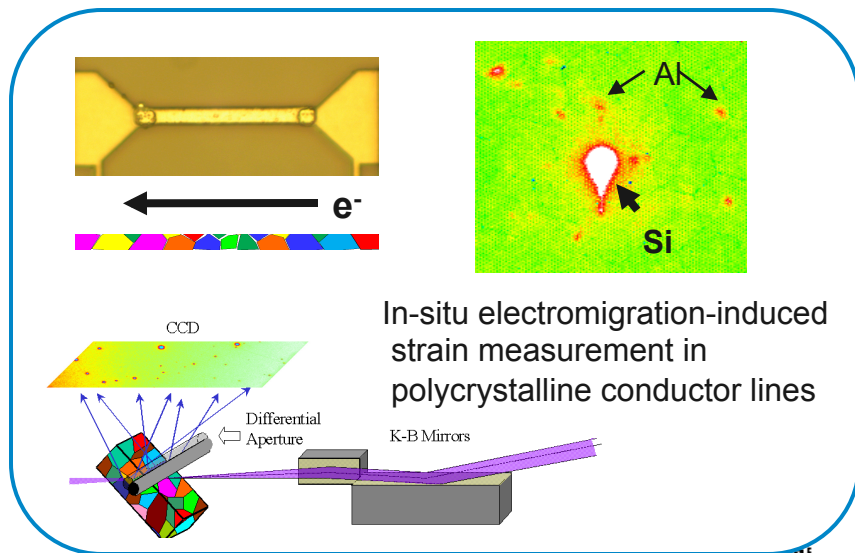
Added value

- Decisive test of models (e.g. strain & neighborhood)
- Grow user community

Laue Diffraction 3D Microscopy at 34-ID

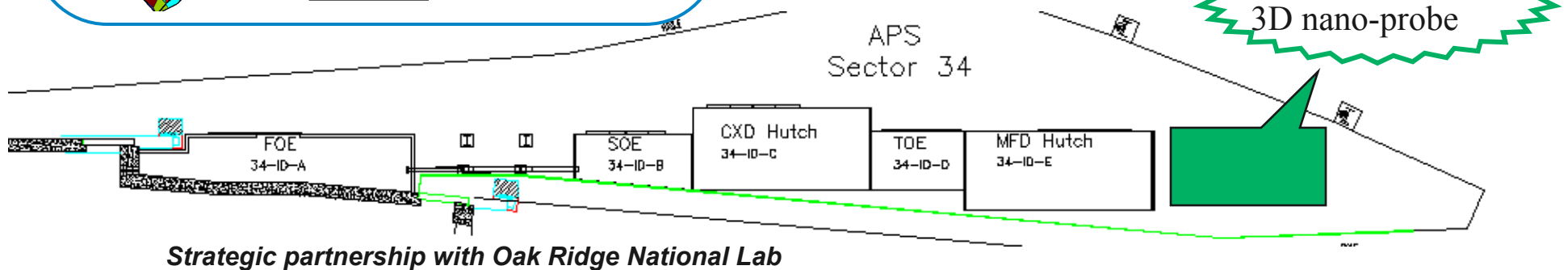
- Crystal structural probe with point-to-point spatial resolution (300 nm current & 50 nm planned) in three dimensions, unique in the world.

For fundamental studies of microstructure and evolution of engineering materials.



Planned upgrade:

- ❑ Build a dedicated polychromatic diffraction nano-probe (50 nm resolution) and hutch.
- ❑ Install triple canted undulators on 34-ID to allow for simultaneous and independent use of polychromatic 3D mesoscale and nanoscale probes and coherent diffraction activities (heavy oversubscription now with growing user community).



Bulk probes: Conclusion

- Proposed instruments match ‘Hierarchical Imaging’ & ‘Real Materials, Conditions, Time’ themes
 - Complementary techniques/instruments
 - Reduce severe over-subscription
 - Globally competitive
-
- High energies are unique APS capability
 - Focusing optics
 - Dedicated instruments
 - Combination of techniques
 - Sample environment
 - Area detectors crucial but no one size fits all
 - Software