Increment 57 / 58 Science Symposium

Advanced Colloids Experiment (Temperature controlled) – ACE-T2

PI: Prof. P. Schall– University of Amsterdam

Presented by:

Dr. William V. Meyer (*a.k.a.* Bill Meyer)

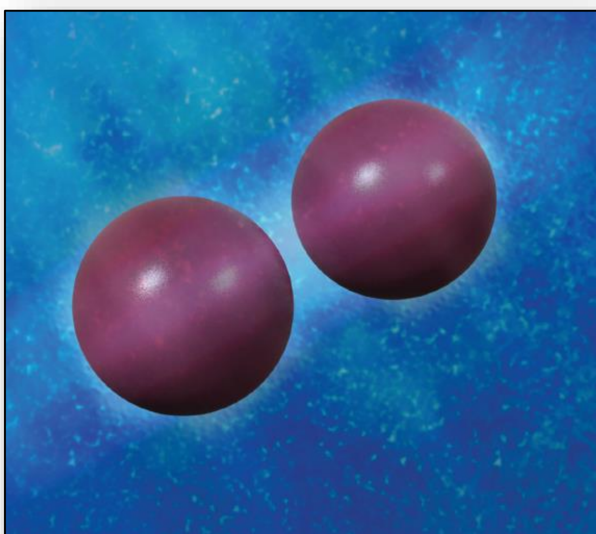
ACE NASA Project Scientist

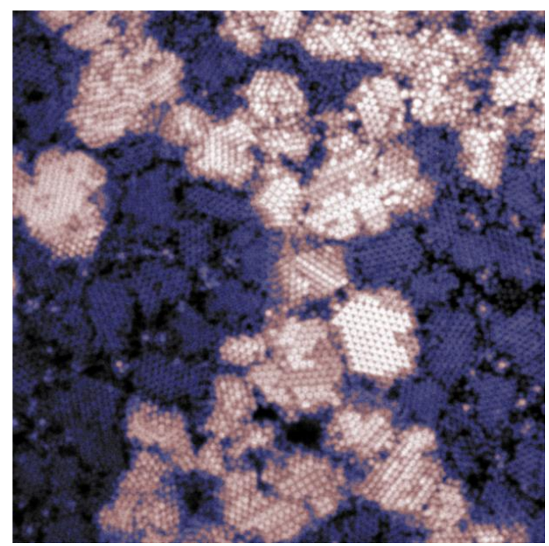
USRA at NASA GRC, Tel: (216) 433-5011, Email: William.V.Meyer@NASA.Gov
ACE NASA Project Manager: Ron Sicker, Tel.: (216) 433-6498
ZIN-Technologies Project Lead: Kelly Bailey, Tel: (440) 625-2251
ZIN-Technologies Science Lead: John Eustace, Tel: (440) 625-2244

May 24, 2018

ASSEMBLY OF COLLOIDAL SUPERSTRUCTURES BY CRITICAL CASIMIR FORCES IN MICROGRAVITY

Piet Swinkels, Gerard Wegdam, Peter Schall
– University of Amsterdam –





Science Team

Amsterdam

Piet Swinkels

Simon Stuij

Prof. G. Wegdam

Prof. P. Schall

Milan

Dr. M. Potenza

ACE-T2 Science Team Contact Information

Peter Schall
Professor Soft Condensed Matter Physics
Master Coordinator Advanced Matter and Energy Physics
Van der Waals-Zeeman Institute
University of Amsterdam

Postal address:
P.O.box 94485
1090 GL Amsterdam, The Netherlands

Visiting address:
Science Park 904, 4th floor
1098 XH Amsterdam, The Netherlands

Tel. +31-20-525-6314
Fax. +31-20-525-5788
Email: Peter Schall (pschall@science.uva.nl)
Email: Peter Schall <ps@peterschall.de>
Web: https://staff.fnwi.uva.nl/p.schall/
https://iop.fnwi.uva.nl/scm/

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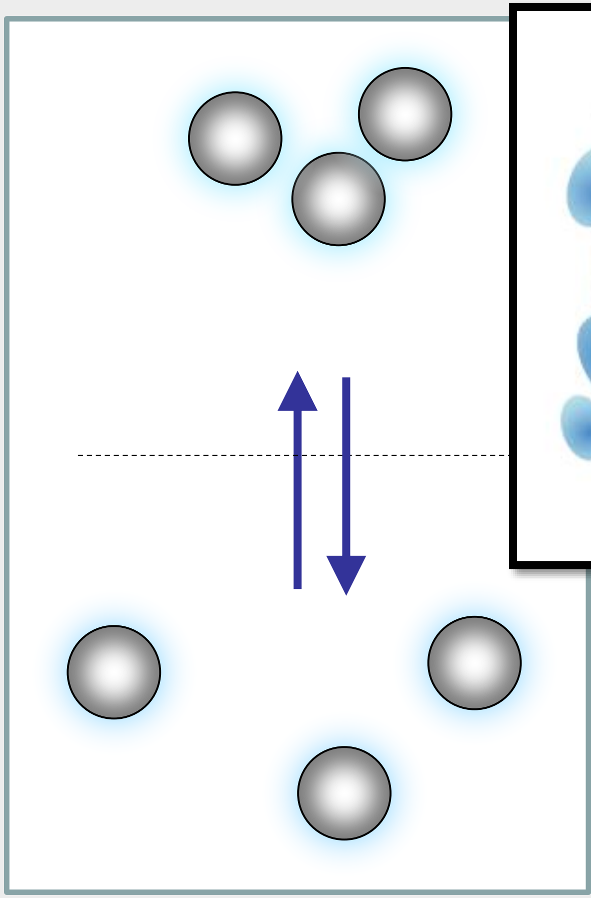
Advanced Colloids Experiment (Temperature controlled) – ACE-T2

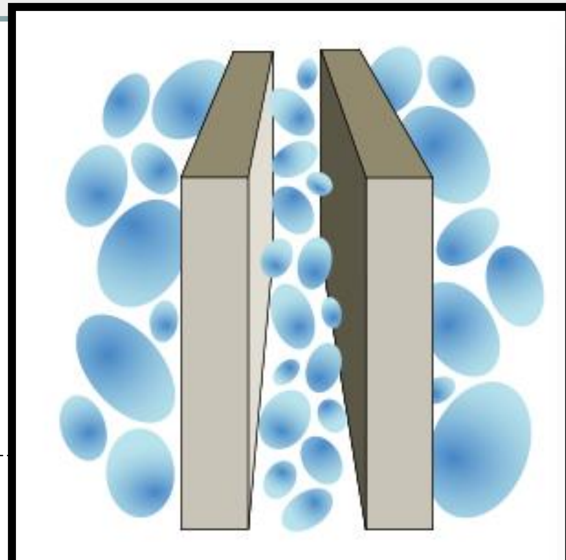
Prof. P. Schall– University of Amsterdam

