Combined levitation and neutron diffraction to investigate liquids and solids at high temperatures

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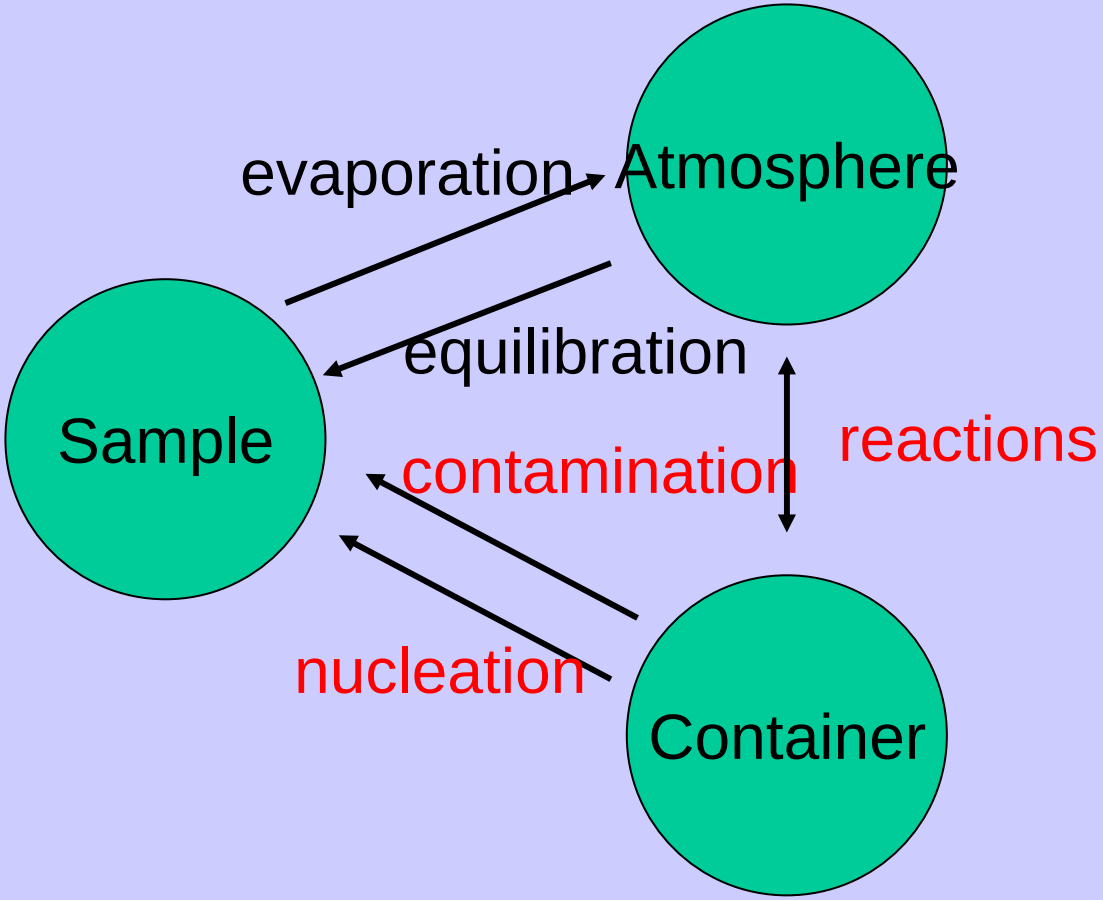
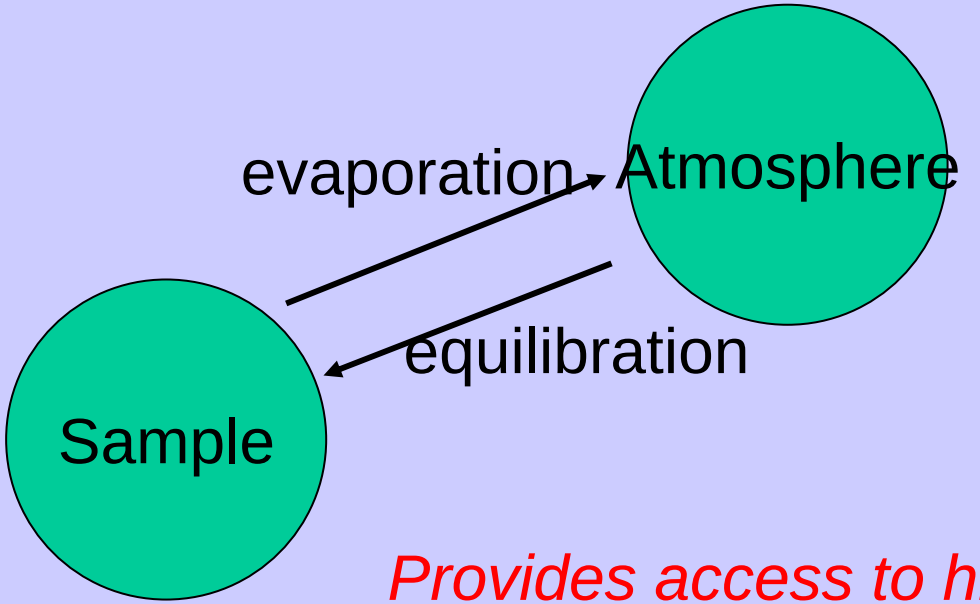
Outline

