Oak Ridge National Laboratory

21 January 2026

Soft Matter I: Integrating Rheometers with Neutron and X-ray Beamlines

What moves, what matters, and how it fits on a beamline

Presented by

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U.S. DEPARTMENT of ENERGY

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Main takeaways for today's lecture

01

Understand what a rheometer is and physically does

02

Recognize the major rheo-scattering modes and why they matter

03

Understand mechanical constraints

04

Know what can go wrong mechanically

05

Feel confident supporting a visiting rheo-scattering user

Why am I the one up here talking to you today?

- **Soft Matter Sample Environment Scientific Associate**
    - Develop, maintain sample environments for soft matter research
    - Polymer solutions, melts, films; Protein solutions
- **Main focus at ORNL:** Rheo-SANS (rheology + small-angle neutron scattering)
- **7.5 years of rheology/ rheometry experience**
    - Learned how to fight with rheometers (and win!)
- **Helped support rheometer experiments at ORNL, PSI, and ANSTO**

University of Minnesota

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SNS SPALLATION NEUTRON SOURCE

HFIR

PSI

Australian Government

ANSTO

What is Rheo-Scattering?

Why would we want to run such a complicated experiment?

Why do we care about soft, squishy materials

Water flows freely

[1]

Ketchup only flows when you shake it

[2]

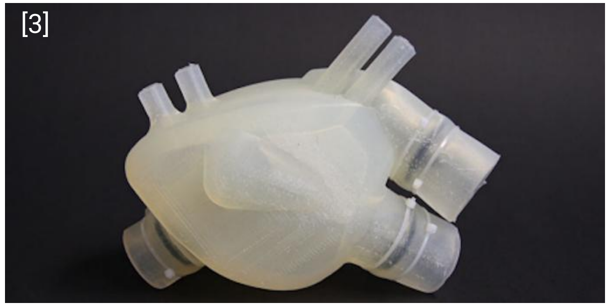
Pudding just sits there…

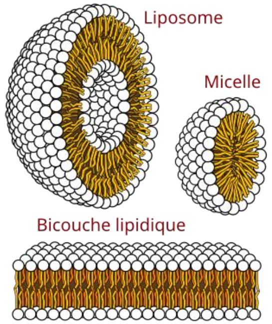
What's going on?

"They have internal structures that respond differently when you push on them."

Soft matter is everywhere!

[3]





How can we quantify how these materials are different?

How can we understand the underlying structural differences?

How do these materials' structures change under deformation (mixing, rolling, etc.)?

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[1] PicsFive/Shutterstock.com [2] Fackelmann/Amazon.com [3] Zurich heart

What is rheology?

- Rheology – study of materials under deformation and flow
  - Typically studying liquids, but also applies to solids
- “How do the properties of materials change under stress/strain?”

Stress is **how much force** to deform material
Strain is **how much it deforms**
Strain rate is **how quickly** it deforms
Viscosity is how **stress relates to deformation**

[1] πανταρει

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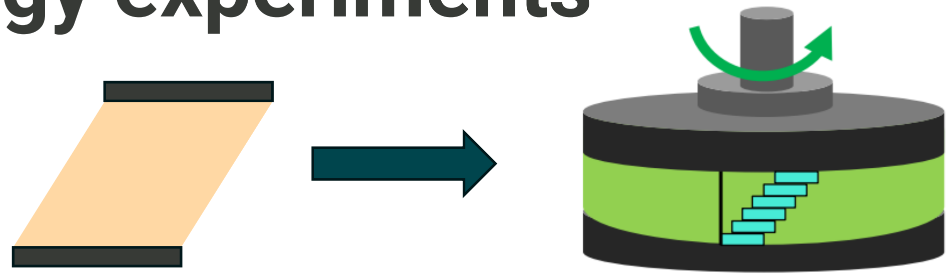
[1] Society of Rheology

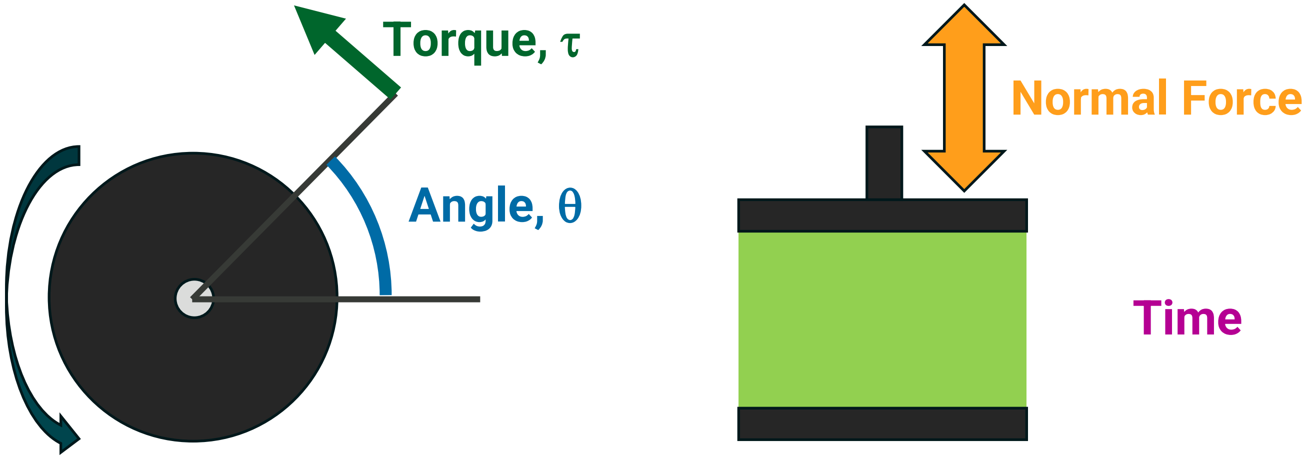
The four signals that matter in rheology experiments

Sliding plates can only move so far...

Rheometer use rotational motion to generate controlled shear

Sample is confined between two surfaces: one fixed, one rotating





Angle: how much did the plates move (relates to strain)

Torque: how much force to rotate plate (relates to stress)

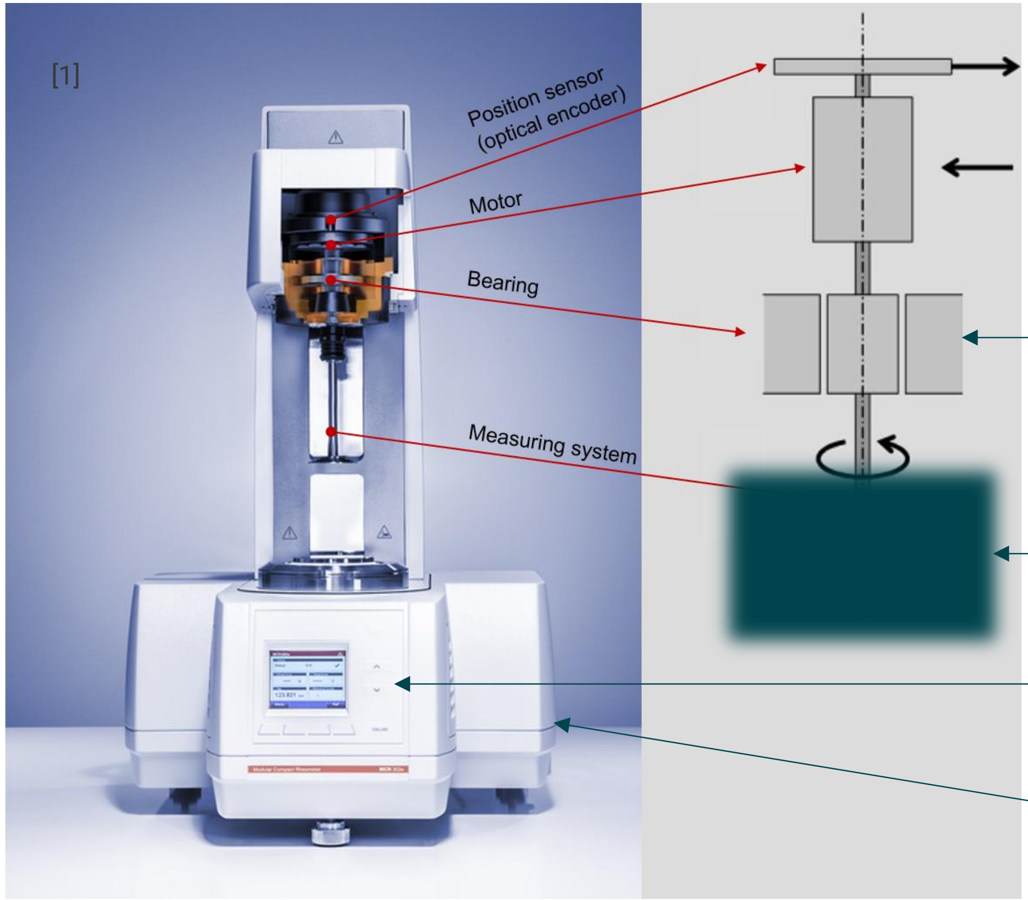
Normal force: is sample pushing on plates (relates to complex material properties)

Time: how long did it take for these to happen?

What makes up a rheometer?

A rheometer is a precision rotary actuator sitting on top of very sensitive force sensors

Most common rheometer for neutron scattering facilities – Anton Paar Modular Compact Rheometer (MCR)



Measures **angle** (θ) and **normal force** (F_N)

Applies **torque** (τ), controls rotation to the sample

Air bearing – what allows the rheometer to measure small torques, not just friction***

Sample cell

Control panel

Electronics

***The air bearing is why rheometers are so sensitive to external forces, and is the most-commonly damaged portion

***Designed to remove internal friction. It cannot remove external disturbances

A rheometer requires a connection to compressed air at ALL TIMES under operation

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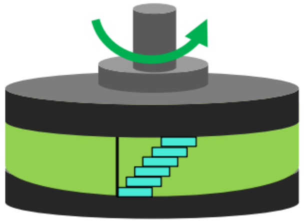
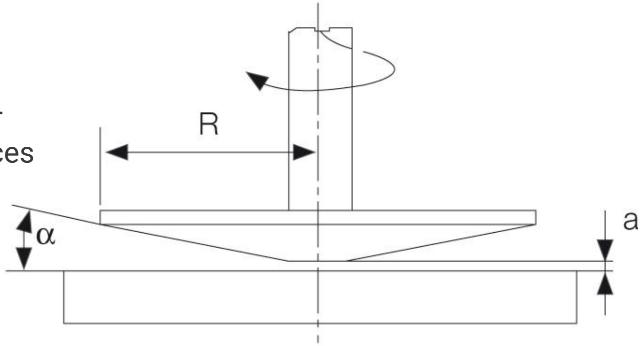
[1] Anton Paar

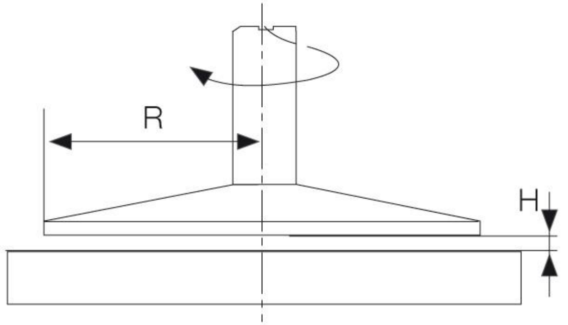
Common rheometer geometries (‘measuring systems’)



Typical for Rheo-reflectometry

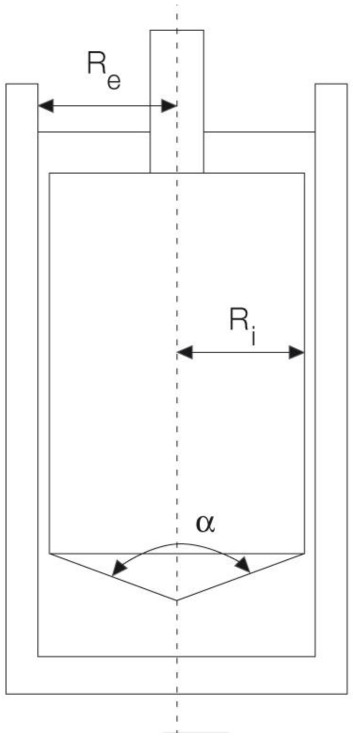


Cone-and-plate
+ Very uniform shear
− Vulnerable to interfaces



Parallel plate
+ Small volumes
+ Great for gels/soft solids
− Complex shear profile

Typical for Rheo-SAS



‘Cup-and-bob’
Concentric Cylinders
High sample contact
+ Great for low-viscosity fluids
+ Minimizes edge effects
− Higher sample requirement

Why Combine Rheology with Scattering?

- Rheometers shears a liquid, measures the material's response
- Only gives us the symptoms of what's happening, not the underlying cause
- Many equations to describe rheological behavior…

$$\eta(\dot{\gamma}) = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (m\dot{\gamma})^n}$$

$$\eta(\dot{\gamma}) = \eta_\infty + (\eta_0 - \eta_\infty)(1 + (\tau\dot{\gamma})^2)^{((n-1)/2)}$$

$$\eta(\dot{\gamma}) = K(\dot{\gamma})^n$$

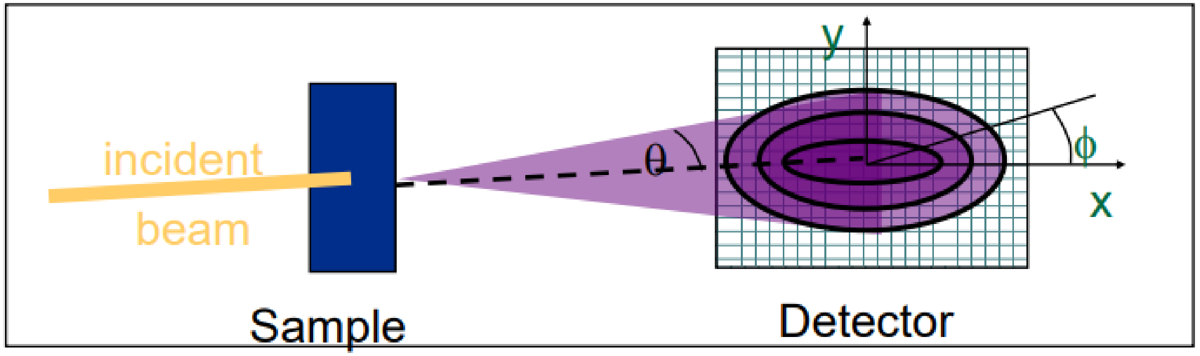
Different phenomena may give similar results

'Rheology without structure is theology'
How can we be more confident in our structure?

Scattering looks directly at nanoscale structures, tells us how molecules, particles, interfaces, or networks are arranged

'Conventional' scattering conducted at rest (static)

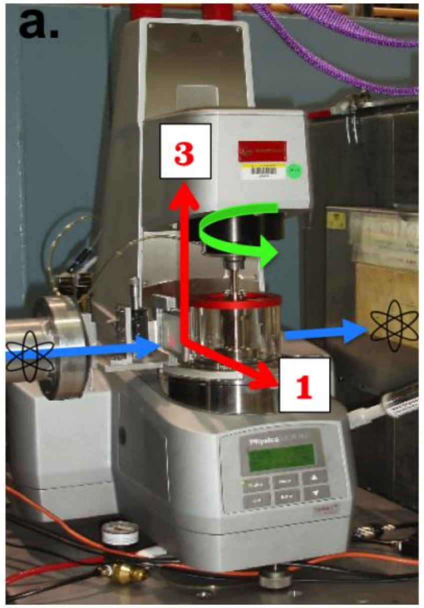
Structure alone doesn't explain how the material behaves/structures *when it flows*



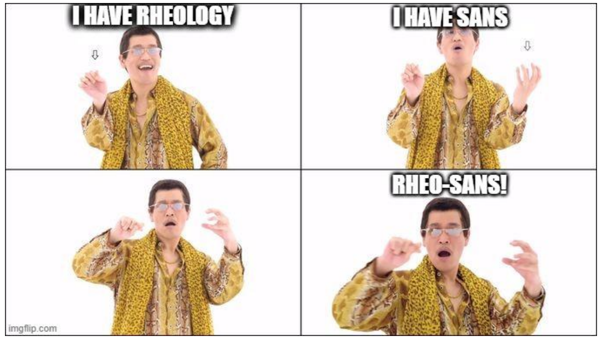
Is there a technique that can help us see structural snapshots DURING flow?

