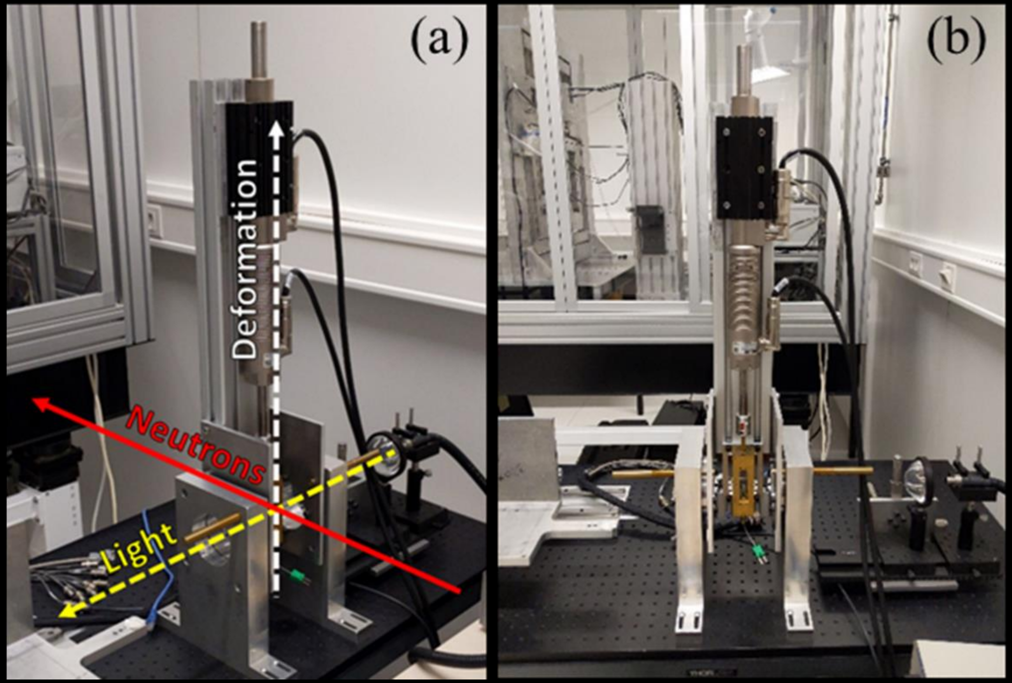
Development of an Extensional Flow SANS Sample Environment for Polymer Solutions

Dr. Matthew Wade

26/11/23



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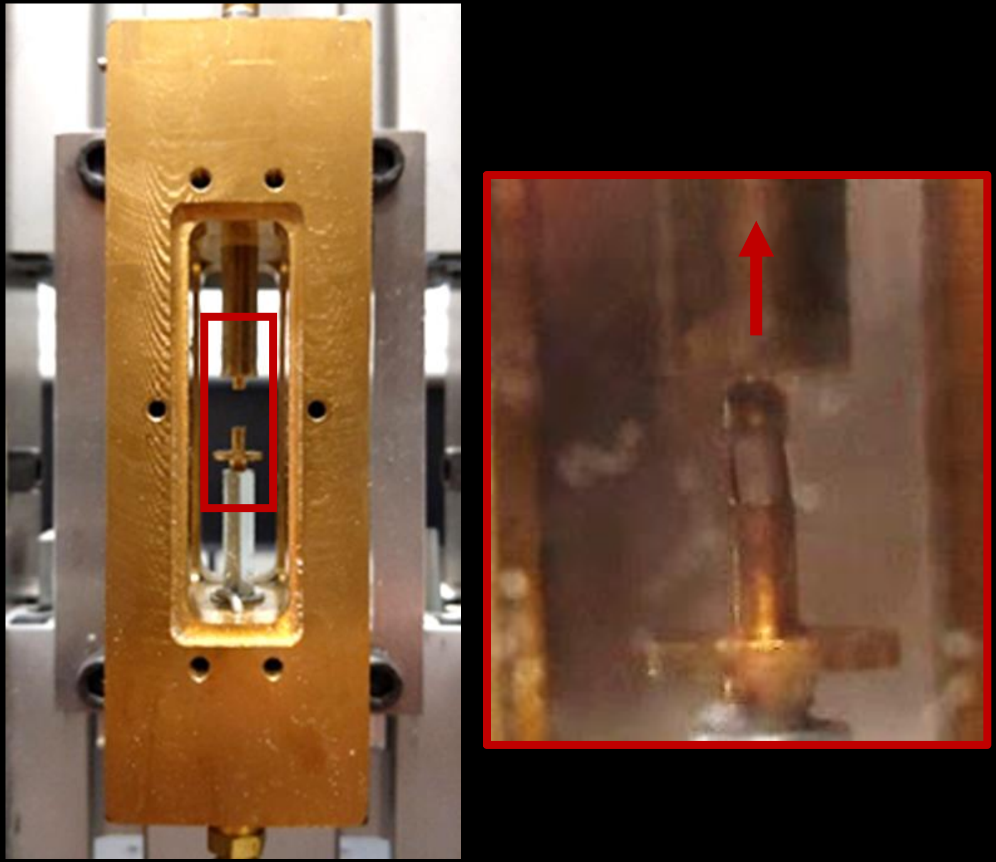
Capillary Breakup Extensional Rheometer

- Standing capillary between two rapidly separated plates with high-speed camera
  - Surface tension + gravity serve as dominating forces
  - Approximation of extensional flow
  - Industrially relevant processes – Ink-jet printing, Direct ink-writing



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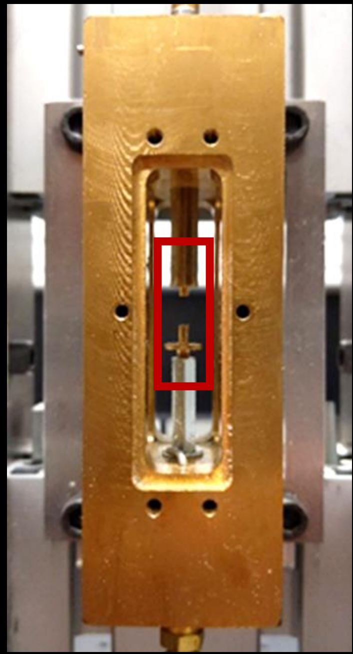
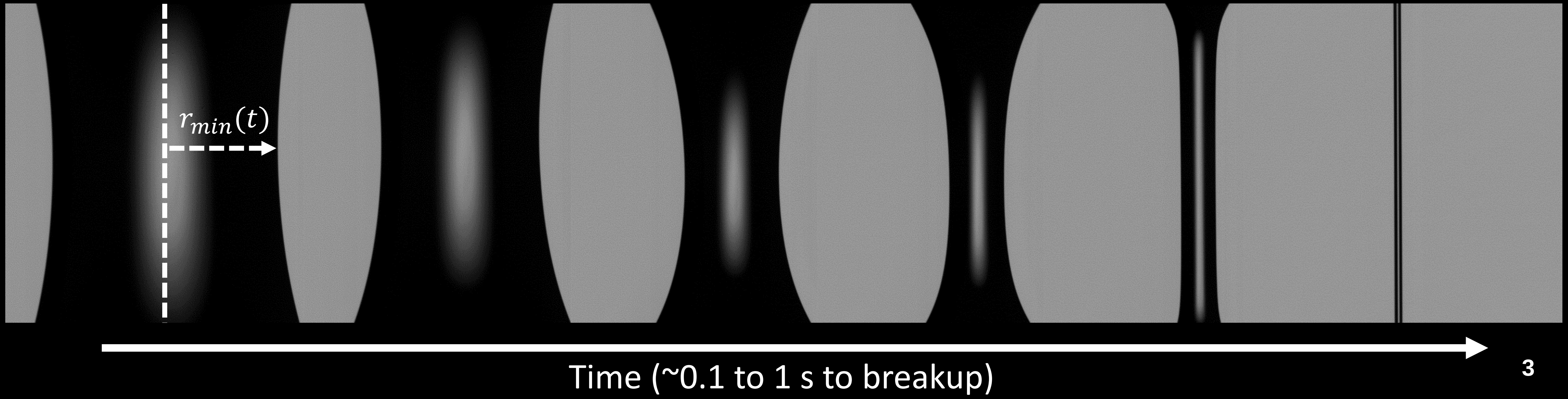
Capillary Breakup Extensional Rheometer