- Containerless processing/levitation at high temperatures
- Instrument design and capabilities
- Experimental results
- Outlook and plans

Containerless processing

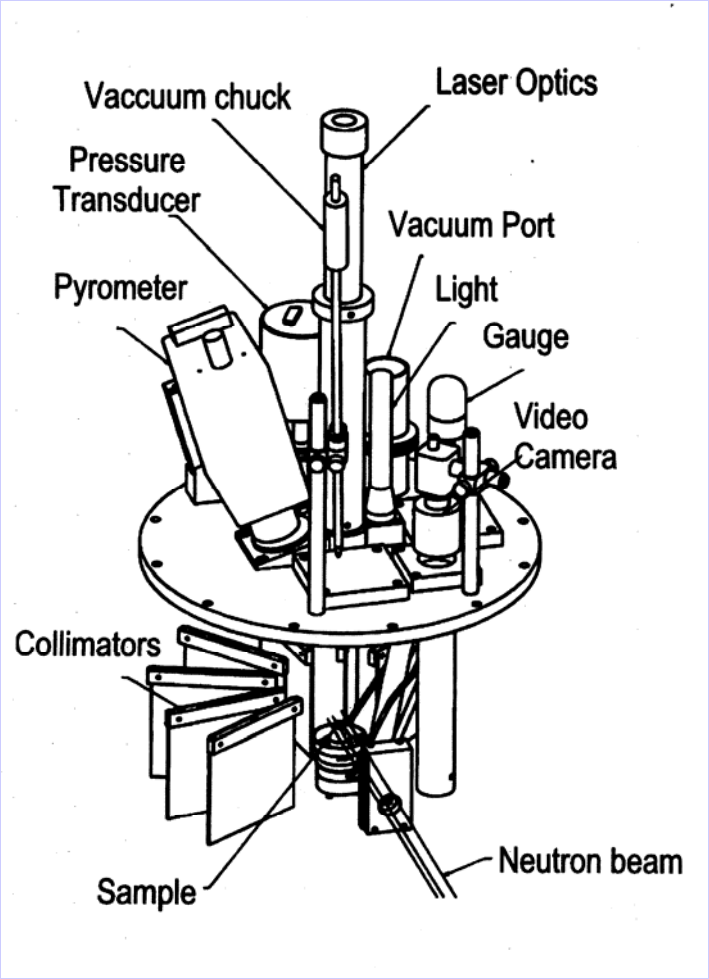


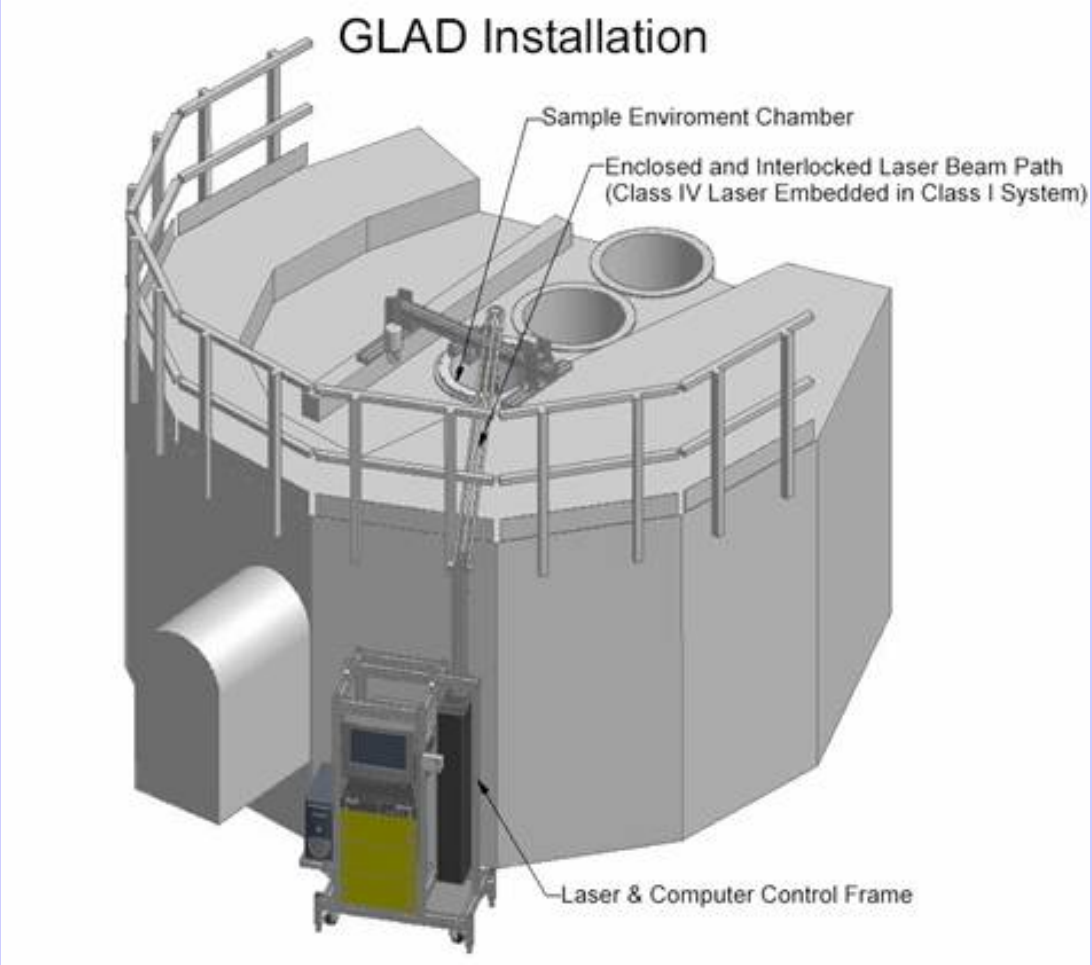
Containerless processing



Provides access to high purity, non-equilibrium liquids and pristine liquid surfaces and maintains purity of solids.

