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solutions for suspensions

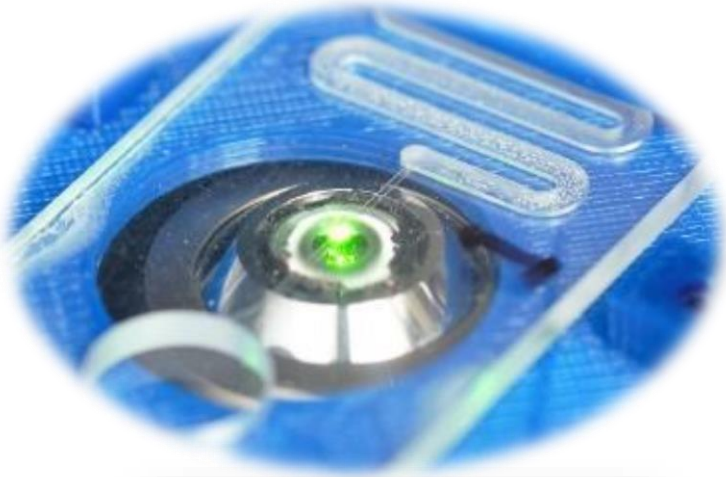
Holographic Characterization of Agglomerates in CMP Slurries

April 2017

Outline

- Total Holographic Characterization® (THC)
- Comparison of THC to other technologies
  - Dynamic Light Scattering (DLS)
  - Scanning Electron Microscopy (SEM)
- Analysis of Silica CMP slurries
  - Thermal cycling studies
  - Dilution
  - Mechanical Stress
  - Ionic strength studies
  - pH studies
- Summary

Total Holographic Characterization®



Capabilities:

- Detect and identify particles in suspensions
- Measure position, size and refractive index of particles on a single particle basis
- Rapid and automated collection of quantitative data
- No special sample preparation required
- 150 µL samples

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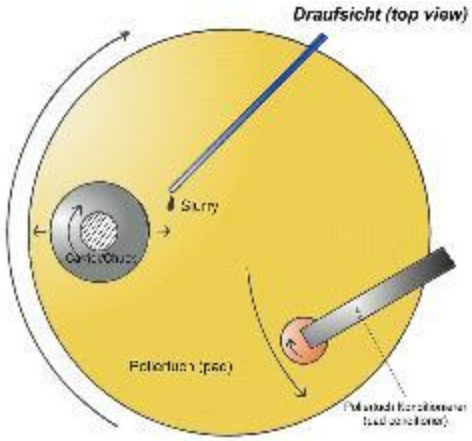
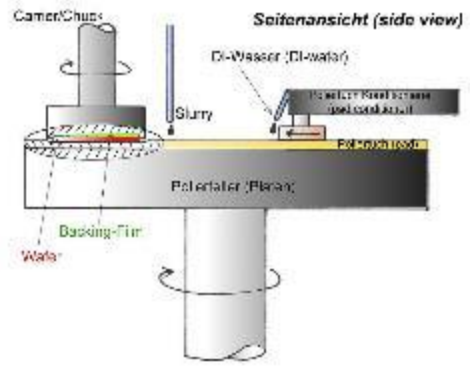
Detection of CMP Slurry Agglomerates

- Chemical Mechanical Planarization (CMP) Slurries
  - Abrasive and corrosive chemical slurries to polish semiconductor wafers
  - Flatness requirements sub-nanometer
  - Semiconductor industry suffers significant losses from Large Particle Contaminants (LPC)
- Spheryx Inc.
  - Total Holographic Characterization® (THC) detects slurry LPC

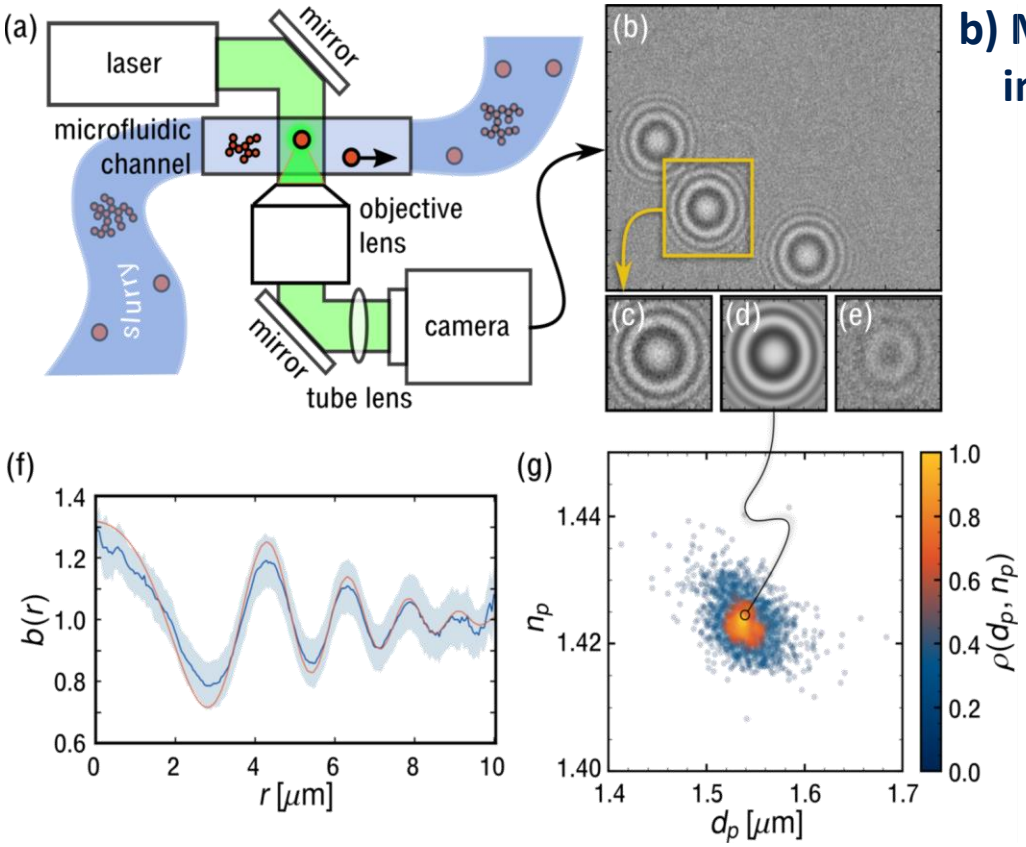




Source: Wikipedia.org

Total Holographic Characterization®

a) Particles flow through laser beam in microfluidic channel



b) Microscope records interference pattern

c) Experimental Data

d) Computation fit

e) Differences between fit and data

f) Compare measurement to scattering theory

Comparison yields:

