# Science Symposium

## Advanced Colloids Experiment (Temperature controlled) – ACE-T10

PIs: Prof. Roberto Piazza & Dr. Stefano Buzzaccaro – Politecnico di Milano - ESA

Presented by:



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February 12 - 14, 2019

# AGING DYNAMICS AND STRESS – RELAXATION IN COLLOIDAL GELS STUDIED IN MICROGRAVITY

POLITECNICO
MILANO 1863


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Stefano Buzzaccaro – Politecnico di Milano

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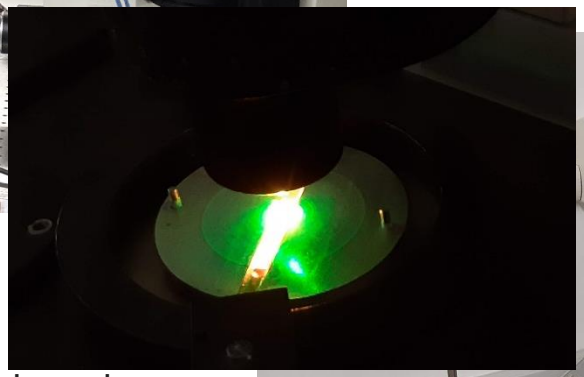




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# ISS Increments 59 and 60 Science Symposium

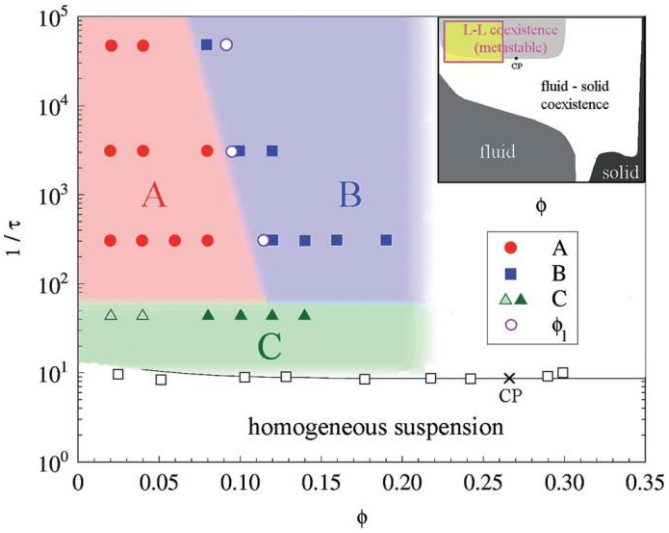
Advanced Colloids Experiment (Temperature controlled) – ACE-T10:

Roberto Piazza – Politecnico di Milano (POLIMI)
Stefano Buzzaccaro – Politecnico di Milano (POLIMI)

- Science Background and Hypothesis
- Investigation goals and objectives
- Measurement approach
- Importance and reason for ISS
- Expected results and how they will advance the field
- Earth benefits/spin-off applications

# Science Background and Scientific questions – 1/2

When the attractive forces induced by the presence of a high molecular weight additive acting as a depletant become strong enough, a colloidal suspension undergoes a liquid–liquid (L–L) phase separation that, if the depletion forces are sufficiently short-ranged compared to the size of the colloidal particles, is metastable with respect to crystallization. As for simple liquid mixtures, a colloid suddenly brought within the L–L miscibility gap undergoes a spinodal decomposition process, consisting of phase separation followed by a progressive coarsening of the two phases. However, spinodal decomposition usually gets arrested by the formation of a colloidal network, that is, of a disordered colloidal solid with a gel-like structure. On earth distinct regions can be clearly set apart within the liquid–liquid coexistence region of the phase diagram where gel formation is observed. When forces are barely sufficient to drive the system within the metastable region, an initial disordered gel hosts the rapid nucleation of crystallites, which stress the gel structure until it fully collapses., leading to the formation of a macroscopic colloidal crystal. For stronger attractive forces, two distinct scenarios are observed, depending on the particle volume fraction of the original suspension. At low volume fractions, the gel breaks after a short delay time into separate clusters, which rapidly settle until they compact in a denser disordered phase. For larger values of the particle concentrations, gel breaking is conversely suppressed, the structure undergoes a continuous compression that resemble a syneresis process, and the microscopic dynamics is characterized by logarithmic correlation functions resembling those found for attractive glasses.




**Scientific questions**

- Does the depletion gel scenario drastically change in the absence of gravity?
- Do internal stress-relaxation mechanisms (syneresis) show up in μ-gravity too?

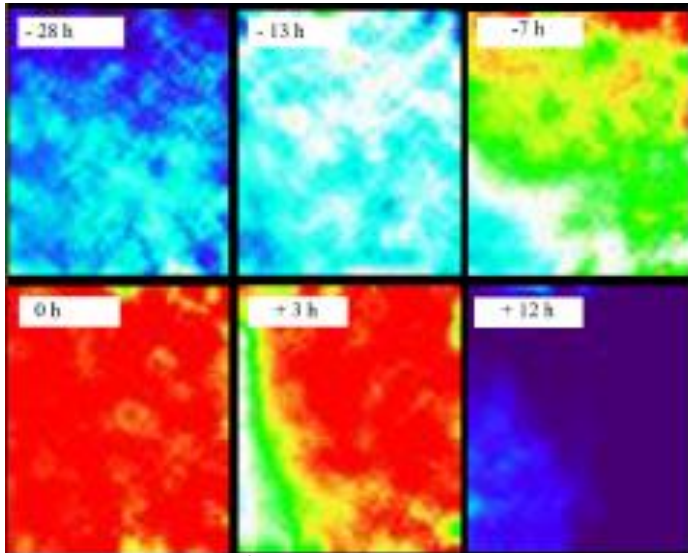
Need for μg

- Gravity stress can mask the internal restructuring and ageing effects in tenuous gels
- On earth, syneresis can hardly be set apart from gravity compression. Getting rid of this problems requires μ-gravity
- In μ-gravity, crystallites grow without breaking the matrix, which allows us studying crystallization in a gel

# Science Background and Scientific questions – 2/2

During the fast gelation process localized stresses are brought into the gel. Besides affecting the rheological properties of the materials such dynamics rules the aging of the thermal systems at rest. We investigated the aging behavior of colloidal gels, characterized by tunable mechanical properties, using photon correlation imaging, an optical technique that allows obtaining the microscopic dynamics of the sample, while retaining at the same time the spatial resolution of imaging techniques. We are able to visualize sudden localized plastic restructuring events of finite spatial extensions. Their complex spatio–temporal correlations increase with the age of gel, eventually causing system-spanning "quakes", heralded by a speeding up of the microscopic dynamics, which originate from well-defined spots and spread at a large but finite speed through the gel. The waiting times between these macro–quakes is not random but scale in a well-defined manner with the age of the sample. The presence of two different type of quakes, suggests a strong connection with stress relaxation in earthquake sequences, where acceleration of seismic moment leading to large events and seismic cycles are observed.

More in general, relaxation of localized stresses brought in an amorphous solid by a rapid solidification process is ubiquitous in nature, occurring from earthquakes, to damage and failure of materials. We are confident that the comprehension of the stress relaxation mechanism in our colloidal model systems can help to rationalize and possibly to predict the damage and failure of amorphous materials.



Macro- quake propagation

**Scientific questions**

- How the relaxation of internal stress couple with the gravitational stress?
- On earth the macroscopic stress relaxation events originate from the top of the sample. Is it the same also in microgravity conditions?

Need for μg

- Gravity stress can mask the internal restructuring and ageing effects in tenuous gels

# EARTH SCIENCE:

## A Photon Correlation Imaging Investigation

# An (old) interesting problem…

## Last decades

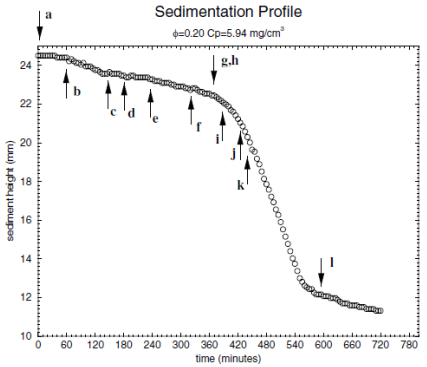
Buscall and White (1987)
Allain, Cloitre, Wafra (1995)
Senis and Allain (1997)
Poon et al (1999)
Starrs (2002)
Derec, Senis, Talini and Allain (2003)
Manley, Skotheim, Mahadevan and Weitz (2005)
Codre, Liguore and Cipelletti (2007)
Besseling, Weeks, Schofield and Poon (2007)
…

$h(t)$ $\Phi(z,t)$

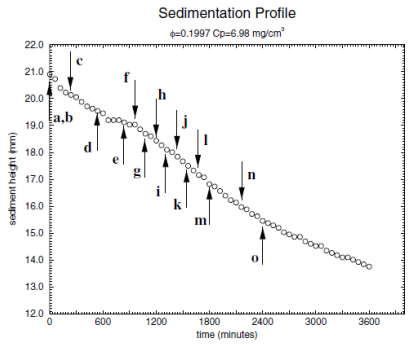
**MACROSCOPIC**

L. Starrs, W. Poon, D. Hibberd and M. Robins, *J. Phys.: Condens. Matter*, 2002, **14**, 2485.

**Delayed sedimentation**



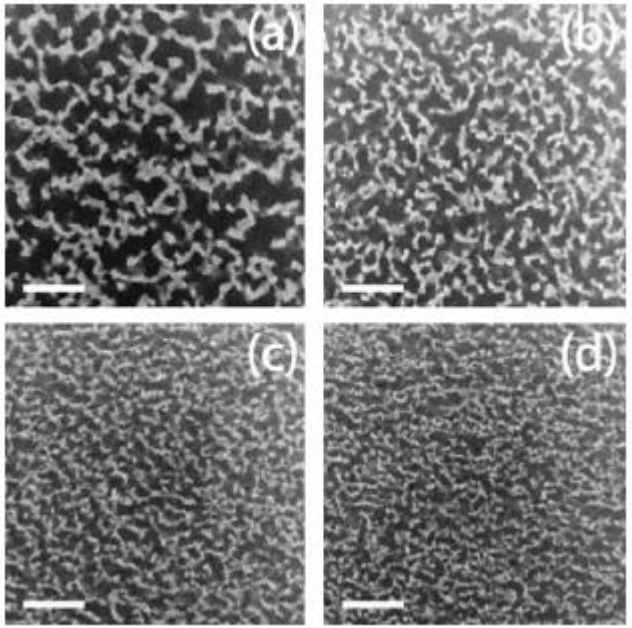

**Creeping sedimentation**



# …still interesting

## Last years

**Teece, Faers  Bartlett, 2010**
**P. Bartlett, L. J. Teece and M. A. Faers , 2012**
**Zhang, Royall, Faers, Bartlett , 2013**
**…**




**S(q,t)**

Micro-rearrangement

**MICROSCOPIC**

L. J. Teece, M. A. Faers and P. Bartlett, *Soft Matter*, 2011, 7, 1341.

# Our contribution

In our experiment (quantitative) on Ground and on ISS (Colis)*

$h(t)$ $v_{sed}(t,z)$ Sedimentation velocity

$\Phi(t,z)$ MACROSCOPIC

$g_2(t,z,\tau)$ ICF

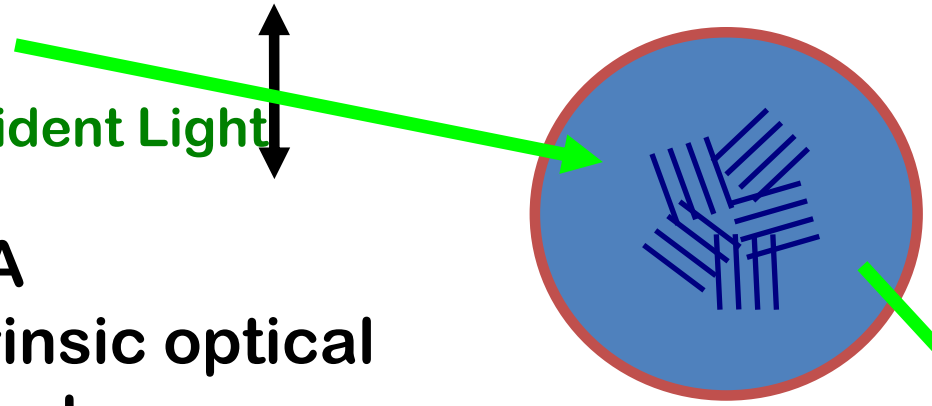
$qR_c \cong 1$

MICROSCOPIC

**QUANTITAVE RELATIONSHIP WITH EQUILIBRIUM PHASE DIAGRAM**

* See later

# How to measure concentration? DepLS




**MFA**
**Intrinsic optical anisotropy**

**POLARIZED COMPONENT**

$I_{VV} \sim N\,P(q)S(q)$

$I_{VH} \sim N\,P(q)$

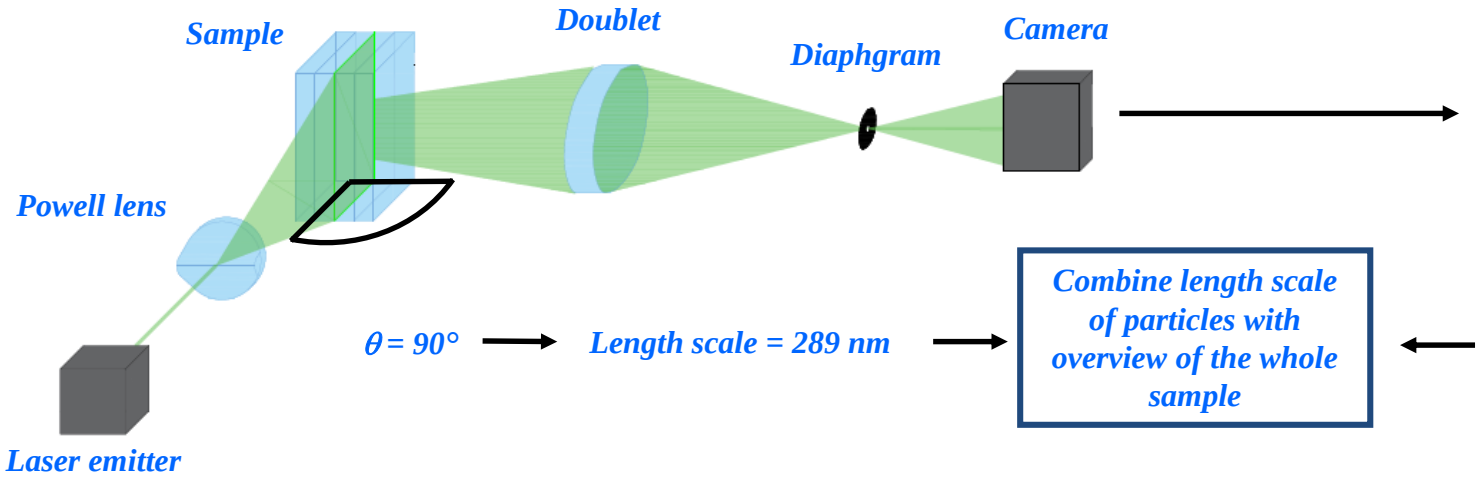
**DEPOLARIZED COMPONENT**

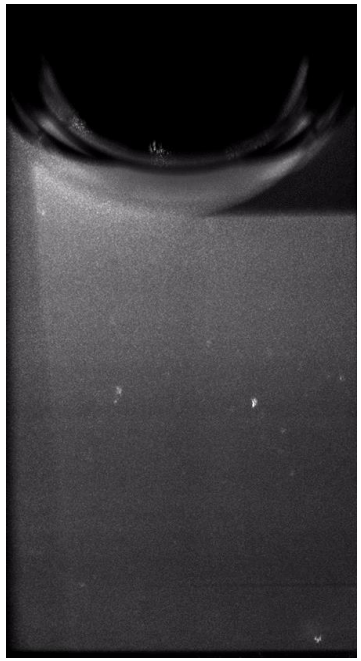
Scattered Light

| STATIC Dep LS | DINAMIC Dep LS | |
|---|---|---|
| $\Phi(z,t)$ | $g_2(t,z,\tau)$ | Rotation<br>Traslation |

# Photon Correlation Imaging

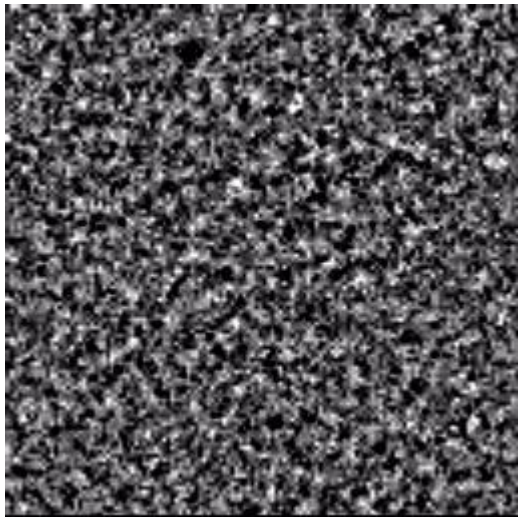
***Localized stress can propagate to far distances: how to study them?***



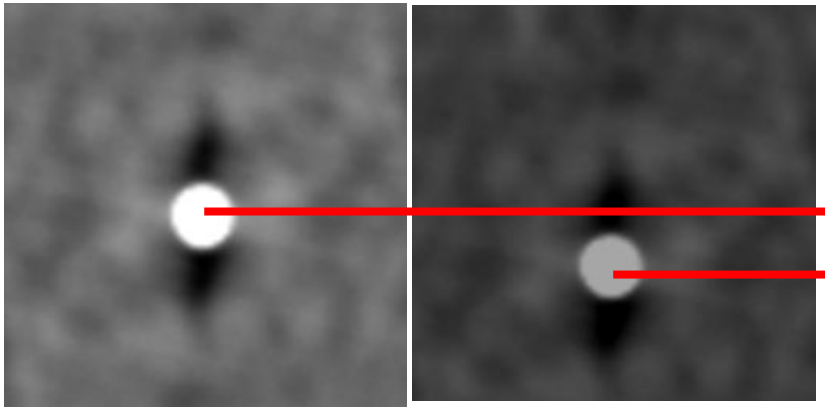



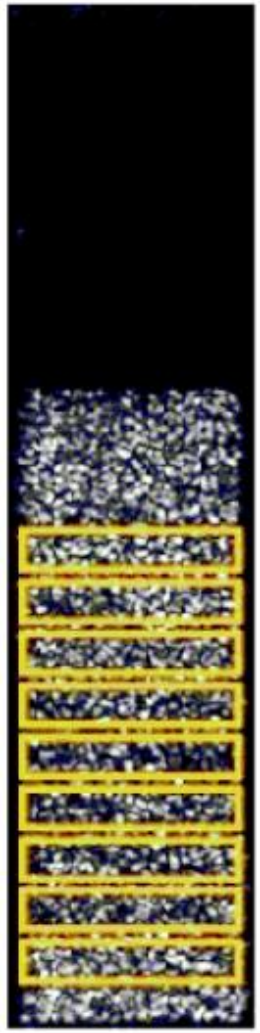
# Multi-speckles correlations

## With sedimentation



$$C_{x,z}(\Delta t, \Delta x, \Delta z) = \langle I(t,x) I(t+\Delta t, x+\Delta x, z+$$





$$C_I(t,\tau) = \frac{\left\langle I_p(t) I_{p+\Delta z}(t+\tau) \right\rangle_p}{\left\langle I_p(t) \right\rangle_p \left\langle I_p(t+\tau) \right\rangle_p}$$