Diagram of levitator

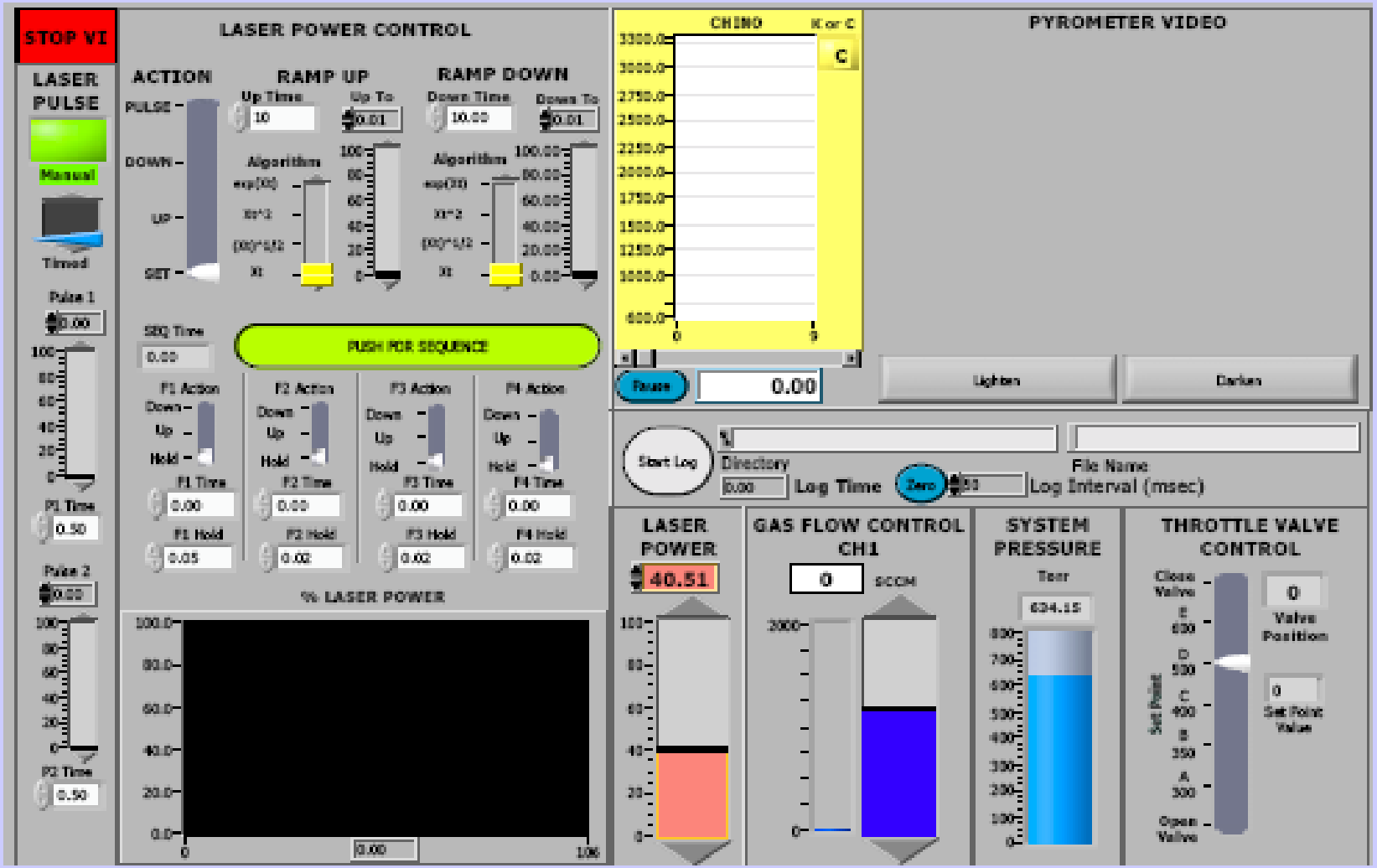




J. Neutron Res., **11**, 113-21 (2003).

Photographs of beamline levitator

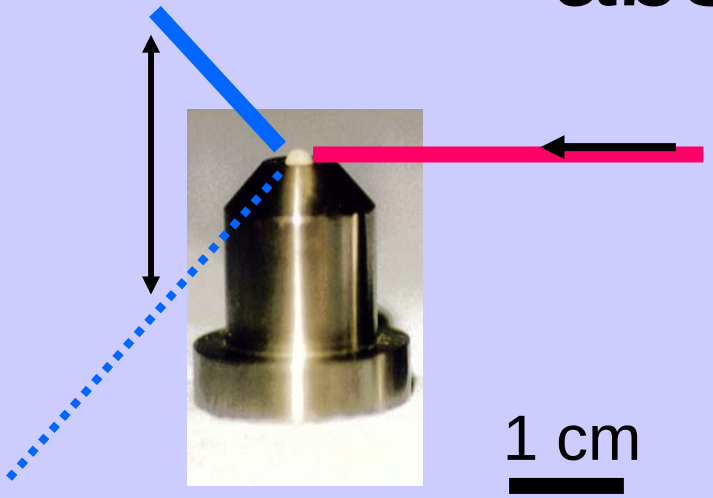
Labview user interface