- size
- refractive index
- 3D position
- symmetry

Size/refractive index distribution: for 2000 silica spheres in slurries acquired in 10 minutes

g) Build statistics:
Each point characterizes one particle, distribution shows homogeneity

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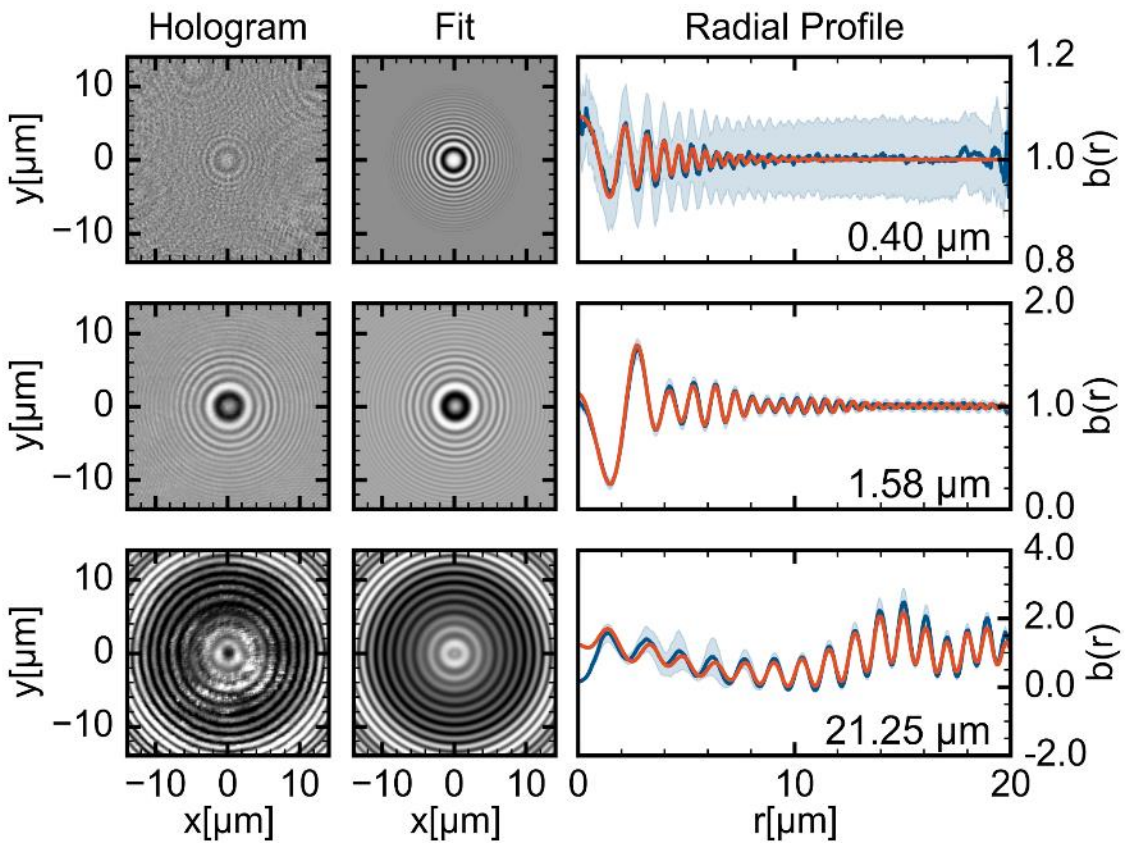
Analysis: From Hologram to Information



- Experimental holograms of various size polystyrene micro-spheres
- Theoretical fits derived using Lorenz-Mie theory
- Radial profiles demonstrating accuracy of the analysis
  - Blue: data
  - Orange: fit
  - Light Blue: std. dev.