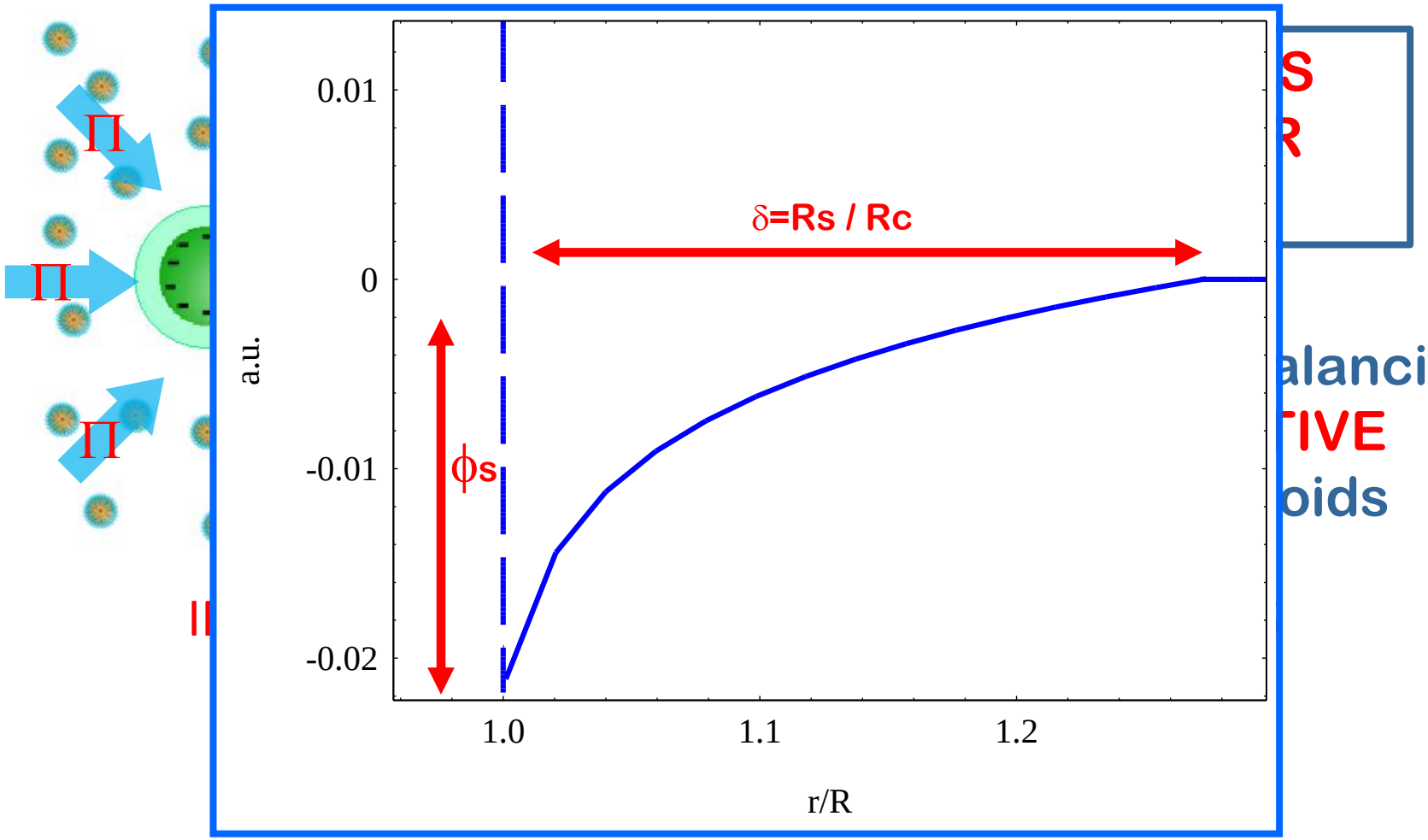
$\Delta t = 0$ $\quad$ $\Delta t \neq 0$

$$g_2(\tau) = \left\langle c_I(t,\tau) \right\rangle_{\Delta t}$$

L. Cipelletti, G. Brambilla, S. Maccarrone and S. Caroff, *Optic. Express*, 2013, **21**, 22353.

# How to tune the interactions?

# Depletion Force

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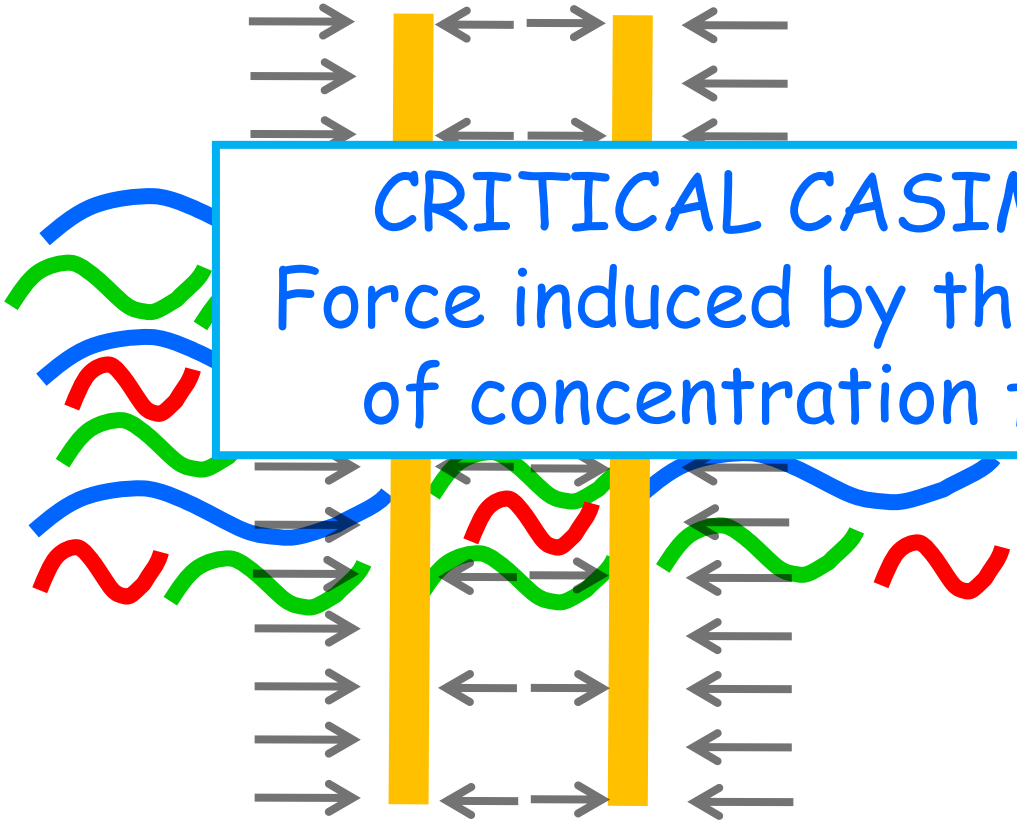
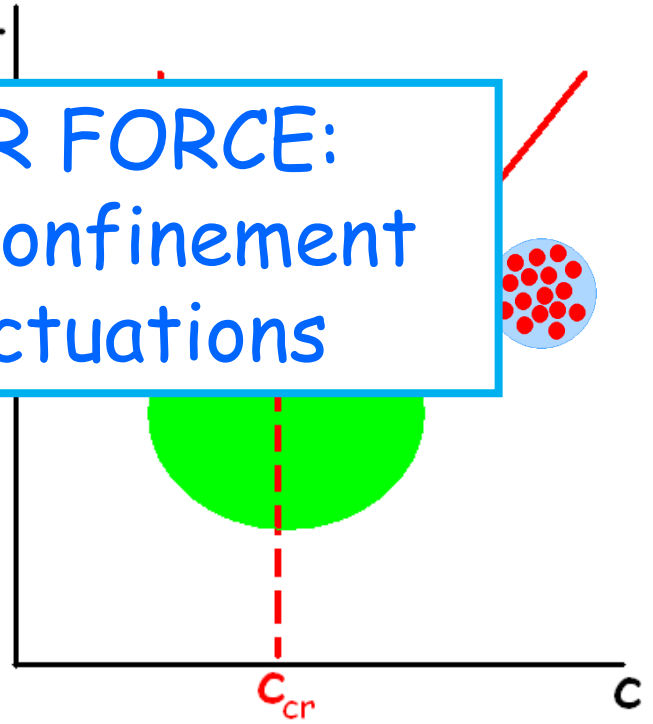
# Critical Casimir Force (CCF)

CLASSICAL CASIMIR FORCE:
Electromagnetic field fluctuactions
(Van der Waals)

CONSOLUTION CURVE
(PHASE TRANSITION)





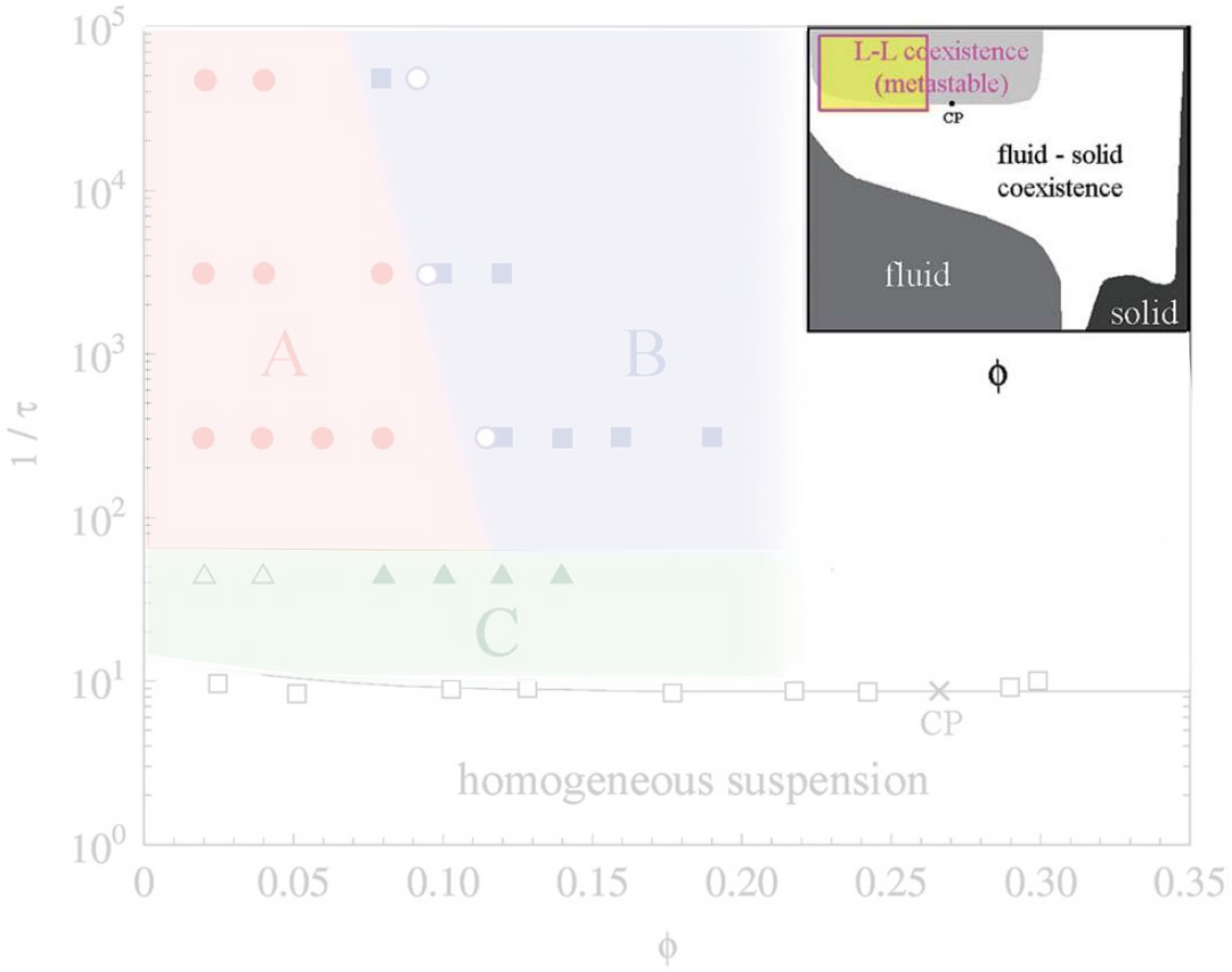

**CRITICAL CASIMIR FORCE:**
**Force induced by the confinement of concentration fluctuations**

CCF can be finely tuned by:

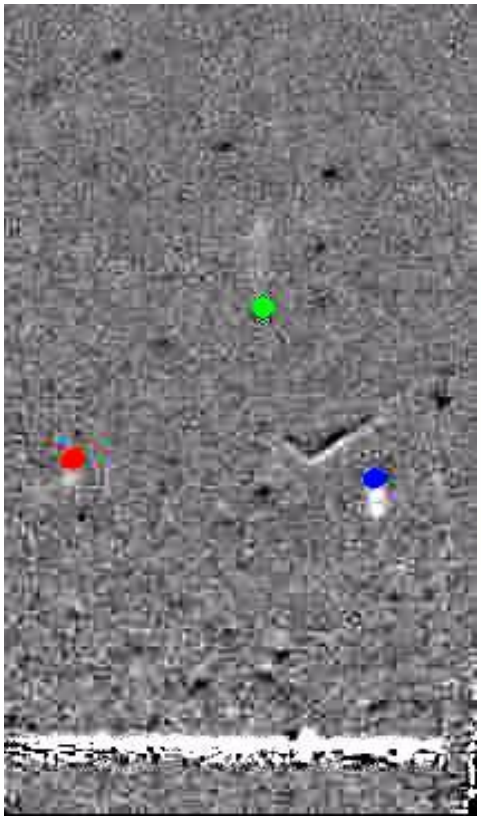
- Type of surfactant;
- Type of Salt Added (at fixed ionic strength 0.5 M of Ammonium Sulphate reduces the T_c more than 30°C);
- Surfactant Volume Fraction (classical depletion)

