Structural studies summary

HEX workshop

APS 9-10th August 2004

Workshop Objectives

1. **Explore new and emerging scientific and technological areas defined in the scope of this workshop.**

2. **Broaden the community interaction by including researchers from various methodologies (e.g., EM, neutron scattering, etc.)**

3. **Identify new scientific proposals/programs specific to the emerging areas which can benefit from the use of High–Energy X-rays that the participants will bring to the APS during next 5 to 10 years. Also evaluate the capital and operational requirements for these proposals/programs.**

4. **In addition to available beamline capabilities at the APS, identify future needs to support research in this area of science and technology.**

5. **Address the need and support for theoretical work to strengthen the experimental research.**

6. **Prepare a summary document for the archival literature to serve as a roadmap for the future applications of high-energy x-rays and suggest the role of the Advanced Photon Source towards this objective.**

Points to discuss

- What energy range do we want?
  - > 50 keV??
- How should the various APS beam lines specialize to most efficiently meet the needs of the users?
  - General PDF/powder beam line, 2D, point det. and microfocus
    - Anomalous scattering/PDF with high resolution optics as an option
  - White light capability
  - High field bend magnet for HE tomography, reaction studies that are not flux limited and suffer from sampling statistics problems
- How do we promote HEX to a wider community?
- Others?

Outline

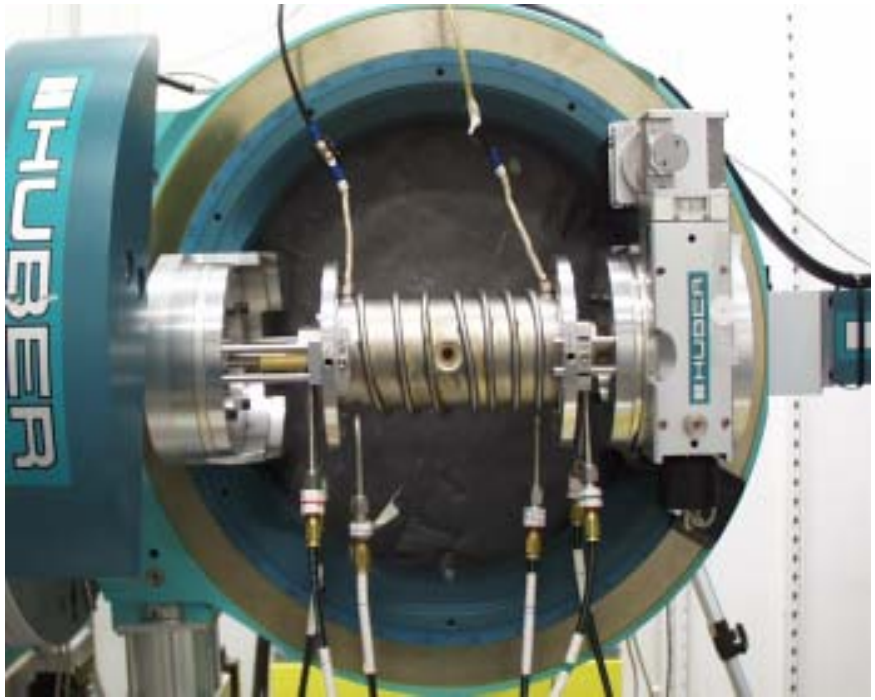
- Motivation for HE X-rays
- Examples
- Future science
- Future needs

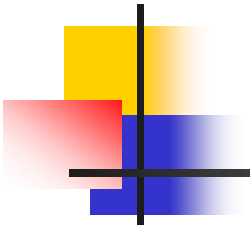
Motivation

- HE x-rays needed to penetrate sample environment and sample
  - Cells for catalysis
  - Stable and uniform furnaces
  - Pressure cells
- HE x-rays give data with smaller systematic error content facilitating weak signal experiments
- Access to high Q for PDF studies and simultaneous PDF/Rietveld work
- Rapid and effective collection of single crystal diffuse scattering data
- Access to high energy absorption edges for resonant scattering work