- Extensional properties from transient filament and breakup
  - Minimum radius as a function of time, $r_{min}\ (t)$
  - Apparent extensional viscosity
  - Extensional relaxation time

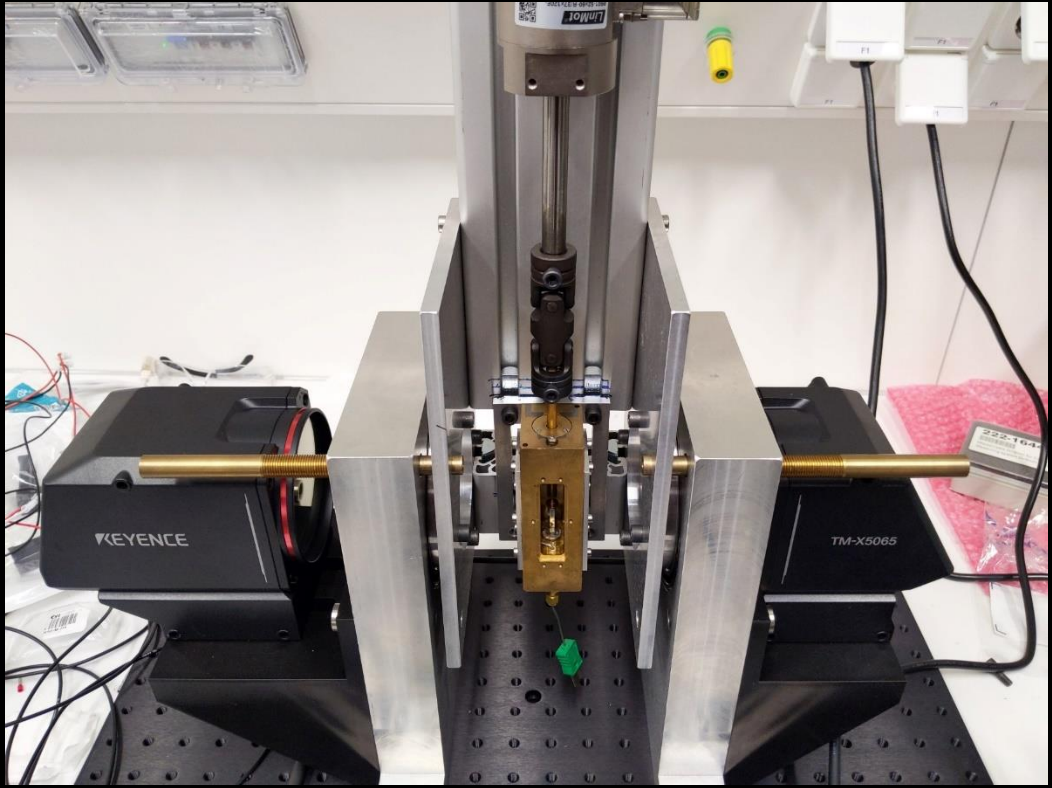
- Ongoing questions involving microscale contributions
  - Impact of small quantities of high MW polymer on capillary formation





CaBER Sample Environment for KWS-II

- Prototype developed as part of GNeuS fellowship
  - Linear drive controlled by Beckhoff PLC
  - Keyence TM-X5000 to capture and process images (loaned for proof of concept)
  - Partially sealed sample environment
  - Syringe loaded sample plate
- System based on custom CaBER instrument
  - Clasen group from K.U. Leuven



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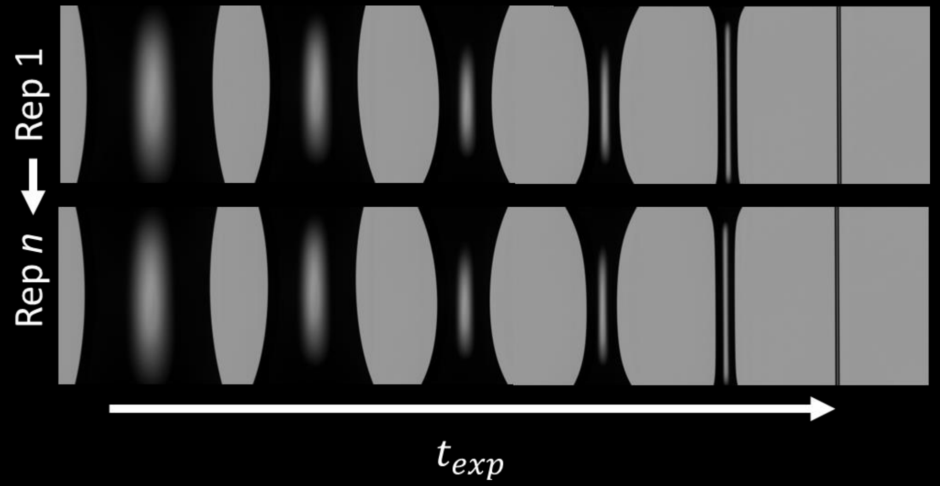
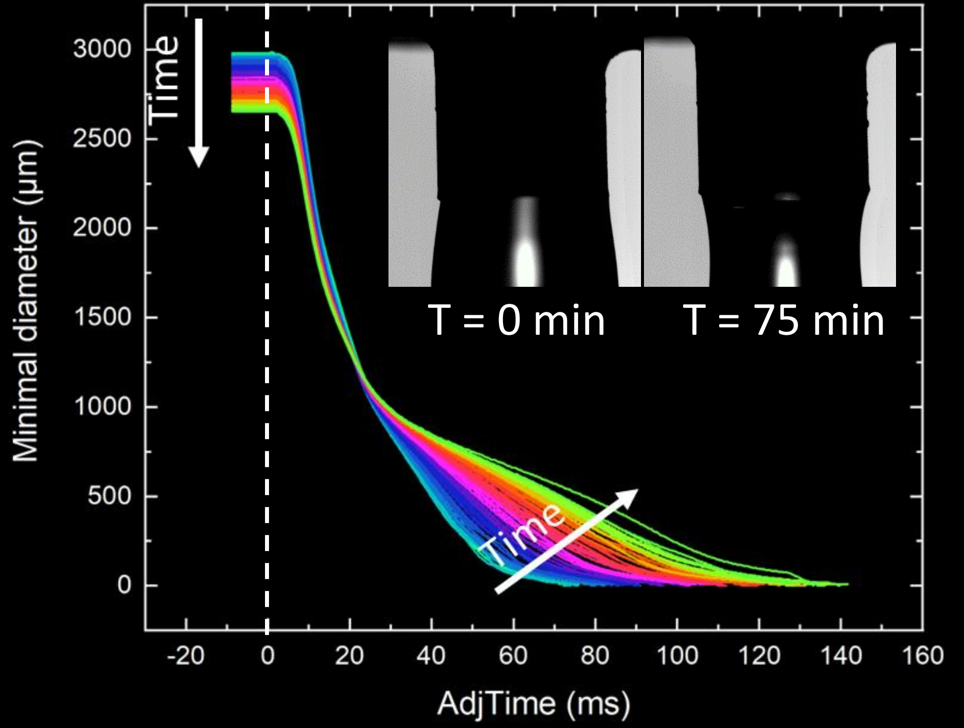
Considerations for CaBER-SANS Measures

- CaBER is inherently transient measurement
  - Transient SANS methods and binning
    - Dependent on material (100 ms to 2-3 s)
  - Automation – repeat test with limited human intervention
  - Repeatability – Comparable flow conditions



- CaBER is usually a “once on per loading” measure
  - Significant changes over 1.5 hrs
    - Evaporation
    - Sample falling off geometry
  - Loading effects



