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... for a brighter future

Levitation: engineering + science at 11 ID-C

U.S. Department of Energy

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Office of Science U.S. DEPARTMENT OF ENERGY

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

Rick Weber, STA at 11 ID-C, XSD.

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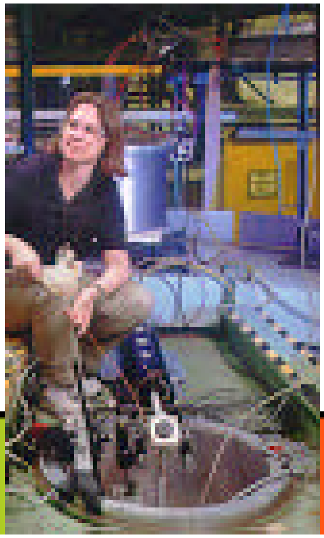
Means of Support

- Chris Benmore, Kevin Beyer, XSD 11 ID-C
- Joerg Neuefeind, SNS
- Martin Wilding, U. Aberystwyth, John Parise, Stony Brook.
- Internal funding: IPNS, Art Shultz, XSD, Brian Toby and Pete Chupas
- External funding: ORNL

Joan Siewenie,
2001 GLAD tests



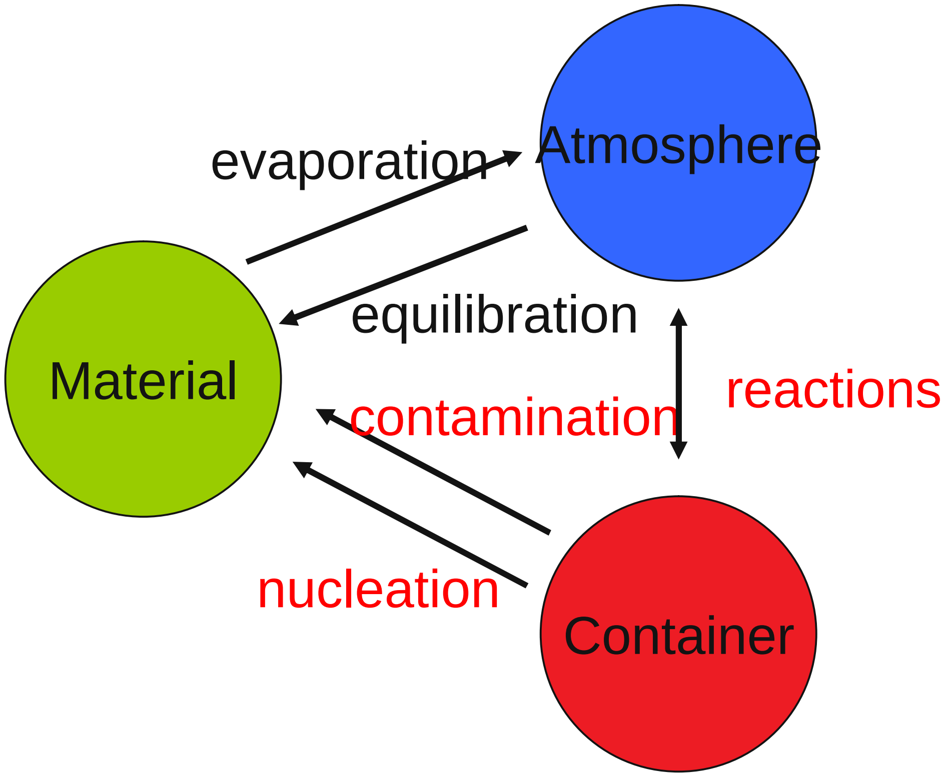
Outline

- Why levitate things?
- Issues specific to levitation experiments
- Methods
- Some applications
- Summary

Containerless processing/levitation

Tendency for chemical reactions increases exponentially with T

Using containerless methods accesses:
- *high purity*
- *non-equilibrium liquids*
- *pristine liquid surfaces*
- *eliminates sample "holder"*



$$F = -mg$$

Issues

- You can't touch the sample with anything
- Non-contact measurements
    - *Beams*
    - *Optical probes*
    - *Spectroscopy*
  - Non-contact temperature control
    - *Lasers/beams – surface heating*
    - *EM – bulk heating*
    - *Microwave – bulk heating*

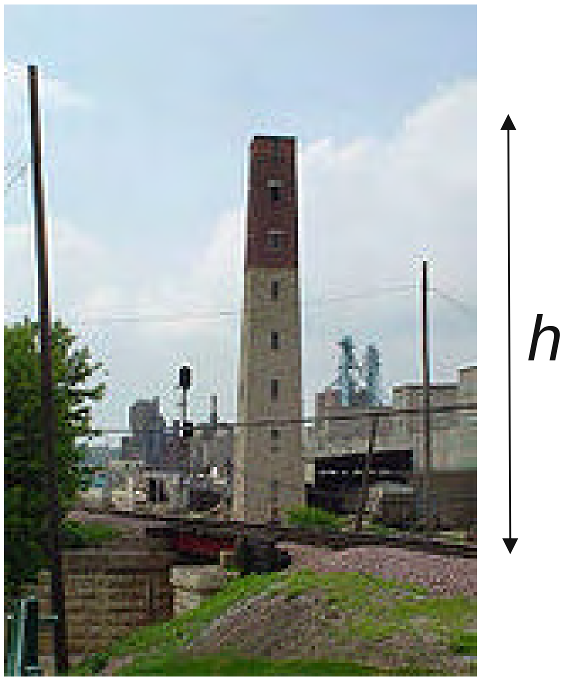
- Sample sizes are limited

- Compositions and materials may be specific to the method

Scientific applications of levitation (containerless techniques)

