



# Increment 41/42 Science Symposium

## Advanced Colloids Experiment (ACE-H-2)



Presented by:

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# SCIENCE PRESENTATION

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

KY 14 NASA EPSCoR-0011



UNIVERSITY OF  
**LOUISVILLE**



**UK**  
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KENTUCKY



# 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



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Science Team



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Science Team



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NASA Glenn Research Center (GRC)



Advanced Colloids Experiments (ACE)

Ron Sicker (Project Manager)  
Bill Meyer (Project Scientist)



# ISS Increments 43 and 44 **Science Symposium** Advanced Colloids Experiment (Heated) – **ACE-H-2**

- 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

# What is Nanoparticle Haloing (NPH)?

- Originally discovered in 2001 by J.A. Lewis and co-workers of UIUC<sup>1</sup>
- Stabilize negligibly charged Silica suspensions through the addition of highly charged Zirconia nanoparticles
- Term derived from gravity settling experiments
- USAX experiments confirmed nanoparticle distance of about 2 nm from silica surface
- Observed in a number of other systems
  - Silica-Polystyrene<sup>2</sup>
  - Silica-Alumina<sup>3</sup>

<sup>1</sup>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

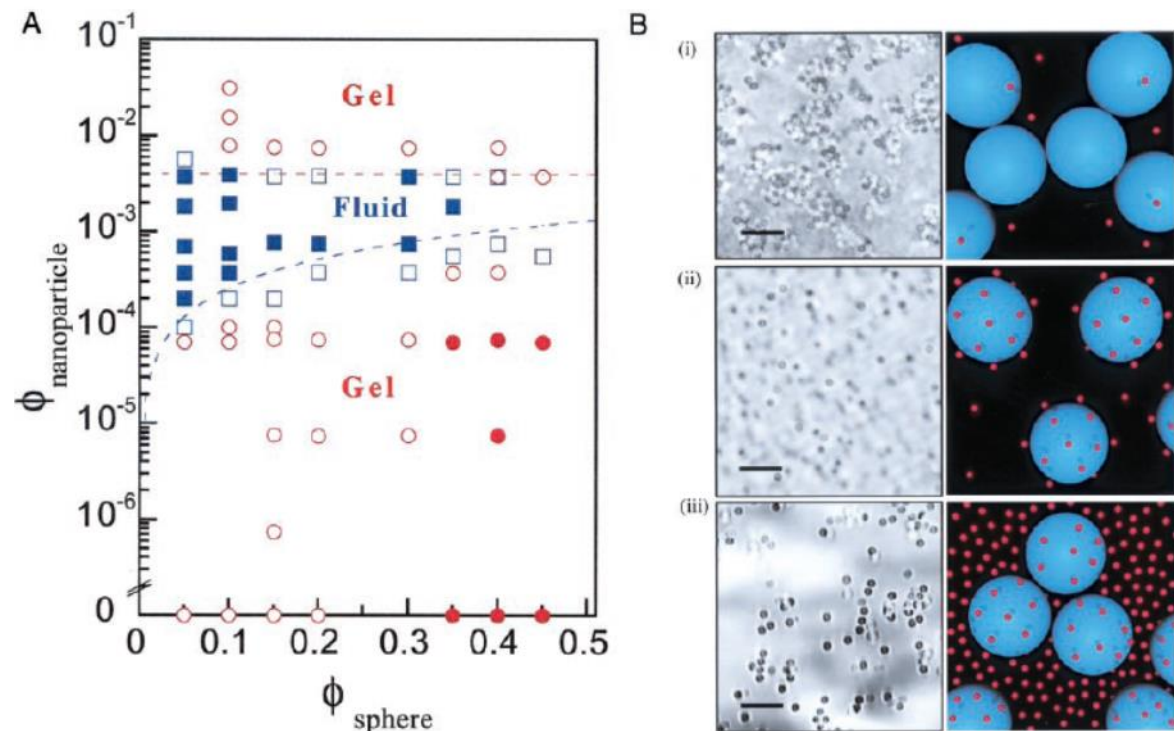
<sup>2</sup>Chan, A. T.; Lewis, J. A. Electrostatically Tuned Interactions in Silica Microsphere–Polystyrene Nanoparticle Mixtures. *Langmuir* **2005**, 21, 8576-8679.

<sup>3</sup>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.