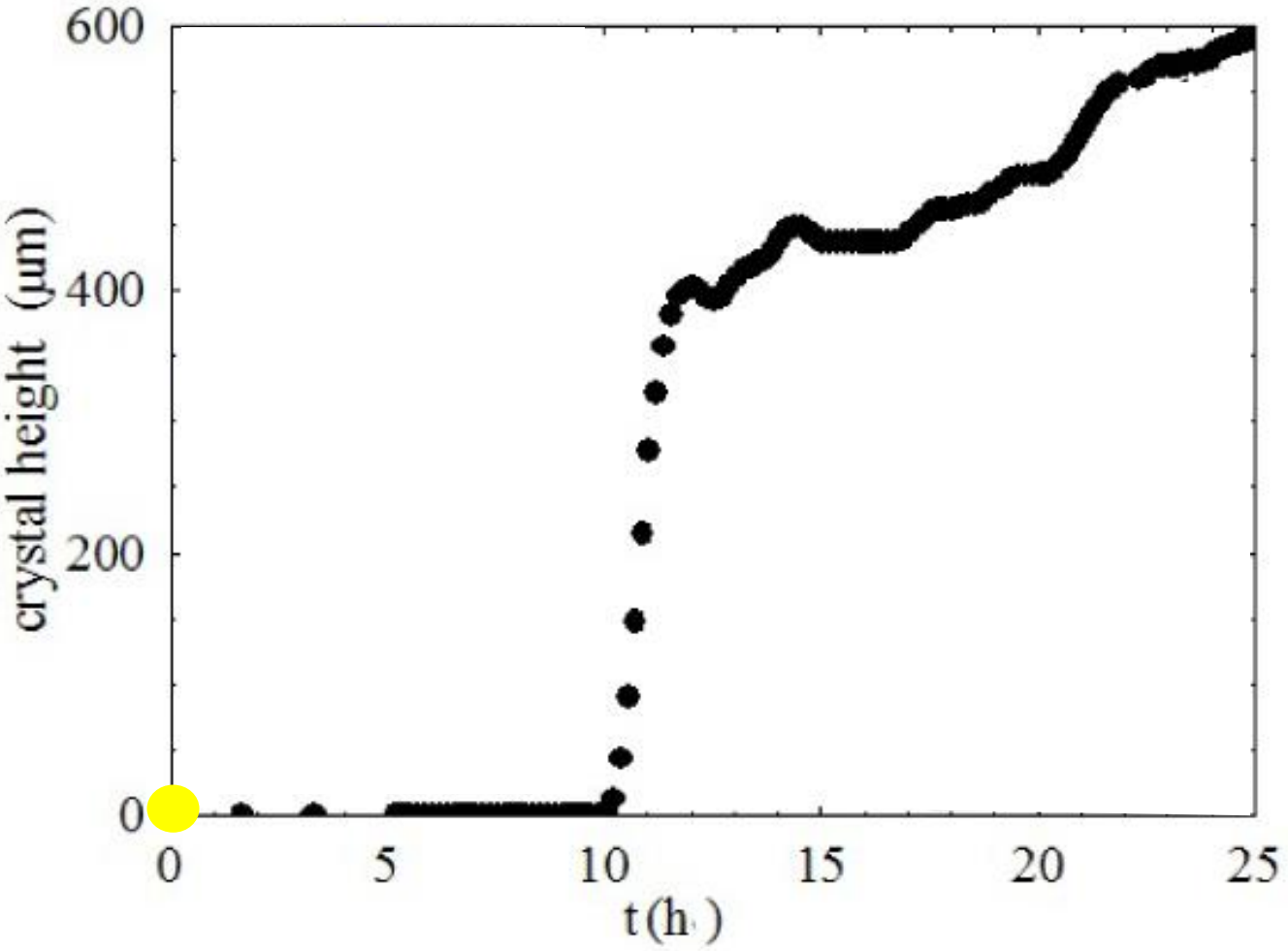
- Temperature

# Phase diagram

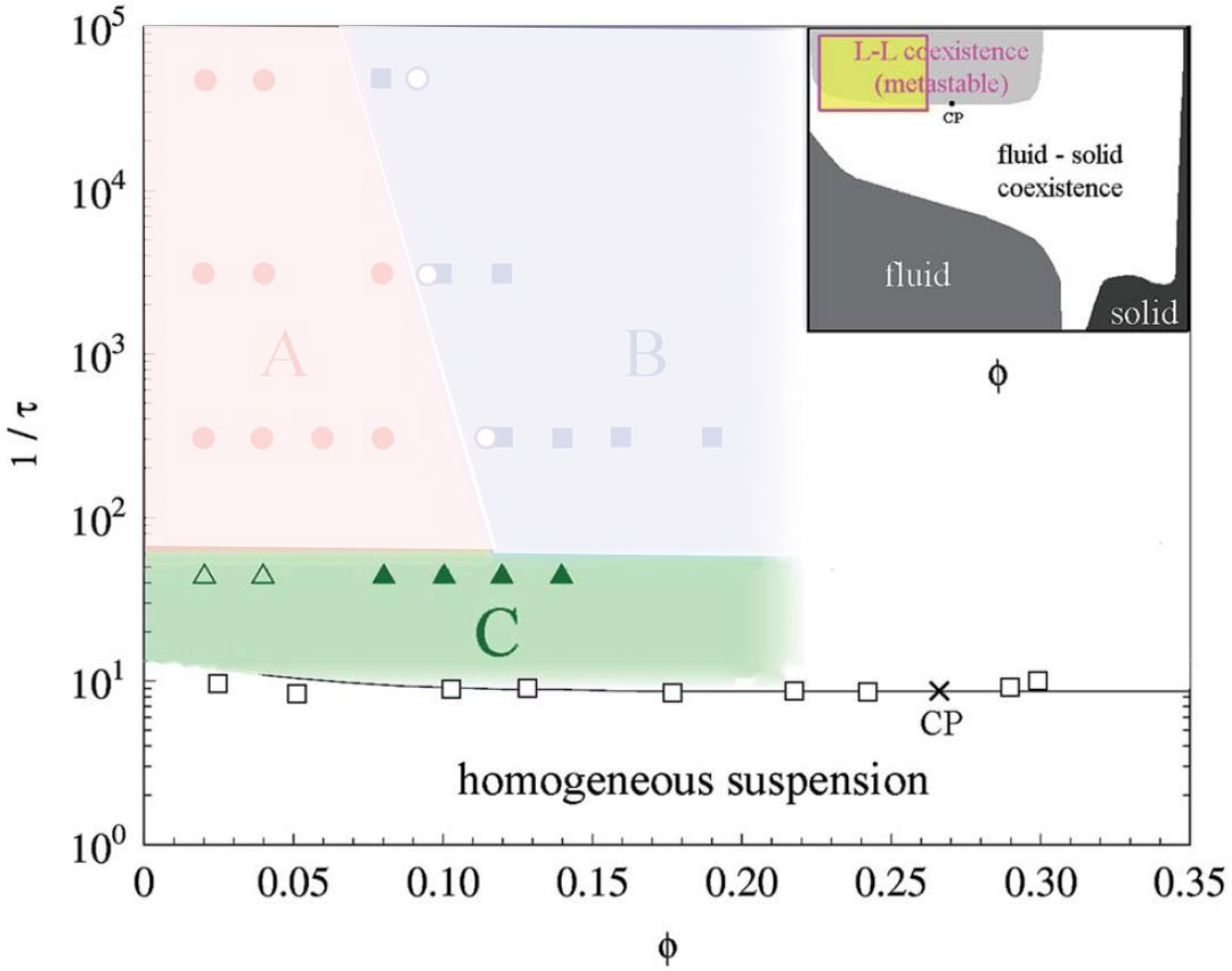


# Crystallizing gels

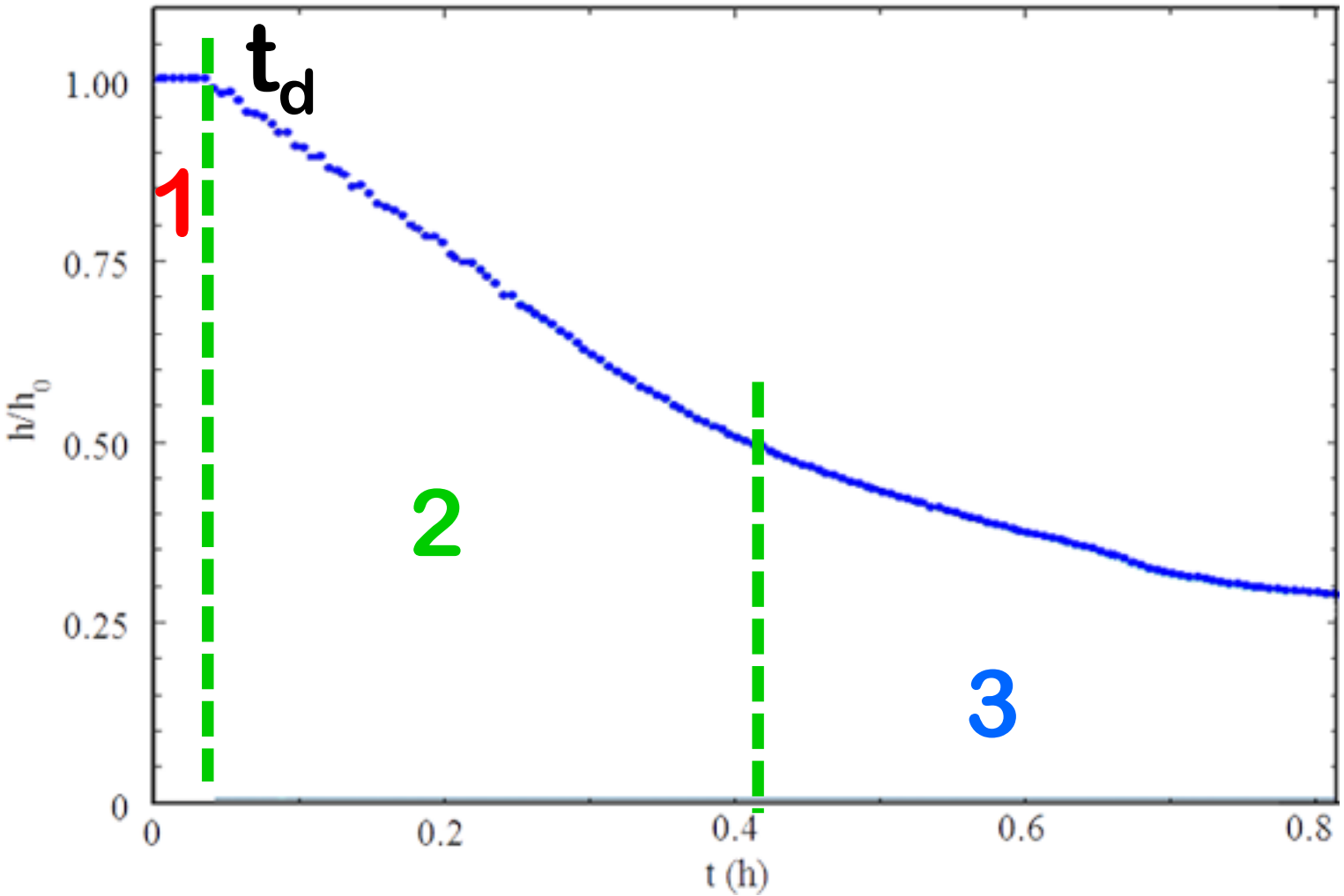


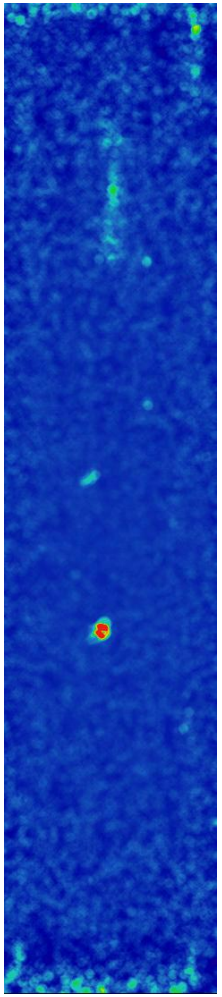


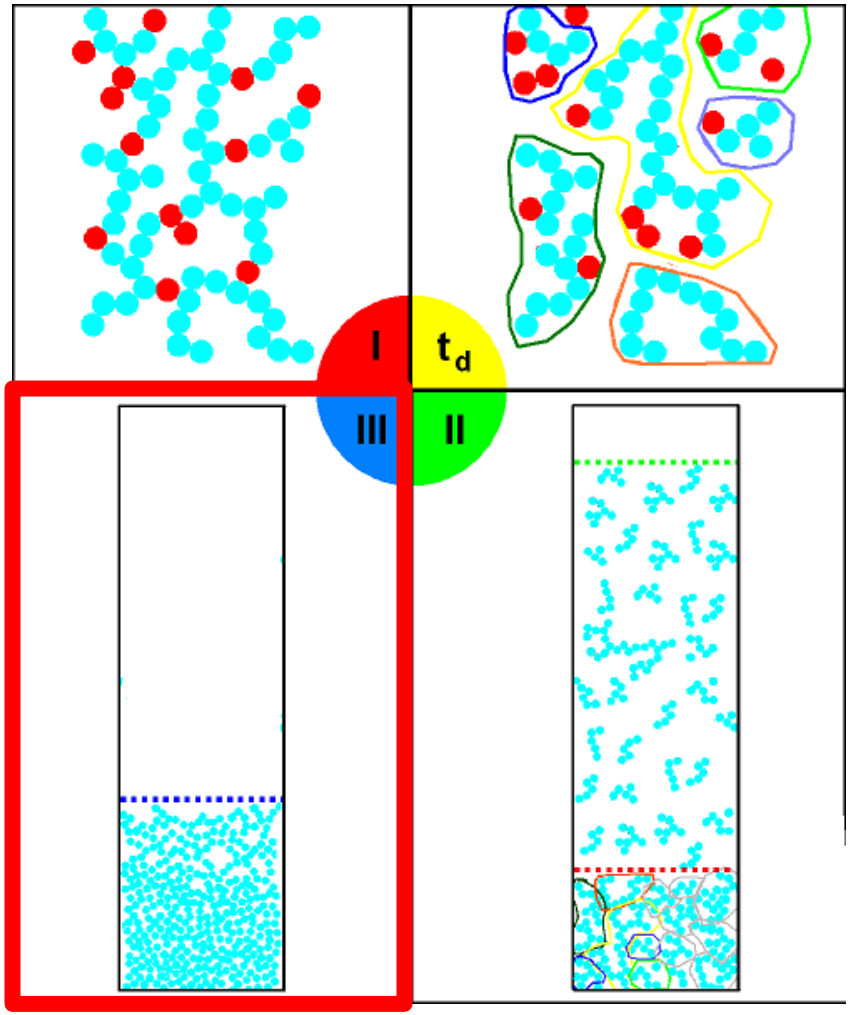
# Phase diagram
# Collapsing gel

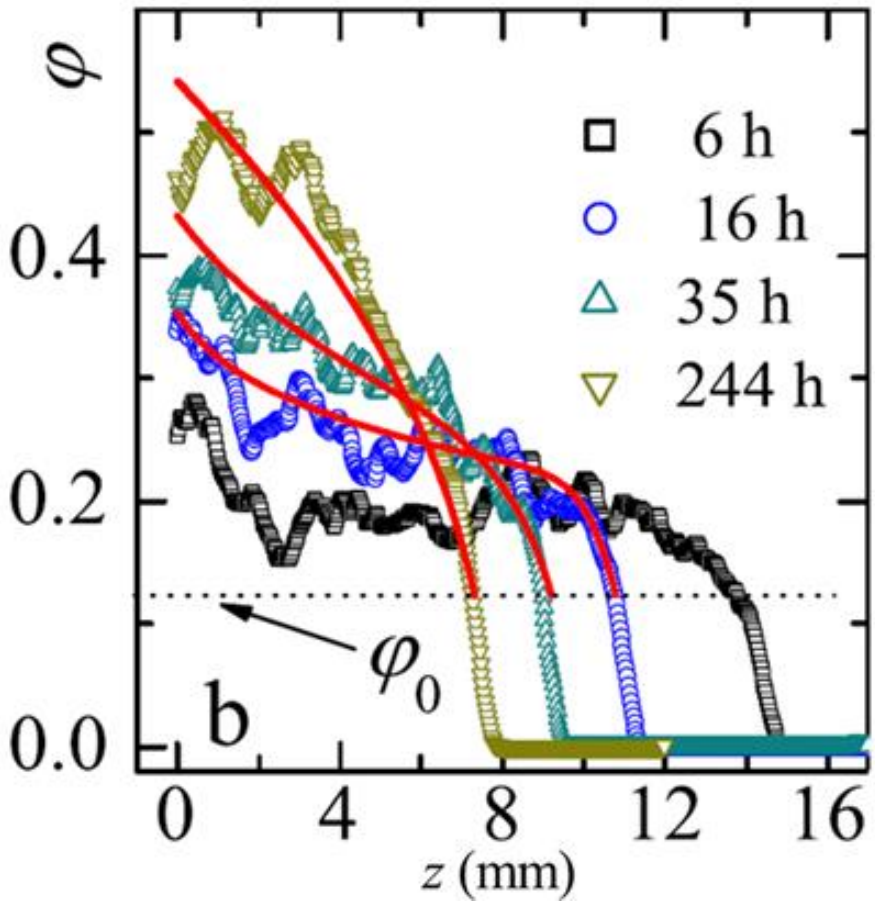


# h(t)

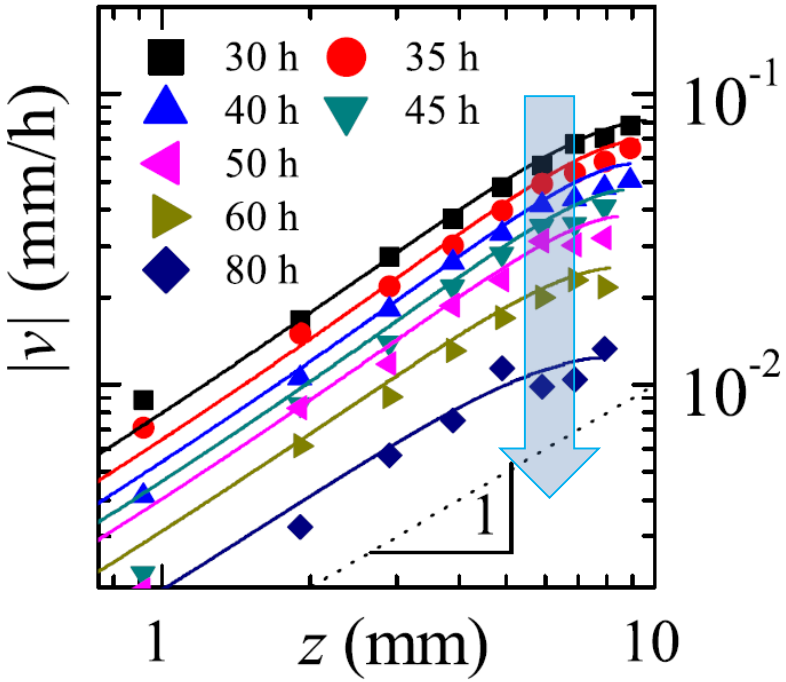


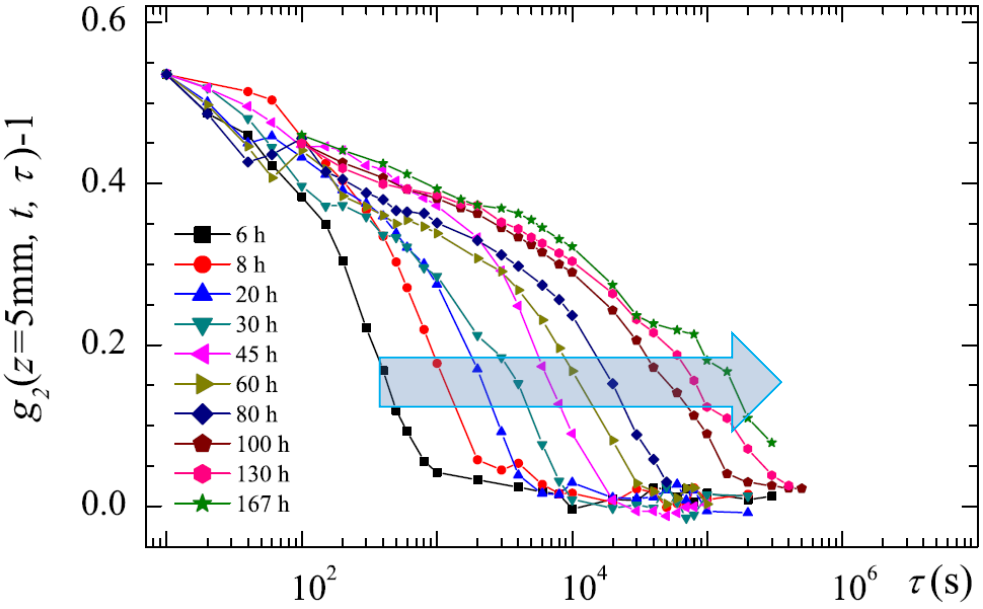





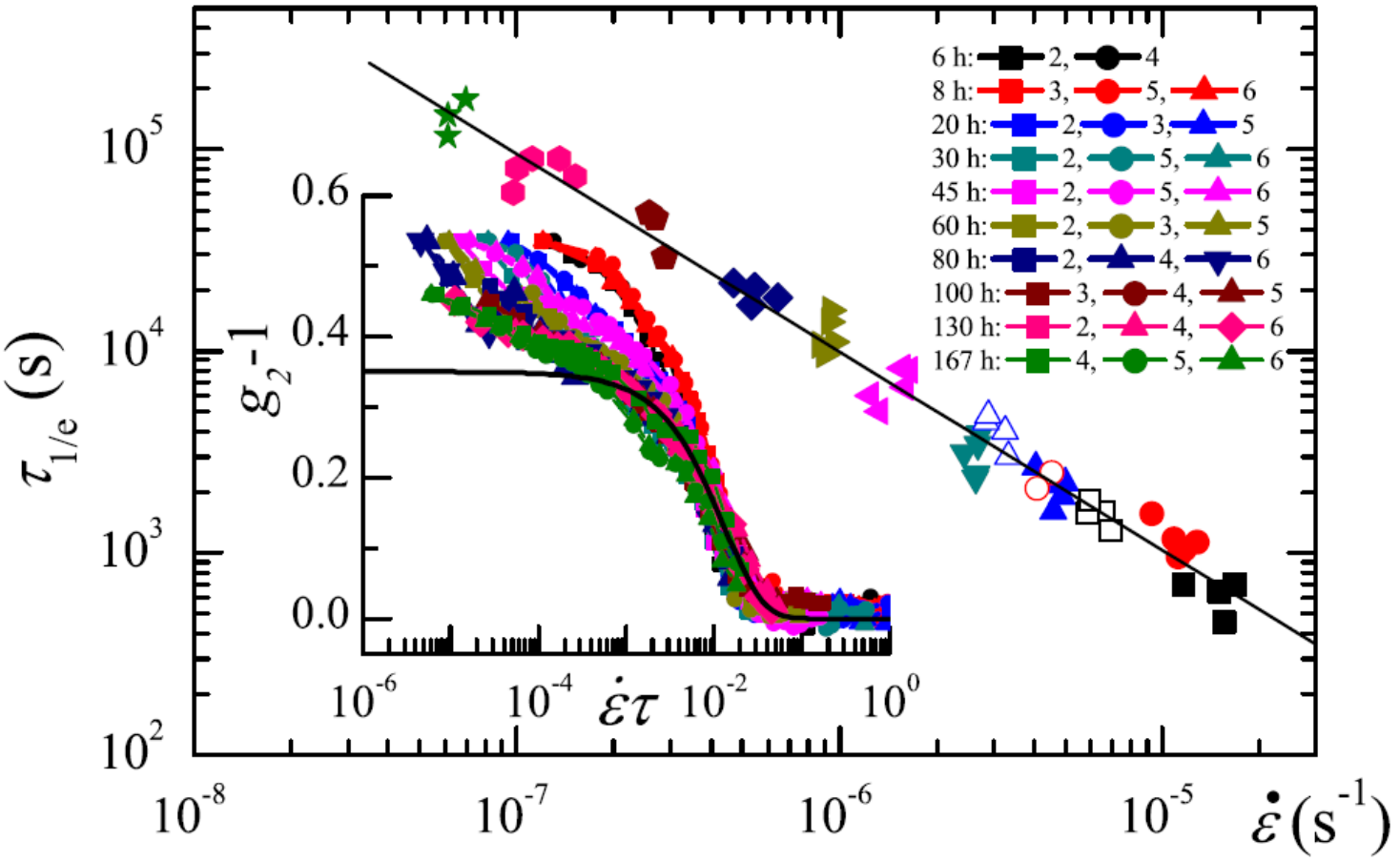

G. Brambilla, S. Buzzaccaro, R. Piazza, L. Berthier and L. Cipelletti, *Phys. Rev. Lett.*, 2011, **106**, 118302.



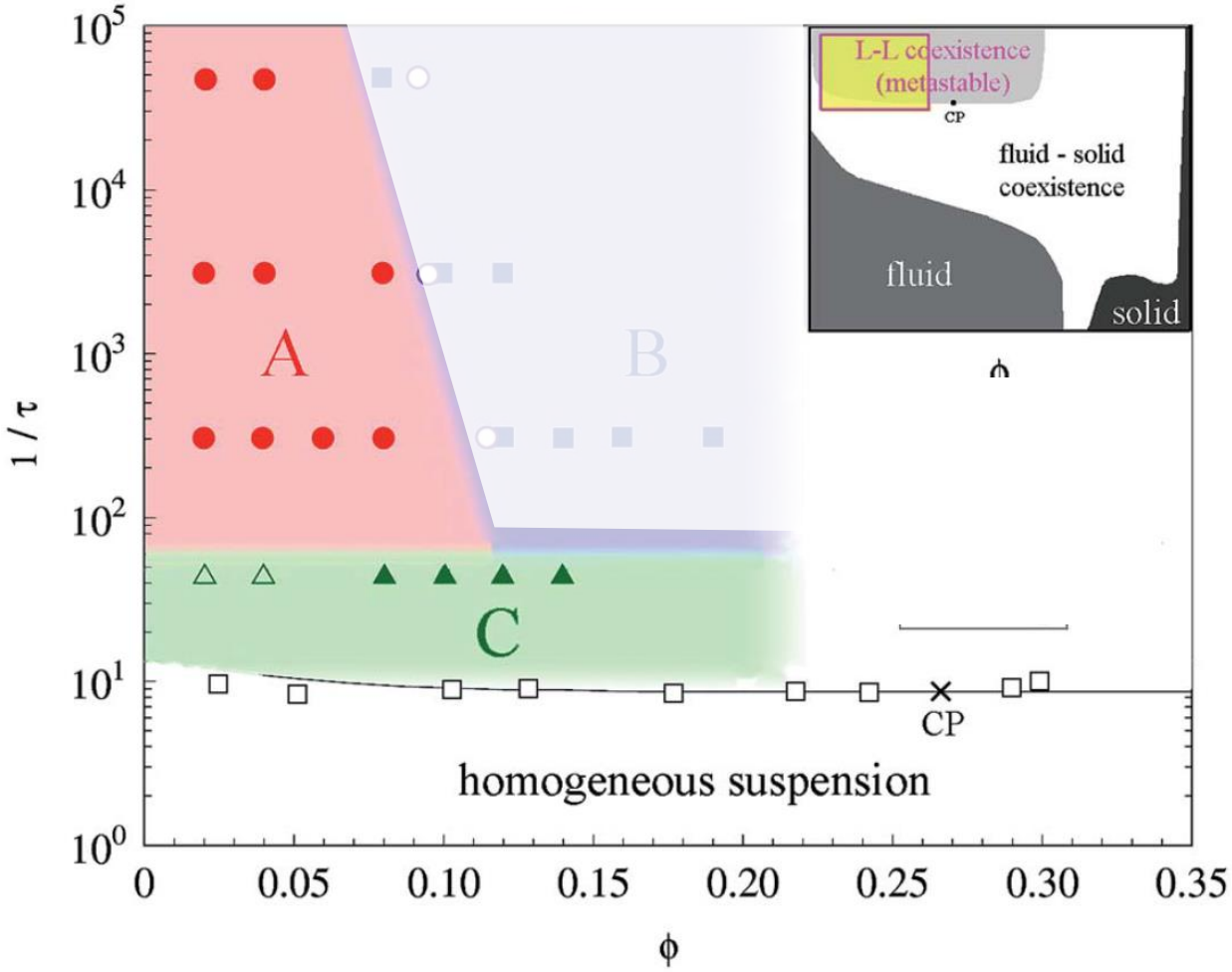

G. Brambilla, S. Buzzaccaro, R. Piazza, L. Berthier and L. Cipelletti, *Phys. Rev. Lett.*, 2011, **106**, 118302.

# $\tau_{1/e}$ vs $\varepsilon(t)$

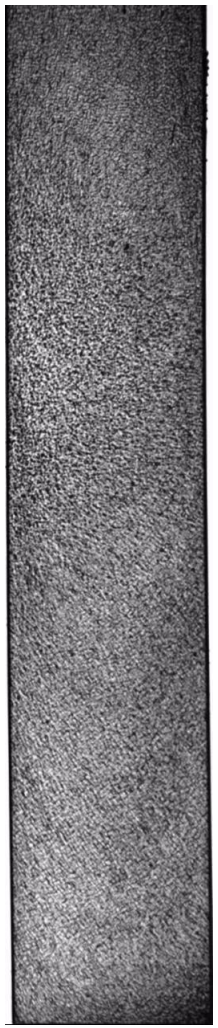


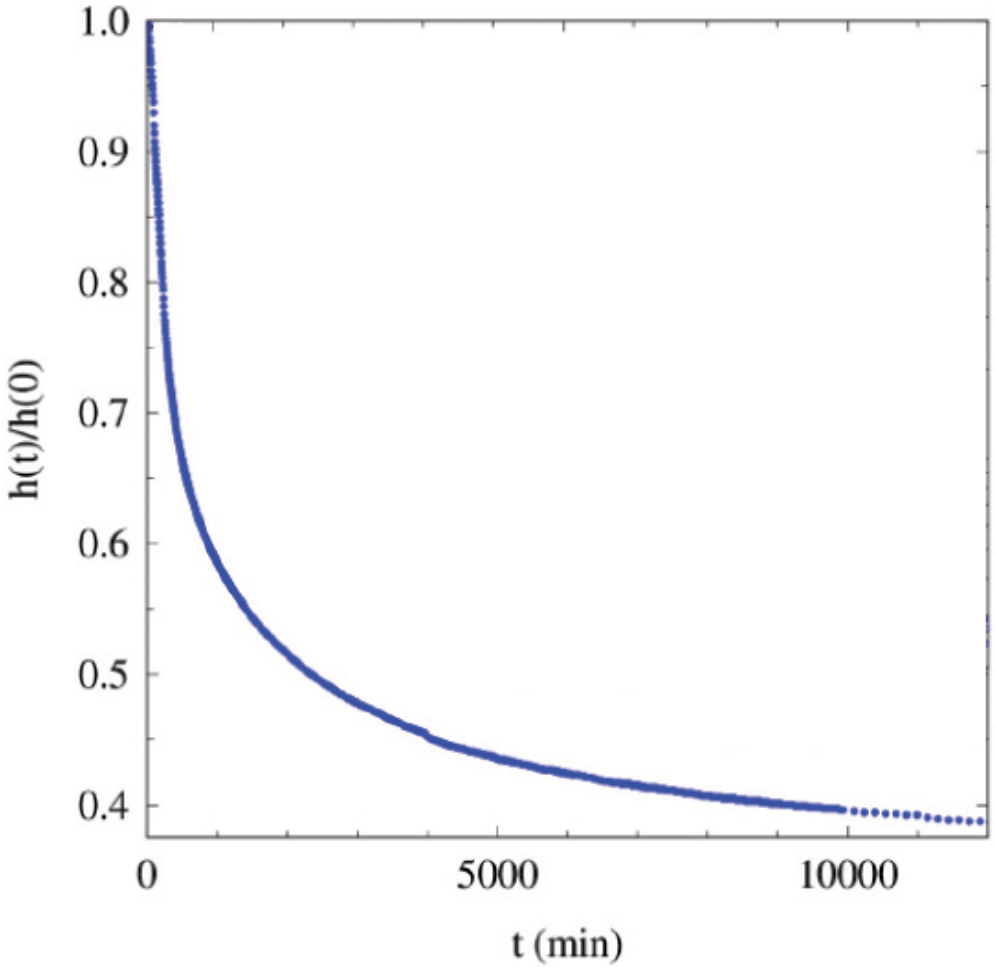

Microscopic structural relaxation driven by macroscopic deformation of the sample

