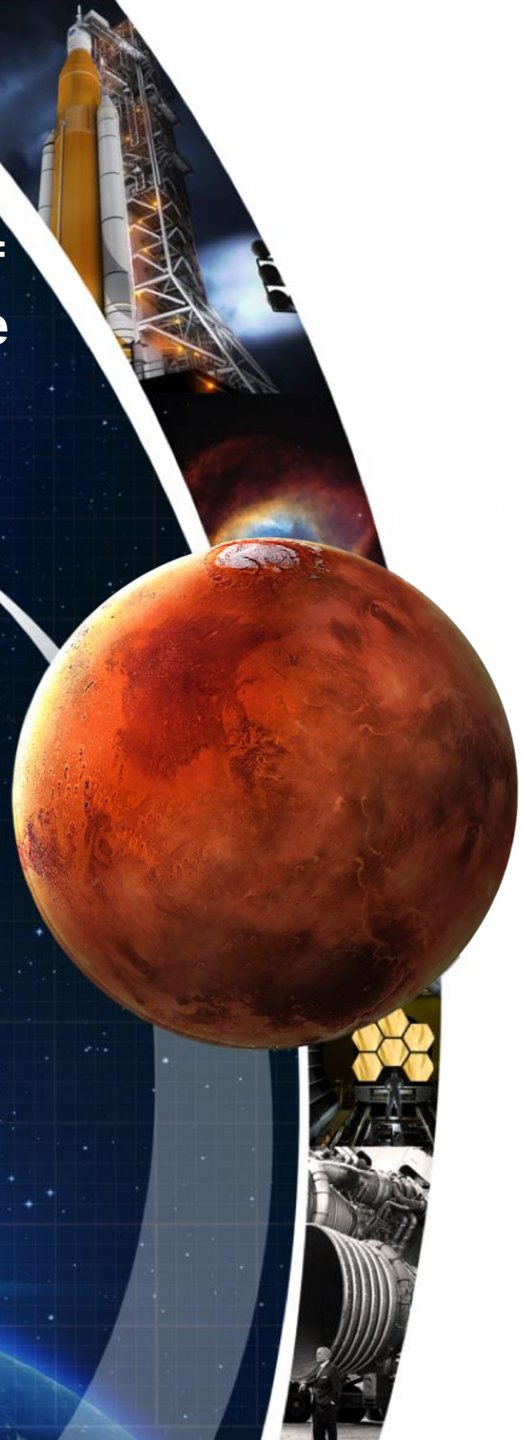
National Aeronautics and Space Administration

Electrostatic Levitation of ZBLAN and Chalcogenide Glasses



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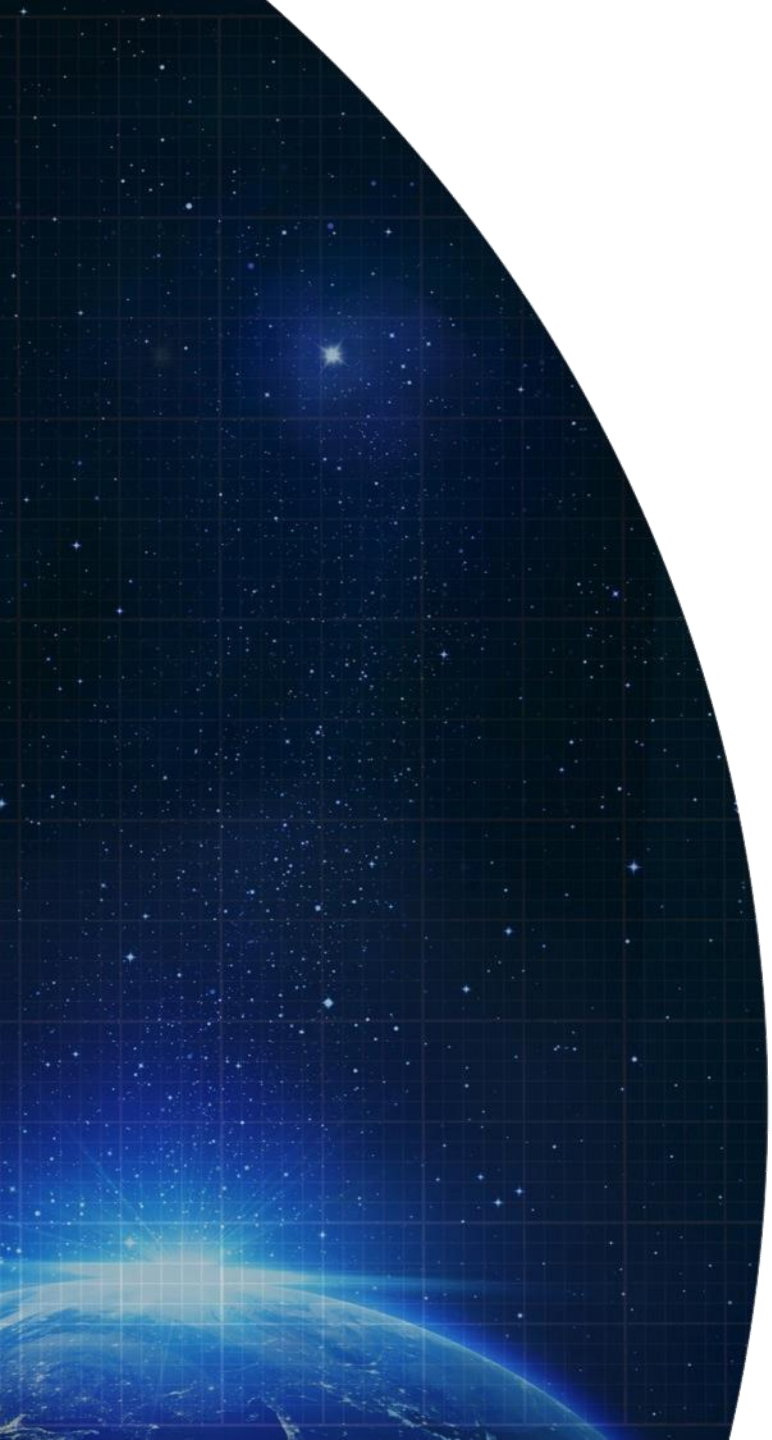


