

# Flows of suspensions of particles in yield stress fluids studied by X-ray microtomography

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# Motivations

## Dense suspensions and rheology



*Oursi, Burkina Faso, courtesy of Xavier Chateau*

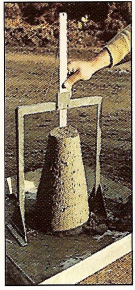
**Can be easily shaped**

**Yield stress fluids**



**Stands on a wall**

*Saint-Gobain Weber*



**Hard to cast...**

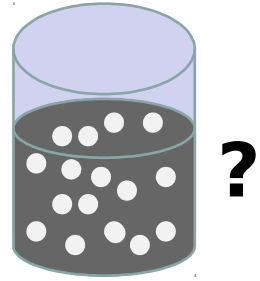
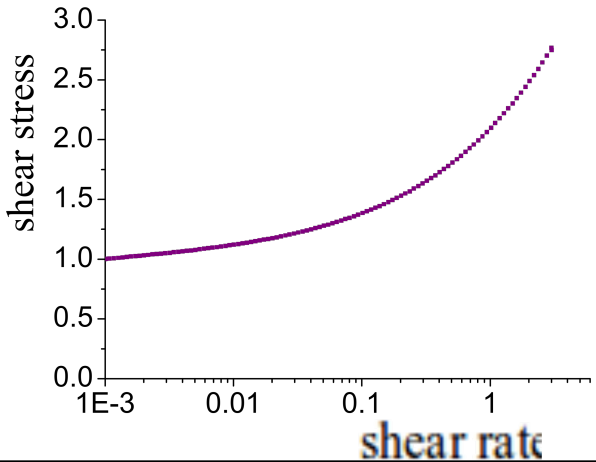
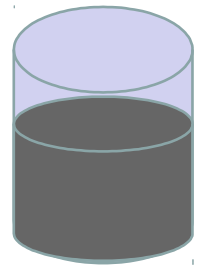
## Suspension of particles in a yield stress fluid



Scale  
Separation

*Other examples: debris flow, coal slurries, food industry...  
Composite materials (reinforced with particles)*

Paste = yield stress fluid

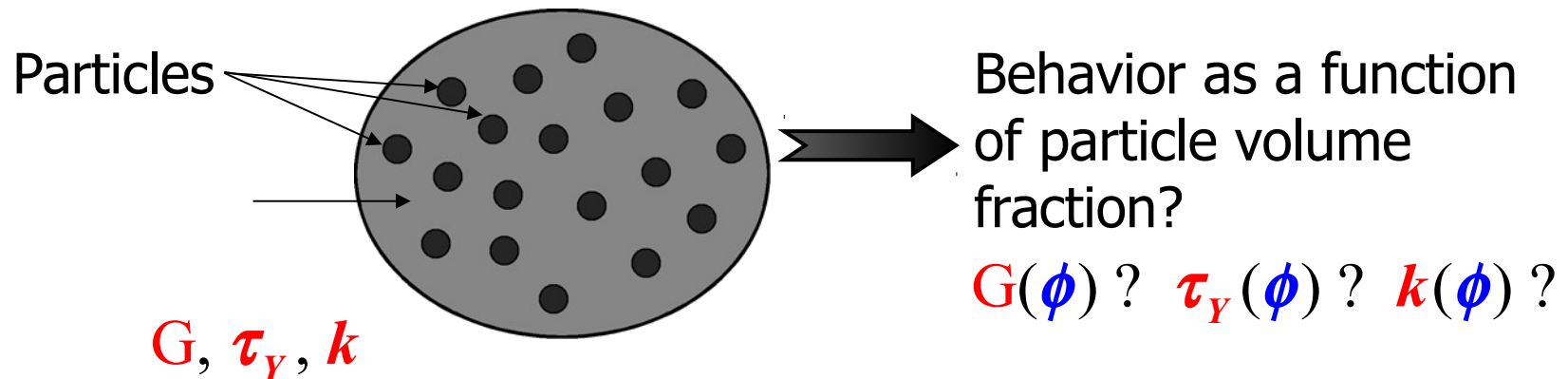


# Model materials

Focus on cases where only **Mechanical Interactions** between particles

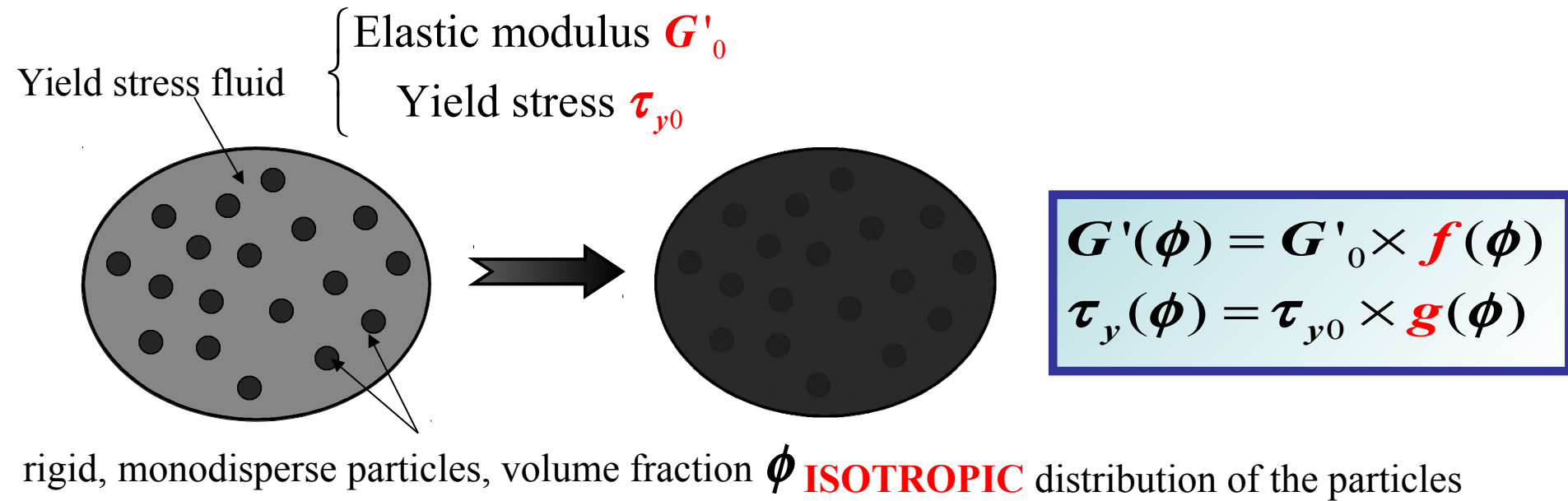
*(i.e. no specific physicochemical interactions between materials)*

- Common framework for **model** and **experiments**

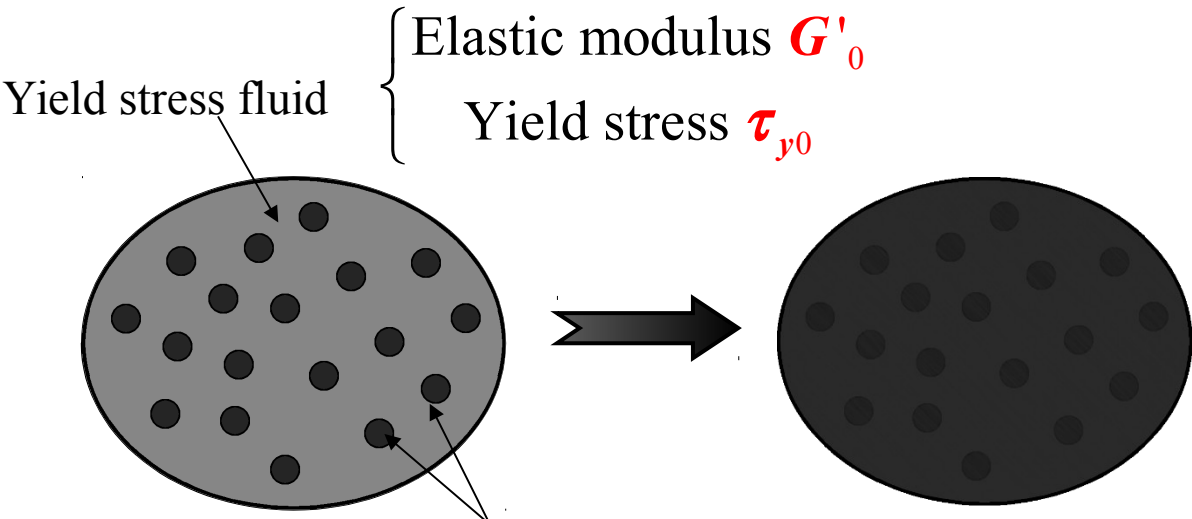




# Role of microstructure



# Role of microstructure

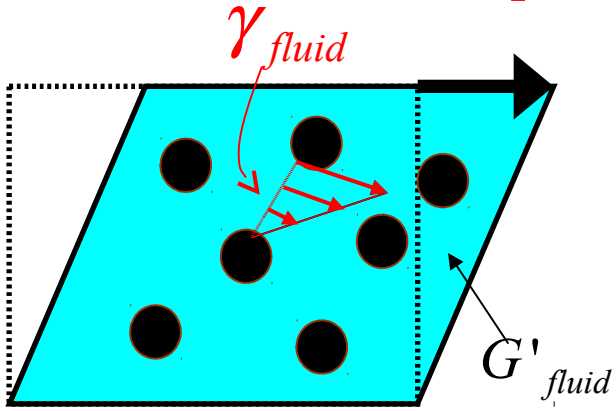


$$G'(\phi) = G'_0 \times f(\phi)$$

$$\tau_y(\phi) = \tau_{y0} \times g(\phi)$$

rigid, monodisperse particles, volume fraction  $\phi$  ~~ISOTROPIC~~ distribution of the particles