Accurate particle size range from 500nm to 10µm

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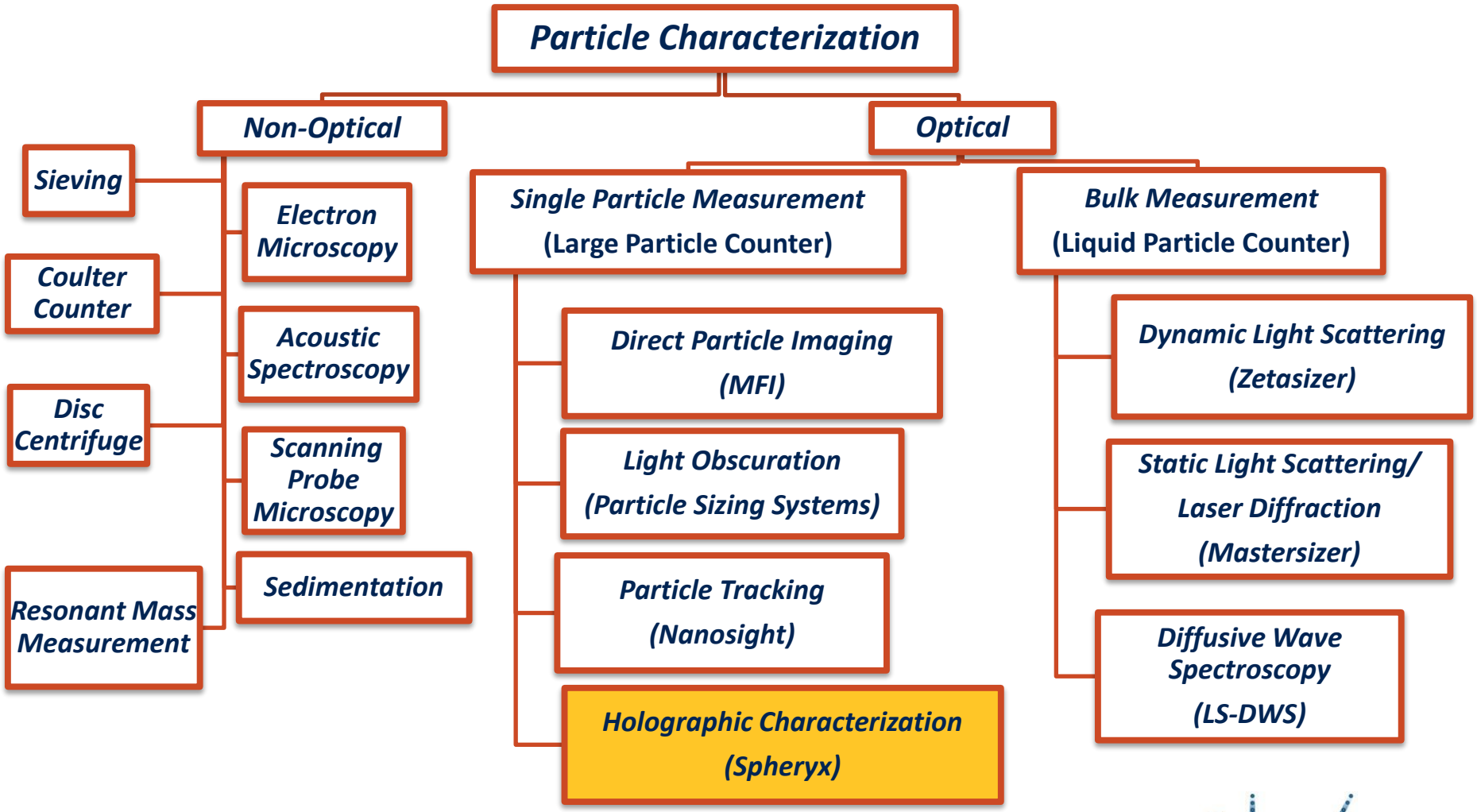
Technology Comparison



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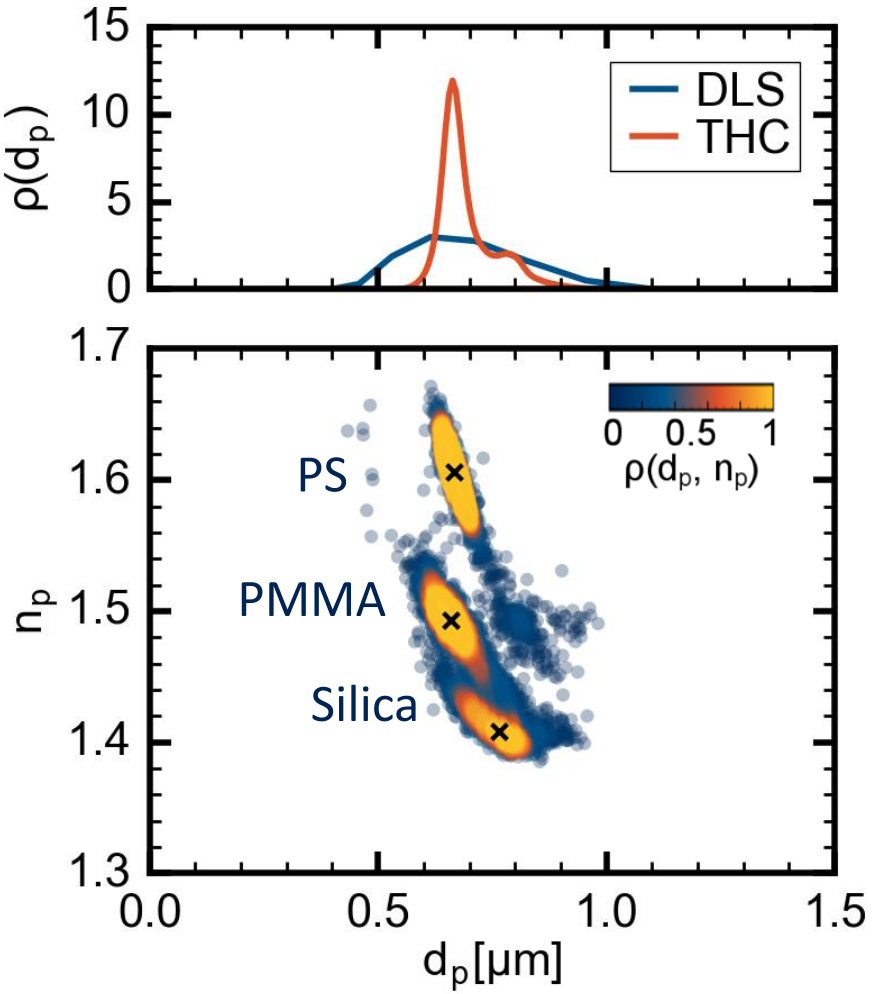
Technology Comparison

	Property characterization	Size	Shape	Density	Porosity	Refractive index	Heterogeneous	Resolution	Sample handling	Wet/Dry
Bulk methods	Dynamic light scattering	X						High	Simple	W
	Static light scattering	X						High	Simple	W
	Analytical disk centrifuge	X		X				Standard	Simple	W
	Acoustic spectroscopy	X					X	Standard	Simple	W
	X-ray scattering	X			X		X	Excellent	Complex	D
	Sedimentation	X		X				Low	Simple	W
	Sieving	X						Low	Simple	D/W
Single particle methods	Direct particle imaging	X	X				X	High	Simple	D
	Coulter counter	X						Standard	Simple	W
	Laser occultation	X						Standard	Simple	W
	Scanning probe microscopy	X	X				X	Excellent	Complex	W/D
	Electron microscopy	X	X		X		X	Excellent	Complex	D
	Particle tracking analysis	X	X	X				High	Simple	W
	Holographic characterization	**X**	**X**	**X**	**X**	**X**	**X**	**High**	**Simple**	**W**

Comparison With Dynamic Light Scattering (DLS)



- Sample containing 3 different 700nm spheres
  - Polystyrene (PS)
  - PMMA
  - Silica