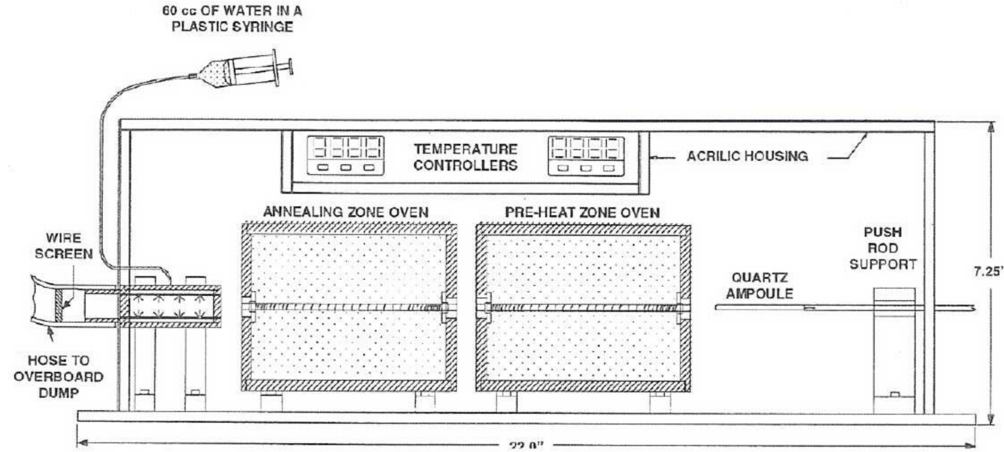
Background

- Heavy Metal Fluoride Glasses have been studied for ~ 35 years
- ZrF_4-BaF_2-LaF_3-AlF_3-NaF (ZBLAN) showed the most promise as optical fiber
- Transmits IR out to ~ 5 microns
- Applications: fiber amplifiers, fiber lasers and nuclear radiation resistant links
- Intrinsic and Extrinsic processes limit light propagation
- Intrinsic: band gap absorption, Rayleigh scatter and multiphonon absorption
- Extrinsic: impurities such as rare-earth ions and crystallite formation
- Theoretical loss coefficient is 0.001 dB/km
- This loss has not been achieved to date due to intrinsic and extrinsic processes

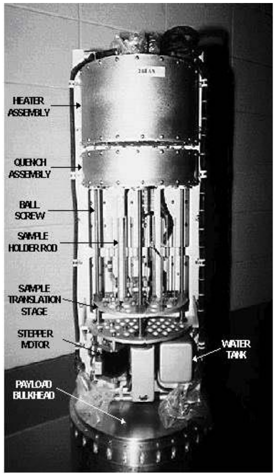
Background

- ZBLAN fibers obtained from Infrared Focal Systems, Inc. and Bell Laboratories
- Fibers were stripped of coating and placed in evacuated quartz ampoules
- These fibers were first flown on NASA's KC135 Reduced Gravity Aircraft
- KC135 produces ~ 25 sec. of reduced gravity per parabola
- One week of flights led to ~ 200 total parabolas
- Fibers were heated to the crystallization temp. during reduced gravity and compared to unit gravity for same amount of time
- Fibers were also flown on board Conquest sub-orbital rocket
- This flight gave ~ 6.5 minutes of reduced gravity

Background

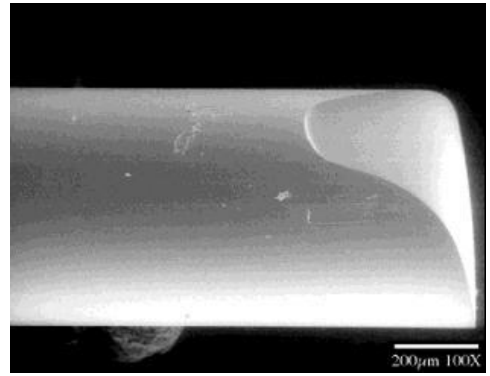


60 cc OF WATER IN A PLASTIC SYRINGE
TEMPERATURE CONTROLLERS
ACRILIC HOUSING
ANNEALING ZONE OVEN
PRE-HEAT ZONE OVEN
WIRE SCREEN
PUSH ROD SUPPORT
QUARTZ AMPOULE
7.25"
HOSE TO OVERBOARD DUMP

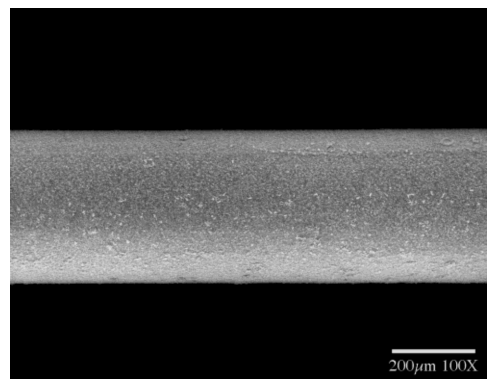


HEATER ASSEMBLY
QUENCH ASSEMBLY
BALL SCREW
SAMPLE HOLDER ROD
SAMPLE TRANSLATION STAGE
STEPPER MOTOR
WATER TANK
PAYLOAD BULKHEAD