# Phase diagram
# Creeping gel

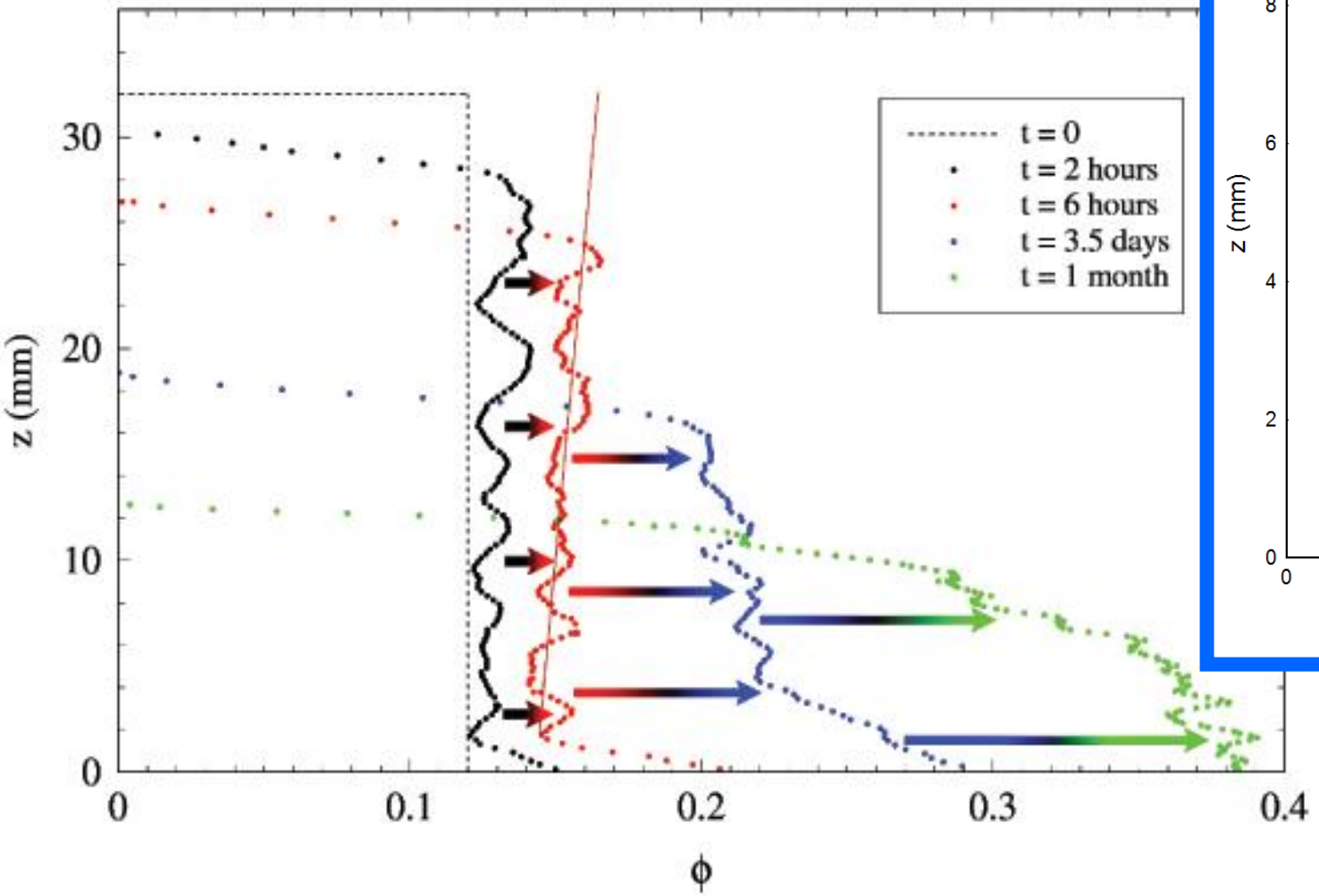


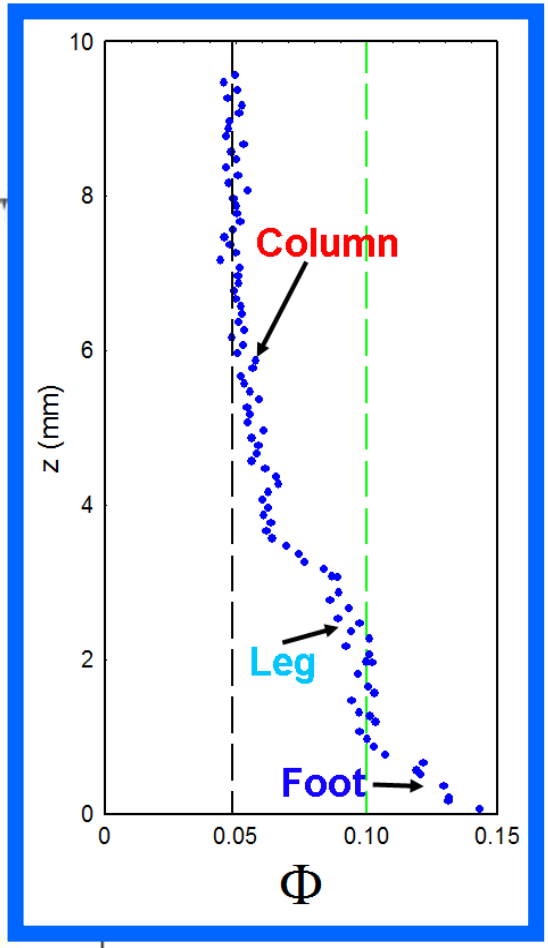
# h(t)



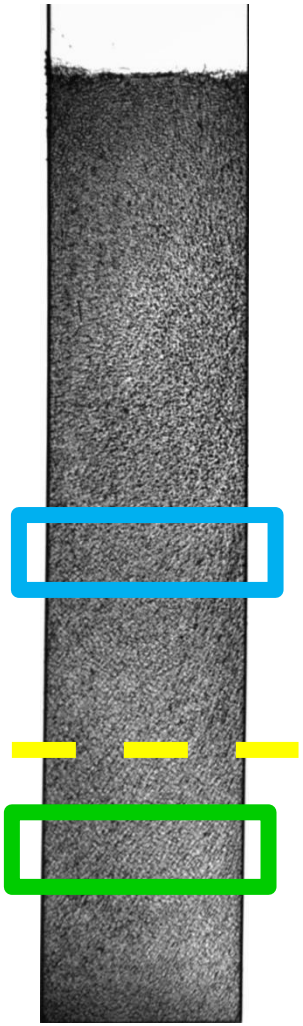


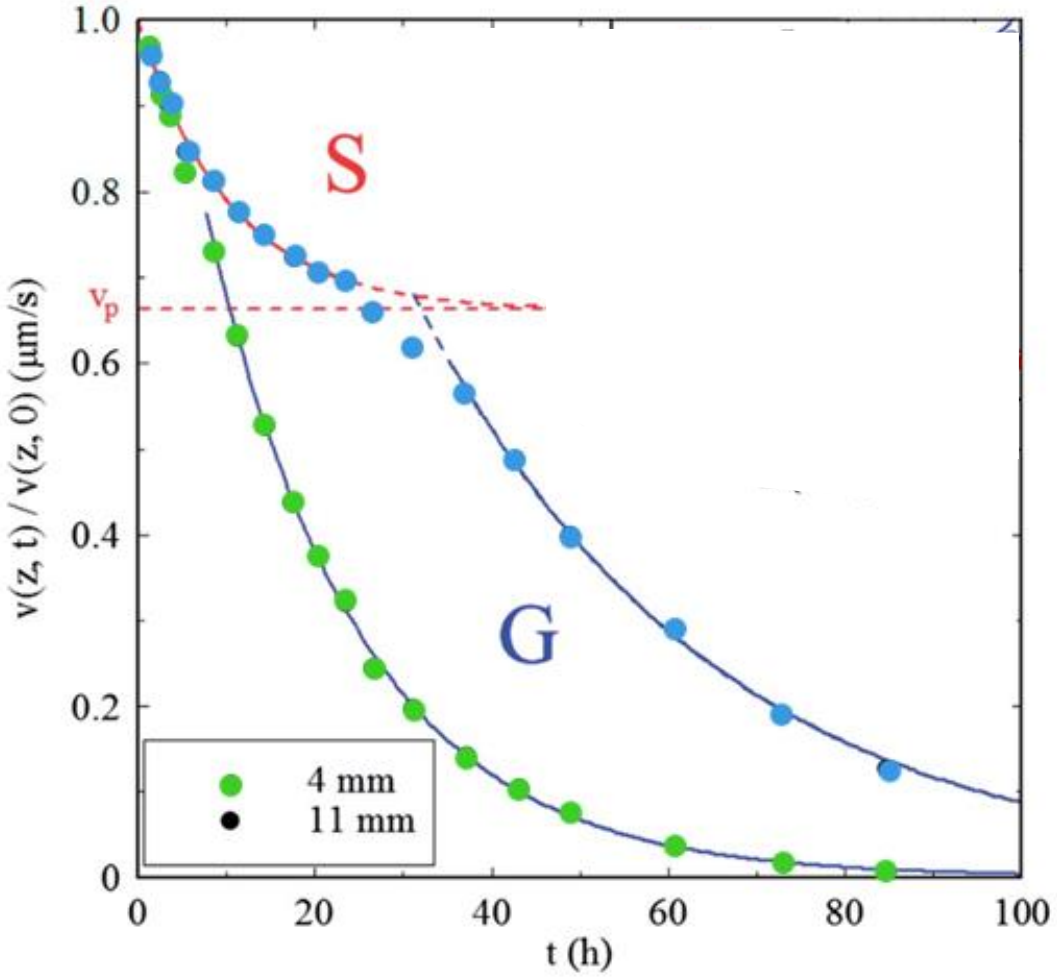
# Concentration Profiles



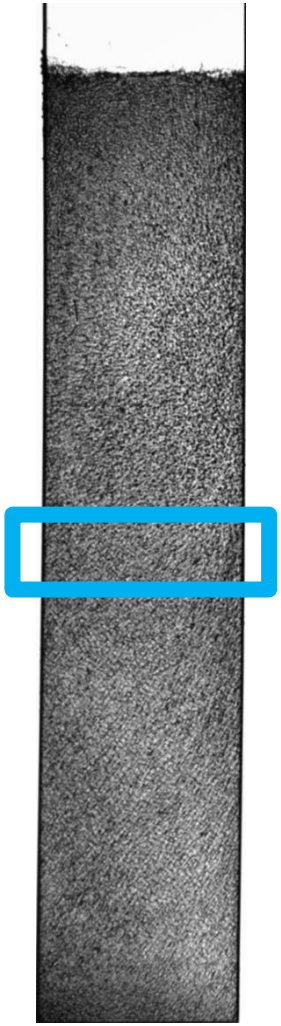



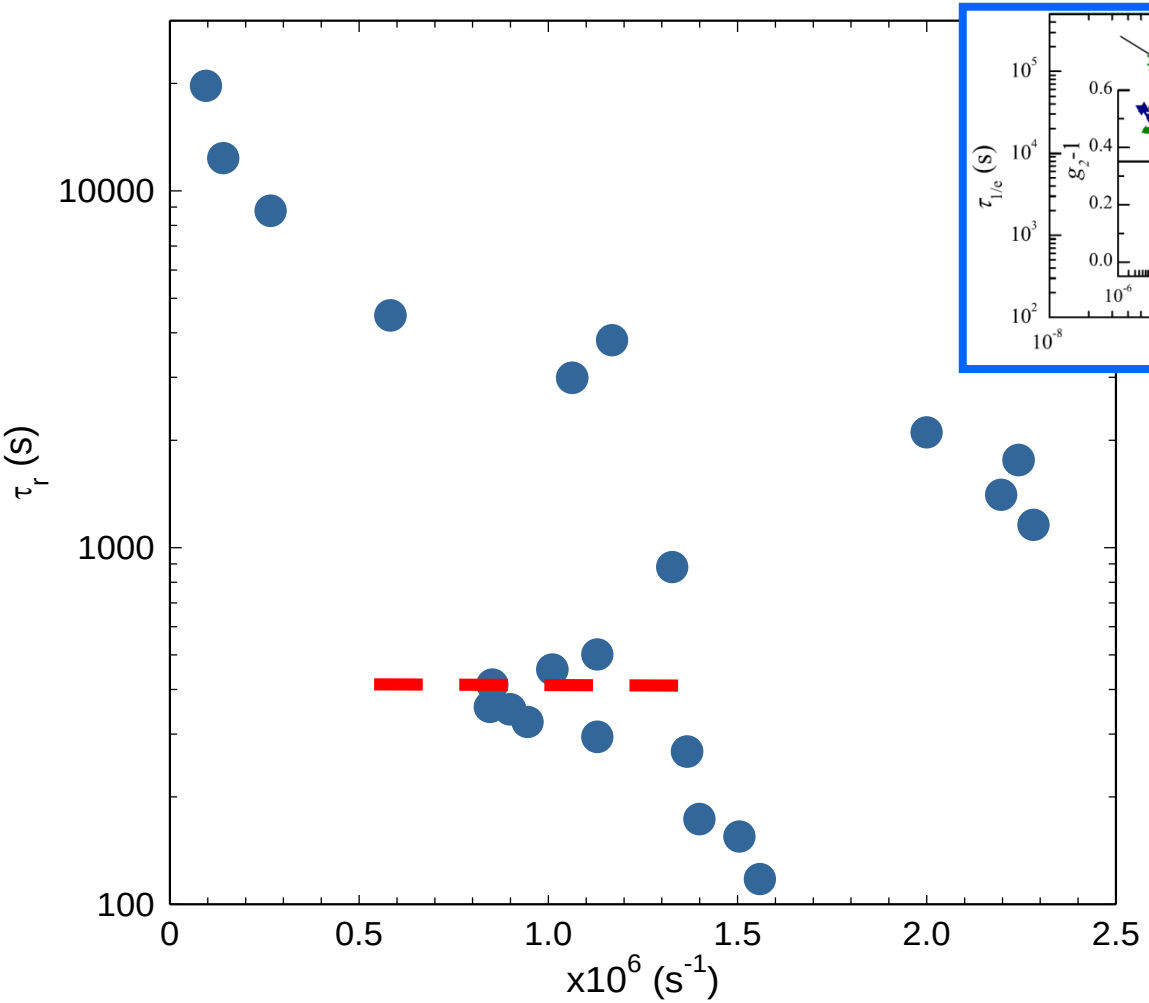
# Velocity

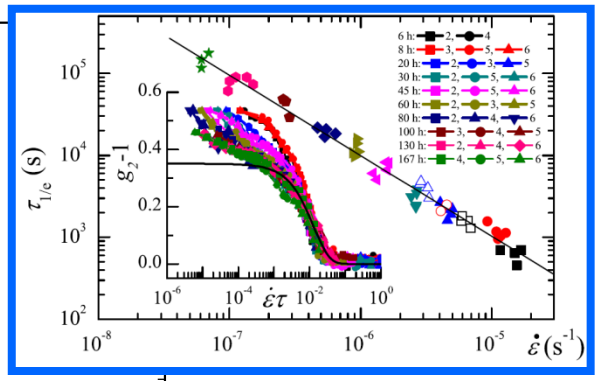




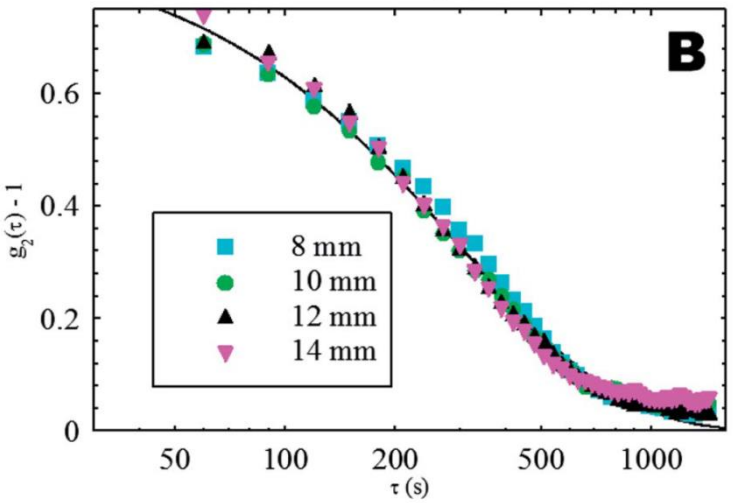
# $\tau_{1/e}$ vs $\varepsilon(t)$





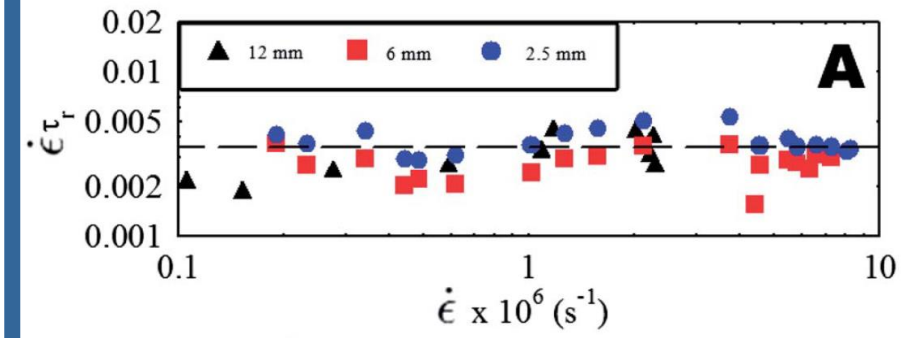



# S




The **whole** correlation functions do not depend on z and is basically unrelated to the macroscopic compression rate

# G




CUMULATIVE STRAIN =3.5 x $10^{-3}$ does not depend on z and comparable to the typical values measured in colloidal systems or molecular glass formers

# A tentative explanation of the apparent “syneresis”

Similarly to what happens for polymers, a newly-formed gel might be in a state of tension, with network strands that are over stretched with respect to their mechanical equilibrium condition.

In this scenario , gravity arguably has the sole effect of breaking the symmetry, favoring gel shrink age along the vertical. Were this true, similar (but in this case isotropic) syneresis effects should be detected in MICROGRAVITY CONDITIONS too.

# Open questions and perspectives

- **What's the origin of gel failure ?**
- **Boundary line location between CLASS A and CLASS B gels depends on gravity?**
- **How does crystalization proceed in Class C gels (Crystal Arrested Phase Separation as in B. Frisken experiments)?**
- **How microstructure influence stress propagation and gel failure?**

**SOLUTION: Microscopy (DDM?) on ISS**

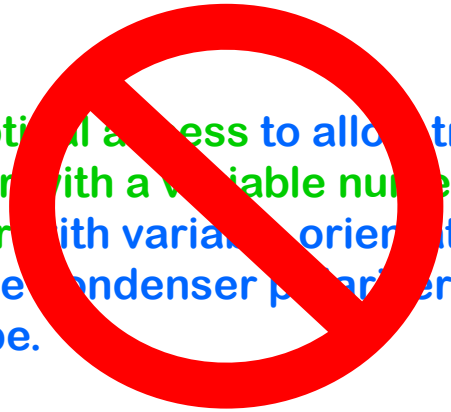
# Ou requirements

1) The ACE-T cooler shall have the ability to be controlled to a temperature +/- 0.1°C (+/- 0.2 is mandatory)

2) The ACE-T shall have the ability to maintain the temperature settings for a period of at least 8 hours with +/- 0.1 °C stability (+/- 0.2°C is mandatory). It should be fine if it is possible maintain the temperature settings for days. In this case it is sufficient to image the sample once for hours.

3) We don't want temperature gradients inside the cell. If each end of the cell have independently controlled variable set points a difference of 0.2°C/mm should be tolerable.

