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# *Sample Environment Instrumentation*

*APS Renewal: Instrumentation Open Forum (January 9, 2009)*

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# *Introduction: Sample environments*

*Complex sample environments are **implicit** in both of the overarching themes.*

- **Mastering hierarchical structures through x-ray imaging.**
  - "Through x-ray imaging, we can illuminate complex hierarchical structures from the molecular level to the macroscopic level, and study how they change in time and in response to stimuli."
- **Real materials in real conditions in real time.**
  - "Hard x-rays are also specially suited to the study of real materials, under realistic conditions and in real-time."

*Complex sample environments are **explicit** in the majority of Science Cases and in multiple Mid-Term Proposals and LOI's*

# *Examples…*

- Geological, Environmental, and Planetary Sciences
  - "… sophisticated sample chambers…maintaining controlled pressure, temperature, and fluid composition."
  - "progress is limited by the availability of materials in situ, in real time, and under a wide range of conditions."
- Interfacial Science: APS Midterm Science Case
  - "… extreme chemical environments is critical to numerous energy technologies ..."
  - Science Drivers: Interfacial reactivity in complex environments.
- Materials Science and Technology
  - "… non-equilibrium phenomena often occur in magnetic, optical and pressure conditions...tools are not available."
  - "… environmental control…require elevated temperatures, corrosive environments, applied loads, or a combination"
  - Key areas: In situ crystal growth, *Dynamic compression, Dynamics in applied fields.*
- Chemical, Atomic and Molecular Physics
  - "A unifying theme…is…understanding of the response of materials to applied fields
- Soft Matter
  - "… It is imperative that X-ray imaging instrumentation [facilitates] routine work at cryogenic temperatures."
- Engineering Applications and Applied Research
  - … Experiments require: "multiple extreme environments…high fields pressures, temperatures, and facilities for reactive or radioactive samples,…". And in situ "sample preparation and processing
  - "What 'design rules' govern soft materials in specific environments (temperature, radiation, chemical, etc).?"
  - "Can we identify and understand quantum critical points?" (requiring sub-K cryogenics.)
- Chemical Sciences and Engineering
  - "measurements under *in situ* condition such as elevated temperatures, pressures, and applied potentials, as well as in reactive and corrosive solutions."
  - "Advanced fuel cells depend critically on the development of new materials [performing] under extreme conditions of high voltage, temperature and corrosive electrochemical environments."

## *APS Renewal Plan for SE Instrumentation*

### *1) What have we done?*

- Starting from the mid-term proposals, we have tried to distil a small number of central SE “themes” representing *key enabling technologies* supporting the science cases.
- Organised these in some kind of priority
- Technical detail not presented here.

### *2) What should you do?*

- Give feedback on choice of themes and priority assessment (in discussion or afterwards)
- Assist with technical detail, where needed.

## *Main Sample Environment Themes*

In order of presentation (…not priority)

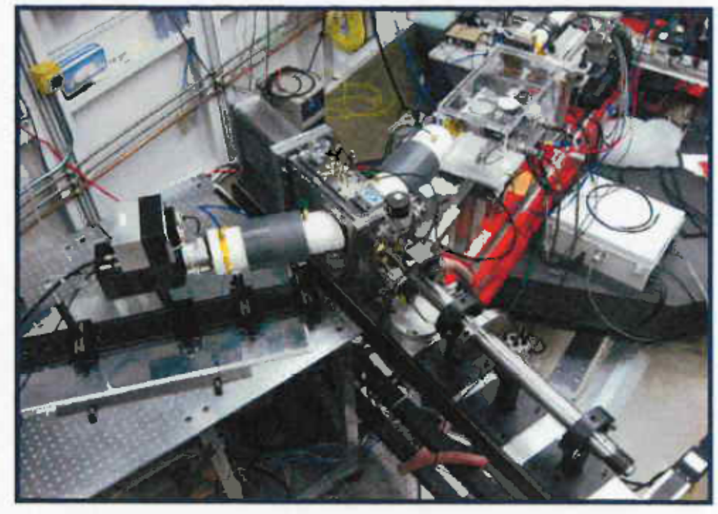
1. Real-time measurements under multiple extreme conditions
2. Nanobeam science at high pressure
3. Next generation diamond anvils and micro-sample preparation
4. Coherent sample environment development and support
5. X-ray science with high magnetic field
6. Extreme Chemical sample environments

(n.b. there is plenty of technical overlap between these themes!)