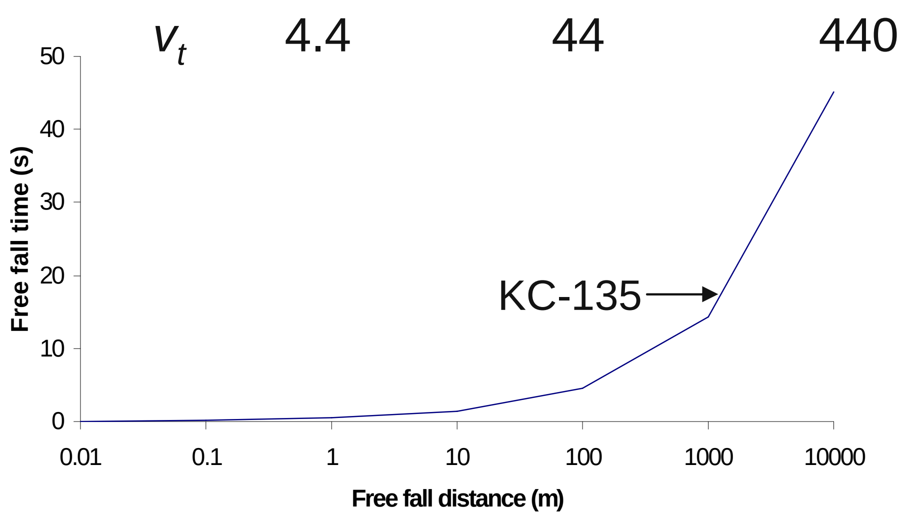
- ***Avoid contamination** of materials at extreme temperatures*
- ***Control melt chemistry** under extreme conditions*
- *Investigate surface reactions with reactive environments*
- *Avoid nucleation to access deeply **supercooled** and non-equilibrium liquids and glasses*
- *Expose pristine **liquid surfaces***
- *Scientific and technological interest derives from:*
  - ***Geo-materials** community: planetary evolution, carbon sequestration, waste storage*
  - ***Fundamental condensed matter physics**: glass transition, fragile liquids, nucleation and ordering in supercooled liquids*
  - ***Bio-materials** community: supersaturated solutions, bio-active phases*
  - ***Energy materials**: materials at extreme temperatures in chemically active gases*
  - *Measurement of **thermophysical properties** of hot liquids: surface tension, viscosity, heat capacity*

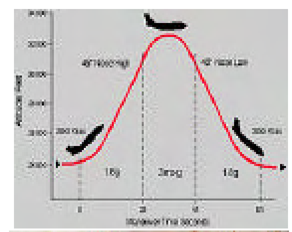
“Transient” methods: Shot tower, William Watts, England, 1782



A shot tower in Dubuque, IA

Free fall time: $$t = \sqrt{\frac{2h}{g}}$$

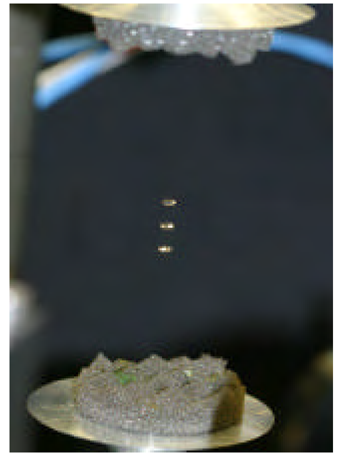




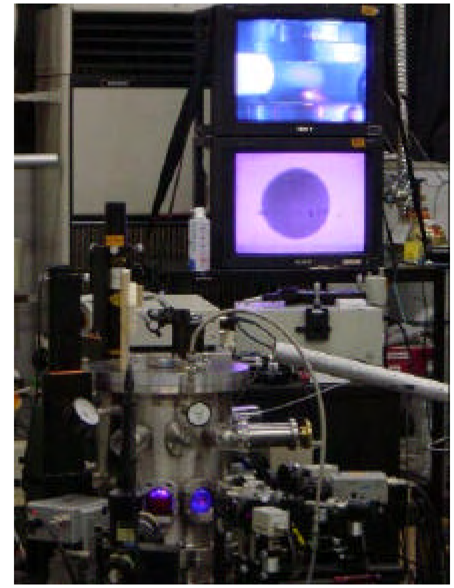
Some methods for “steady-state” containerless or levitation

Aero-acoustic Levitation

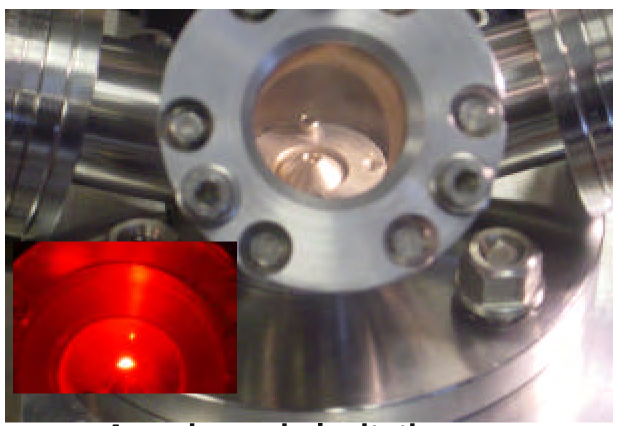
Electromagnetic Levitation



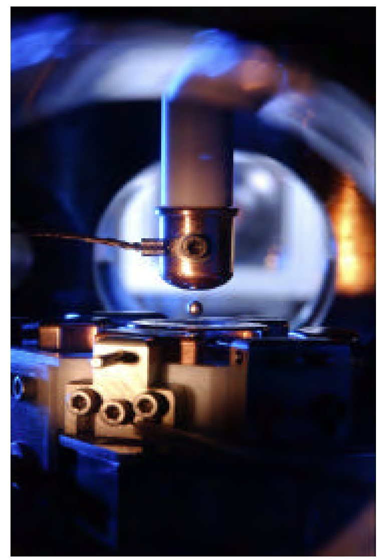
Acoustic levitation



High pressure electrostatic levitation



Aerodynamic levitation



UHV Electrostatic levitation

Magnetic levitation

Comparison of levitation techniques

Method	Sample size (mm)	Sample type	Atmosphere (bar)	Heating	Optical access	Footprint	Relative Price
Acoustic[1]	0.2-3.5	Most	0.5-1 most gases	External radiant	Excellent	Medium	Low
Aero-acoustic[2]	0.8-3.5	Most	1 most gases	External radiant	Excellent	Large	High
Aerodynamic[3]	0.5-4.0	Most#	0.5-5 most gases	External radiant	Good (>50%)	Small	Moderate
Electromagnetic[4]	**3-8***	Metallic conductor	UHV-pressure	EM, external	Poor	Small	Moderate
Electrostatic[5]	0.2-2.5	Material-specific	UHV or >3	External radiant	Good	Medium	High
Gas Film[6]	Up to 20	Low melting	1	Susceptor/EM	Poor	Medium	Moderate
Magnetic[7]	Up to 10	Diamagnetic	UHV-pressure	External radiant	Poor	Large	High
Optical[8]	<0.1		UHV	External (laser)	Good	Medium	Moderate

*Special field shaping coils have levitated up to ~1kg of aluminum.