Unique ability to distinguish by size & composition

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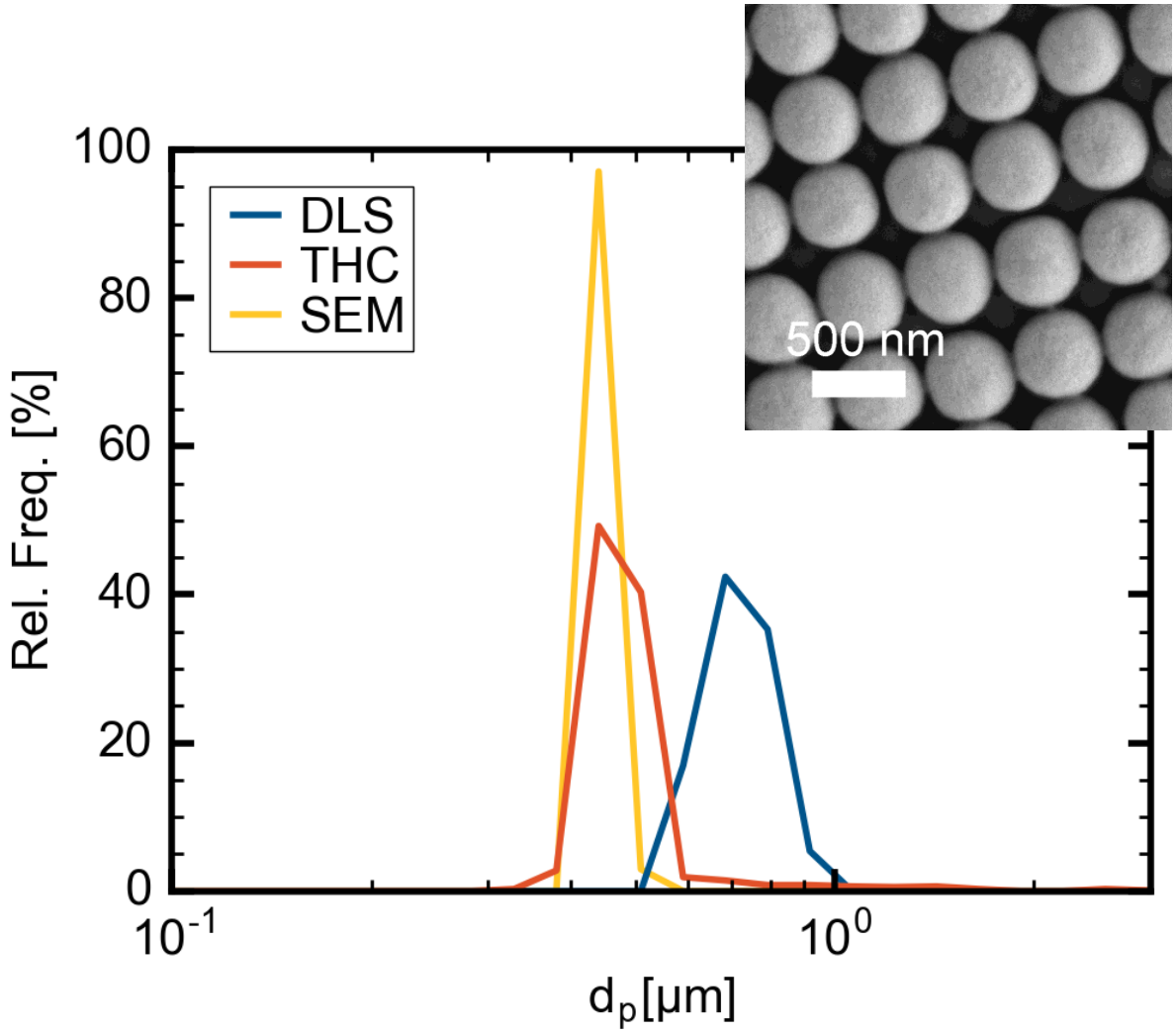
Confirmation with Scanning Electron Microscopy (SEM)