[1] Anton Paar

Rheo-Scattering: Bridging Mechanics and Structure



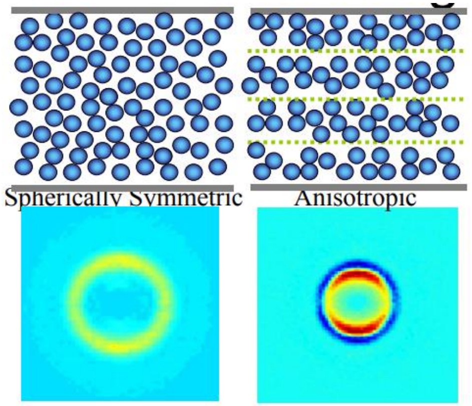
- Simultaneous measurements of flow properties and nanostructure
- Material deformed by rheometer, studied *via* scattering
- Same sample, same time, same conditions
- *In-situ,* real-time correlation between structure and macroscopic properties



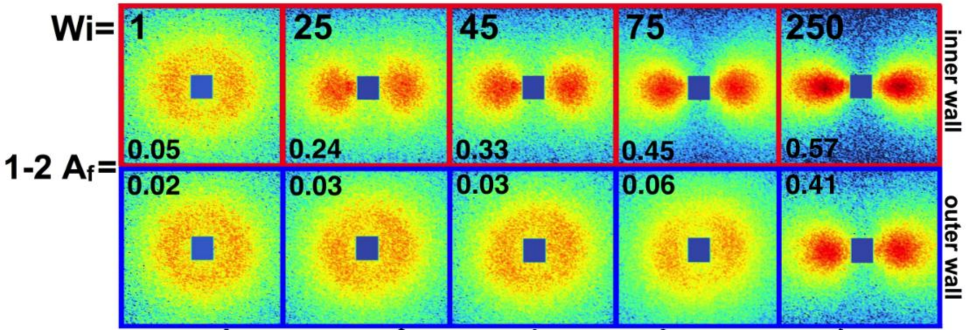
Rheology tells us how the sample flows.
Scattering tells us why.

Rheo-Scattering bridges nano → micro gap in soft matter characterization

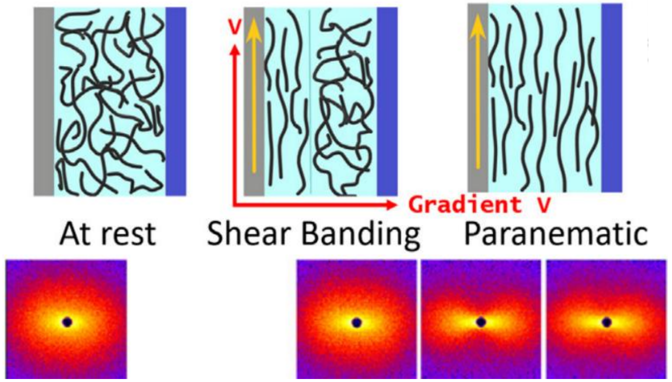
Deformation and breakup[1]



Alignment and Orientation[3]



Explain nonlinear behavior[4]



[1] Calabrese and Wagner, *RCS Soft Matter*, 2017 [2] N. Wagner, *ACNS*, 2008 [3] M. Calabrese and N. Wagner, 2016 [4] B. Arenas-Gómez et. Al, *J. Colloid. Interf. Sci.*,2020

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Where is Rheo-Scattering used?

Rheo-SANS

q~1E-3 A^{-1} to 1 A^{-1}
Bulk structural changes under shear
~1 nm – 1 µm
Polymer micelles, proteins

Rheo-USANS

1E-5 A^{-1} to 1E-3 A^{-1}
0.1 – 100 µm
Colloids, flocs, cements under shear

Rheo-NR

q_z ~ 0.005 – 0.3 Å⁻¹
1 – 200 nm
Interfaces under shear
Brush polymers

Rheo-SAXS/WAXS

q~1E-3 A^{-1} to 1 A^{-1}
Great for ordered systems
~1 nm – 1 µm
Block copolymers, liquid crystals

Australian Government
ANSTO
HFIR
PSI
Argonne NATIONAL LABORATORY
ESRF The European Synchrotron
SNS SPALLATION NEUTRON SOURCE
OAK RIDGE National Laboratory
ill NEUTRONS FOR SOCIETY
NSF CHRNS CENTER FOR HIGH RESOLUTION NEUTRON SCATTERING NIST
MAX IV

ORNL’s Rheo-SANS setup – Concentric cylinders

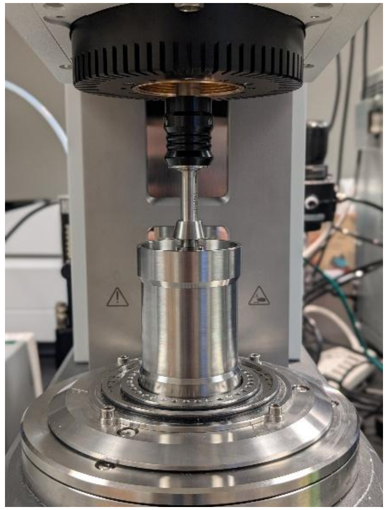
Bob diameter	Cup diameter	Total sample pathlength	Max shear rate	Volume
[mm]	[mm]	[mm]	[s-1]	[mL]
28	30	2	4000	3.74
29	30	1	8000	1.87
48	50	2	6000	21
49	50	1	13000	15

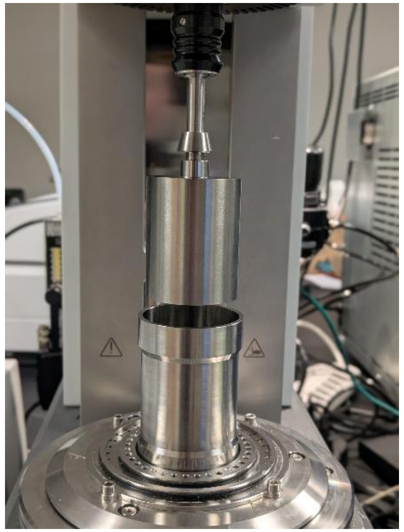
Anton Paar MCR 702e Space rheometer

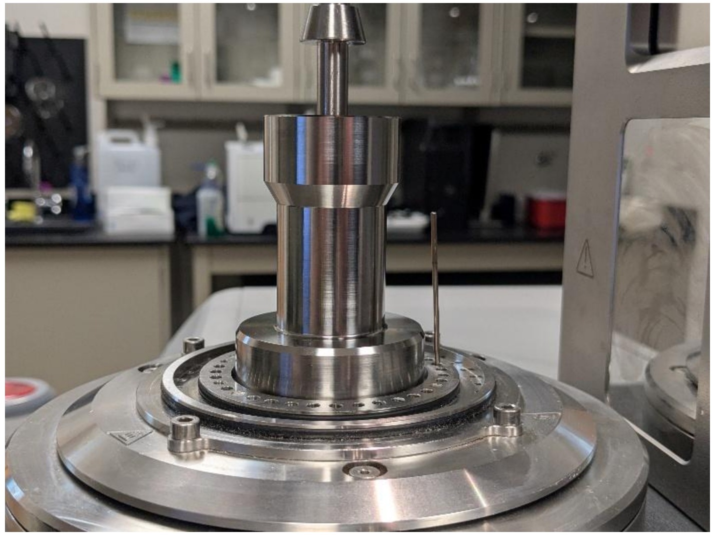
Air-temperature controlled lower measuring system (CTD-200/GL)

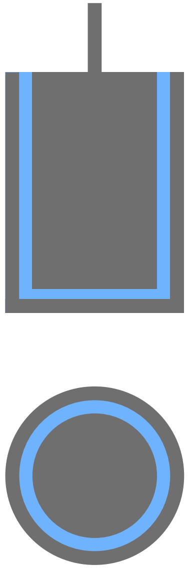
Concentric cylinder design (typically titanium, quartz available)

Multiple sizes of cup and bob – flexibility in V_{sample}, max shear rate, neutron pathlength, etc.









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[1] Anton Paar

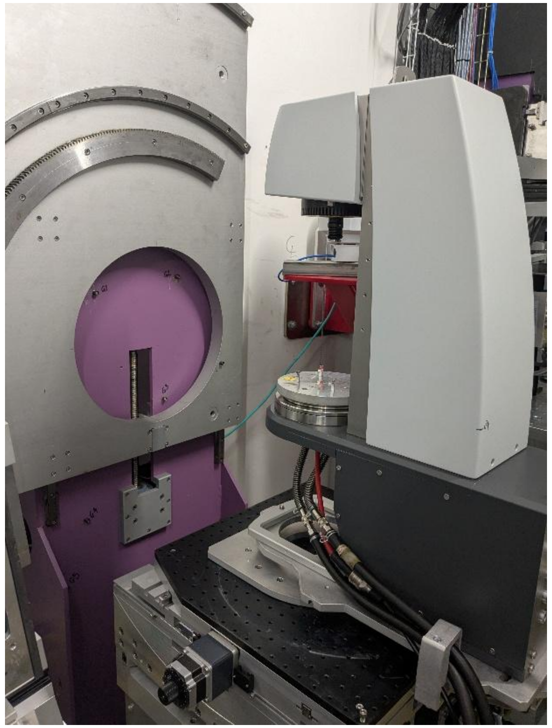
ORNL's Rheo-reflectometry setup

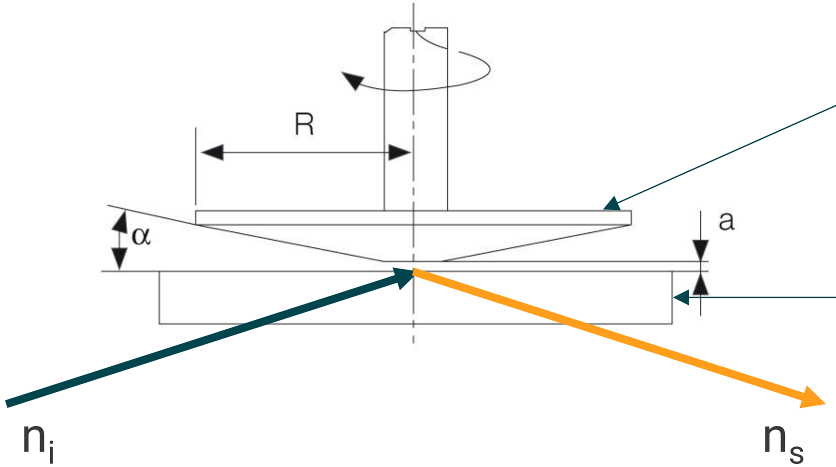
Anton Paar MCR 702e Space rheometer mounted to beamline goniometer

Peltier-temperature controlled lower measuring system (PTD-200)

Cone-and-plate design (titanium)

Multiple sizes of cone-and-plate – flexibility in V_{sample}, beam size, etc.





Ti cone measuring system

Si wafer

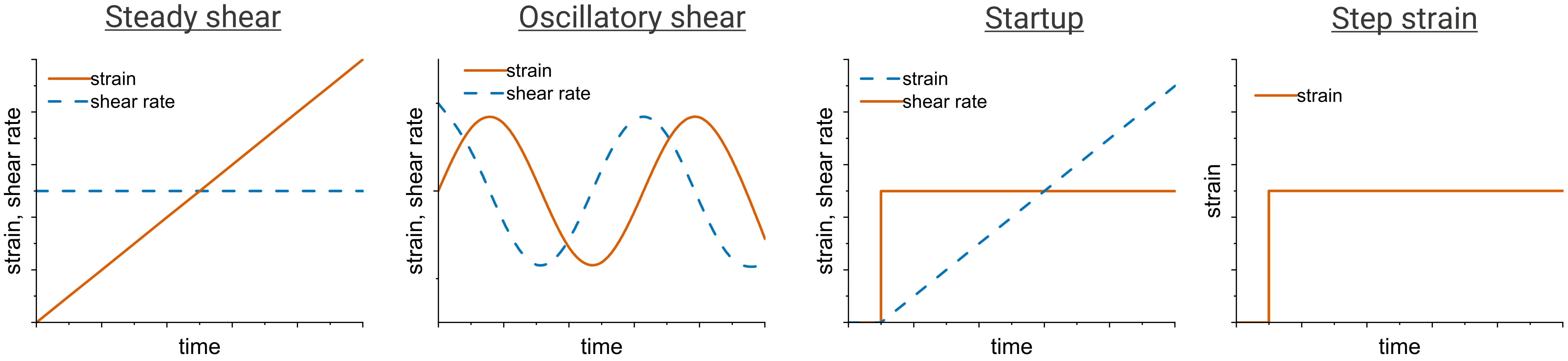
Tilt entire rheometer to access different q ranges.
Rheometer must bolt down tightly to goniometer plate

Rheo-Scattering Measurement modes

What kind of measurements are typically run?

Most common types of rheo-scattering

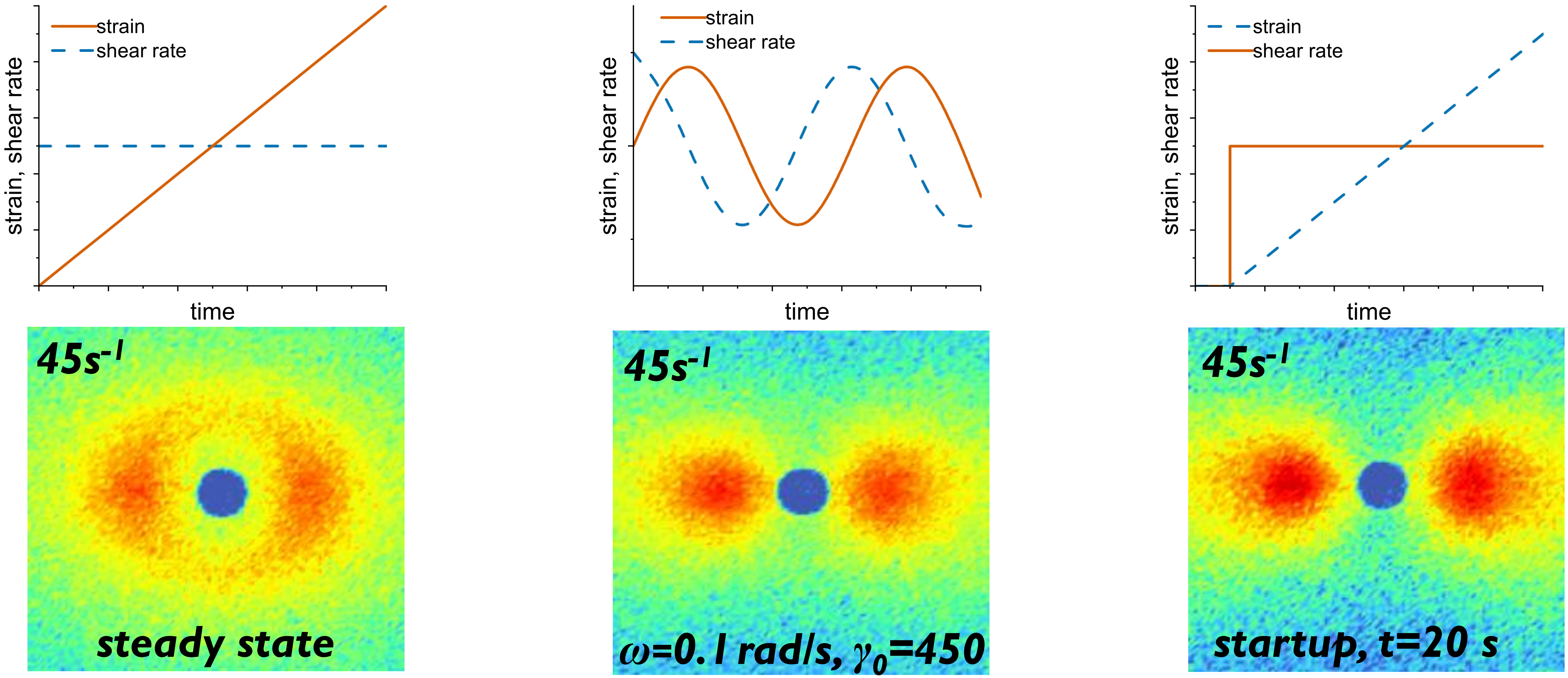
Careful selection of shear type probes structure as function of desired variable



Allows one to determine structure as a function of….