- 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 Hypothesis – 1/5

The Critical Casimir effect

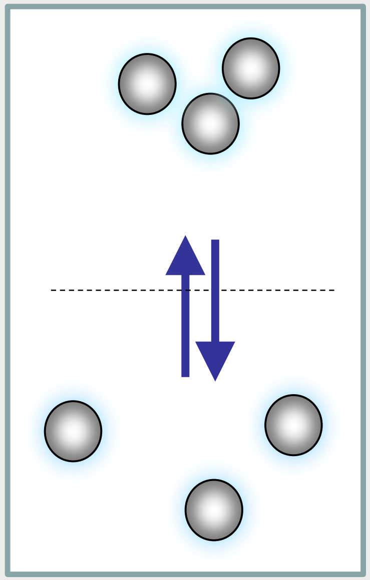


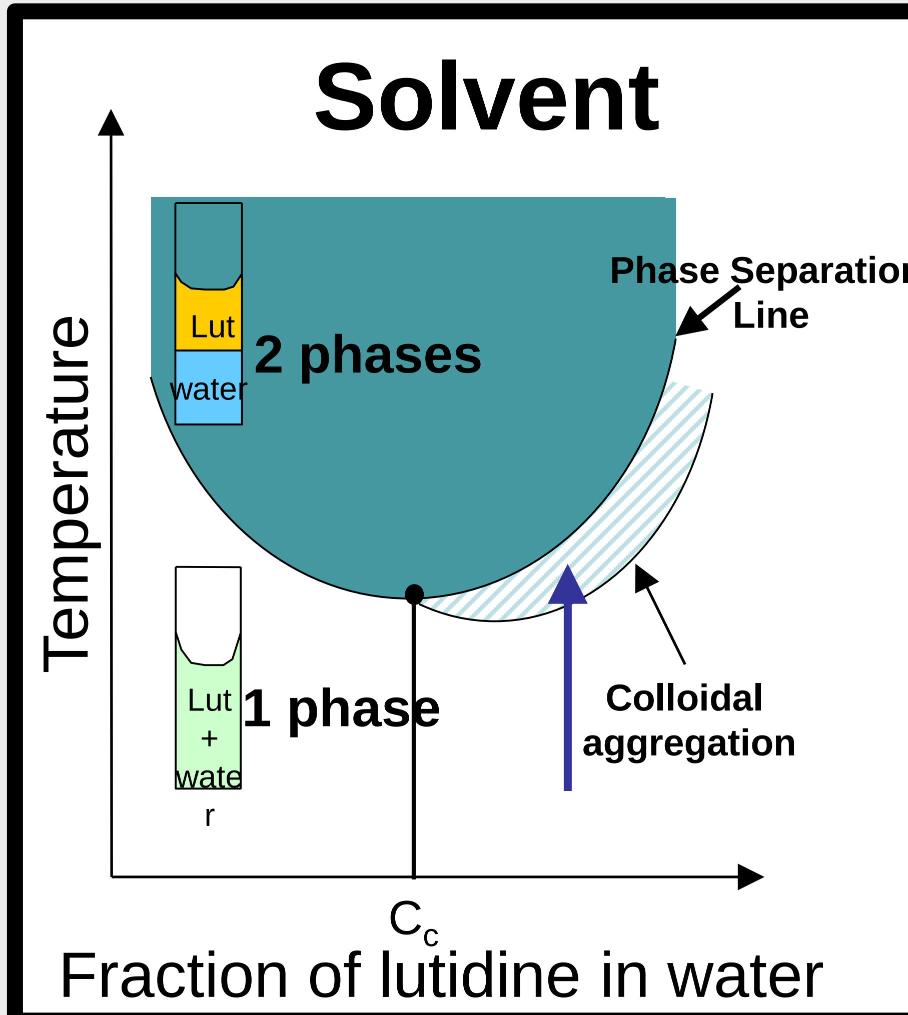


Science Background and Hypothesis – 2/5

The Critical Casimir effect

Concentration fluctuations will become larger as you increase the temperature towards the phase separation line. As the fluctuations grow bigger, the attraction will become stronger and longer ranged and colloids will start to aggregate. See “Notes page” – below.





Science Background and Hypothesis – 3/5

Particle interactions

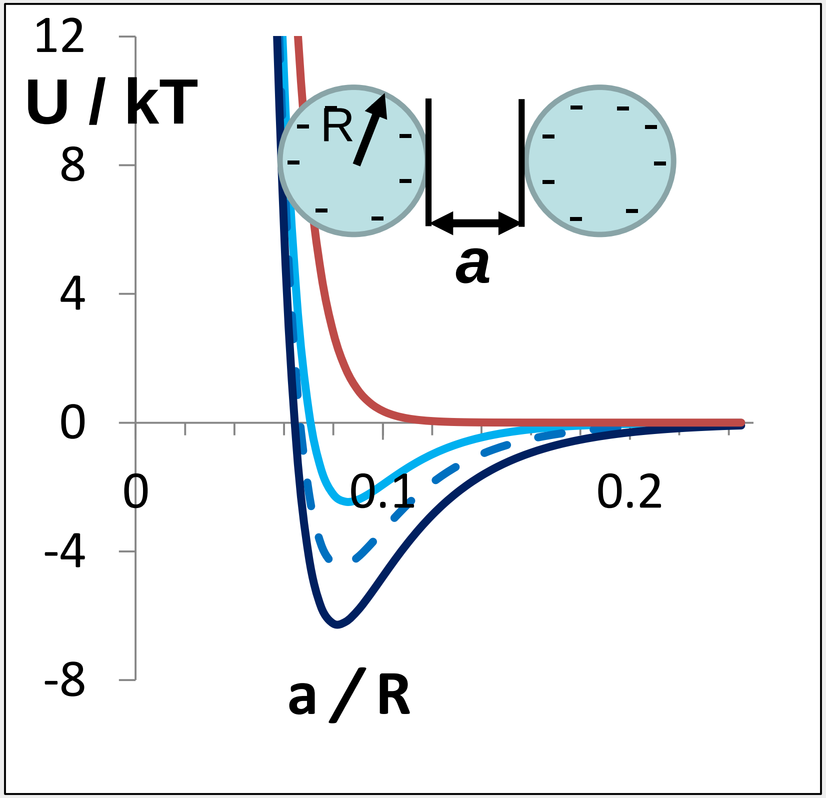
Electrostatic repulsion

$$V_{el}(a) \propto \lambda_d^2 e^{-a/\lambda_d}$$

+

Critical Casimir attraction

$$V_{casimir}(a) \propto -\frac{1}{\xi} e^{-a/\xi}$$

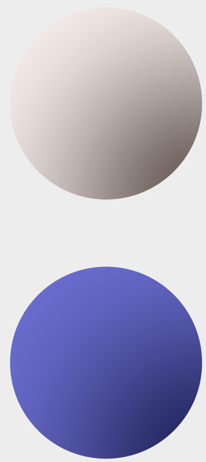


where ξ is the bulk correlation length of the solvent

Schall et al., PRL **103**, 156101 (2009)

Science Background and Hypothesis – 4/5

Tuned Interactions: A/B particle mixtures



Particle A

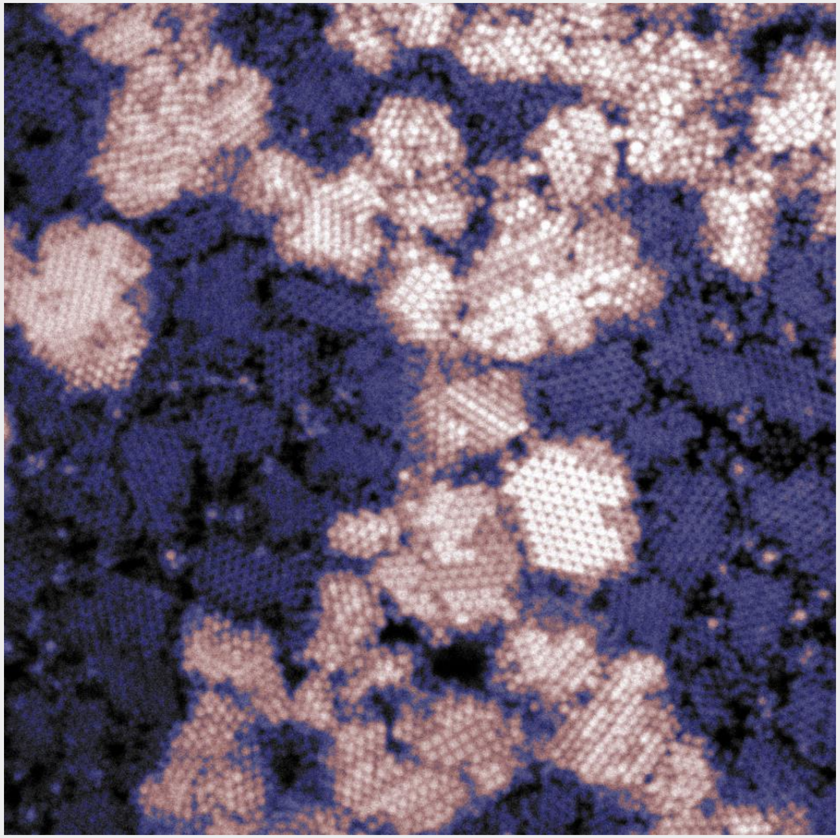
Particle B

Interactions

$u_{AA} > u_{AB} > u_{BB}$

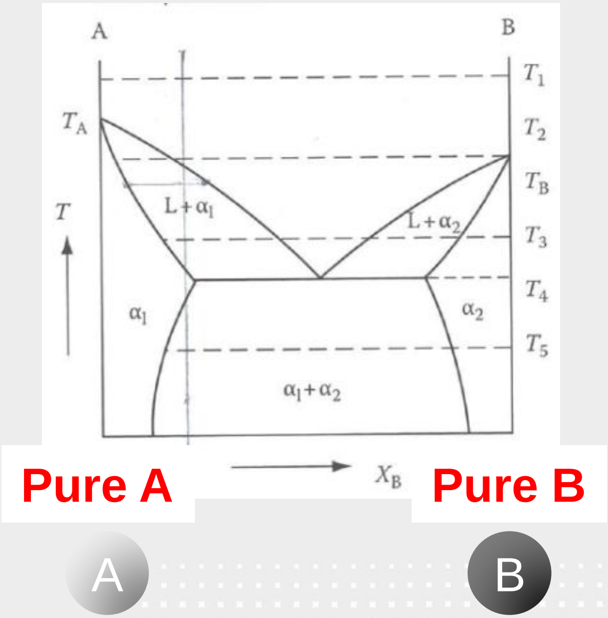
Science Background and Hypothesis – 5/5

Tuned Interactions: A/B particle mixtures



Model intermetallic phases,

e.g. Eutectic mixtures



Investigation goals and objectives – 1/3

Study Critical Casimir assembly
Use temperature control

→ Tune attractions
→ Follow structure formation
→ "Complex crystallisation kinetics"

