UNIVERSITÀ DEGLI STUDI DI MILANO

A microscale approach to yield stress fluids: investigation of nonlinearity and yielding with an optofluidic micro-rheometer

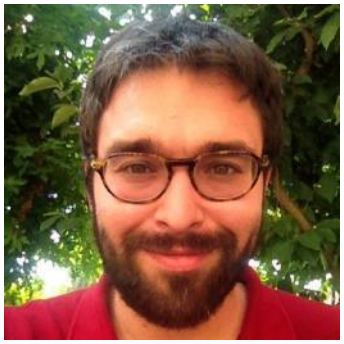
Giuliano Zanchetta

Dipartimento di biotecnologie mediche e medicina traslazionale

Complex fluids and molecular biophysics lab

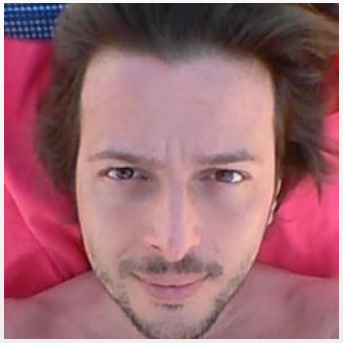
Thanks to…

Andrea Corno



Giovanni Nava

Tommaso Bellini



Valerio Vitali
Paolo Minzioni

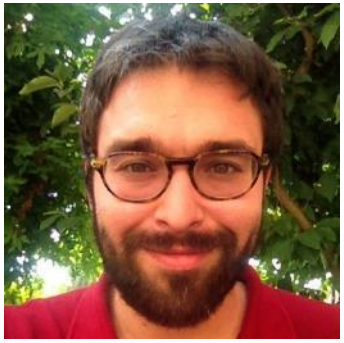
Francesca Bragheri
Roberto Osellame

UNIVERSITÀ DEGLI STUDI DI MILANO

Thanks to…

BIOMETRA

Andrea Corno



Giovanni Nava

Tommaso Bellini

Complex Fluids and Molecular Biophysics Lab

Biomolecules

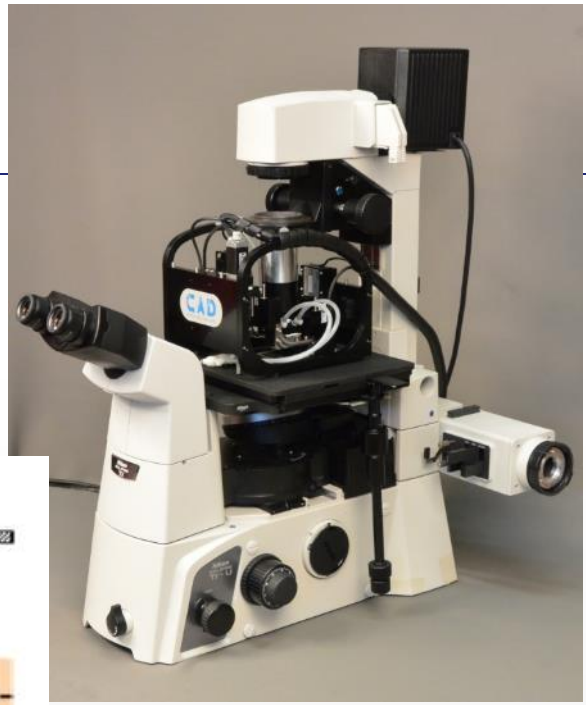
Soft materials

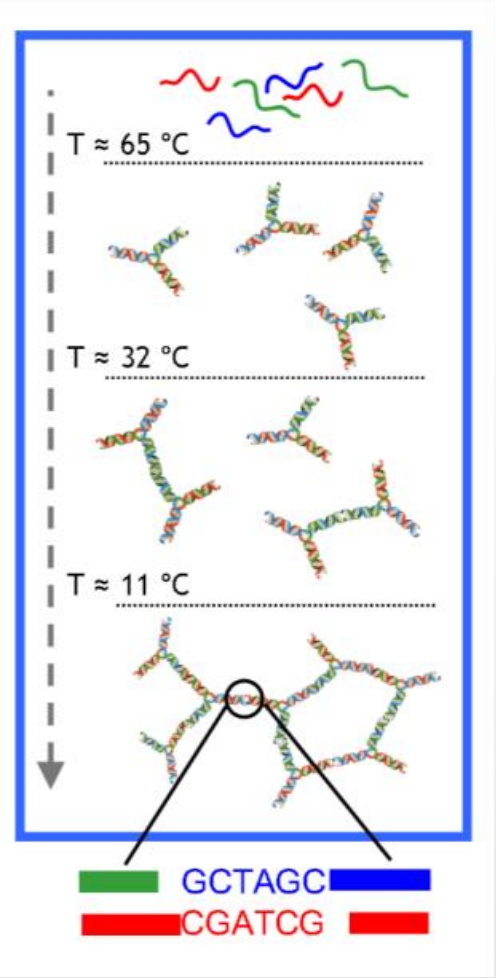
Optical techniques

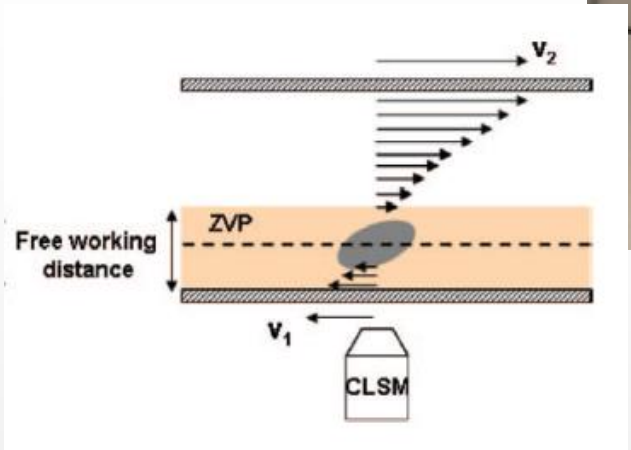
UNIVERSITÀ DEGLI STUDI DI MILANO

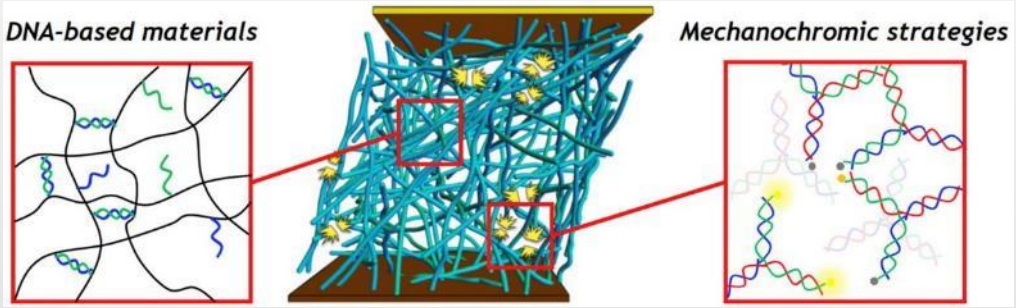
DNA-based hydrogels

Microscopy tools to study phase behaviour & response to mechanical perturbation



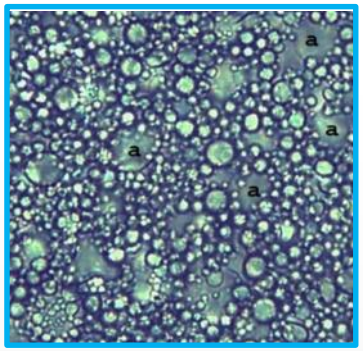


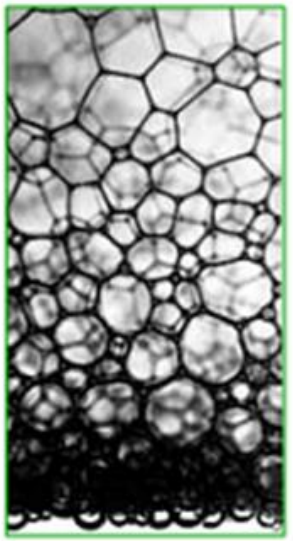


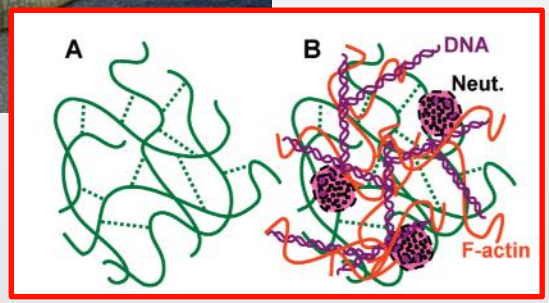


Biffi *et al. PNAS* (2013)
Zanchetta *COCIS* (2019)

Viscoelastic materials







Most interesting materials are neither (only) solid nor (only) liquid, but display multiple length and time scales dependent on microstructure