## *Real Time Measurements Under Multiple Extreme Conditions*

**The vast majority of Extreme Conditions experiments are static, the next frontier is real-time Dynamic measurements.**

Two basic kinds of dynamic measurements:
1) Periodic
2) Single-Event



**Periodic experiments** synchronise oscillating fields (H,E,P etc.) with pulsed x-ray source (SPX Facility)
Stroboscopic effect gives temporal resolution up 1ps through, say, structural transitions or chemical reactions.
Couple multiple probes essential:
1Å to >100 nm lengthscales,
spectroscopic measurements, etc.

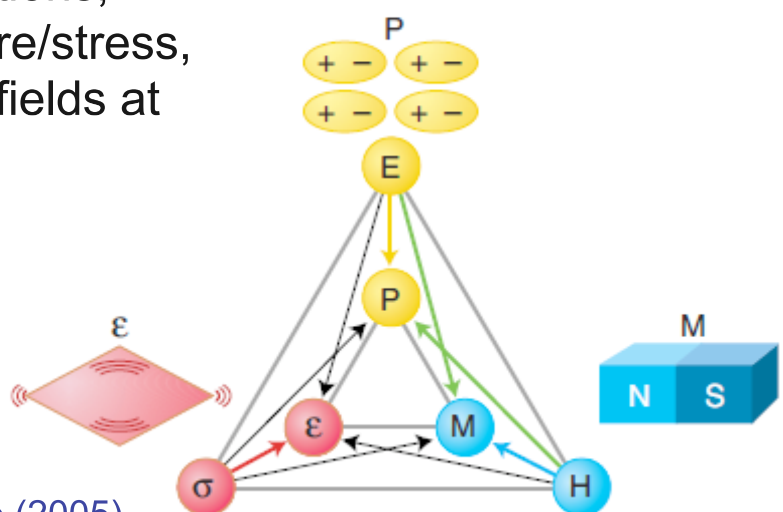
**Single-event experiments**, such as dynamic shock (DC-CAT LOI) place systems far from equilibrium. Measurements must be made in real-time (high flux, careful collimation, fast detectors).

# *Real Time Measurements Under Multiple Extreme Conditions*

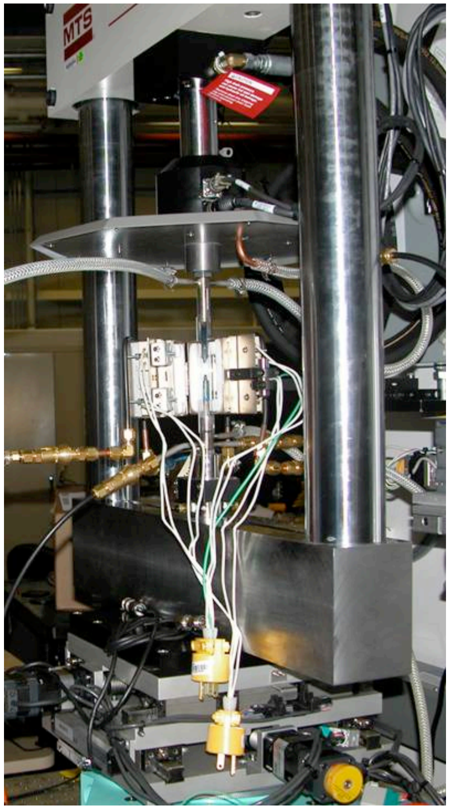
"Materials are central to every energy technology, and future energy technologies will place increasing demands on materials performance with respect to extremes in stress, strain, temperature, pressure, chemical reactivity, photon or radiation flux, and electric or magnetic fields." (DOE, BES Workshop Report (2007), ***Basic Research Needs for Materials under Extreme Environments.***

Understanding the interplay between the elastic, electronic and magnetic properties of materials

**Novel hard x-ray experiments** at multiple extreme conditions, combining high pressure/stress, high electric/magnetic-fields at extremely low (or high) temperature




Spaldin & Fiebig, Science (2005)