Vary Temperature → Vary attraction strength

Vary Rate of change → Eqilibrium vs. out-of-equilibrium

Reverse Temperature → Repeat Experiment

Investigation goals and objectives – 2/3

Investigation of assembly of complex structures from micron-scale colloidal particles interacting via tunable attractive interactions.

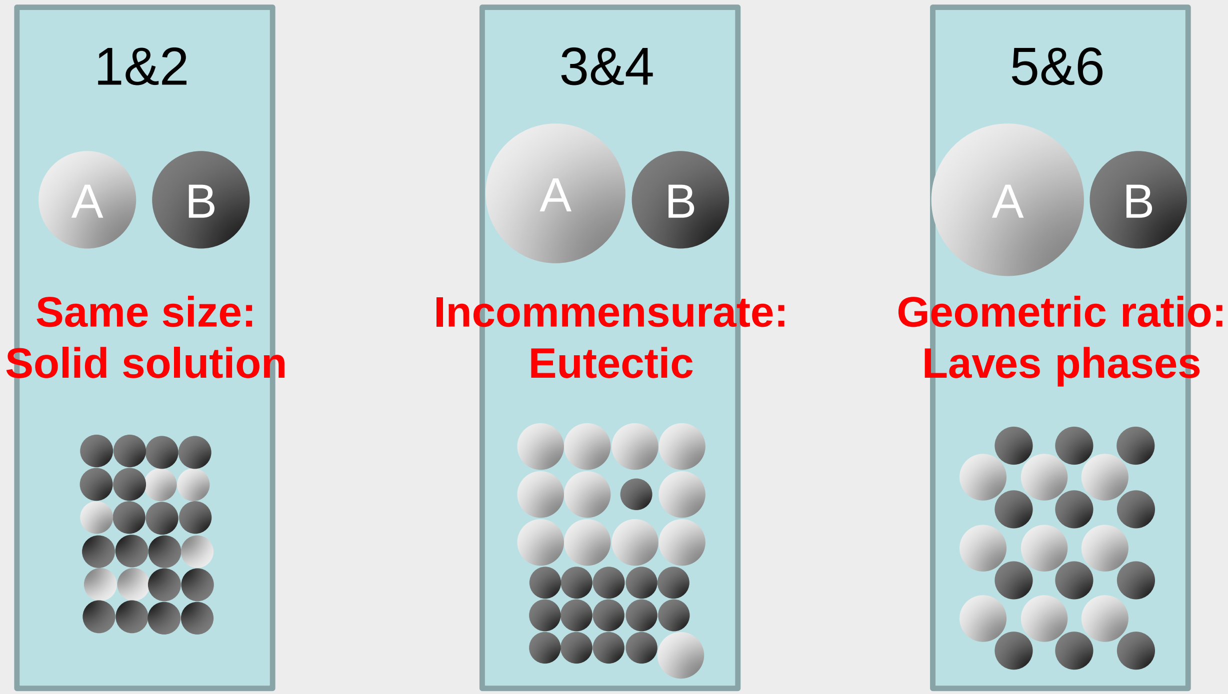
Samples contain suspensions of particle type A and B (trifluoroethyl methacrylate, FEMA colloidal particles) in binary solvents of water (H_2O, 68%mass) and lutidine (32%mass), that upon nearing the critical solvent temperature (T_c~32°C) give rise to critical Casimir interactions between the particles.

Regulating temperature enables control of particle A, B interactions.

The goal is to understand how complex interactions lead to complex structures, and to understand the dynamics of growth of these structures.

Investigation goals and objectives – 3/3

Proposed Experiment - Samples



- Particles in solvents of Lutidine and water + sugar for index match
- Particle volume fraction ~ 10%
- Small amount of salt (NaCl, 2mM)
- Sample details provided in back-up slides

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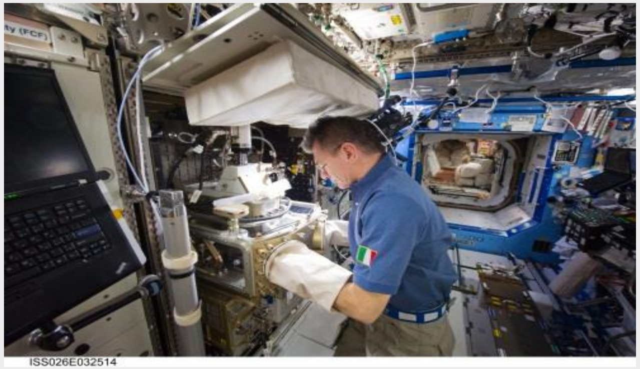
Measurement approach – 1/5

Particle A, B mixtures have been recently investigated by us on ground. Suspended in binary solvent, at temperatures around 28-30°C, they show complex liquid and crystal phases, reminding of complex intermetallic phases.

Structures depend on the particle number and size ratio, as well as on temperature setting their respective attractive strength u_{AA}, u_{AB}, and u_{BB}.

These structures and their formation kinetics will be directly followed with the LMM microscope on the ISS.



Using the Light Microscopy Module (microgravity microscope) aboard the International Space Station (ISS).

Measurement approach – 2/5

Operational Requirements:

Overall, one experiment (capillary cell) is conducted every two days, depending on the number of *z*-layers and speed of growth of the structures. The experiment is repeated until all capillaries are tested. Sample modules are switched if air bubbles are too big. Use the rest of the week to analyze data, re-write scripts, and adjust parameters. The number of capillaries per experiment is limited by data bottlenecks on IPSU (internet protocol setup utilities) and IOP (input/output operations per second). We will need to check *XYZ* position repeatability and oil availability. More specifically, we will need to return to the same Region of Interest (ROI), *e.g.*, single particle crystallite – or to maintain *XYZ* coordinates during an experiment. This requirement implies maintaining one capillary posiStion. If this requirement takes an excessive amount of time, we will need to find a solution.

Operational Protocols: Descriptive overview of the investigation on orbit procedures. Experiment Steps:

The general experimental protocol includes imaging the dynamics of colloidal assemblies in each capillary. We will inspect all samples before mixing the colloid using the *in situ* mixer. Next, define *XY* offsets. Locate and image particles, adjusting the camera parameters using the 2.5x objective and Texas red (or FITC) filter (fluorophore = Cy3MM, Harvard Fluorophore, Cy3-like). Survey capillary at 2.5x, scanning in the *X* direction over a range of at least 10 millimeters. Determine bubble locations and possible primary / secondary Regions of Interest (ROI). If the 2.5x objective is difficult to switch in and out with the 63x air objective, then find ROI capillary cells before using 100x oil objective. If both ends of the capillary can be used (no big air bubble), then apply immersion oil to half of the capillary to allow imaging with 100x oil *and* 63x air objective without astronaut intervention. Select primary locations away from stir bar or bubble.

[Continued]

Measurement approach – 3/5

Operational Protocols [Continued]:

A. Determine aggregation and solvent phase separation temperature

Use low magnification lens to determine particle aggregation temperature, T_{AA}, and solvent phase transition temperature T_c. To do so, survey and record best Z-depth at each primary test location. Ramp temperature slowly from ~27°C to ~31°C during 4h (ramp rate 1°C/hour) while recording microscope video. Note aggregation temperature T_a=T_{AA} when particle aggregates start to appear, and phase separation temperature T_c, where the solvent separates into two components.