#Demonstrated for reactive metallic liquids, [J.J. Wall, J.K.R. Weber, J. Kim, P.K. Liaw, and H. Choo, "Aerodynamic Levitation Processing of a Zr-based Bulk Metallic Glass," *Mater. Sci. Eng. A*, **445-446**, 219-22 (2007)].

1. E.H. Trinh, "Compact acoustic levitation device for studies in fluid dynamics and materials science in the laboratory and microgravity," *Rev. Sci. Instrum.* **56,** 2059–65 (1985).

2. J.K.R. Weber, D.S. Hampton, D.R. Merkley, C.A. Rey, M.M. Zatarski and P.C. Nordine, "Aero-acoustic Levitation - A Method for Containerless Liquid-phase Processing at High Temperatures," *Rev. Sci. Instrum.*, **65**, 456-65 (1994).

3. S. Krishnan, J.J. Felten, J.E. Rix, J.K.R. Weber, P.C. Nordine, M.A. Beno, S. Ansell and D.L. Price, "Levitation Apparatus for Structural Studies of High Temperature Liquids Using Synchrotron Radiation," *Rev. Sci. Instrum.*, **68**, 3512-18 (1997).

4. S. Krishnan, G.P. Hansen, R.H. Hauge and J.L. Margrave, "Observations on the dynamics of electromagnetically levitated liquid metals and alloys at elevated temperatures," *Met. Trans. A*, **19**, 1939-43 (1988).

5. W. K. Rhim, M. Collender, M. Hyson and D. D. Elleman, "Development of an Electrostatic Positioner for Space Materials Processing," *Rev. Sci. Instrum.*, **56**, 307-15, (1985).

6. M. Papoular and C. Parayre, "Gas-Film Levitated Liquids: Shape Fluctuations of Viscous Drops," *Phys. Rev. Lett.*, 78, 2120-23 (1997).

7. A.K. Geim, M.D. Simon, M.I. Boamfa, L.O. Heflinger, "Magnet levitation at your fingertips", *Nature*, **400**, 323-24 (1999).

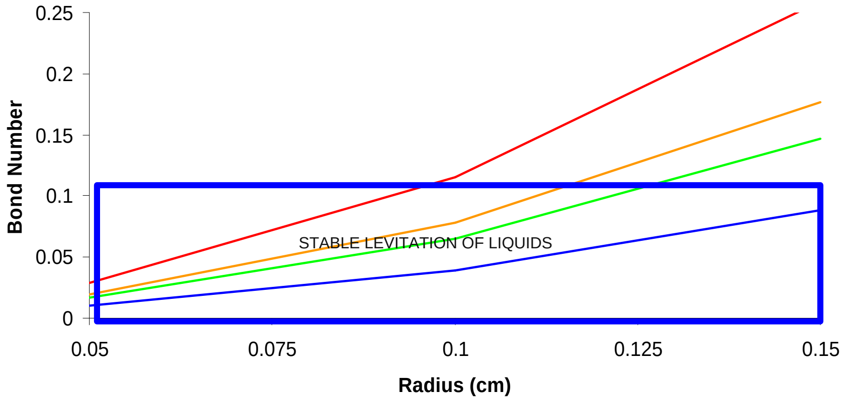
8. A. Ashkin, "Acceleration and Trapping of Particles by Radiation Pressure," *Phys. Rev. Lett.*, 24, 156-59 (1970)

Aerodynamic levitation

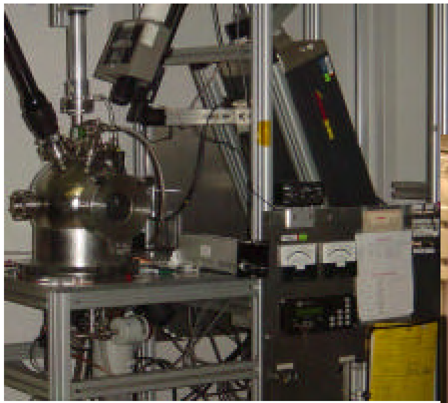
Plenty of force available
Size limit results from fragmentation of liquids

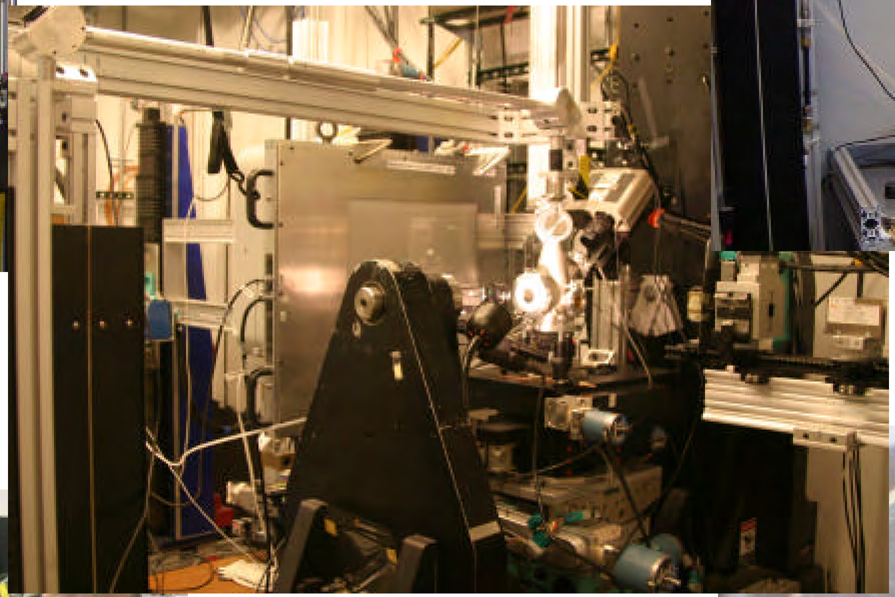
Antonov-225, payload 550,000 lbs
Takeoff wt. ~4 x 10^8 grams