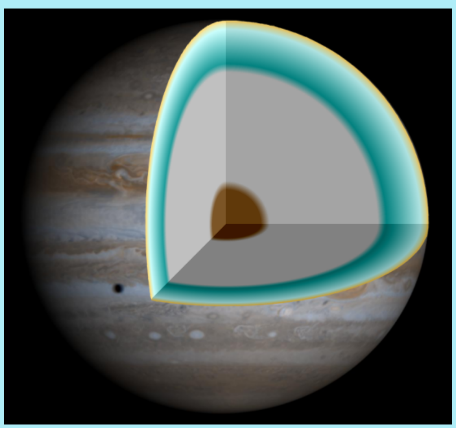
J. Almer

## *Nanobeam science at High Pressure*

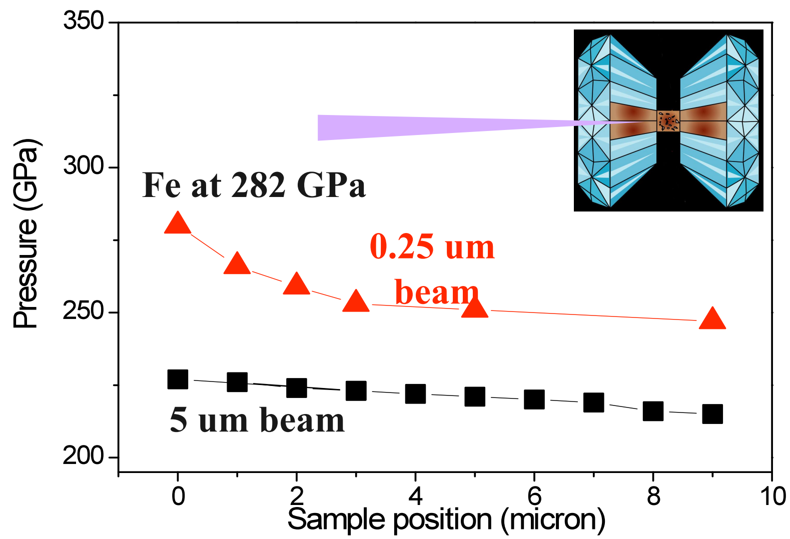
Typical cutting edge high pressure measurement use ~5um beams

**Reducing this by at least a factor of 10 to 200-500 nm will be transformative**

1) Prerequisite for static pressures in terapascal (TPa) regime (1000 GPa) and uniform T approaching 10,000 K
   - Planetary interiors (GEP science case)
   - Novel matter (CMMP science case)
2) High spatial resolution – nanoscale mineral inclusions, minimise P gradients
3) In-situ nanoscale imaging (e.g. by TXM) of samples under extreme conditions (entirely new field of research)
4) Diffraction from single nano-crystals.



Centre of Jupiter is ~ 4.5 TPa.
Core is surrounded by thick layer of liquid metallic hydrogen



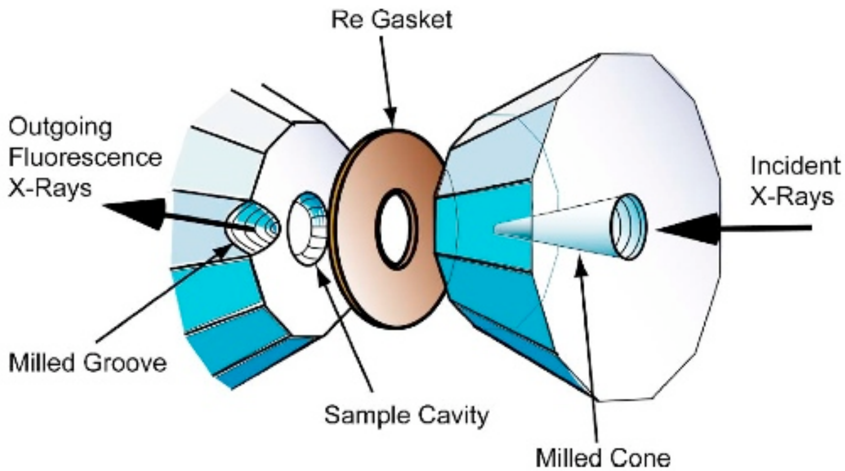
# Next Generation High P anvils and micro sample preparation – a laser ablation facility at the APS

