Advanced Colloids Experiment (Temperature controlled) – ACE-T12

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Presented by:

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SCIENCE PRESENTATION

INFLUENCE OF GRAVITY ON ELECTROKINETIC AND ELECTROCHEMICAL COLLOIDAL SELF-ASSEMBLY FOR FUTURE MATERIALS

NNX14AN28A (NASA EPSCoR)

UNIVERSITY OF LOUISVILLE® WKU® UK® UNIVERSITY OF KENTUCKY® NASA

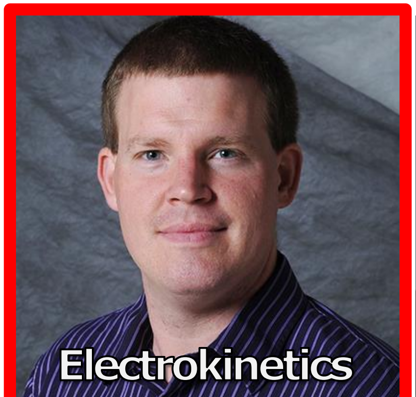
NASA EPSCoR

- **EPSCoR**: Experimental Program to Stimulate Competitive Research

- Establishes partnerships with government, higher education and industry that are designed to effect lasting improvements in a state's or region's research infrastructure, R&D capacity and hence, its national R&D competitiveness.

- The awards enable faculty development and higher education student support.

Science Team



Stuart J. Williams
UL, Science PI

Suzanne Smith
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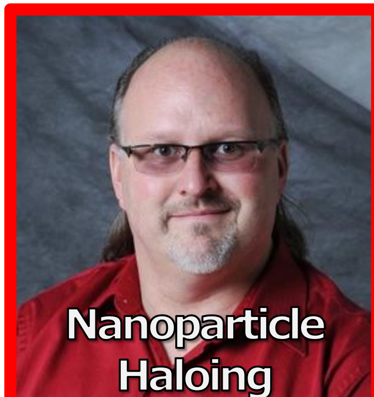
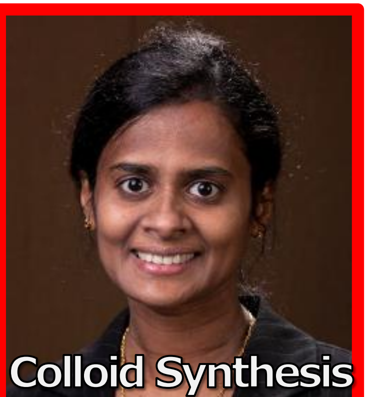
Science Team



Gerold Willing
UL, Co-I

Science Team



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ACE-T-12

- 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 (1/3)

What is Nanoparticle Haloing (NPH)?