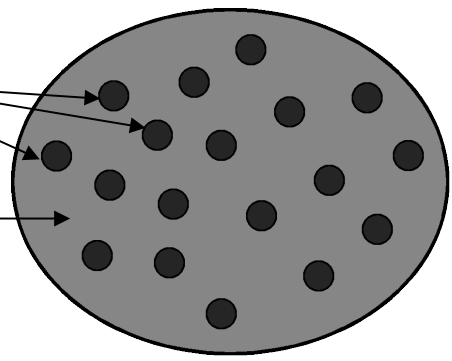
strain in the interstitial fluid **depends on microstructure**



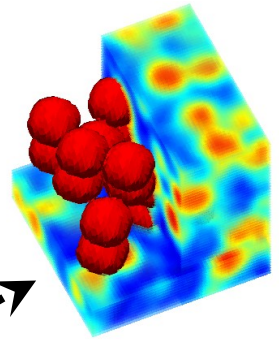
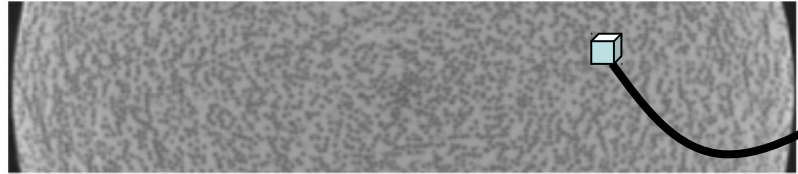
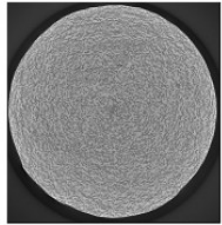
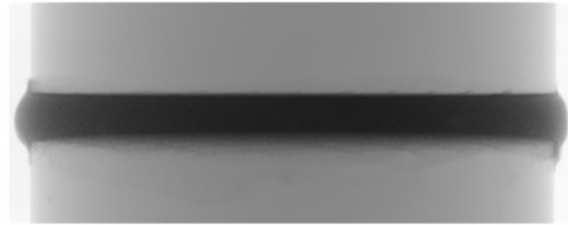
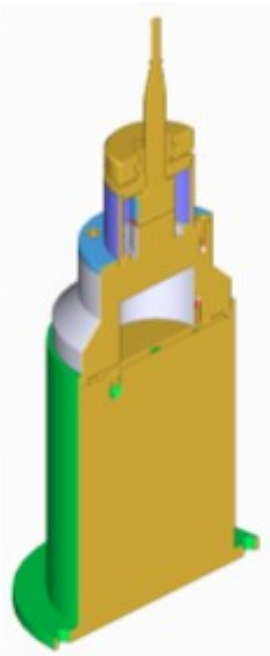
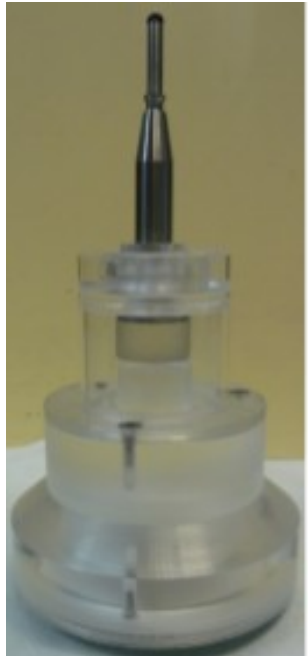
# Material & methods

Monodisperse PS Particles (140  $\mu\text{m}$ )

Concentrated Emulsion  
(80% NaI solution in dodecane)



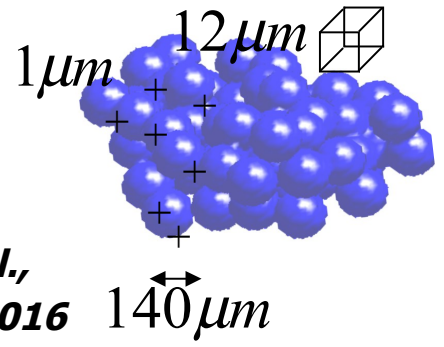
1- **finding** a good particle/fluid contrast material + stability for 3D scan  $\rightarrow$  dodecane with NaI + PS beads (140 $\mu\text{m}$ )



2- **loading/shearing** done ex-situ with a parallel plate configuration  
Dia. 20mm & height 2mm



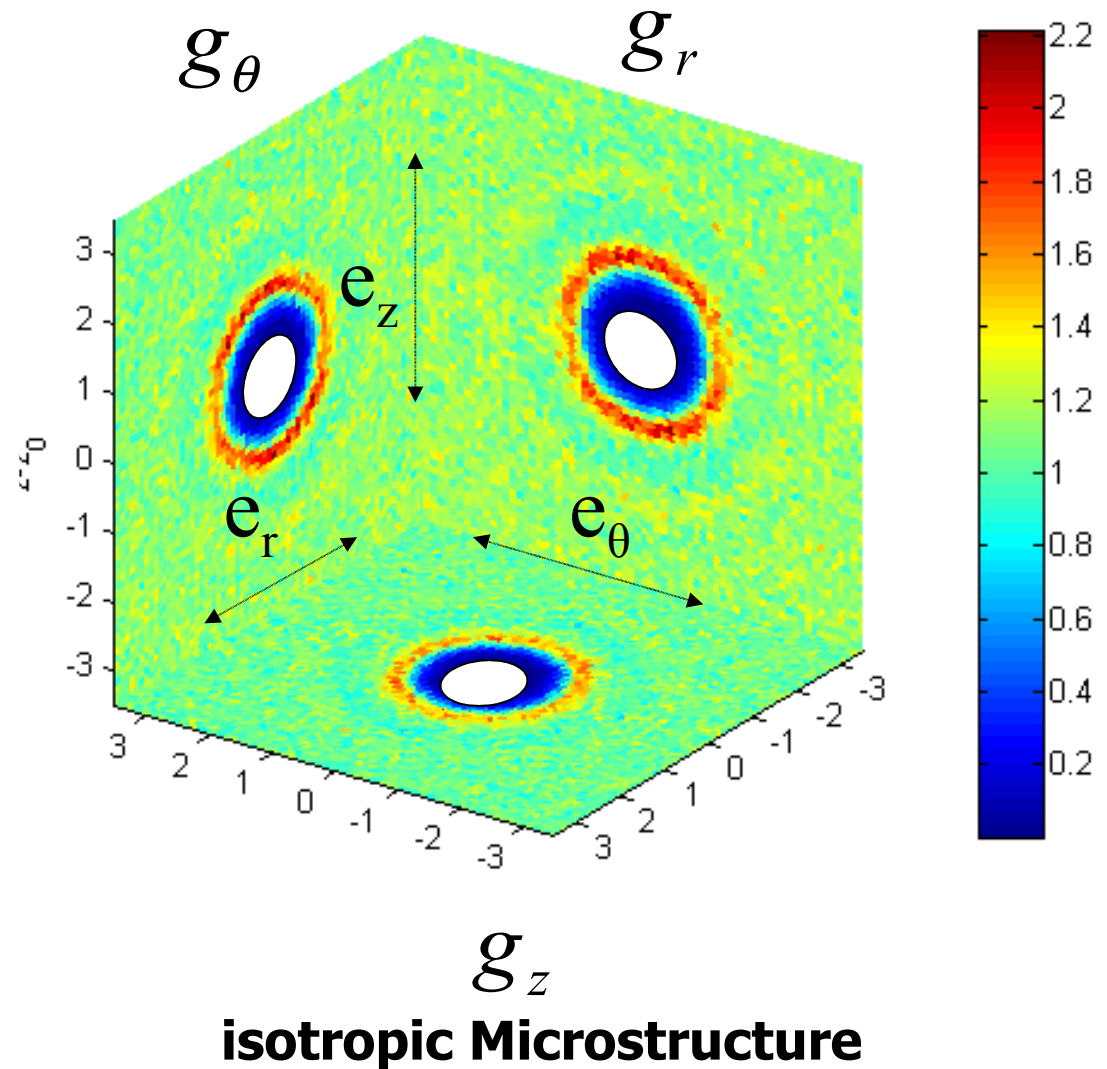
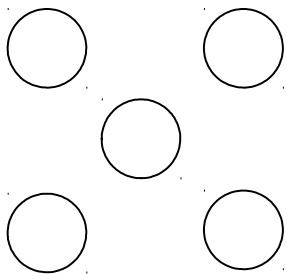
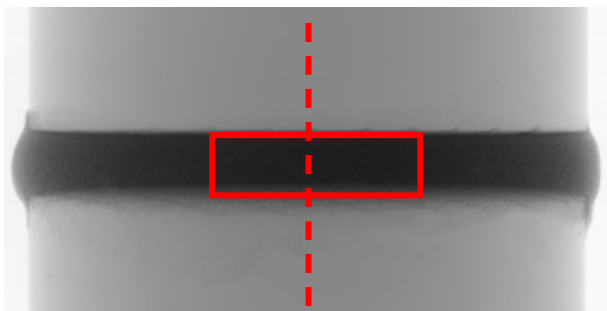
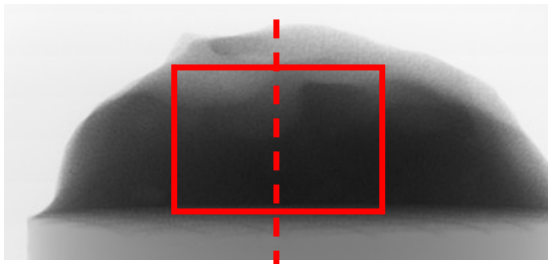
3- **scan** : 1 hour with a 12 $\mu\text{m}$  voxel size