## Next Gen Anvils

Anvils formed from hardest materials – limited to simple geometrical shapes

A laser ablation facility could:

- Optimise highest stable pressures
- Maximise sample volumes
- Minimise pathlengths through anvils (e.g. for fluorescence measurements, or Background/flux limited high Q diffraction studies) e.g. "holey" and "groovy" anvils (W. Bassett)




[Mayanovic *et al* Rev Sci Inst. 78, 053904 (2007) ]

## Micro Sample Preparation

As samples tend towards 1 um and smaller, 'by hand' sample preparation no longer feasible

- Precision cutting single crystals to match gasket hole in DAC's
- Precision cutting gasket hole to match.
- Improvement in surface finish (of sample) minimise contaminant scattering

# *Coherent sample environment development and support (the "Socialist" model)*

## Centralised support for specialised sample environment within XOR (and CATS?)

Coherent provision/development of multiple SE
(e.g. coupling P and T, or P and H)

Key focus would be *portable* systems:
Single beamline can't keep up with cutting-
Edge synchrotron developments.

Shift base level responsibilities
away from instrument scientists (link to ESAF?)

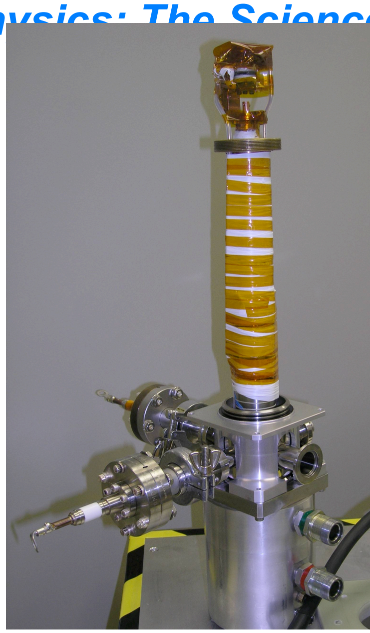
Cutting edge technical
developments (e.g.
P cells, HT levitation system)
exploit proximity of synchrotron

Multiple tiers of support –
**e.g. Cryostats**

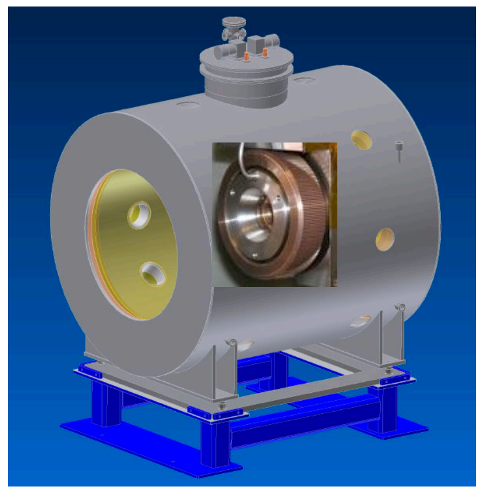
1. Base level technical: Installation on beamline, maintaining cryogens, minor repairs
2. High Level technical: Managing finite resources, complex technical Troubleshooting /Repairs,
3. Scientific/Engineering level: Science driven development of next-gen cryostats

## *X-ray science with High Magnetic Fields*

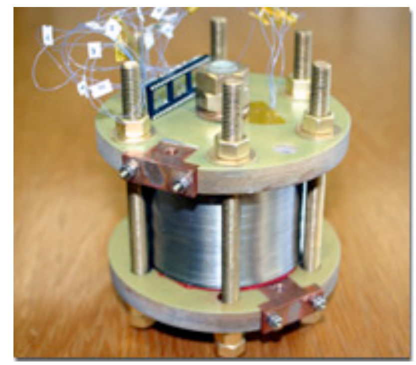
- ➔ **"New instrumentations for studying the neutron- and <u>x-ray-scattering properties of materials in high magnetic fields should be developed in the United States</u>."  (National Research Council (2005), *Opportunities in high magnetic-field science.***
- ➔ **"DOE should exploit fully the existing third-generation synchrotron; … This also means proceeding with <u>the high-magnetic-field sample environment for APS</u>."  (National Academies Press (2007), *Condensed-Matter and Materials Physics: The Science of the World Around Us.***