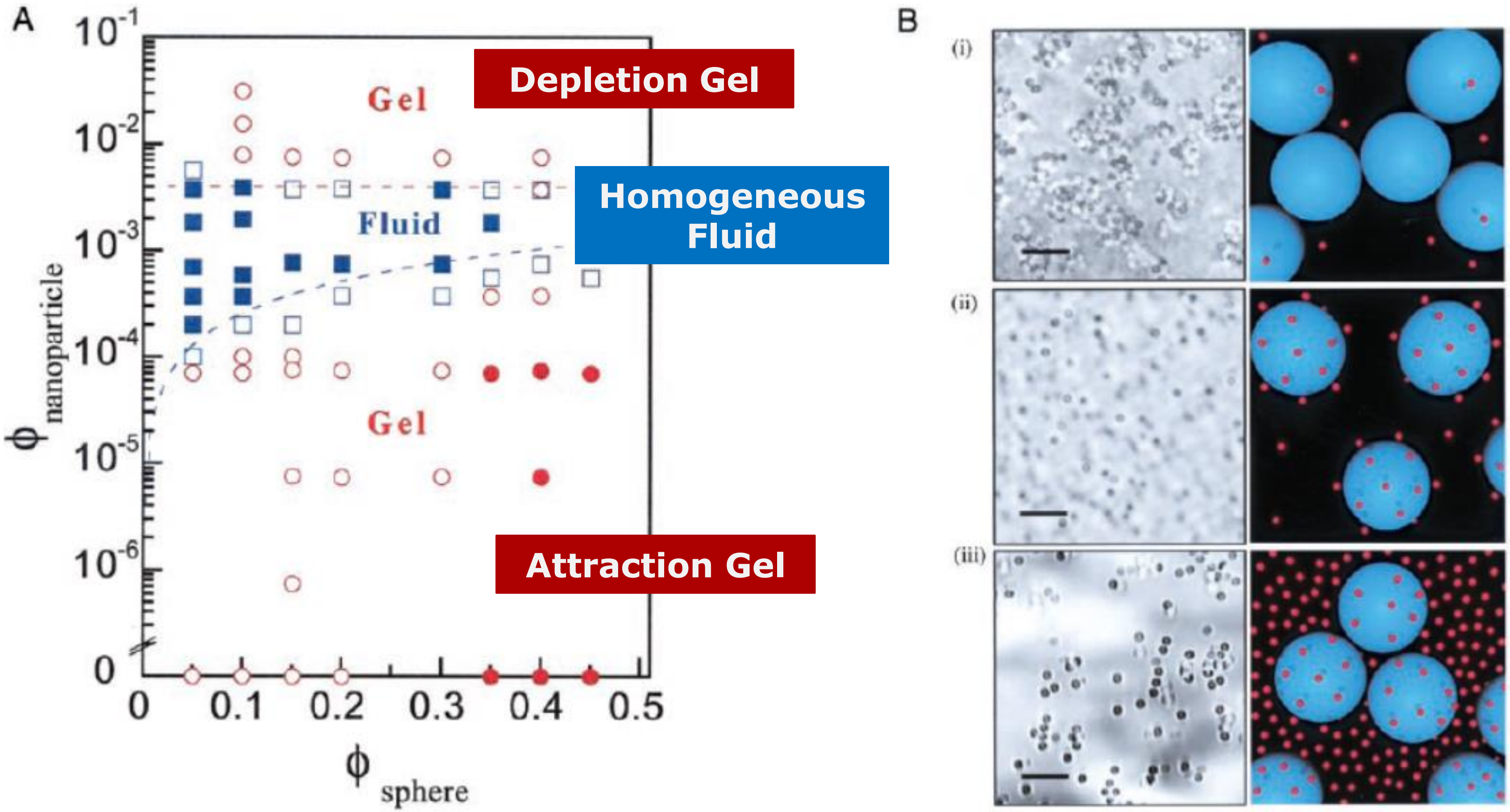
- Originally discovered in 2001 by J.A. Lewis and co-workers of UIUC[1]
- Stabilize negligibly charged Silica suspensions through the addition of highly charged Zirconia nanoparticles
- "Heavy" particles: *gravity settling* experiments
- USAX experiments confirmed nanoparticle distance of about 2 nm from silica surface
- Observed in a number of other systems
  - Silica-Polystyrene[2]
  - Silica-Alumina[3]

[1]Tohver, V.; Smay, J. E.; Braem, A.; Braun, P. V.; Lewis, J. A. Nanoparticle Halos: A New Colloid Stabilization Mechanism. *Proc. Natl. Acad. Sci. U.S.A.* **2001**, 98(16), 8950-8954
[2]Chan, A. T.; Lewis, J. A. Electrostatically Tuned Interactions in Silica Microsphere−Polystyrene Nanoparticle Mixtures. *Langmuir* **2005**, 21, 8576-8679.
[3]Kong, D. Y.; Yang, Y.; Wei, S.; Wang, H. B.; Cheng, B. J. Dispersion Behavior and Stabilization Mechanism of Alumina Powders in Silica Sol. *Mater. Lett.* **2004**, 58, 3503-3508.

Science Background and Hypothesis (2/3)