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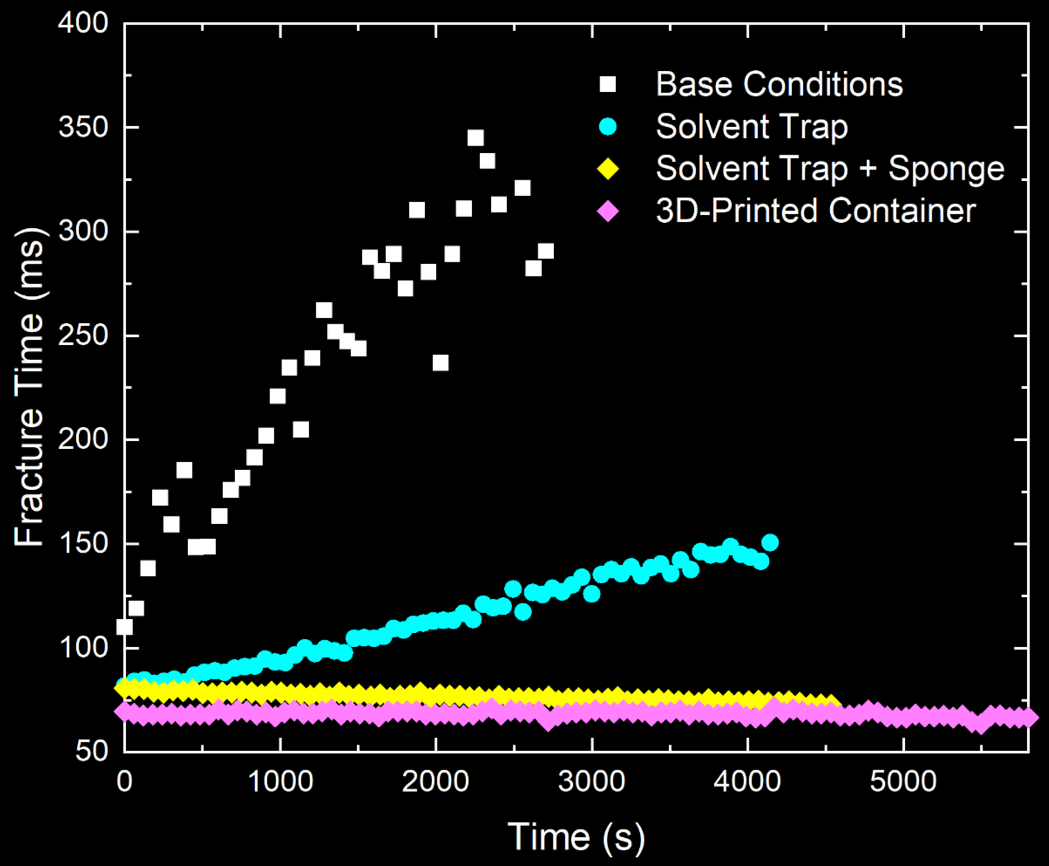
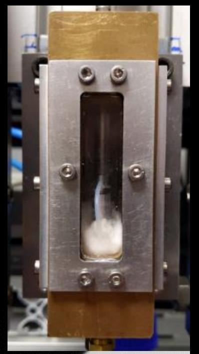
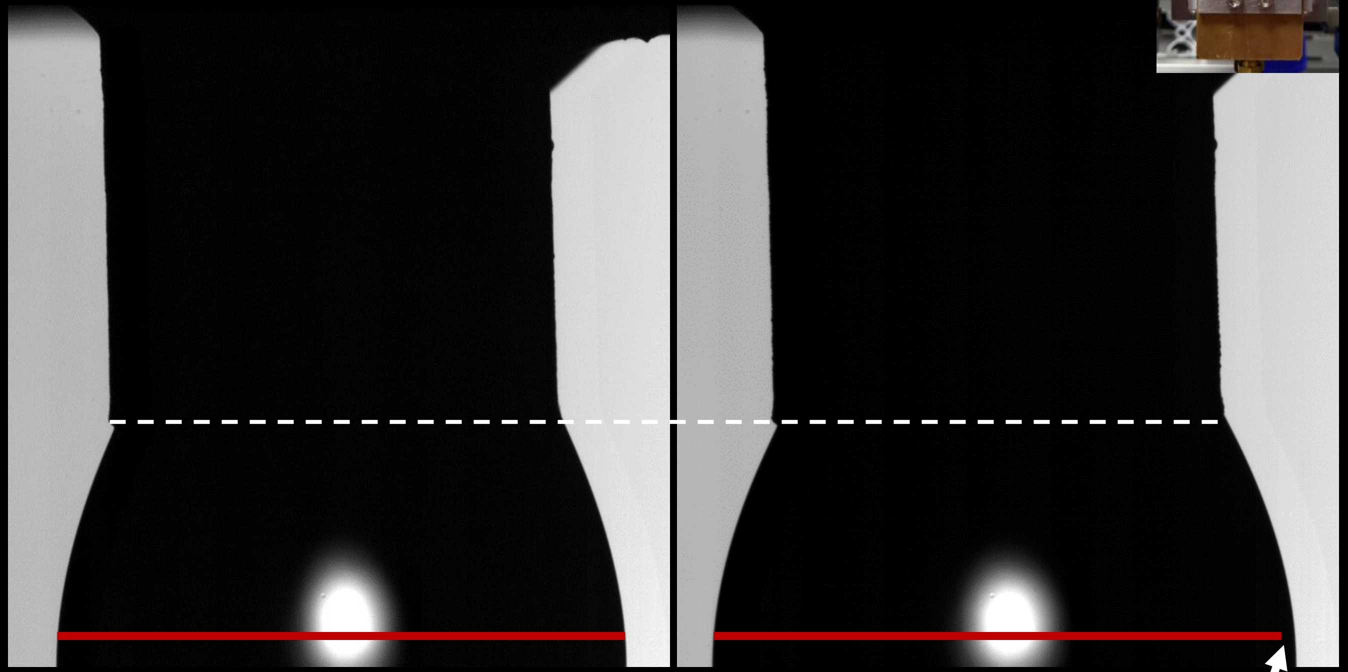
Repeatability - Controlled Environment







- Cotton saturated with water sealed with sample in CaBER
- Total test time = 1.5 hrs (longer times are possible)
- Relatively stable compared to prior conditions (swelling instead of evaporation)

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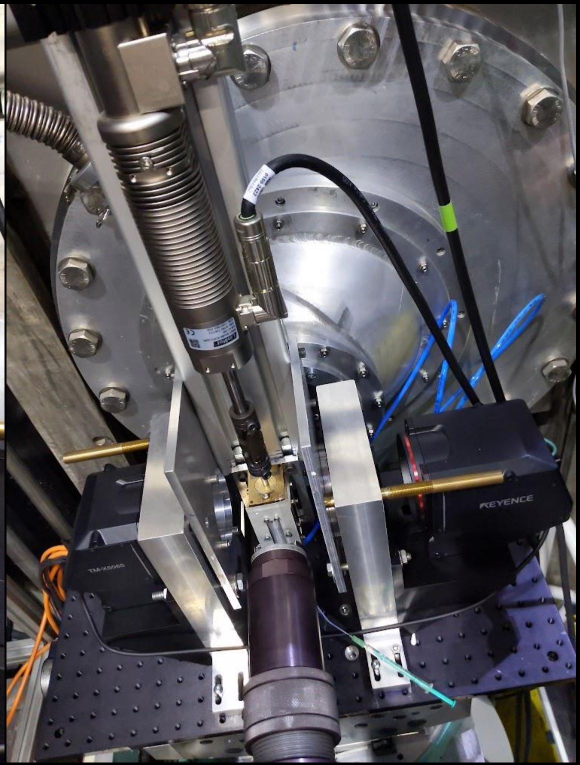
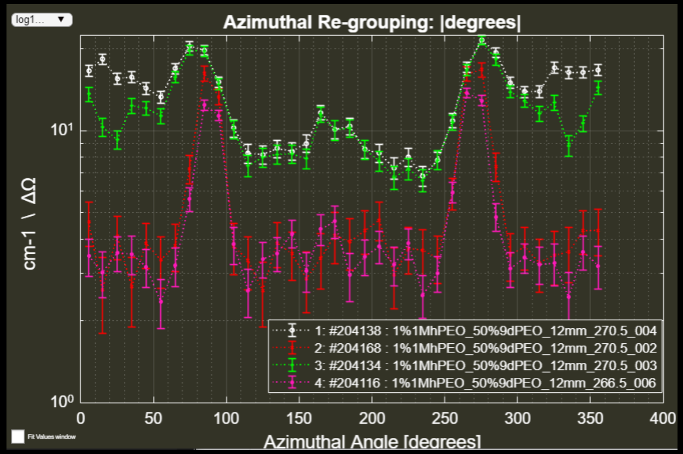
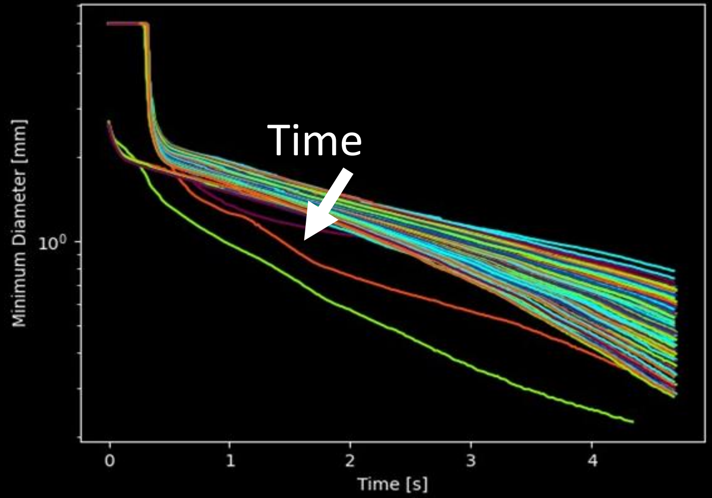
Proof of Concept at ILL D-22