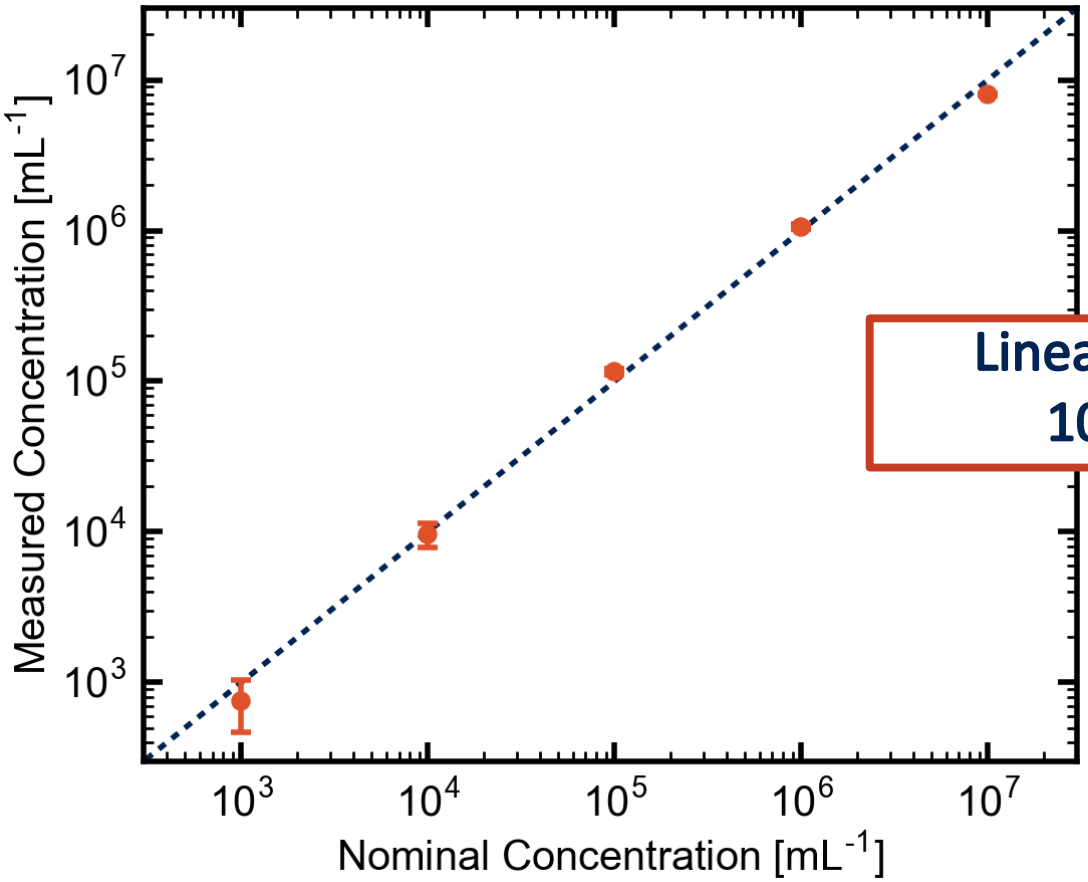


THC vs SEM

- 500 nm polystyrene spheres
- THC agrees with SEM results
- THC - no sample preparation needed
- Particle resolved
- No input assumptions

THC Concentration Measurements

Concentration = Particle Counts/Fluid Volume



No calibration or conversion required

Linear concentration range: 10^4 - 10^6 particles/mL

NIST Traceable PS

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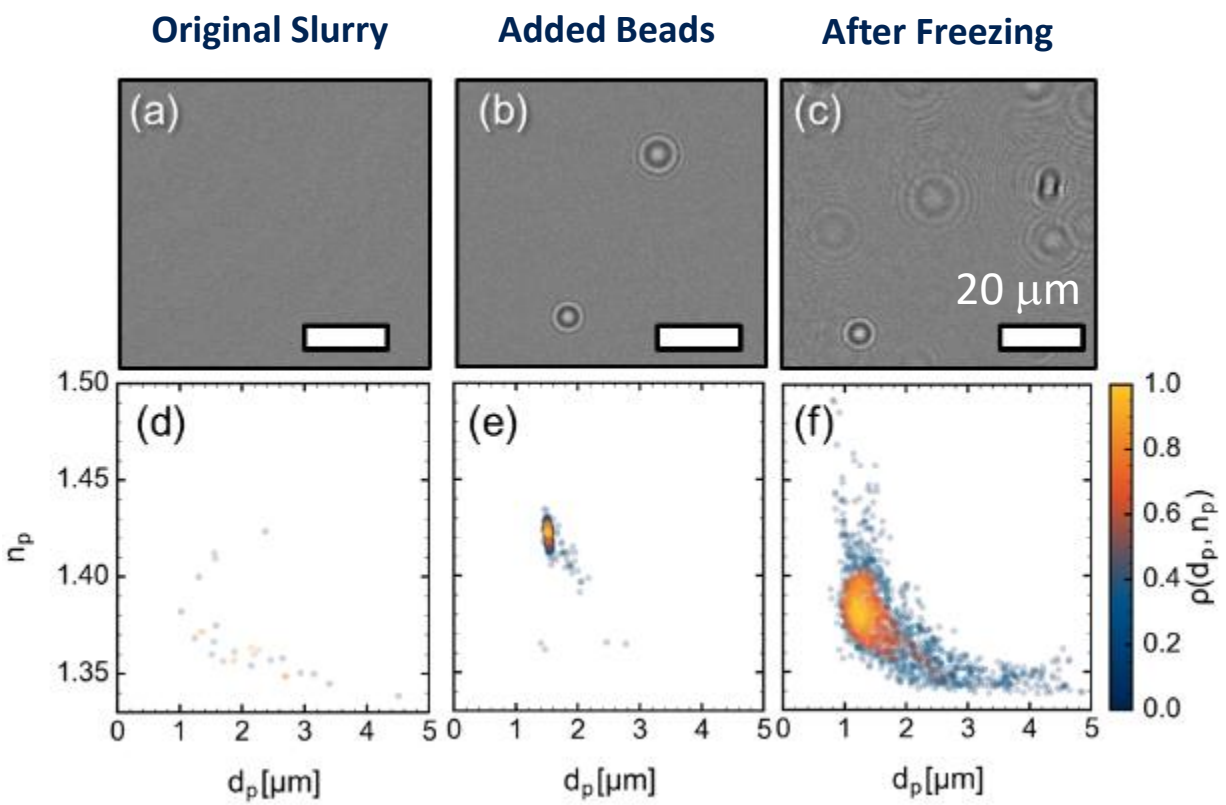
Agglomerate Detection in Turbid Media

- Difficult to detect and identify contaminants in turbid medium using conventional microscopy
- Total Holographic Characterization®
  - Scattered light from small particles ($d_p << \lambda$) contributes only to background
  - Effective in turbid medium to detect and characterize large contaminants

GE&R 120nm and 80nm silica slurry (10% wt)