Sample environment

- Fast data acquisition
- Good S:N
- Furnace Design
  - Eulerian Cradle
  - Very low lateral and radial thermal gradient (~ ±2°C over 4 mm distance)
  - ~1800 K
  - Inert to Oxidizing
  - Sample rotation for improved powder averaging
  - Sample Containment
  - Uniform Heating

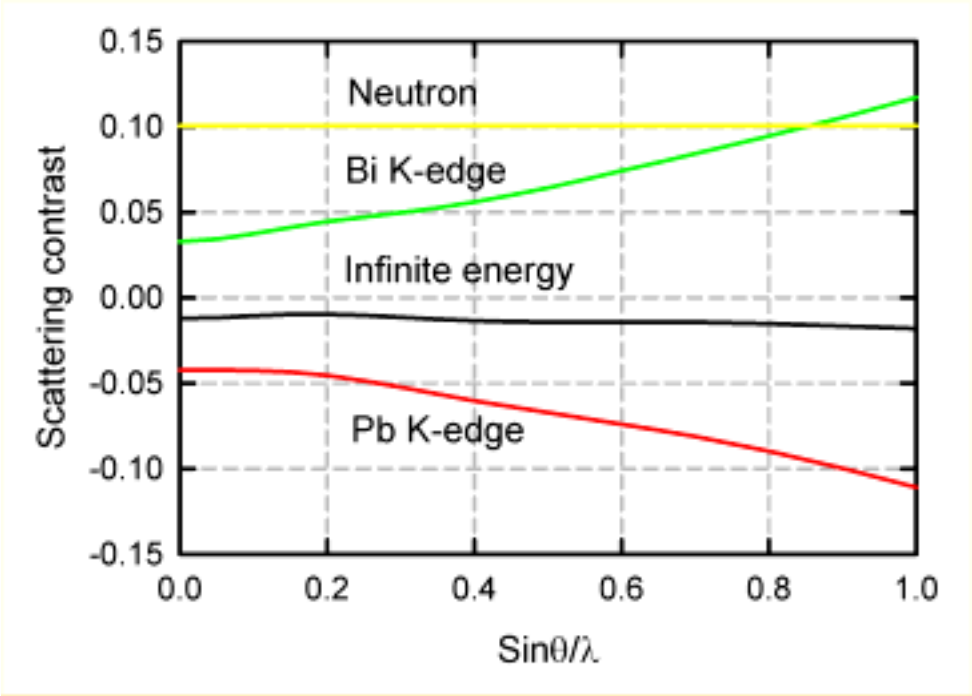


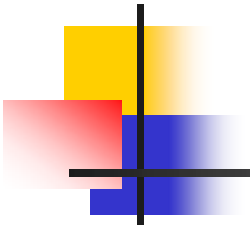


- In-situ studies will be a continuing area of interest
  - Better time resolution
    - Current experiments are not flux limited
      - Could be done on BM?
    - BETTER DETECTORS NEEDED
      - With better detectors might be flux limited
  - More extreme environments

Contrast in Pb/Bi studies

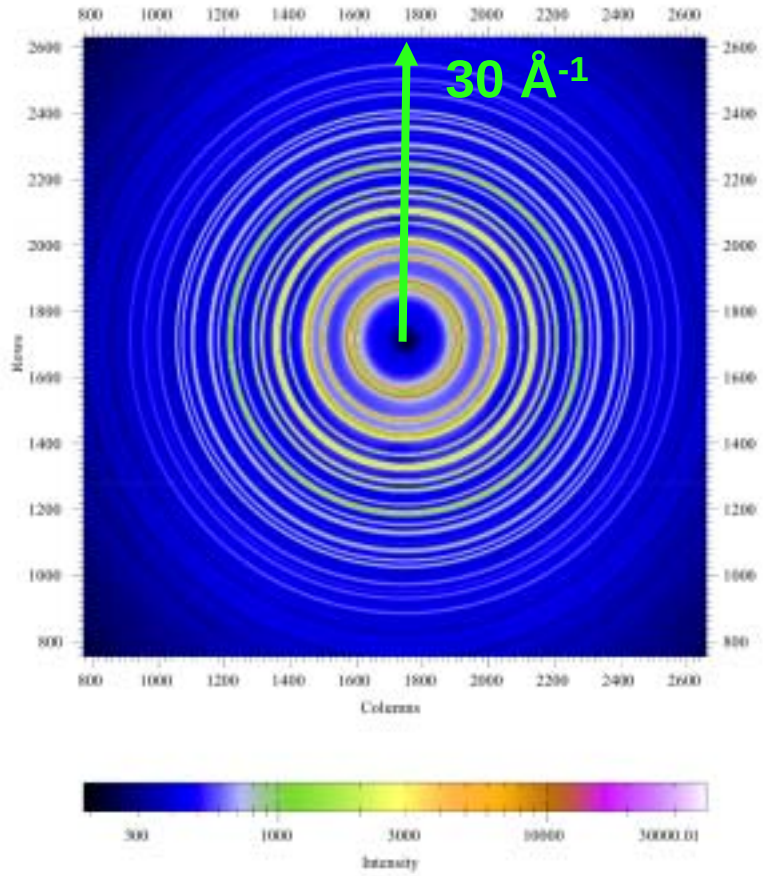
- The achievable scattering contrast in Pb/Bi K-edge studies is quite low
  - Contrast comparable to the neutron measurement, but data available at multiple contrast levels