# Nanoparticle Concentration Effects

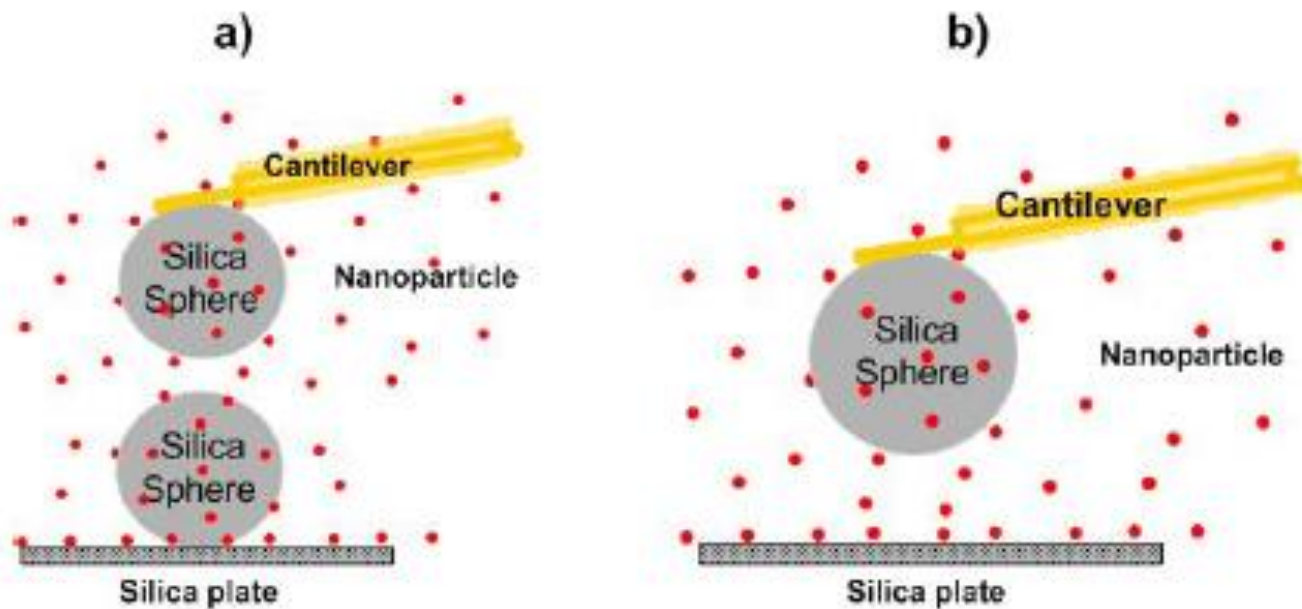
- Three stability regimes based on Nanoparticle volume fraction

- Attraction Gel
- Homogeneous fluid
- Depletion Gel



# 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



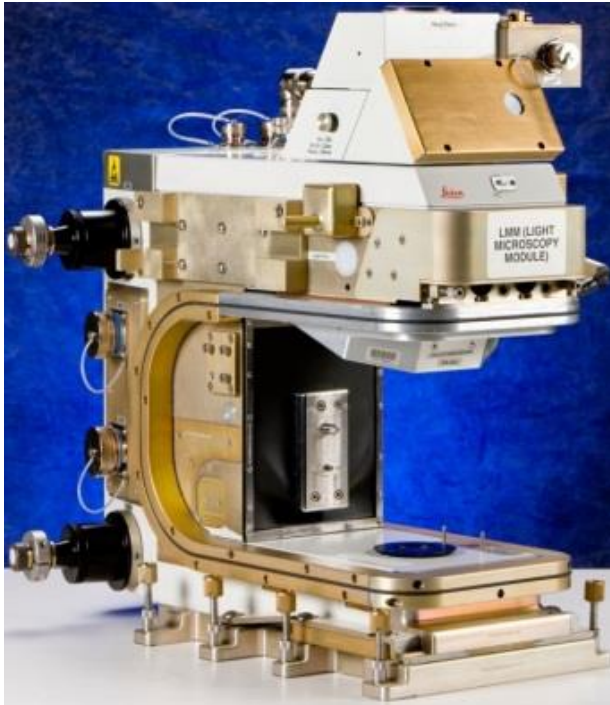


# Investigation goals and objectives

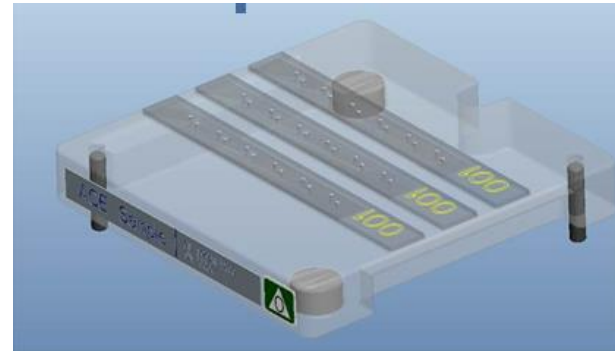
## Unresolved NPH Questions

- Does the Halo exist?
- Do nanoparticles and microparticles act individually or as a single unit?
- How does the diameter of the Halo change with the addition of nanoparticles?
- Will NPH suspensions behave in other gradients as they do under gravity?
  - Thermal
  - Electric Field
- How does the Halo form or reform if disturbed?
- Can NPH be used to create composite colloidal crystals?
- How does nanoparticle charge impact Halo formation?