Steady shear	Oscillatory shear	Startup	Step strain
Shear rate or stress	Time, frequency, amplitude, etc.	Transient structure as f(γ)	Relaxing structure as f(t)
Steady alignment/ ordering	Reversible vs. nonlinear dynamics	Transient buildup, yielding	Relaxation, recovery

Scattering response varies with shear profile



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Credit: Prof Michelle Calabrese, UMN

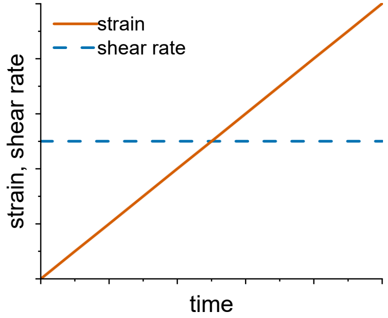
Oscillatory shear is mechanically unforgiving

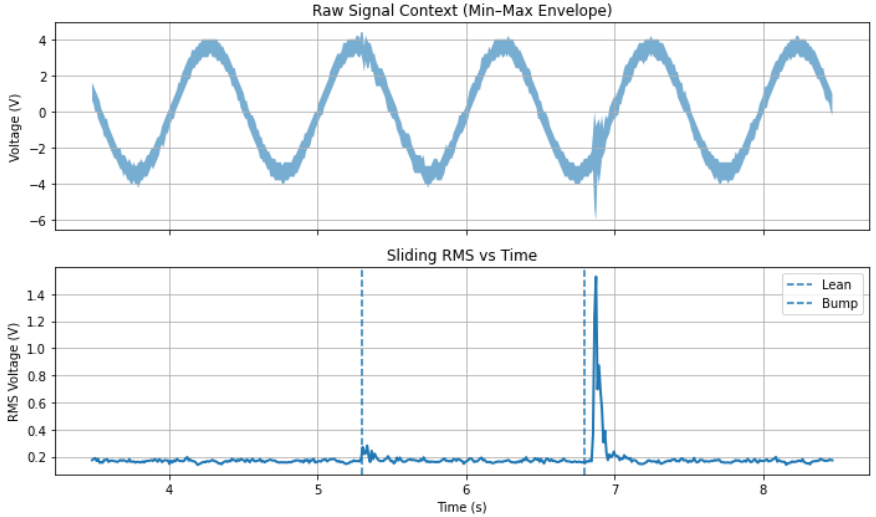
Small vibrations and compliance show up immediately in oppressive oscillatory data

<u>Steady shear</u>

Motor is just pushing along

Errors largely average out



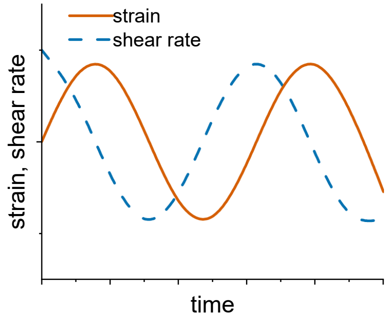


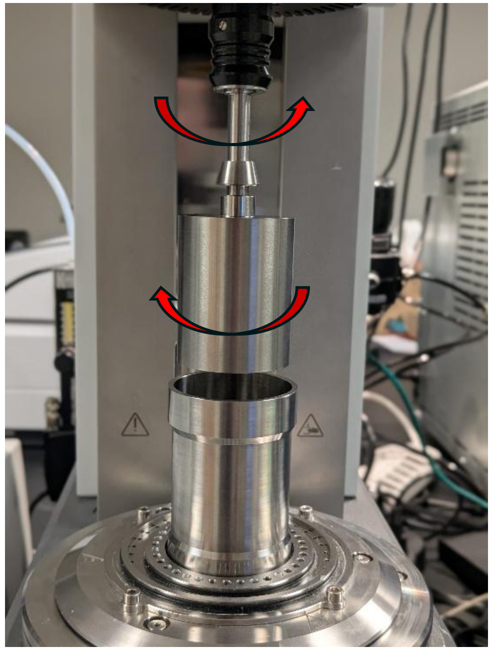
<u>Oscillatory shear</u>

Deformation is *back-and-forth, often ~10-100 Hz*

Harmonics arise, parts flex and bend

Weird stuff happens





Bumping table/instrument causes CLEAR affects on deformation/response

If steady shear is a cruise on a smooth road, oscillatory shear is driving over speed bumps at 60 mph; every bump shows up in the recording.

Mechanically integrating rheometer

How to fit the rheometer ON the beamline?

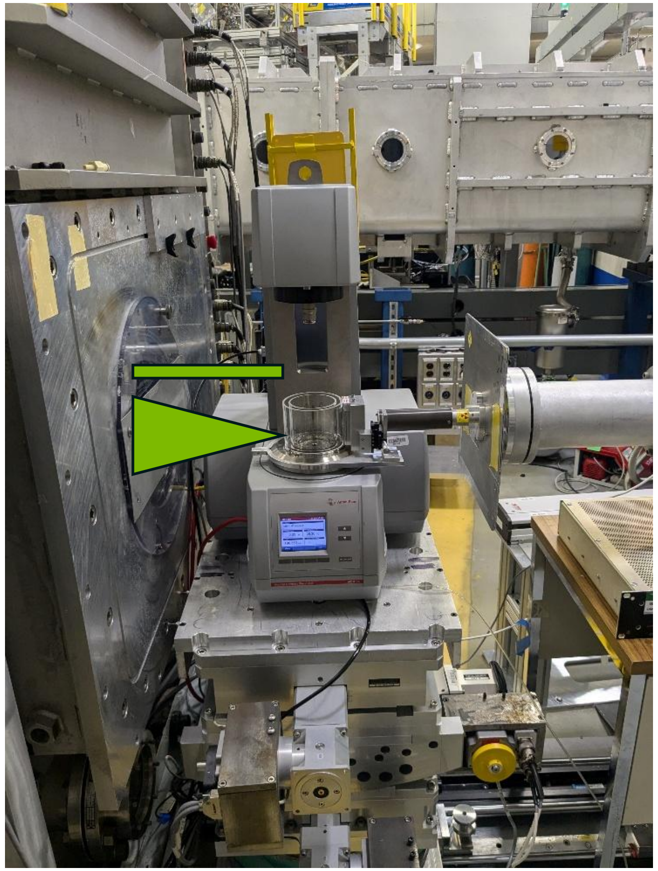
Perpendicular to neutron beam for free path

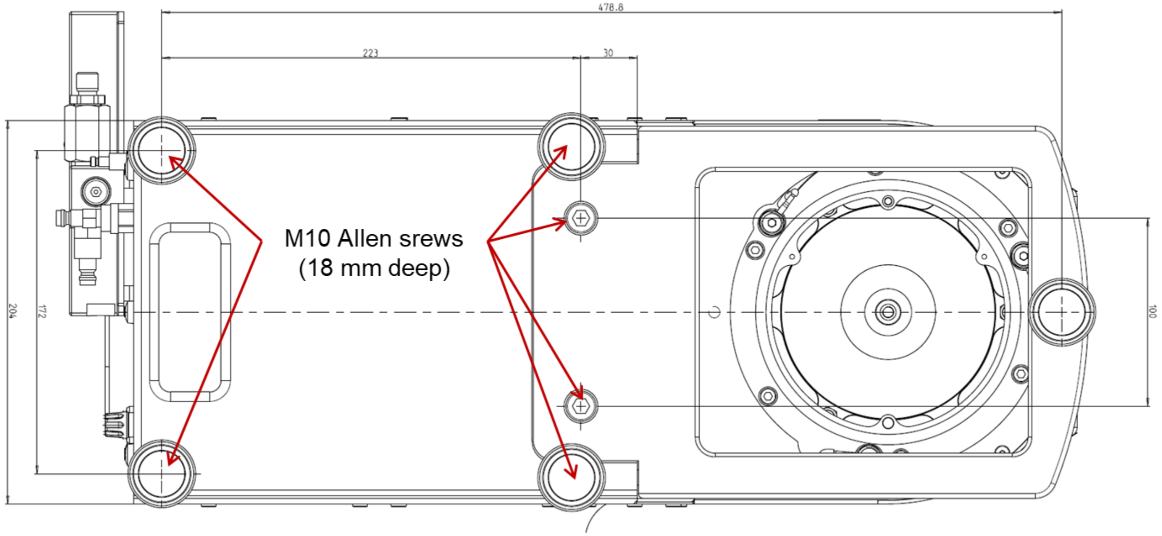
Rheometers relatively wide– allows for beam divergence, ↓ q_{max}

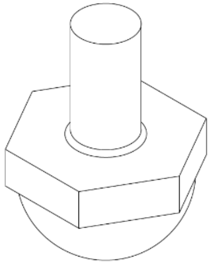
Place rheometer as close to detector tank as possible

Replacing rheometer feet with kinematic mounts makes placement reproducible!

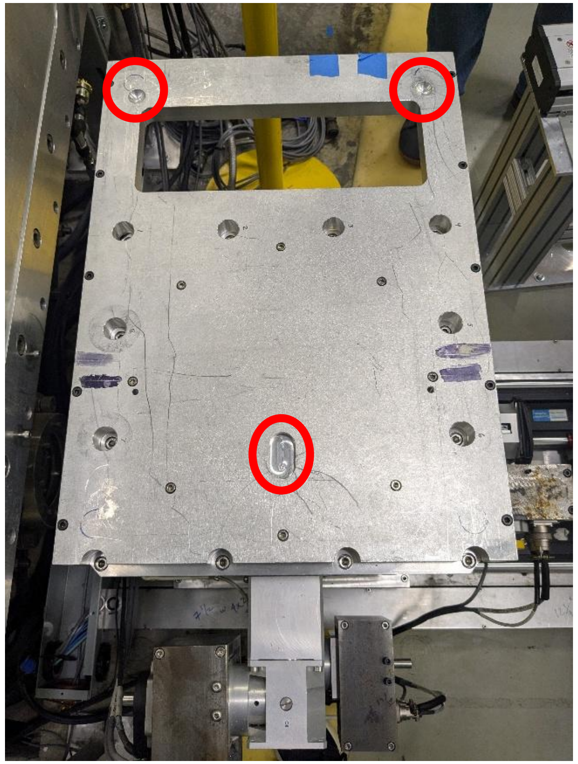
<u>MCR 302 mounted on CG-2 (GP-SANS)</u>







Kinematic feet for reproducible placement

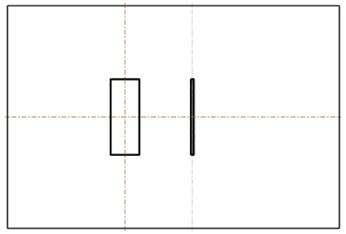


Shear planes in Couette Rheo-SANS

Couette geometry results in three separate 'shear planes'

The choice of plane determines which structural features we can resolve

Aperture with radial, tangential slits



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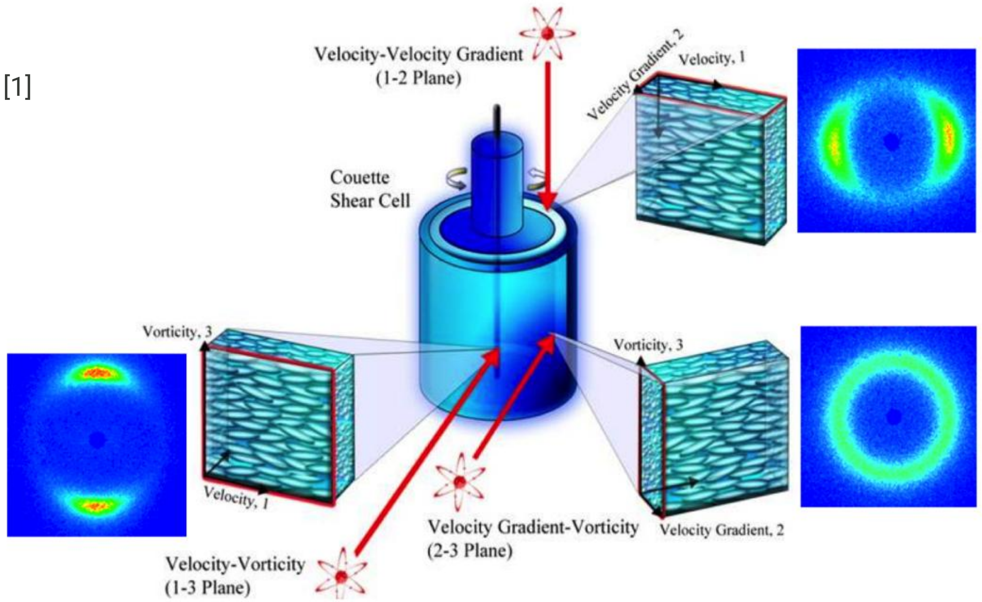

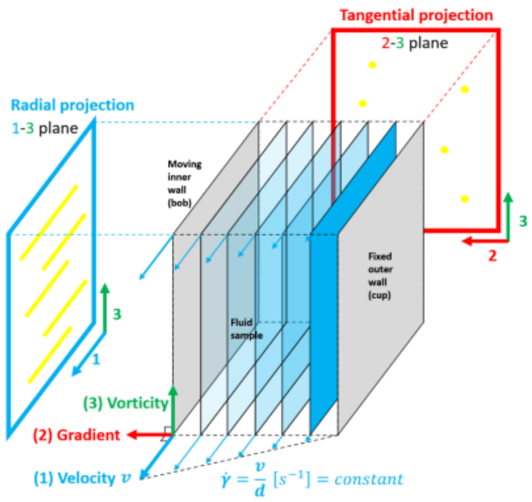

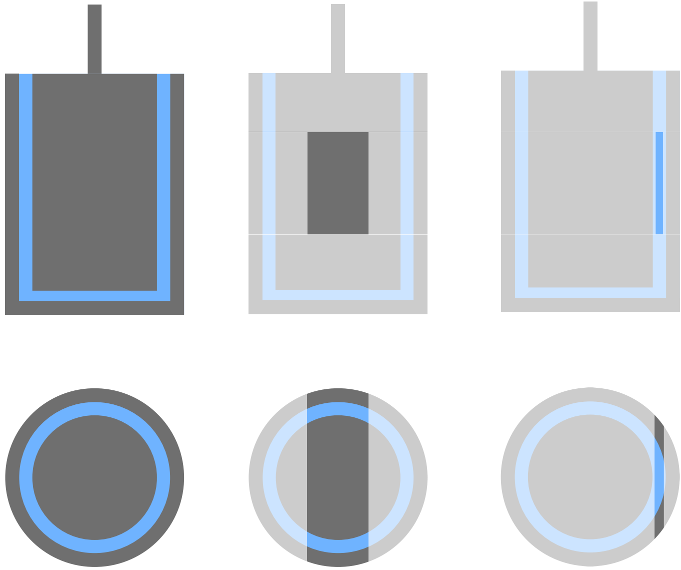

<u>Flow-Vorticity (1-3 plane, 'radial')</u>

- Probes structural changes in flow direction
- Simple interpretation of alignment
- Limited info about gradient direction

<u>Gradient-Vorticity (2-3 plane, 'tangential')</u>

- Probes structural changes perpendicular to flow
- Sensitive to shear-induced anisotropy
- Difficult to interpret for strongly aligned systems

Ability to translate rheometer relative to beam critical

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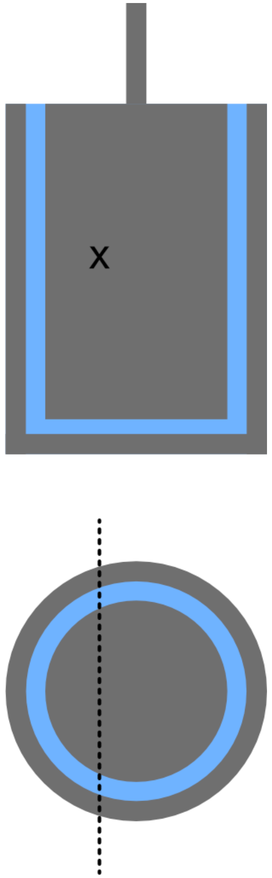
[1] M. Calabrese and N. Wagner, adapted from L. Porcar et al., *Rev. Sci. Instru., 2011*

Alignment axes – lots of moving parts!

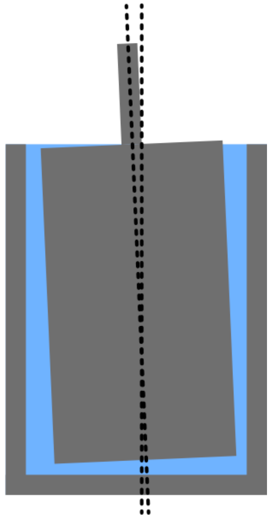
Radial concentricity and axial parallelism the most critical!