Yield stress materials

Solid-like

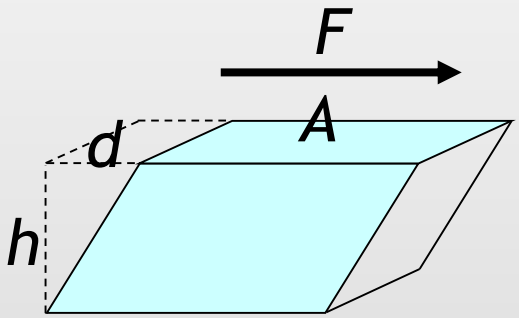
Liquid-like

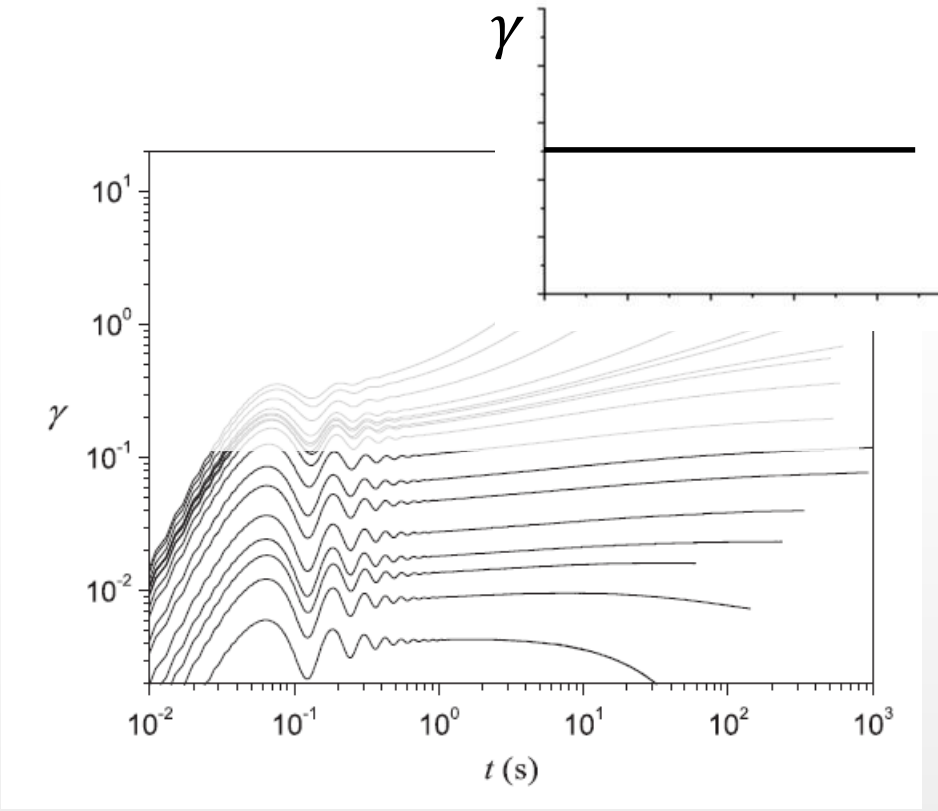
F

Yield stress materials: creep test

$\sigma = F/A$

$\gamma = d/h$



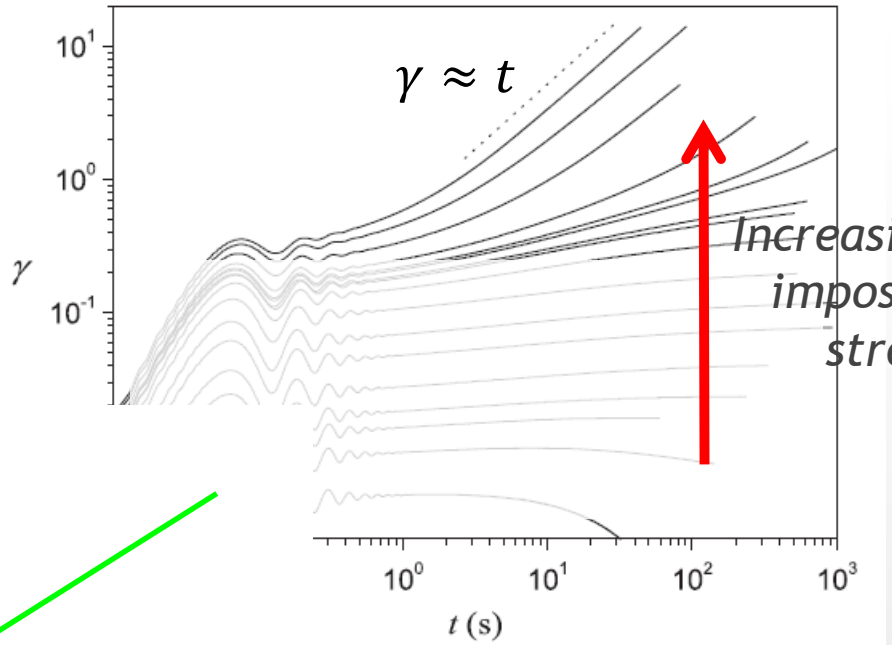


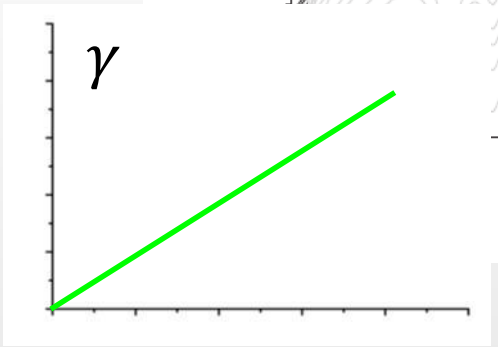
$\sigma < \sigma_y$ *solid regime* $\gamma = \frac{\sigma}{G}$

Balmforth et al., Annu.Rev. Fluid Mech. (2014)
Bonn et al. Rev. Mod. Phys. (2017)
Coussot, Rheol. Acta (2018)

Yield stress materials: creep test

$\sigma > \sigma_y$ *fluid regime* $\dot{\gamma} = \frac{\sigma}{\eta}$

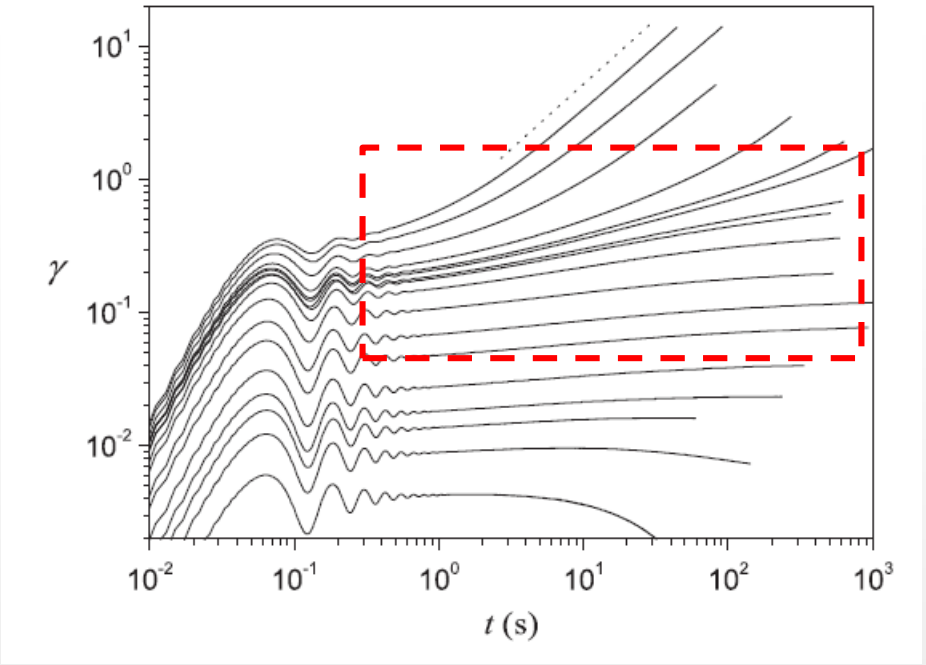




Balmforth et al., Annu.Rev. Fluid Mech. (2014)
Bonn et al. Rev. Mod. Phys. (2017)
Coussot, Rheol. Acta (2018)

Yield stress: what happens at the transition?

$\sigma \approx \sigma_y$ *creep regime* $\gamma \approx t^{\alpha}$ $\alpha \ll 1$



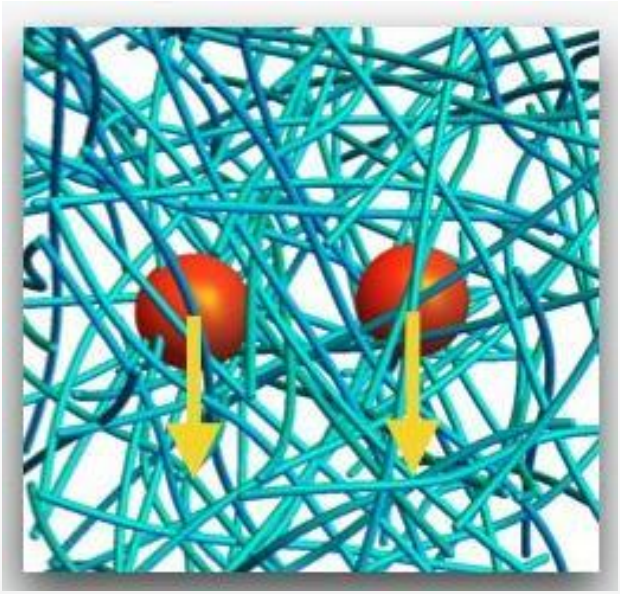
- *Delayed yielding*
- *Are there structural/dynamic precursors of failure?*
- *Interplay with flow localization/aging/boundary conditions…*

Balmforth et al., Annu.Rev. Fluid Mech. (2014)
Bonn et al. Rev. Mod. Phys. (2017)
Coussot, Rheol. Acta (2018)

Yield stress at the local scale

Particle stress < σ_y

Trapping!



N.Gnan

Particle stress > σ_y

Flow

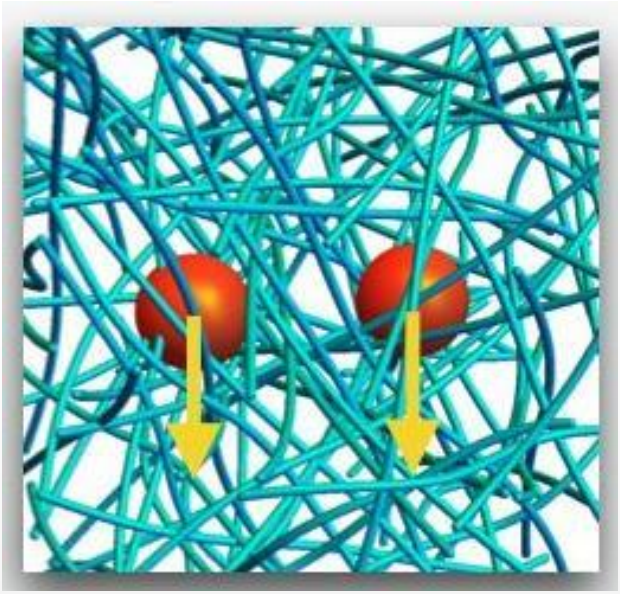
Beris et al. J. Fluid Mech. (1985)

…

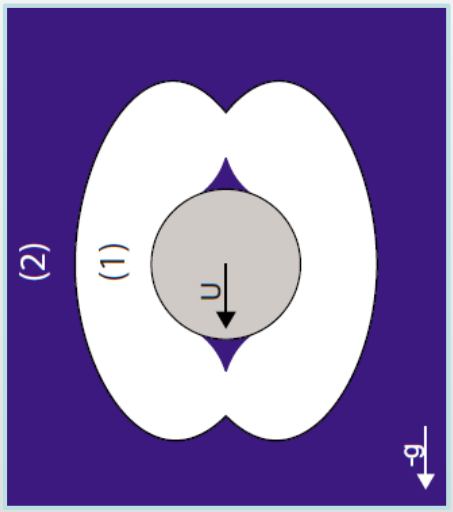
Putz, Phys. Fluids (2008)

Holenberg et al., PRE (2012)

Yield stress at the local scale



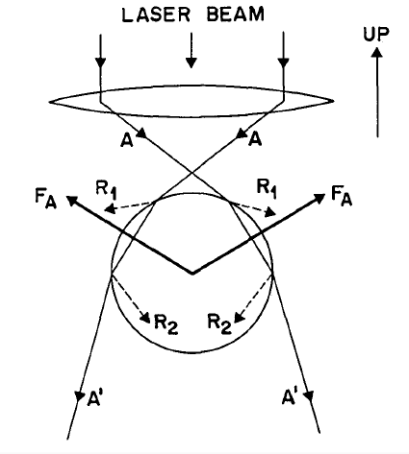
N.Gnan



- *Conditions for stable trapping?*
- *Comparison with macroscopic rheology?*
- *Extent of fluidized region?*
- *Transient vs. steady state*
- *Fine control over stress…*

Beris et al. J. Fluid Mech. (1985)
…
Putz, Phys. Fluids (2008)
Holenberg et al., PRE (2012)