**Deboeuf et al., unpublished 2016**

# Pair distribution function: prepared suspension

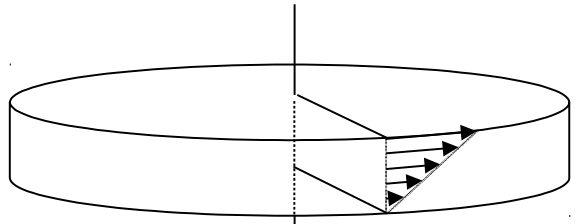
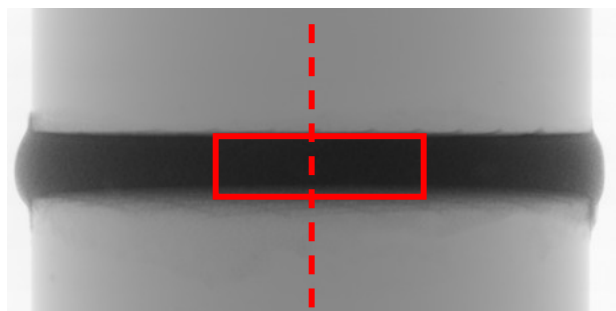
No preshear and load



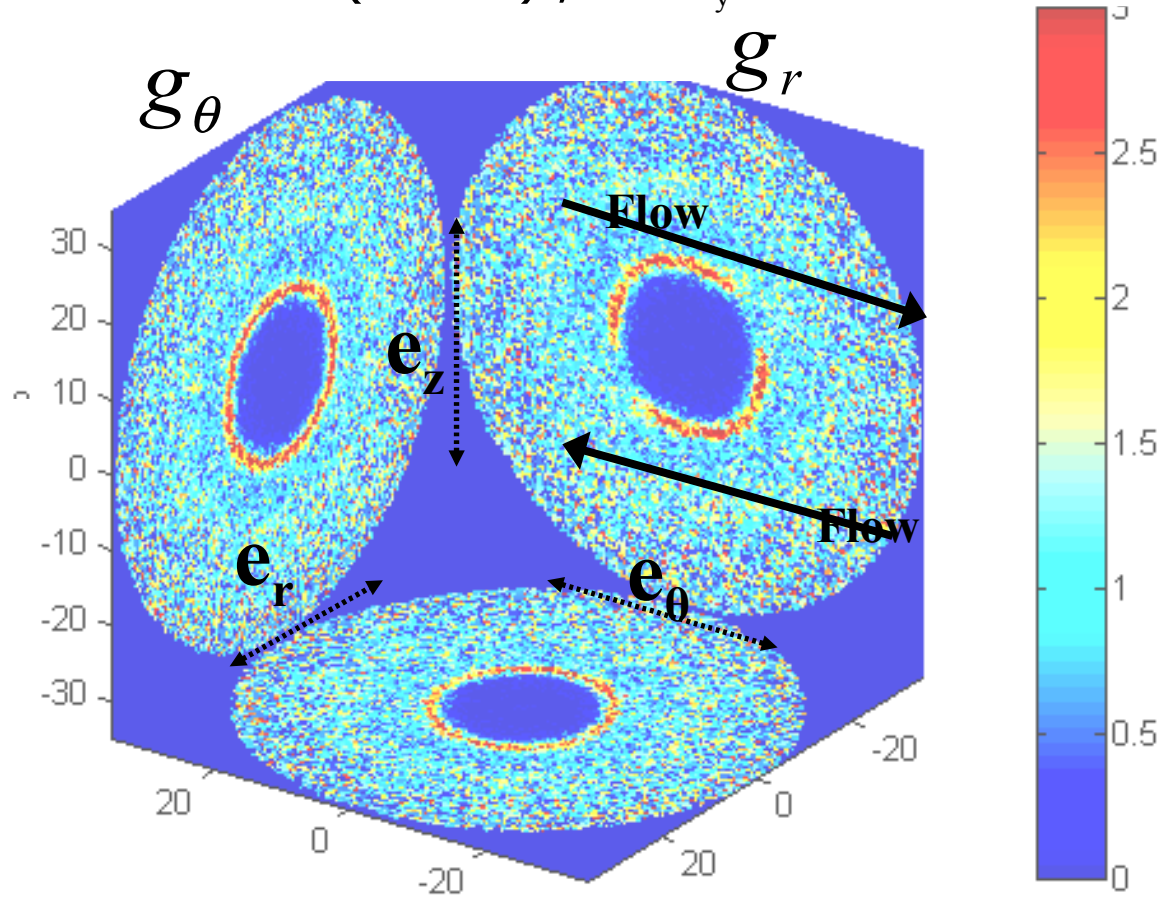
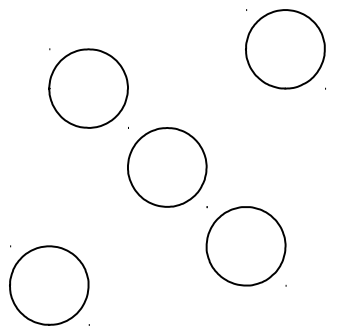


# Pair distribution function: sheared suspension

Steady state at low rate ( $10^{-2} \text{ s}^{-1}$ ) ;  $\tau \approx \tau_y$



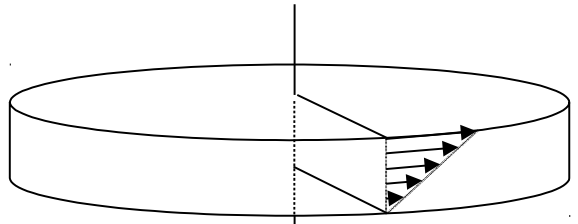
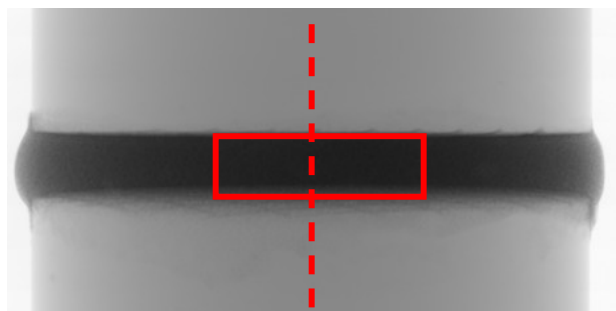
Azimuthal flow  $v_\theta(r, z)$



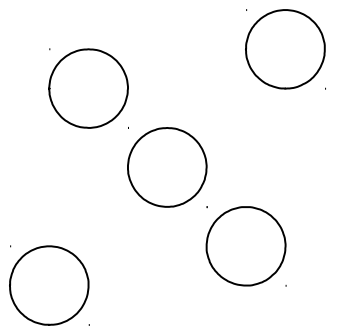
$g_z$   
**anisotropic in shear plane**  
**isotropic in other planes**

# Pair distribution function: sheared suspension

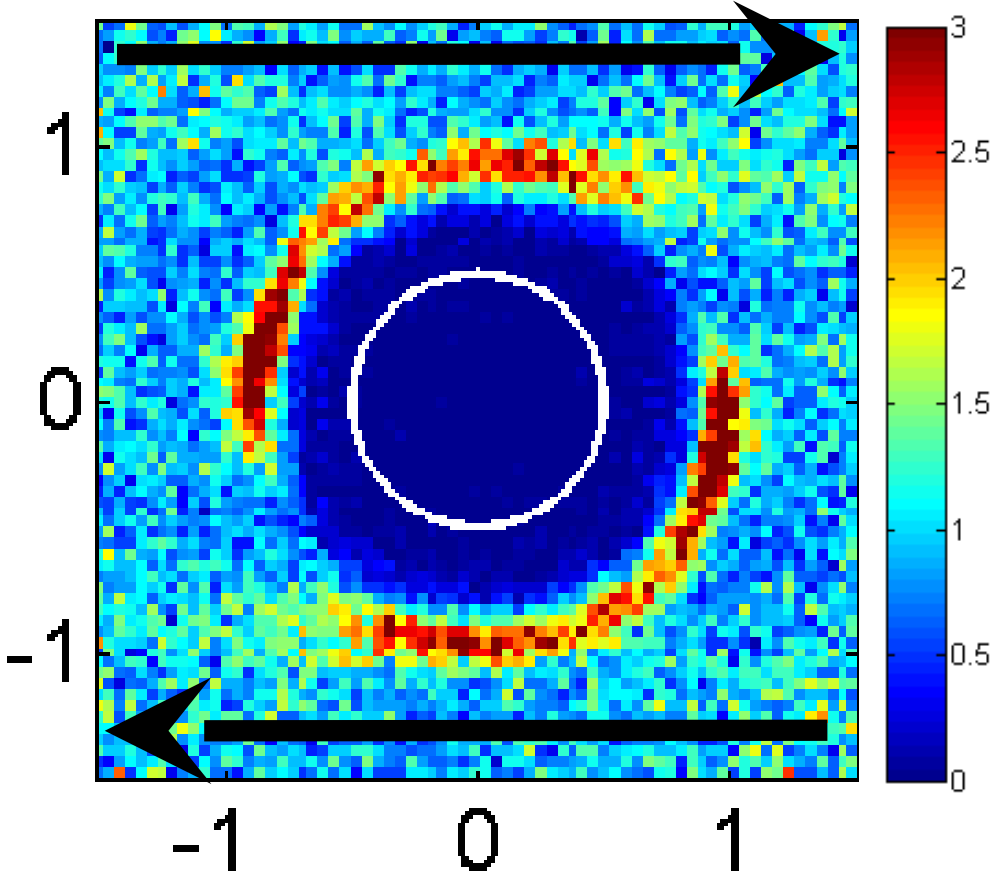
Steady state at low rate ( $10^{-2} \text{ s}^{-1}$ ) ;  $\tau \approx \tau_y$