Nanoparticle Concentration Effects

- Three regimes based on nanoparticle volume fraction



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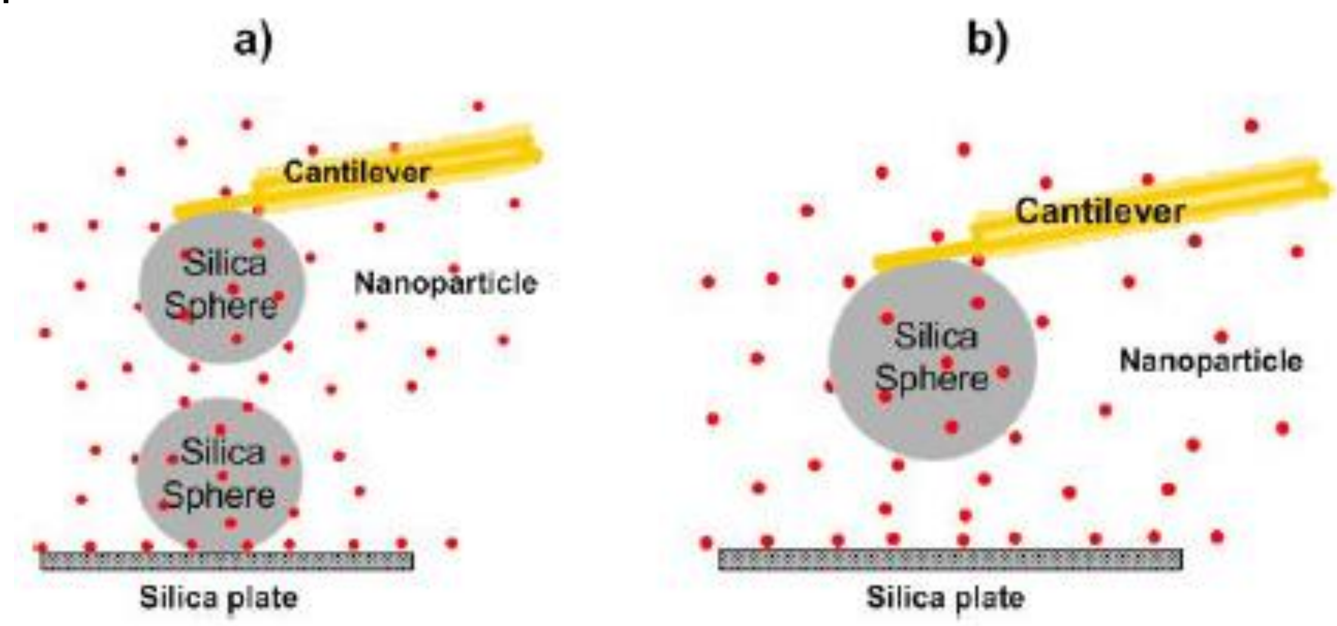
Science Background and Hypothesis (3/3)