Background

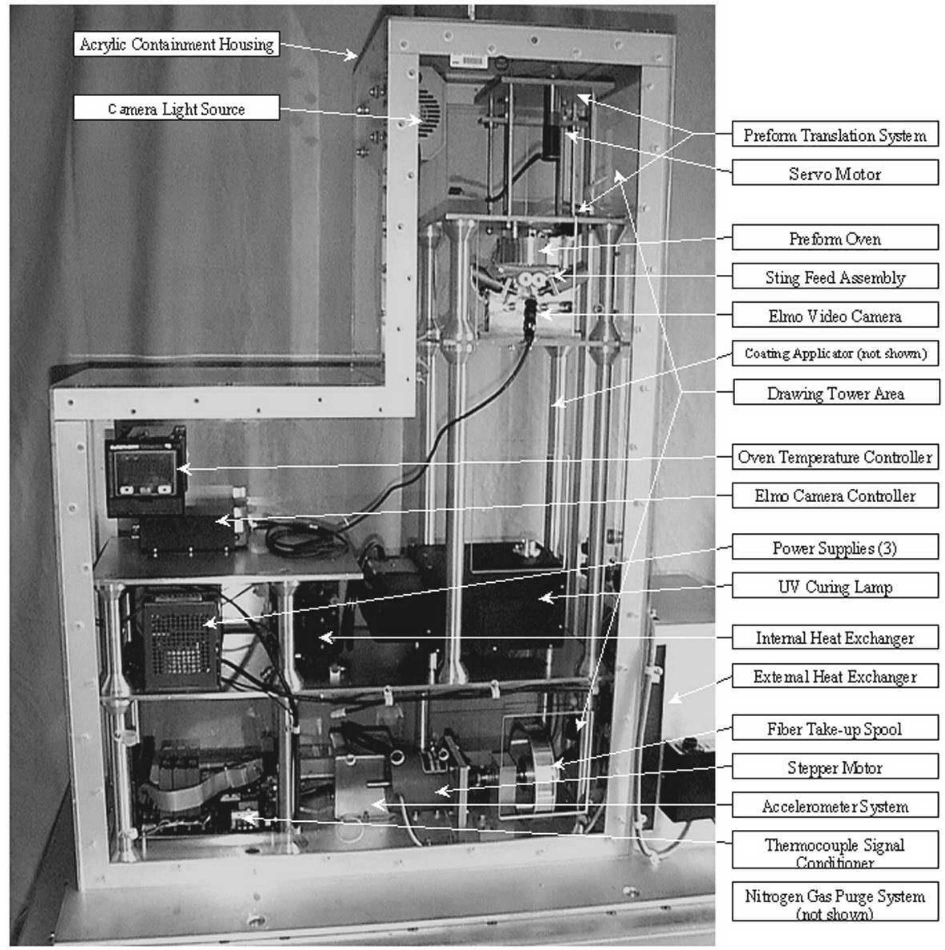


Zero-g



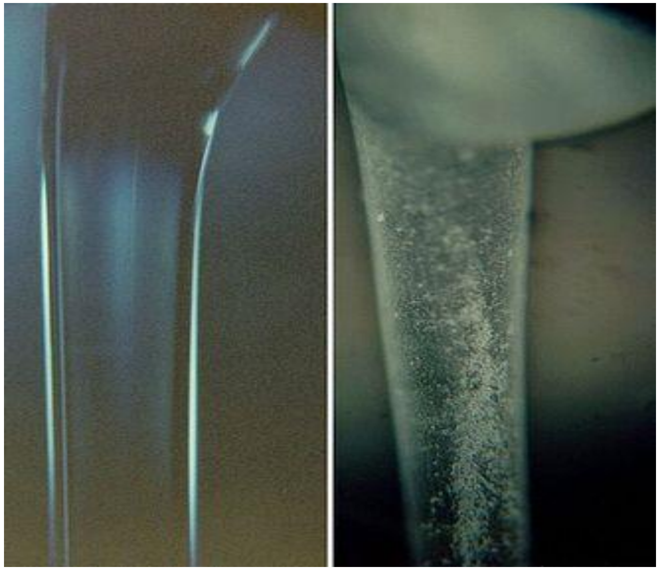
Unit-g

Background

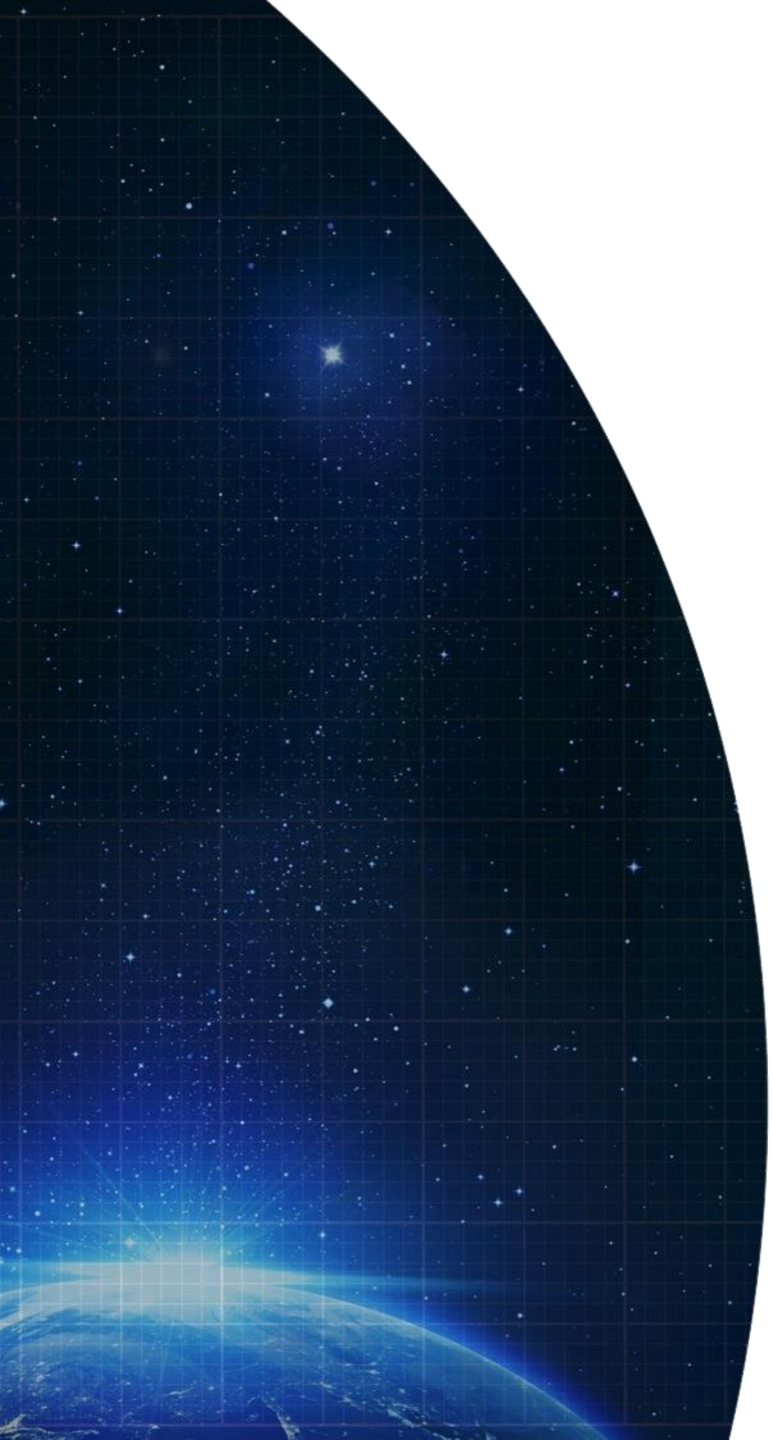


Acrylic Containment Housing
Camera Light Source
Preform Translation System
Servo Motor
Preform Oven
Sting Feed Assembly
Elmo Video Camera
Coating Applicator (not shown)
Drawing Tower Area
Oven Temperature Controller
Elmo Camera Controller
Power Supplies (3)
UV Curing Lamp
Internal Heat Exchanger
External Heat Exchanger
Fiber Take-up Spool
Stepper Motor
Accelerometer System
Thermocouple Signal Conditioner
Nitrogen Gas Purge System (not shown)