- Scattering data collected 2 weeks ago
  - Significant reflections where features expected
  - Thin vertical beam centered on capillary
- Data are still being organized and processed
  - Notable changes in WLM at short times
  - Slight changes in PEO solutions
- CaBER results highlight changes
  - WLMs exhibit significant variation
  - At short times data are similar
  - Evaporation, temperature change?







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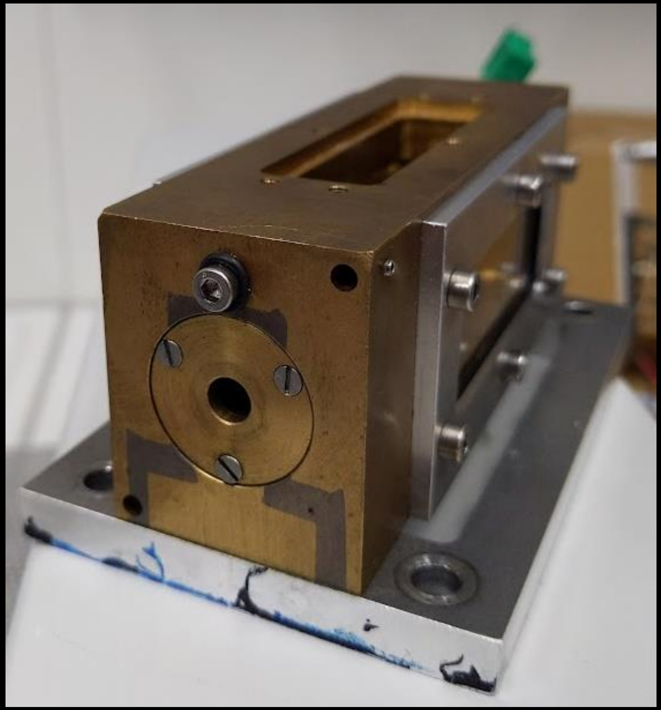
Future Iterations of CaBER-SANS

- Overhaul of sample chamber
  - Humidity and temperature control
    - Evaporation stabilized specific humidities and temperatures
    - Depending on material – 80% humid or more
  - Larger volume
  - Larger windows for lower scattering angles
  - Easier access to internal plates
  - Aluminum instead of copper

- Proof of concept tests got lucky
  - Consistent weather (rain)
  - Stable temperature



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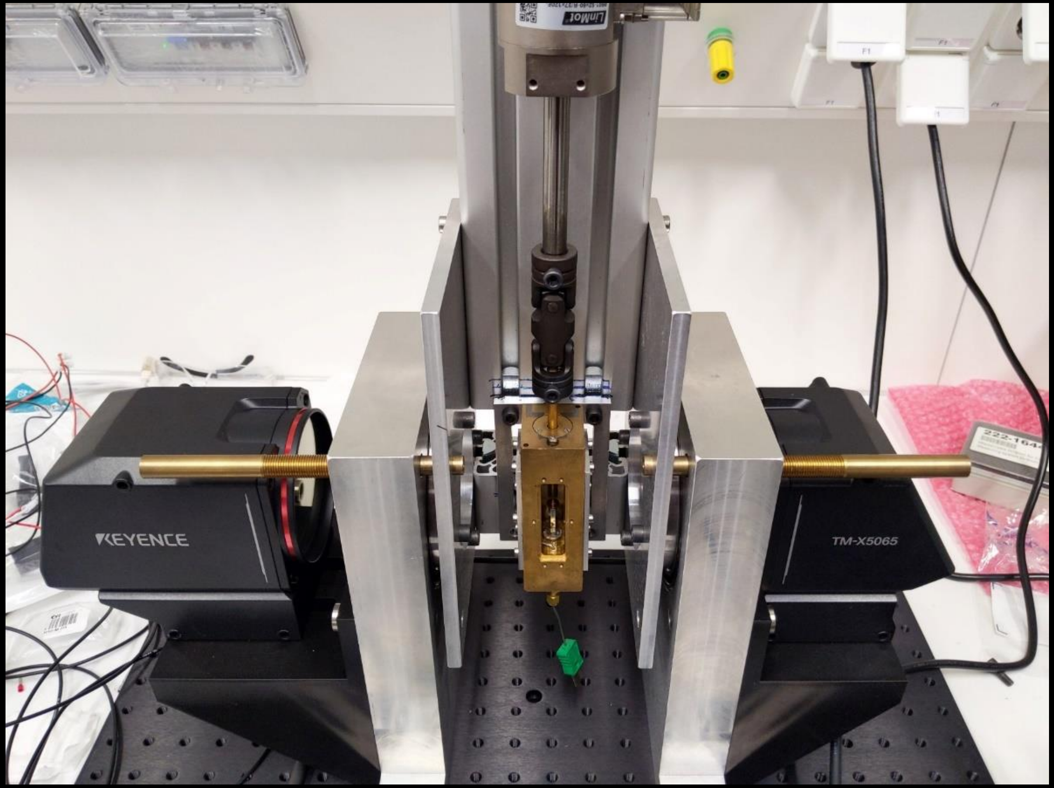
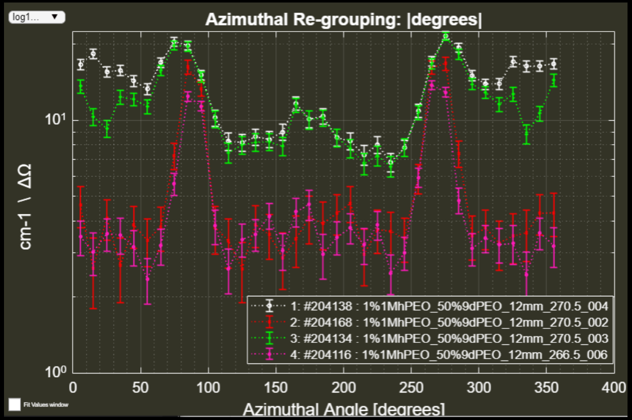
Ongoing Development of CaBER SANS

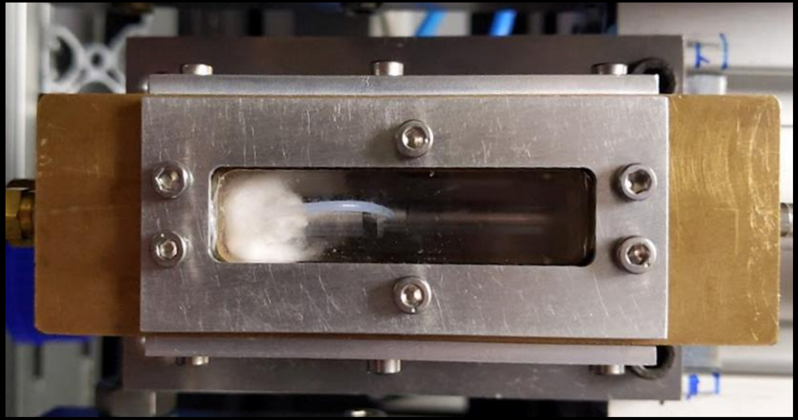
- CaBER is not an easy test to run on a beamline
  - Rapid, time-resolved phenomena
  - Challenges with sample stability