# Measurement approach – 1/7



Light Microscopy Module  
(LMM)



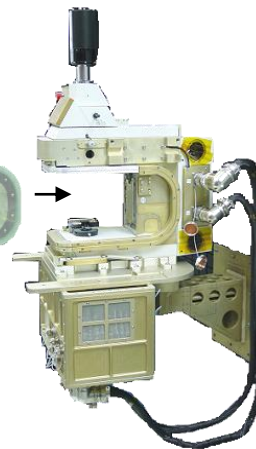
ACE Sample Assembly with  
Workhorse Style Cells

# Measurement approach – 2/7

## 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.



### 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 – 3/7

## 1. Aggregate Identification & Visualization

- Purpose
  - Identifying the structure of NPH aggregations as a function of nanoparticle concentration
- Overview
  - The number of “clusters” will be identified, counted, and imaged
  - The number of 2  $\mu\text{m}$  colloids within each cluster will be determined
    - If applicable, their crystalline arrangement will be determined
- Significance
  - 3D NPH aggregation studies have not been conducted
  - Will compare and contrast to (published) planar/2D results

# Measurement approach – 4/7

## 1. Aggregate Identification & Visualization

- Experiment Steps & Required Data
  - Methodically scan the chamber with a 10x objective using a DAPI filter. Identify colloidal aggregations, save the coordinate location for each respective aggregation, and determine the total number of identified aggregations for the sample well.
  - Return to each identified aggregation and acquire high resolution images of the whole aggregation (20x, 40x, or 63x air, depending on the size of the total aggregate). Count the number of particles within each respective aggregate.
  - Acquire each image set at 5 frames per sec, no pixel binning, 8 bits per pixel (highest supported), full frame images.
  - An out-of-plane scan may be required if larger colloidal aggregations occur.

# Measurement approach – 5/7

## 2. Thermal Shock

- Purpose
  - The integrity of 3D NPH aggregations will be assessed
- Overview
  - Inherent ACE-M heating capabilities will induce a temperature shock (uniformly and rapidly heating the sample).
  - The relative position of 2  $\mu\text{m}$  particles will be tracked.
  - Brownian motion via particle tracking will quantify NPH stability.
- Significance
  - For the first time, stability (and temperature-dependent halo disruption) will be demonstrated.

# Measurement approach – 6/7

## 2. Thermal Shock

- Experiment Steps & Required Data
  - Return to one colloidal aggregate and image the whole aggregation at high resolution (20x, 40x, or 63x air, depending on the size of the total aggregate).
  - Set the prescribed temperature setting (hot side). Acquire images continuously (5 frames per sec) for the duration of the temperature change. Simultaneously, acquire surface temperature measurements at the chamber.
  - After the temperature has stabilized, allow the sample cell to return to ambient conditions (cooling rate is arbitrary). Image acquisition during this time is not required.
  - Repeat for a second, similarly-sized aggregate within the same sample well.
  - (If time allows, repeat for additional aggregates.)

# Measurement approach – 7/7

## Additional Notes

- Preliminary inspection of each sample well will be performed (2.5X) to observe for air bubbles
- Data will only be acquired on sample wells with identified NPH-induced aggregates.
  - Not all samples are expected to contain aggregates as NPH is aggregate dependent.



# Importance and reason for ISS - 1/3

## Need for Microgravity NPH Research

- To answer the existing NPH questions, there is a specific need to visualize a NPH suspension
- Even with the highest end of currently available technology, particle sizes would 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 - 2/3

## How Microgravity can Impact NPH Research

- Does the Halo exist?
  - Can utilize particles that can be directly observed
- Do nanoparticles and microparticles act individually or as a single unit?
  - Direct observation of particle dynamics in an NPH suspension without the influence of gravity
  - Particle tracking to observe differences in nanoparticle and microparticle Brownian motion
- How does the diameter of the Halo change with the addition of nanoparticles?
  - Separation distances can be directly measured
  - Variations in these distances should change with nanoparticle concentration
    - Nanoparticle to Microparticle distance
    - Nanoparticle to Nanoparticle distance

# Importance and reason for ISS - 3/3

## How Microgravity can Impact NPH Research

- Will NPH suspensions behave in other gradients as they do under gravity?
  - Fundamental changes induced by thermal or electric field 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 be used to create composite colloidal crystals?
  - Gravity settling can create crystals from the 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-H-2, 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.

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

**BACKUP SLIDES**

# Mission Success Criteria for ACE-H-2

Complete success is the achievement of all of the science requirements (nanoparticle visualization, thermal shock, and Brownian motion analysis). There will be sufficient information to provide a crosscheck of all data and calculated factors. Sample processing and properly setting up image acquisition parameters are as important as the measurements during the experiments themselves. Adequate conclusions can still be drawn even if a significant (~33%) portion of the sample cells are compromised.

Success Level	Accomplishment
Minimum Success	Homogenize the samples and acquire images of the samples such to identify and count the formed NPH aggregations within the sample cell. A majority (> 75%) of the aggregations have been identified. The size of each aggregation is estimated based on its overall fluorescence.
Significant Success	A majority (> 90%) of the aggregations have been identified for several colloidal concentration levels (at least 9 of 15 samples). The number of colloids within each aggregation is determined. The crystallinity and geometry of each colloidal aggregation is determined.
Complete Success	Accomplish the above for most concentration levels (at least 13 of 15 samples) with the inclusion of the “Thermal shock” measurements. Multiple experimental runs will assess reproducibility.