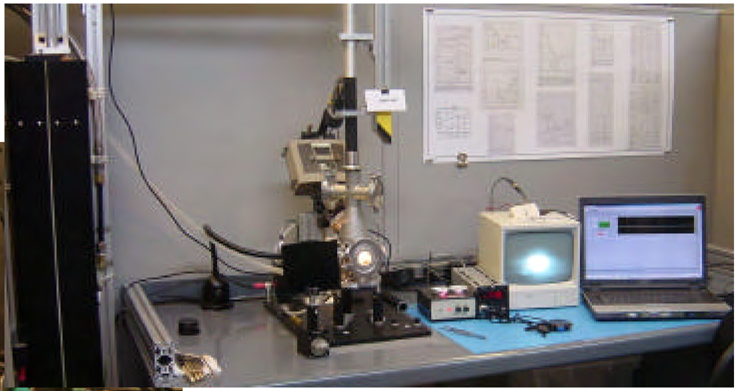
$$B_o = \frac{rgL^2}{g}$$

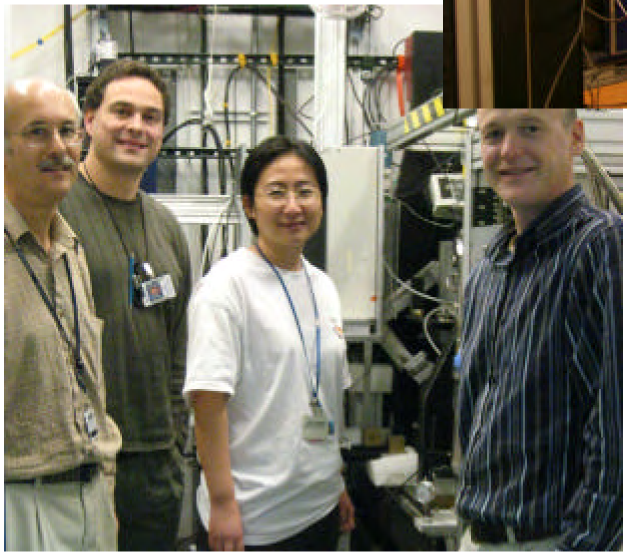


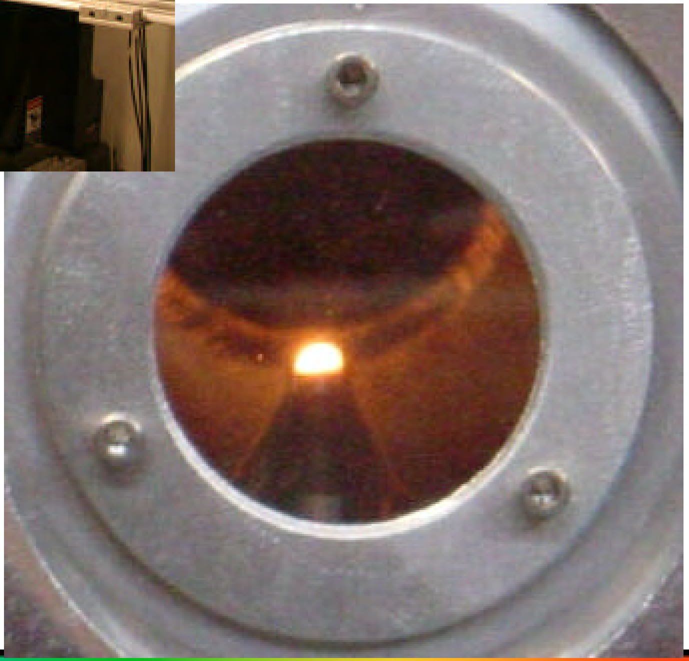
Operated as a Class I laser system with embedded 250 Watt (Class IV) CO2 laser in the lab and at 11 ID-C





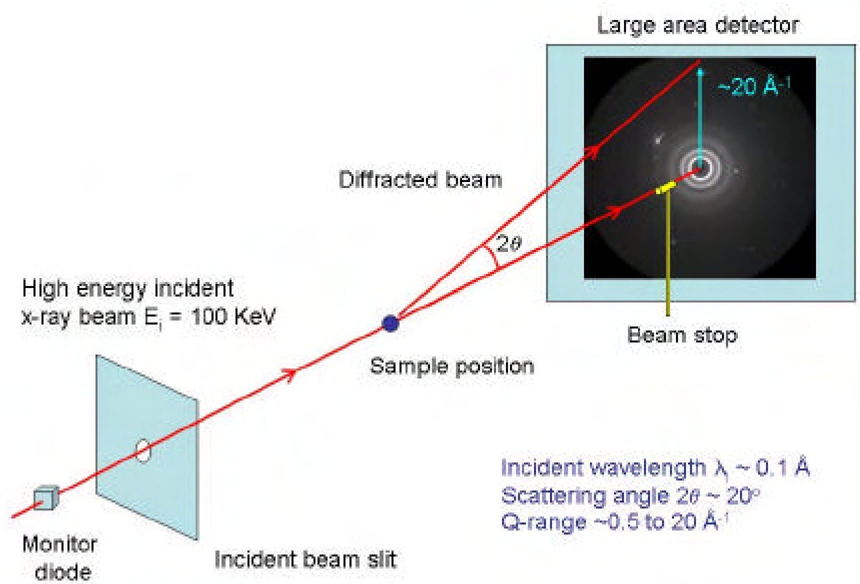






X-ray set up

Mar 345, PE 1600, GE Revolution



1 mm square beam
Area detector
High energy

Desirable features of an APS research levitation system

- Beamline and lab. based systems to enable sample synthesis and testing
- Non-contact temperature measurement
  - Optical diagnostics and beam probes
- Class I laser operation
  - Enhanced safety
  - More accessible to users
- Ability to change process atmospheres
  - Oxidizing, neutral, reducing, reactive
- Basic lab support:
  - semi-micro balance, density meas., mixing and grinding equipment, technical support