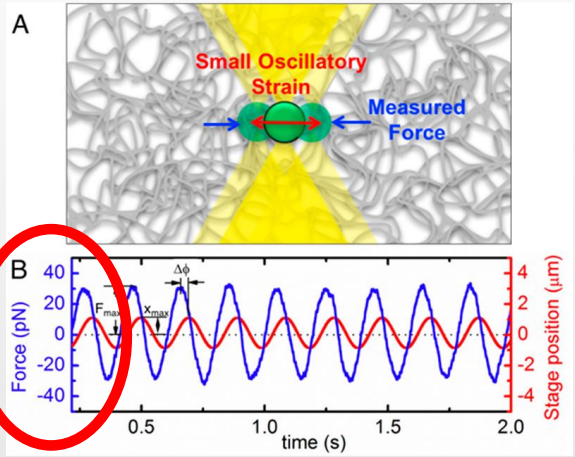
Optical tweezers for microrheology



Ashkin et al. Optics Lett. 1986

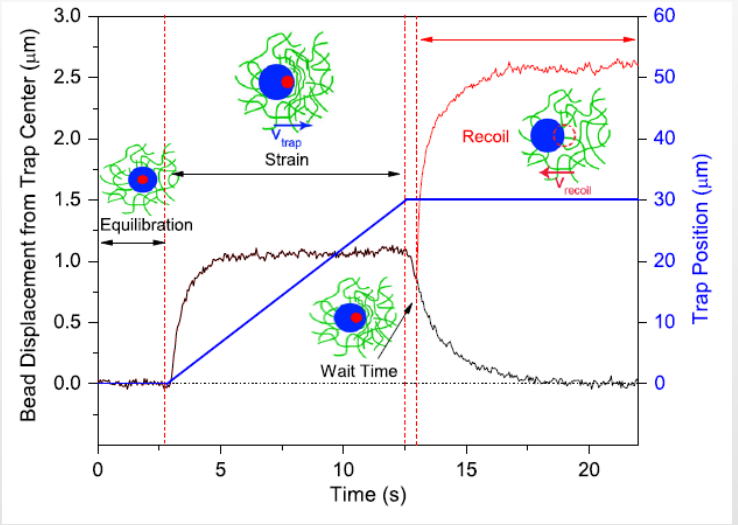
Nobel prize Physics 2018

Oscillatory rheology



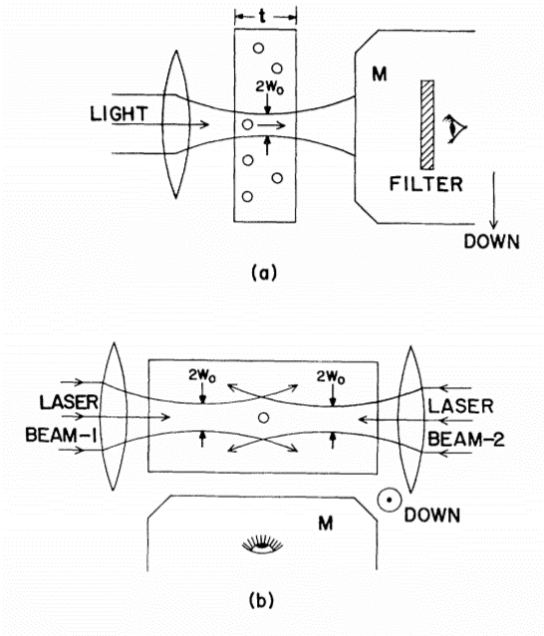
Forces are limited

Constant shear rate & recoil



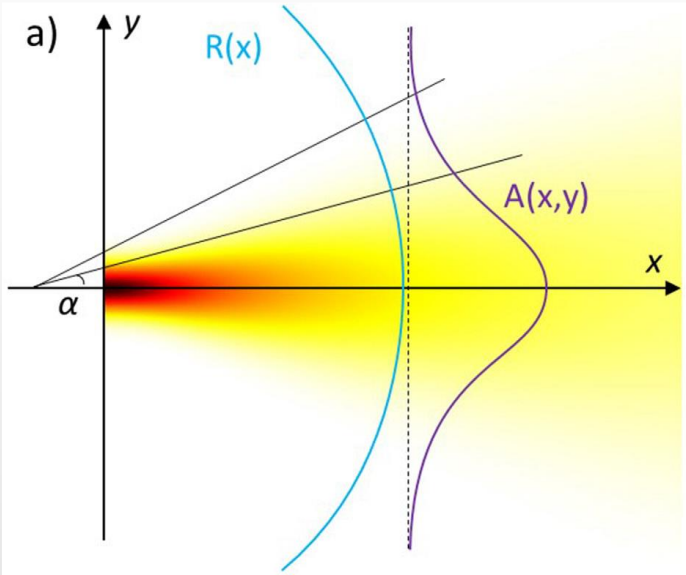
Chapman & Robertson-Anderson, PRL (2014)
Robertson-Anderson, ACS Macro Lett. (2018)

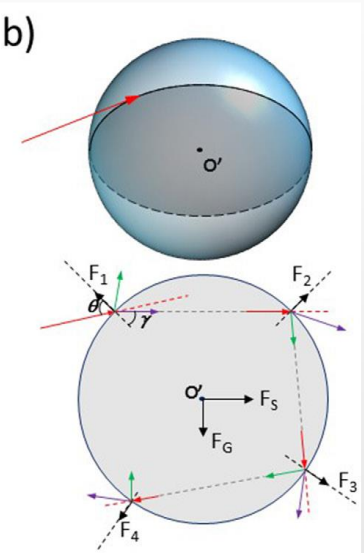
Before optical tweezers



Scattering forces:

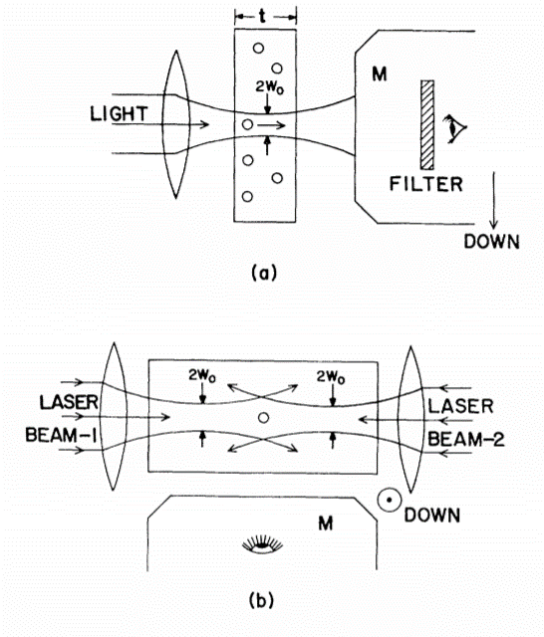
- *Stronger*
- *Less control*



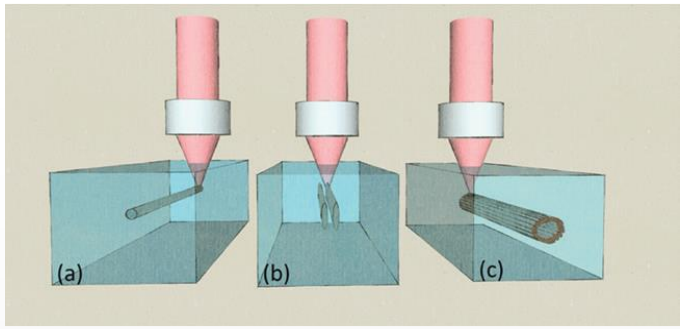


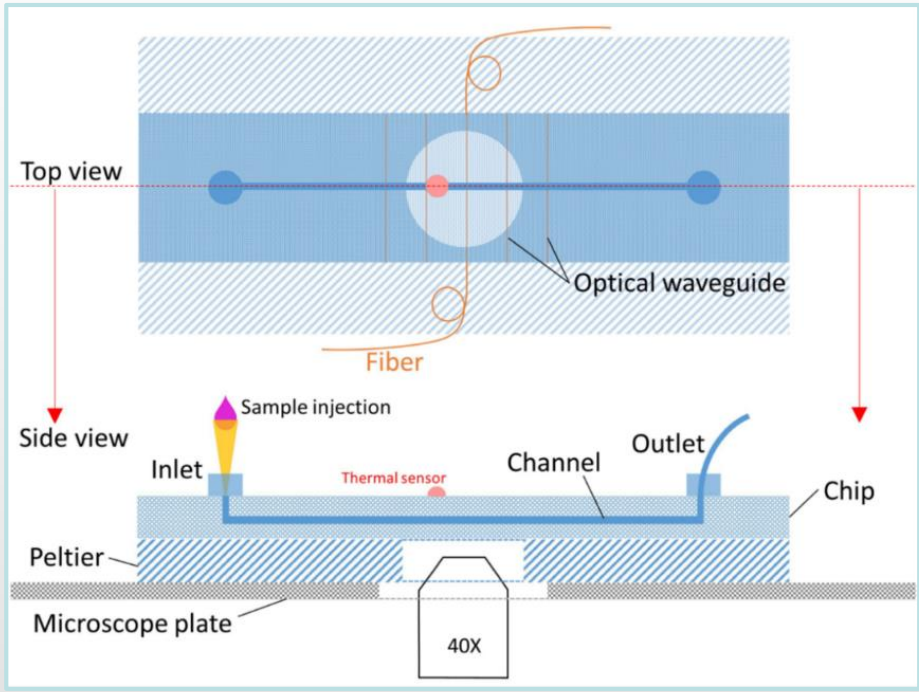
Ashkin PRL (1970)
Bragheri et al. J. Biophot. (2010)
Yang *et al. J. Micromech. Microeng. 27 (2017)*

Optofluidic micro-rheometer



Femtosecond laser writing of waveguides in glass

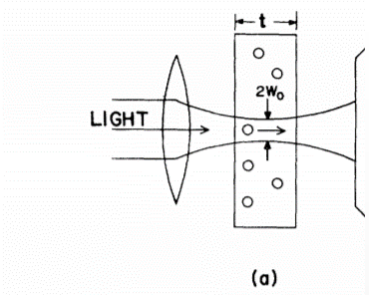


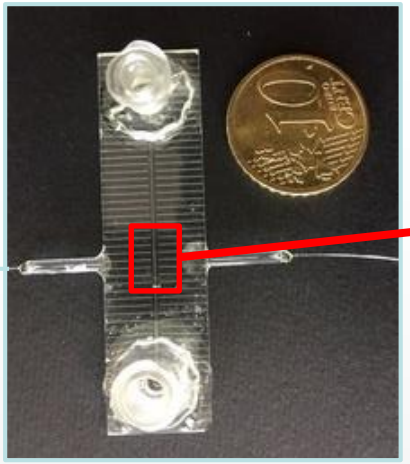


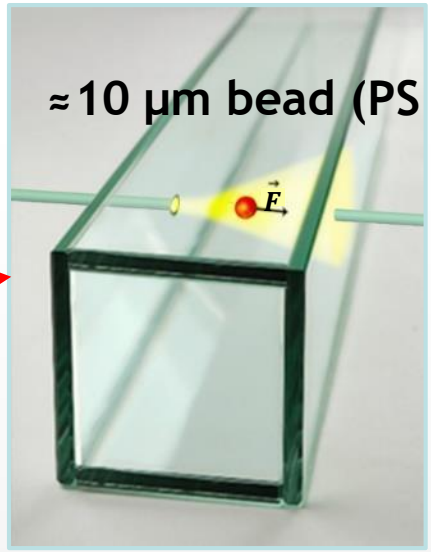
Sima et al. Nanophotonics (2018)
Yang et al. Micromachines (2017)

Optofluidic micro-rheometer

Shooting on one side

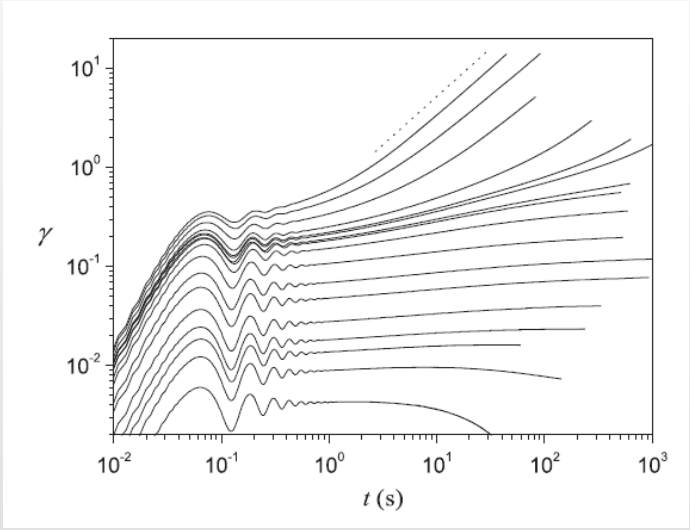






Shutter

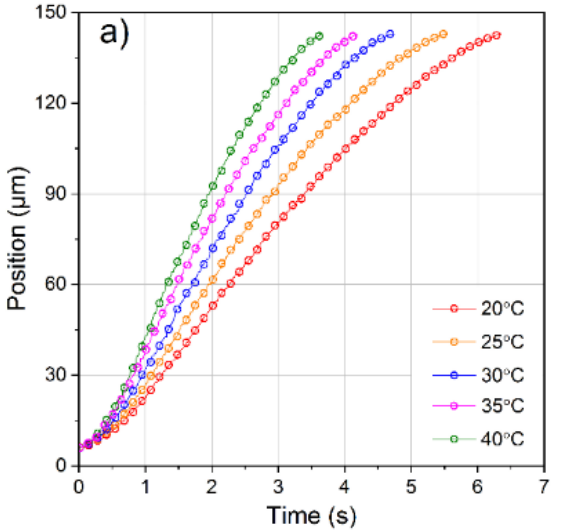
LASER
1064 nm

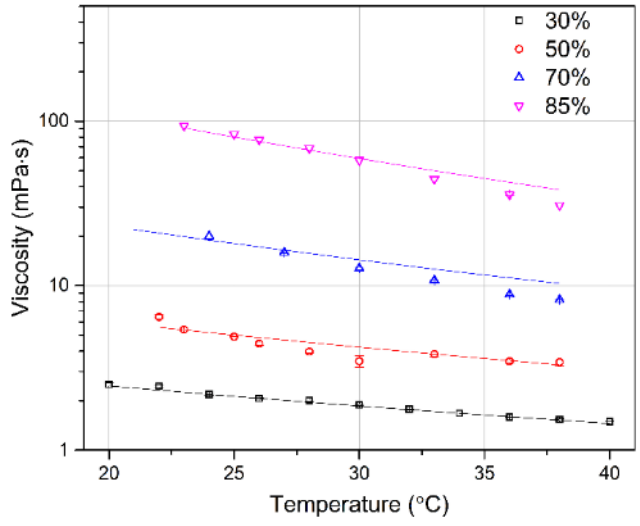


Nava et al. Soft Matter (2018)