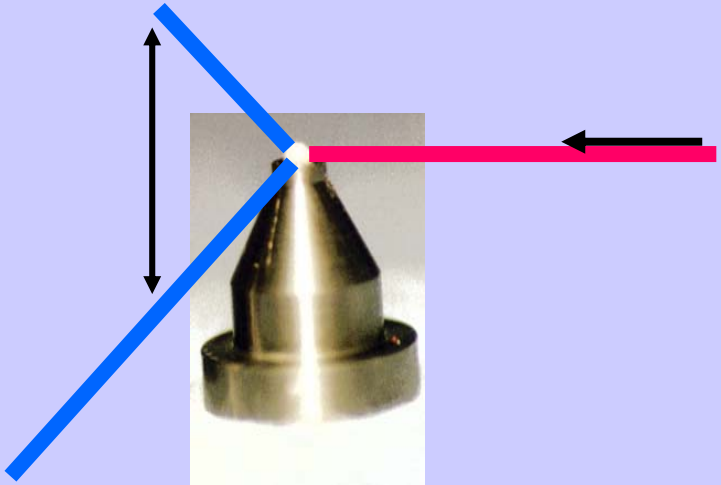


Levitation nozzles

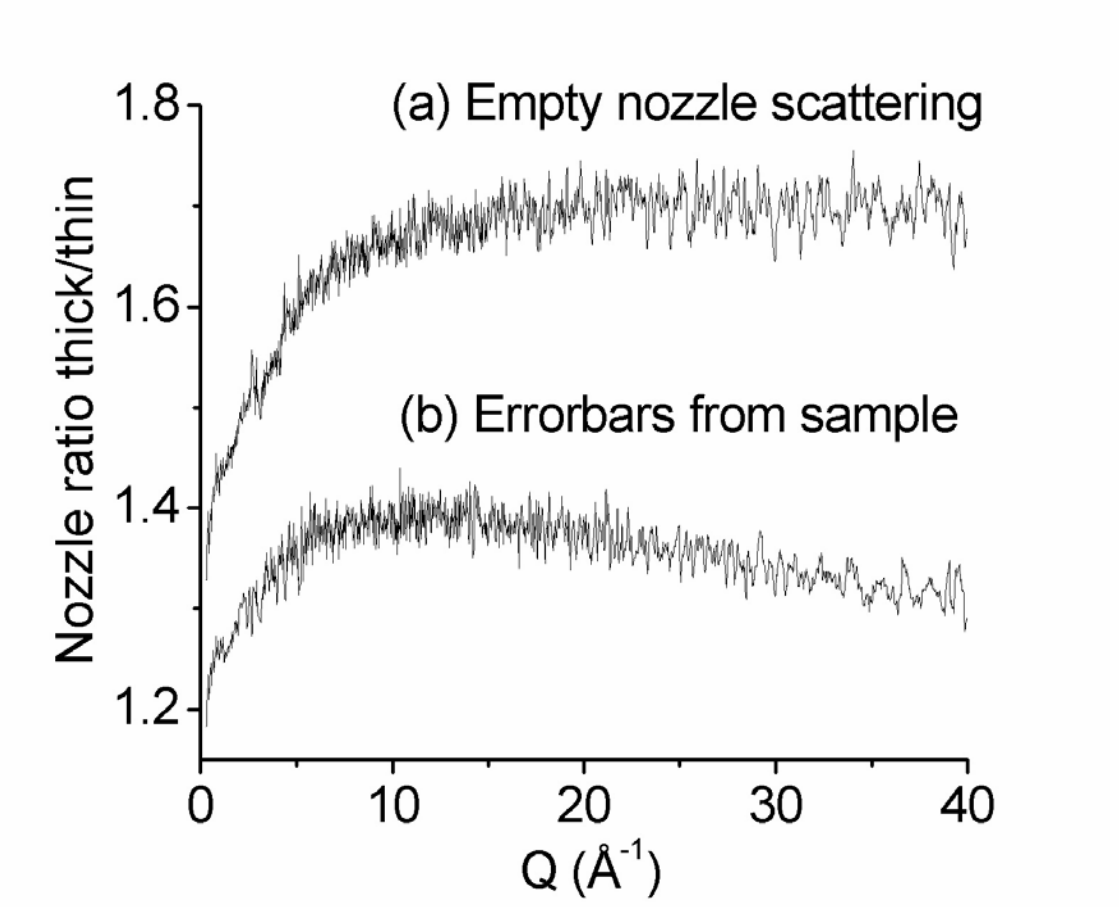
Vanadium levitation nozzles, side view of levitated molten drop, top view into nozzle