- Resonant scattering at high energy is potentially useful
  - PDF and crystal structure studies
  - ASAXS
    - Needs very stable small band pass optics with energy scanning capabilities

Area Detectors and the PDF method



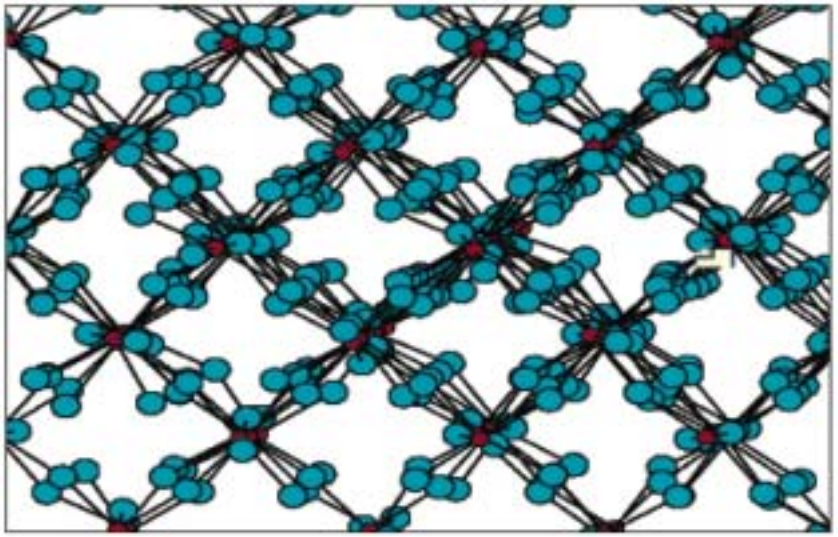
Why use *Area Detectors*?

- With an area detector we can collect all data simultaneously.
- High energy X-rays are a necessity to probe large values of Q over small scattering angles (small detector areas).
- Counting statistics; averaging over a large solid angle.
- A conventional measurement takes ~8 hours… we need better time resolution for in-situ studies

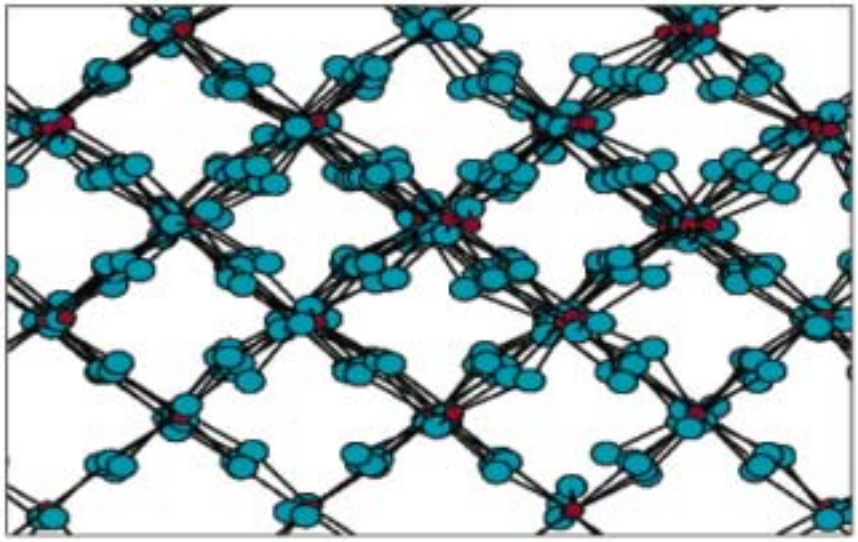
Chupas, Qiu, Hanson, Lee, Grey, Billinge ***Journal of Applied Crystallography*** **36 (2003) 1342.**