Azimuthal flow  $v_\theta(r, z)$



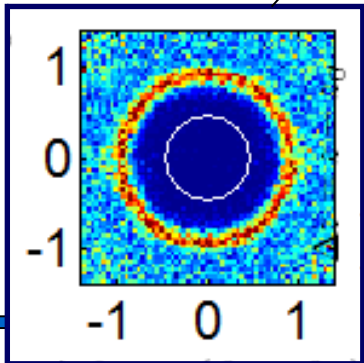
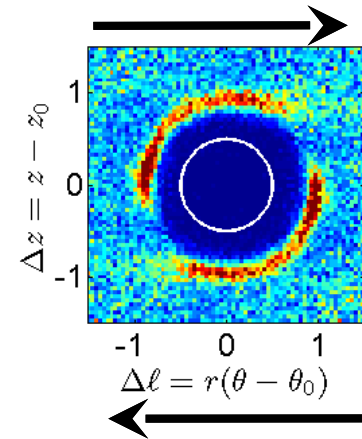
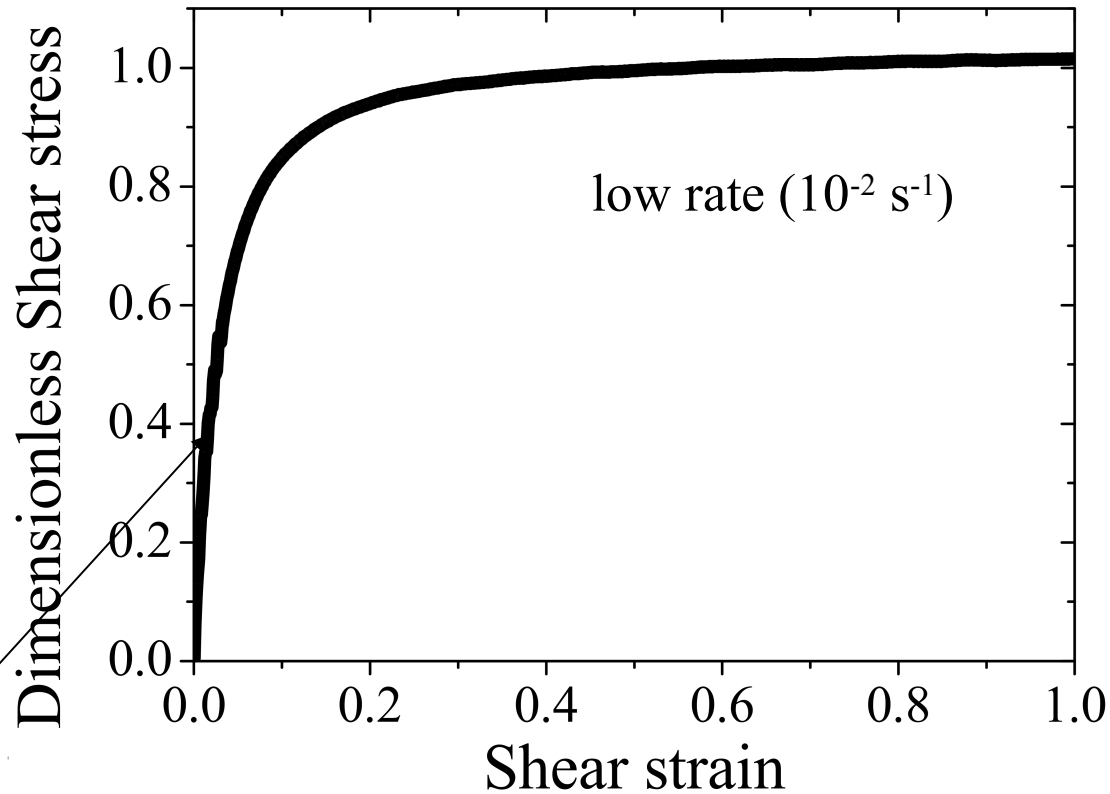
In the shear plane ( $e_\theta, e_z$ )



**anisotropic in shear plane**  
**isotropic in other planes**

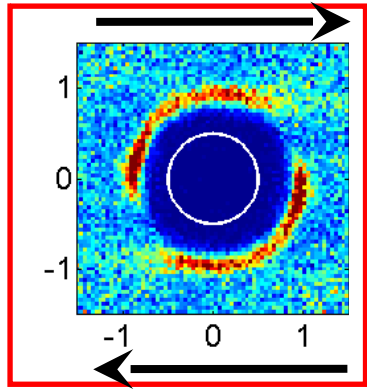
# Dependence on shear history

Elastoplastic response **from initial state (isotropic)**

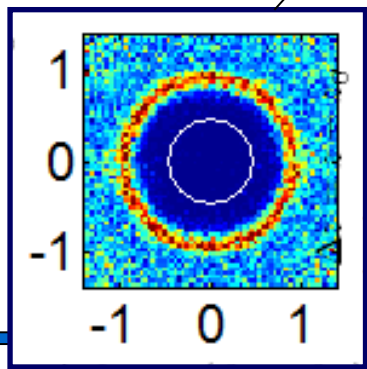
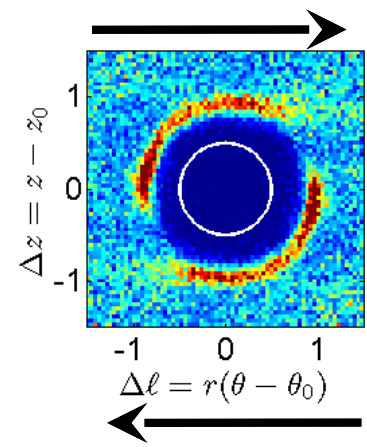
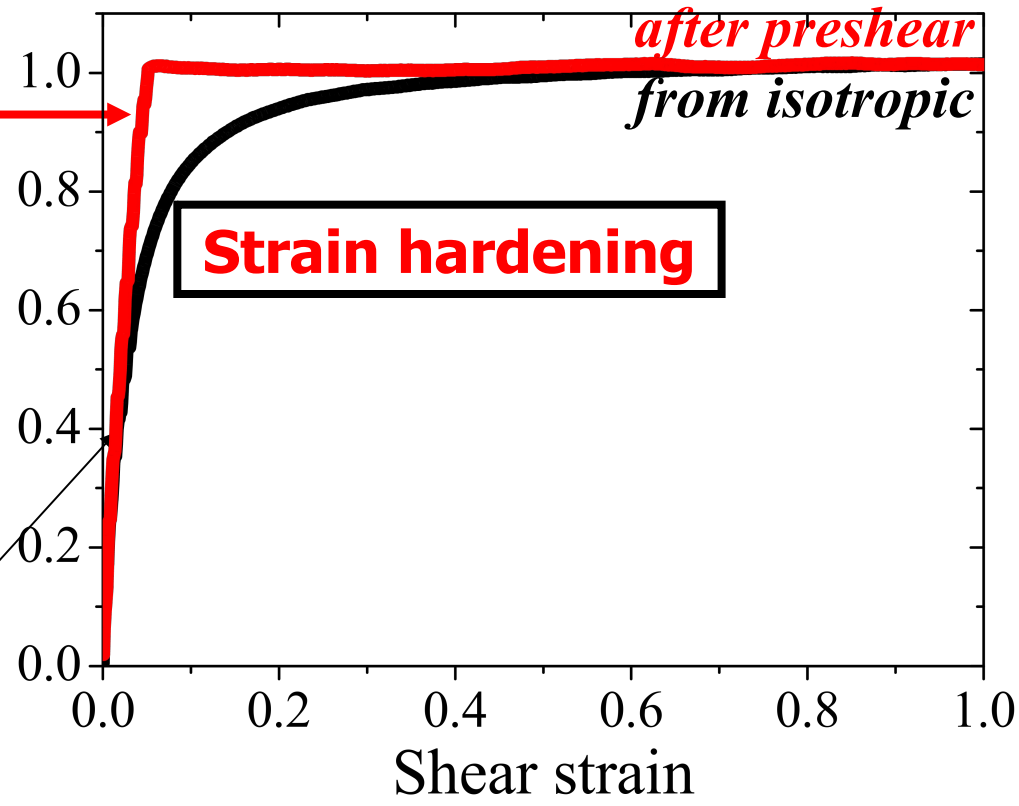