You can 'get away' with some misalignment in the others

Radial concentricity
(bob vs. cup centerlines)

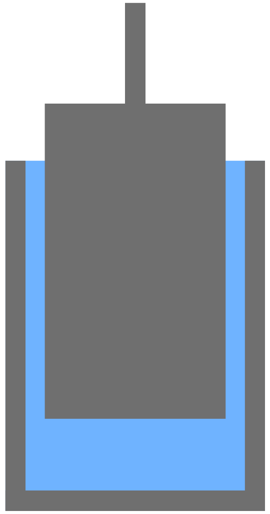


Global positioning relative to beam

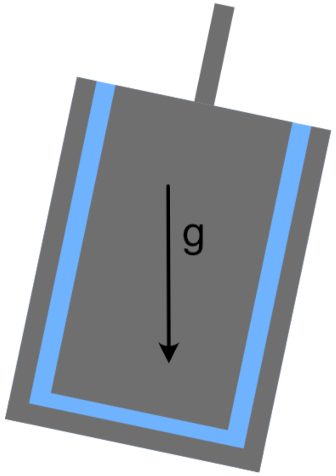


Axial parallelism
(bob vs. cup axes)

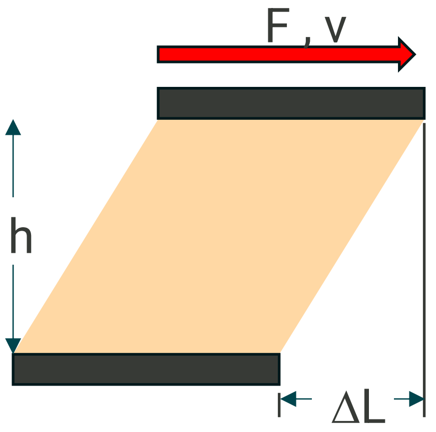
Vertical positioning/ immersion depth



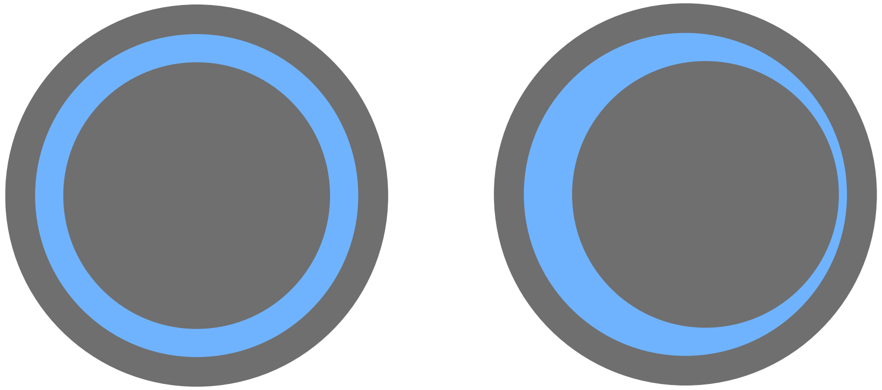
Tilt of entire instrument relative to gravity



Alignment: What matters and what doesn't

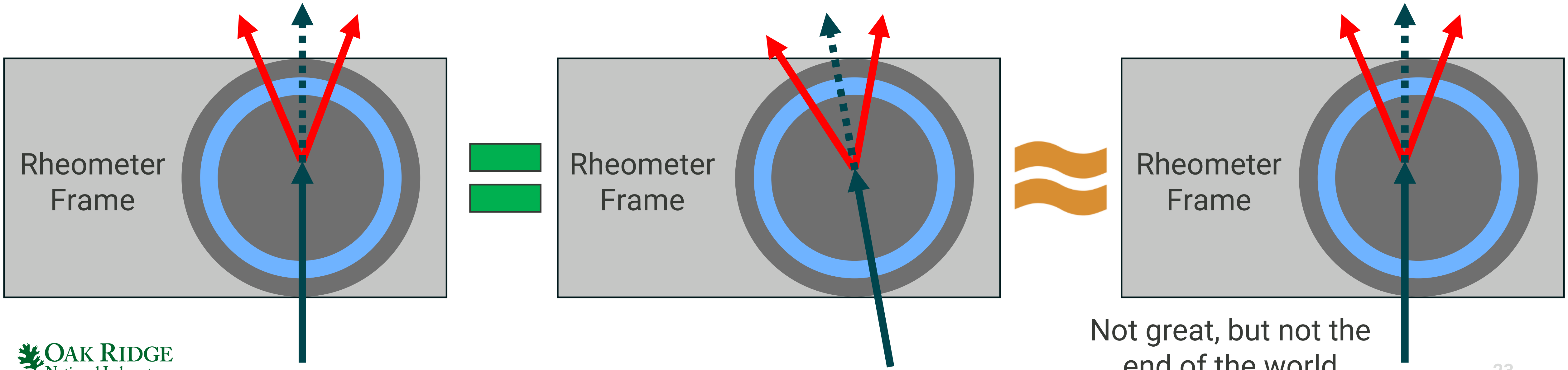


Strain rate, $\dot{\gamma} = \frac{v}{h}$



Varying gap = varying strain rate = varying structure
VERY VERY BAD

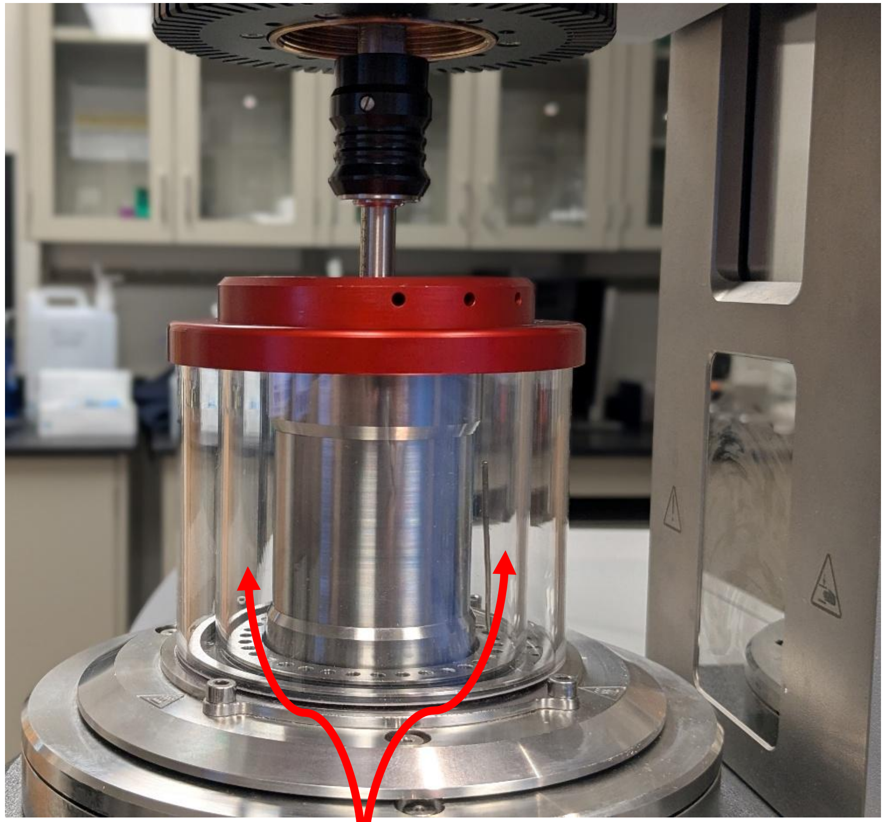
If the cup and bob are truly concentric **and** parallel, the experiment is far more tolerant to where the rheometer sits on the beamline than you'd expect

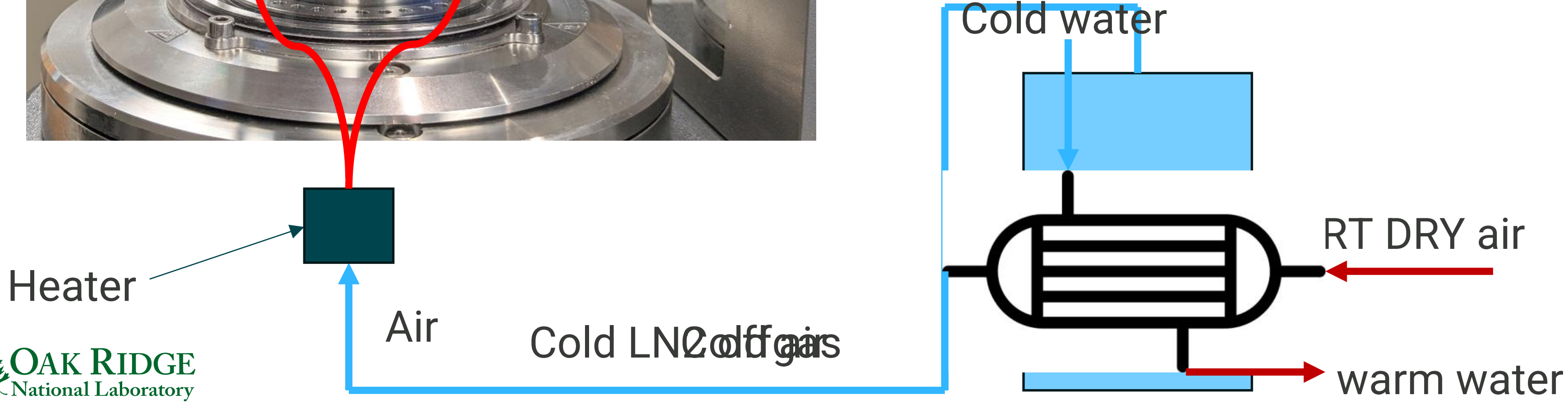


Not great, but not the end of the world

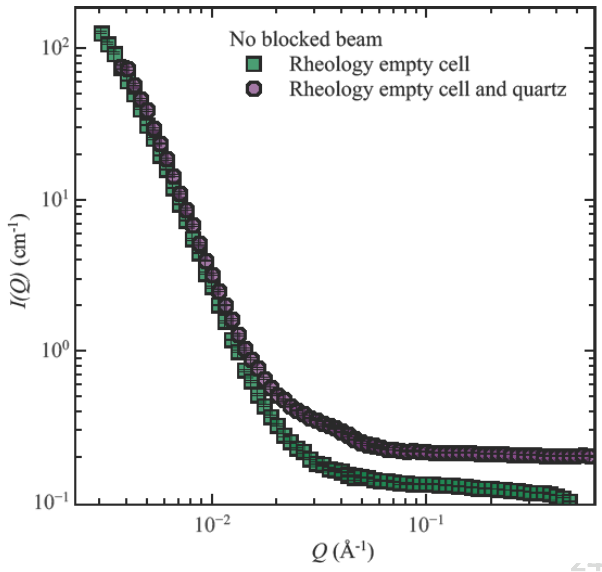
Environmental control on the Rheo-SANS setup

Rheo-(U)SANS: Convection via *heated* air/nitrogen





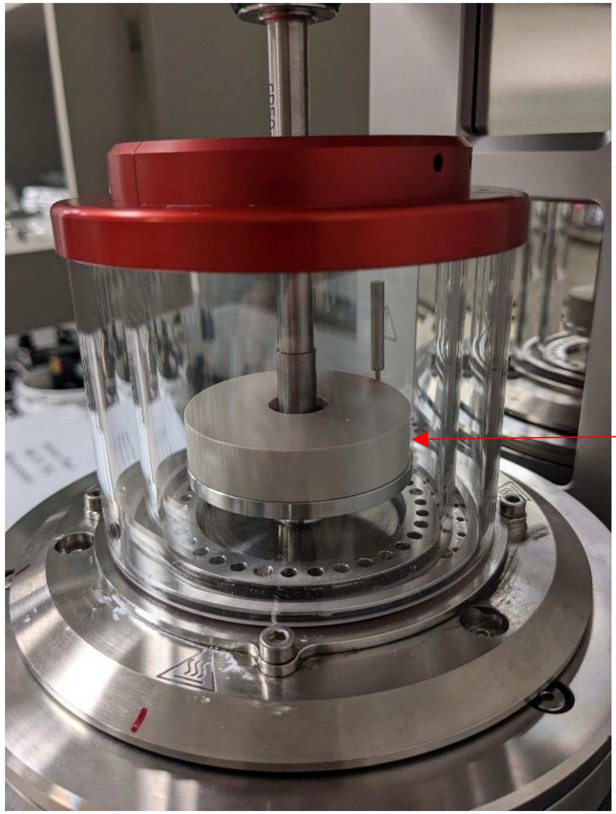
- Accessible temperature range – Ambient to ~200 ºC
- No cooler – can only raise temperature
  - Anton Paar recommends using LN2
    - ~100- 230 L LN2/day
  - For ~10 ºC-ambient, use heat exchanger + chiller
- Slow changes, ESPECIALLY when cooling!
- Quartz rings do add some background scattering



Temperature control on neutron reflectometry + rheology

Anton Paar sells Si wafer mount to use with convective temperature control system

At ORNL, we prefer mounting on Peltier-temperature control unit below Si wafer



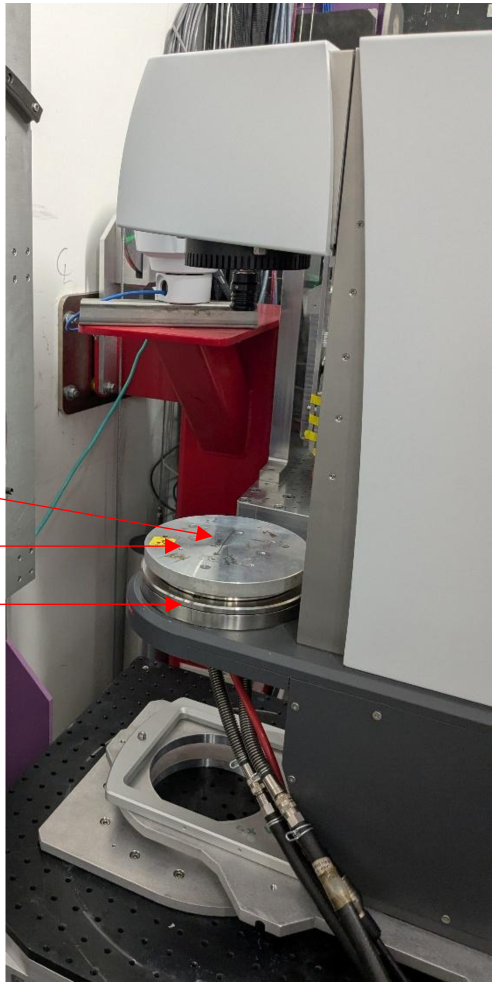
(missing Si wafer)

Both systems have advantages, Peltier is faster to respond

(missing Si wafer)

Aluminum plate w/ mounting arms

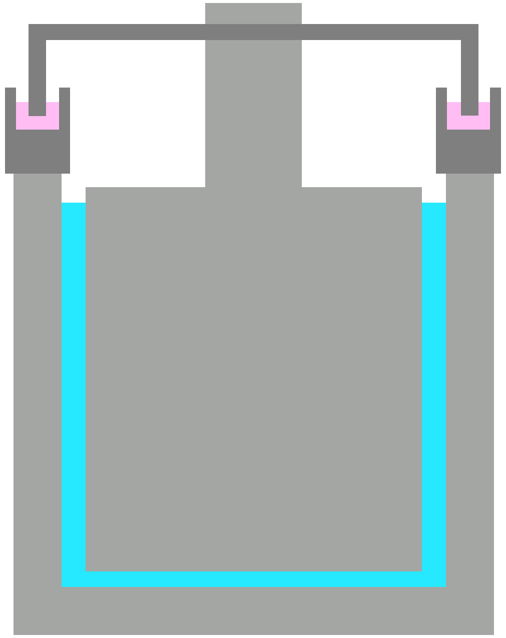
P-PTD200 Peltier System

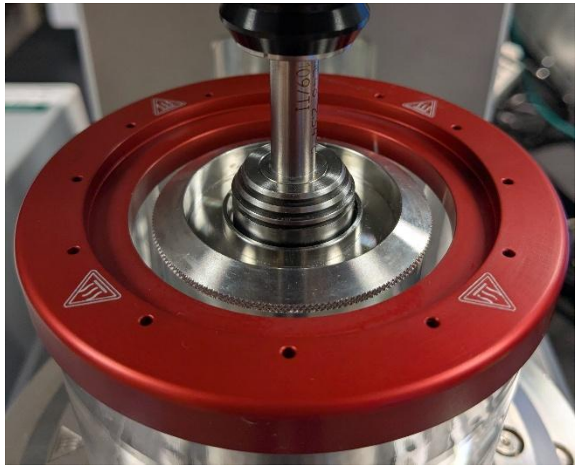


In both cases, temperature gradients possible

Preventing sample evaporation during experiments

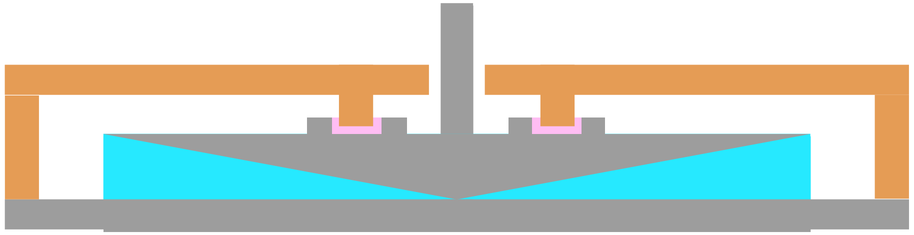
- Rheo-Scattering measurements are often slow, can take over a day for one sample
    - Volatile solvents need extra care
        - Solvent trap forms saturated atmosphere to limit evaporation
        - Even so, consider refreshing your sample every so often