4) ACE-T shall have the ability to provide cooling to a sample cell that can achieve a temperature of at least 5°C below the ambient temperature and at least 35°C (20°C is mandatory) above the ambient temperature. We should to know the averaged/estimated ambient temperature before the fly to tune the samples critical temperature.



5) The cell should have two optical access to allow transmission configuration imaging. We need a condenser with a variable numerical aperture equipped with a polarizer. A second polarizer with variable orientation (at least two position: parallel or perpendicular to the condenser polarizer) has to be located in the imaging side of the microscope.

6) In our case an air objective with 60x and 0.5 NA is sufficient to achieve our goal. We don't need oil objectives.

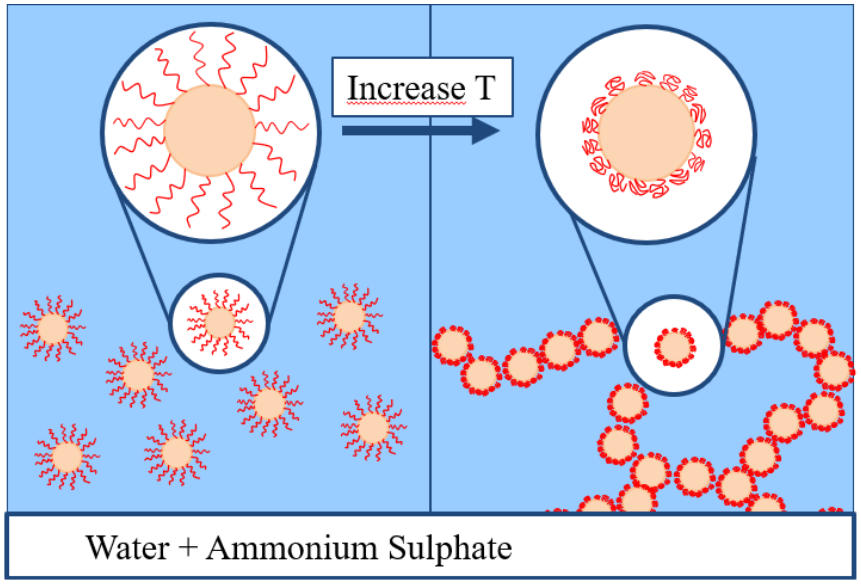
7) The ACE-T heater and cooler shall have the capability of controlling the rate of heating and cooling the sample to the required setpoint. ACE-T heater shall have the capability of reaching any setpoint with a rate of 5°C /min. ACE-T cooler shall have the capability of reaching any setpoint with a rate of 1°C /min (for our experiments cooling rate is not critical).

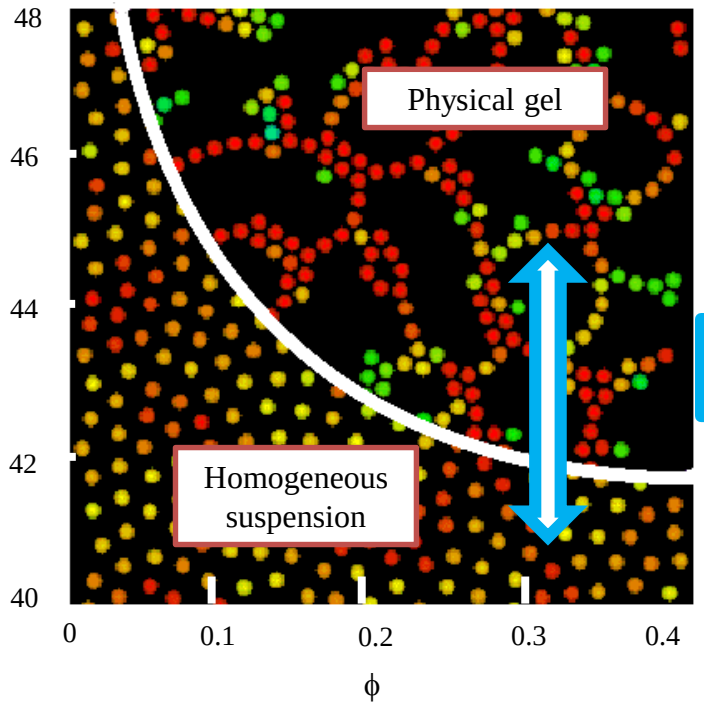
# Need to develop a new fluorescent colloidal system

# The new system

HFIPM nanoparticles stabilized with (PEG2000)

- Fluorescent
- Tunable Radius
- n=1.38
- Tunable Tgel with Ammonium Sulphate

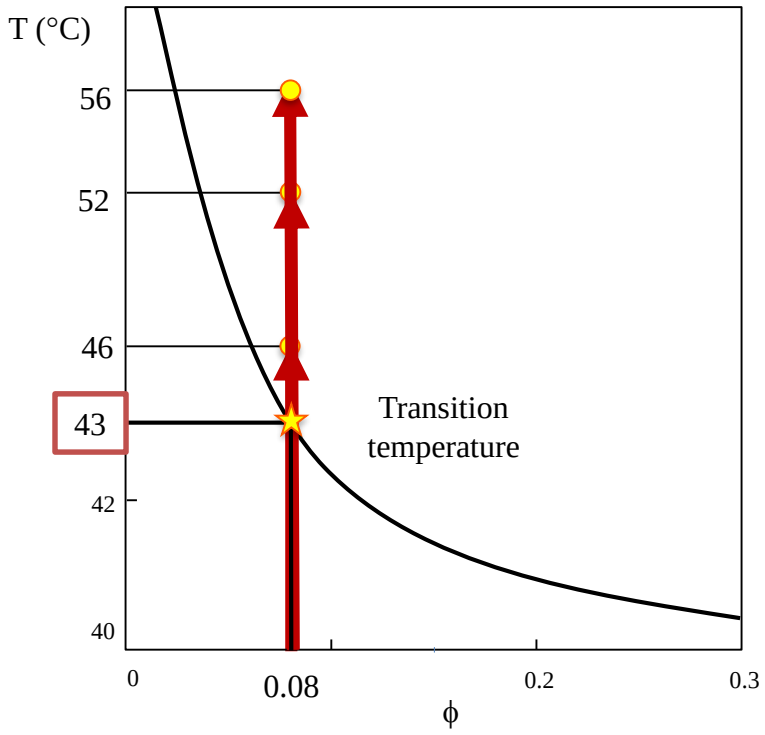


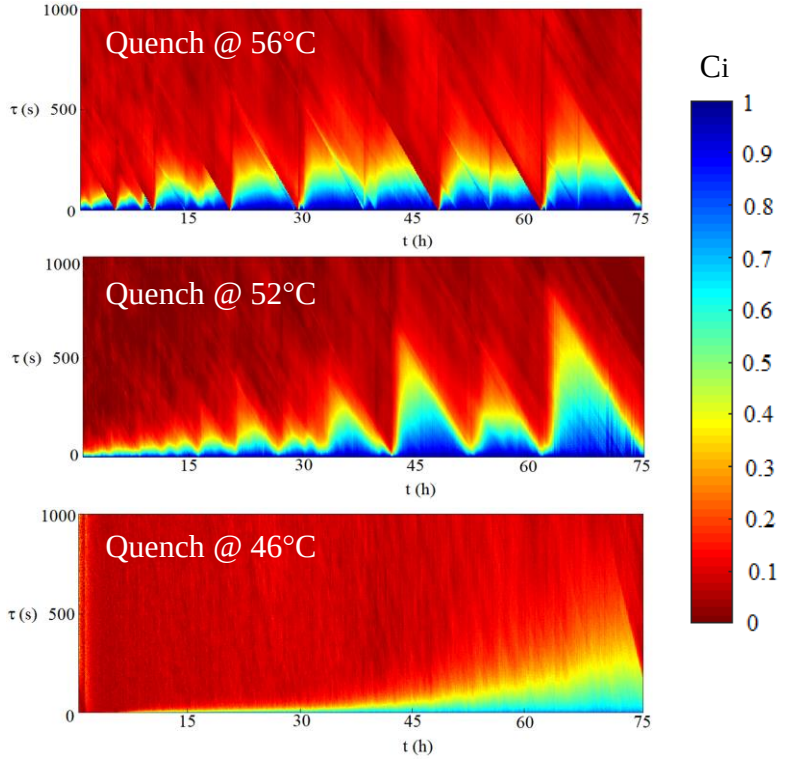




Palmiero U C, Agostini A, Lattuada E, Gatti S, Singh J, Canova C T, Buzzaccaro S and Moscatelli D.
Use of RAFT macro-surfmers for the synthesis of transparnt aqueous colloids with tunable interactions, 2017

# START of unpublished data

# Microscopic dynamics at different T

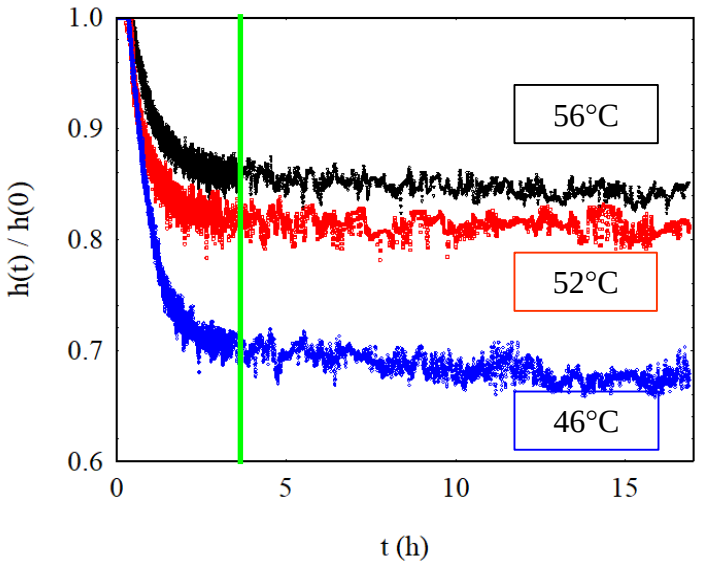


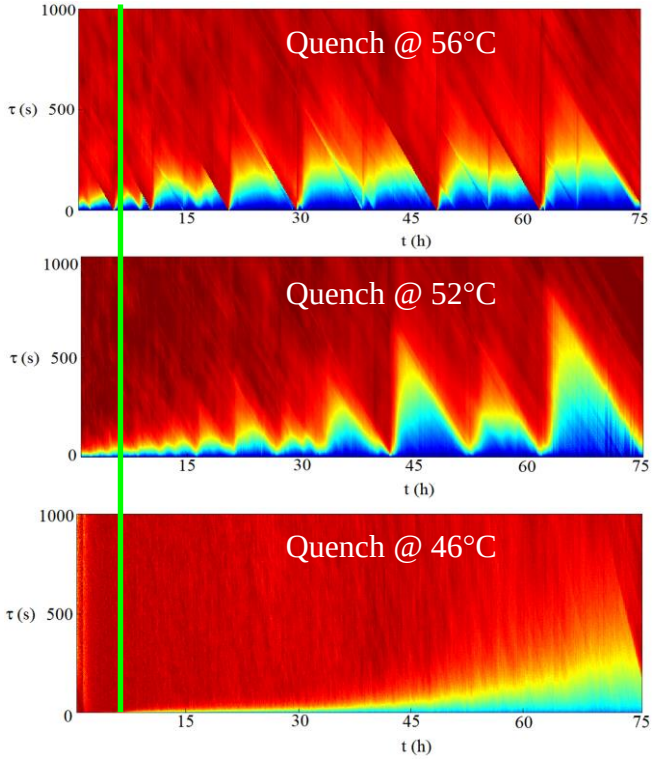



Change in the mechanical properties

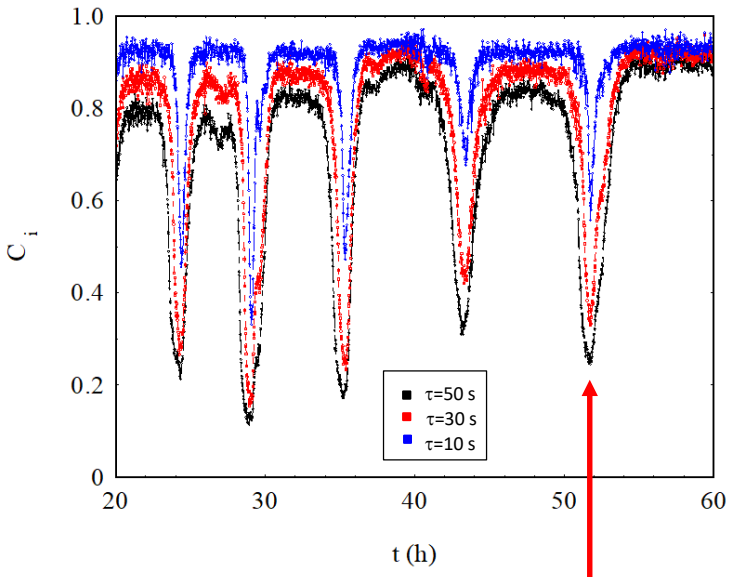
Temperature – Bomd Strength

Descent of the meniscus

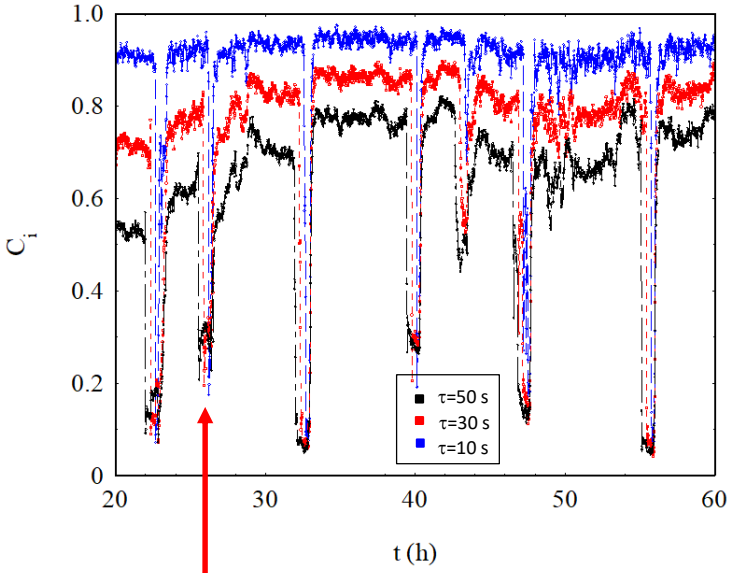


Moderate quench (52°C)



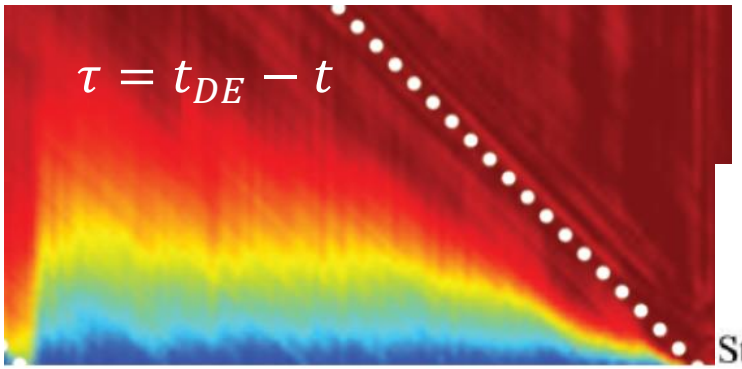

Strong quench (56°C)

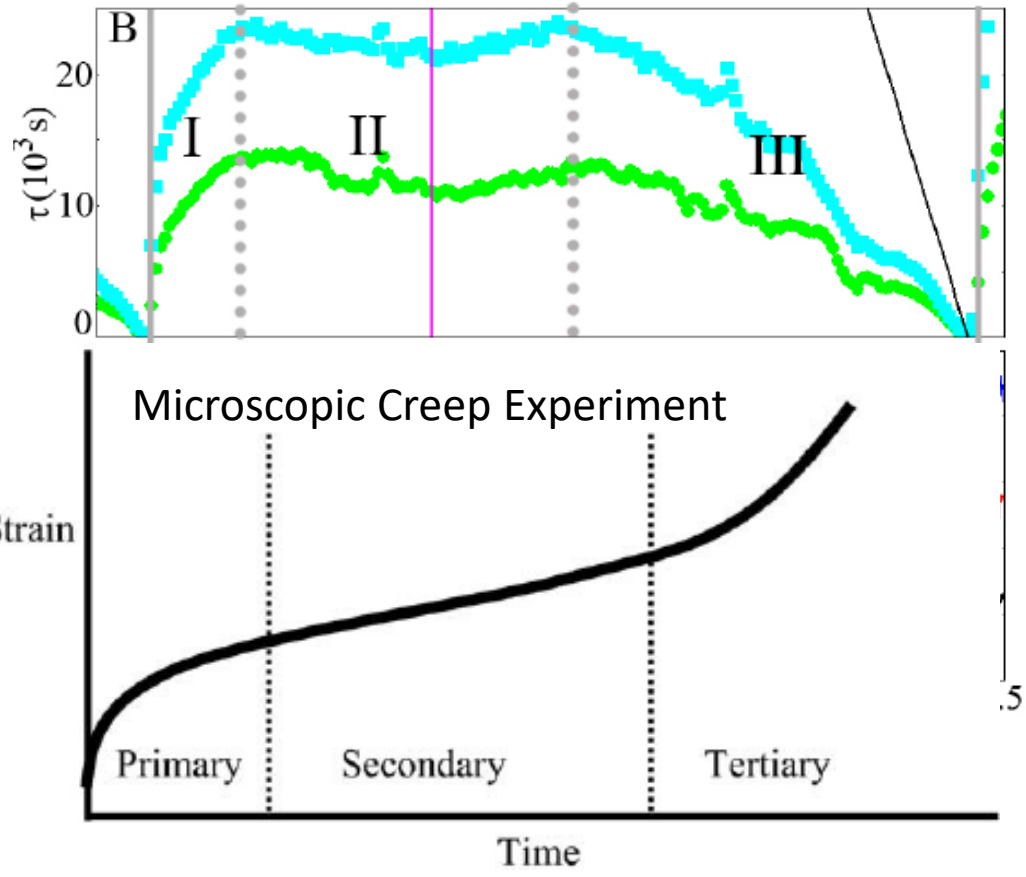



Complete re-arrangements of the structure

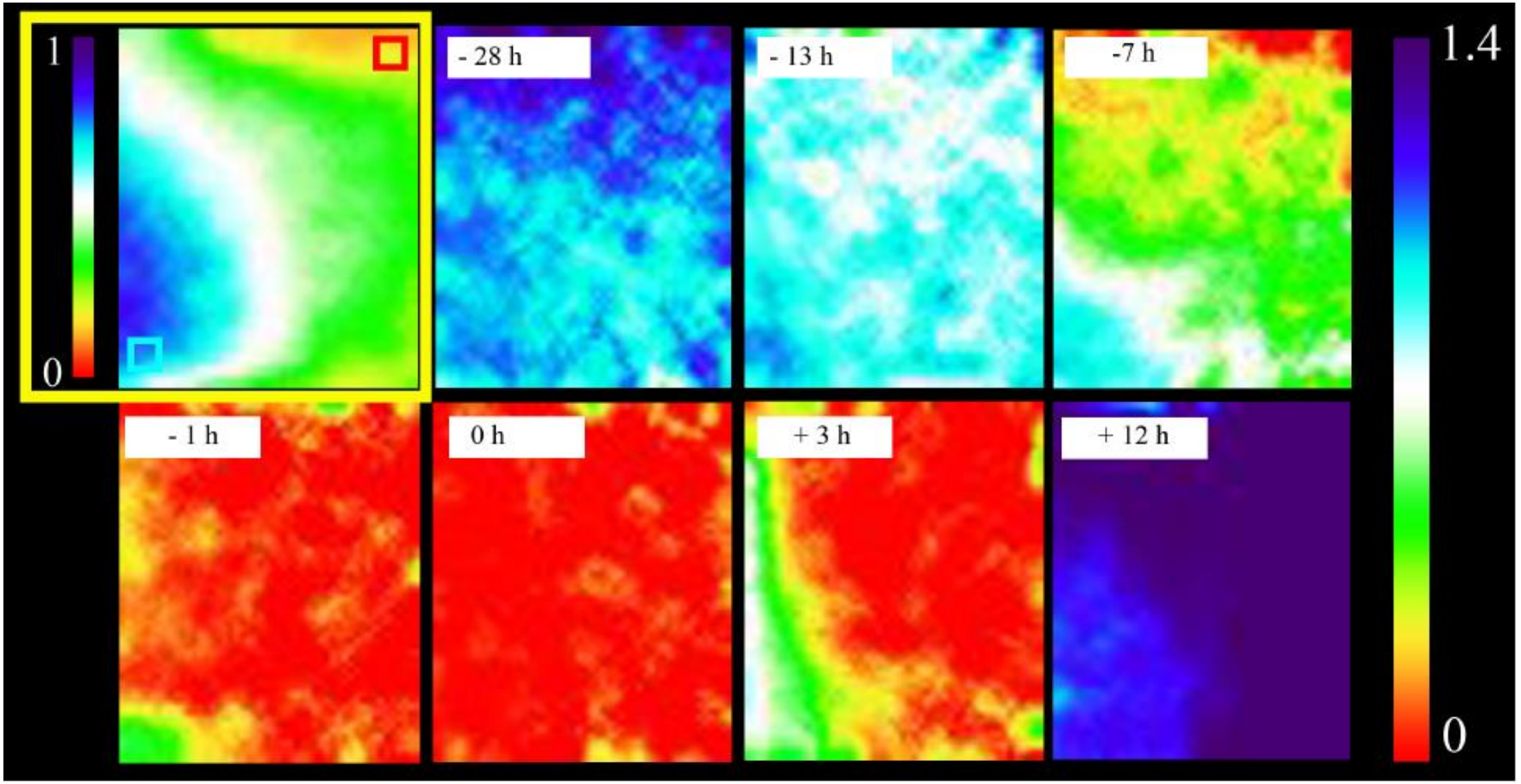
# Precursors of the “ failure”