Background



Zero-g High-g

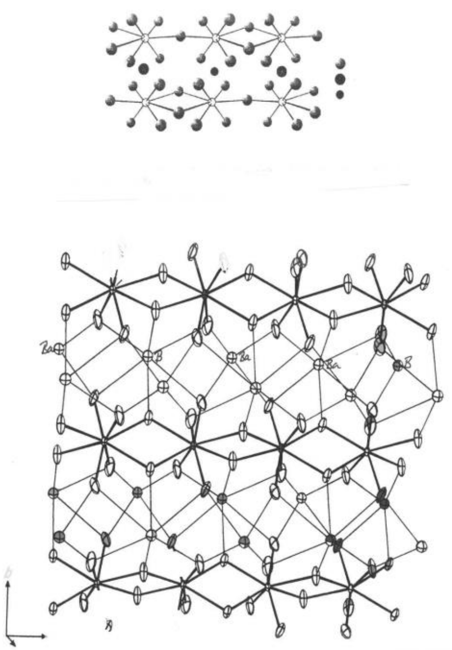


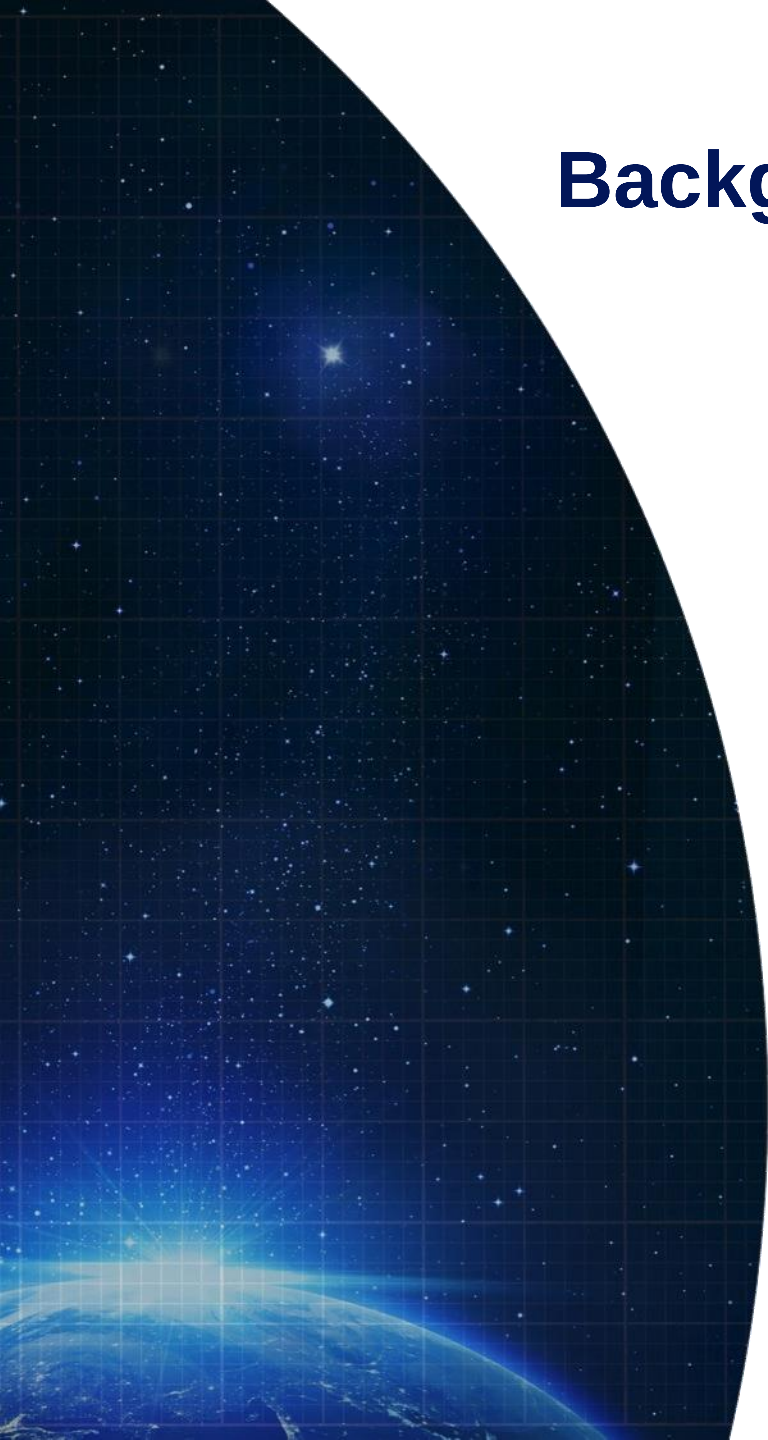
Background

- What causes the suppression of crystallization in zero-g?
- Nucleation and Growth Rates are inversely proportional to viscosity
  - $I \sim 1/\eta$ and $U \sim 1/\eta$
- Ethridge proposed shear thinning present in unit gravity is absent in reduced gravity
- This has been observed for some polymer melts
- Under conditions of shear thinning, effective viscosity decreases with increasing shear rate, thus,
  - $\eta = \eta(\varepsilon)$, and
  - $I(\varepsilon)$ and $U(\varepsilon) \sim 1/\eta(\varepsilon)$
- Low gravity processing is known to reduce convection, which reduces shear
- Thus, crystallization will be reduced in low gravity

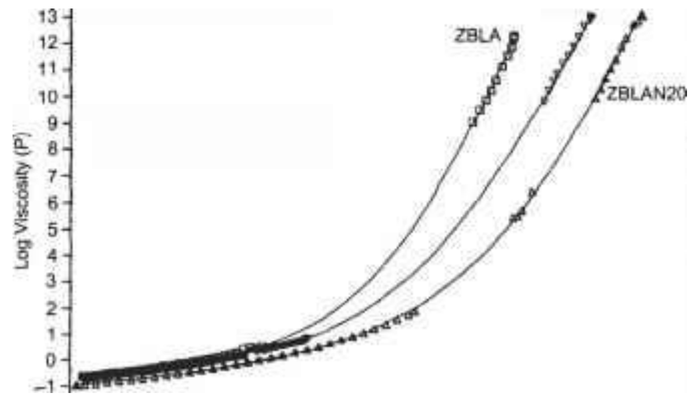
Background

- May be more subtle – Lateral diffusion of barium ion a few nm will induce crystallization – so any perturbation could cause crystallization.