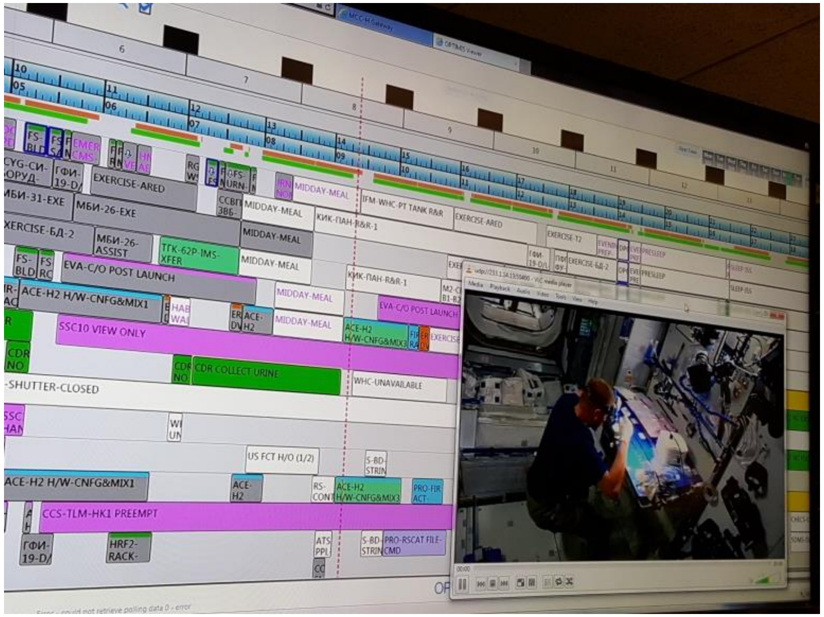
CP-AFM Applied to Nanoparticle Haloing

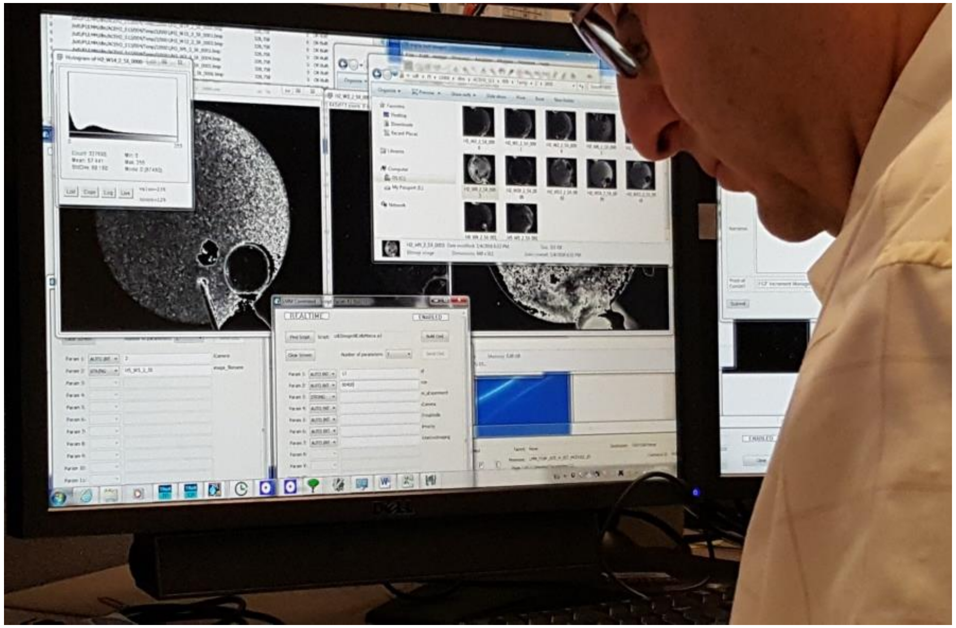
- Can be performed in any fluid environment, including nanoparticle suspensions
- Choice of geometries to study
  - Sphere on Sphere
  - Sphere on Plate



ACE-H2

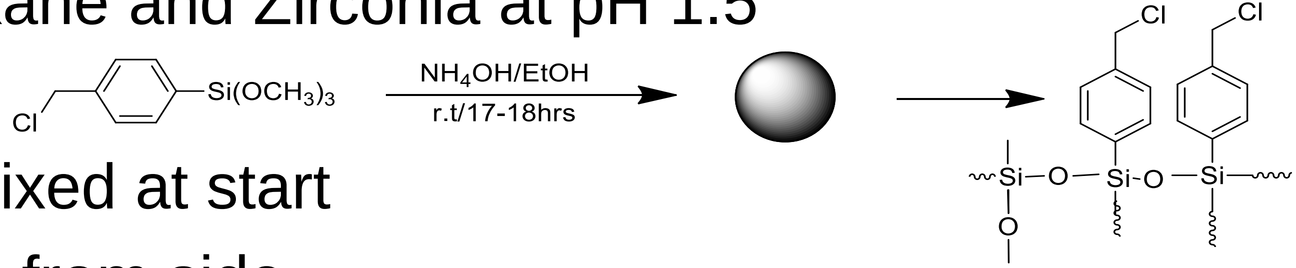
- Orb 4 launched on Dec. 6, 2015
- Experiments started on Jan. 4, 2016