Effects of nozzle on absorption





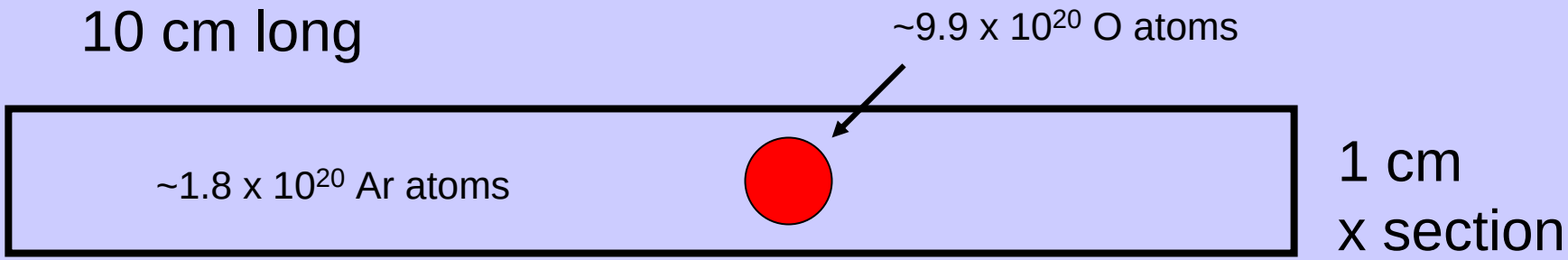
(a) Ratio of the empty nozzle scattering for the thick/thin walled vanadium nozzle. (b) Ratio of errorbars from the sample scattering only.



Instrument specifications/parameters

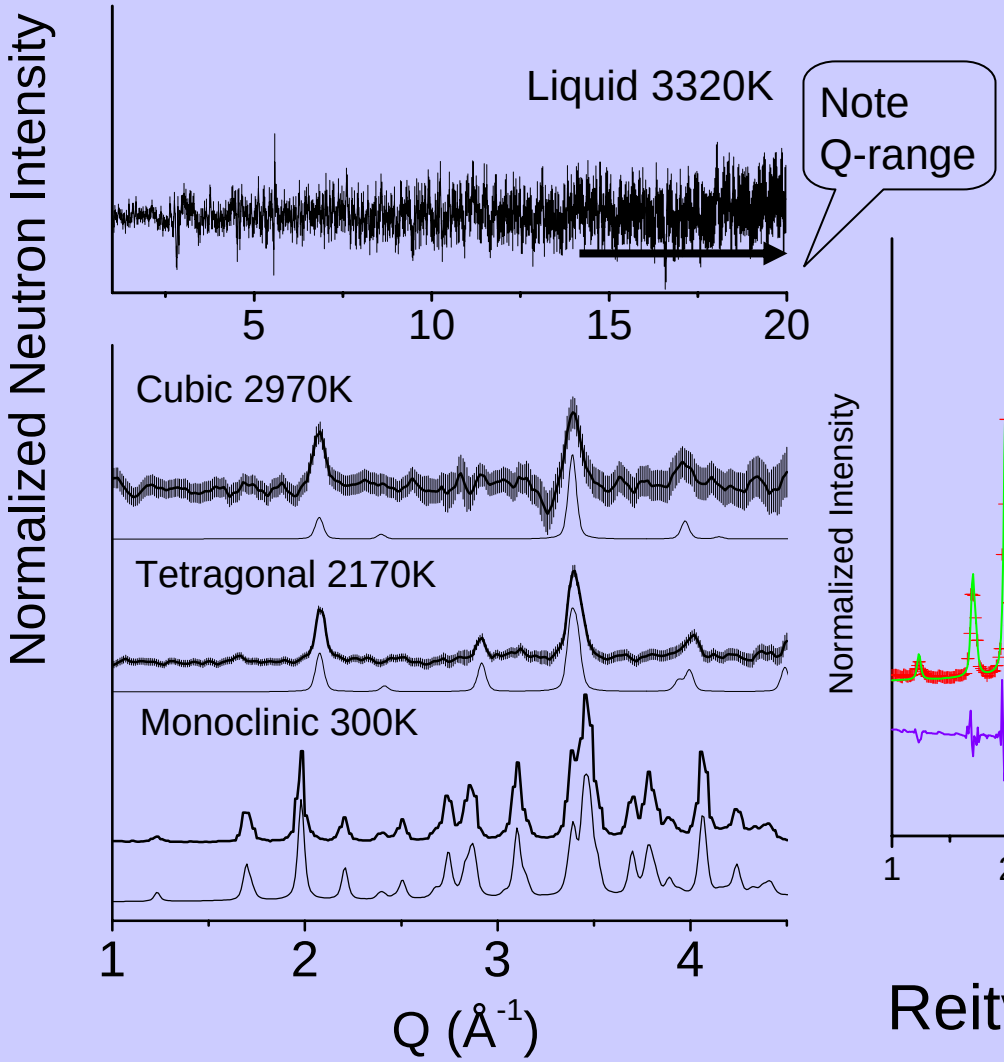
- Ability to investigate liquids and hot solids under non-equilibrium conditions and at high purity.
- Ability to work in a controlled atmosphere O_2, N_2, redox, reactive, pressure 0.5-5 bar.
- Adequate heating power.
- Class I laser system to minimize user training requirements.
- Easy installation/deinstallation at beamlines.
- Complementary bench top facility for sample characterization/glass synthesis.
- Ability to obtain “rough”G(r) quickly in order to plan ongoing experiments.
- *Fast measurements (<1 hour) to enable studies of T-dependence and transient effects in non-equilibrium liquids.*