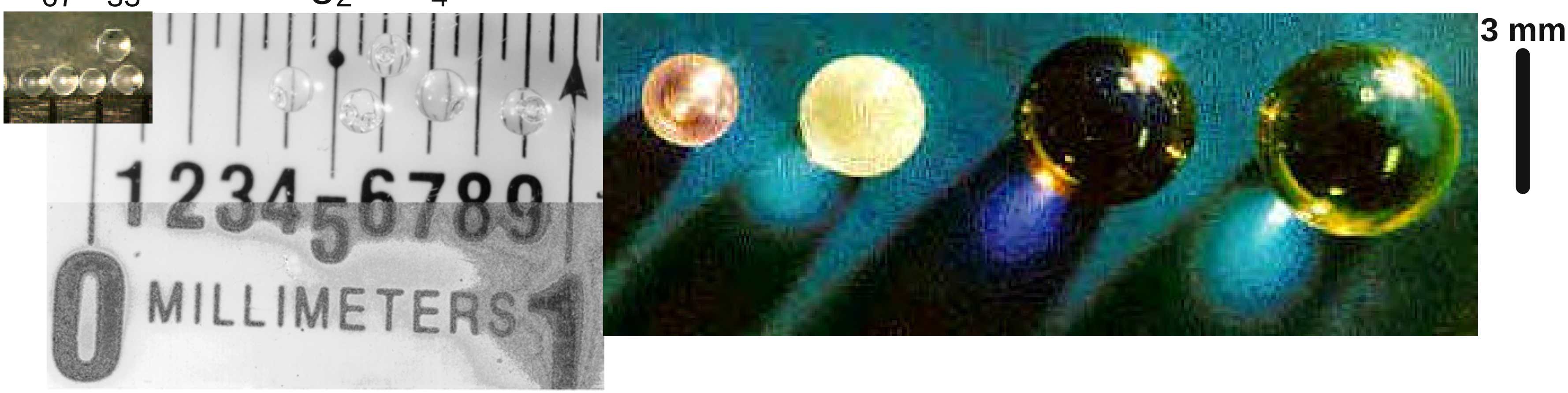
Some examples of reluctant glass formers made using levitation melt processing

- Al_2O_3-CaO 50-75 % CaO
- Al_2O_3-RE oxide 20-50 % RE oxide (includes RE garnets and perovskites)
- Al_2O_3-SiO_2 up to 67 % Al_2O_3 (includes 60/40 mullite)
- CaO-SiO_2 up to 50% CaO
- MgO-SiO_2 up to 67 % MgO (includes enstatite and forsterite)
- Calcium phosphates
- Zr-Cu-Ni-Al-Ti alloys

Cooling rate limited by surface area:volume

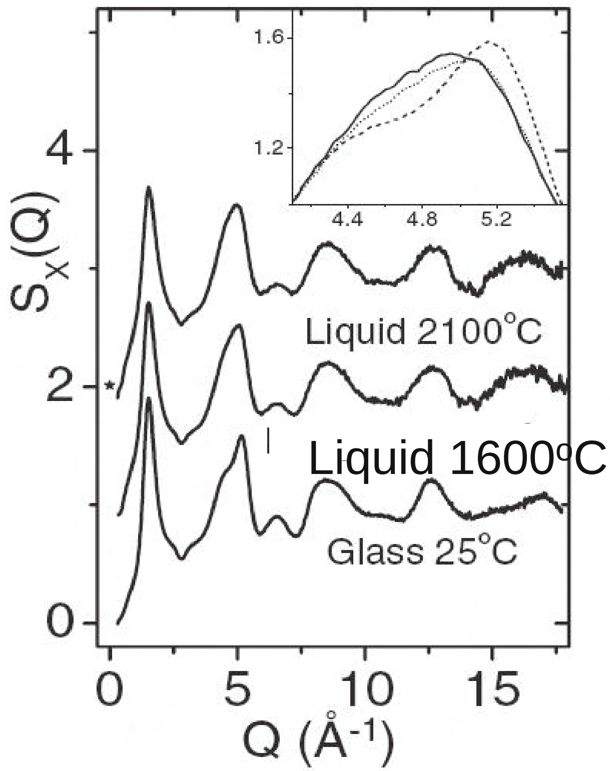
1000 — 300 — 100 — 30 K/s @ 1300K

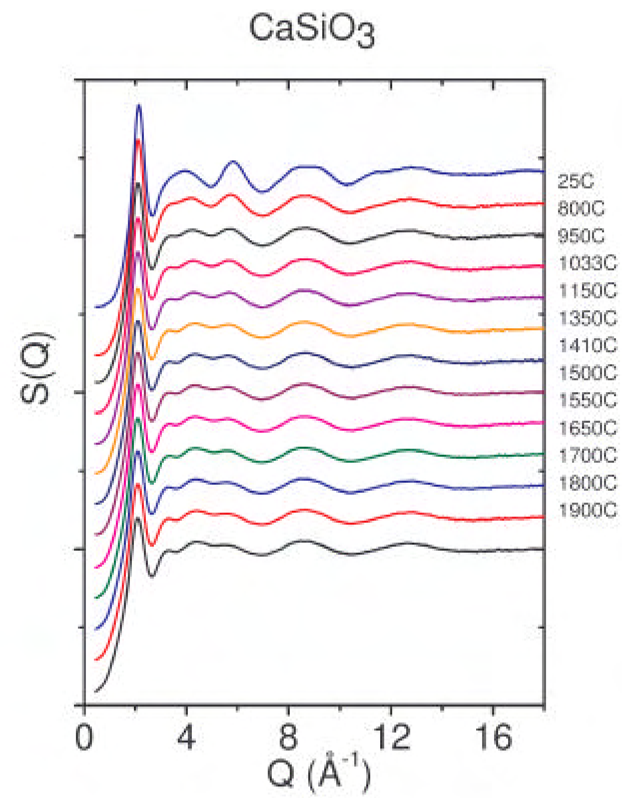
$A_{67}S_{33}$ — Mg_2SiO_4 — ErAG — YAG — LAG