- Prototype assembled and tested on D-22, ILL
  - Data analysis are ongoing, but promising
  - CaBER results highlight variation in sample behavior

- Sample chamber overhaul
  - More accessible
  - Temperature and humidity control
  - Larger volume







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Acknowledgements

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GNeuS Global Neutron Scientists

KU LEUVEN

materialise

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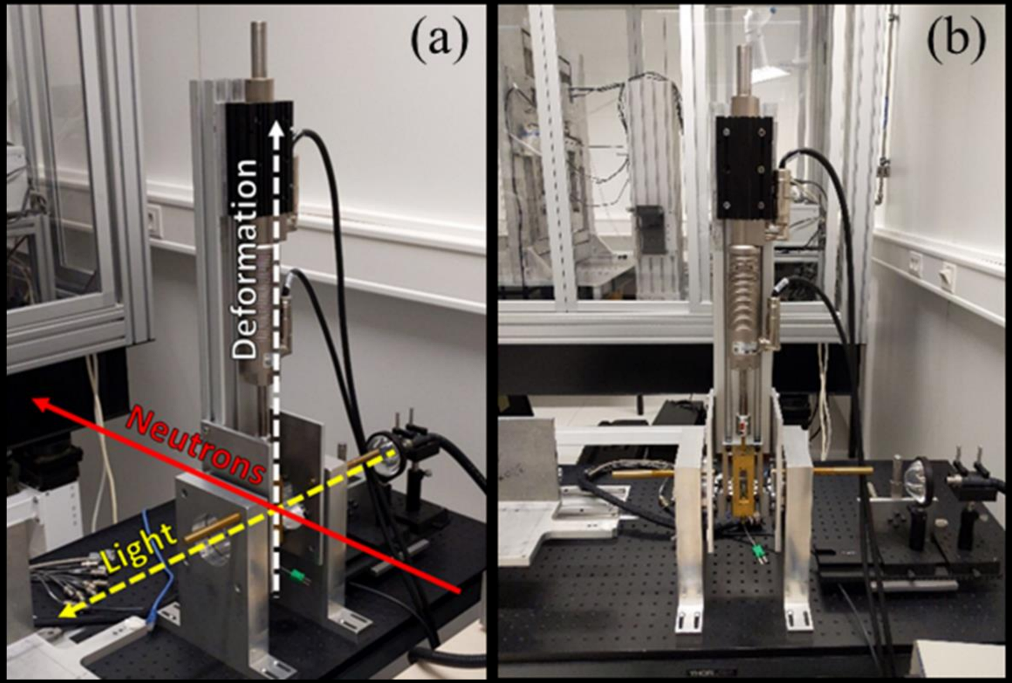

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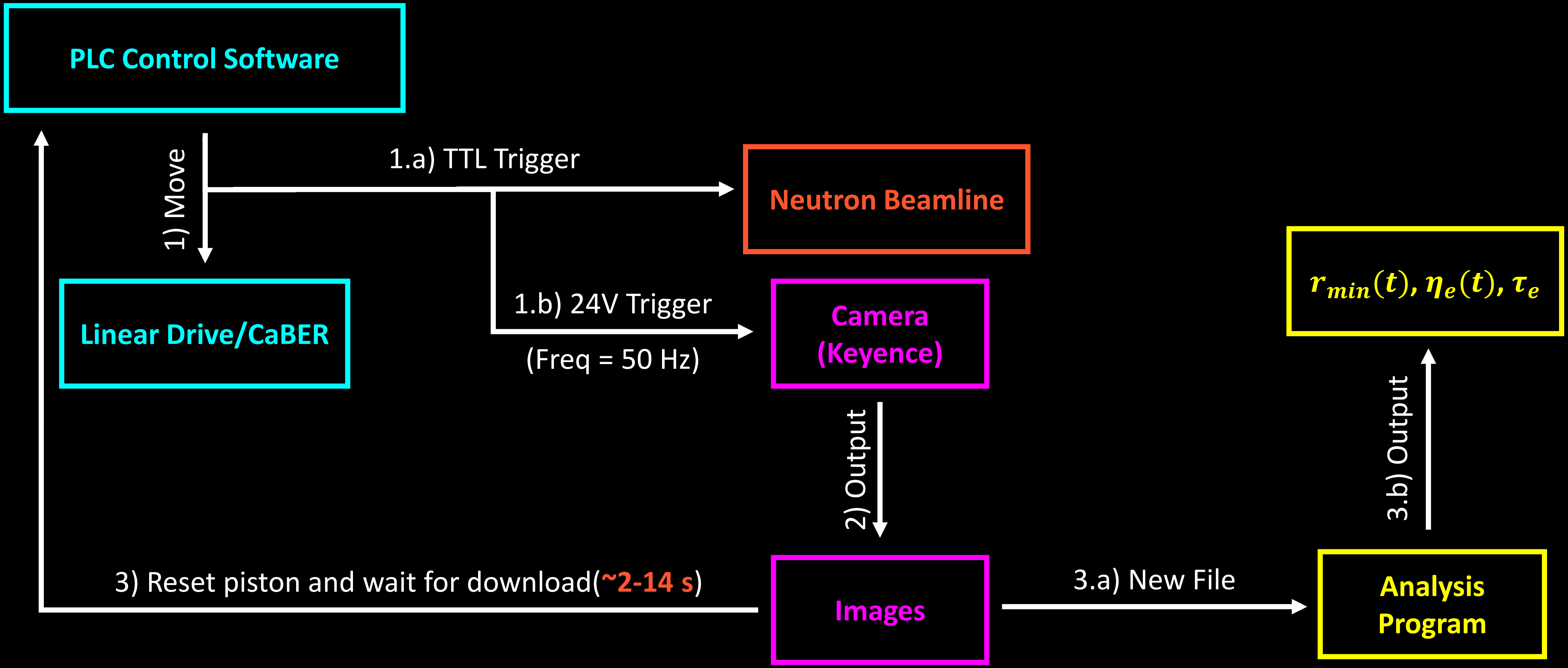
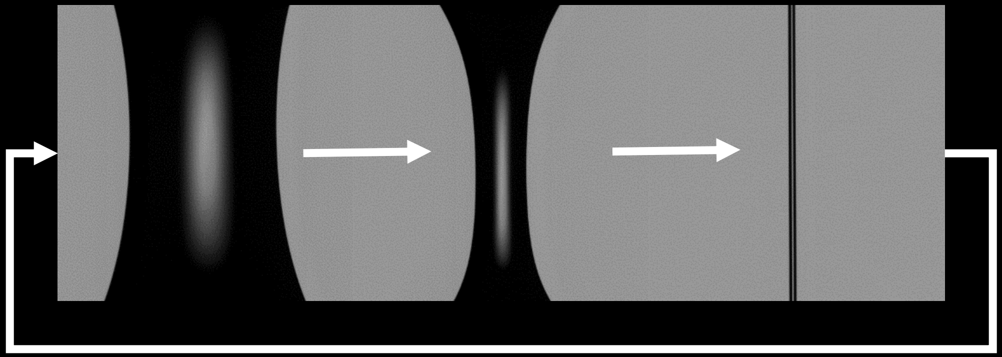
Automation – Triggering and Control Scheme