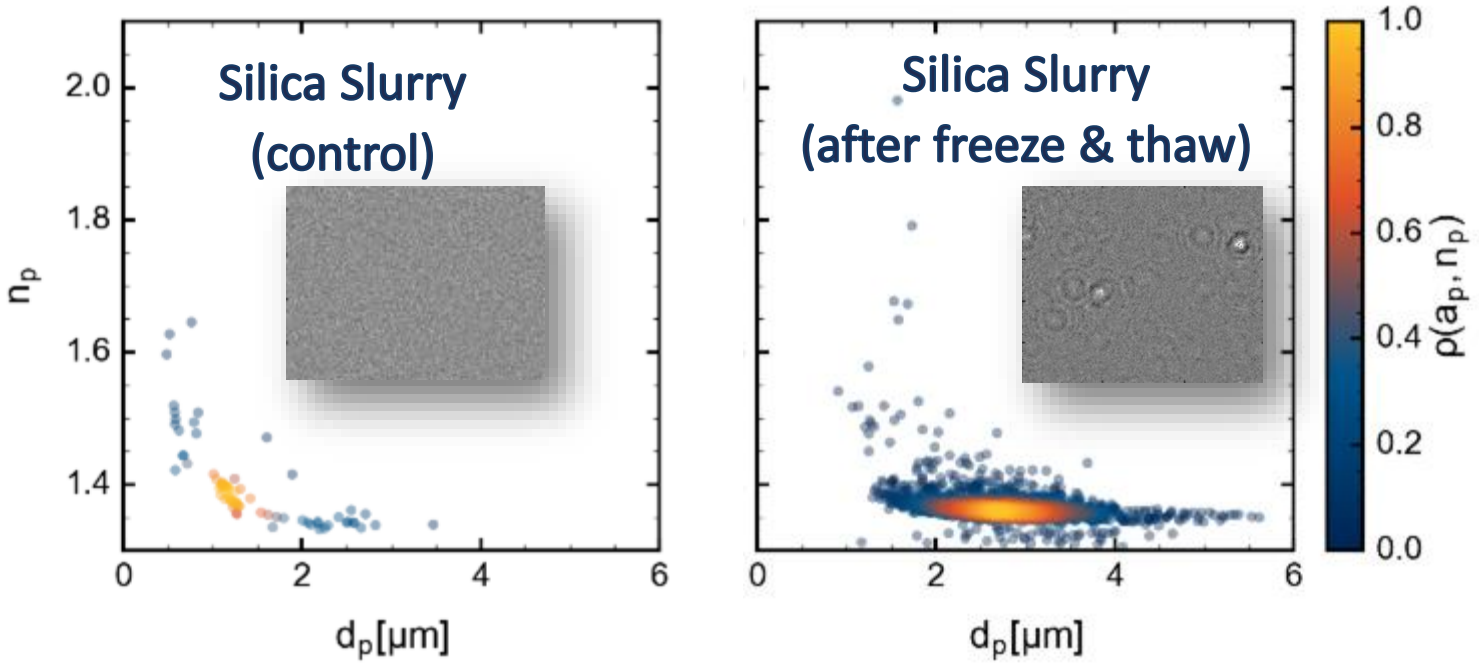
THC: Agglomerate Detection



- Single frames from holographic videos
- Silica slurry: uniform background
- Colloidal mixture:
  - Silica slurry doped with 1.5 μm particles
- Silica slurry after thermal cycle

THC can detect large contaminants in slurries

Silica Slurry After Thermal Cycling

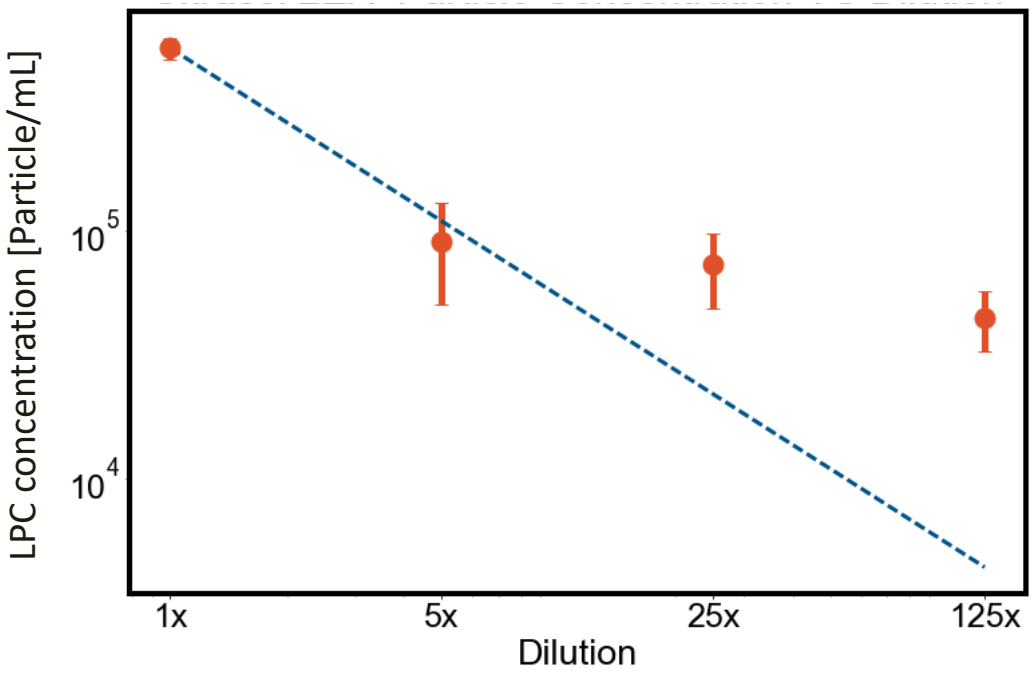


Measurements of Dow Ultra-Sol silica slurry (30% wt) yield:

- Few LPC were detected at original concentration
- More and larger LPC present after freezing and thawing