Temperature dependent structure of liquids

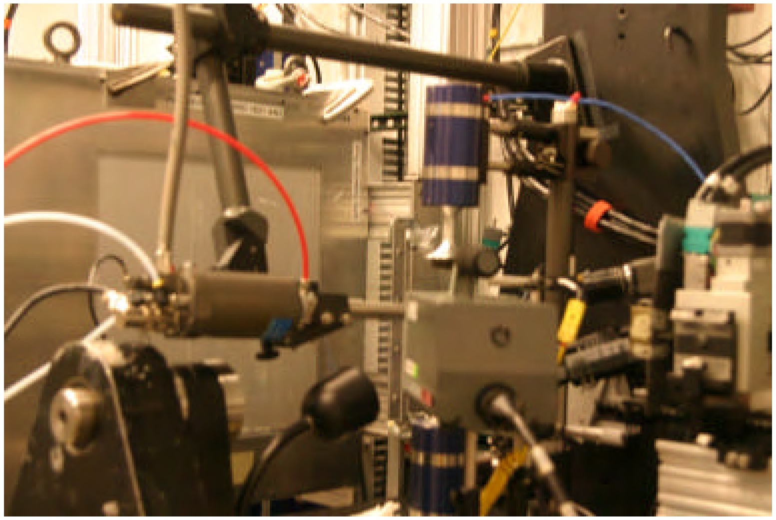
SiO_2

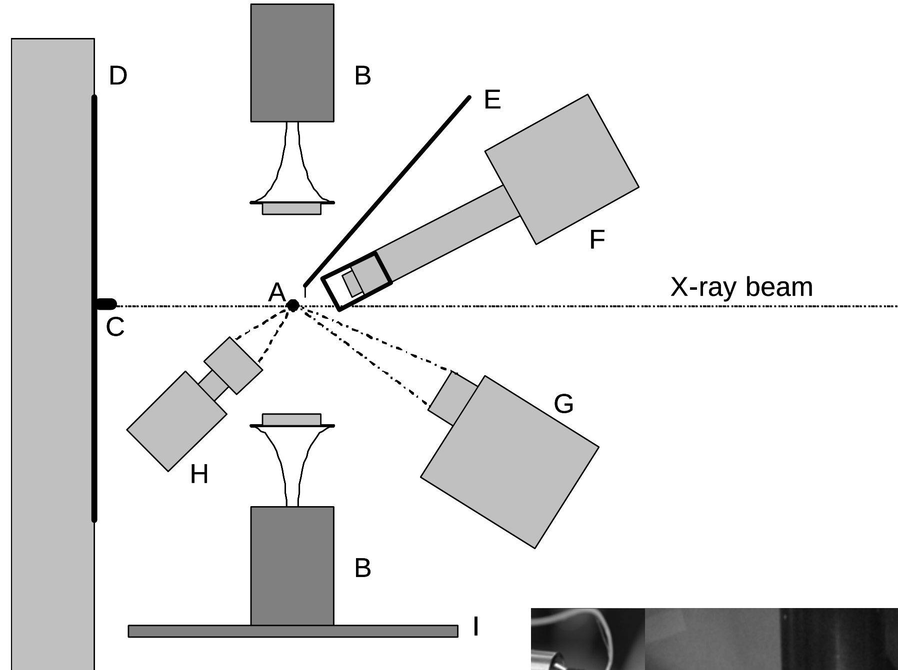




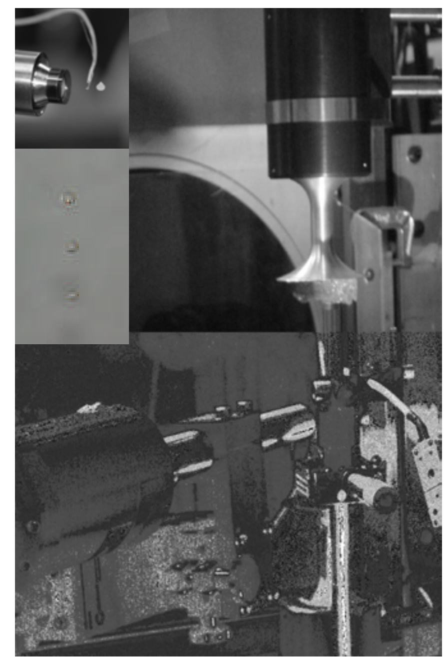
Mei, *et al*, PRL, **98**, 057802 (2007).