Snapshots from the Molecular Dynamics Simulations

- Molecular Dynamics simulations (Santanu Chaudhuri, Mark Wilson, Paul Madden)
- MD is ideally suited for comparison with total scattering methods.



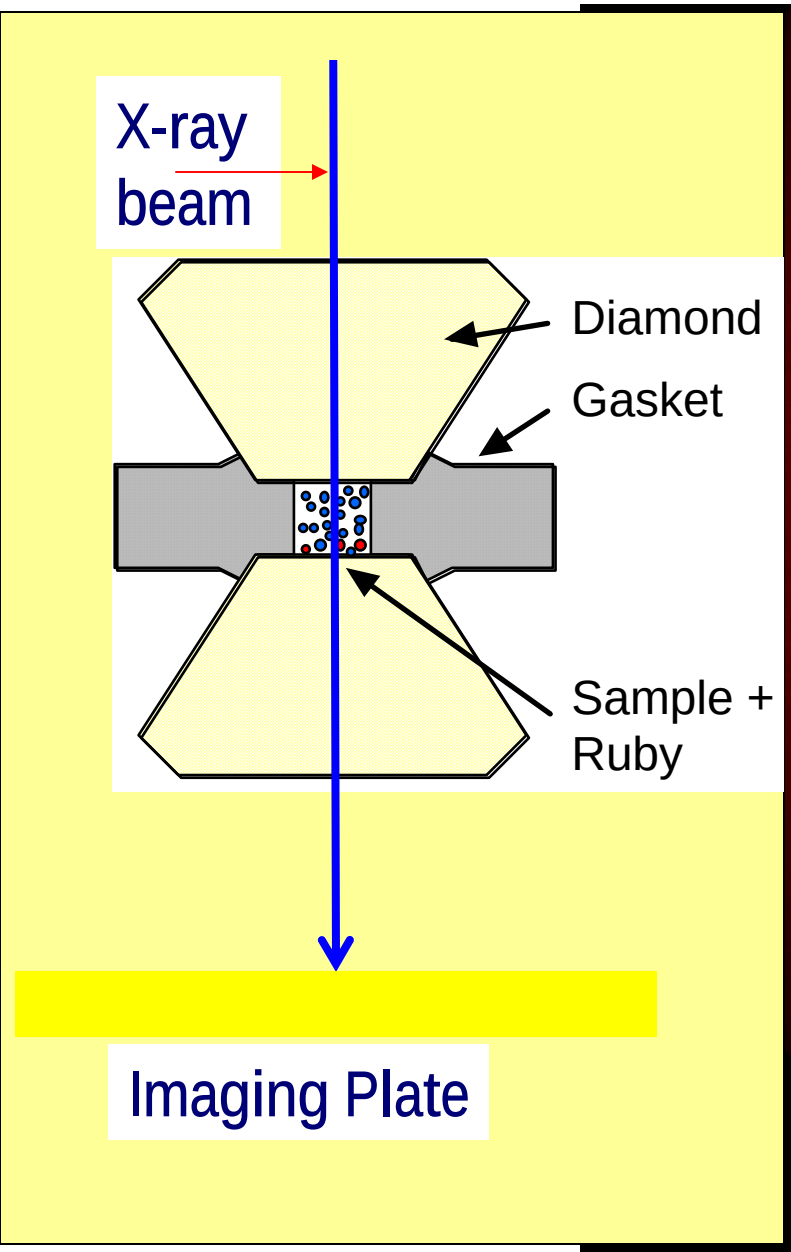
500 K (Rhom)