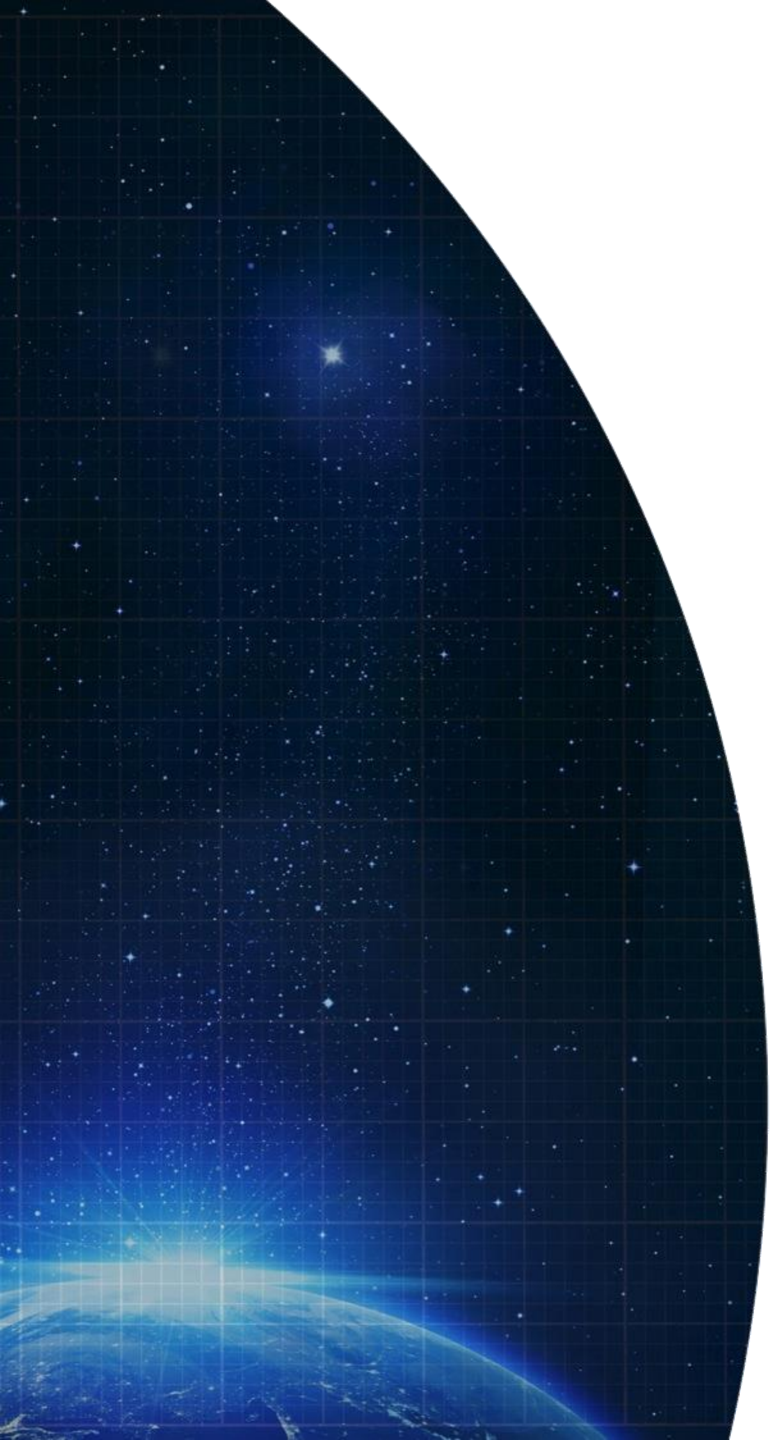




Background-log viscosity vs. 1/T



Viscosity of ZBLAN is highly non-linear
Proposed that processing in zero-g flattens viscosity curve



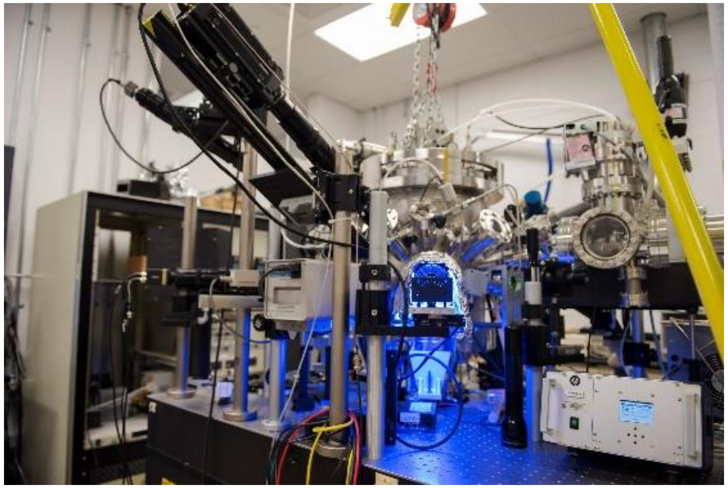
Experimental

- **It is proposed to use electrostatic levitation (ESL) to perform viscosity and crystallization studies of pure ZBLAN and selected chalcogenide glasses**.
- Viscosity is inversely proportional to nucleation and growth and is the only measurable quantity in these equations, all others being thermodynamic.

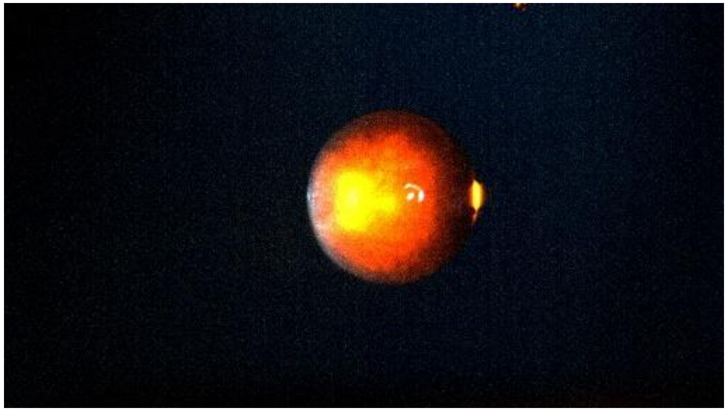
Experimental – Chalcogenide Samples

- Sample 1 : **As39Se61**
- Sample 2 : **As38Se60I2**
- Sample 3 **: As38Se58I4**
- Sample 4: **As38Se56I6**
- Sample 5 **: As38Se60I2 doped Pr3+.**
- Using Arsenic, Selenium and Iodine: have seen crystals during fiber draw