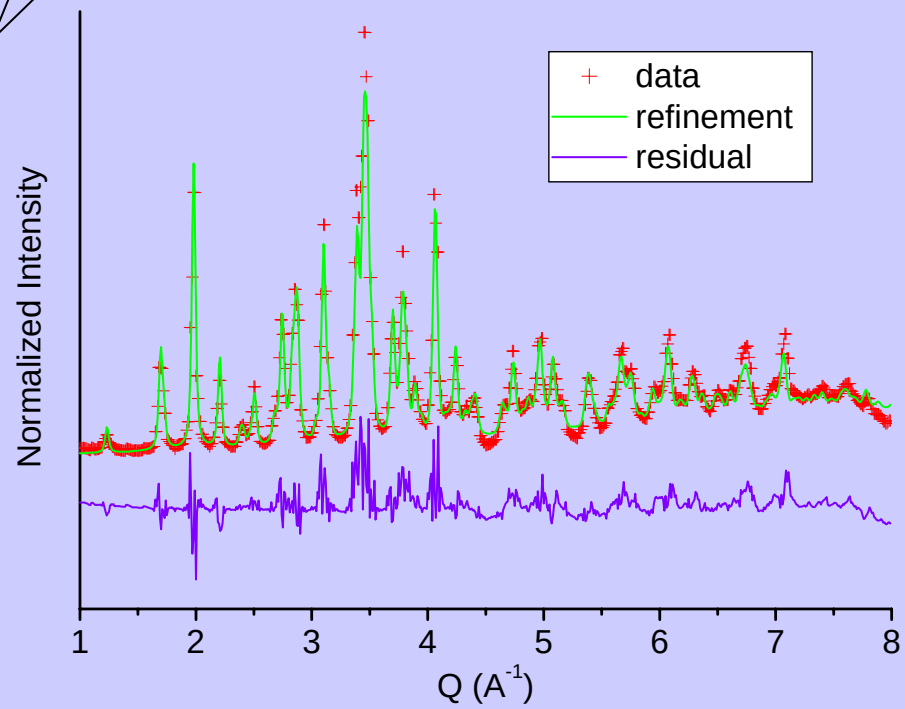
Effect of scattering by the ambient gas



The coherent scattering cross sections of oxygen and argon are 4.232 and 0.683 barn respectively. Scattering contribution by argon would be about 2% of that from oxygen.