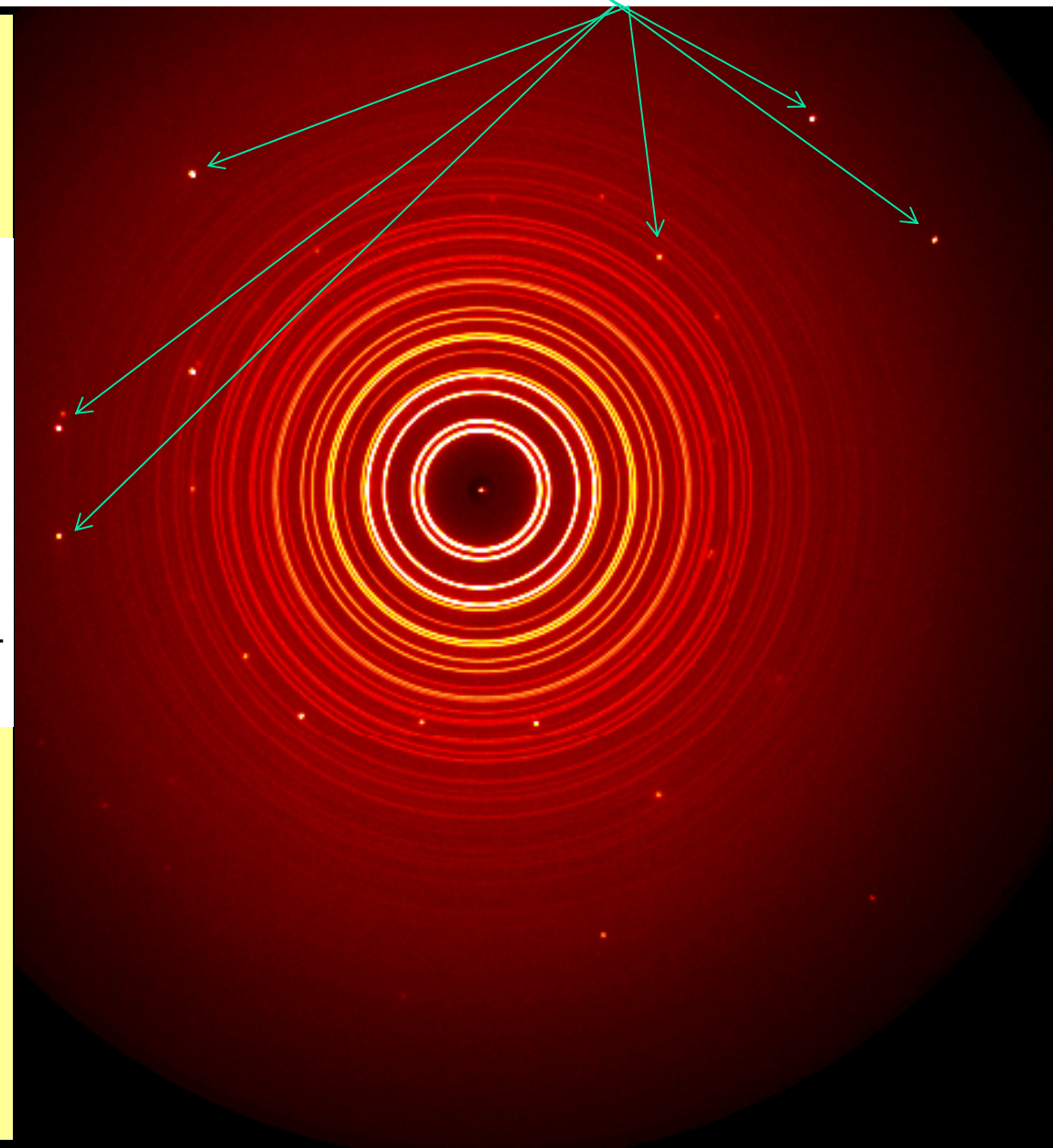


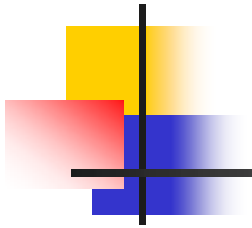
900 K Cubic

Chaudhuri, Chupas, Wilson, Madden, Grey *Journal of Physical Chemistry* 108 (2004) 3437.