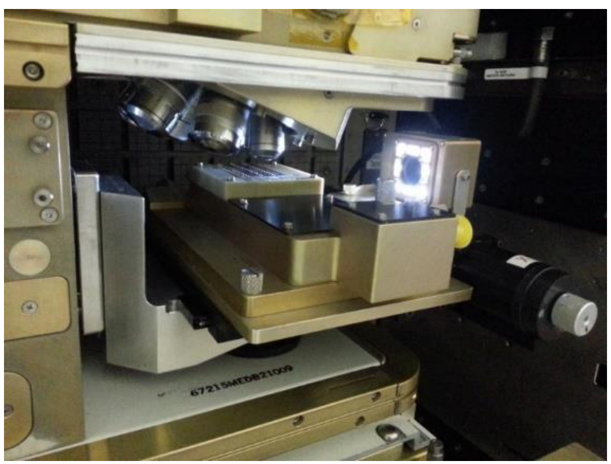


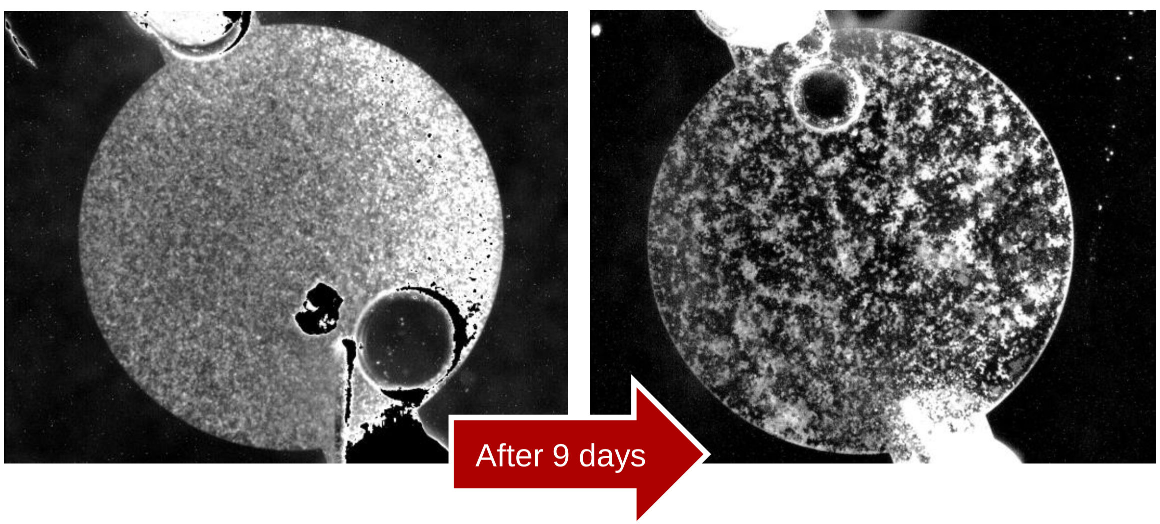
ISS Experiments

- Silsesquioxane and Zirconia at pH 1.5



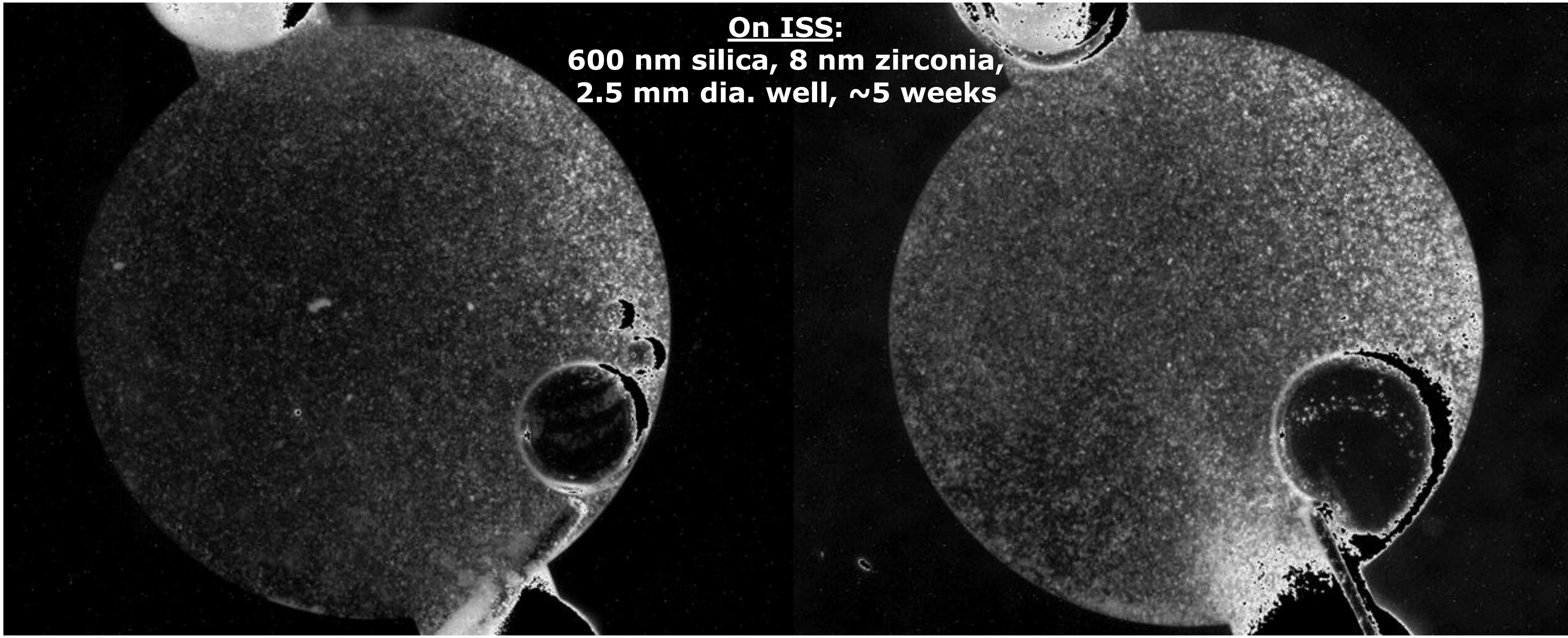
- Samples mixed at start
- Illumination from side
- Rapid agglomeration observed