Moderate quench (52°C)

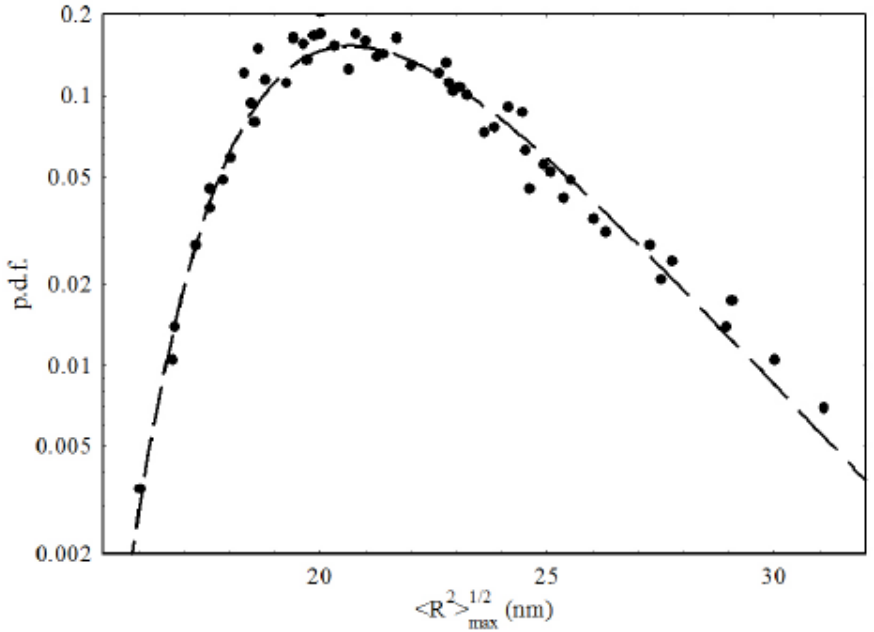


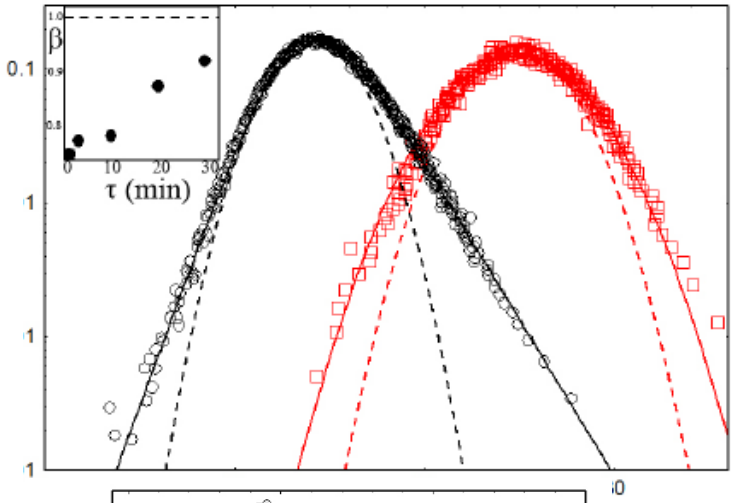



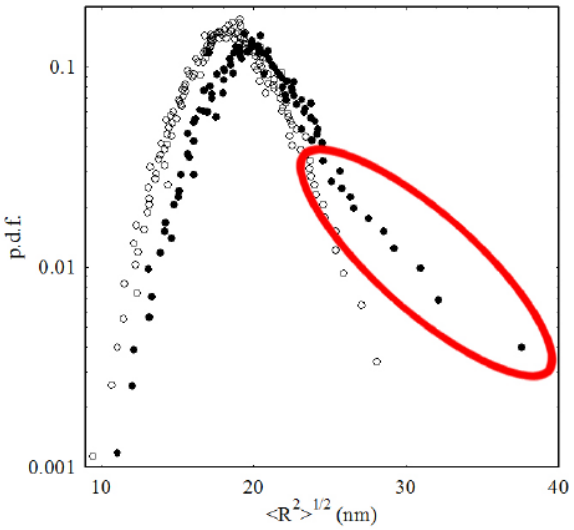
# Spatial propagation of the ‘‘failure’’



# Statistics of the quakes

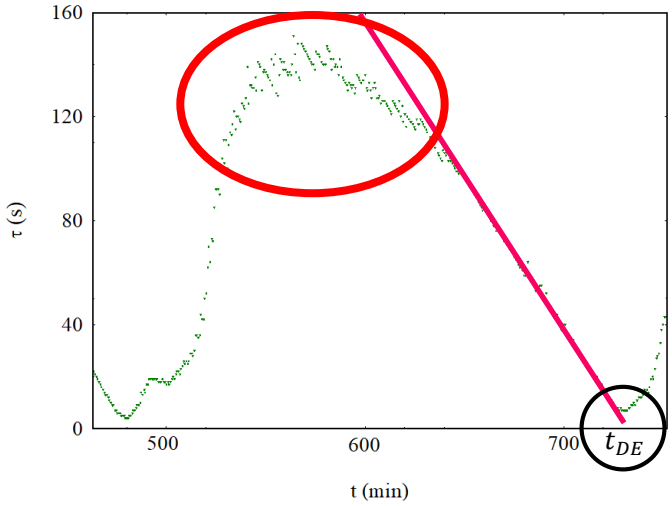


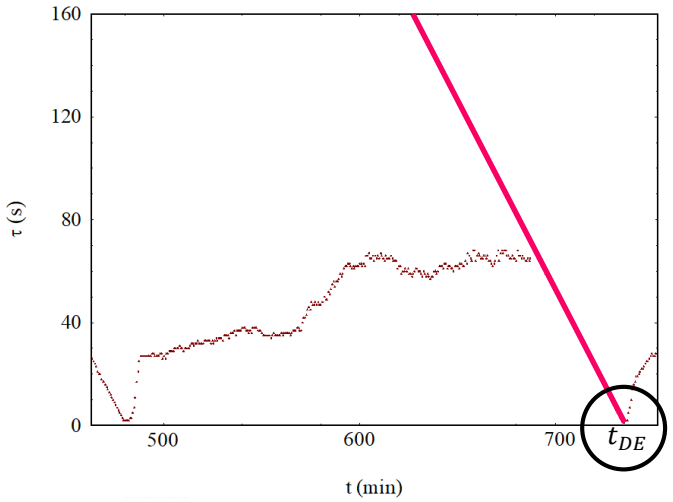
# Ductile to brittle transitions

Moderate quench (52°C)




Acceleration of the dynamics

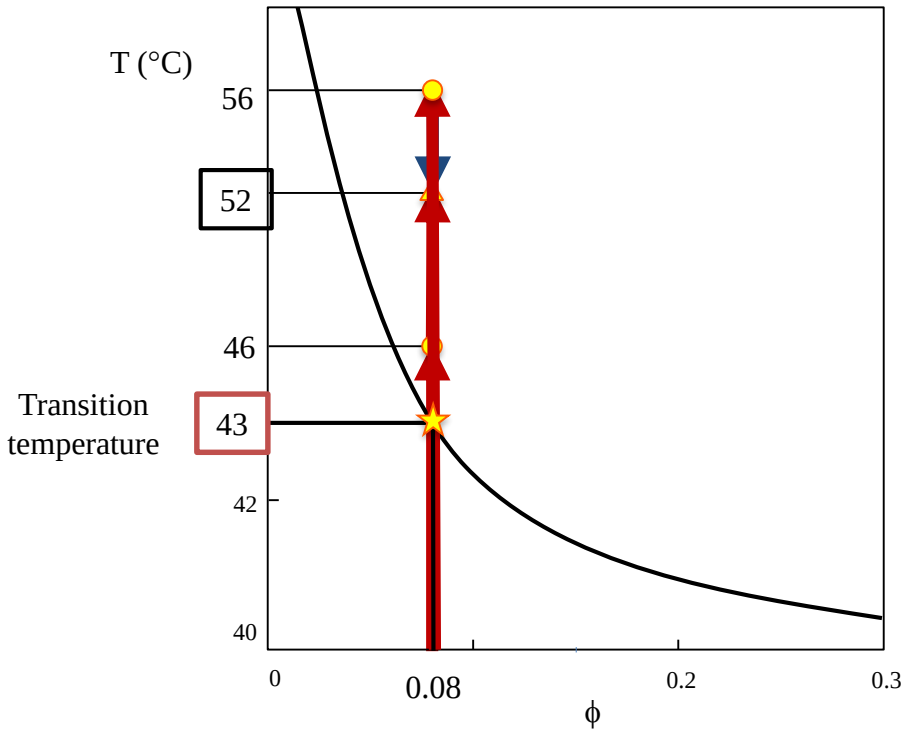
Strong quench (56°C)

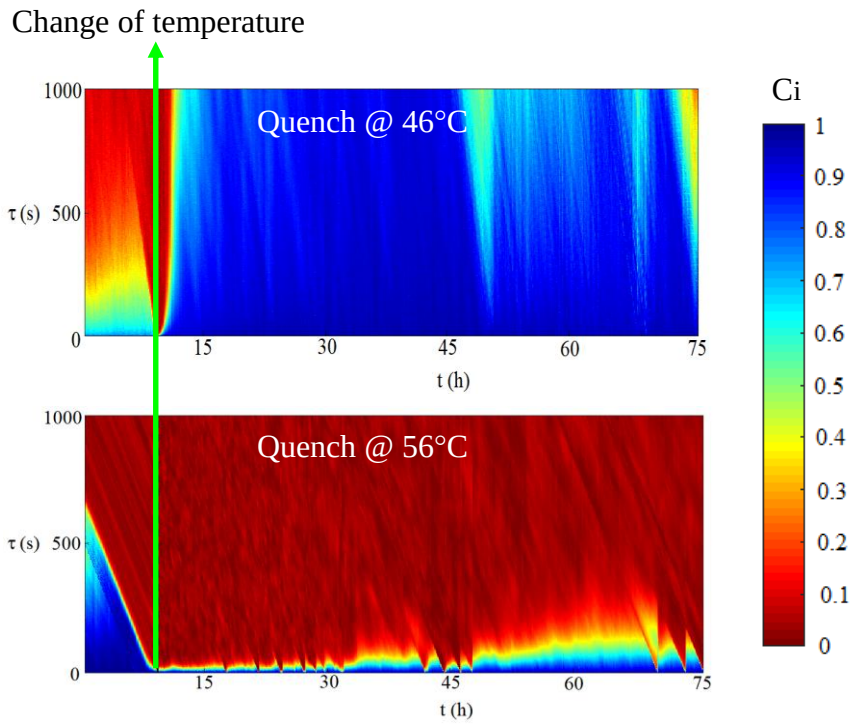



No substantial change in the dynamics

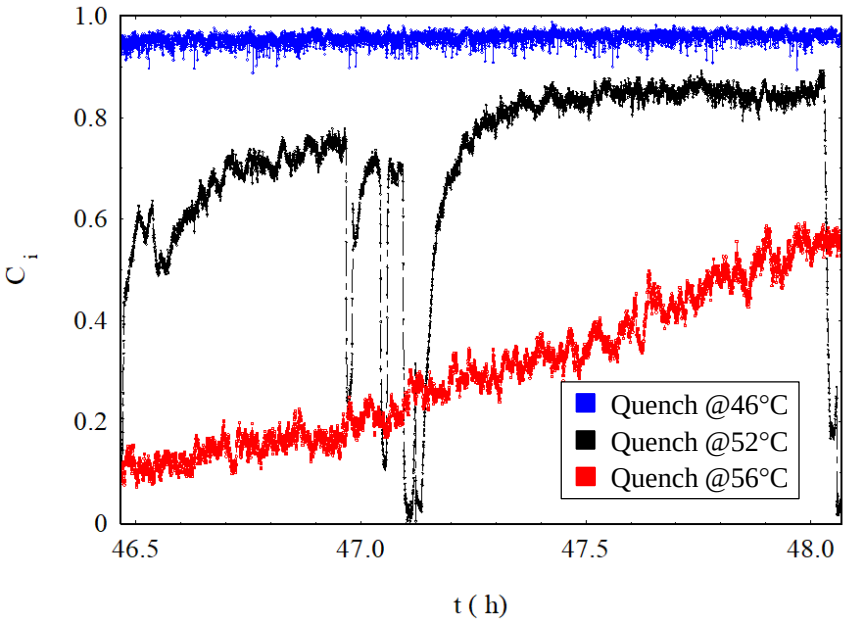
«Ductile – Brittle Transition»

# Change of temperature after the quench



# Change of temperature after the quench
# Memory of the system



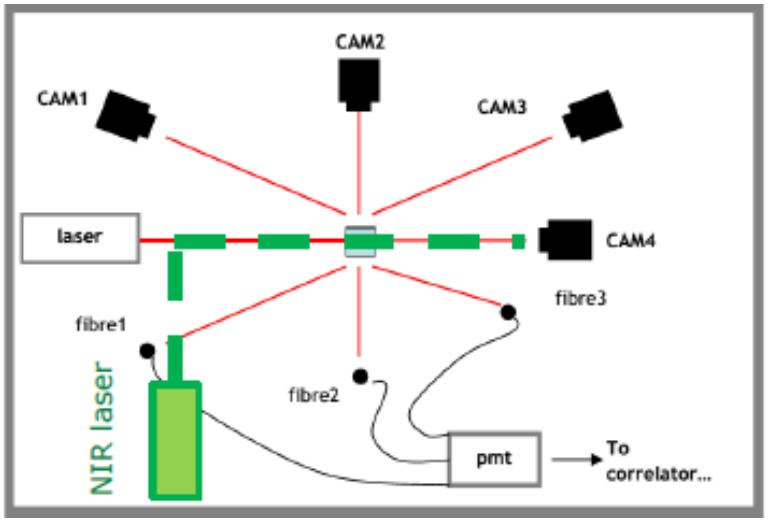

The gel keeps the memory of the temperature formation (accumulated stress)

Prince Rupert's Drop


Deep quench brings high internal residsual stress

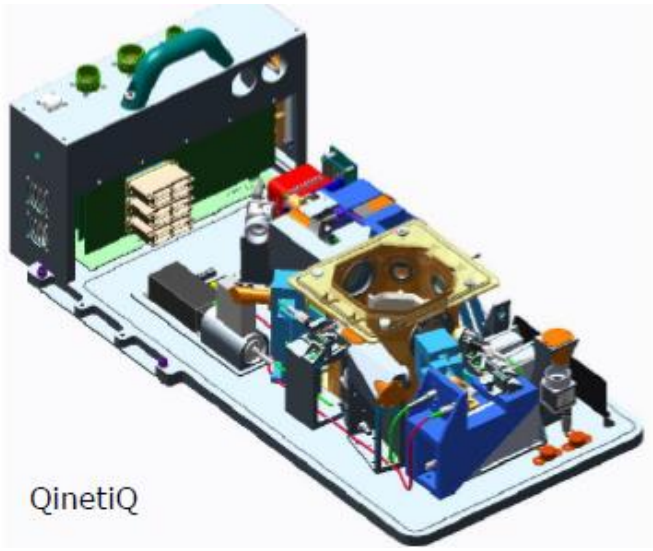
END of unpublished data

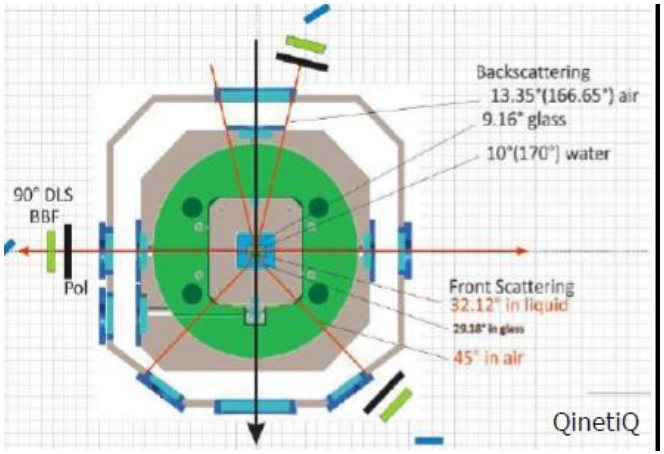
# PCI on ISS: Colloidal Solids Setup




**Roberto** Piazza is the PI of the **Esa** project **«Colloidal Solids» .**
In collaboration with **Stefano Buzzaccaro** and **Luca Cipelletti (Univ. Montpellier**) it was developed an versatile, state-of-the-art, and flexible light scattering apparatus.

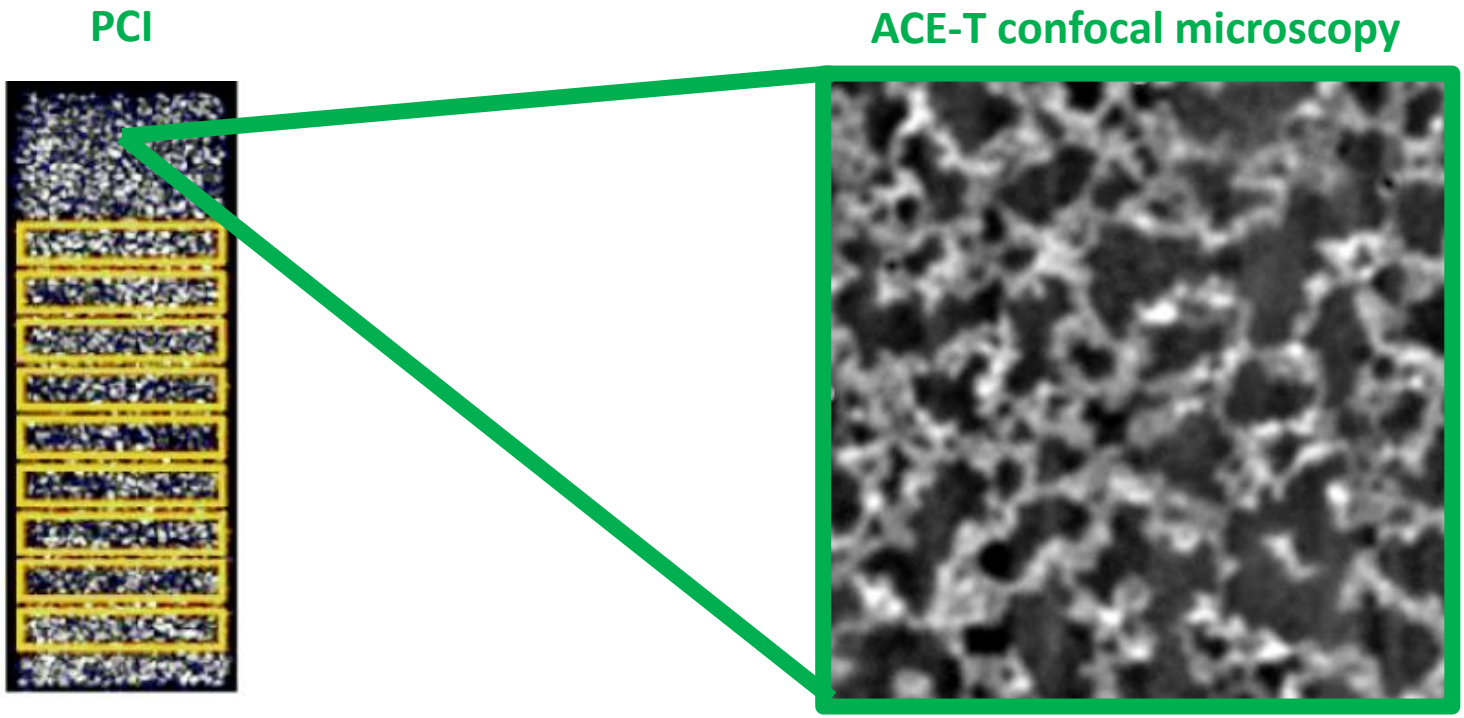
It allows multi-angle static and dynamic light scattering onboard the ISS + multiangle PCI.
In addition the sample can be mechanically stressed by using an infrared laser beam that it's partially absorbed by water.