**Pulsed magnetic-field up to 60T with 30 ms width for time-resolved exp**

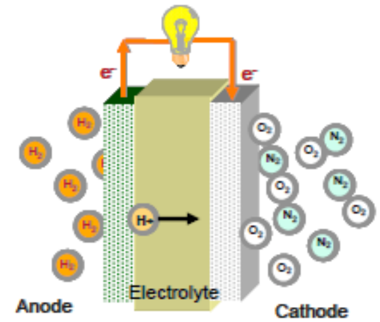


**Large bore magnet (10T) for combining HP, LT/HT and other conditions**

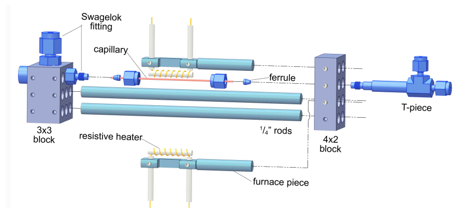


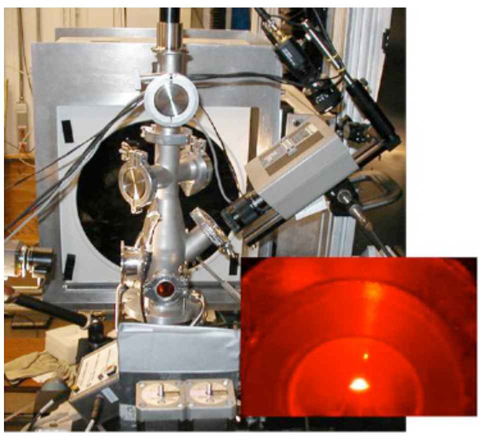
**Superconducting magnet (26.8T, NHMFL recent record) for x-ray imaging, scattering**

# In-situ study of catalytic processes, corrosive reactions, fuel cell and battery performance, high -temperature material synthesis and crystal growth… (controllable T, P, chemical …)



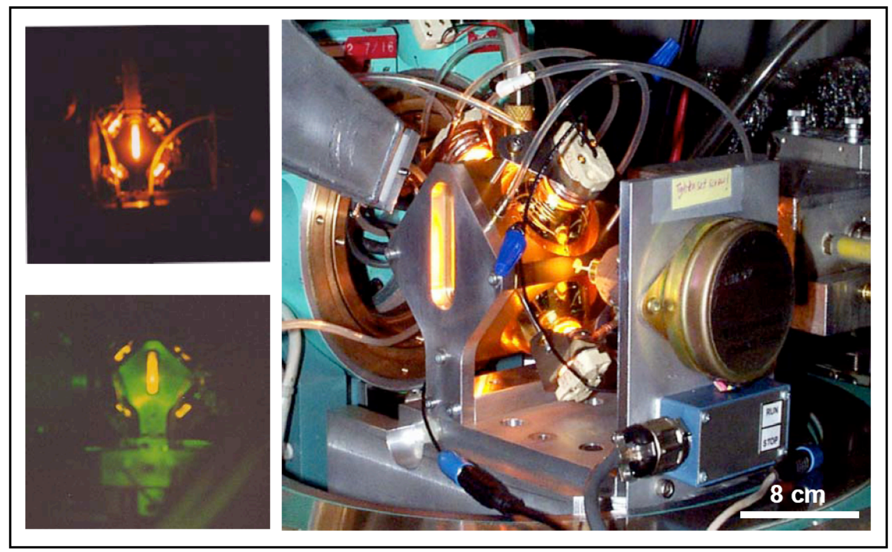

*Schematic of an $H_2$ fuel cell. (Image courtesy D. Myers, ANL)*



Greaves et al., Science (2008)

**Laser-heating lavitation for higher T (>3000 C) with various gases**




Trudy Kriven, UIUC

## *Summary*

Multiple extreme sample environment developments are essential and critical for the two overarching themes and for most of the proposals for future x-ray science and beamline upgrade.

1. Real-time measurements under multiple extreme conditions
2. Nanobeam science at High pressure
3. X-ray science with high magnetic field
4. Coherent sample environment development and support
5. Next generation diamond anvils and micro-sample preparation
6. Extreme Chemical sample environments

Please email suggestions to:
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