B. Confocal video, time series

Go back to room temperature, and stir for 30min. After that, switch in 63x air objective. Increase temperature to T_a-0.3°C. Record microscope video (confocal imaging), while slowly ramping temperature to T_a+0.3°C during 6 hours (ramp rate 0.1°C/hour). These direct particle-scale observations should allow determining the colloidal liquid condensation and crystallization temperatures T_{liq} and T_{cryst}. If possible, record confocal z-stacks of the characteristic liquid phase (at T>T_{liq}) and crystal phase (at T>T_{cryst}). Each z-stack should consist of ~200-300 images, 0.1μm apart, starting from ~10-20μm into the capillary (above the glass wall). The *Z*-scanning rate should be set as fast as possible, potentially 10 frames per second. No pixel binning, 8 bits per pixel (highest supported), full frame images. After z-stack is taken, continue recording microscope video (at same location and depth as before)

[Continued]

Measurement approach – 4/5

Operational Protocols [Continued]:

C. Confocal stacks

Go back to room temperature, and stir for 30min. Use 63x air objective. After aggregation temperatures have been refined in (B.), go to T_{liq} and keep the temperature constant. Record confocal stack time series (each z-stack consisting of ~200-300 images, 0.1µm apart, starting from ~10-20µm into the capillary, taken with high scan rate). Record 1 z-stack per 2 min, in total 50 z-stacks over 100 minutes. After this time series, when colloidal liquid has formed, go to T_{cryst} and repeat the same z-stack time series, again taking one z-stack every ~2min, 50 –stacks total over 100min.

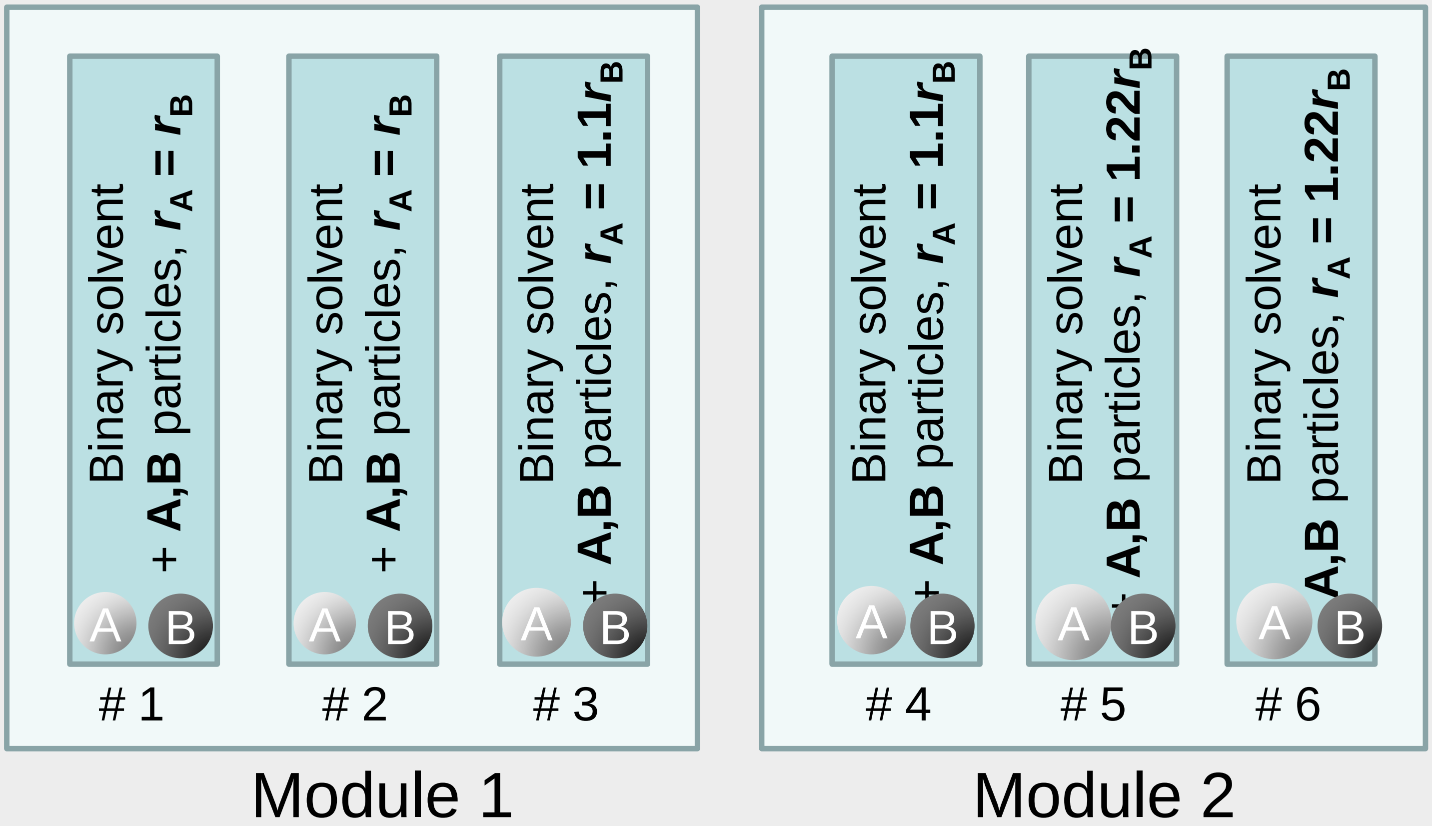
D. Imaging with 100x oil

For better image quality, imaging with 100x oil objective is preferred. However, this will introduce a heat gradient as heat is sucked away from the imaging location into the objective. The heat gradient may also induce particle flows, which are detrimental for the study of particle assembly. To find T_{liq} and T_{cryst} with the 100x air objective, start from T_a and repeat ramping procedure in (A), ramping to ~31°C during 4h (ramp rate 1°C/hour). After aggregation temperatures have been found, refine with a slower ramp rate 0.1°C/hour. Cool down and stir for 30min. Then, go to refined T_{liq} and record confocal z-stack time series as described in (C). After that, go to T_{cryst}, and again record confocal z-stack time series.

Repeat A. – D. for all sample capillaries.

Measurement approach – 5/5

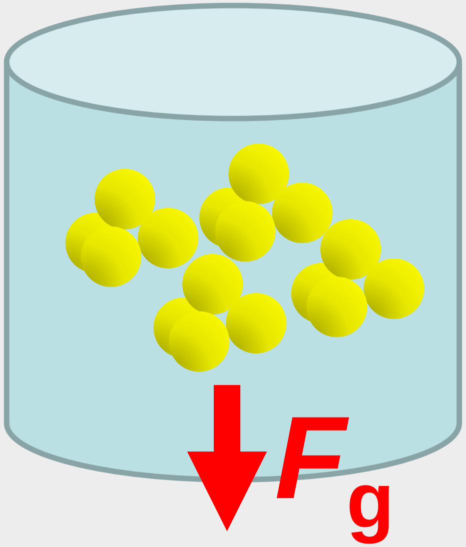
Pictorial summary



FEMA particles (trifluoroethyl methacrylate) in water (68%wt) and lutidine (32%wt)

Importance and reason for ISS – 1/2

1. Growth of large structures



$$F_g = \Delta\rho \, g \, V$$

where:

F_g [force of gravity]

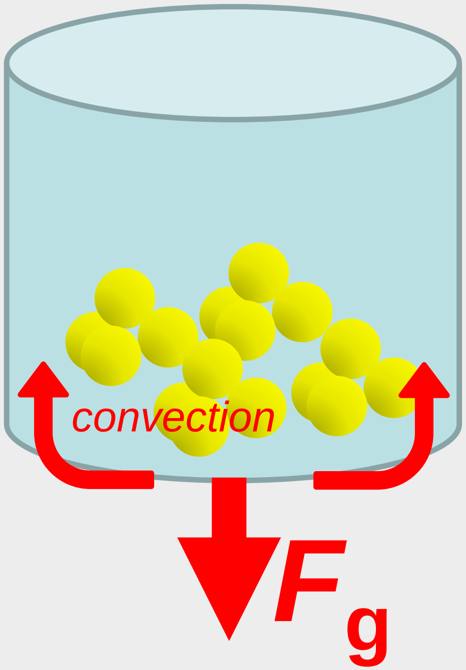
$\Delta\rho = \rho_{Colloid} - \rho_{Solvent}$ [density]

g [gravity]

V [Volume]

Importance and reason for ISS – 2/2

2. Temperature is the control parameter



- Change Temperature
  $\Delta\rho$ changes
- Temperature → Convection disrupts growth

These structures and the growth process are strongly affected by sedimentation and convection. The goal of the ACE-T-2 experiment is to study the diffusion-limited growth of these complex structures without the disturbing effects of gravity.