Acoustic Levitation



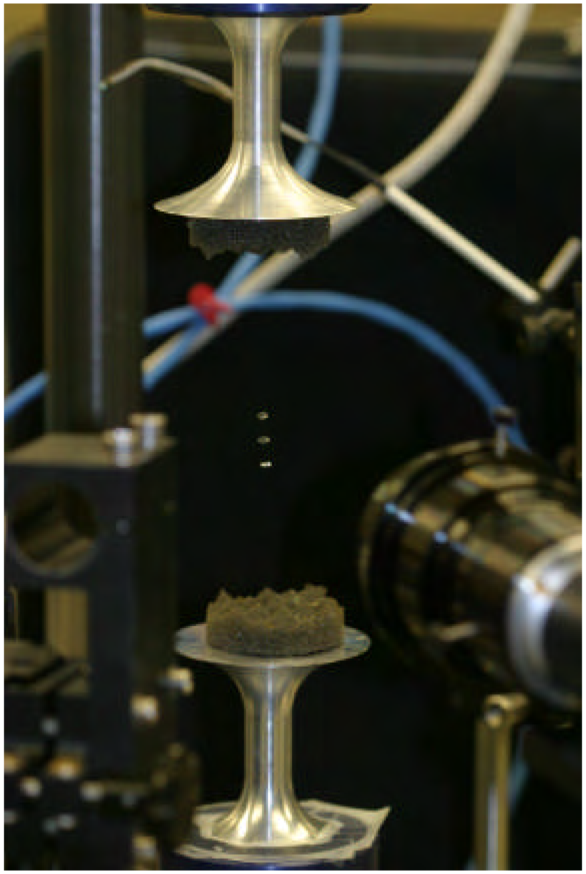


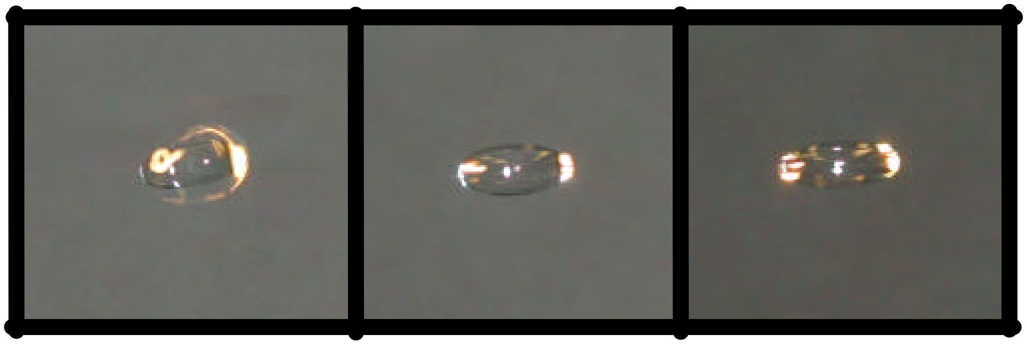
VIDEO

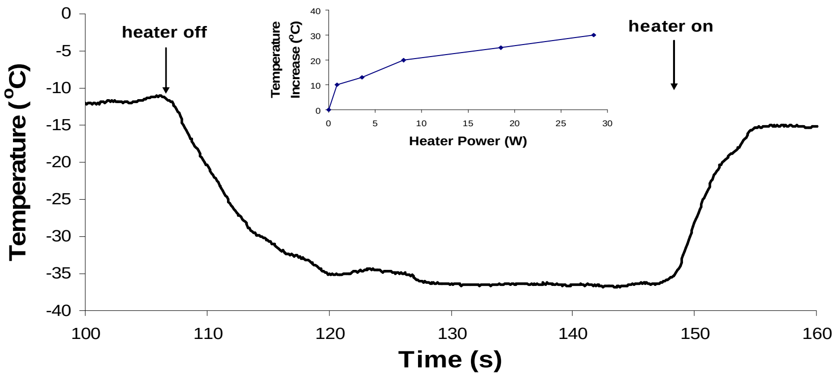


RSI, **80**, 083904 (2009).

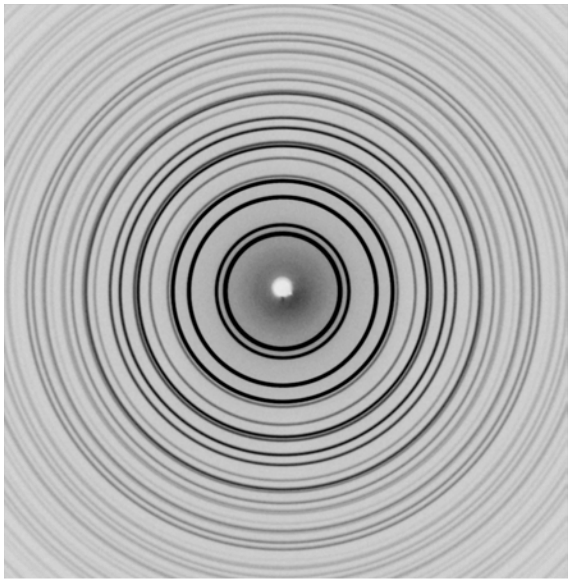
Acoustic Levitation

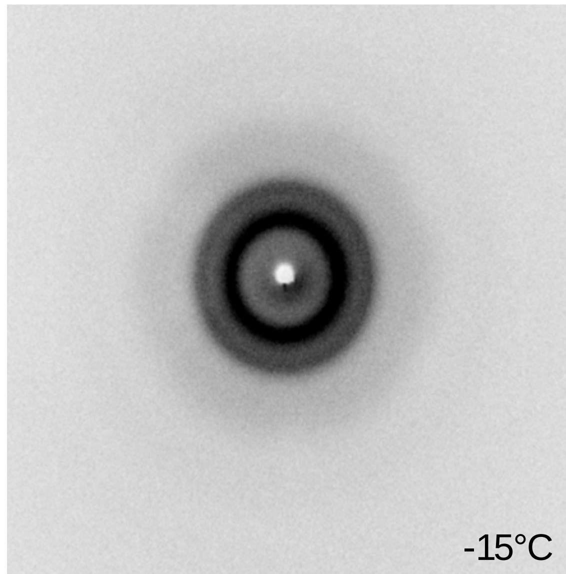


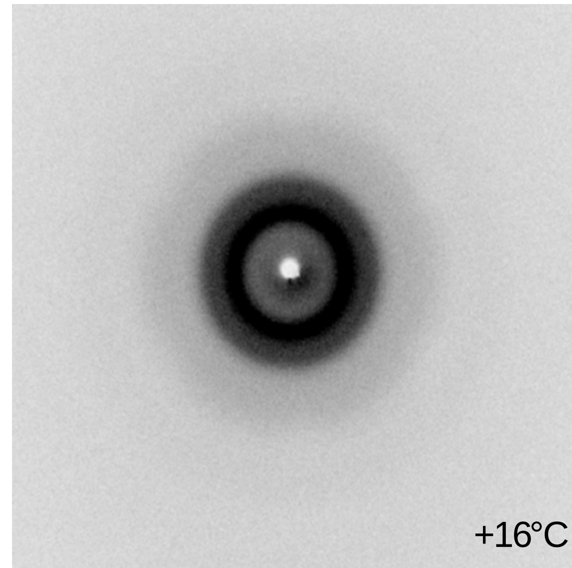




What the image plate "sees"