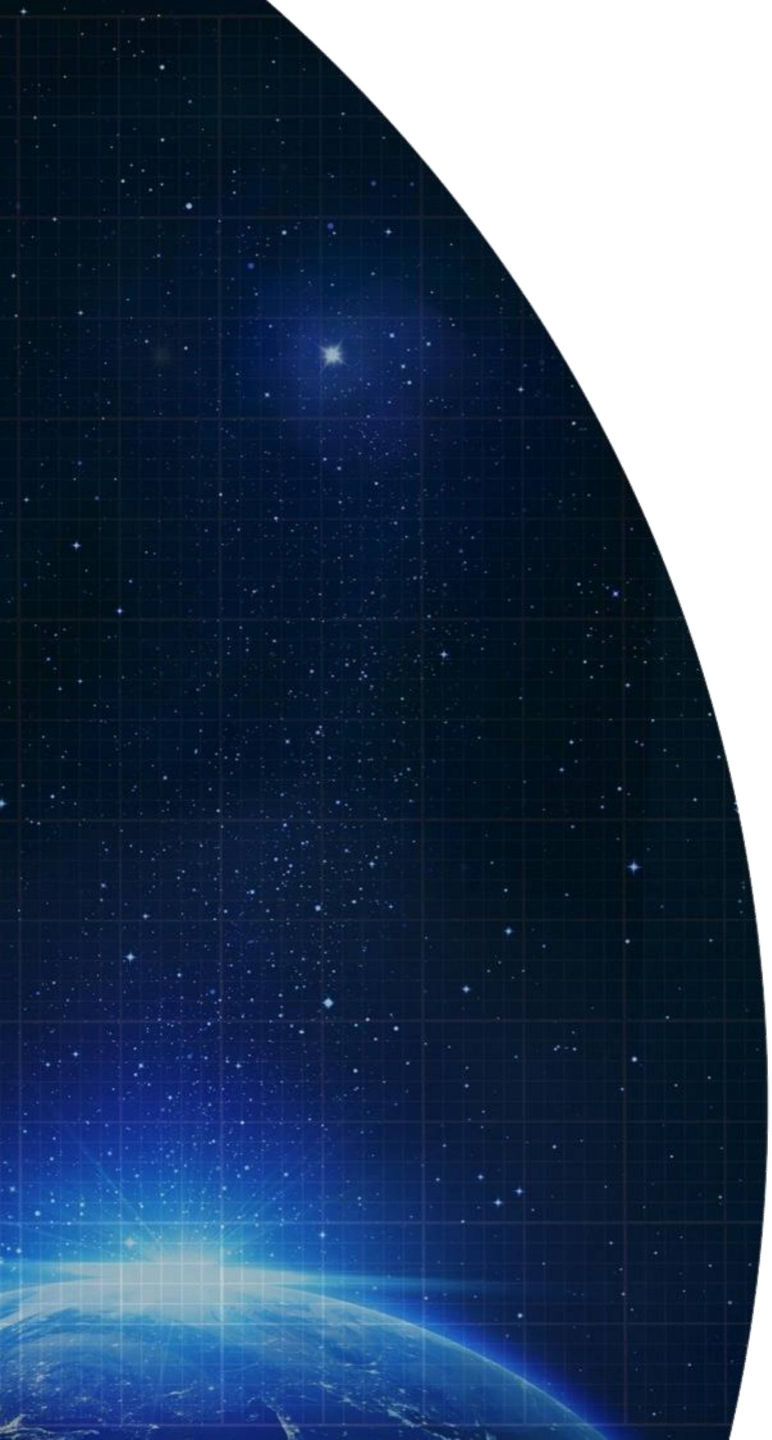
ESL of ZBLAN



MSFC ESL Facility



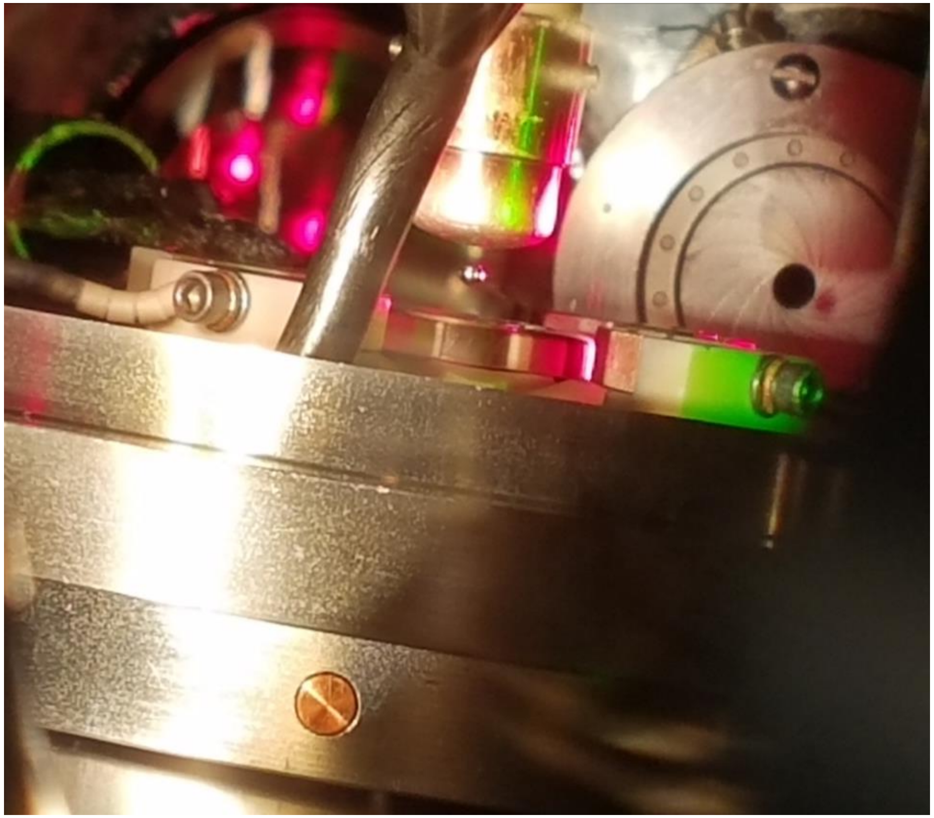
ZBLAN levitated ~ 500C



Electrostatic Levitation

- ESL uses an electrostatic field to levitate charged specimens
  - Typically 2-3mm spheres
- The field is created between 6 electrodes
  - 3 are grounded
  - 3 are connected to high-voltage DC amplifiers
- Charge is maintained by irradiating the sample with UV light
  - Deuterium arc lamp
  - As samples are heated volatile components tend to evaporate as positively charged ions
    - Surface ionization
- Samples are heated and melted with a laser
  - The ESL lab has Nd:YAG, fiber optic, and CO_2 lasers available
- Temperature is measured with a pyrometer
  - InGaS (~1.5um) typically