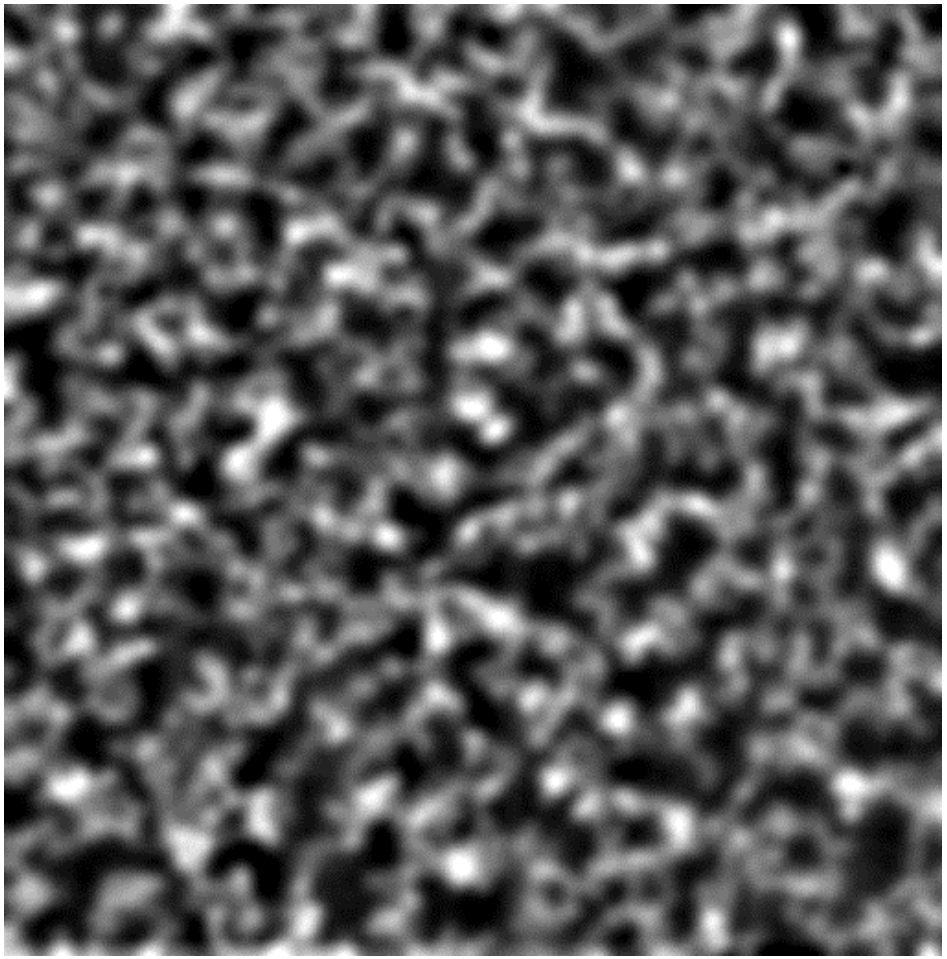
# Investigation goals and objectives (1/3)

Our PCI investigation allows to link localized plastic rearrangements to macroscopic stress relaxation but we don't have access to gel microstructure. One pixel of our PCI images corresponds to a volume inside the gel of around 50 x 50 $\mu m^2$




The ACE-T10 confocal microscopy data will allows us to follow the gel microstructure evolution, elucidating the role of gel strands break-up with the plastic rearrangement of the system.

# Investigation goals and objectives (2/3)



An example of what
we expect to see with
the ISS confocal microscope

# Investigation goals and objectives (3/3)

## ACE-T10 Measurements / Observables

Basic measurements/observables that we will probe with these experiments.
(Note, the samples are designed to tune the inter-particle forces changing the sample temperature T.)

## For each T

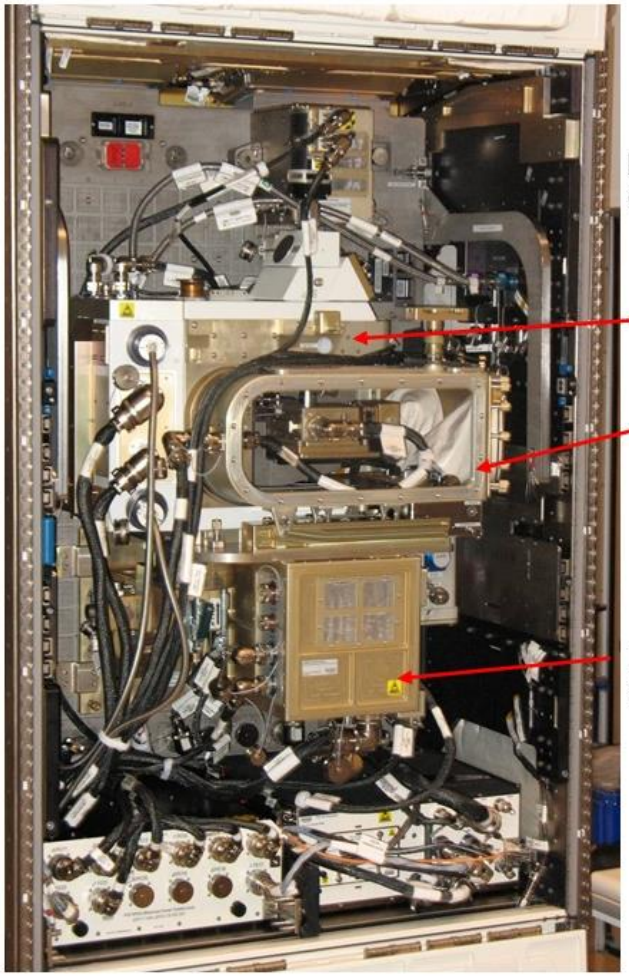
- **Time evolution of the gel structure factor S(q)**
- **Statistic of the rupture/joining events**
- **Study of gel evolution using the Confocal Differential Dynamics Microscopy (Conf DDM)**
- **Gel characterization using Minkowski Functionals (MFs):** An elementary question in porous media research is in regard to the relationship between structure, function and macroscopic properties. In the context of statistical physics it was highlighted the importance of the use of the MFs for the geometric characterization of porous media. The calculation of MFs is possible on ACE-T10 thanks to the capability to 3D-reconstruct the gel structure.

# Measurement approach – 1/17

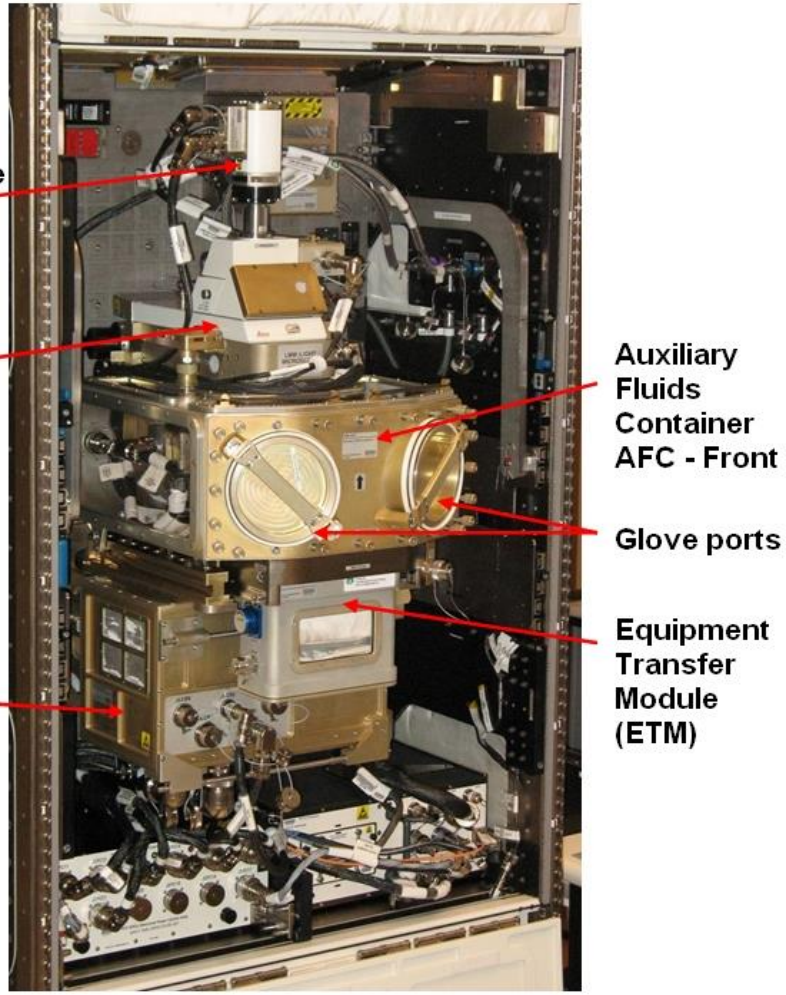
We will be using a motorized flight-hardened Commercial-Off-The-Shelf (COTS) Leica DM-RXA microscope
with a Yokogawa spinning disk confocal head
[pictured later]
and
ACE-T sample modules
[pictured later]

# Measurement approach – 2/17

Light Microscopy Module (LMM) in the Fluid Integrated Rack (FIR)



LMM in the Closed Position or Operating Configuration




LMM in the Open Position or Installation/Service Configuration

# Measurement approach – 3/17

## LMM Implementation Philosophy

Philosophy: Maximize the scientific results by utilizing the existing LMM capabilities. Develop small sample modules and image them within the LMM

Payload specific and multi-user hardware customizes the FIR in a unique laboratory configuration to perform research effectively.

**Light Microscopy Module**

### Payload Specific Hardware

- Sample Cell with universal Sample Tray
- Specific Diagnostics
- Specific Imaging
- Fluid Containment

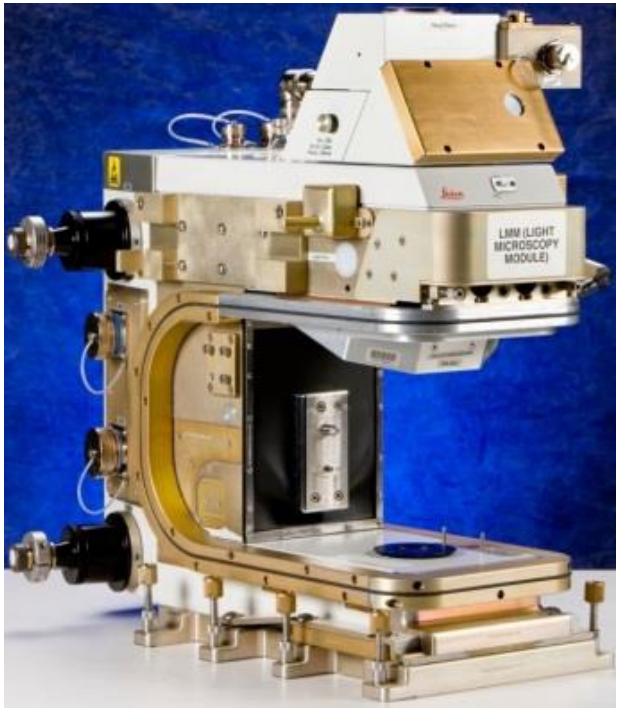
### Multi-Use Payload Apparatus

- Test Specific Module
- Infrastructure that uniquely meets the needs of PI experiments
- Unique Diagnostics
- Specialized Imaging
- Fluid Containment

### FCF Fluids Integrated Rack

- Power Supply
- Avionics/Control
- Common Illumination
- PI Integration Optics Bench
- Imaging and Frame Capture
- Diagnostics
- Environmental Control
- Data Processing/Storage
- Light Containment
- Active Rack Isolation System (ARIS)

# Measurement approach – 4/17




Light Microscopy Module (LMM)

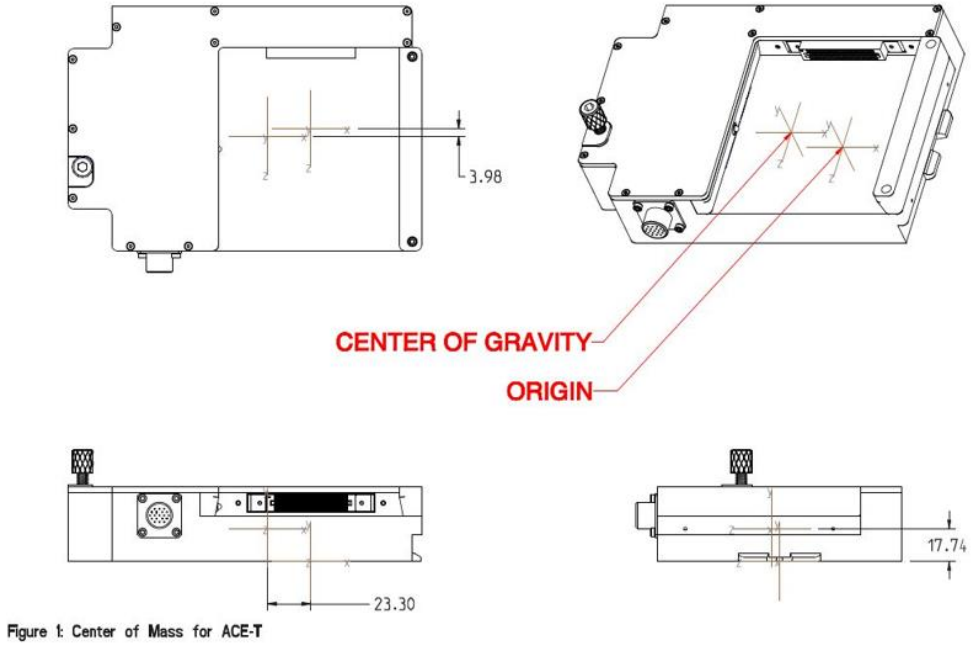
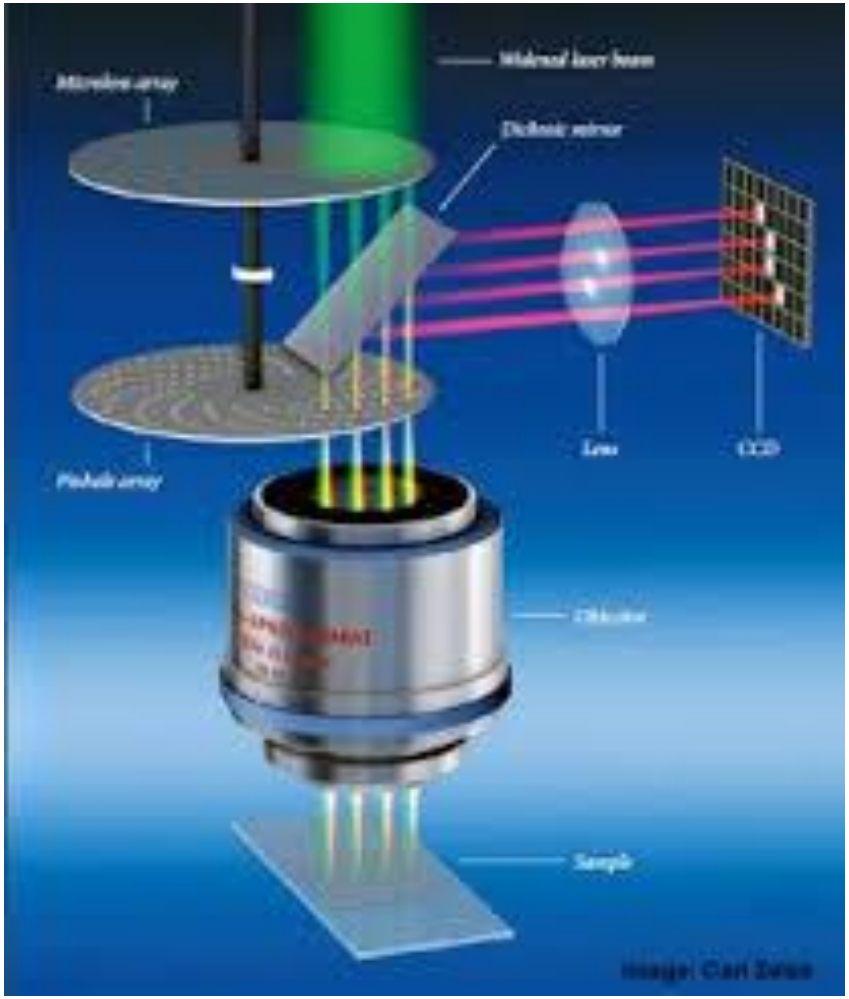
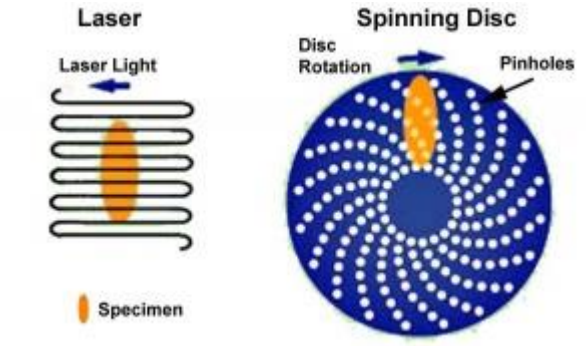



Figure 1: Center of Mass for ACE-T

ACE Sample Assembly with Removable ACE-T Sample Tray that will contain a row of 3 temperature controlled capillary cells

# Measurement approach – 5/17






Using confocal microscopy to acquire 3D images.

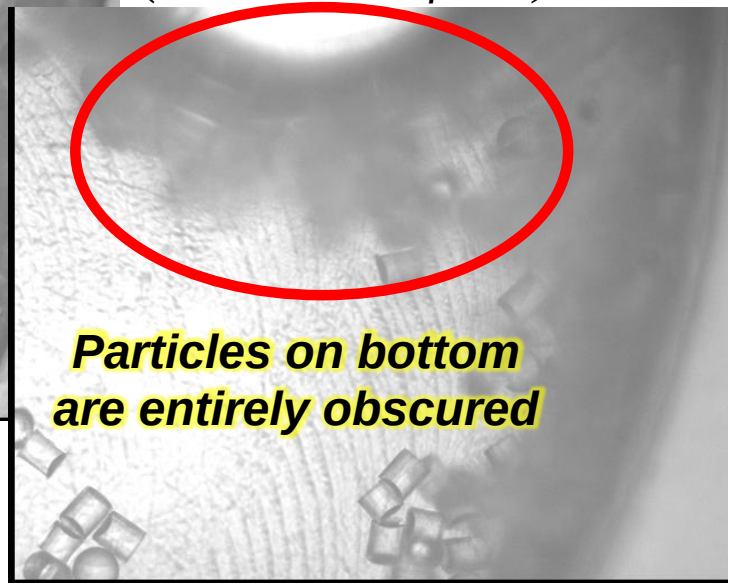
# Measurement approach – 6/15

## The difference between traditional and confocal microscopy

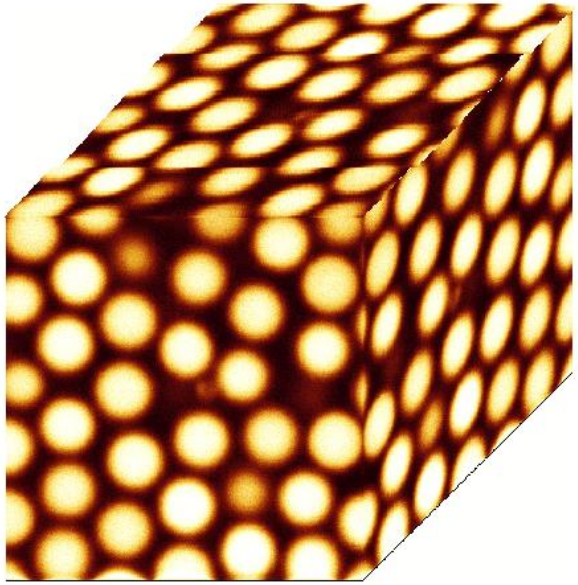
***Traditional microscopy doesn't see through objects well; out-of-focus light obscures in-focus light***

*Focus 50 µm below cover slip*


*Focus 200 µm below (near bottom of well)*




***Confocal microscopy rejects out-of-focus light, to look through semi-transparent objects***



***3D image is built out of 2D XY sections while stepping through Z axis.***

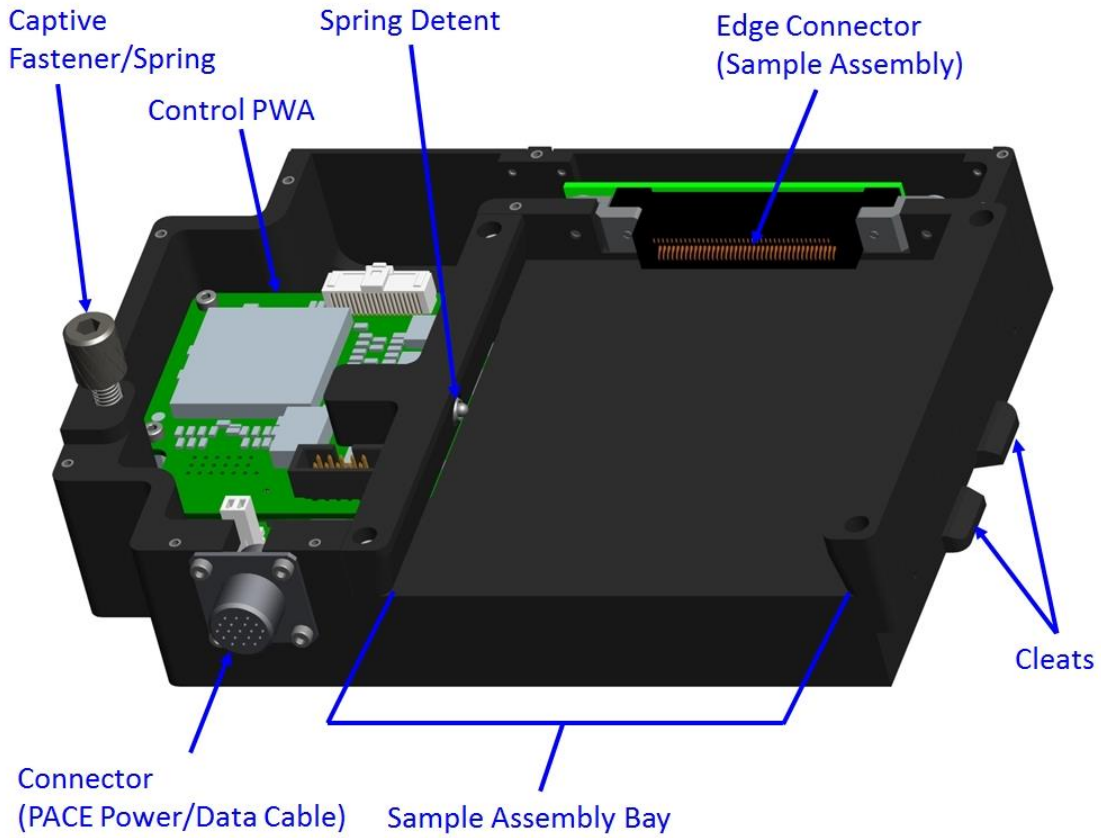
# Measurement approach – 7/17

# Mechanical Design Highlights

- Modular sample assemblies
  - Allows for multiple sample configurations.
    - Easier Sample replacement
    - Decreased “ACE-T” up-mass in comparison to ACE-H