# Dependence on shear history

Elastoplastic response **after preshear**



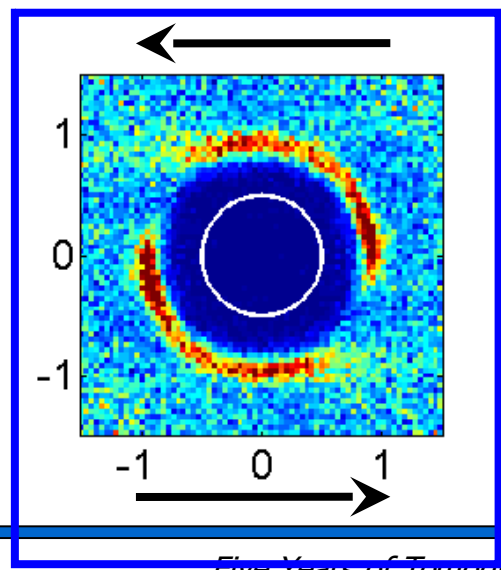
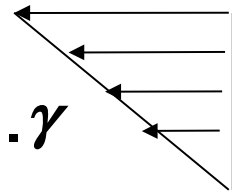
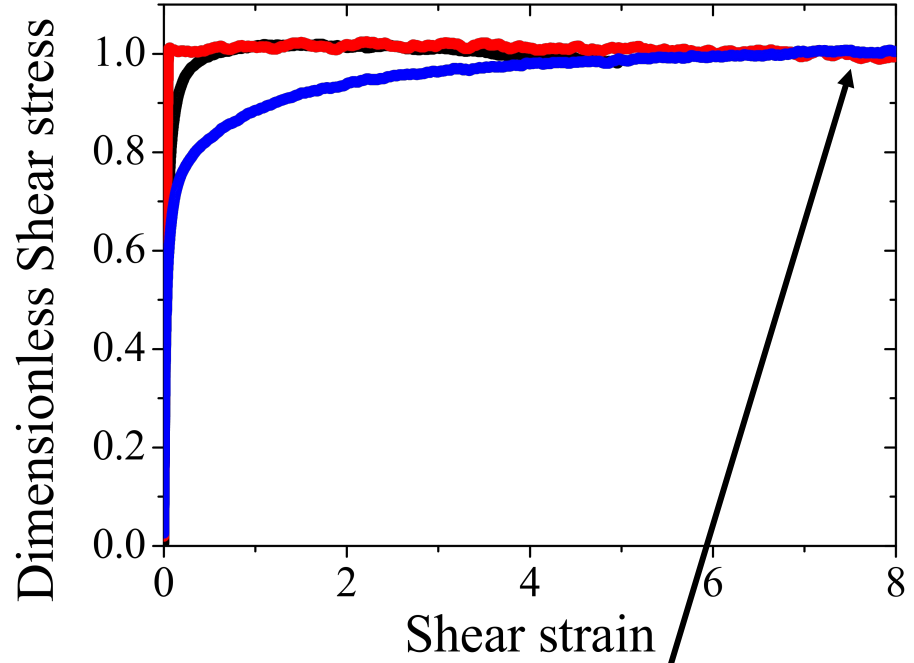
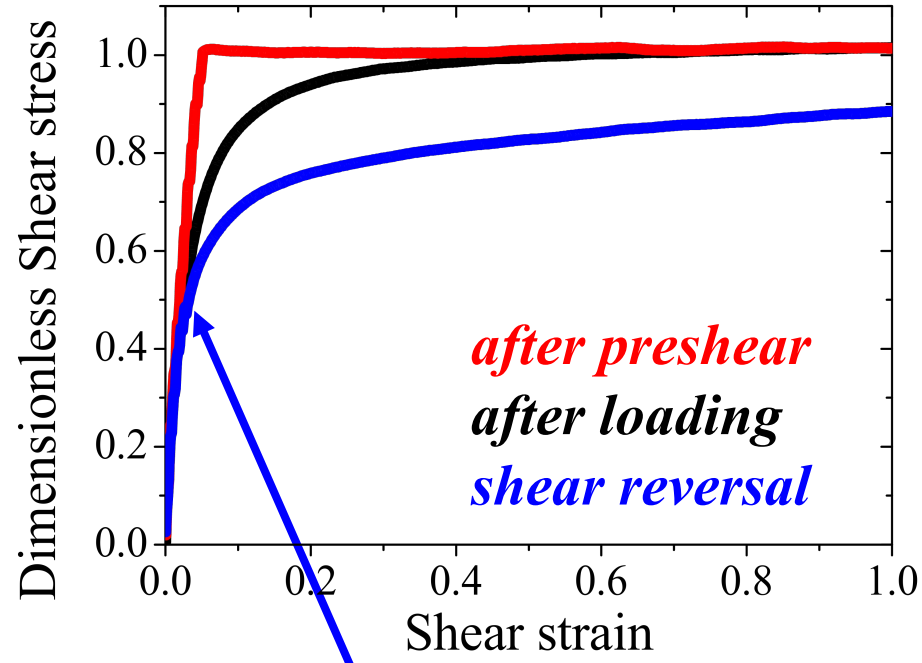
Dimensionless Shear stress



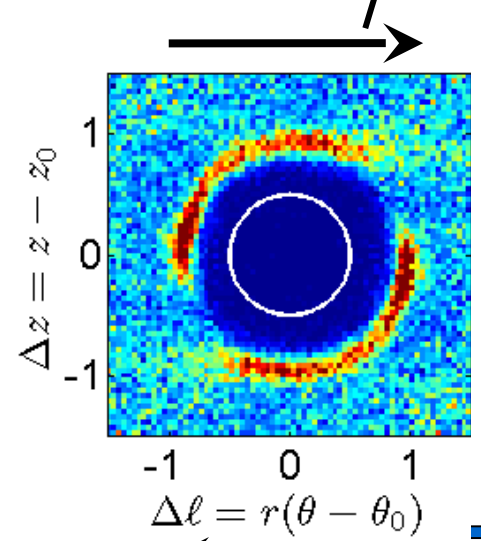
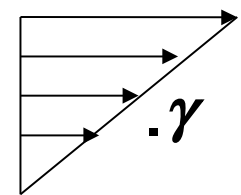
Initial microstructure = final microstructure  
Almost perfect elastoplastic behavior