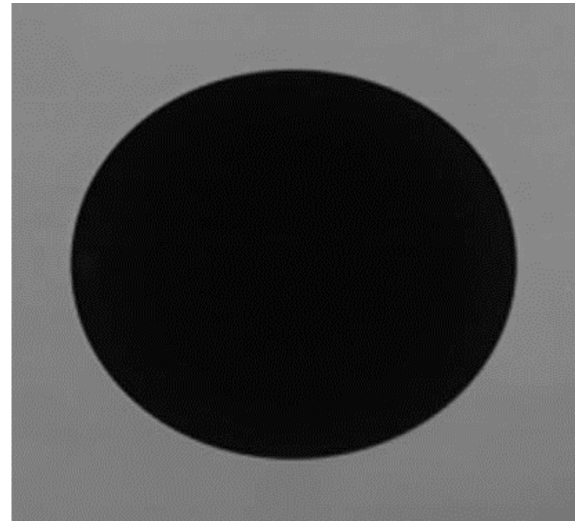
Electrostatic Levitation



Viscosity Measurements

- Oscillating drop method
- Surface oscillations are induced in the molten sample by modulating the electrostatic field near the resonant frequency
- Once stable mode 2 oscillations have developed
  - – Modulation is turned off
  - – Natural frequency of the resulting oscillations and the damping are recorded with high-speed video
    - Typically 1000fps
- Surface tension calculated from natural frequency
- Viscosity calculated from damping coefficient

Viscosity Measurements



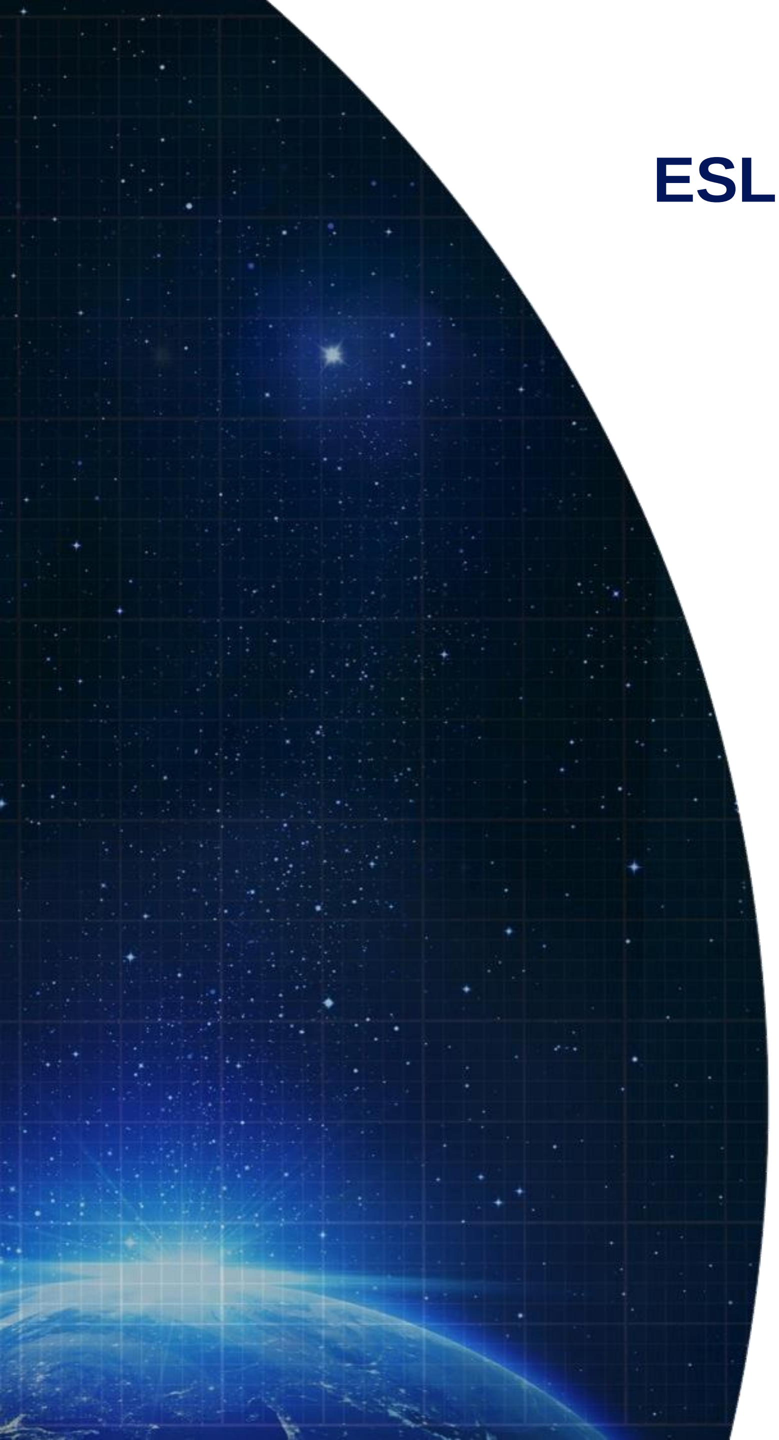
Example of an oscillating sample at its resonant frequency.

$$\omega_l = \sqrt{\frac{l(l-1)(l+2)\gamma}{\rho R_o^3}}$$

Rayleigh (1879)

$$\tau_l = \frac{\rho R_o^2}{(l-1)(2l+1)\mu}$$

Lamb (1881



ESL Experiments with ZBLAN To Date

- The MSFC ESL lab has made several attempts to process ZBLAN samples
- The lab has been able to melt samples and oscillate a couple of samples
- ZBLAN has proven to be a difficult material to work with
  - But not impossible
- More time perfecting the techniques for ZBLAN is required
- Thermal gradients have been an issue
  - Heating from 2 directions, with 2 CO_2 lasers should help