Calibration with Newtonian fluids

$$F = 6\pi r \eta \dot{x} \qquad \sigma = \eta \dot{\gamma}$$





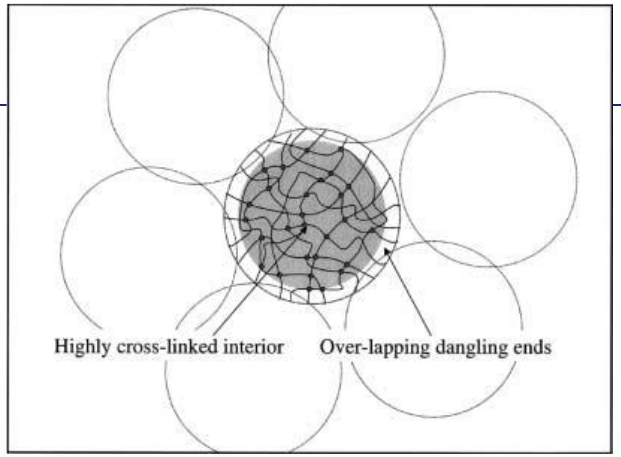
Glycerol-water mixtures

F ∽ [1-1000] pN (10x optical tweezers)

$$\gamma = \frac{\Delta x}{2r} \qquad \sigma = \frac{F}{12\pi r^2}$$

Yang et al. Micromachines (2017)

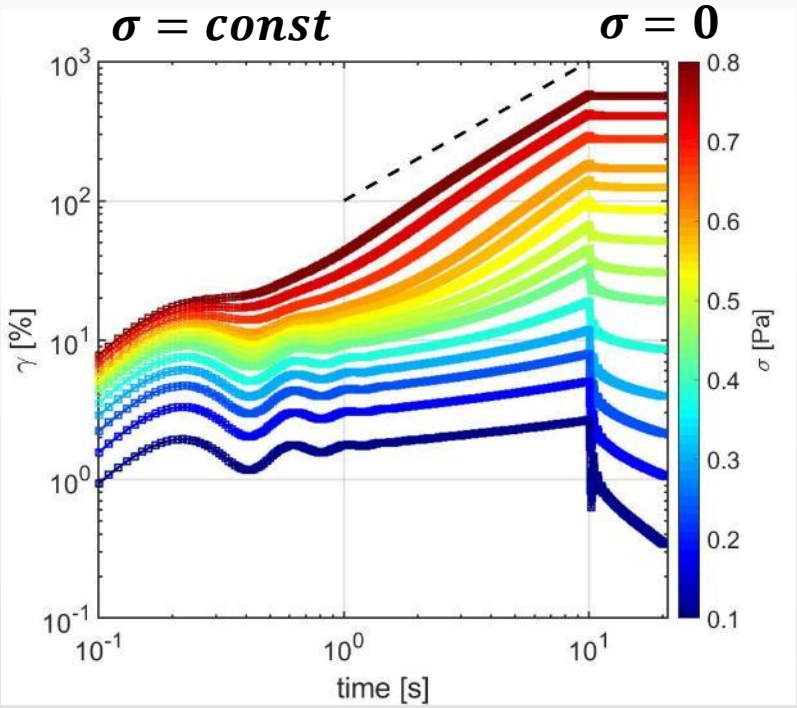
Swollen microgels



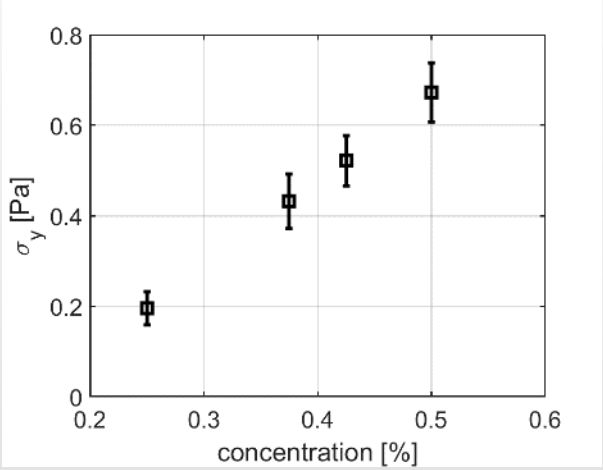
Carbopol Aqua SF-2 Lubrizol

Cross-linked linear polyacrylic acid chains (≈ 1 μm)

Bulk rheology: creep & recovery



- *Solid-creep-fluid*
- *Drop of recoverable strain*

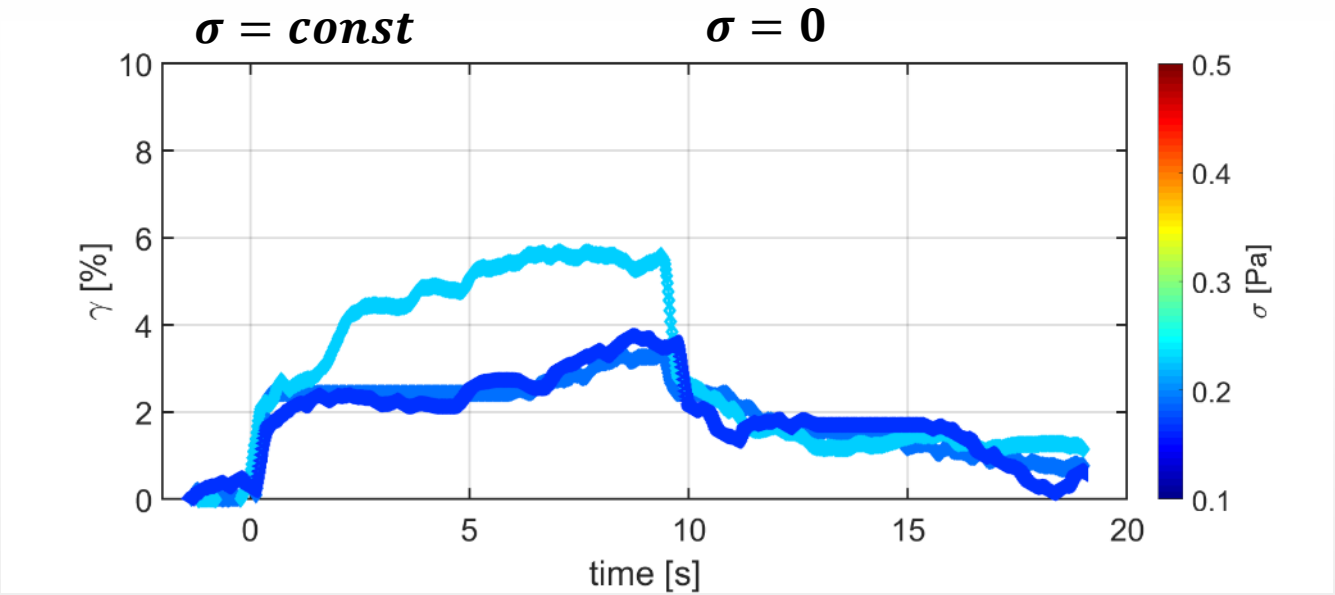


0.375%

Roberts & Barnes Rheol. Acta (2017)

Micro-rheometer

Creep & recovery



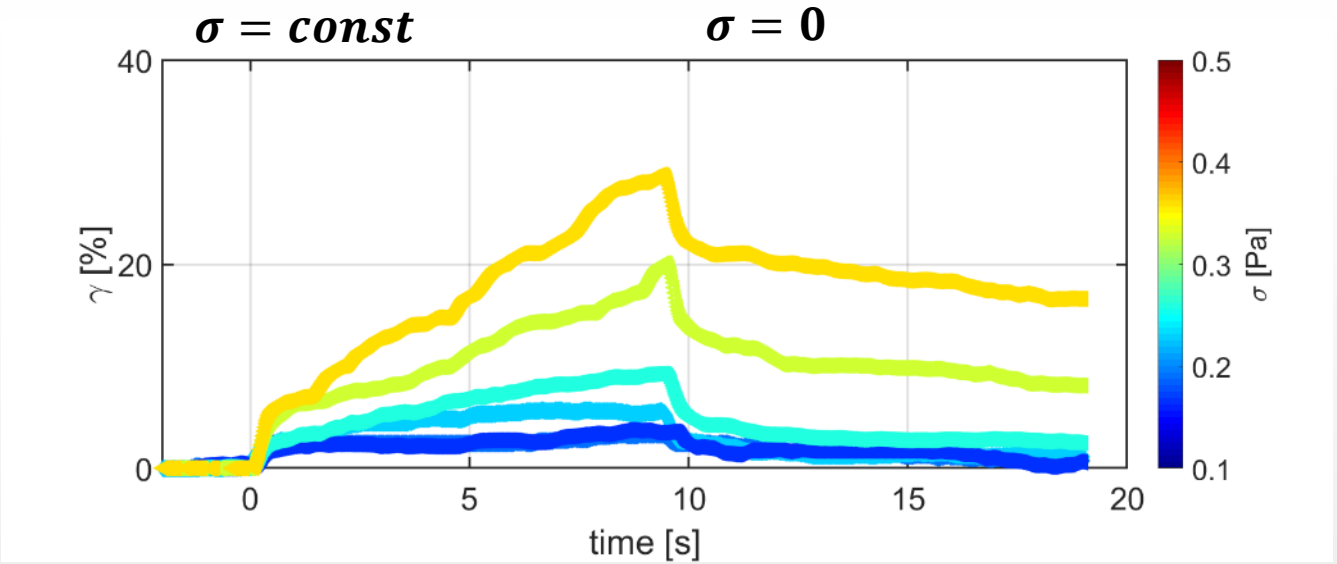
Low stress:

- *Sub-micrometer displacement*
- *Almost elastic response*
- *Full recovery*

Aqua 0.375%

Micro-rheometer

Creep & recovery



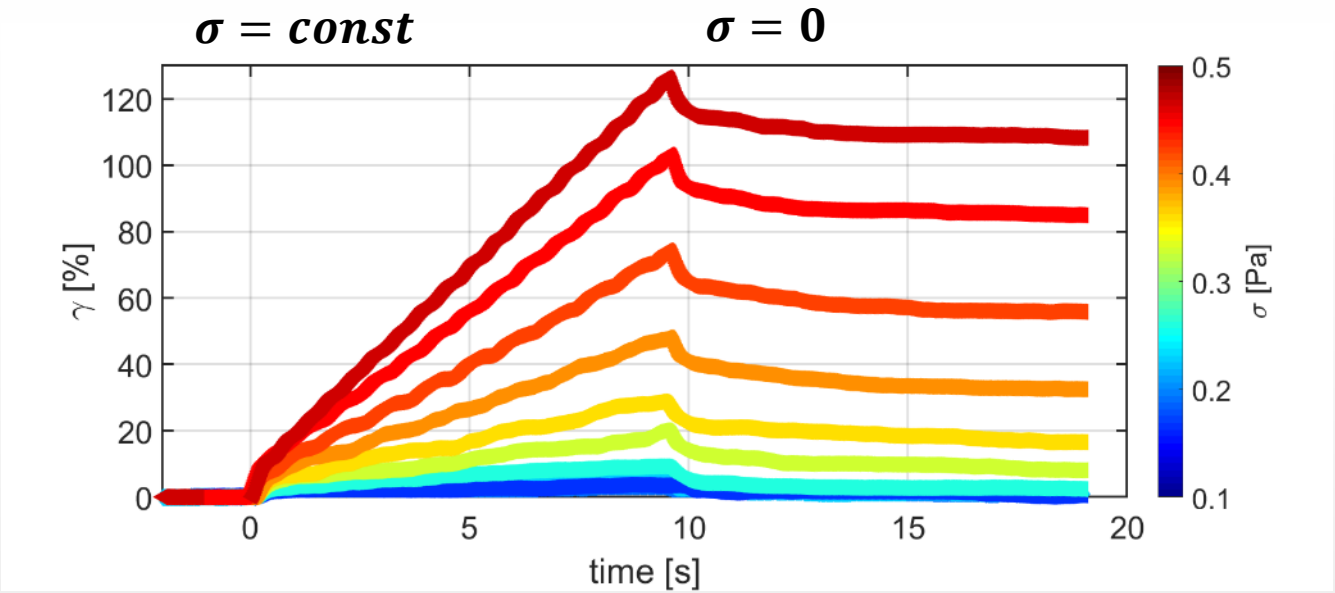
Increasing stress:

- *Small, intermittent yielding events*
- *Progressive loss of recovery (plasticity)*

Aqua 0.375%

Micro-rheometer

Creep & recovery



High stress:

- *Transition to continuous (bumpy) flow*
- *Persistent recovery*

Aqua 0.375%

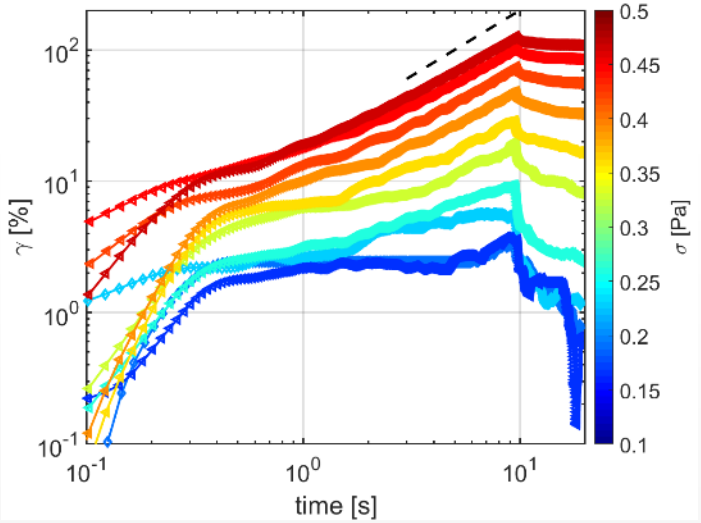
Micro-rheometer: analysis

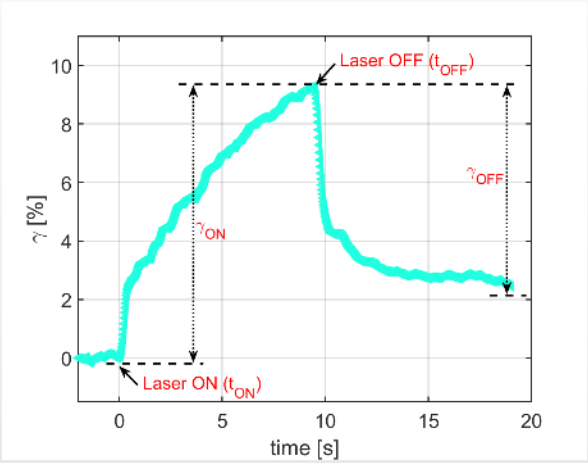
Creep & recovery

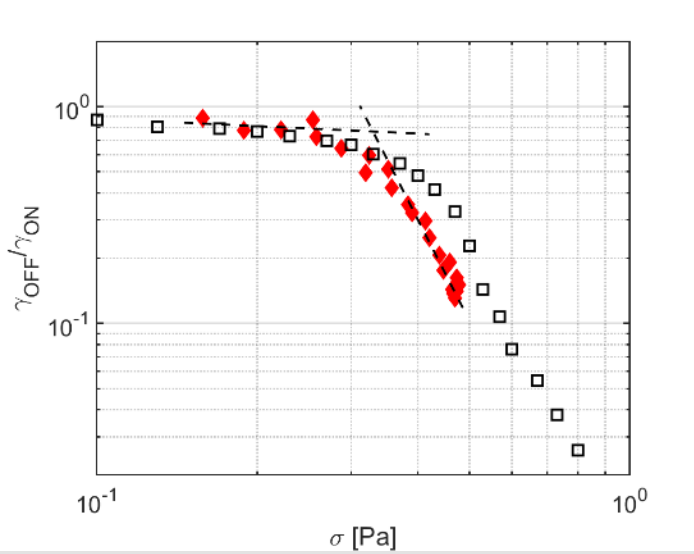
Two criteria for yield stress determination (for both bulk and micro):

- Transition to $\gamma \approx t$

- Fraction of recovered strain: crossover



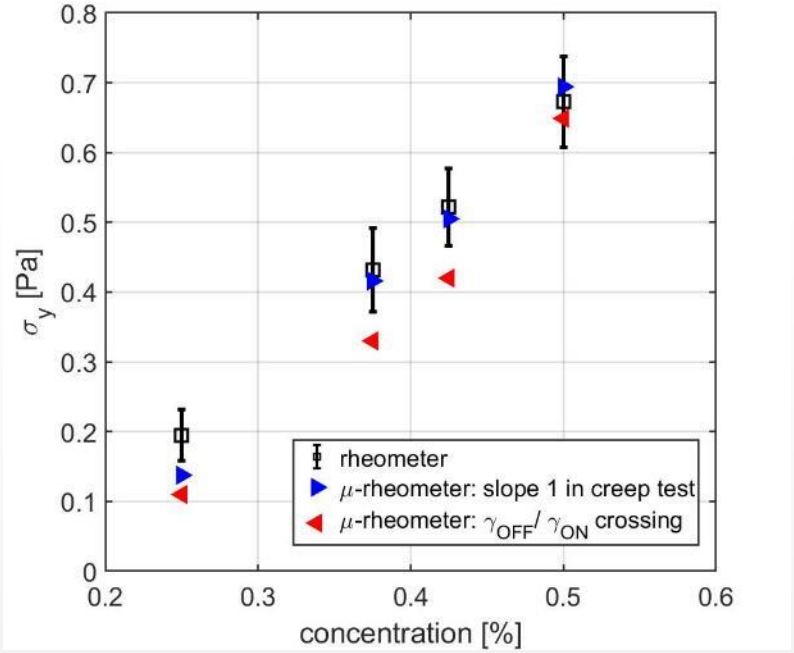




Aqua 0.375%

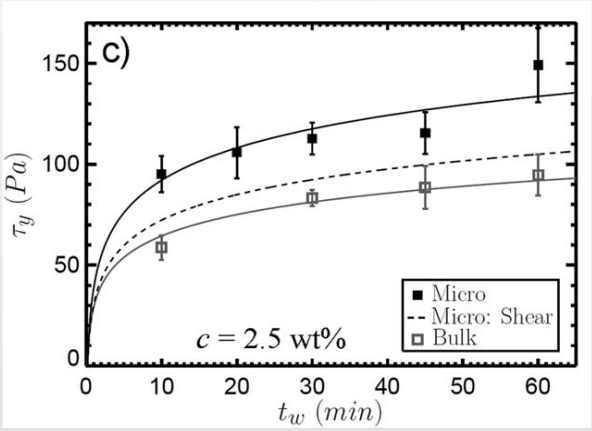
Dinkgreve *et al. J. Non-Newt. Fl. Mech. (*2016)

Micro vs. macro yield stress



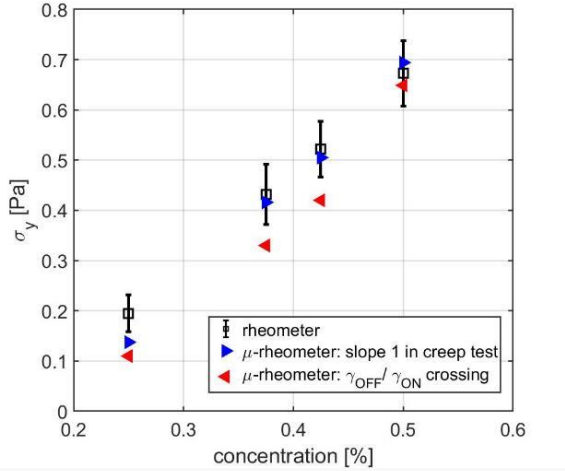
Agreement: not obvious…

Magnetic tweezers on Laponite



Rich et al., Soft Matter (2011)

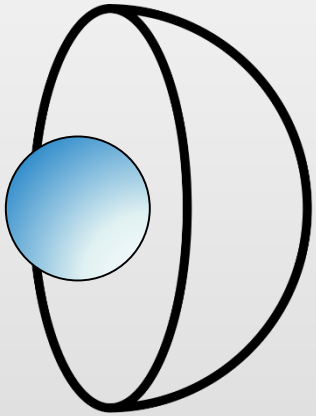
Micro vs. macro yield stress



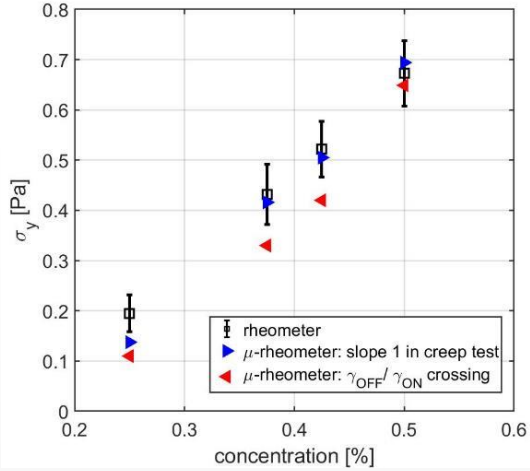
Stress definition (extension of Stokes drag)

$$\sigma = \frac{F}{12\pi r^2} = \frac{F}{2\pi * 6r^2} = \frac{F}{2\pi r_{eff}^2}$$

$$r_{eff} \approx 2.5r$$



Micro vs. macro yield stress

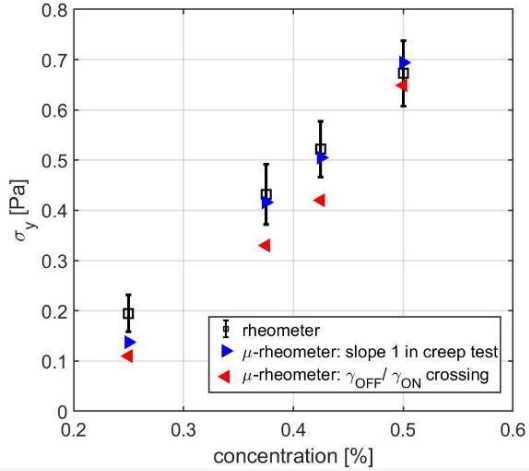


Threshold for particle trapping in yield stress materials

$$Y = \frac{2\pi r^2 \sigma_y}{\frac{4}{3}\pi r^3 (\rho_p - \rho_l) g} \approx 0.15$$

Beris et al. J. Fluid Mech. (1985)
Tabuteau et al. J. of Rheol. (2007)
Emady et al., J. of Rheol. (2013)
Holenberg et al., PRE (2012)