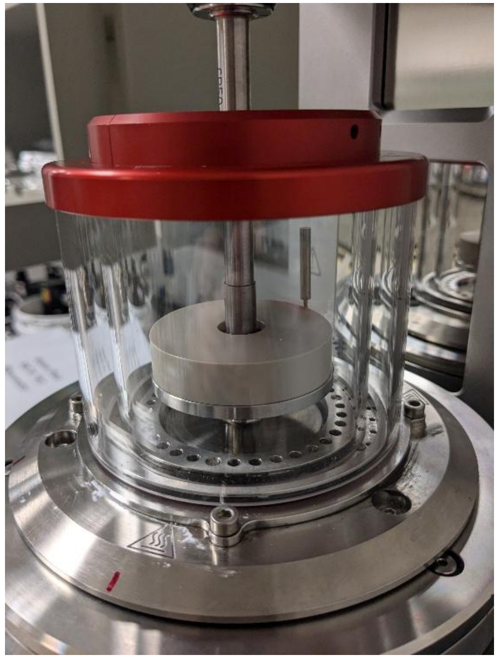




Evaporation control protects both your sample and your torque signal

<u>Rheo-NR PEEK evaporation blocker</u>

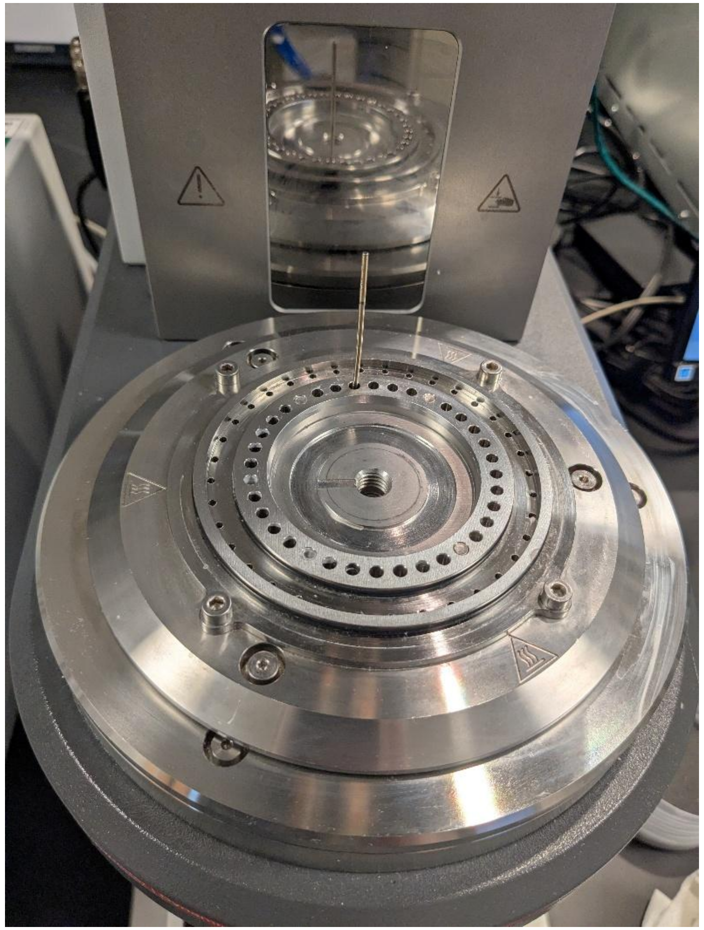




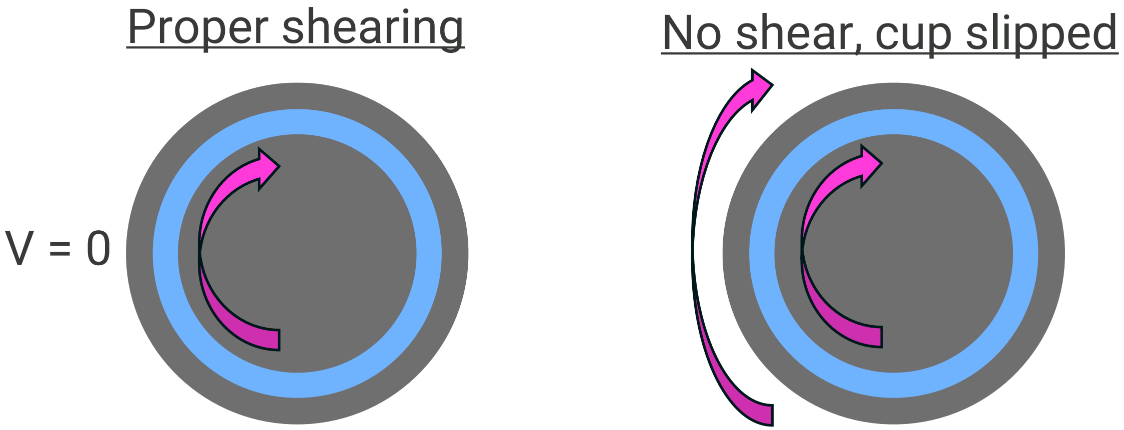
Practical advice for running Rheo-SANS

What to do when stuff goes wrong

Use a strong vacuum pump to hold down your sample cell

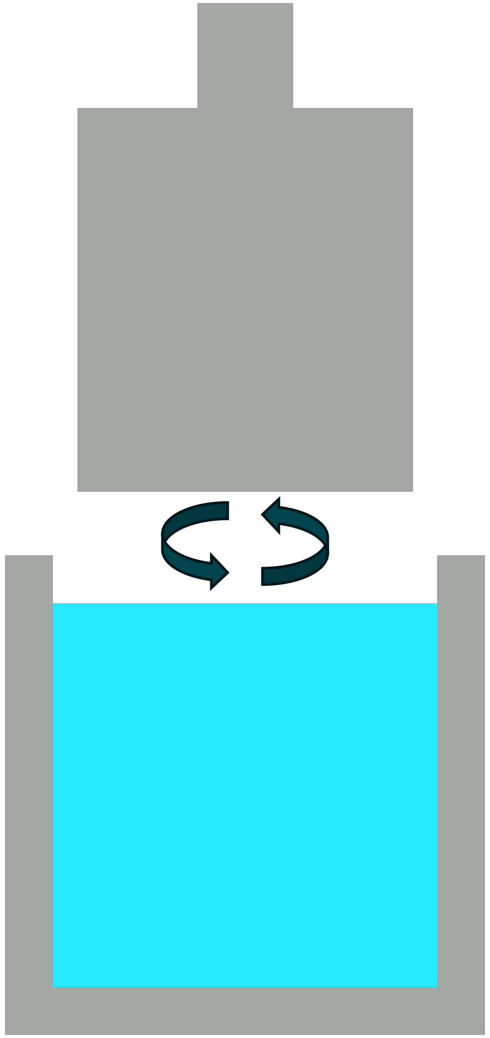


- Sample cup/wafer often held in place by a vacuum chuck
  - No screws, clamps, friction
- Dirty bottom of cup/wafer, weak vacuum pump, debris/ leak in vacuum lines undo all your alignment work



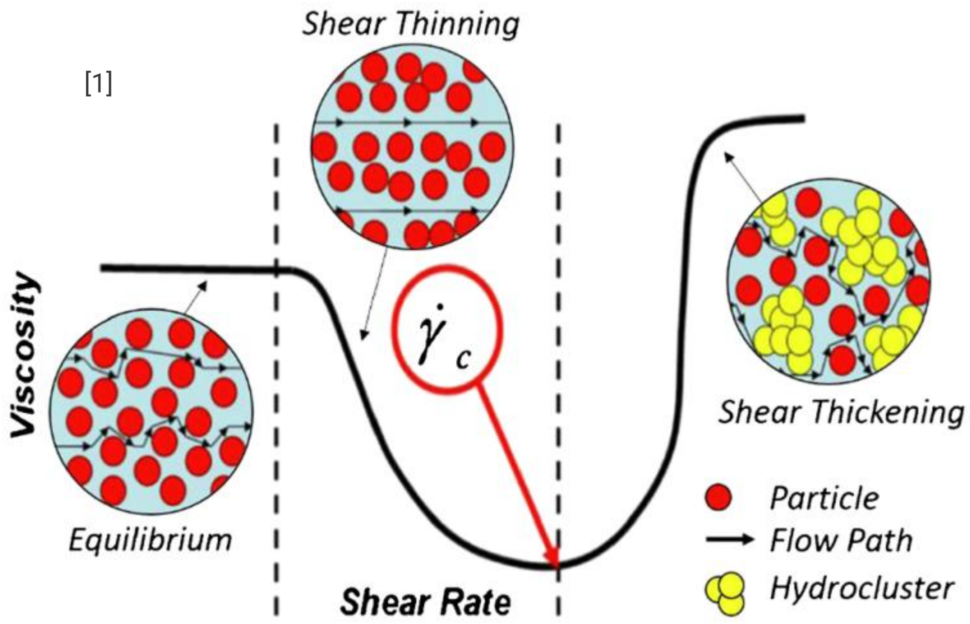
Even a perfectly aligned rheometer can produce garbage data if the sample cup slips.

Sample loading for thick (shear thickening) materials



- Load cup first, then bring bob down into sample
- Very high viscosity, gelled samples induce high normal forces on bob
    - Can overwhelm rheometer normal force
    - Shear thinning? Rotate bob while lowering
        - Don’t overdo it, or it could shear thicken!
- Consider loading effects on sample
    - Consider thermally cycling, allow time for relaxation and/or bubbles to disappear

[1]



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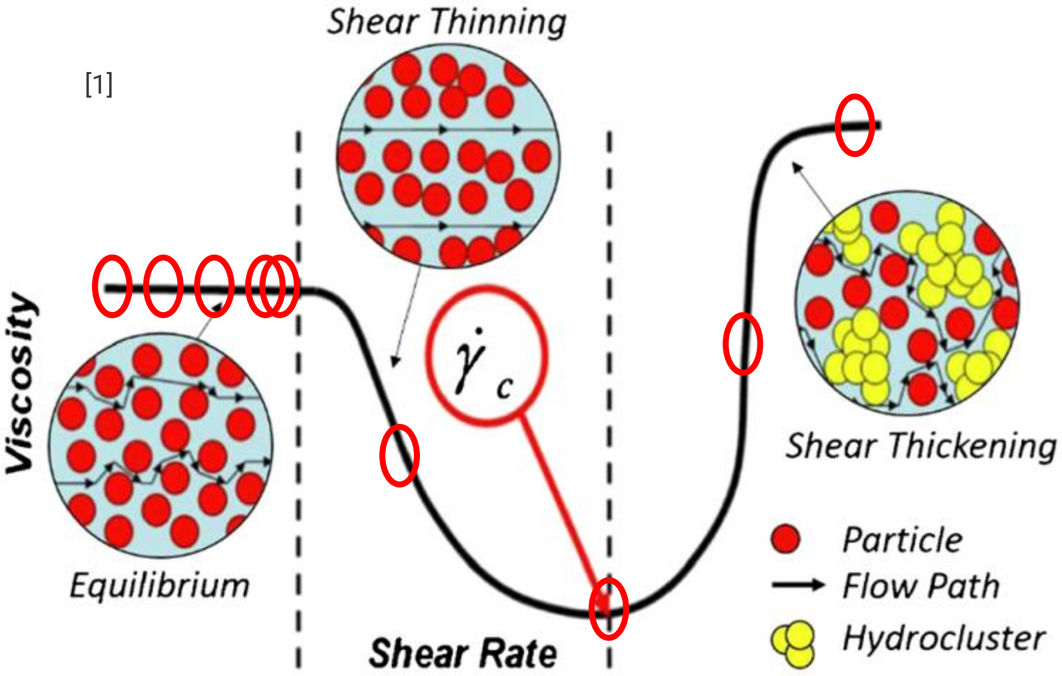
[1] Pinto and Meo, *Appl Compos Mater*, 2017

Know the system!

- PLEASE ask users to study the rheology of their sample THOROUGHLY prior to beamtime

Samples don't tend to change much at low shear rates
Know conditions that give the most information about microstructural changes



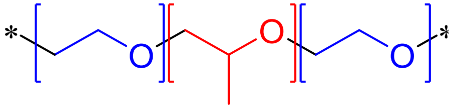
LETTER | DECEMBER 20 2024

A Pluronic block copolymer in H_2O and D_2O: The isotope effect on phase transition

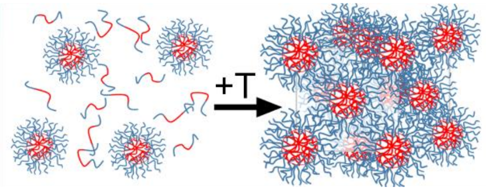
Nicola Antonio Di Spirito; Finizia Auriemma; Odda Ruiz de Ballesteros; Nino Grizzuti; Rossana Pasquino

Check for updates

Physics of Fluids 36, 121712 (2024)
https://doi.org/10.1063/5.0247190



PEO = poly(ethylene oxide)
PPO = poly(propylene oxide)



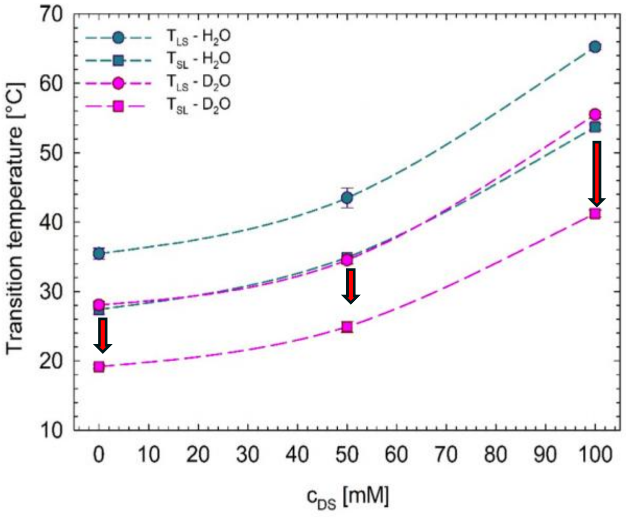


FIG. 2. Liquid-to-solid and solid-to-liquid transition temperatures as a function of diclofenac sodium concentration for the Pluronic F68/H_2O and Pluronic F68/D_2O solutions with various amounts of diclofenac sodium, at 5 °C min^{-1}. The error bars are evaluated as standard deviations of multiple experiments.