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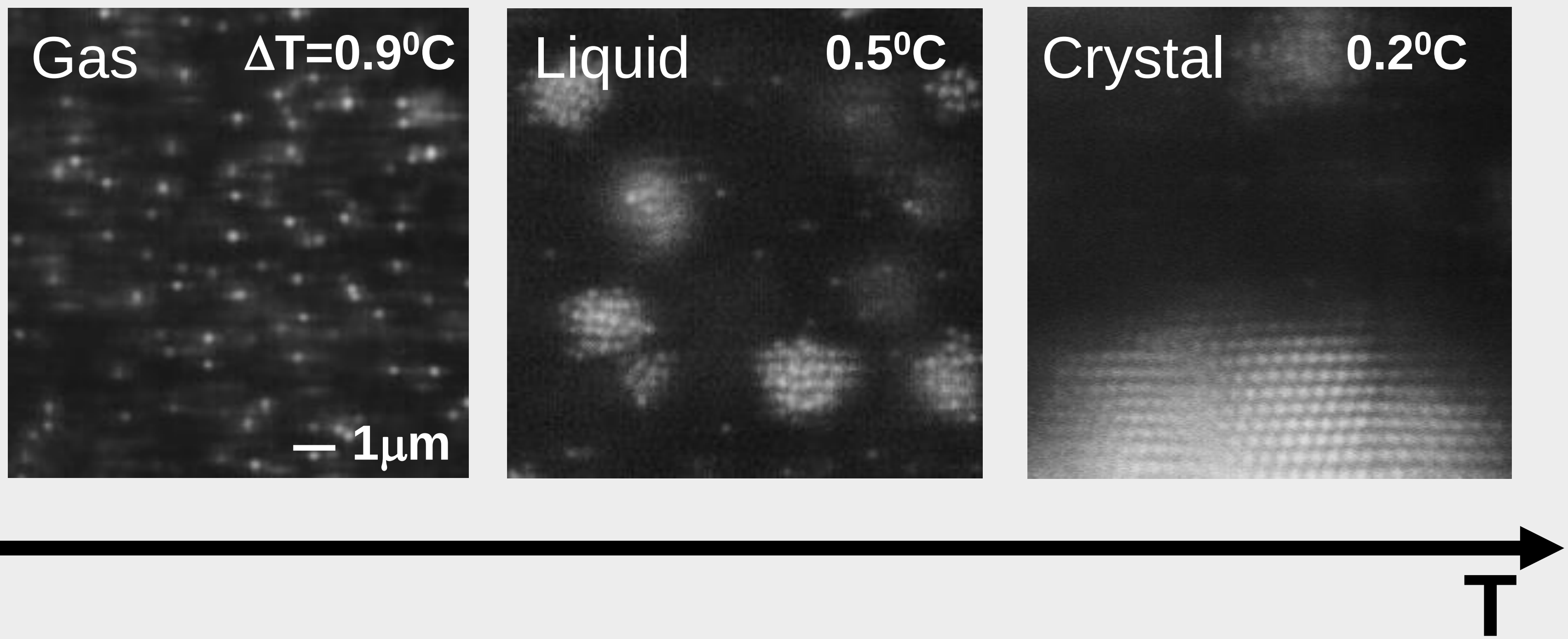
Expected results and how they will advance the field

1-Component system



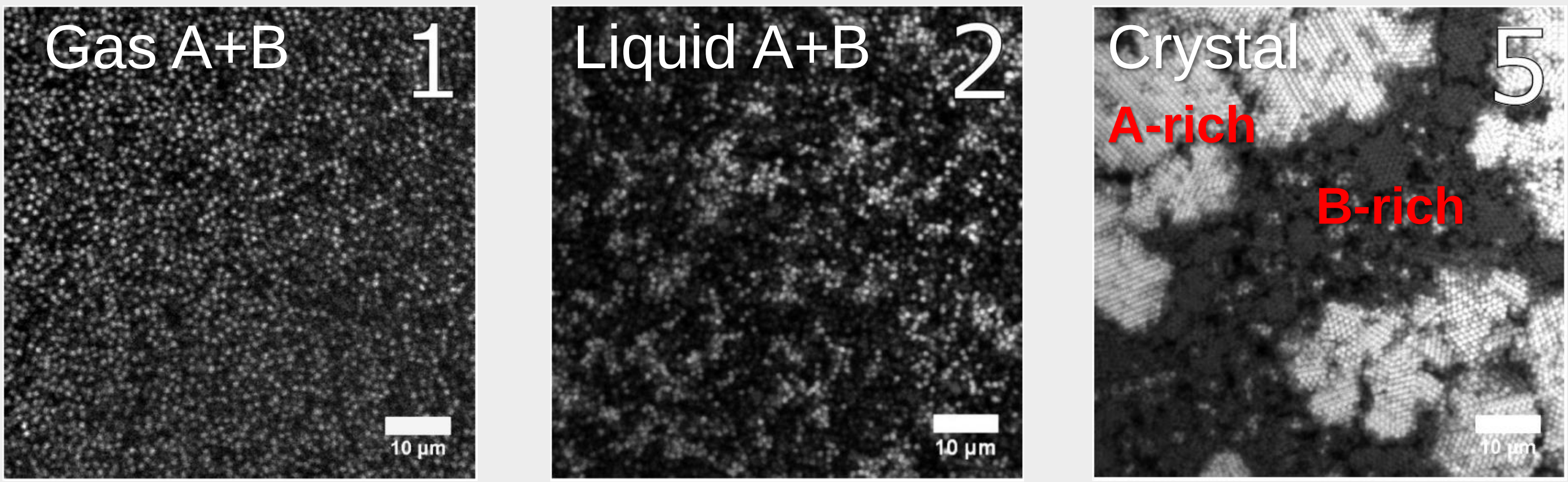
Microscopy: Ground-based work

Expected results and how they will advance the field

2-Component system

Particle A

Particle B



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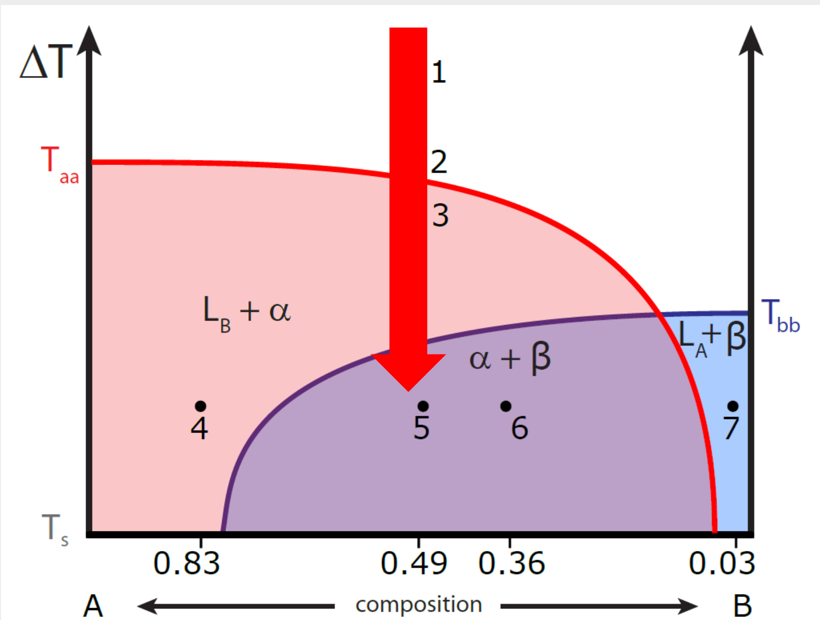
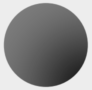
Microscopy: Ground-based work