PDF measurements in a DAC on nanocrystalline Au (1-ID-C, E = 80 keV, λ = 0.10557 Å). Image plate data: few diamond spots







- Support from simulation very important
- Data processing/analysis software
- Need better availability of optics for focusing of HE x-rays to 20 μm to go through diamond cells etc

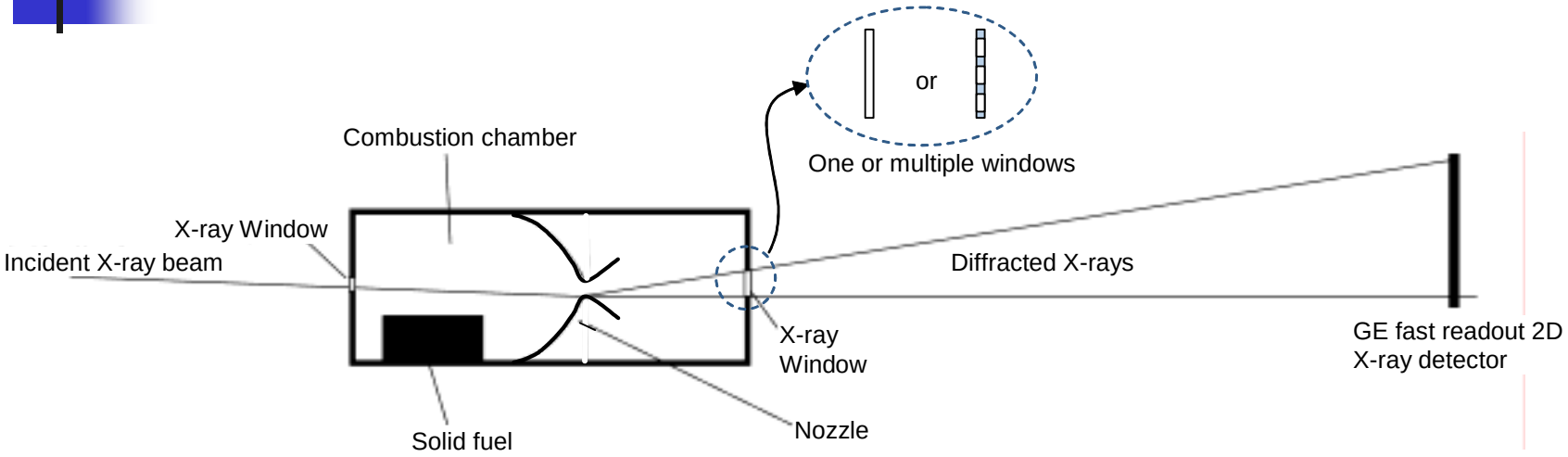
Diffuse scattering

- Single crystal diffuse scattering provides vital information on complex disorder, and can make important contributions to a large number of scientific fields.
- Advances in instrumentation and analysis tools will open the technique up to a much larger range of scientific community.
  - Detector technology must cope with a very wide dynamic range
  - Computational analysis must cope with huge volumes of data
- High-Energy X-rays are a vital part of this development because
  - large volumes of reciprocal space can be measured efficiently.
  - absorption and background corrections are minimized.
- This should be pursued in parallel with complementary developments in neutron instrumentation.

Future experiments

- More in-situ studies often combining PDF and Rietveld
  - But with better time resolution
  - Optics for small beams should be widely available for high P work
  - Currently detector limited
    - But future studies may be flux limited as well
- Single Xtal diffuse scattering
- Perhaps more high energy resonant scattering
  - Need optimized optics

Real time study of solid fuel rocket motor nozzle erosion



- Require very high fluxes of high energy x-rays
  - must penetrate two windows that are subjected to high pressures and temperatures
  - “sample environment” only provides a small angular opening
- Need very rapid read out detectors

Future needs

- Better detectors
  - Dynamic range, low noise, and readout time
- Optics for focusing, optimized band pass etc.
- Specialized beam lines for different experiments
  - PDF/powder and resonant including APDF, white beam
- Software for data reduction and analysis
- Coupling between simulations and experiments