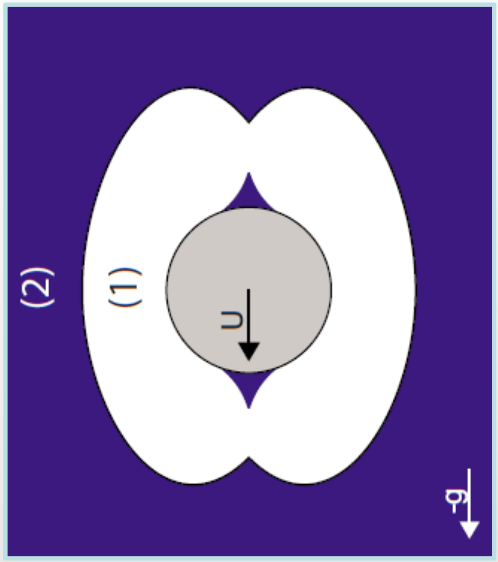
Micro vs. macro yield stress



Threshold for particle trapping in yield stress materials

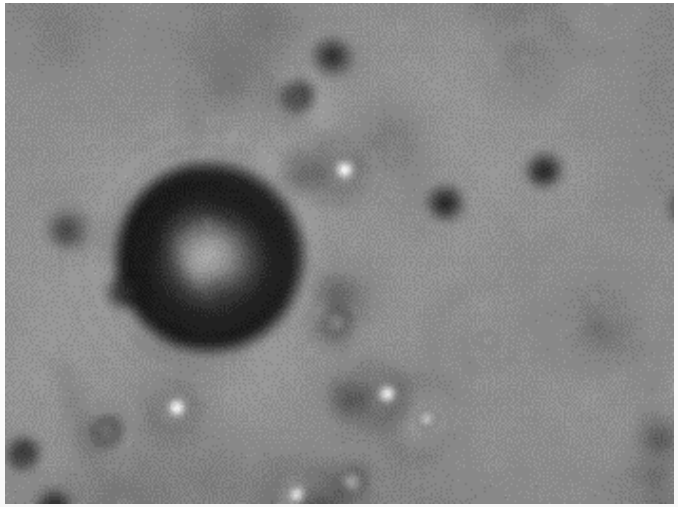
$$Y = \frac{2\pi r^2 \sigma_y}{\frac{4}{3}\pi r^3 (\rho_p - \rho_l) g} \approx 0.15$$

$$r_{eff} \approx 2.5r$$

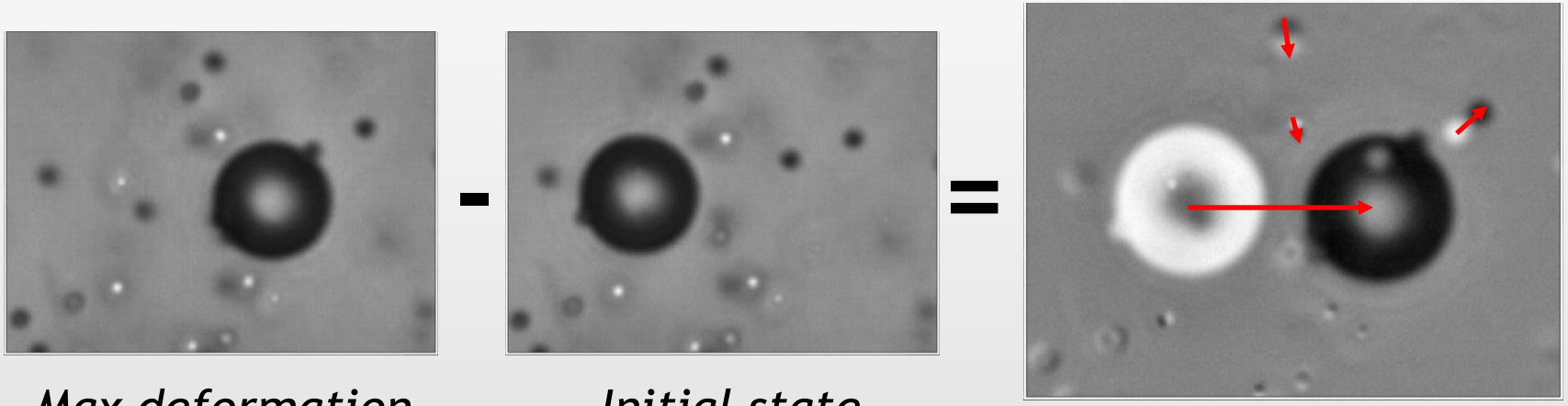


Beris et al. J. Fluid Mech. (1985)
Tabuteau et al. J. of Rheol. (2007)
Emady et al., J. of Rheol. (2013)
Holenberg et al., PRE (2012)

Imaging



Sub-micron tracer particles

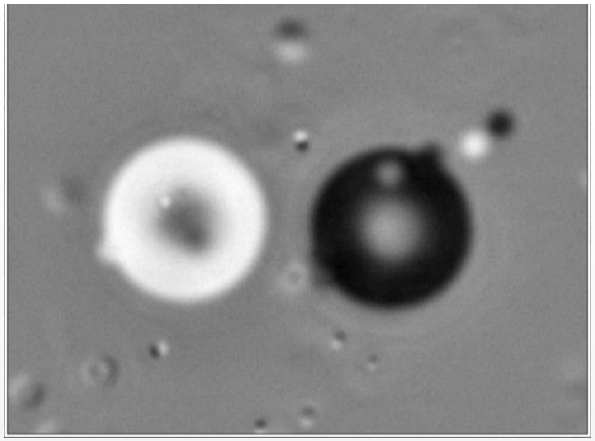


Max deformation *Initial state* *Deformation 'field'*

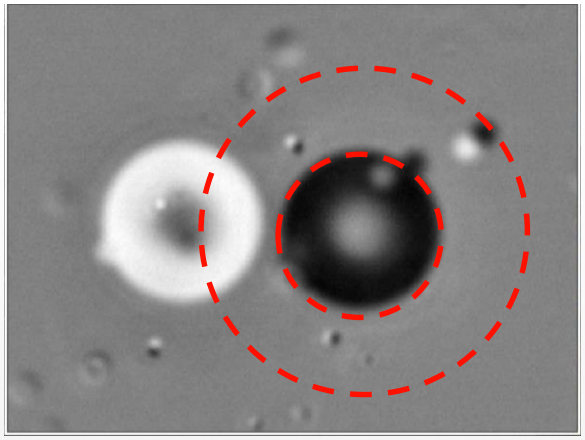
Aqua 0.375%, σ = 0.5 Pa

Imaging

Deformation 'field'



At max deformation

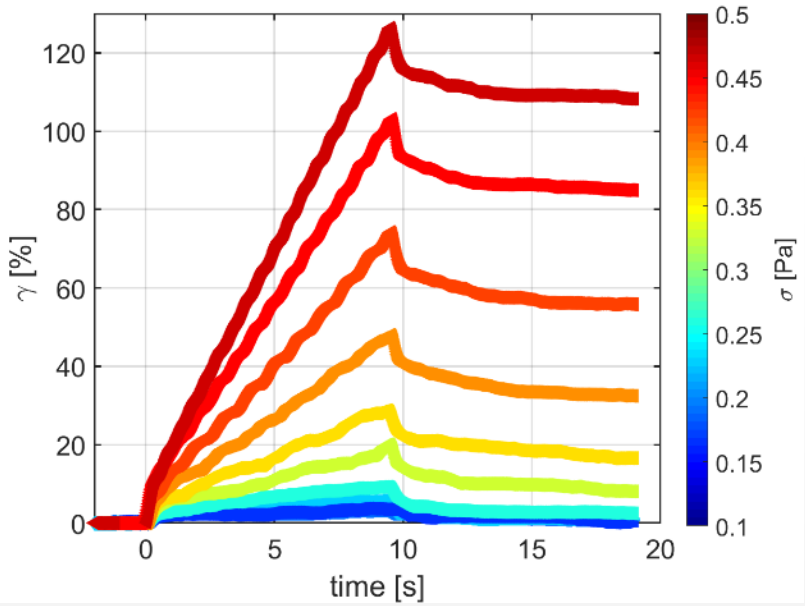


After recovery

The moving microbead permanently displaces regions of the material at least 2r away

Aqua 0.375%, σ = 0.5 Pa

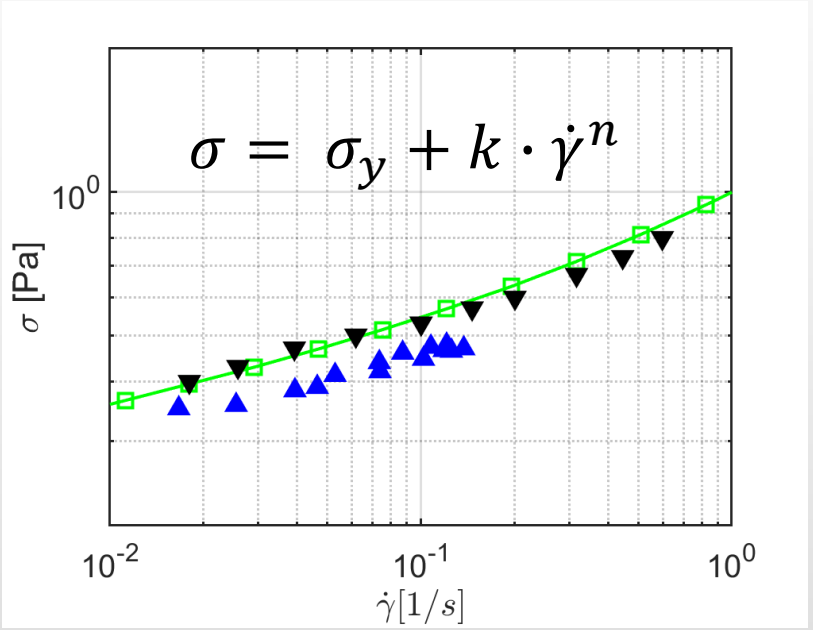
Fluid regime



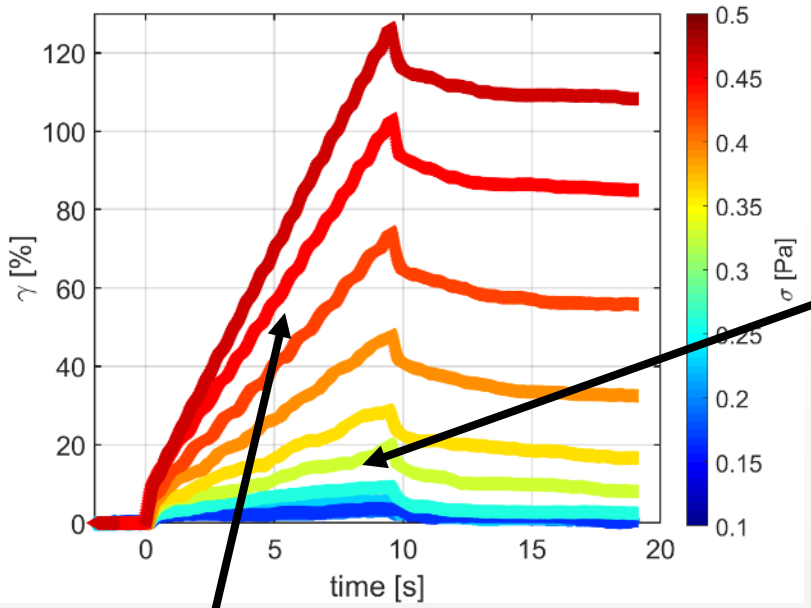
$$\dot{\gamma} = \frac{v}{2r}$$

(average strain rate around the particle)

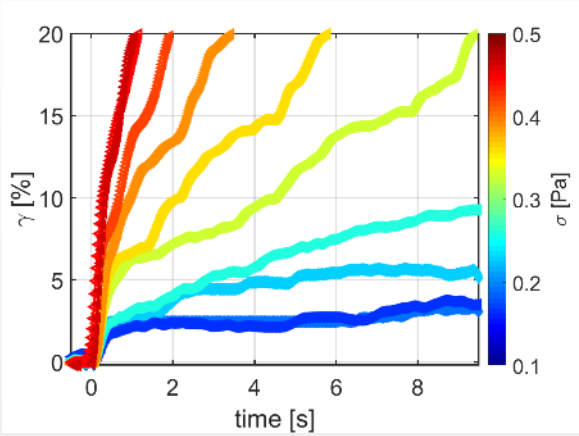
Similar values and scaling to ***bulk creep*** *and* ***bulk teady shear***