Phase diagram and behavior



Pure A

Pure B



Liquid phases

L_A: liquid, A-rich

L_B: liquid B-rich

Crystal phases

α: crystal, A-rich

β: crystal, B-rich

Crystallization temperatures

T_{aa} : crystallizaton of A

T_{bb} : crystallizaton of B

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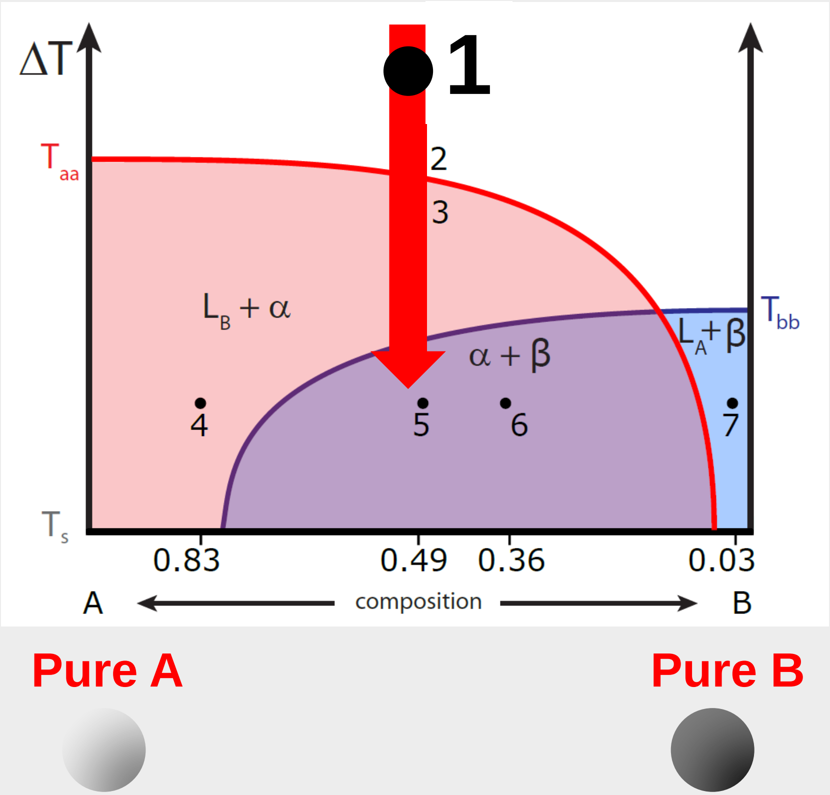
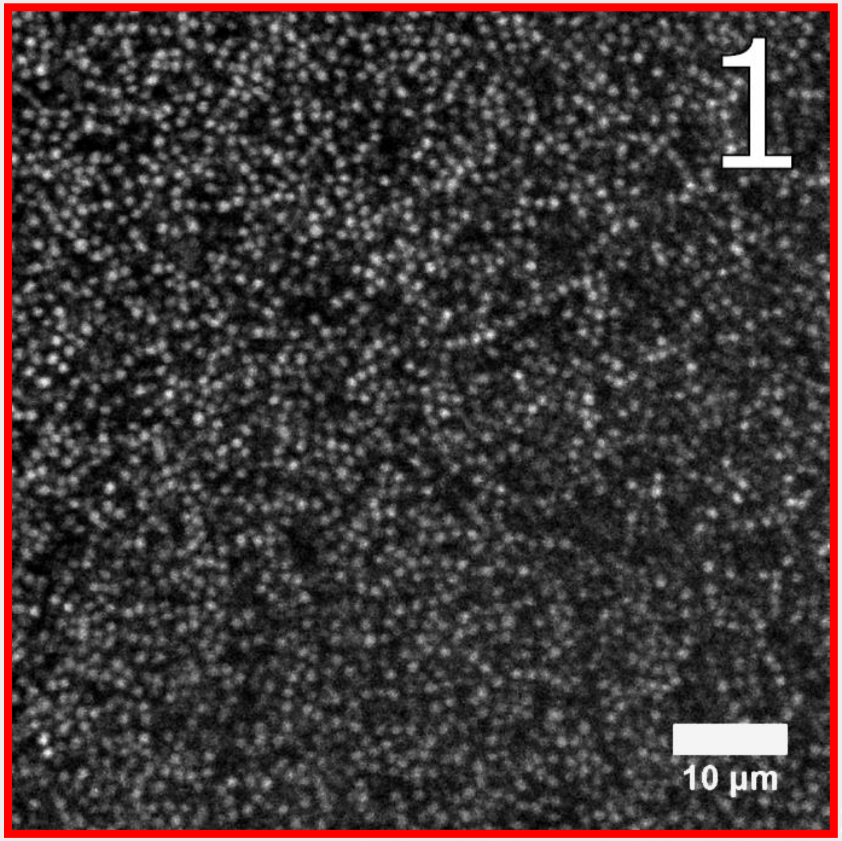
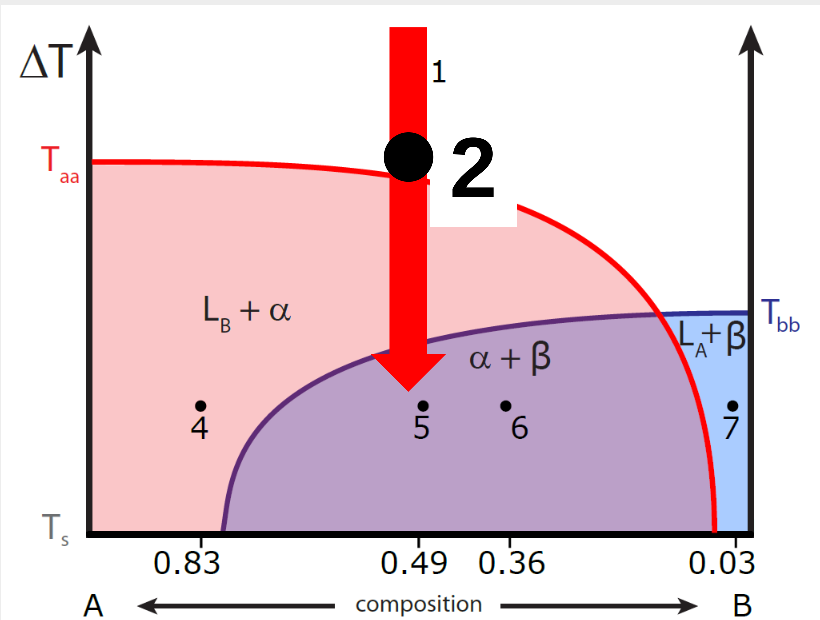
Phase diagram and behavior