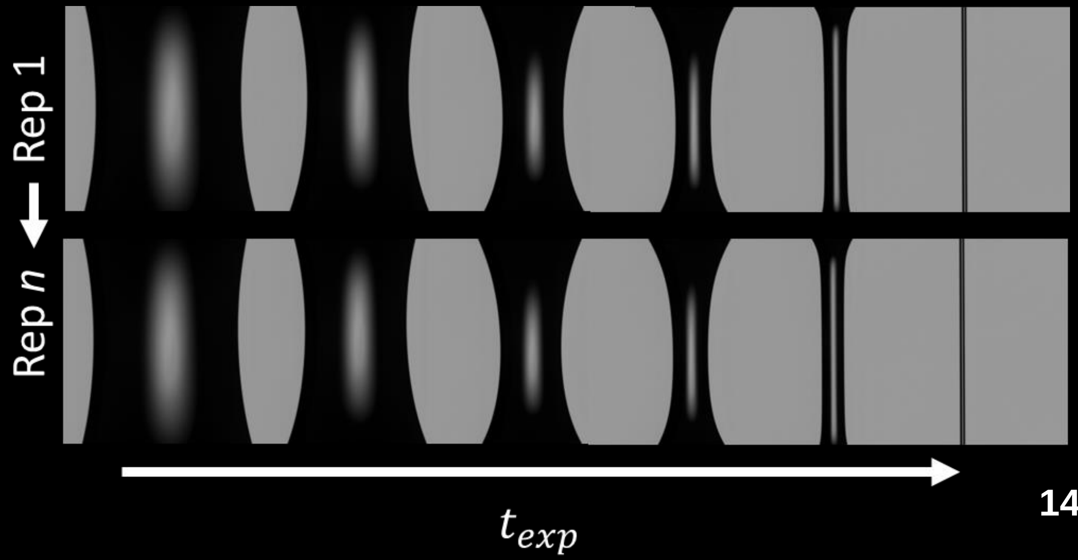
- **Repeat time limited by time to reset piston and download data or raw images**

Putting a CaBER Instrument on a Beamline

- CaBER is inherently transient
- **Automate** – Repeat test with limited human intervention
  - Limited time at beamlines
  - Sync multiple components
    - Beamline and CaBER
    - CaBER components



- **Consistent CaBER tests** – Ensure comparable flow conditions
  - Changes in sample
    - Volume
    - Composition
  - Changes in loading conditions
    - Volume
    - Edge effects



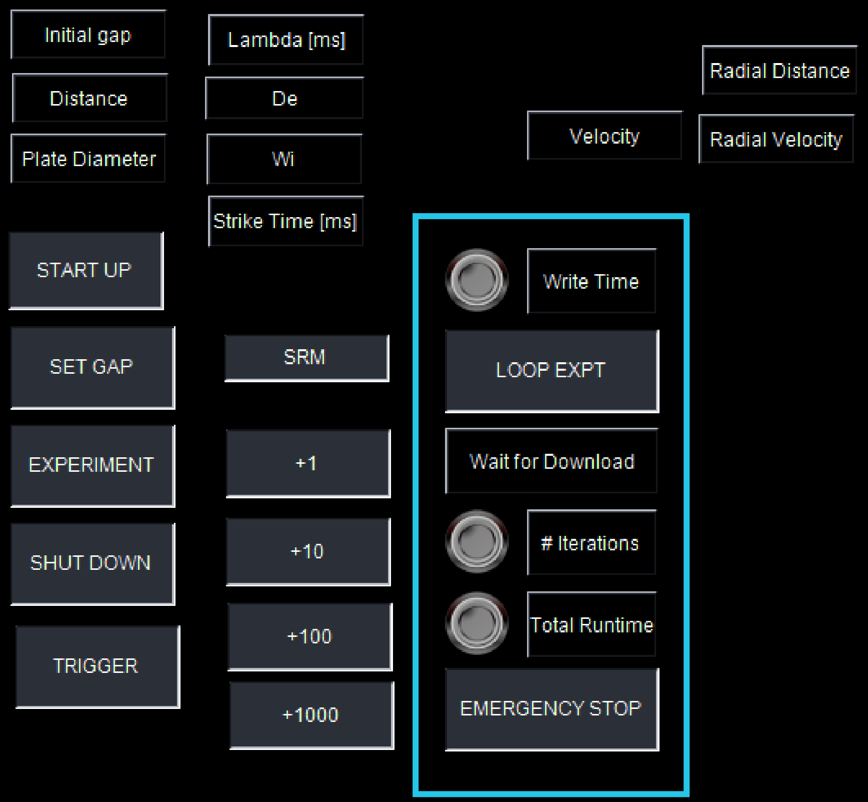
Automating CaBER – TwinCAT Looping Expt.

- Loops “experiment” and “set gap” steps
  - Wait time for download (manually entered)
  - Run until:
    - Set number of iterations
    - Time limit
  - Option to record time when “experiment” started

- Writing files on local machine from PLC
  - Specify sNetid as IP for local machine (as seen by PLC
  - Default value writes to PLC (inaccessible)

- Tests are completely automated!

TwinCAT Program Interface



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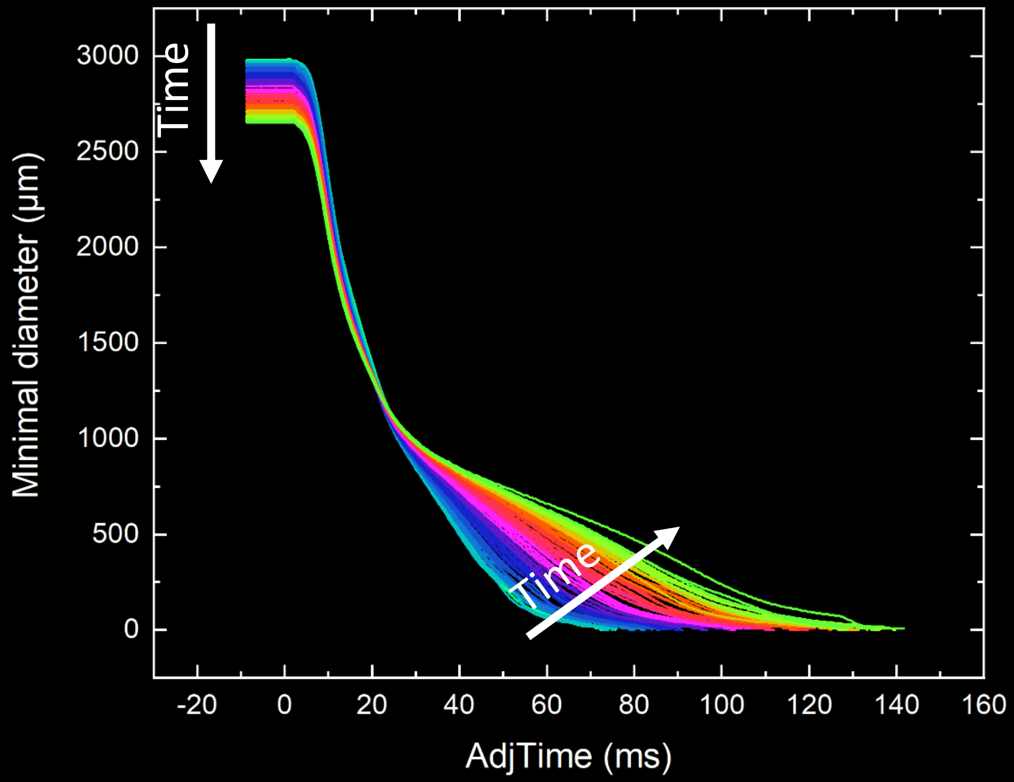
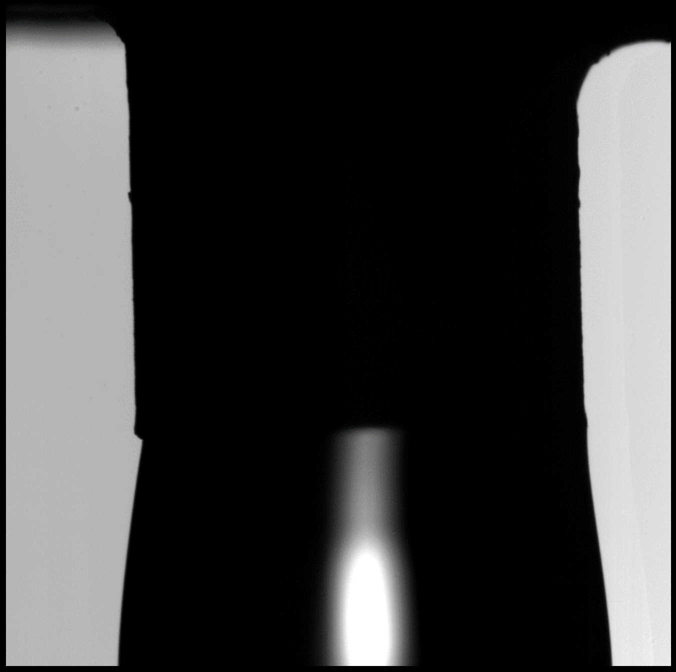
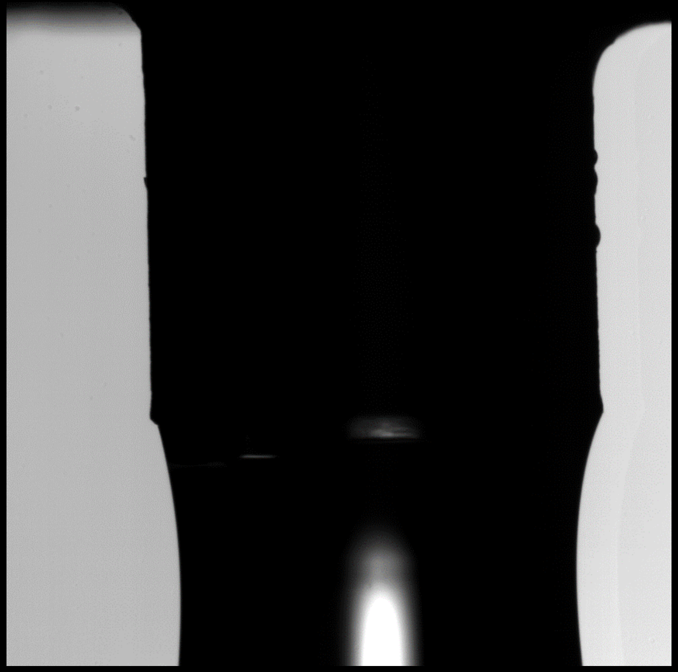
Repeatability – Sealed Environment