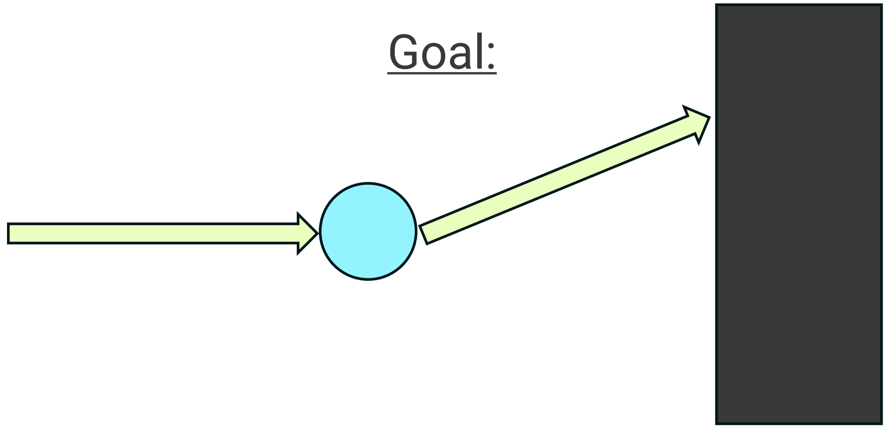
Deuterating solvents can dramatically impact hydrogen bonding & resulting microstructures

[1] Pinto and Meo, *Appl Compos Mater*, 2017

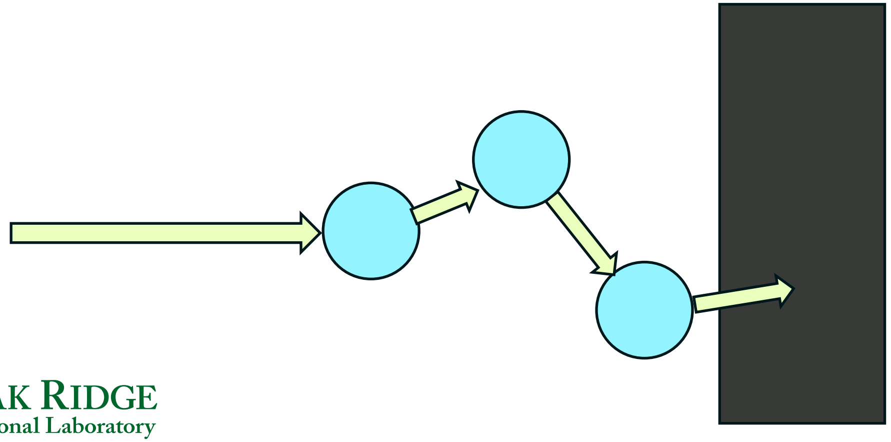
Pitfalls – multiple scattering

- In strongly scattering samples, neutrons may scatter more than once before reaching the detector – BAD!
    - Distorted scattering, unexpected gain/loss of features, low signal

Goal:



Multiple scattering:



- Use NIST scattering calculator:
  https://www.ncnr.nist.gov/resources/activation/

Material

CH2CH2OCH2CHCH3OCH2CH2OH

Absorption and Scattering

Density: 1 | Thickness: 0.2 | Calculate

Source neutrons: 3 Ang | Source X-rays: Cu Ka

Scattering from CH2CH2OCH2CHCH3OCH2CH2OH

Source neutrons: 3.000 Å = 9.09 meV = 1319 m/s
Source X-rays: 1.542 Å = 8.042 keV
Sample in beam: $CH_2CH_2OCH_2CHCH_3OCH_2CH_2OH$ at 1.00 g/cm^3

1/e penetration depth (cm)		Scattering length density (10^{-6}/Å^2)		Scattering cross section (1/cm)		X-ray SLD (10^{-6}/Å^2)	
abs	29.216	real	0.320	coh	0.001	real	9.372
abs+incoh	0.189	imag	-0.000	abs	0.034	imag	-0.019
abs+incoh+coh	0.189	incoh	20.658	incoh	5.243		

Neutron transmission is 34.80% for 0.2 cm of sample (after absorption and incoherent scattering).
Transmitted flux is 3.480e+7 n/cm^2/s for a 1e8 n/cm^2/s beam.
Contrast match point: 12.7% D_2O by volume (real SLD = 0.320×10^{-6}/Å^2)

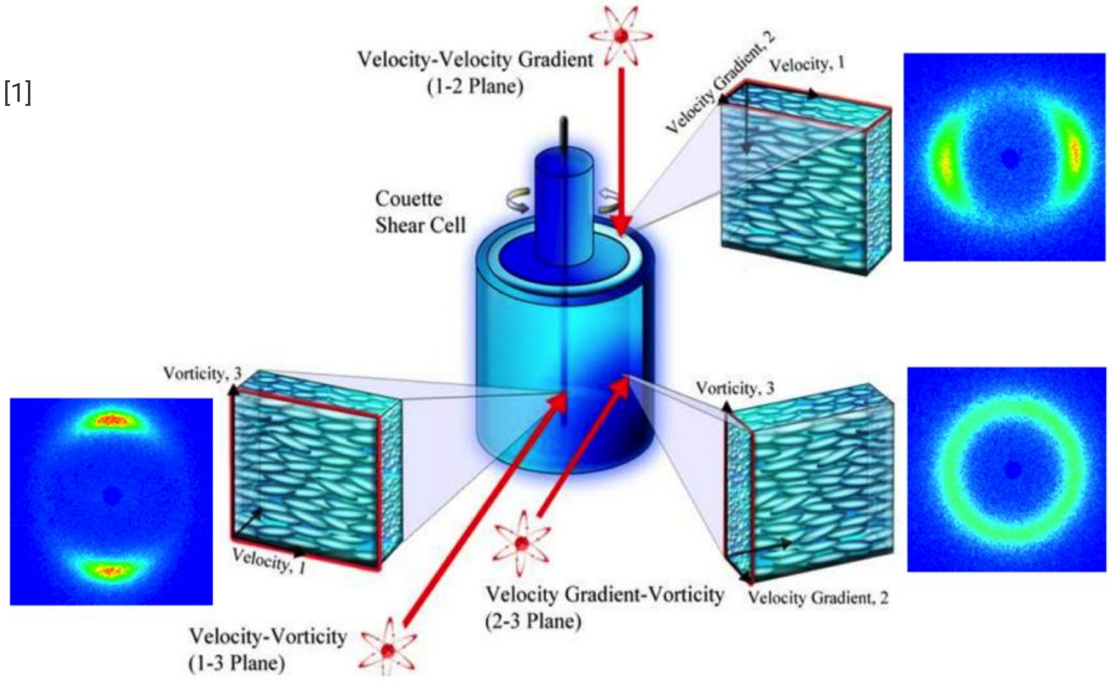
Target transmission ~90%, but discuss with your instrument scientist!

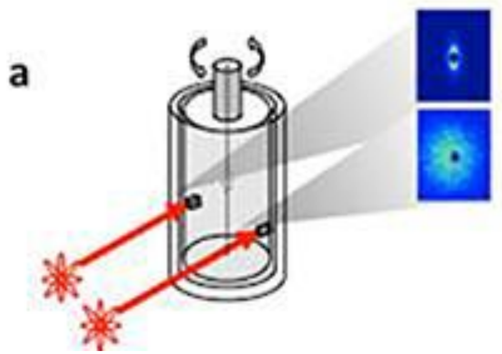
Complementary Techniques

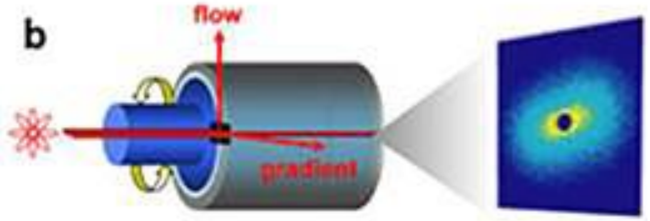
How can we further support user experiments?

The enigma: the 1,2- shear plane

[1]

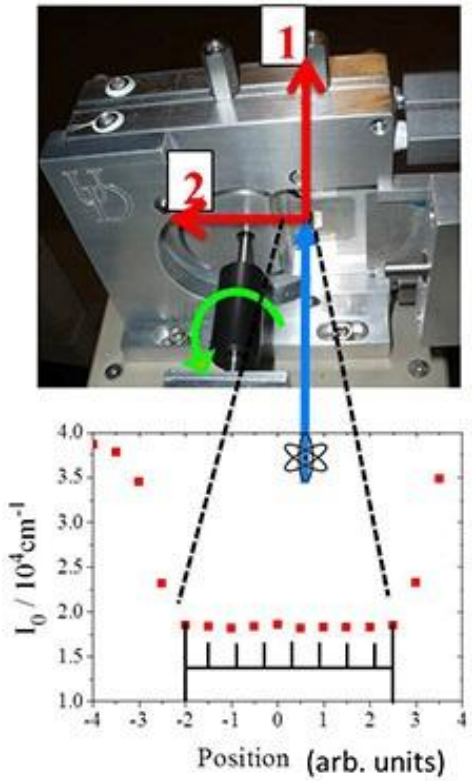






<u>Flow-gradient (1-2)</u>
Probes structural changes across shear gradient
Captures shear-induced layering or banding
Complex scattering pattern, instrumentationally difficult

[2]



For a 'full' picture of a material under shear, a 1,2-shear cell is necessary

Oak Ridge National Laboratory

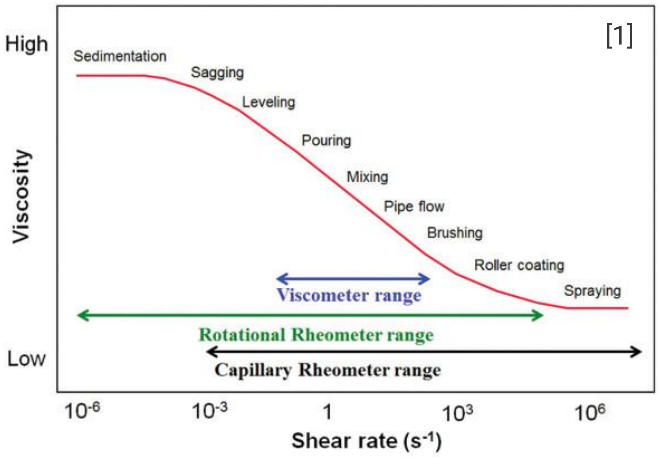
[1] M. Calabrese and N. Wagner, adapted from L. Porcar et al., *Rev. Sci. Instru., 2011* [2] K. Gurnon et al., *J. Vis. Exp.* 2014

Capillary Rheo-SANS: accessing high shear rates

Limit of shear rate in Couette cell ~ 10^3 s^{-1}

Many real-world processes ~10^4-10^7 s^{-1}

$$\uparrow \dot{\gamma} = \frac{v}{h} \downarrow$$



[2]

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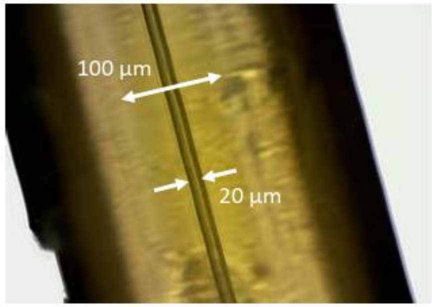
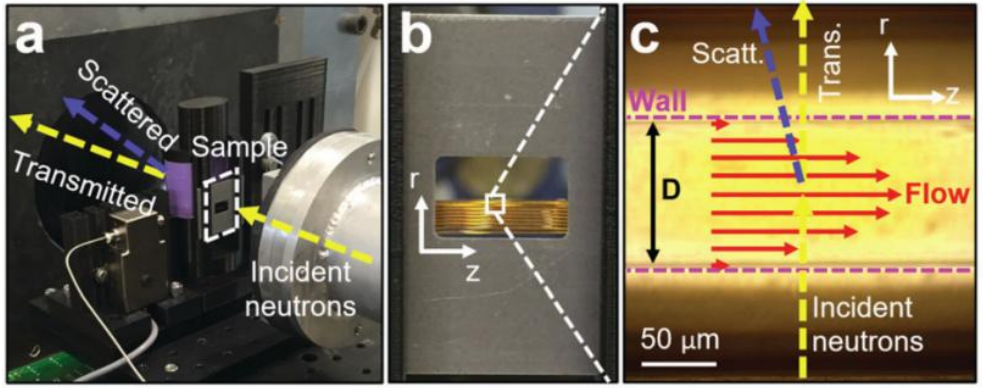
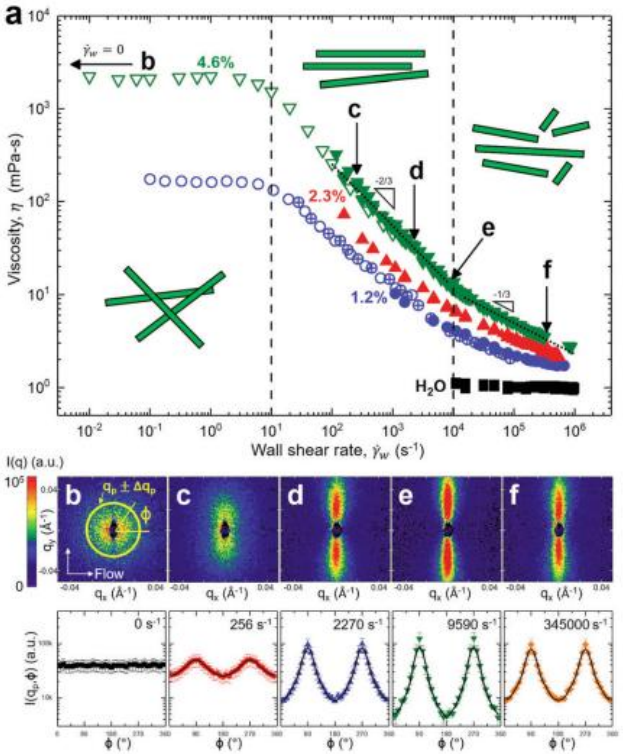
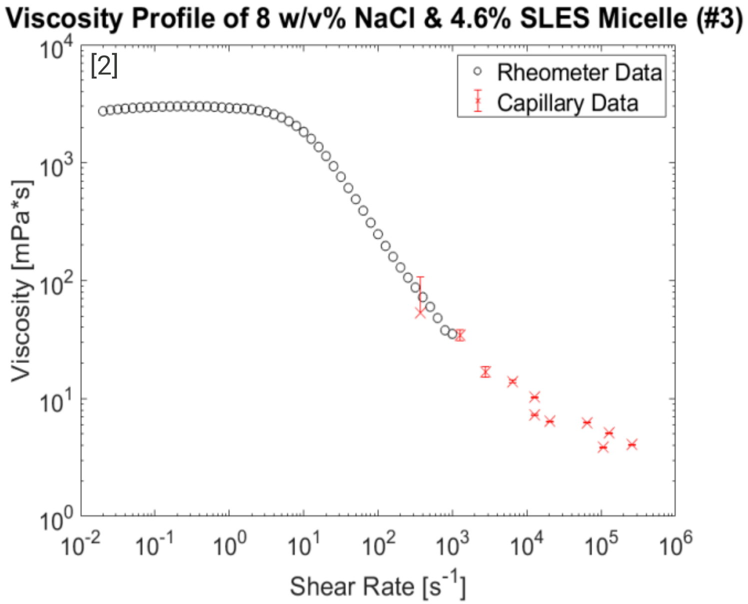






$$\sigma_w = \frac{(\Delta P - P_{end})R}{2L} \qquad \dot{\gamma}_w^c = \frac{4Q}{\pi R^3} - \frac{4v_{slip}}{R}$$



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[1] Duffy, *AmericanLaboratory.com*, 2016 [2] Reidel et al., *CHRNS*, 2020, all others [3] Murphy et. al, *Soft Matter*, 2020

Rheo-SAXS

Strong scattering from electron density contrast

Higher typical flux

- Synchrotrons: $10^{12} - 10^{15}$ photons/s/mm²
- Neutrons: $10^{8} - 10^{10}$ neutrons/s/mm²

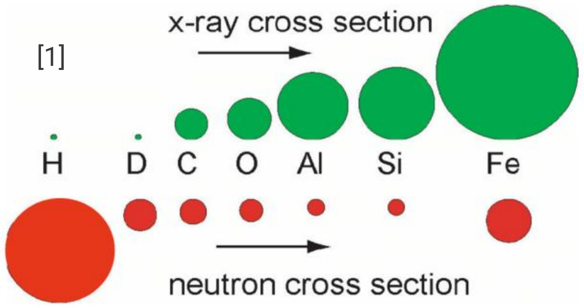
Rheo-SAXS: Higher time resolution (ms-s vs. s-min)

- Kinetics of faster reorientations may be observed

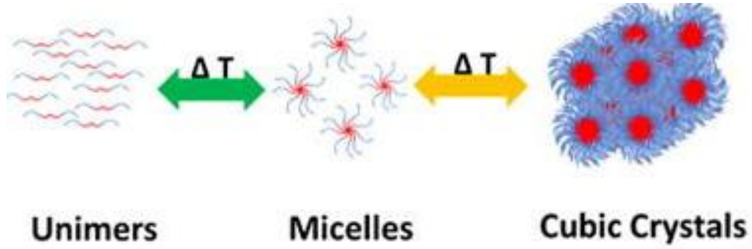
SAXS better quantifies local packing, crystalline order, periodicity

> Rheo-SANS and Rheo-SAXS probe different but overlapping windows of structure under flow