Dow Ultra-Sol 2EX silica slurry: 70nm (30.5 % wt) original

Dilution and Agglomeration

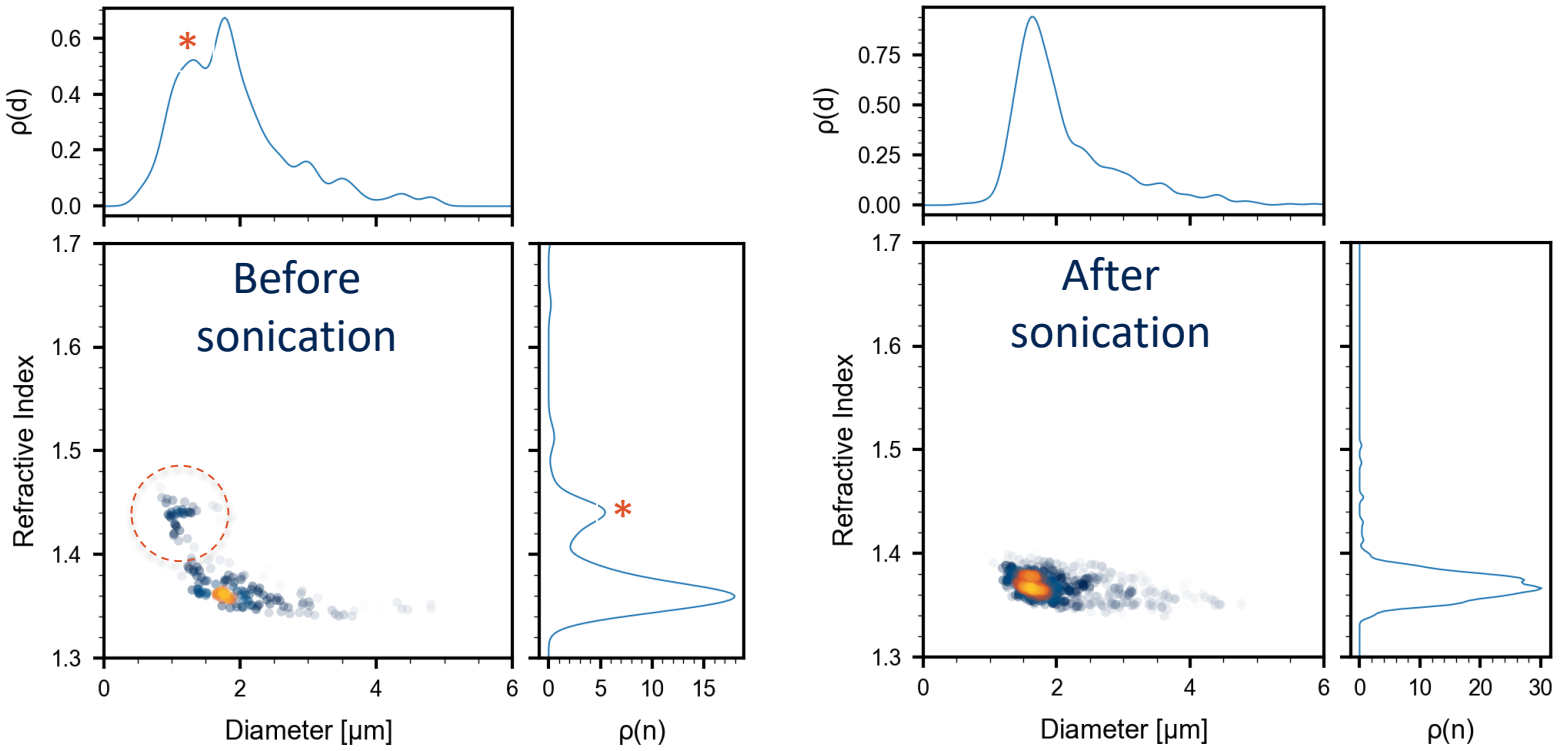


- Diluting commercial silica slurries with DI water
- Blue dotted line represents the expected linear reduction of LPC with dilution

Concentration of LPC does not scale linearly with dilution

Dow Ultra-Sol 2EX silica slurry: 70nm (30.5 % wt) original

Mechanically Induced LPC
Case 1



- Shear forces (sonication) increase the LPC concentration
- Shear forces REDUCE high index LPC concentration

Dow Ultra-Sol 2EX silica slurry: 70nm (30.5 % wt) original

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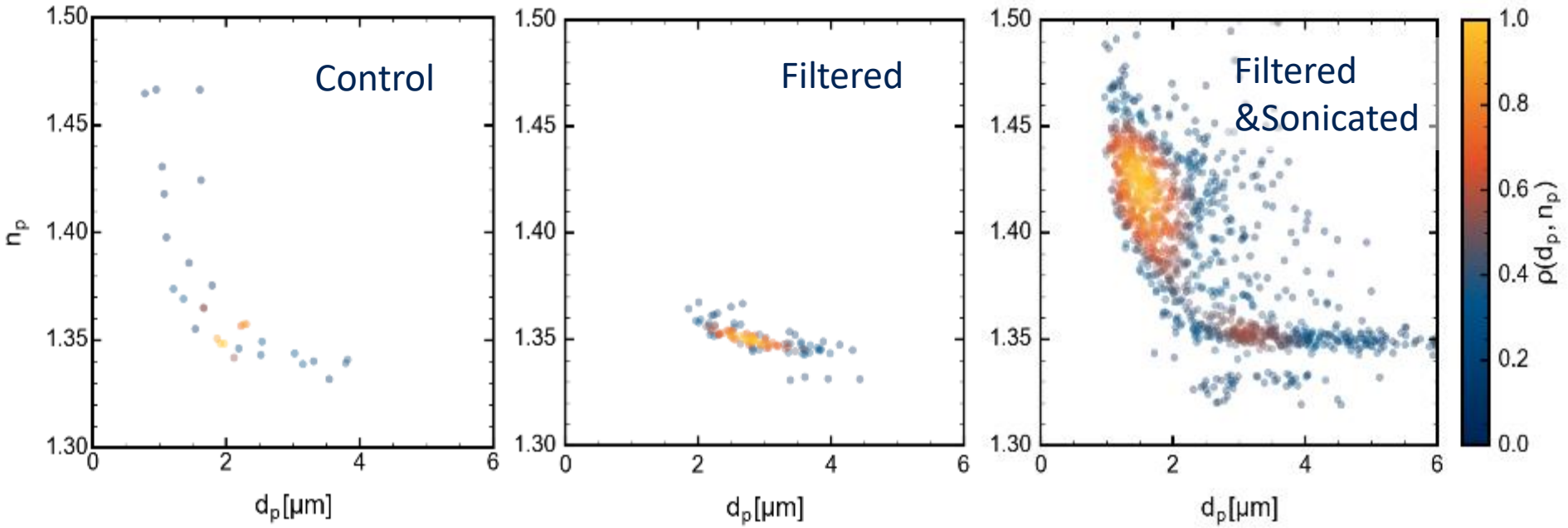
Mechanically Induced LPC
Case 2



- Shear forces (by sonication or filtration) increase the LPC concentration
- Shear forces INDUCE high index LPC concentration
- High index LPC depends on formulation
- Open question: Which type of LPC cause wafer damage?