Test 0, Frame 0

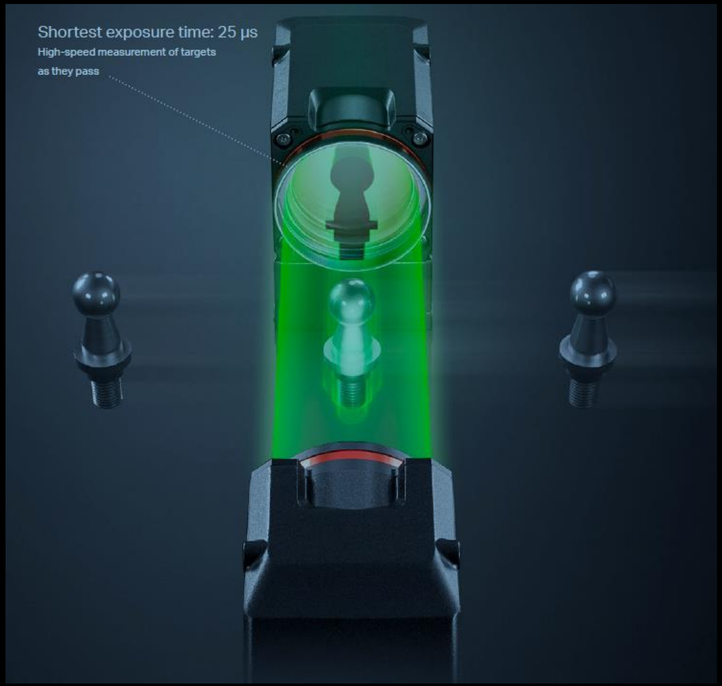


Test 66, Frame 0

- Total test time = 75 minutes
- Significantly more stable than prior tests
- Setting up sealed environment requires partial disassembly of CaBER (modify of sample chamber?)

Future Work on CaBER-SANS

- Processing ~2 TB of data from secondment
  - Stability over longer times (up to 5 hrs)
  - Alternate compositions and materials
  - “Bulk” geometries
    - Impact of loading conditions
    - Wetting effects
    - Solvent evaporation/absorption

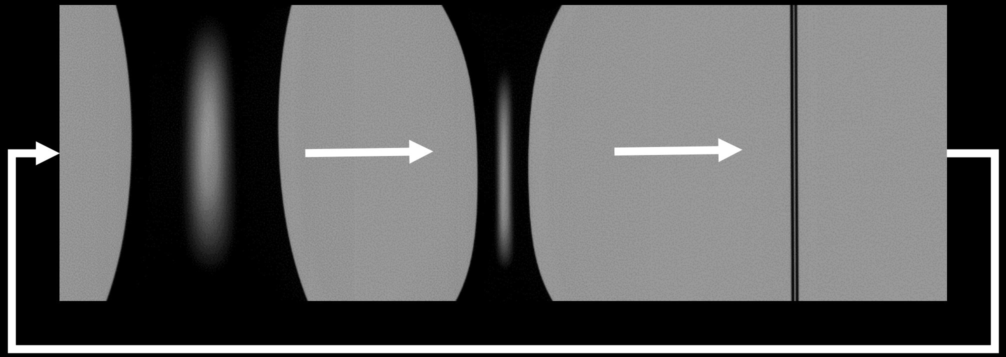


- Temporarily moving the CaBER setup + constructing new CaBER setup in Garching
  - Easier/cheaper to transport by car
  - Exchanging U.S. license for German license (Illinois and Germany have reciprocity agreement)
  - Replacement for highspeed camera
    - Camera for CaBER in Leuven also used on other instruments
    - Optical micrometer from Keyence (15-20k €)
    - Lower frame rate high-speed camera from Chronos (8-10k € with lens)

Putting a CaBER Instrument on a Beamline

- CaBER is inherently transient

- **Automate** – Repeat test with limited human intervention
  - Limited time on beamline
  - Sync multiple components
    - Beamline and CaBER
    - CaBER components



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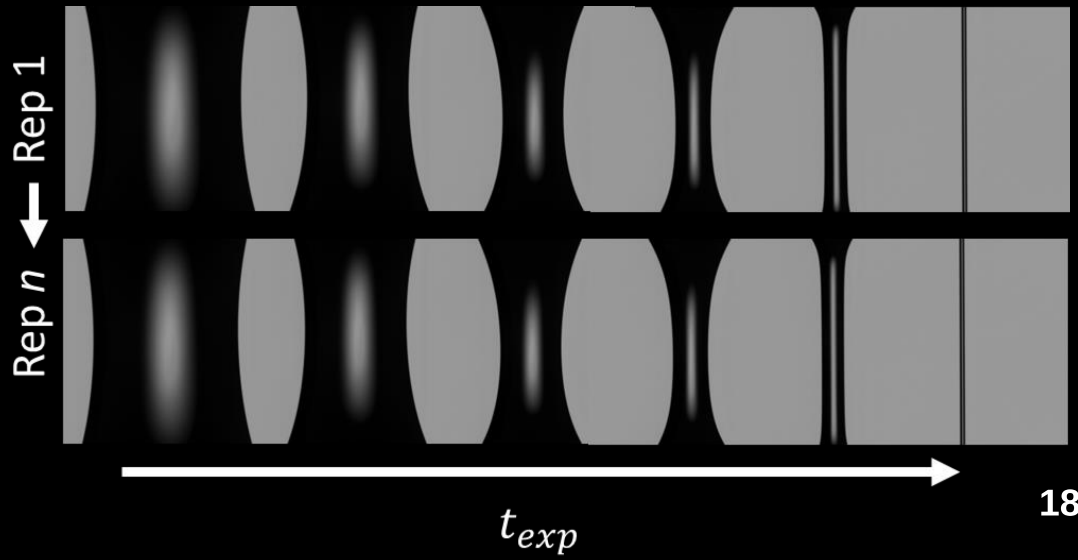

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- Tests are completely automated as of last week!!