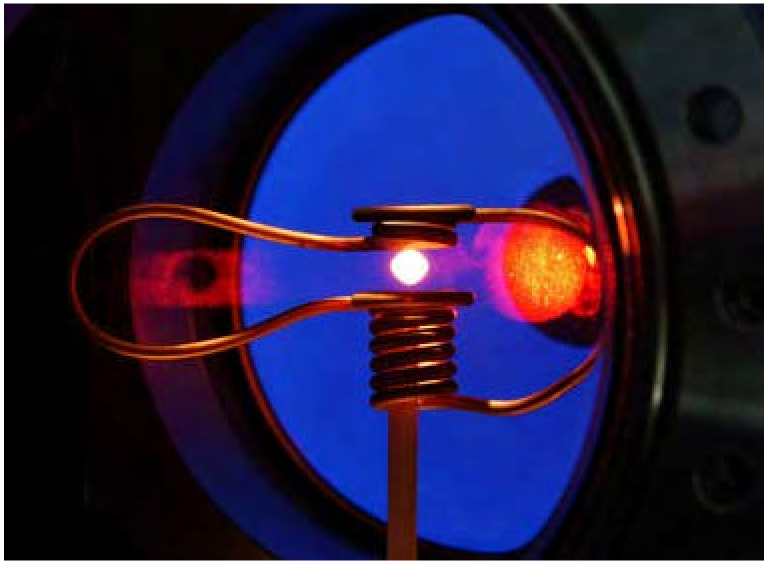




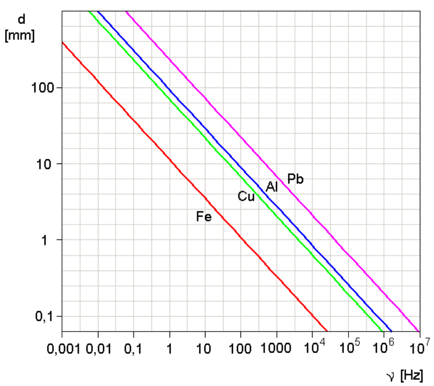


Small (sub-millimeter) sample motion does not measurably affect the data quality

Electromagnetic levitation



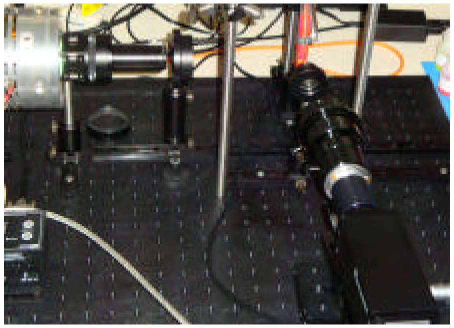
- Need skin depth, d, < sample x-section
- Force is proportional to (*field gradient*)^2
- Heating is proportional to (*induced* (current) *field*)^2
- Typically 450 kHz generator (Lepel, Inductotherm, Ameritherm)
- Need to match impedance of load
- Need to avoid FCC frequencies
- Heating and levitation are **coupled**
- Cooling gas or beam heating can extend T range
- Used in a demo expt. at GLAD ~1993

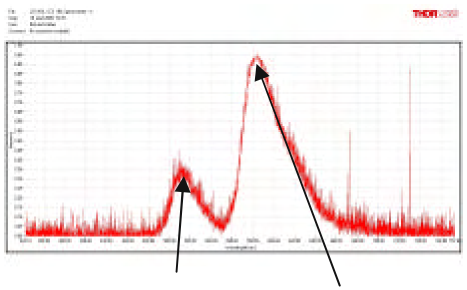


$$d \propto C\sqrt{\frac{r}{n}} \qquad Force \propto \frac{dB^2}{dz} \qquad Heat \propto B^2$$

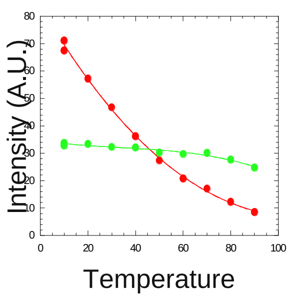
Non-contact temperature measurement

Low T





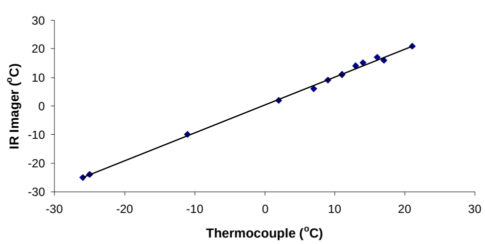
5,6 carboxyfluorescein rhodamine B



FL emission ratio measurements
Ross *et al*, Analyt. Chem., 4117-23, **73** (2001).

Inframetrics 760, 8-12 μm thermal imaging camera

Peak in thermal emission spectrum at 300 K is ~10 μm



High T

Optical pyrometry at a wavelength where emission is strong. Peak ~3000/T (in K)

Emissivity corrections may be needed.

$$\frac{1}{T_{abs.}} - \frac{1}{T_{app}} = \frac{\lambda \ln(\varepsilon_{\lambda})}{C_2}$$

Summary

- *Good coverage of "sample environment space"*
- *Ability to investigate liquids under non-equilibrium conditions and at high purity*
- *Class I laser system maximizes safety, minimizes user training requirements*
- *Complementary bench top facility enables characterization and synthesis*
- ***Neutron + X-ray** needed to deconvolute structure and constrain models*
- ***Work needed** on low T measurement*
- ***Work needed** on fast measurements in transient conditions – high flux is essential*

sample environment space