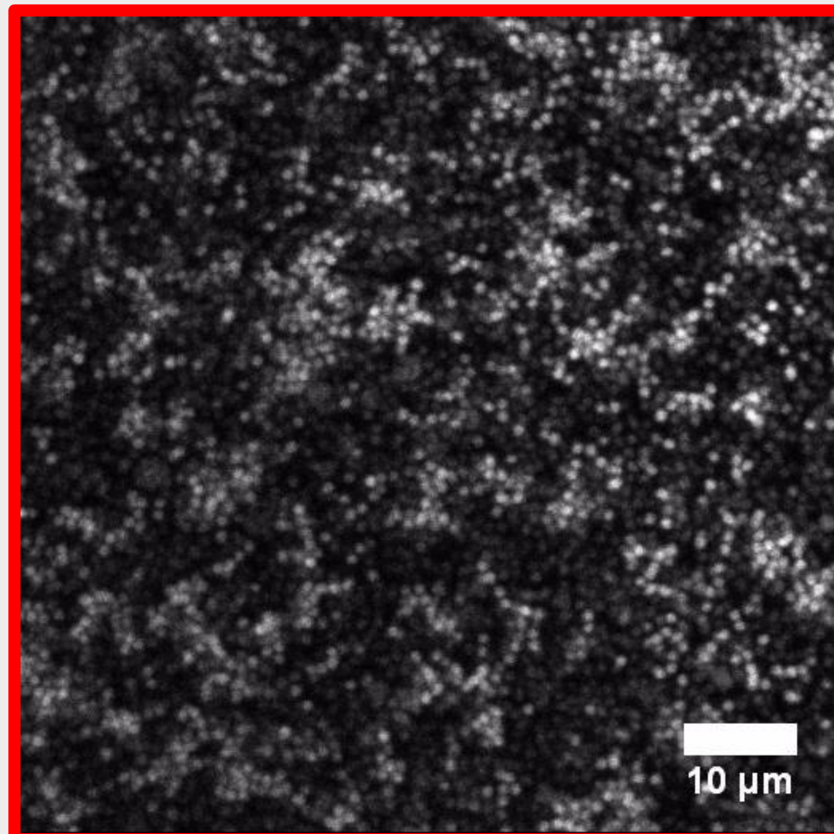


Phase diagram and behavior

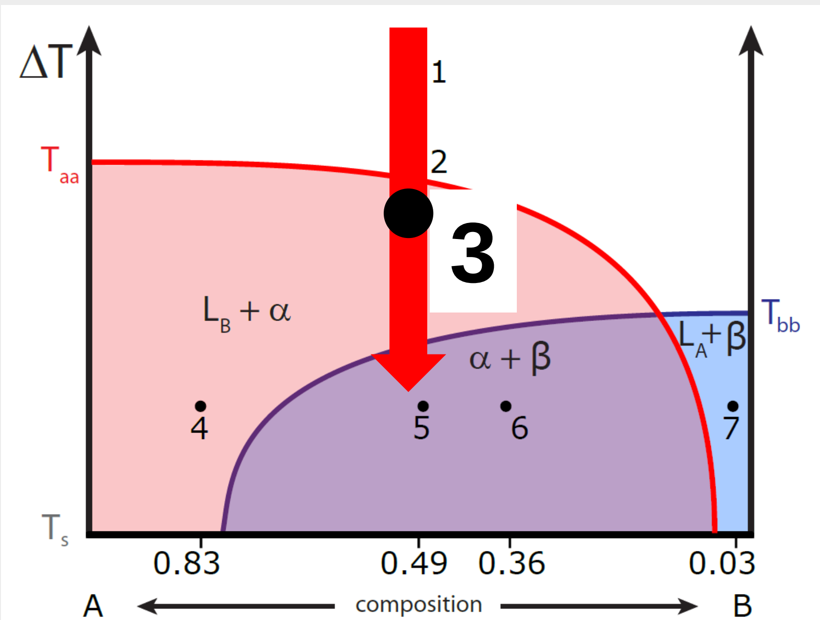


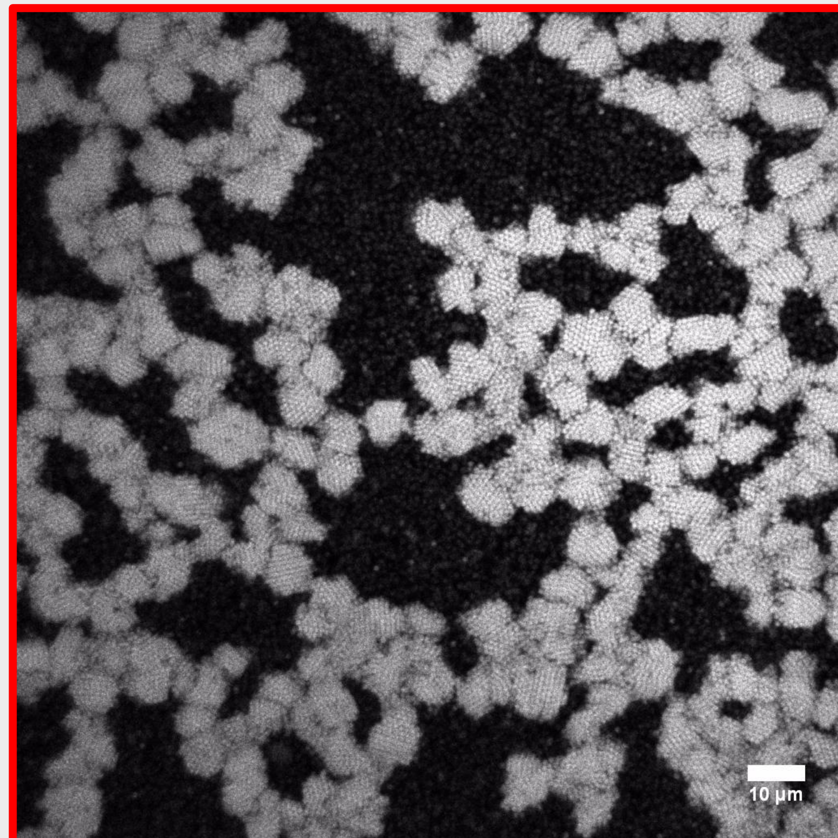
Pure A

Pure B



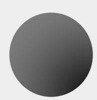
Phase diagram and behavior





Pure A

Pure B



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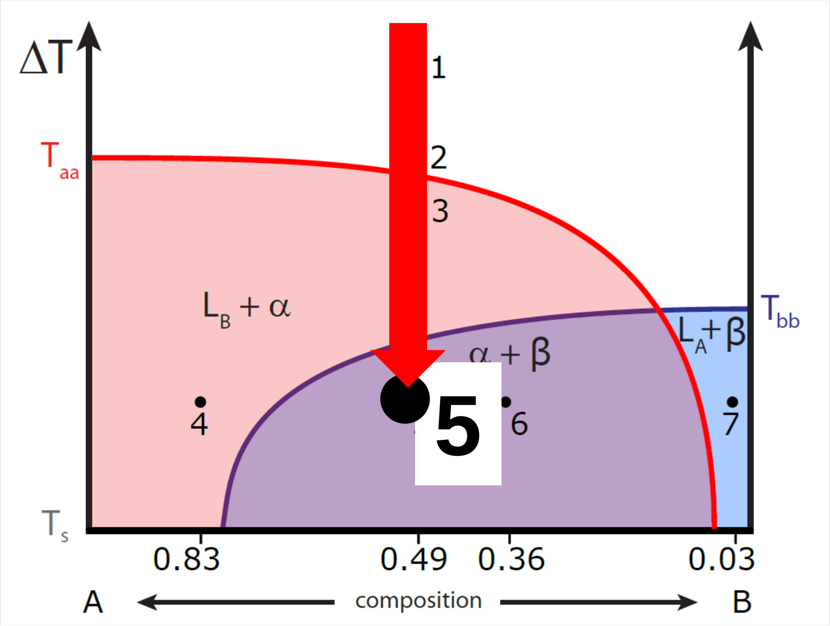
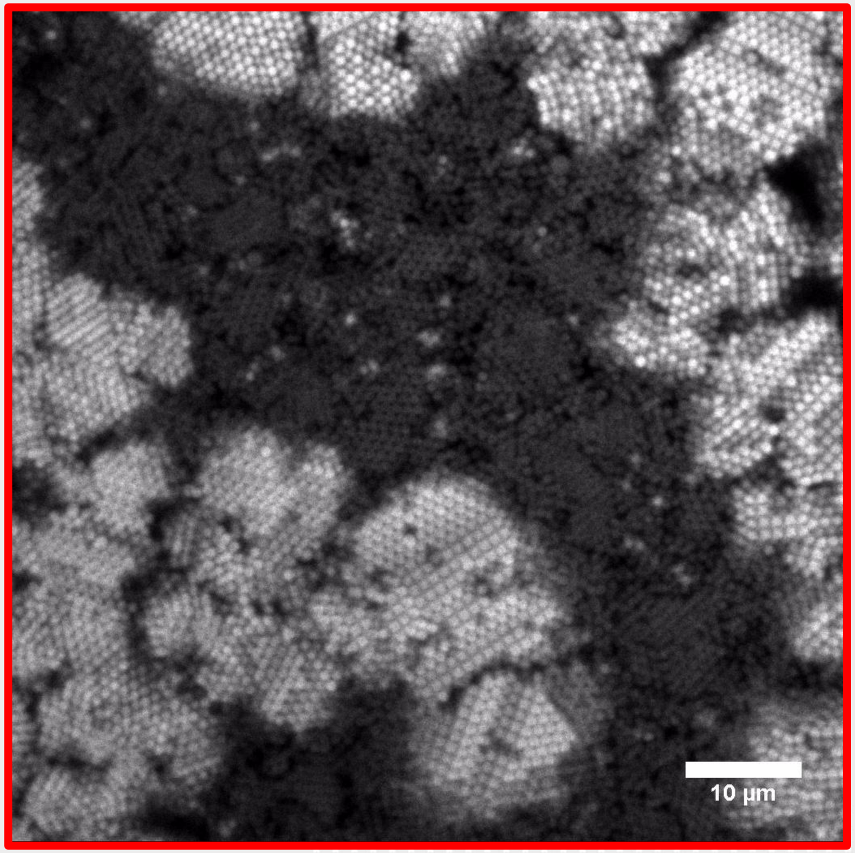
Phase diagram and behavior