TwinCAT Program Interface

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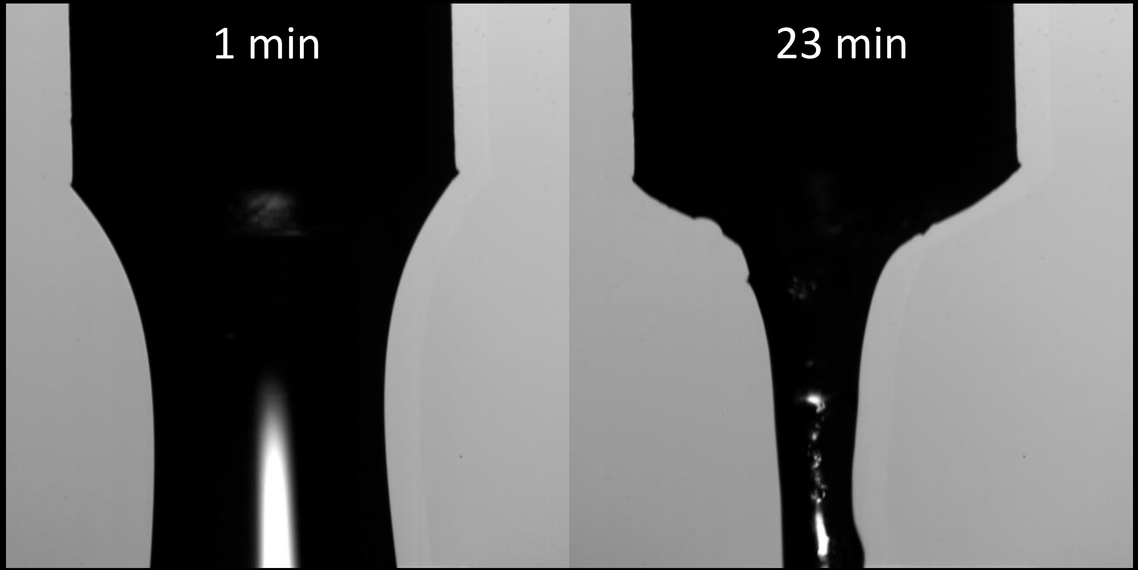
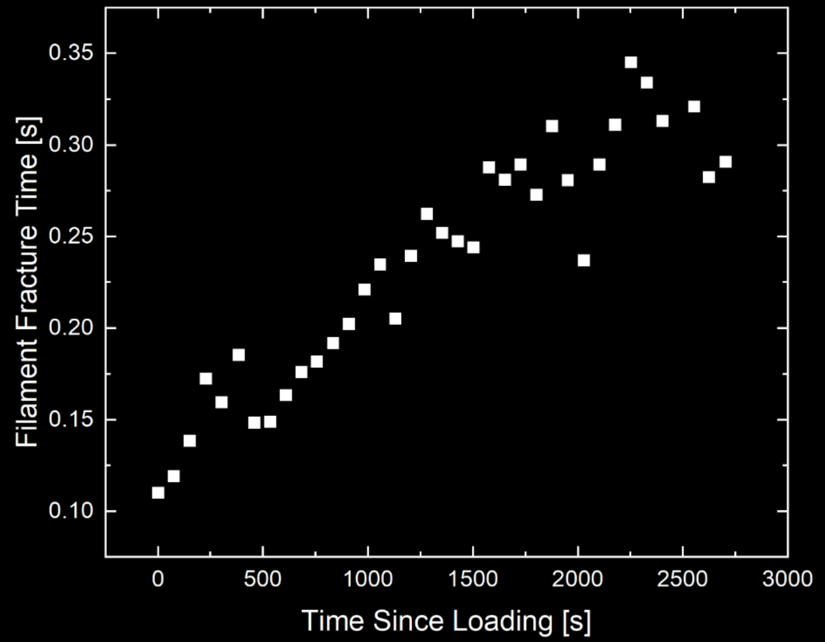
Consistent Tests – Sample Changes with Time

- CaBER experiments repeated by hand
  - One loading - 8 MDa PEO + 35 kDa PEO solution
  - 33 tests over 45 minutes

- Solvent evaporation is a significant issue
  - Significant changes in material behavior
  - Changes to loading of material

- Possible solutions
  - Solvent trap (modified liquid – liquid cell)
  - Solvent bath near geometry
  - Higher boiling point solvent





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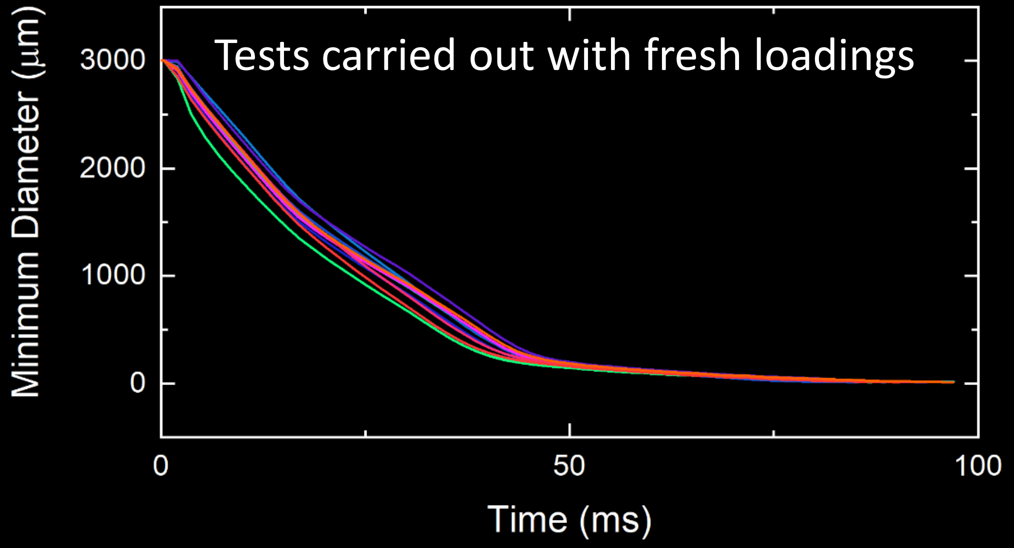
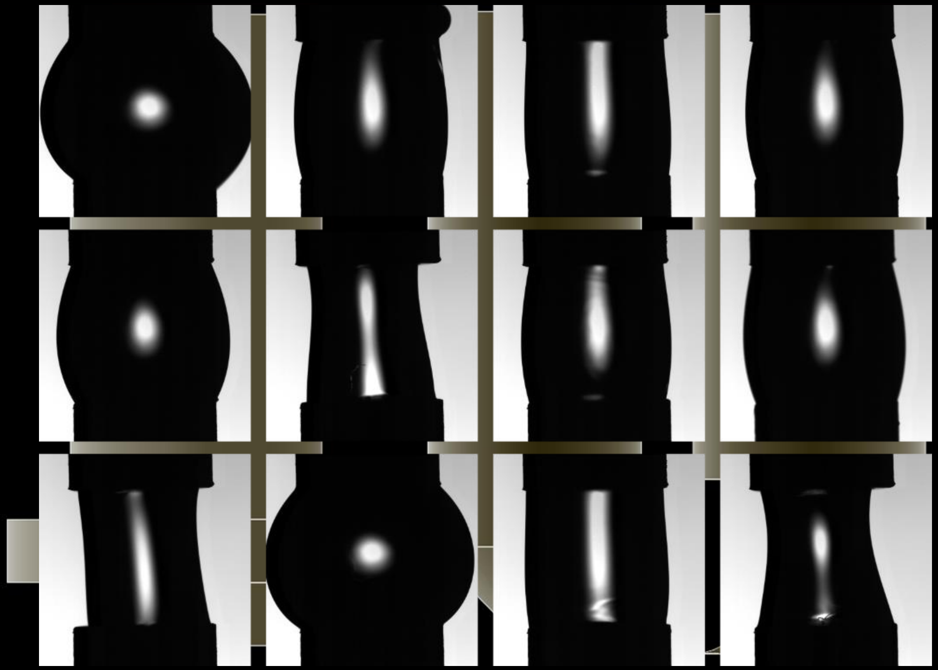
Consistent Tests – (Re)Loading Effects

- Loading has a significant impact on CaBER
  - Droplet volume
  - Centering of droplet
  - Edge wetting
- Typically run multiple tests with fresh loadings
  - Median or average as representative data set
- Possible solutions
  - Featured geometries – consistent pinning/wetting
  - Dip and pull from bulk solution
- Challenges deconvoluting from evaporation





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