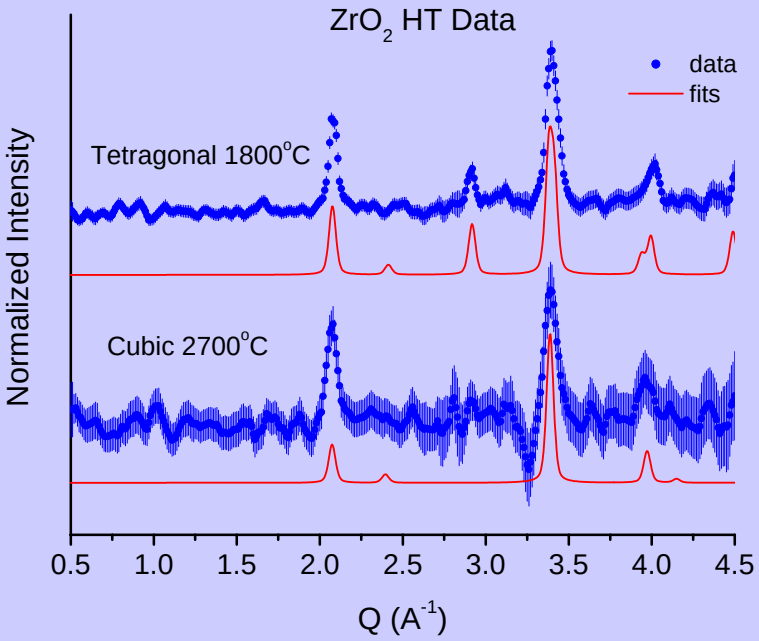
Zirconium dioxide

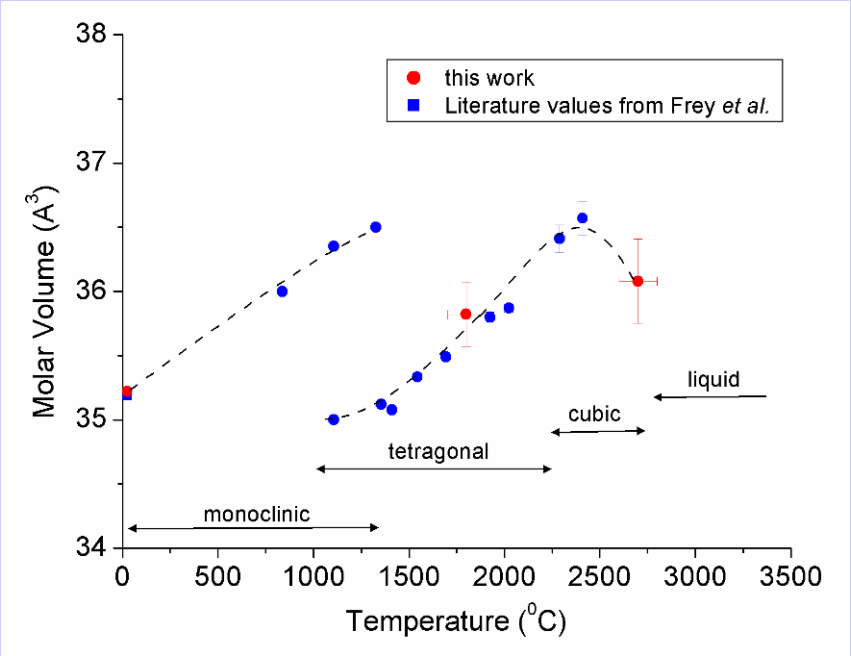




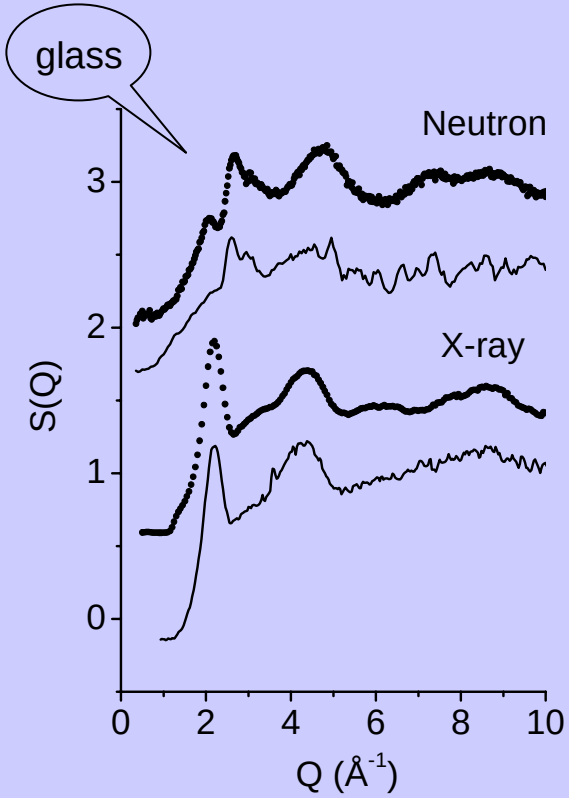
Reitveld refinement (300 K data)

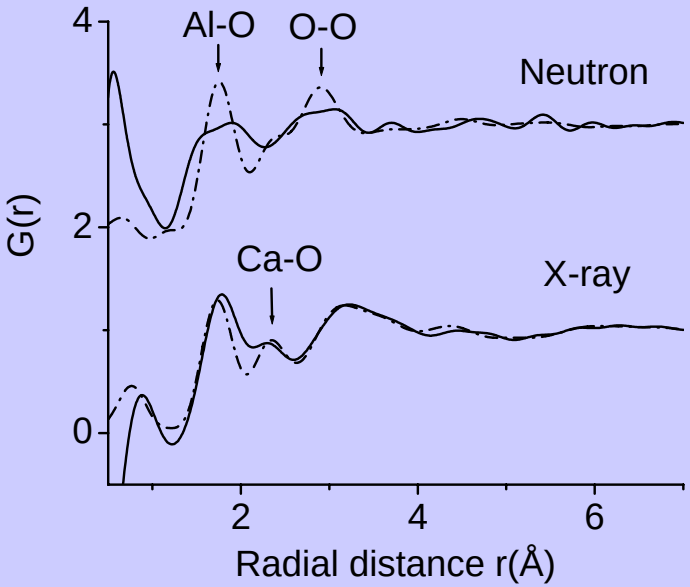
Zirconium dioxide analysis of high temperature data