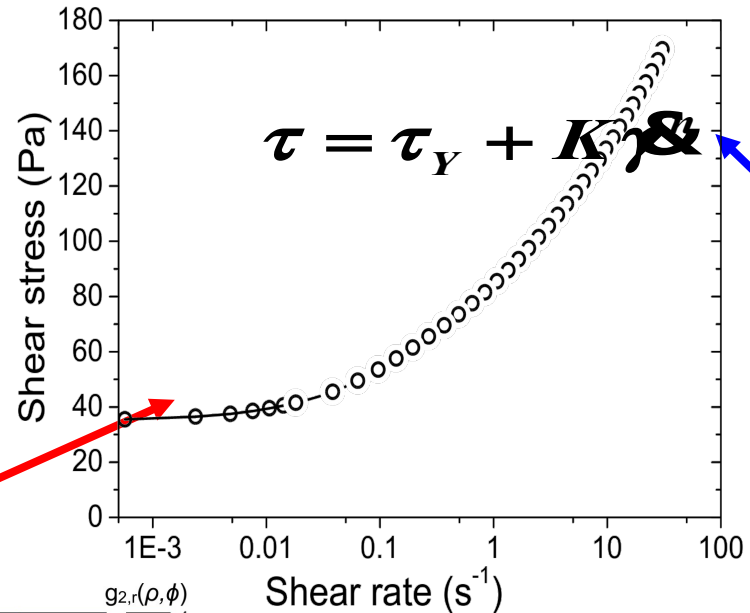
# Dependence on shear history



*shear reversal*



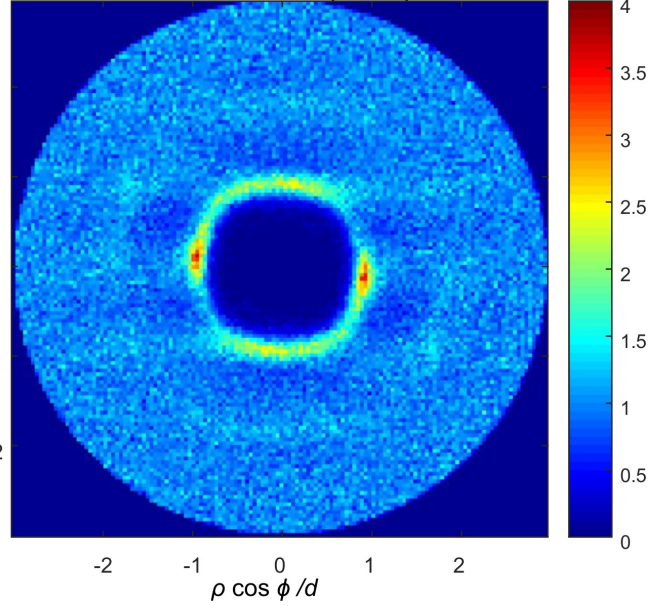
# Dependence on shear rate



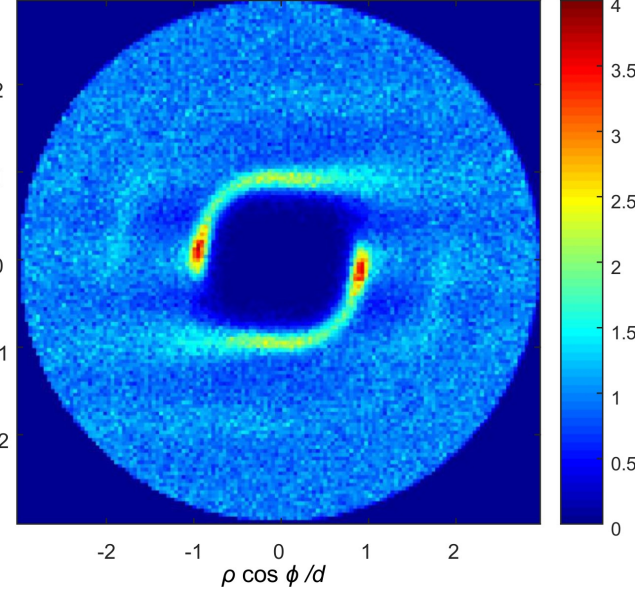
$\tau_Y$   
Low shear rate

$K\dot{\gamma}^n$   
High shear rate

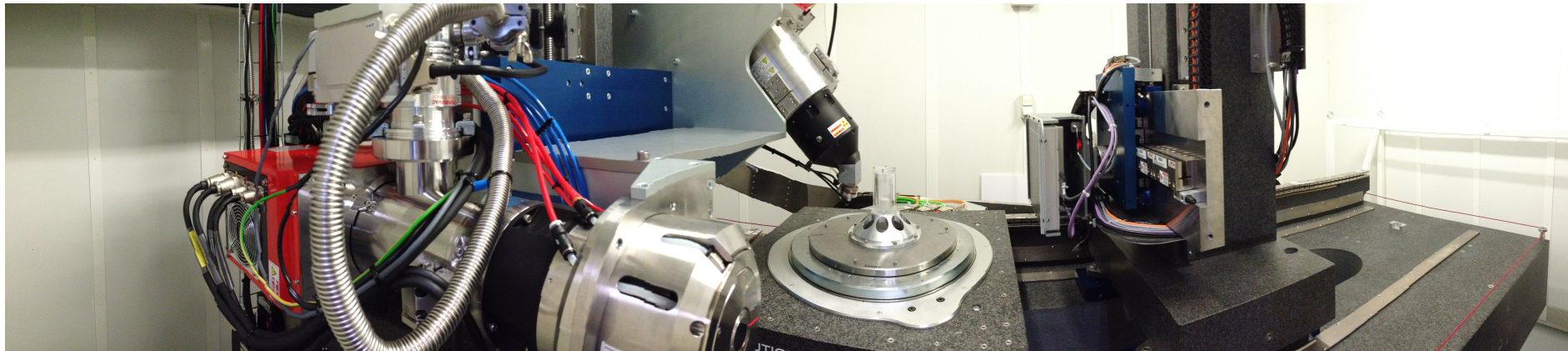
After low shear ( $10^{-2}s^{-1}$ )



After high shear ( $10s^{-1}$ )



# Development of an in situ rheometer



## Multi-configurations : Couette & parallel plates

*David Hautemayou (Navier)*

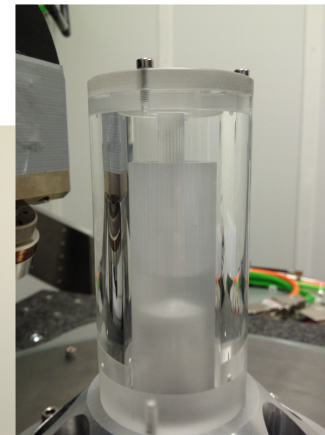
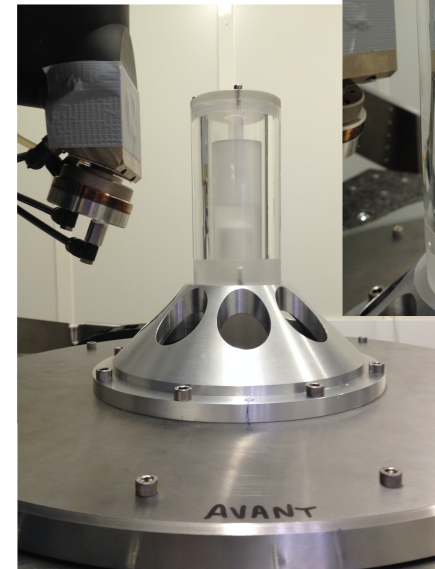
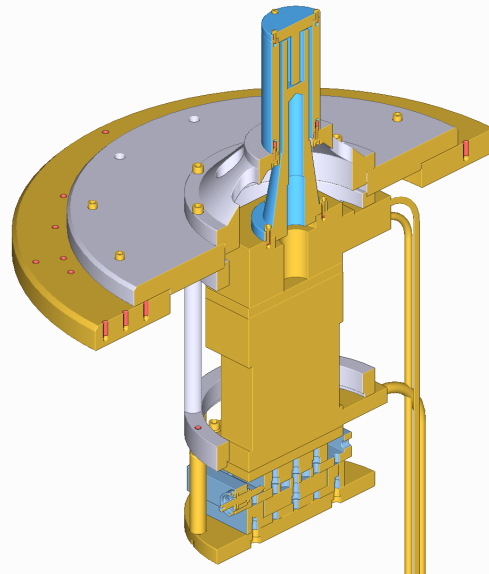
*Couette* : ext dia. 20mm, int. dia. 10mm, height 30mm

*Parallel* : dia. 20mm, height 2mm

Load capacity : 14N

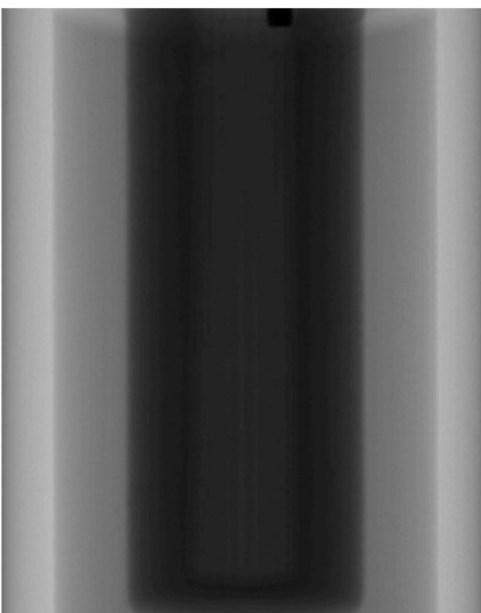
Displacement : range 30mm, rate 10 mm/s, resolution 50 $\mu$ m

Rotation: max rate 720 $^{\circ}$ /s , max torque 0.42Nm at 0 $^{\circ}$ /s, load 100N, repeatability 0.2  $\mu$ m, concentricity  $\pm$  1.5 $\mu$ m

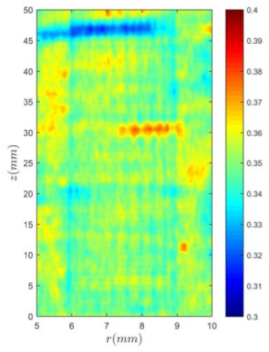




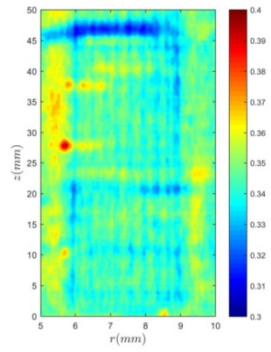
# 2D/3D validation



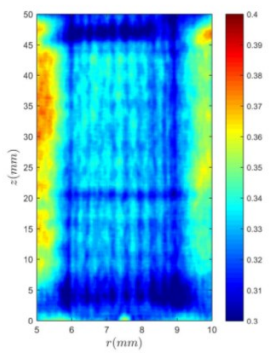
Same material with reference radiographs  
and then, a radiograph every 0.5s  
+ some 3D scans time to time