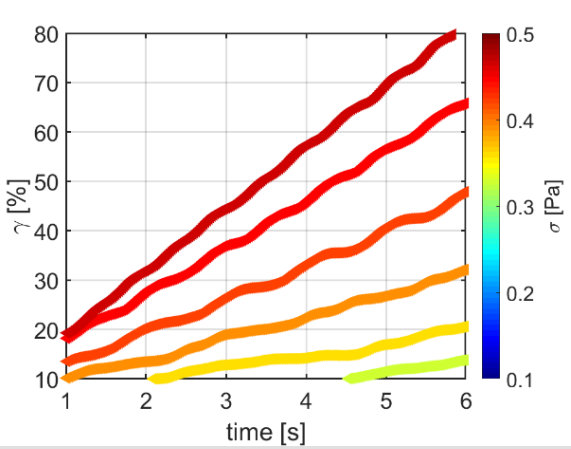
Fluid regime



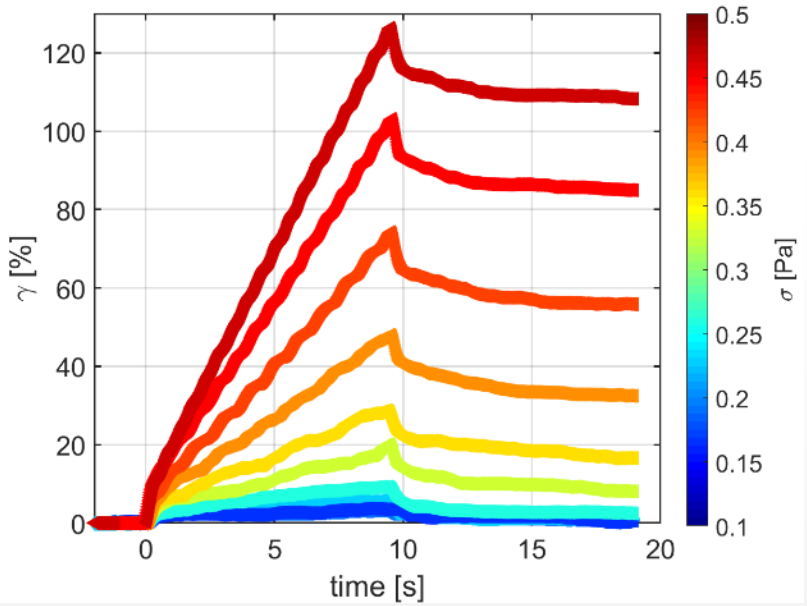
Intermittent jumps

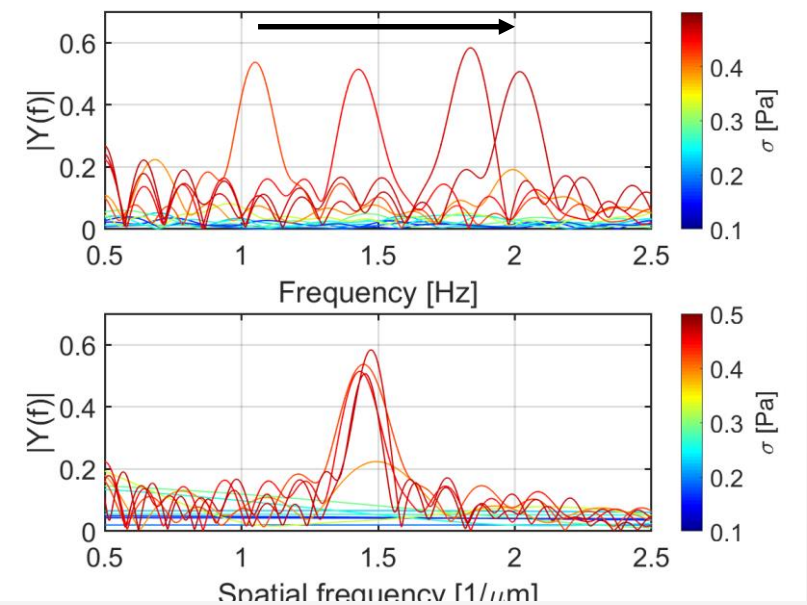


Periodic bumps



Fluid regime

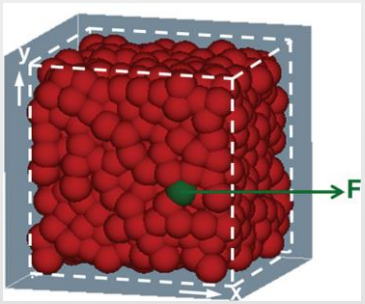


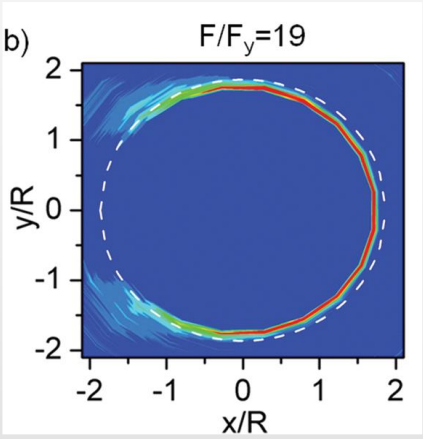


Temporal & spatial Fourier modes of velocity

- *Increasing frequency with stress*
- *Regular steps (≈700 nm)*

- *Compatible with accumulation and rearrangement of microgels in front of the microbead*





Mohan et al. J. of Rheol. (2014)

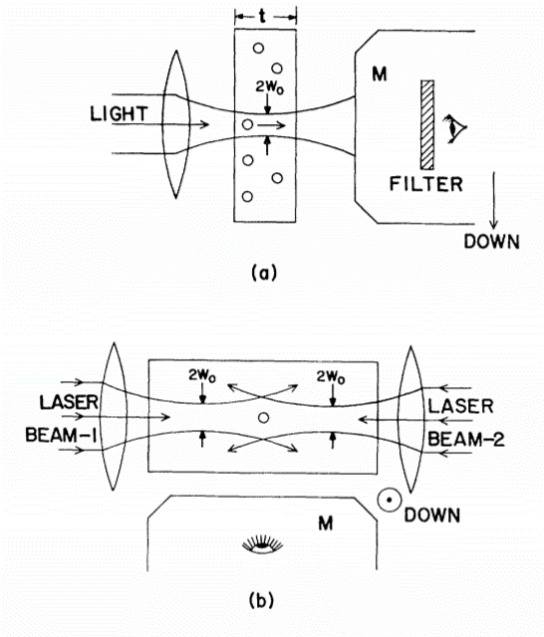
Conclusions

- *An integrated optofluidic micro-rheometer is proposed for creep experiments, with forces up to 1 nN*
- *We report the first optical measurement of local yield stress values, in good agreement with bulk estimates*
- *We find agreement also in the fluid regime, but with distinct signatures of microscopic rearrangements*
- *We investigate the material around the microbead and find a plastically deformed/fluidized region consistent with previous observations with large spheres and with mean stress definition*

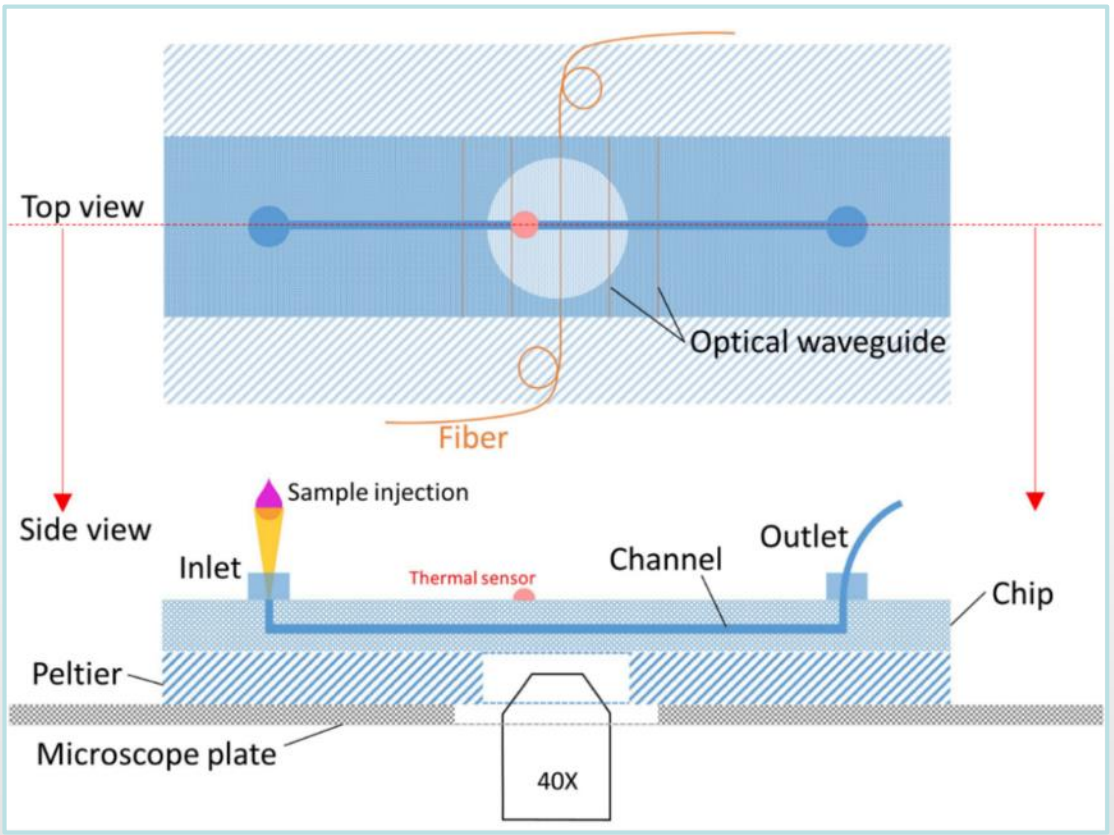
What's next?

- *Build-up of the fluidized region*
- *Oscillatory, nonlinear measurements*

Optofluidic micro-rheometer



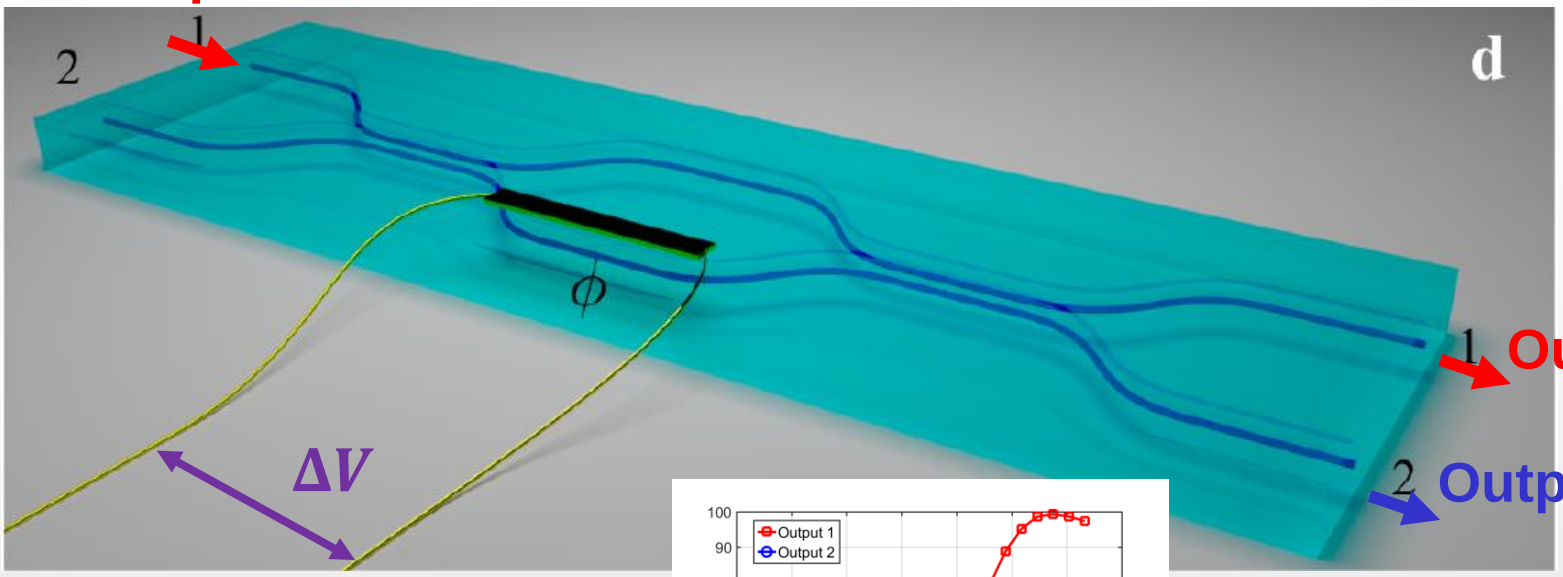
Convenient geometry for time modulation!