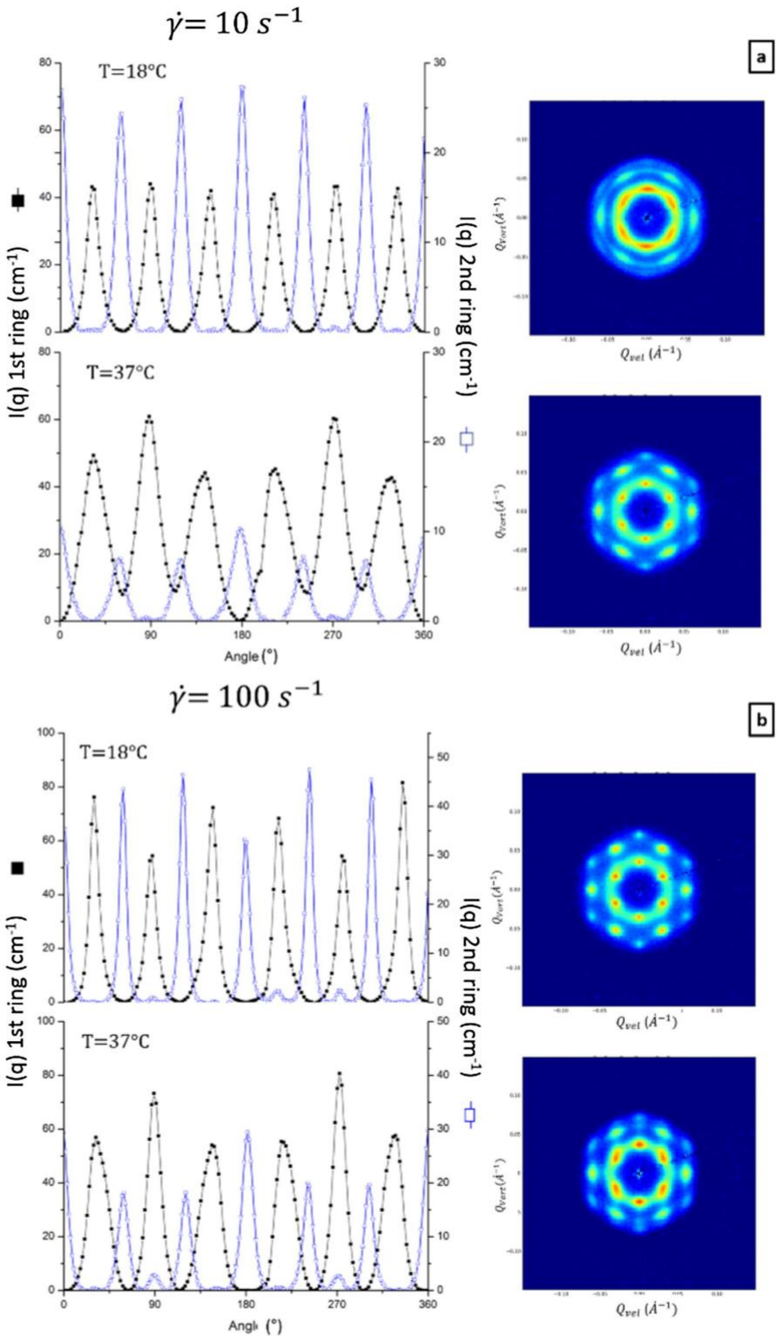
[1]



[2]



[2]



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[1] Daniel Hussey, National Institute of Standards and Technologies, 2010 [2] Shriky et. Al, *J. Colloid. Interf. Sci.*, 2020

Dielectric RheoSANS: *in-situ* dynamics measurements!

ELSEVIER
Current Opinion in Colloid & Interface Science
Volume 42, August 2019, Pages 110-120

Dielectric RheoSANS: a mutual electrical and rheological characterization technique using small-angle neutron scattering

Jeffrey J. Richards [1], John K. Riley [2]

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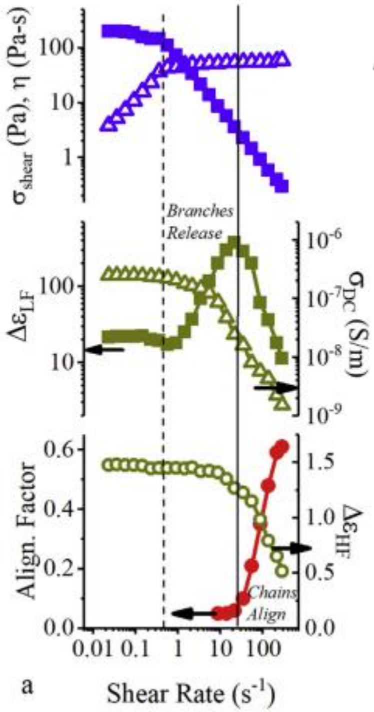
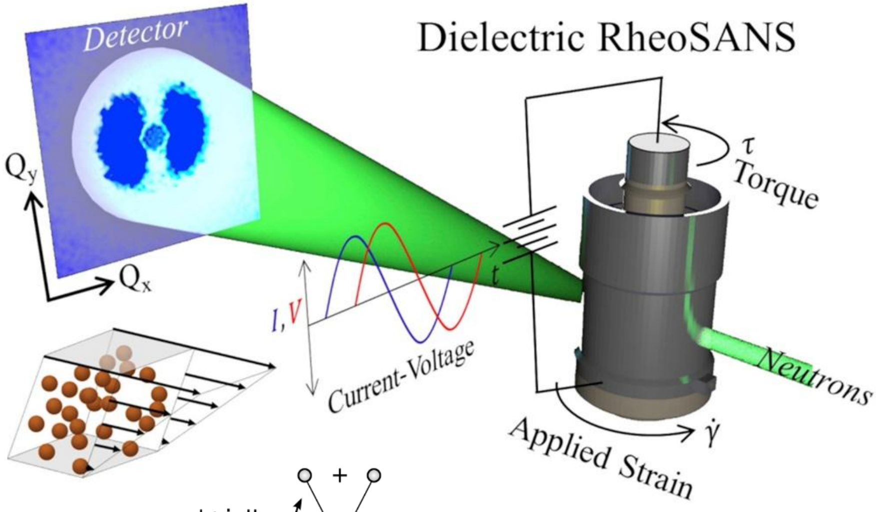
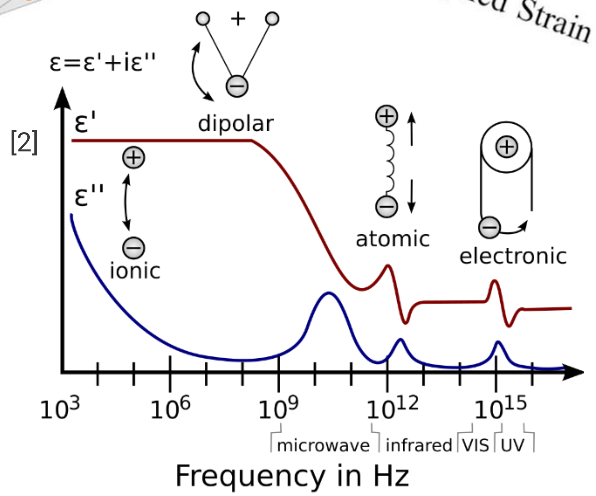
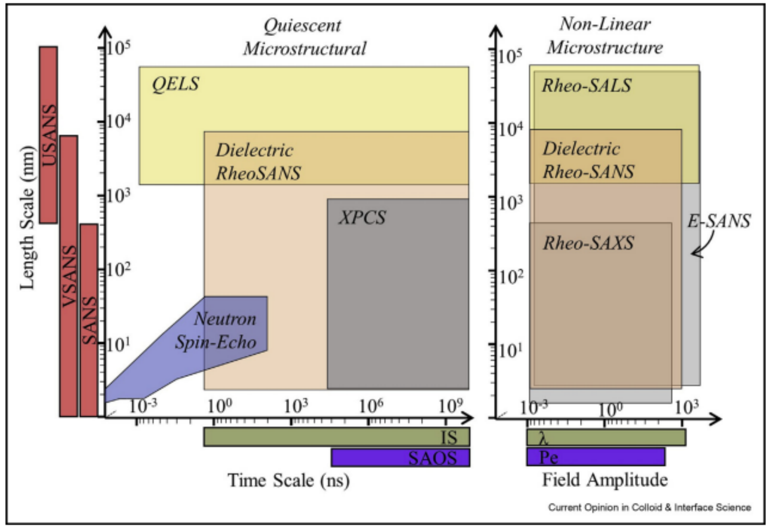
https://doi.org/10.1016/j.cocis.2019.05.002





Dielectric spectroscopy is much like a rheological frequency sweep, but using electrical potential and measuring dynamics!





- Converts cup and bob into electrodes
- Tracks flow (rheology), structure (SANS), and polarization dynamics (dielectric) simultaneously!
- Useful for systems where charge transport couples to structure (ionic surfactants)

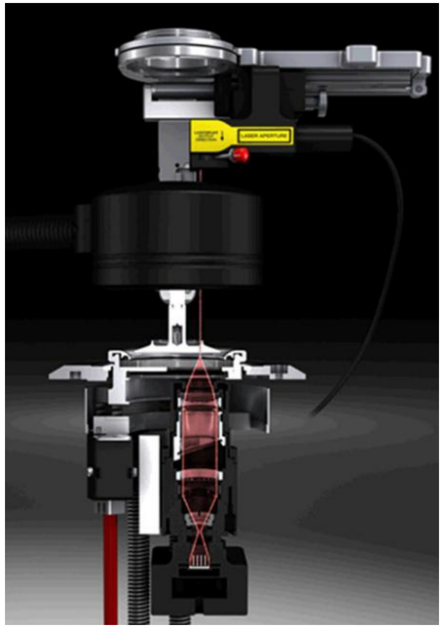
[2] Dr. Ken Mauritz, *U. Southern Mississippi*

Rheo-SALS

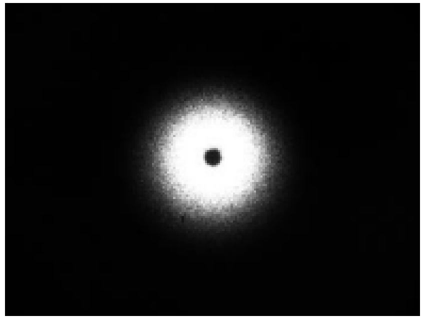
Combines rheology + small-angle light scattering

Commercially available through both Anton Paar (MCR series) and TA Instruments (DHR-2 and DHR-3 series)

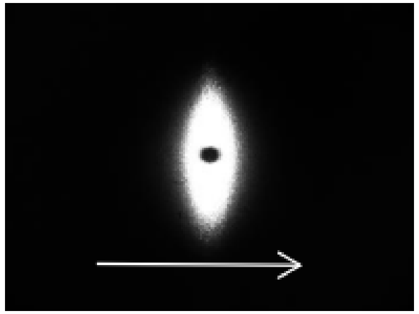
Anton Paar



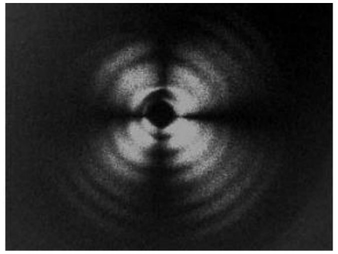
0.39 mW 658 nm beam
-20 to 300 °C
q ~ 0.3 μm^{-1} to 2 μm^{-1}
~3.0 to 21 μm

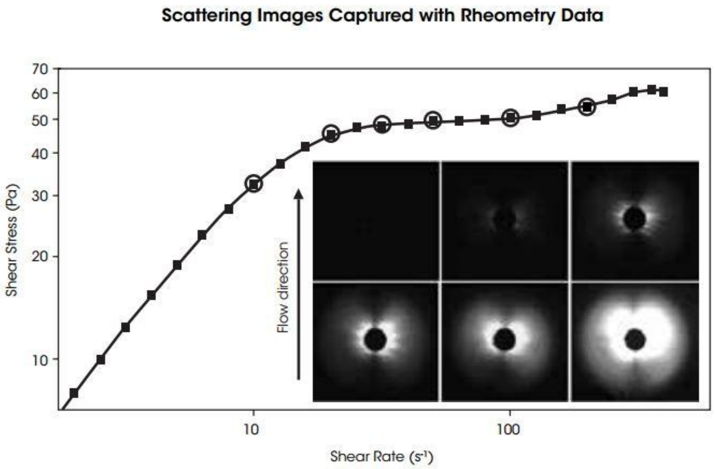


0) rest



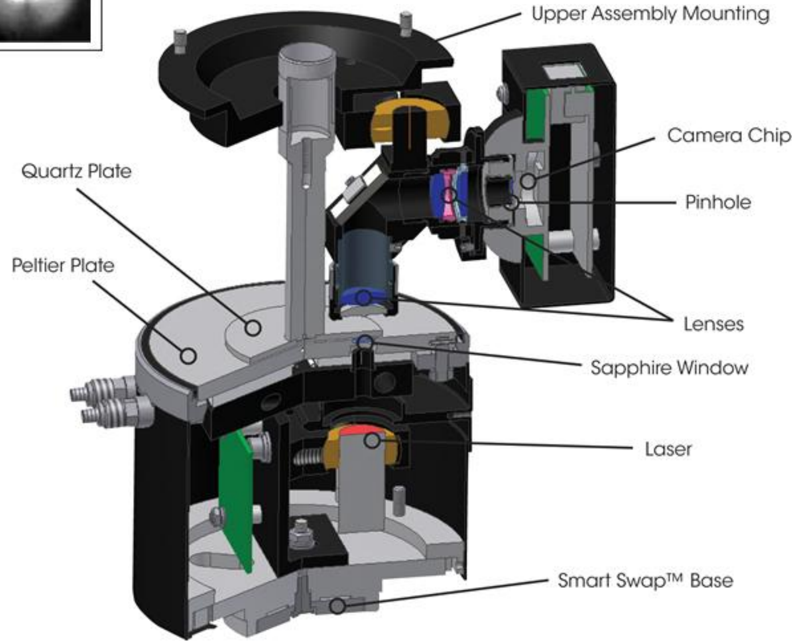
e) $\dot{\gamma}$= 120 s^{-1}





0.95 mW 635 nm beam
5 to 95 °C
q ~ 1.38 μm^{-1} to 6.11 μm^{-1}
~1 to 4.6 μm

TA Instruments



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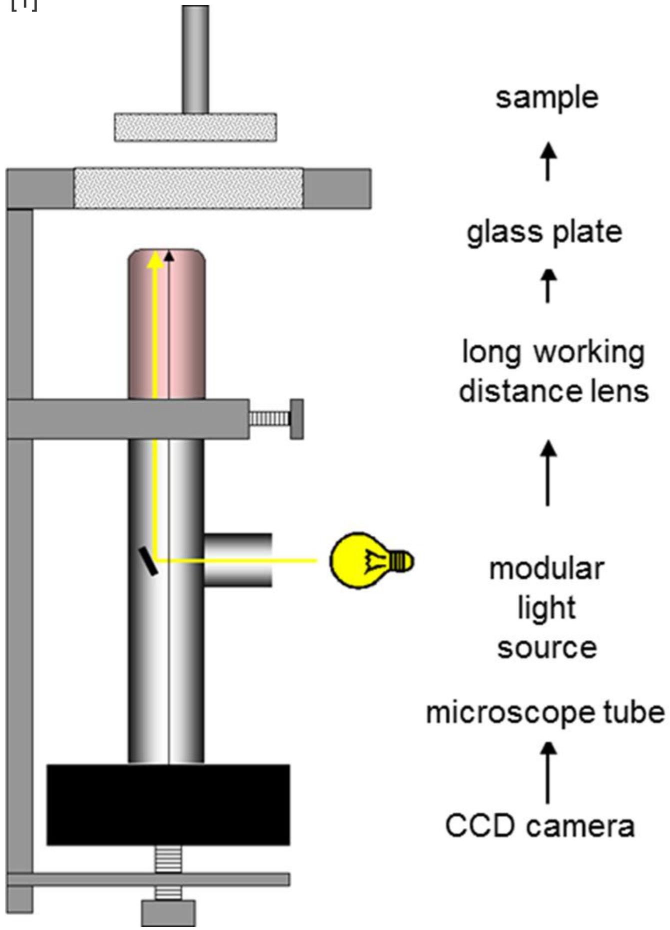
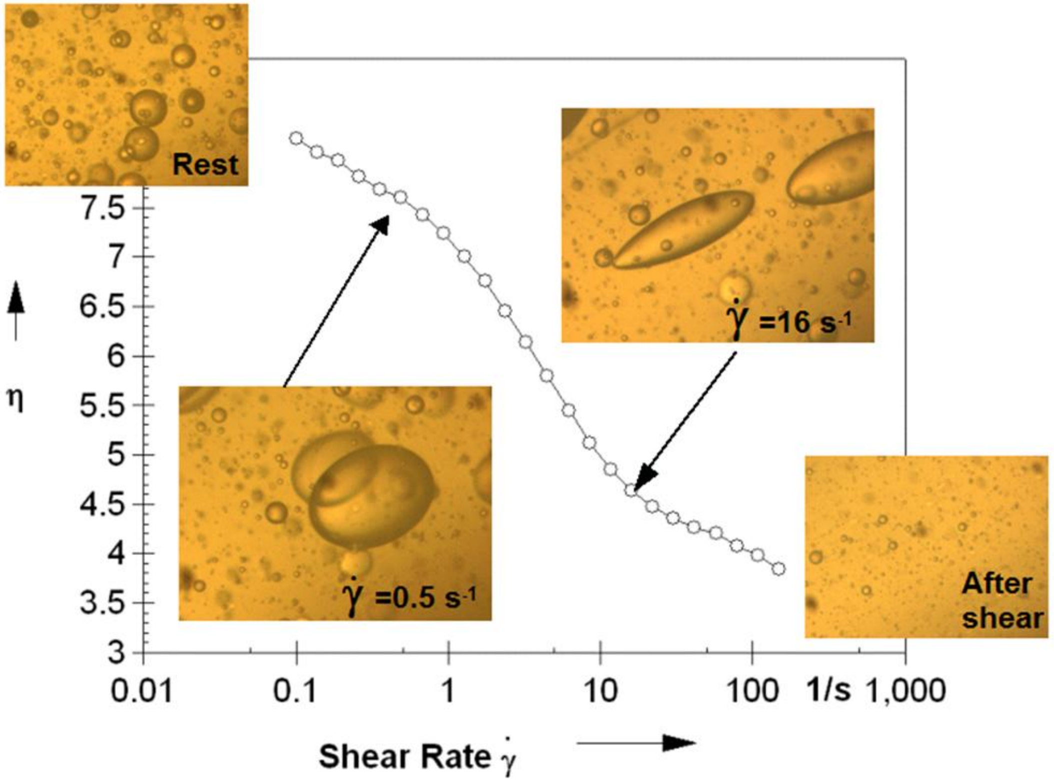
Rheo-Microscopy