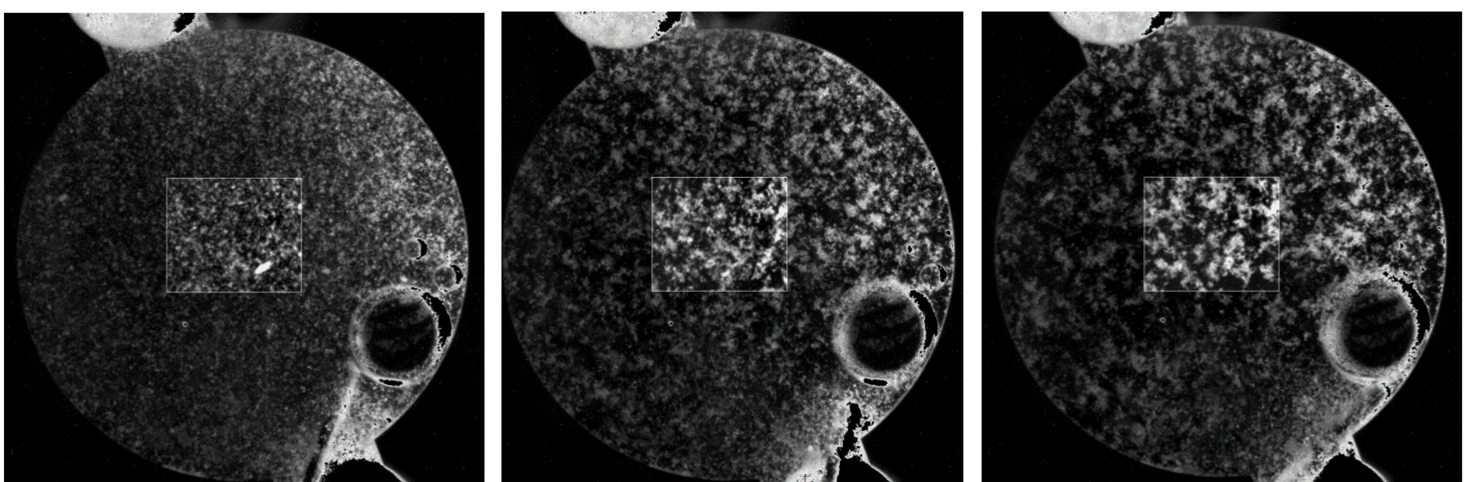


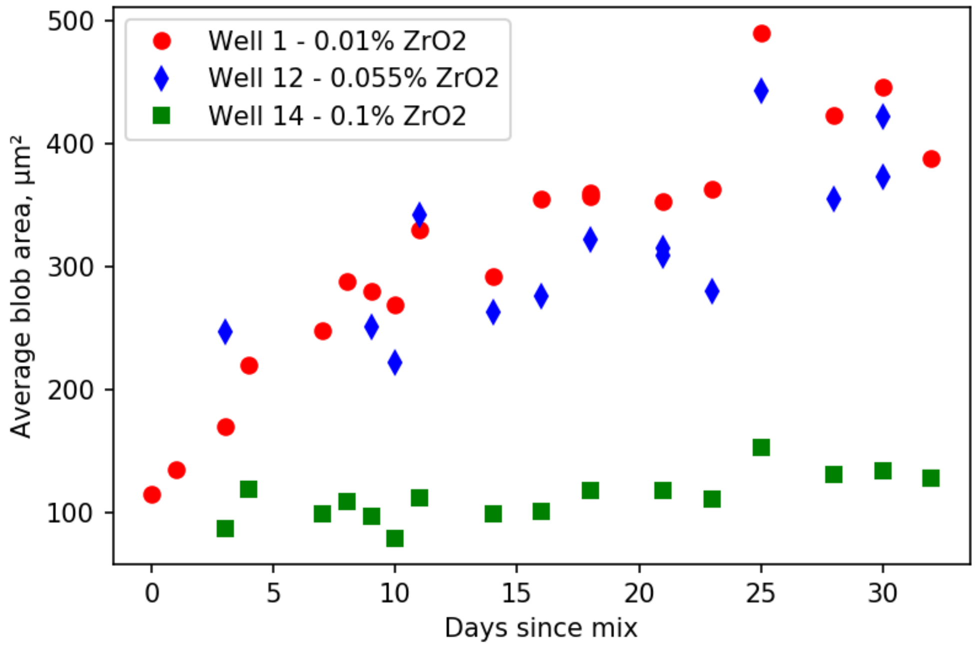


UNSTABLE
(0.01% nanoparticle solution)