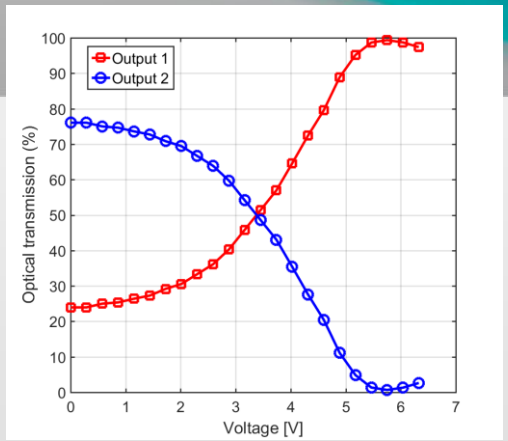


Yang et al. Micromachines (2017)

Optical modulator

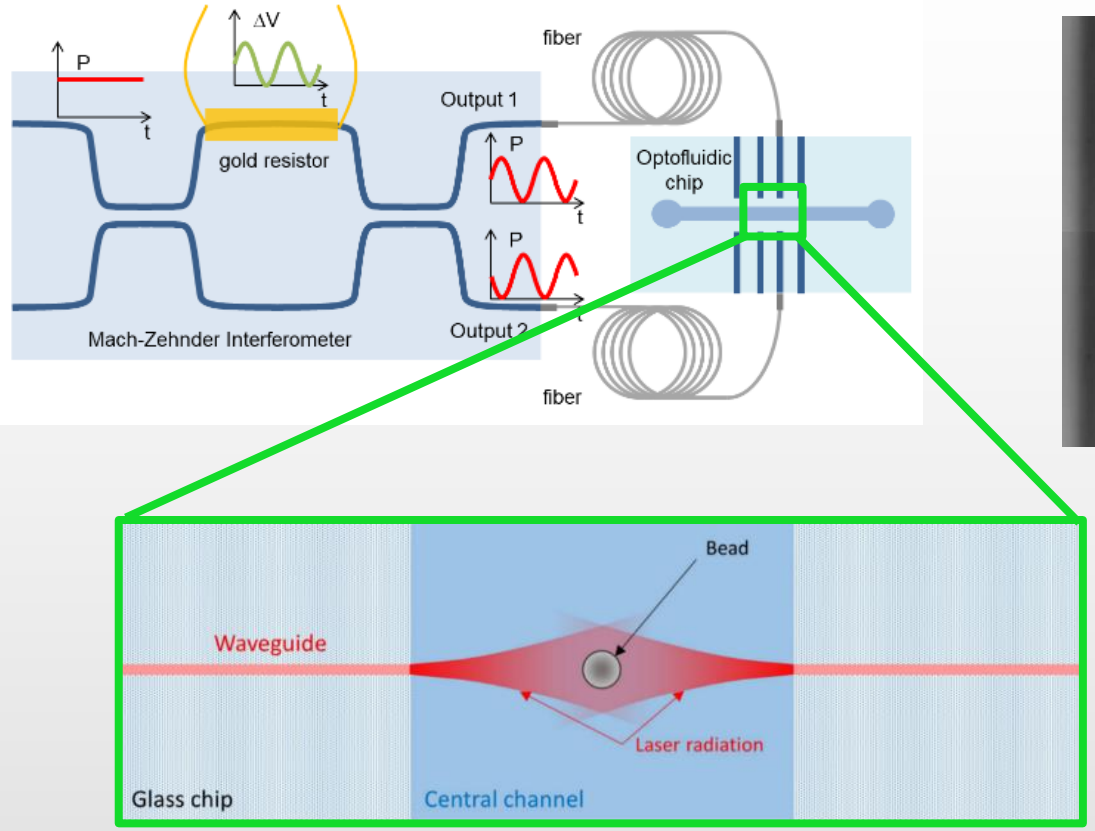
Integrated Mach-Zehnder interferometer

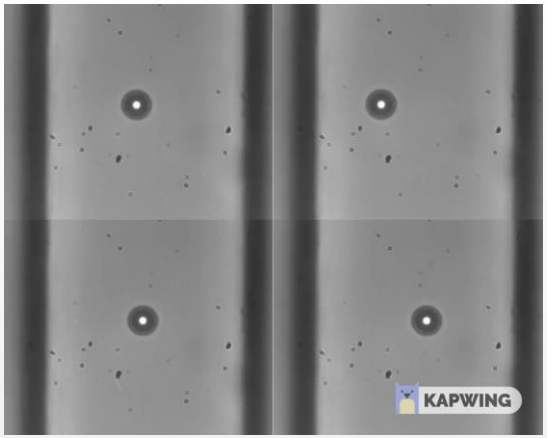




Oscillatory micro-rheometer

Counter-propagating beams +
integrated optical modulator

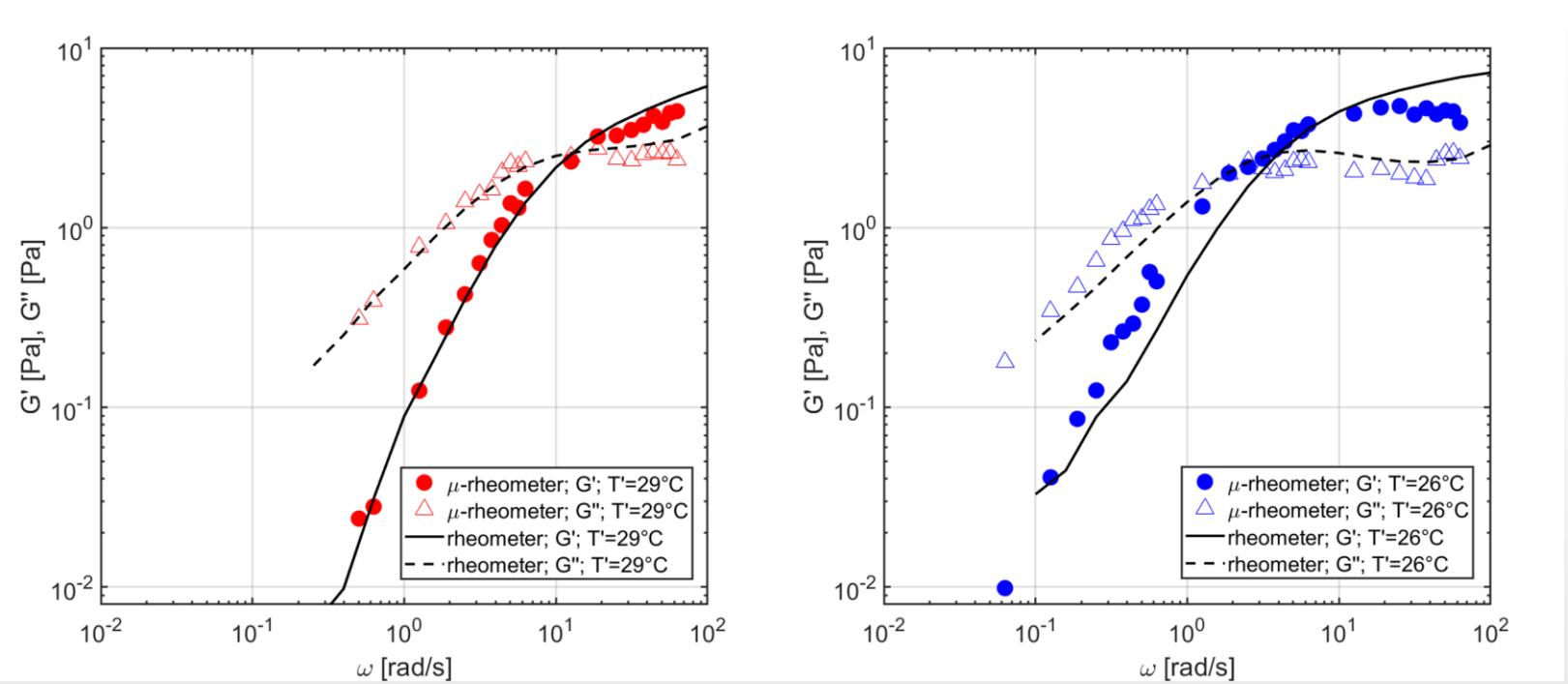




Vitali et al., Sci.Rep. (2020)

Surfactant wormlike micelles

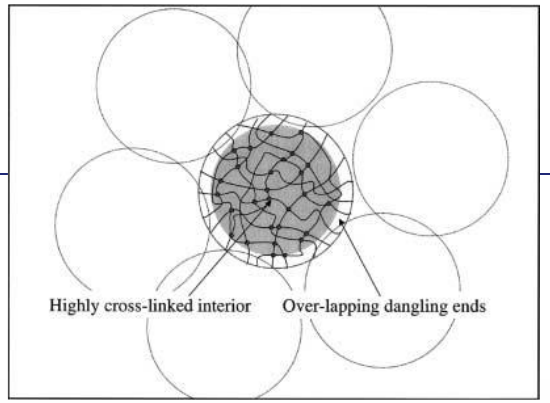
Linear oscillatory rheology

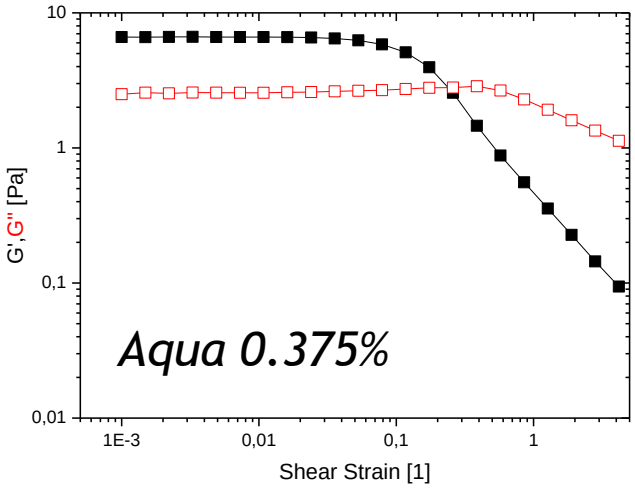


100 mM CpyCl + NaSal

Vitali et al., Sci.Rep. (2020)

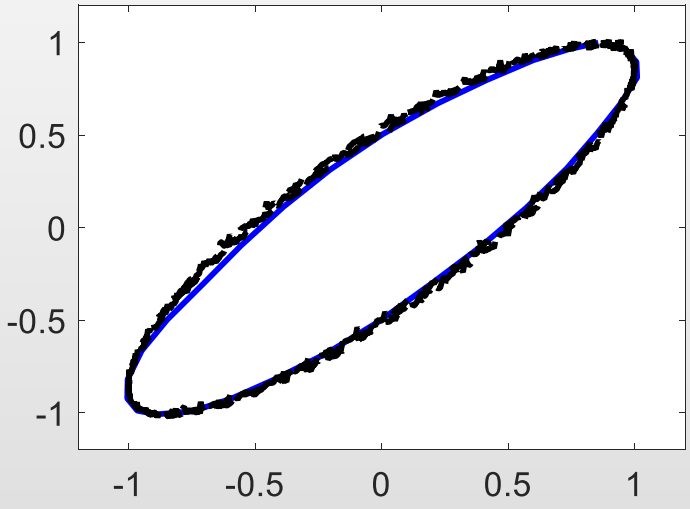
Beyond linear regime



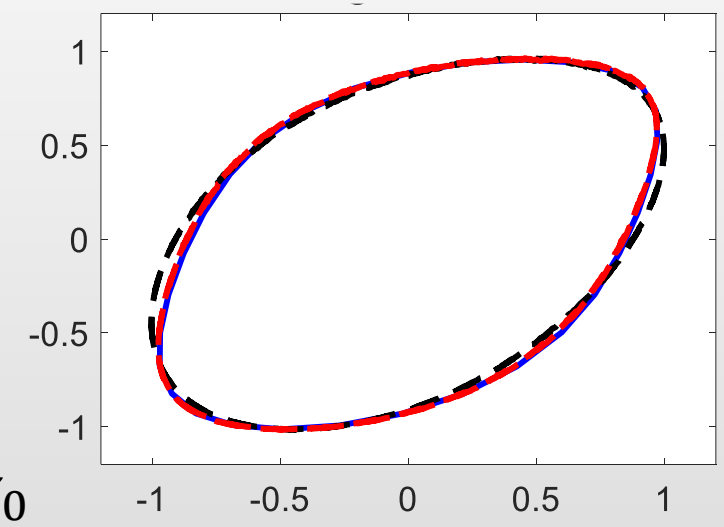


σ/σ_0

Low stress



High stress



γ/γ_0

Conclusions

- *An integrated optofluidic micro-rheometer is proposed for creep experiments, with forces up to 1 nN*
- *We report the first optical measurement of local yield stress values, in good agreement with bulk estimates*
- *We find agreement also in the fluid regime, but with distinct signatures of microscopic rearrangements*
- *We investigate the material around the microbead and find a plastically deformed/fluidized region consistent with previous observations with large spheres and with mean stress definition*

What's next?

- *Build-up of the fluidized region*
- *Oscillatory, nonlinear measurements*

Grazie!