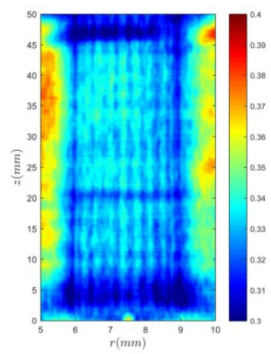
(a) Image number 314



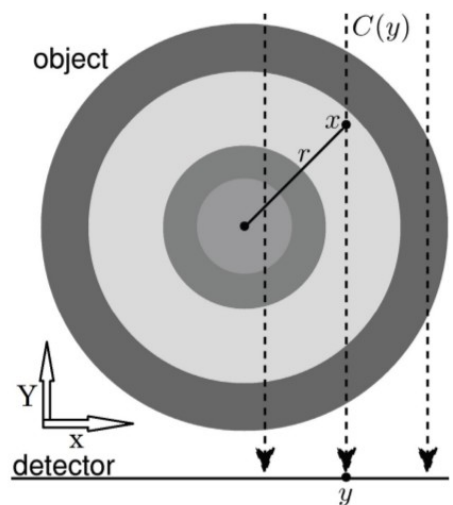
(b) Image number 491



(c) Image number 608



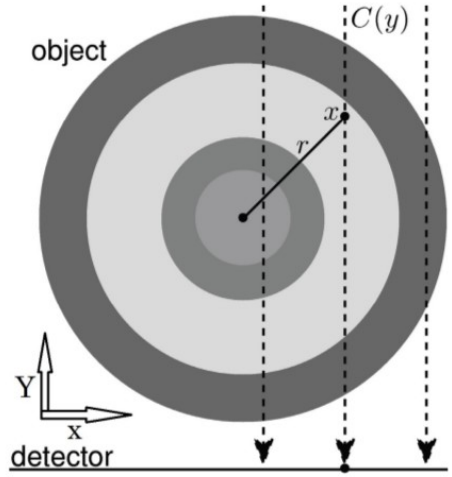
(d) Image number 7199



*Average particle concentration*



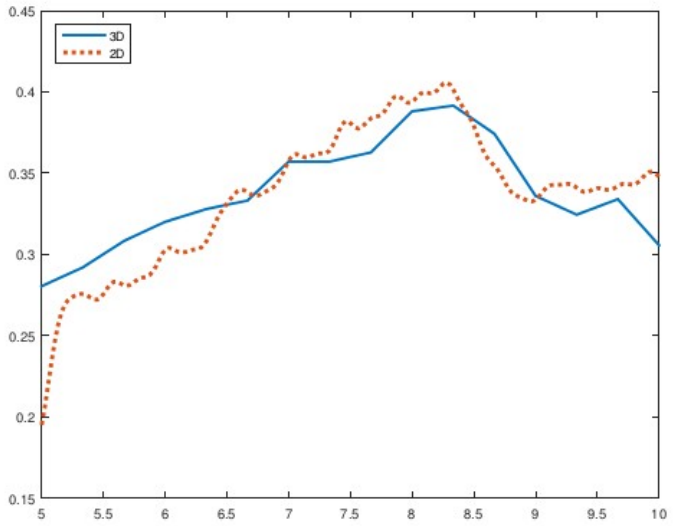
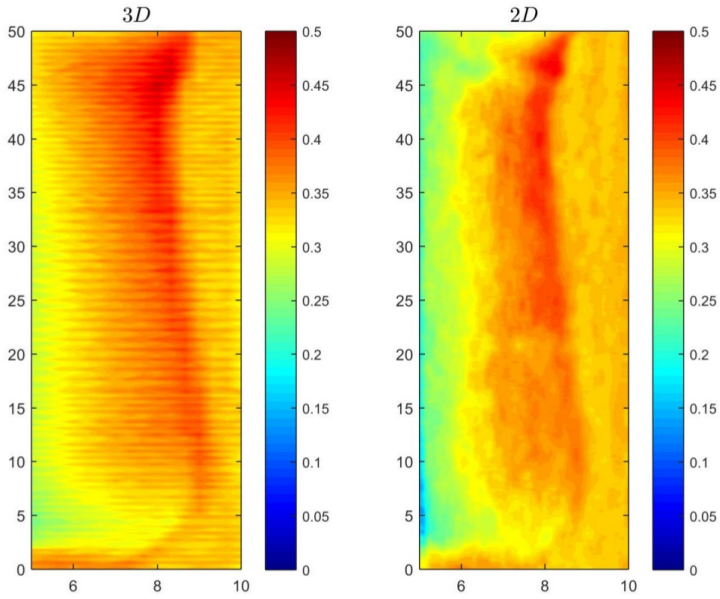
# 2D/3D validation



Work by Mohammad Gholami (Ohio University)

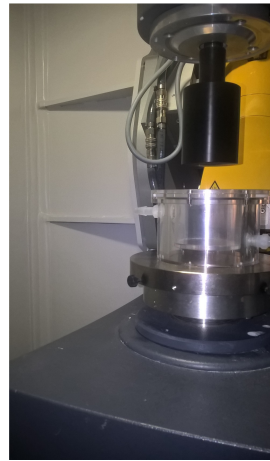
Using Abel transformation, calculating the volume fraction from the 2D radiograph)

Done in Matlab



2D/3D comparison of the volume fraction

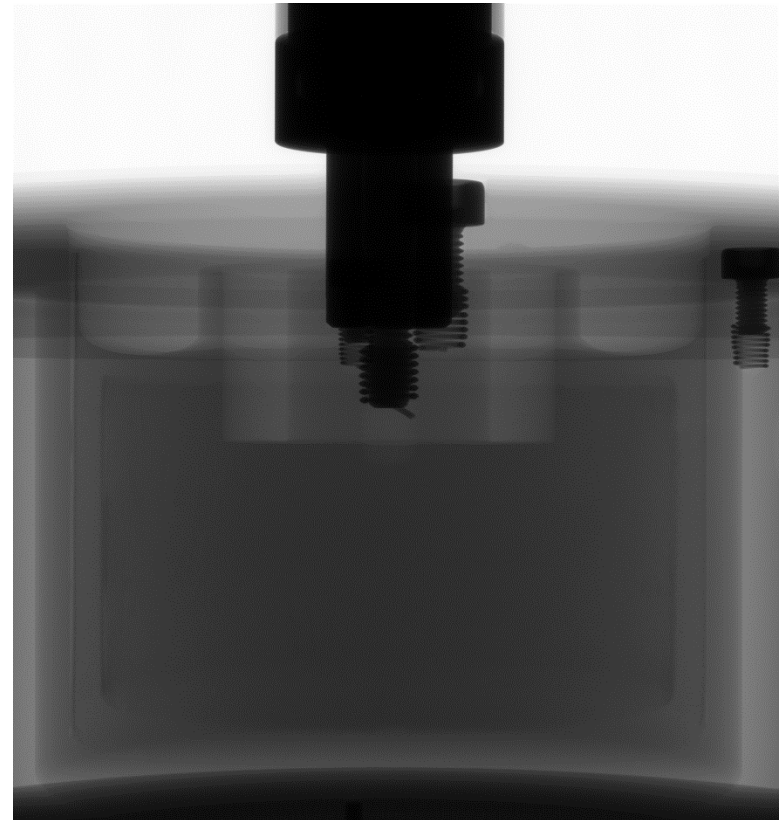
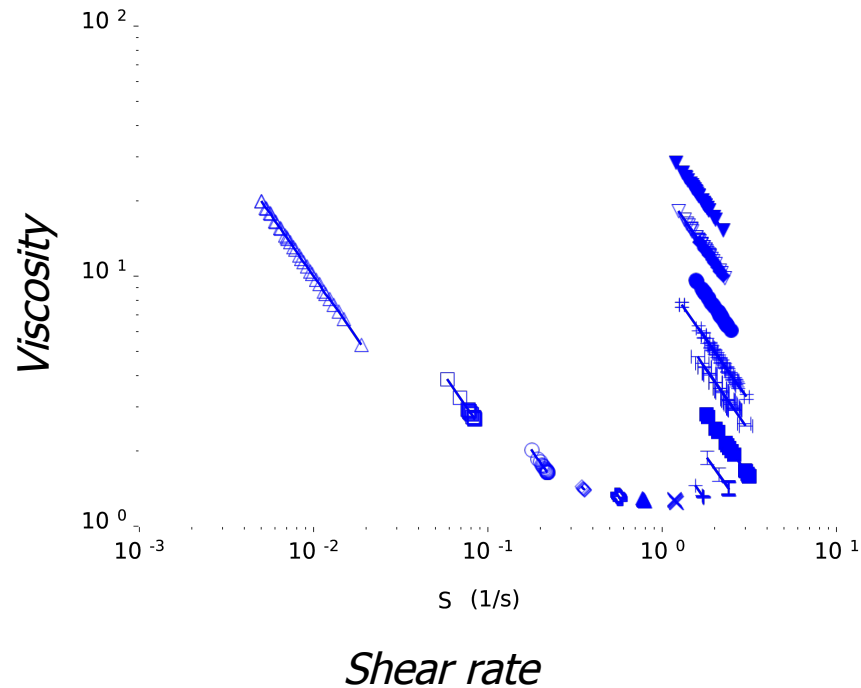
# Time-resolved shear thickening



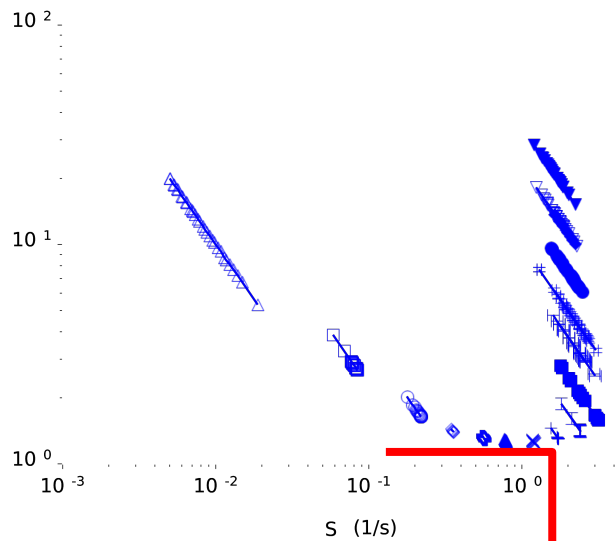
41% of cornstarch in water/CsCl/glycerol,  
small gap Couette, stress controlled