STABLE
(0.1% nanoparticle solution)



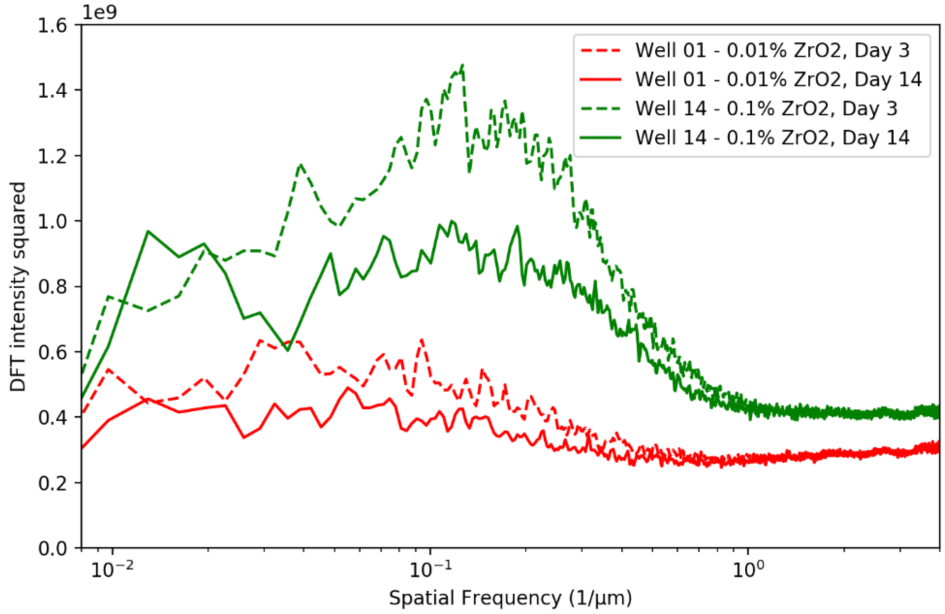




"Blob" Size:

More stable with 0.1% nanoparticle concentration.

ISS experiments & terrestrial "settling" were qualitatively similar (data not shown)



Dynamic Structure Factor:

Insight into how domain size changes with time

Well 14 "bump" indicates structure movement at < 10 μm scale over a 3 second interval.
(not the case for Well 1)

Investigation goals and objectives

Unresolved NPH Questions

- How will NPH suspensions behave in other gradients (thermal, electrical) as they do under gravity?
- How does the halo form or reform if disturbed?
- Can NPH (and manipulation thereof) be used assemble and reconfigure colloidal crystals?
- How does nanoparticle charge impact Halo formation?

Measurement Approach (1/8)

Light Microscopy Module (LMM)

ACE sample assembly

<u>Sample 1</u>: 1% vol. 1-2 μm silsesquioxane fluorescent particles, 0.1% vol. zirconia nanoparticles

<u>Sample 2</u>: 1% vol. 1-2 μm silsesquioxane fluorescent particles, 0.055% vol. zirconia nanoparticles

<u>Sample 3</u>: 1% vol. 1-2 μm silsesquioxane fluorescent particles, 0.01% vol. zirconia nanoparticles

Measurement Approach (2/8)

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 (3/8)

1. Aggregate Identification and Visualization:

Purpose

- Identify the structure of NPH aggregations as a function of nanoparticle concentration

Overview

- Particle aggregations will he identified and characterized

Significance

- 3D NPH aggregation studies have not been conducted
- Will compare and contrast to (published) planar/2D results

Measurement Approach (4/8)

1. Aggregate Identification and Visualization:

Experiment Steps & Required Data

- A sample will be mixed and visualized (2.5X to 20X).
- Two to four locations will be selected on each capillary.
- Locations will be imaged intermittently (2.5X to 20X), up to three hours for each location before mixing.
- Acquire each image set with no pixel binning, highest bits per pixel, full frame images.

- Aggregation dynamics will be analyzed similarly to ACE-H4

Measurement Approach (5/8)

2. Temperature Shock

Purpose

- The integrity of 3D NPH aggregations will be assessed

Overview

- ACE-T capabilities will induce a uniform temperature shock
- Aggregation stability will be monitored

Significance

- For the first time, stability (and temperature-dependent halo disruption) will be demonstrated

Measurement Approach (6/8)

2. Temperature Shock

- A sample will be mixed and visualized (2.5X to 20X).
- Two to four locations will be selected on each capillary.
- Locations will be imaged intermittently prior to and during heating.
- A capillary will be uniformly heated over a period of 10 minutes to 60 ^{o}C.
- The temperature will be held for two minutes, then cooled to ambient temperature.
- Acquire each image set with no pixel binning, highest bits per pixel, full frame images.