[1]



Rheo-SANS
- nm - µm
- Ensemble-averaged scattering

Rheo-Microscopy
- µm - mm
- Real-space imaging
- Spatial heterogeneity visible



- Rheo-SANS and Rheo-Microscopy: one quantifies nanostructure, the other visualizes real-space morphology and heterogeneity
- Fuller picture of structure-flow relationships

[1] Anton Paar

Particle Tracking Velocimetry

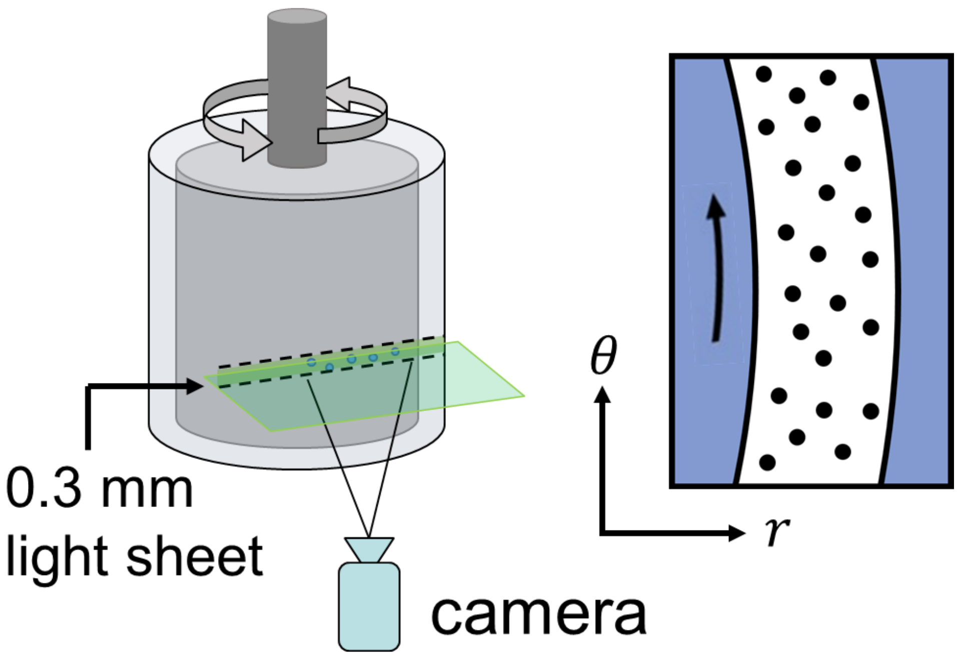
RESEARCH ARTICLE | MARCH 21 2023

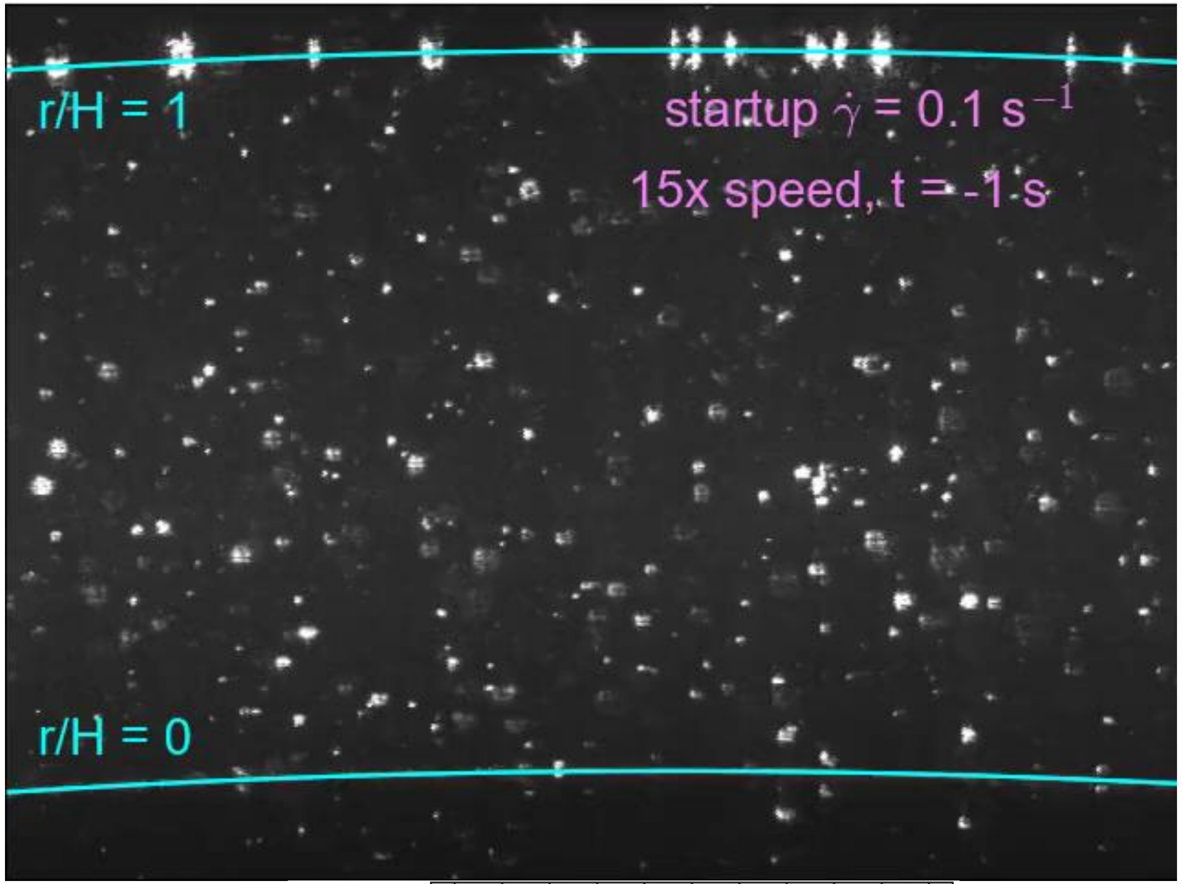
Evolution of flow reversal and flow heterogeneities in high elasticity wormlike micelles (WLMs) with a yield stress

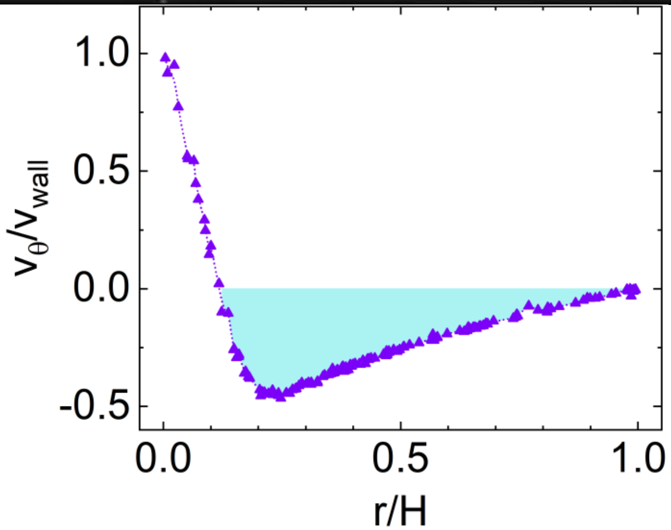
Patrick J. McCauley ; Christine Huang ; Lionel Porcar; Satish Kumar ; Michelle A. Calabrese

Check for updates

J. Rheol. 67, 661–681 (2023)
https://doi.org/10.1122/8.0000535





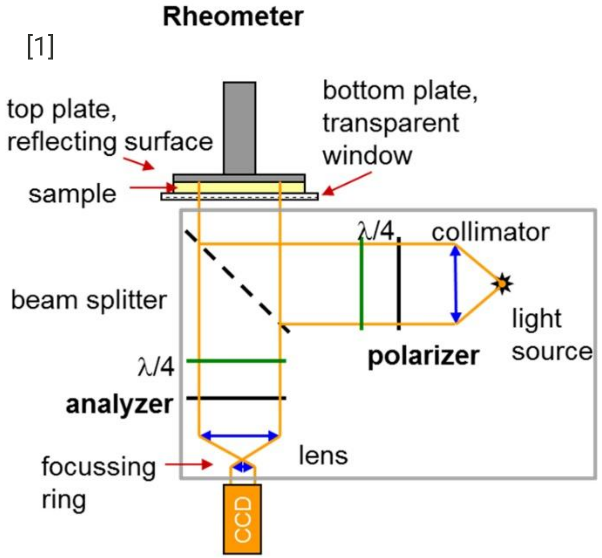


Rheometry with Polarized Light Imaging

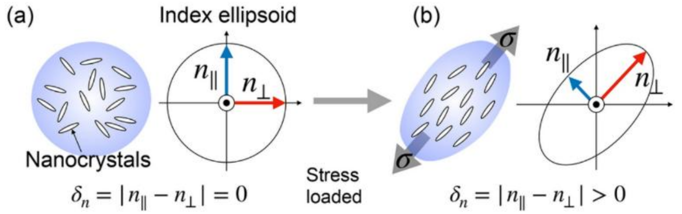
- Flow-induced anisotropy in complex fluids often linked to microstructural alignment
- Need to connect bulk optical anisotropy with nanostructural orientation from scattering
- Shear-induced polarized light imaging (SIPLI) observes birefringence in materials under deformation
- Birefringence: constructive/deconstructive interference of light through materials with anisotropic index of ref.

Birefringence rheology provides fast, bulk-sensitive signatures of flow-induced anisotropy, providing complementary view of alignment dynamics

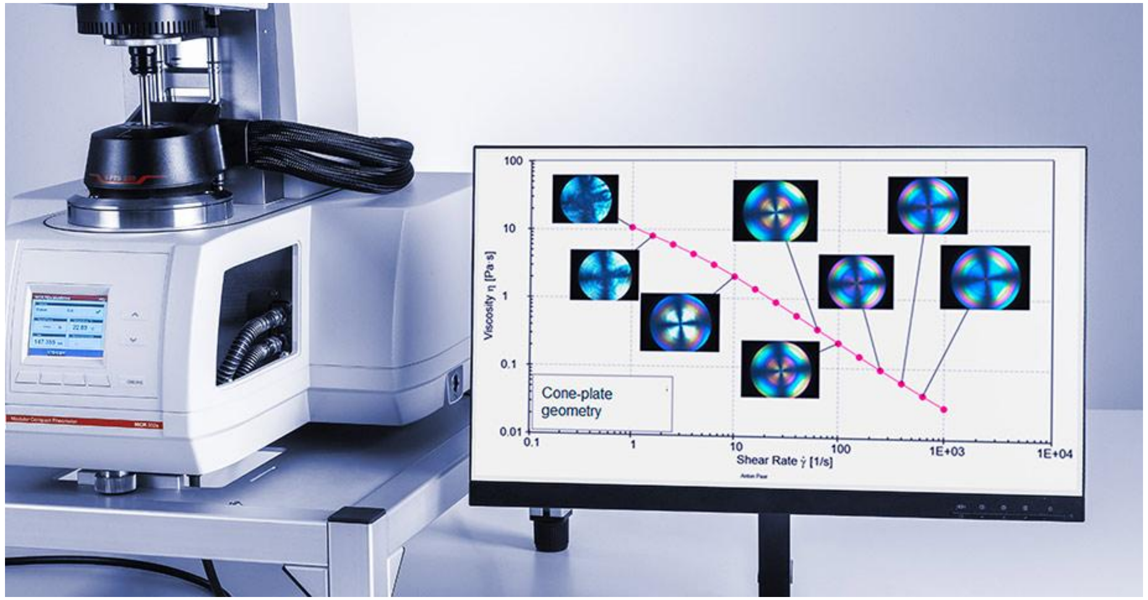
[1]



[2]



[1]



[1] Anton Paar [2] Worby et. al., *Sci Rep.*, 2024

Rheo-Scattering: Key Takeaways

Rheometers induce rotational shearing motion on soft materials

Rheo-Scattering involves bombarding samples with neutrons while under flow or deformation

Rheology describes materials' behavior under flow

Scattering provides average description of nanostructure

Why combine rheology and scattering?

- Probes **microstructure under deformation**.
- Links **macroscopic mechanical response** to **nanostructure**.

Rheometers are **complex** sample environments

Rheo-SANS uniquely links flow, alignment, and composition, providing a quantitative window into the microscopic origin of soft matter behavior

Acknowledgements

Oak Ridge National Laboratory


Luke Heroux

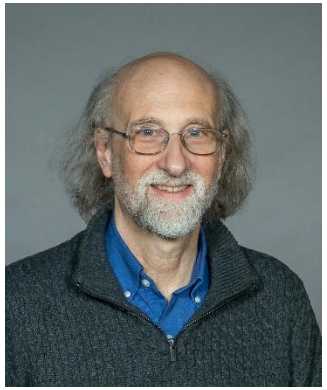

Gernot Rother


Wei-Ren Chen


Changwoo Do

NIST Center for High-Resolution Neutron Scattering


Katie Weigandt


Paul Butler


Ryan Murphy

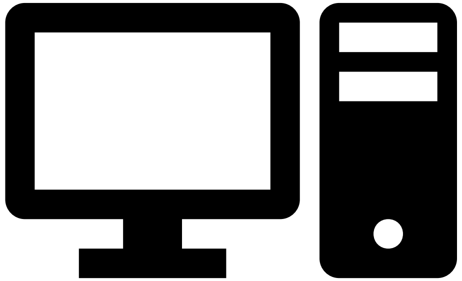
Oak Ridge National Laboratory

NSF CHRNS CENTER FOR HIGH RESOLUTION NEUTRON SCATTERING NIST

Anton Paar

TA Instruments

Tomorrow: Making it all talk to each other

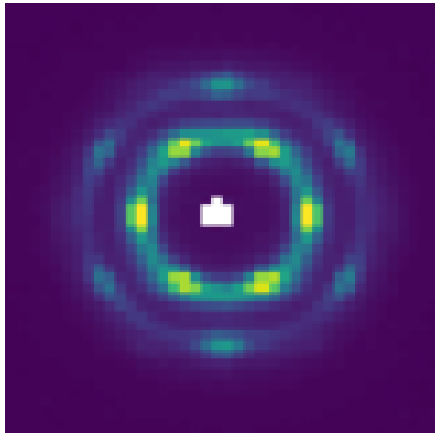


1. control shear

2. log data output

3. time resolution

Detector



Motion is only half the experiment;
Timing is the other half

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