**2D X-ray imaging**

**2D volume fraction profiles**

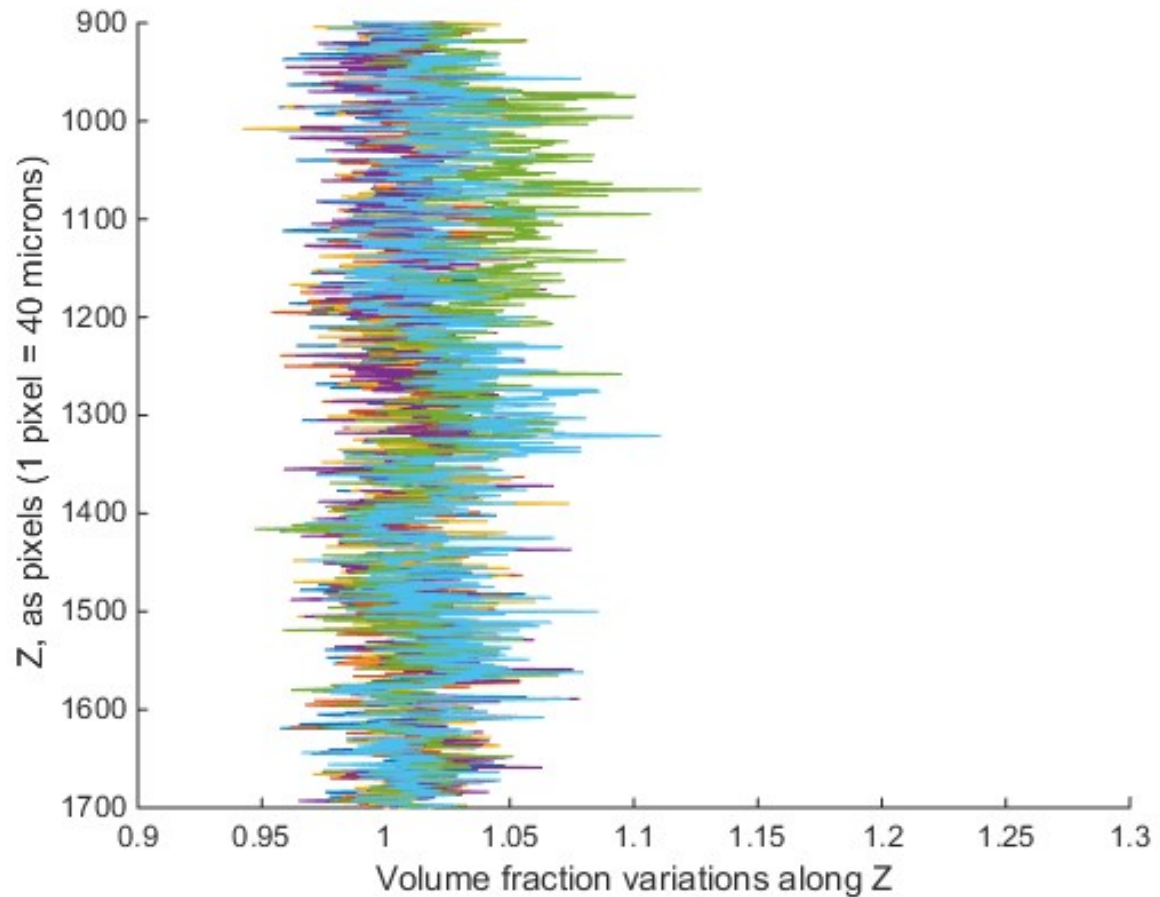


# Time-resolved shear thickening

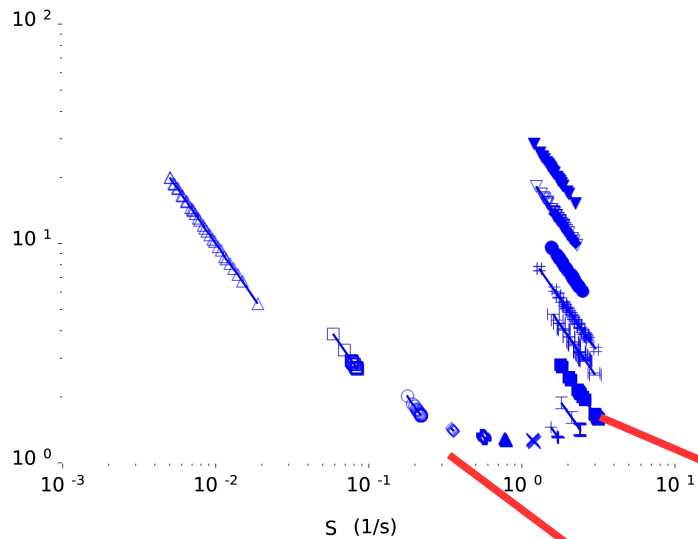


**Before DST:  
homogeneous**

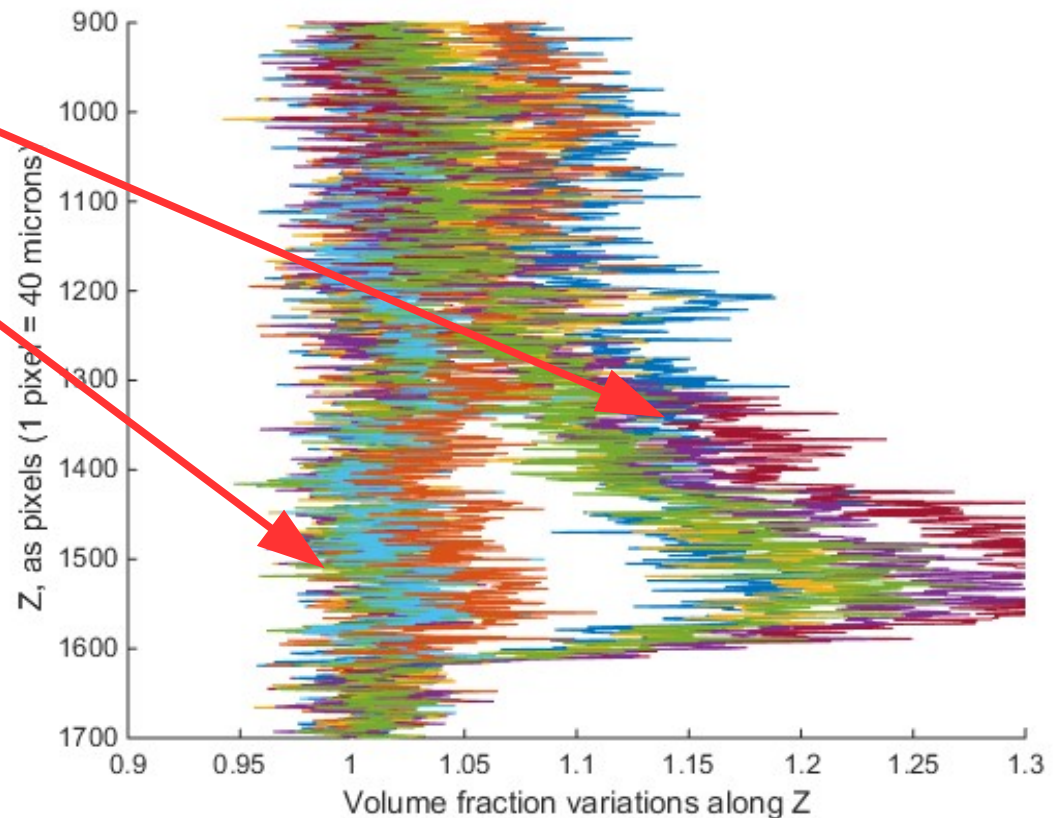
*Vertical volume fraction  
profile, near the rotor*



# Time-resolved shear thickening



*Vertical volume fraction profile, near the rotor*



**Formation of a jammed (close to RCP) granular material block near the rotor, at the bottom balanced by decrease near stator**

**How can anything be interpreted on the intrinsic behavior???**



# Conclusions

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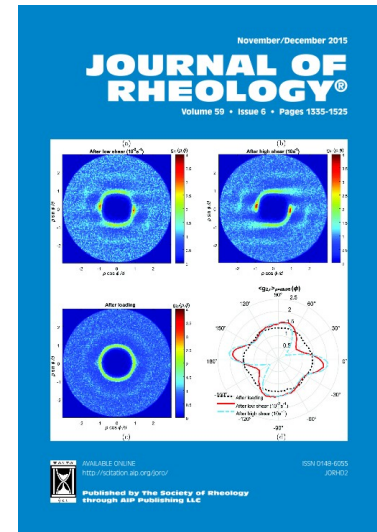
- Development of new experimental methods for yield stress fluids with non-colloidal particles
- 2D validated by 3D → in some cases, no need of a special in situ rheometer
- Not so simple behaviour
- New experimental perspectives for many different materials (both models and real materials) → if you've got **a good candidate you're interested on**, send it !!

**Ovarlez et al. (2015), *Flows of suspensions of particles in yield stress fluids*, Journal of Rheology.**

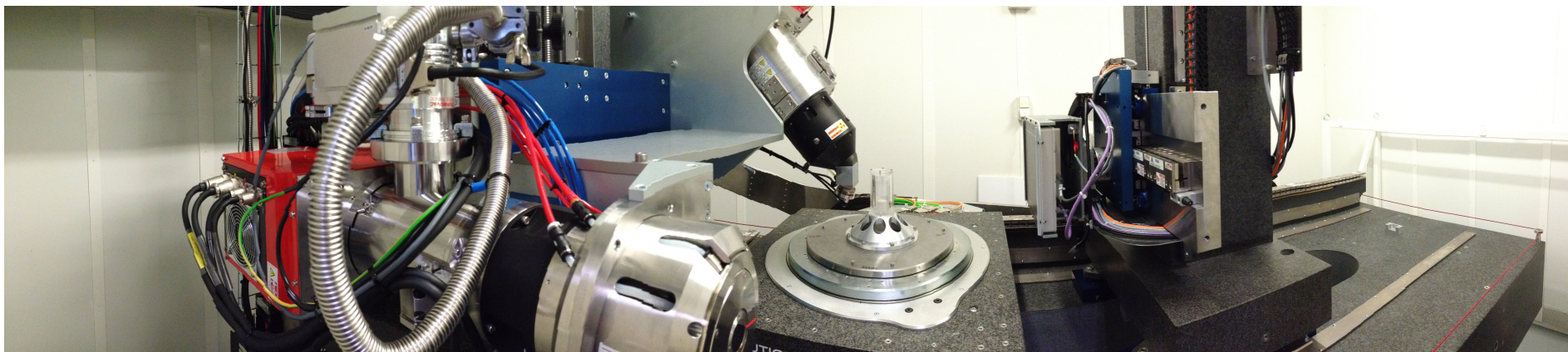


Programme ANR : Sciences de l'information, de la matière et de l'ingénierie : Sciences de l'ingénierie, matériaux, procédés, énergie (JCJC SIMI 9) 2010 : projet SUSPASEUIL

Référence projet : ANR-10-JCJC-0905



**THANKS FOR YOUR ATTENTION!!**



*Five Years of Tomography at Laboratoire Navier, the 8<sup>th</sup> of July 2016*