Measurement Approach (7/8)

3. Temperature Gradient

Purpose

- Acquire a “phase diagram” of NPH stability as a function of temperature

Overview

- ACE-T will induce a temperature gradient across the capillary
- Aggregation stability will be monitored across the capillary

Significance

- Stability and aggregation dynamics as a function of temperature will be assessed.

Measurement Approach (8/8)

3. Temperature Gradient

- A sample will be mixed and visualized (2.5X to 20X).
- Locations will be imaged intermittently prior to and during heating.
- A temperature gradient will be produced, centered around 45 ^oC with a 15 ^oC gradient (37.5 ^oC to 52.5 ^oC).
- Particle aggregations will be imaged across the capillary, providing a temperature-dependent “phase diagram”
- After imaging, the capillary is cooled to ambient temperature.
- Acquire each image set with no pixel binning, highest bits per pixel, full frame images.

Importance and Reason for ISS (1/2)

Need for Microgravity NPH Research

- To answer existing NPH questions, there is a specific need to *visualize a NPH* suspension

- Even with the available technology, particle sizes would be too large to remain in suspension for any significant length of time

- The only way to remove this issue is to perform experiments in an environment where gravity is reduced significantly

Importance and Reason for ISS (2/2)

How Microgravity can Impact NPH Research

How will NPH suspensions behave in other gradients (thermal, electrical) as they do under gravity?

- Fundamental changes induced by thermal gradients need to be measured with minimal impact from gravity

How does the halo form or reform if disturbed?

- Requires an environment where long term observation is possible
- Requires the ability to precisely disturb the suspension

Can NPH (and manipulation thereof) be used assemble and reconfigure colloidal crystals?

- Gravity settling can create crystals from microparticles only
- Other gradients may allow for nanoparticle incorporation
- Nanoparticle concentration may also play a role

How does nanoparticle charge impact Halo formation?

- Should have a direct impact on all the previous questions
- Also needs a method to create particles with a tunable charge

Expected results and how they will advance the field

- In ACE-T-12, fundamental insight will be gained into the interaction of smaller nanoparticles with larger colloids, i.e. the “nanoparticle haloing” (NPH) phenomenon, as a function of particle concentration. Crystallization behavior of the larger colloids will also be observed whose structure is a function of the size and concentration of nanoparticles. In the microgravity, we hope to observe unobstructed NPH interactions which would otherwise be significantly hindered by gravity on earth due to sedimentation issues (high density contrast between particles and fluid).

- This work will pursue the fundamental studies of order and particle interactions in nanoparticle haloing and subsequent colloidal structure stability and crystallinity. Understanding this is needed for technologies that will underlie complex processes like self-assembly and motility. With understanding comes specificity, control, and reversibility in interactions for materials with submicron-features.

Earth benefits / spin-off applications

Ultimately, the ability to design colloidal particles with a variety of well-controlled three-dimensional bonding symmetries opens a wide spectrum of new structures for colloidal self-assembly, beyond particle assemblies whose structures are defined primarily by repulsive interactions and shape.

Such materials might include photonic crystals with programmed distributions of defects. Optical technology utilizing such materials may offer intriguing solutions to unavoidable heat generation and bandwidth limitations facing the computer industry.

ACE-T Objectives

Objective 3: Demonstrate temperature-dependent nanoparticle haloing stability

- Los Alamos study observed that a higher temperature (48 ^{o}C) was needed to disrupt the aggregates
  - ACE-H2 limit of (38 ^{o}C); updated ACE-T limit is 60 ^{o}C.

Mission Success Criteria for ACE-T12

Minimum Success

- Homogenize the samples and acquire images such to identify the general geometry and shape of colloidal aggregations. Such measurements were acquired for 2/3 capillaries.

Significant Success

- All samples (3/3) imaged as previously mentioned.
- Temperature-dependent aggregation stability is demonstrated from "Temperature Shock" experiments.

Complete Success

- Detailed confocal images provided insight on 3D colloid formation, structure, and crystallinity with time.
- A phase diagram of temperature-dependent aggregation crystallinity is acquired from "Temperature Gradient" experiments