Molten Ca and Sr aluminates

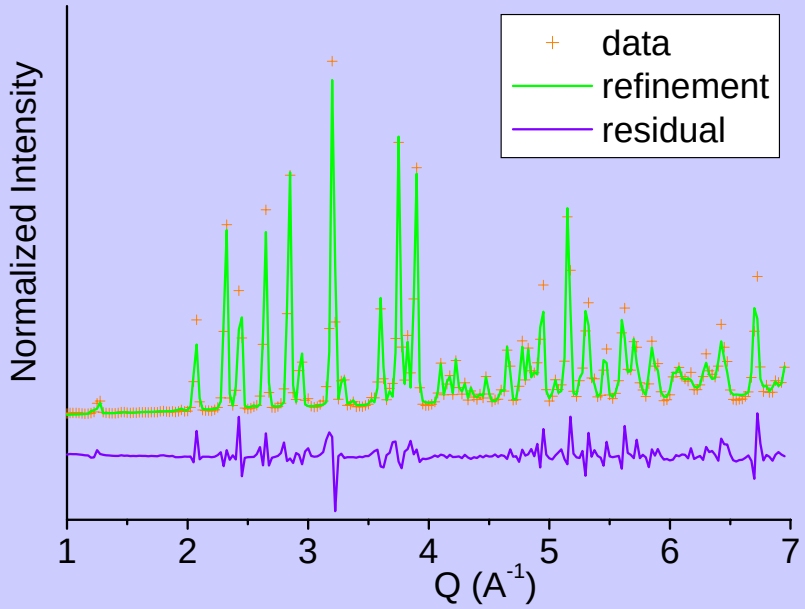


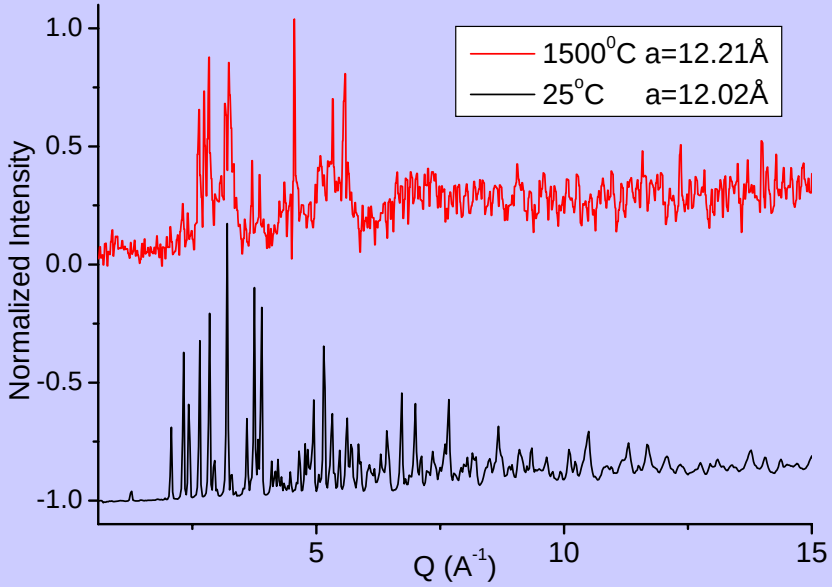


Al is 4 and higher-CN in liquid, Sr is 8-CN, Ca is 6+-CN (based on O-O separation)

Preliminary neutron results on YAG

25°C

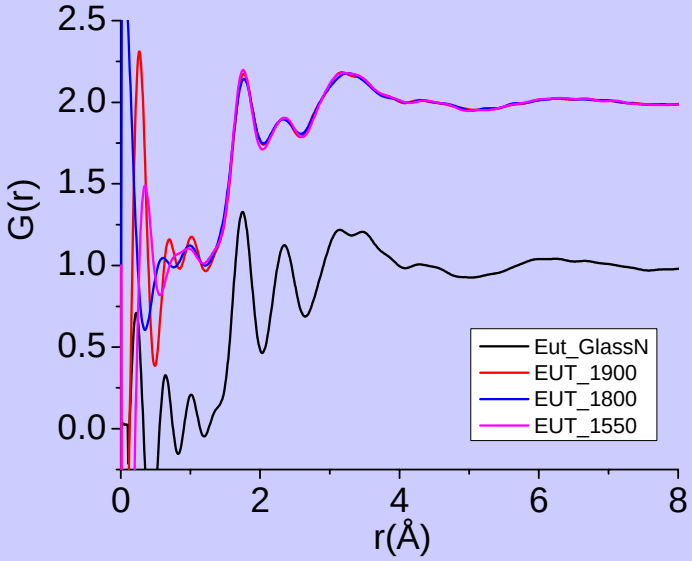




Thermal expansion of solid - 10 x 10^{-6}/K

No evidence of $Y_3Al_5O_{12} \rightarrow 3\ YAlO_3 + Al_2O_3$ decomposition

Recent X-ray work at APS



Trends: Ca CN increases in liquid *vs*. glass – MD suggests Al triclusters are forming that “free” oxygen to increase Ca-O coordination

Plans

- Complete development and integration of a beamline instrument
- Further experiments on YAG at GLAD
- Install a system at SNS (NOMAD beamline ?)
- Complementary HEX instrument at APS (in progress)

Acknowledgments

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