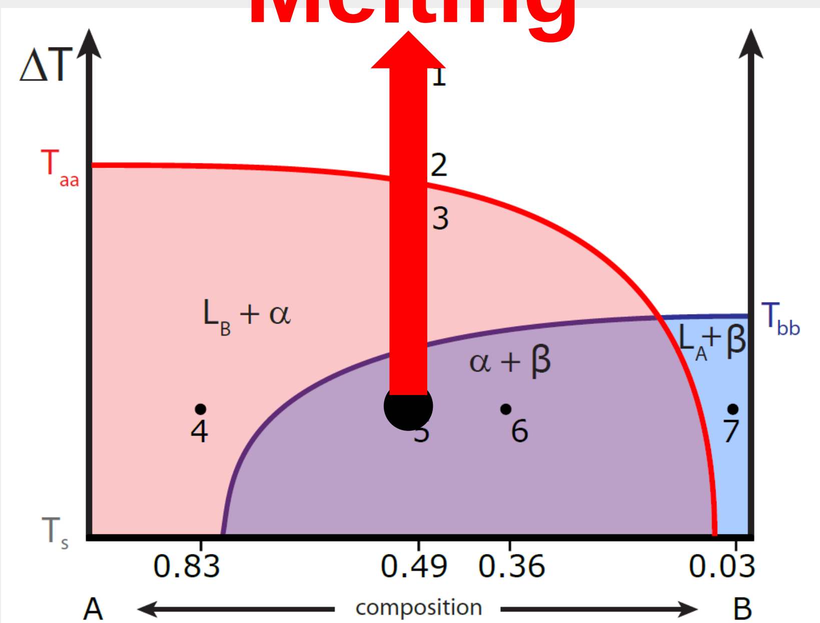
Pure A

Pure B



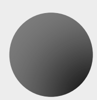
Phase diagram and behavior

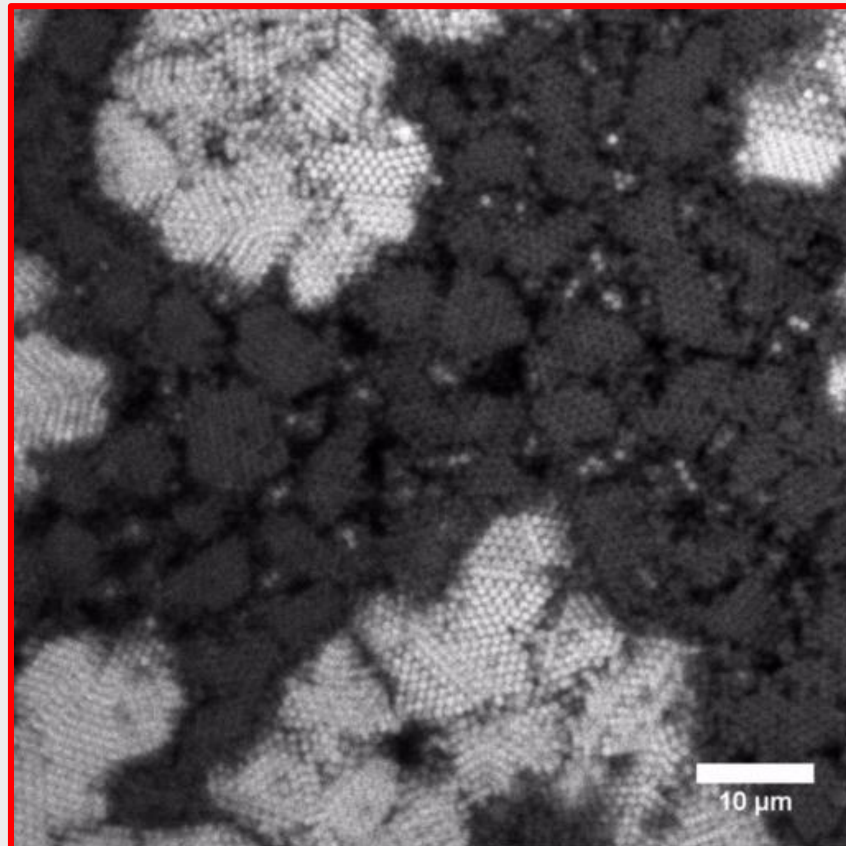
Melting



Pure A

Pure B





Expected results and how they will advance the field

Space-based (microgravity) work – 3D

- What do we expect to see?
  - Observation of *temperature - controlled assembly*
  - Structures as function of A/B mix and quench rate
  - Dynamics of "eutectic" crystal formation

Earth benefits / spin-off applications

Understanding self-assembly processes will teach ways to grow complex nanostructured materials. Furthermore, these experiments will provide fundamental insight into the nature of self-assembly, the relation between complex interactions and complex equilibrium and non-equilibrium structures, and the dynamics of both equilibrium and non-equilibrium growth processes.

Collaborators

- Tom Kodger (Wageningen)
- Peter Bolhuis (Amsterdam, Simulations)
- S. Dietrich (MPI Stuttgart)
- Ania Maciolek (MPI Stuttgart)

The International Space Station (ISS)
Light Microscopy Module (LMM)

ACE-T2 BACKUP SLIDES

Mission Success Criteria for ACE-T2

Complete success is the achievement of all of the science requirements. This means that there will be sufficient information to provide a crosscheck of all data and calculated factors. Processing, manipulation and characterization of the samples in micro-gravity are as important as the measurements during the experiments themselves. *e.g.*, sample homogenization is essential to conduct of any of the flight experiments. This allows for the homogenization of the crystallites or any structures formed from phase separation or gelation that have occurred in 1g before launch, and provides a proper starting point in micro-g.

Success Level	Accomplishment
• Minimum Success	• Homogenize and observe at least 4 of the 6 capillary samples. This includes observing the time evolution with confocal microscopy (using bright field and fluorescent microscopy as needed) imaging for several days to weeks, depending on rates of change determined in real-time as data is downlinked to Earth (these cannot be predicted accurately ahead of time in the 1g environment). • Have sufficient data (both in terms of frequency and duration) from microscopy of sufficient quality to observe, characterize and quantify the rates of growth of structures formed as a result of the physical process of interest in microgravity, including but not limited to self-assembly, the relation between complex interactions and complex equilibrium and non-equilibrium structures, and the dynamics of both equilibrium and non-equilibrium growth processes. • We hope that these processes will generate new structures formed in microgravity, that may direct further earthbound studies and inspire new directions for materials synthesis and fundamental physics understanding.
• • Significant Success	• Accomplish the above for 5 of the 6 different types fluid samples launched.
• Complete Success	• Accomplish the above for all launched samples, with multiple runs to repeat the experiment and assess reproducibility.

ACE-T2 samples

Description: UvA ACE-T2 Flight Sample #1
P/N: UvA_T2_Flight_Sample1
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H_2O, with 40%wt sucrose in the H_2O fraction + 2 mM salt (KCl).
(binary particle mixture A, A' (same radius) with number ratio 20/80%)

Description: UvA ACE-T2 Flight Sample #2
P/N: UvA_T2_Flight_Sample2
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H_2O, with 40%wt sucrose in the H_2O fraction + 2 mM salt (KCl).
(binary particle mixture A, A' (same radius) with number ratio 50/50%)

Description: UvA ACE-T2 Flight Sample #3
P/N: UvA_T2_Flight_Sample3
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H2O, with 40%wt sucrose in the H2O fraction + 2 mM salt (KCl).
(binary particle mixture A, A' (same radius) with number ratio 80/20%)

Description: UvA ACE-T2 Flight Sample #4
P/N: UvA_T2_Flight_Sample4
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H2O, with 40%wt sucrose in the H2O fraction + 2 mM salt (KCl).
(binary particle mixture A, B (R_B=1.2 R_A) with number ratio 20/80%)

Description: UvA ACE-T2 Flight Sample #5
P/N: UvA_T2_Flight_Sample5
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H2O, with 40%wt sucrose in the H2O fraction + 2 mM salt (KCl).
(binary particle mixture A, B (R_B=1.2 R_A) with number ratio 50/50%)

Description: UvA ACE-T2 Flight Sample #6
P/N: UvA_T2_Flight_Sample6
Total volume: 1 ml
Suspension of 10%vol FEMA particles dyed with Cy3MM in a binary solvent of 32% Lutidine and 68% H2O, with 40%wt sucrose in the H2O fraction + 2 mM salt (KCl).
(binary particle mixture A, B (R_B=1.2 R_A) with number ratio 80/20%)