# Measurement approach – 8/17

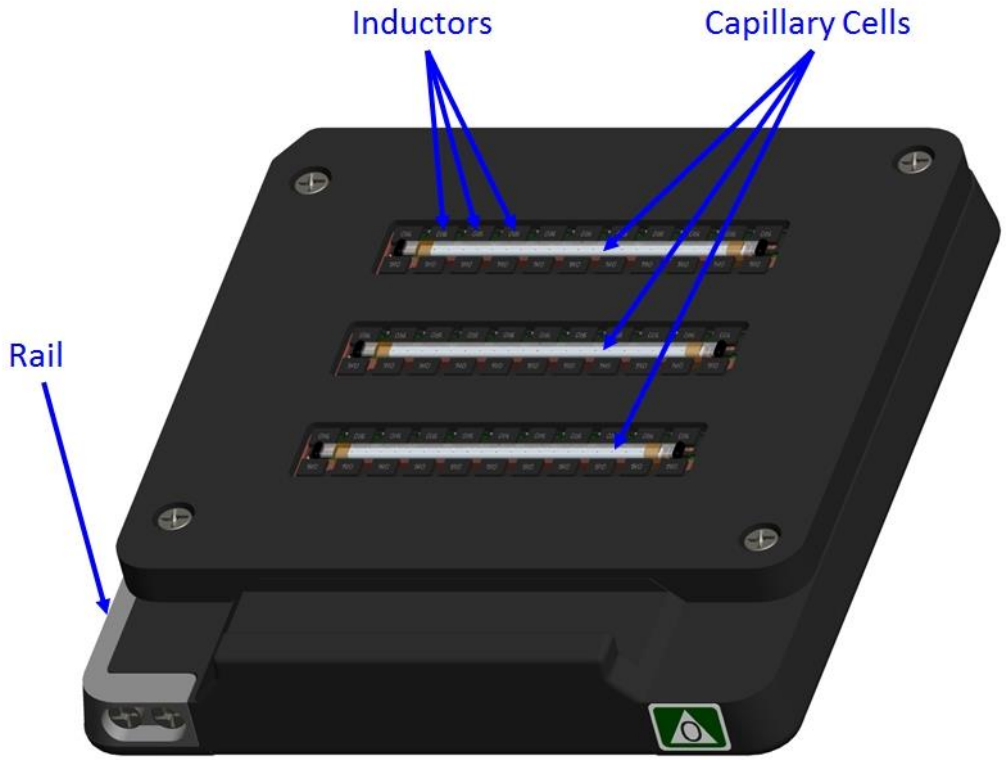
# Mechanical Design Highlights



# Measurement approach – 9/17

## Mechanical Design Highlights

- In-situ mixing (details in electrical section)
- Black Hard Anodize Surface Coat
  - Reduction of any errant light within the AFC
  - Increased wear resistance

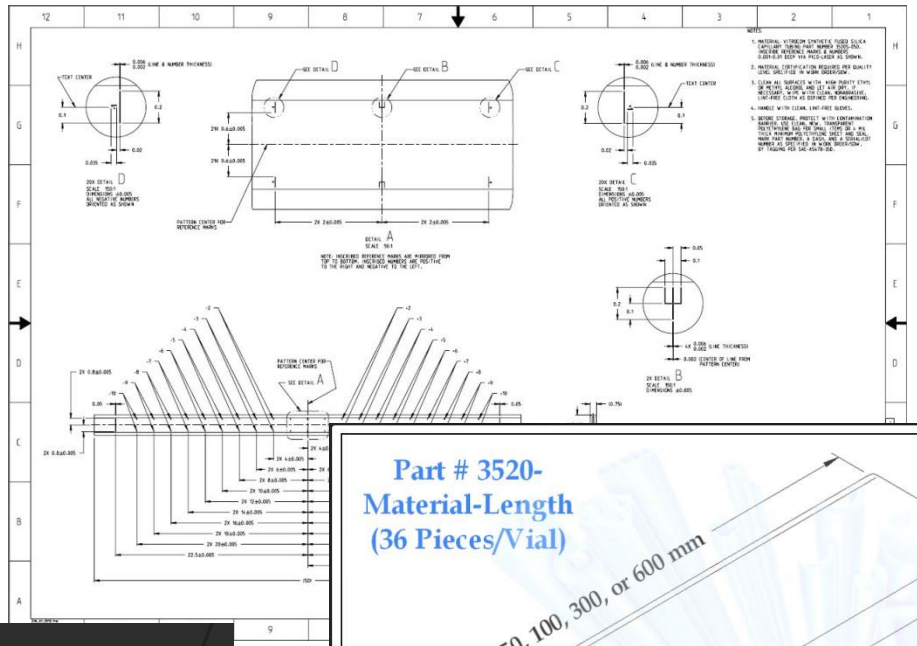


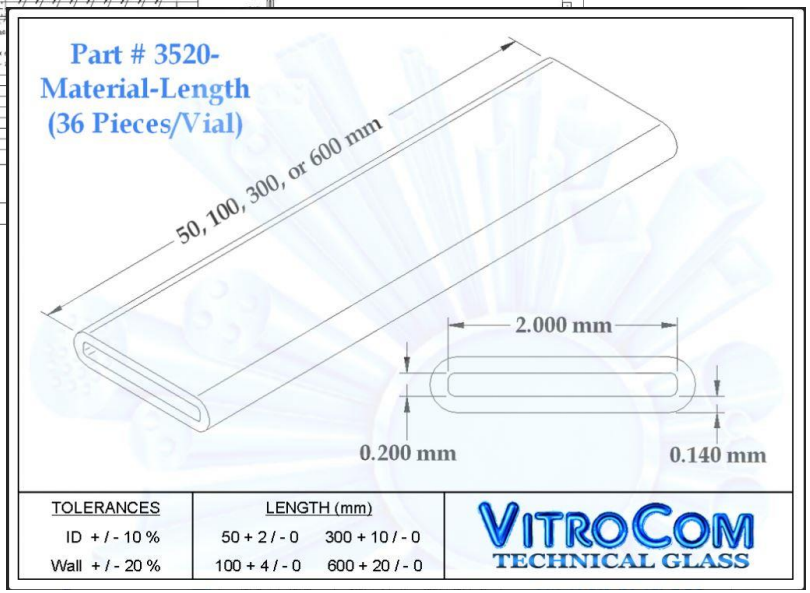
# Measurement approach – 10/17

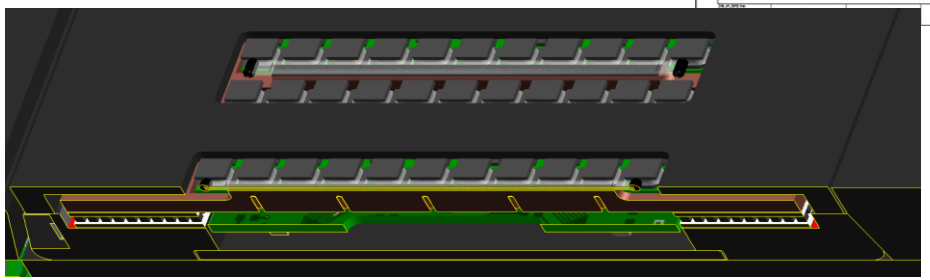
# Mechanical Design Highlights

## Capillary cell

- Purchased through VitroCom.com
- Material
  - Borosilicate (3520-050)
  - Fused Silica by request (3520S-050)
- COTS
- 50mm length
- Reference Marks
  - Secondary Process to ease positional awareness





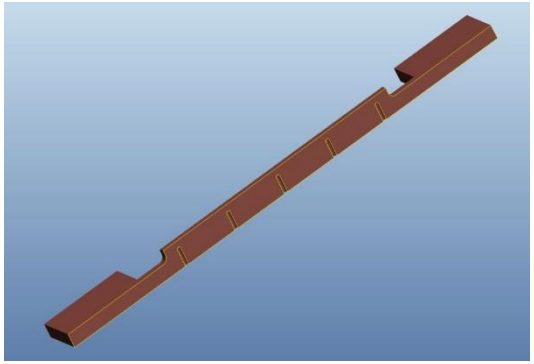



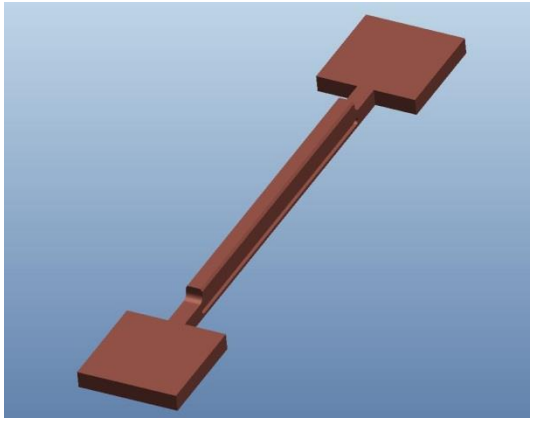
Two capillary cells surrounded by inductors that are used for walking a turning stir-bar for sample mixing.

# Measurement approach – 11/17

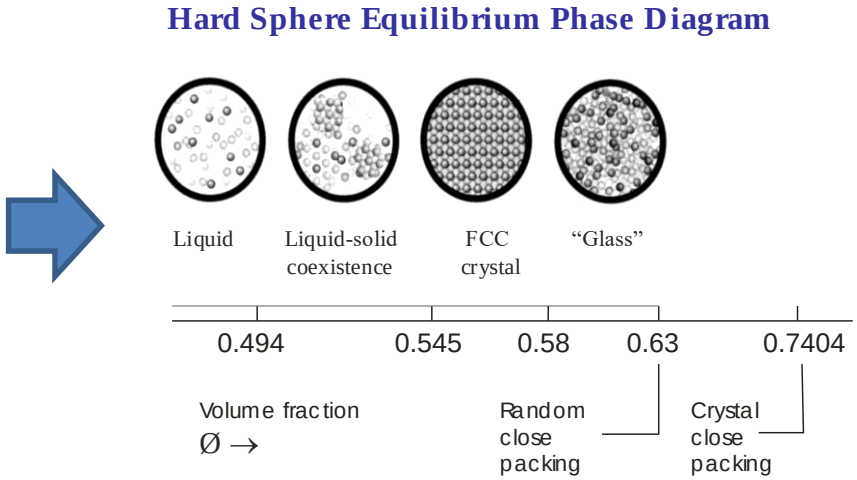
# Temperature gradient option

- Thermal bridge
  - Material: Copper
  - Bridges thermal energy between TEM's
  - Constrains Thermistor Positioning
  - Thermal symmetry across X and *Y Axis
    *When set-points are equal





Bonus information: ACE-T, in general, will enable temperature control that can either be linear across the capillary - or a temperature gradient across the capillary. A temperature gradient will form a density gradient! You can now march through a phase diagram using a single capillary and have a common error bar for all measurements.



# Measurement approach – 12/17

**Procedure to determine the primary and secondary ROI (steps 1 to 6)**

1. Locate the desired sample under the LMM microscope.
2. Mix the sample in sample module using the in-situ mixer for 15 minutes or greater to achieve homogenization.
3. Inspect the sample with the 2.5x objective.
4. Select an exposure time based on the observed luminescence.
5. Determine a ROI for the preliminary study. Also, locate a secondary ROI.
    a. The ROI will not have dust or a bubble right next to it, nor will the stir bar be visible, but it will appear as a uniform field on the screen
    b. Consult with a member of the Science team.
6. Examine the selected ROI with the 20x and the 63x (air) objectives to insure that the area selected is uniform under those magnifications.

# Measurement approach – 13/17

**Procedure to determine Tg (steps 7 to 20)**

7. Acquire 100 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary, at 1 fps
8. Heat sample to 29$^{o}$C for 2 minutes then observe at 63x.
9. Acquire 100 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary, at 1 fps. Use this information to determine the Image Average Background (IAB).
10. If sample does not appear to begin coarsening (or alternately, the variance between images is not increasing as analyzed with the script provided by the Science team from the Image J software and the IAB), then heat sample to 31$^{o}$C for 2 minutes then observe at 63x.
11. Acquire 100 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary, at 1 fps.
12. Repeat Steps 10 and 11 as needed at 2$^{o}$C increments until rapid coarsening is observed and sample shows rapid change when analyzed using Image J software. Do not exceed 49$^{o}$C unless directed by the Science team.

# Measurement approach – 14/17

13. The temperature where the rapid increase of the variance occurs is Tg1, namely a first approximate guess for Tg.
14. Cool the sample down to 25°C
15. Mix sample using automatic mixing until re-homogenized.
16. Set the sample temperature to Ts= Tg1-2°C for 2 minutes then observe at 63x.
17. Acquire 100 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary, at 1 fps.
18. Repeat steps 16 to 19, starting from Ts+0.5 in steps of 0.5°C up to Tf= Tg1+2°C (8 measurements in total)
19. Determine the temperature at which the images show the largest changes/coarsening with respect to the image taken at the previous temperature step (namely, the largest increase of the image variance, calculated with a ImageJ script). This temperature is assumed to be **Tg**.
20. Check whether the Science team, after having examined the collected images, agrees with this value for Tg.

# Measurement approach – 15/17

**Procedure to study the gelation dynamics (steps 21 to 36) at fixed Texp**

21. Cool the sample down to 25$^{o}$C
22. Mix sample for 15 minutes or greater to achieve complete homogenization
23. Acquire 100 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary, at 1 fps.
24. Set the sample temperature to T=Tg-1$^{o}$C for 2 minutes then observe at 63x.
25. Check exposure time versus luminescence for the 63x and 20x magnifications with the ACE-T10 Science Team. This exposure time will be utilized for observation until notified to change by the ACE-T10 Science team.
26. Set the imaging system to acquire 1000 images at the ROI with 63x magnification at a plane 50 microns below the top inner surface of the capillary at the highest frame rate possible.
27. Simultaneously, set the Sample Heater to Texp=**Tg**+1°C or + 2$^{o}$C (based on the ACE-T10 Science team choice). The aim is capturing any changes to the sample that happen very quickly.

# Measurement approach – 16/17

The temperature in the following steps will be held steady as long as it takes to complete the acquisition of the required imagery.

28. Once completed, initiate another series of 1000 images at the same height and location at a rate of 0.5 fps (~2000 seconds).

The final series of images will be taken with the Confocal Imaging system at 63x (or 20x, see below) with a z stack separation as noted.

Downlink images during the interims, as possible.

If the sample material in the ROI should become bleached, switch to the secondary location selected in step 5, based on input from the Science team

Send or make the imagery available to the Science team for their evaluation. It is possible that the extended exposure to temperature could be made shorter or extended based on the ongoing results.

29. After completing the last set of stationary plane imagery, utilize the Confocal Imaging system with either the 63x or 20x objective (as determined by the Science team) to take a 51 image stack of images at the highest frame rate possible every 40 seconds. The images should have a nominal separation of 1.0 microns and should range from 25 microns above to 25 microns below the previous image plane (50 microns below the top inner surface of the capillary).
30. Obtain 1000 image stacks. This will take in excess of 11 hours.
31. Adjust the exposure times as required, based on Science team input.

# Measurement approach – 17/17

32. After completing the acquisition of 1000 image stacks at 40 second intervals, change the interval to 8 minutes and acquire one 1000 image stacks at 63x and another at 1000 image stacks at 20x as described in step 31. Completing this step will require in excess of 6 days if fully completed.
33. The number of image stacks obtained in step 34 may be altered by the Science team.
34. Once the nominal (lighted) imaging has been completed, take 100 images at the level of the original plane (50 microns below the top inner surface of the capillary) without external illumination utilizing the previous frame rate and exposure setting.
35. Ask to the ACE-T10 Science team how to proceed: Repeat the procedure with a new Texp (CASE A ) or with the next highest priority sample (CASE B).
36.
    a. CASE A: Repeat the process (steps 23 – 37) with a new Texp decided by the Science Team (Typically New Texp = Previous Texp + 2°C). If the sample material in the ROI should become bleached, switch to the secondary location selected in step 5, based on input from the Science. If necessary repeat steps 1-6 to locate new ROIs.
    b. CASE B: Repeat the whole process (steps 1- 37) for the next highest priority sample.

# Importance and reason for ISS

- Colloidal stability is critical to soft matter systems, as it relates to products (see P&G ACE- T experiments);
- These structures change in time, by processes know as coarsening - meaning the particle move under thermal motion to compact. This compaction continually changes this structure;
- When the structures created in the products can longer support the gravitational stresses (*e.g.* buoyancy) on the structures, they collapse. The collapse is often abrupt and without warning (delayed collapse). This is the essence of product instability;
- For some systems the gel collapse can be anticipated by precursors, which detection can potentially allow us to forecast the gel failure.

# Expected results and how they will advance the field (1/2)

This research will have significant impacts in two areas:

- First, in the scientific community, is not clear how microscopic structure and its dynamics is related to the gel mechanical properties. The structure evolution controls the aging of the gel and, on earth, this eventually lead to collapse. This work will significantly enhance the knowledge necessary to advance this important area of science.

# Expected results and how they will advance the field (2/2)

- Second, relaxation of localized stresses brought in an amorphous solid by a rapid solidification process is ubiquitous in nature, occurring from earthquakes, to damage and failure of materials. Besides affecting the rheological properties of the materials such dynamics rules the aging of the thermal systems at rest. The detection of failure precursors, as the ones discovered in our colloidal gel, can potentially allow us to forecast the material failure before catastrophic rupture.

# Earth benefits/spin-off applications

Understanding the behavior of weak gels and their aging can help materials scientists to make consumer product with longer shelf lives.

In addition, because several feature of the failure dynamics of disordered materials are system independent, we are confident that the results obtained using our model colloidal system can be generalized to other materials.
This potentially can help to develop new way to characterize aging material and suggest their replacement before catastrophic failure.

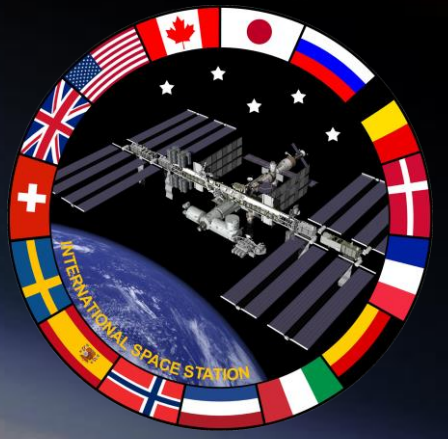
Genoa bridge collapse

National Aeronautics and Space Administration

# ACE-T10

## Increment 59/60 Science Symposium

## BACKUP SLIDES



# ACE-T10 samples

| | Well # | Capillary Cell Contents |
|---|---|---|
| Module 1 | 1 | Water+7% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 500 mM of Ammonium Sulfate |
| | 2 | Water+12% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 500 mM of Ammonium Sulfate |
| | 3 | Water+3.5% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 500 mM of Ammonium Sulfate |
| Module 2 | 4 | Water+1.5% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 240 mM of NaCl + 10% of Triton X100 |
| | 5 | Water+7% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 500 mM of Ammonium Sulfate |
| | 6 | Water+12% of HFIPM particles (80nm in diameter) stabilized with PEGMA2000 surfmer dyed with Rhodamine B + 500 mM of Ammonium Sulfate |

Notes:
HFIPM -> 1,1,1,3,3,3- hexafluoroisopropyl methacrylate
PEGMA2000 -> Poly(ethylene glycol)methyl ether methacrylate ( Mn 2000 Da) with 4-cyano-4-(phenylcarbonothioylthio)-pentanoic acid (CPA) as RAFT agent

# Microgravity Justification

- Formation of colloidal structures is profoundly affected by gravity via sedimentation processes. Chaikin and Russel have already demonstrated this effect in space experiments exploring the simplest of all entropic transitions, the hard-sphere liquid-solid phase transition.

- Sedimentation causes particles to fall so rapidly that there is insufficient time for particles to explore the full phase space of positions and velocities that are required for thermodynamic assembly processes. A substantial particle concentration gradient arises in the earthbound sample.

$$h = \frac{k\,T}{\Delta\rho\, V\, g}$$

h= gravitational height
K T = Thermal Energy of system
$\Delta\rho$ is the density difference between the particles and the background fluid
V is the particle volume
g is the gravitational acceleration

h ranges from a few microns for the case of polystyrene in water to a fraction of a micron for most of the other particles we consider. Our particles are usually of order 1 micron in diameter.

# Microgravity Justification (continued)

- In addition, the shear forces of fluid flow due to the sedimenting particles is often sufficient to break structures that are forming thermodynamically.
- The solvents we plan to use (such as water) are restricted by various factors, for example by our need to fix the colloidal structures in space. Almost all of the particles of future interest are either too heavy or too light compared to water.
- Sample equilibration often requires ~1 to 12 hours. Structure growth sometimes continues for one to two more weeks after the initiation process. These processes are too slow for a drop tower or an airplane.
- Space station or space shuttle provides an environment where microgravity is sustained long enough to allow these experiments to be conducted. The samples can be homogenized, and then allowed to develop in the microgravity environment. Their structures and optical properties can be measured. For most samples we are contemplating, the density mismatch between particle and background fluid is large (*e.g.* > 1.1 x). Microgravity dramatically reduces these differences and permits true equilibrium processes to occur.