General Engineering & Research silica slurry: 80nm (5 % wt) in DI water

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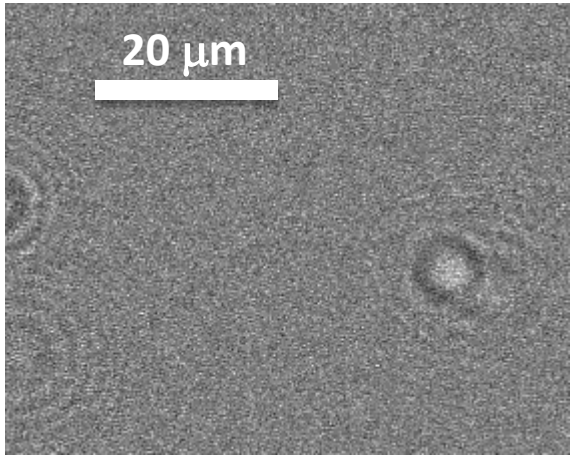
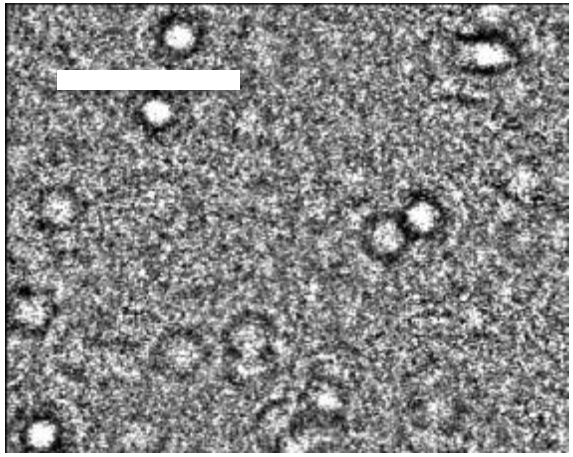
Ionic Strength Studies in Silica Slurries



120 nm Silica Slurry

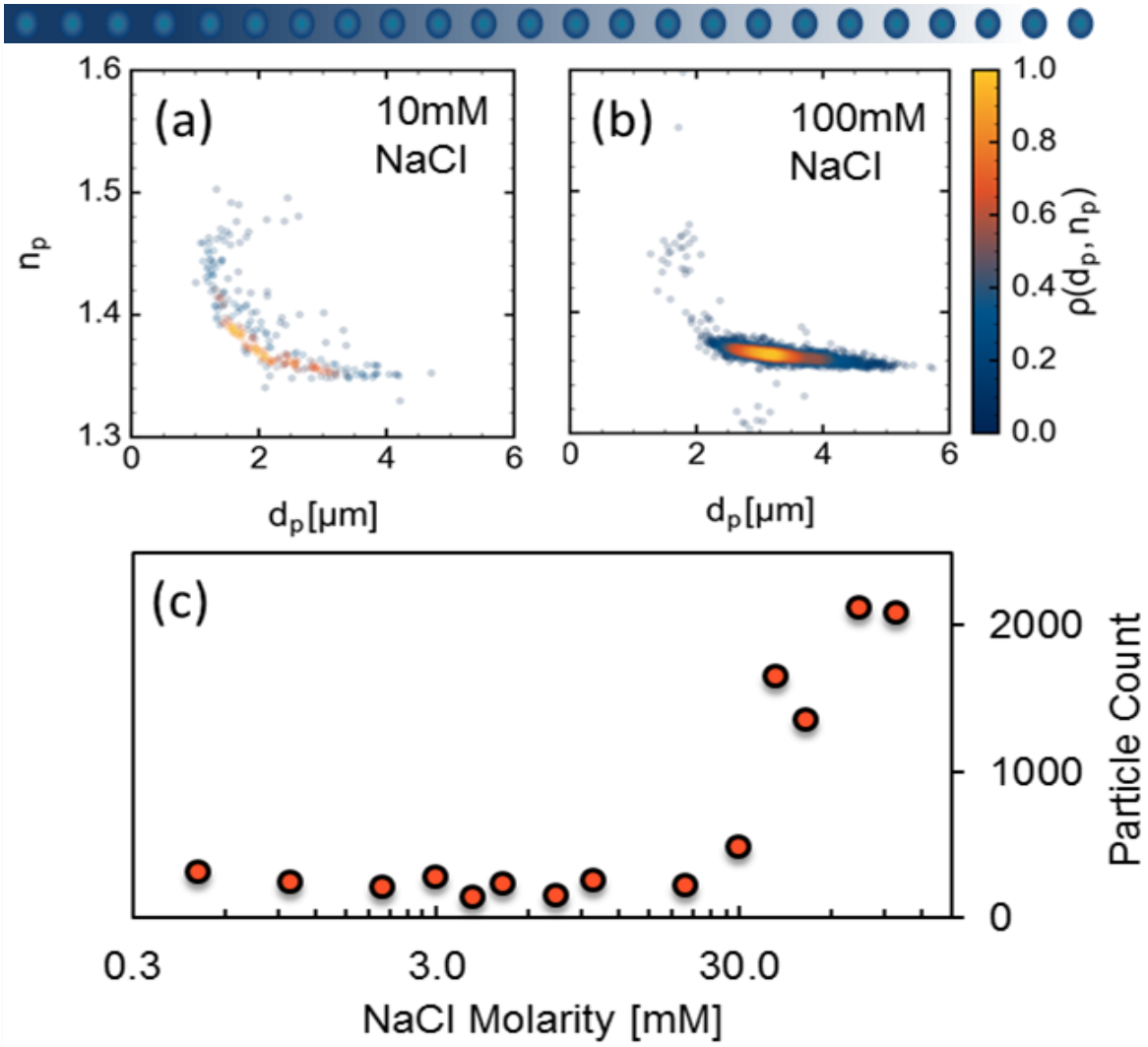


120 nm Silica Slurry
in 0.1M NaCl Solution

- Adding salt changes the electrostatic screening length
- Increasing ionic strength increases agglomeration
- THC measures sizes and refractive indices of LPC

General Engineering & Research silica slurry: 120nm (5 % wt) in DI water

Ionic Strength Studies in Silica Slurries



Total Holographic Characterization® of 120 nm silica slurry (5 wt%):

(a) 10 mM of NaCl

(b) 100 mM of NaCl

(c) Number of LPC as a function of salt concentration

General Engineering & Research slurry: 120nm (5% wt) in H_2O at pH = 6.5

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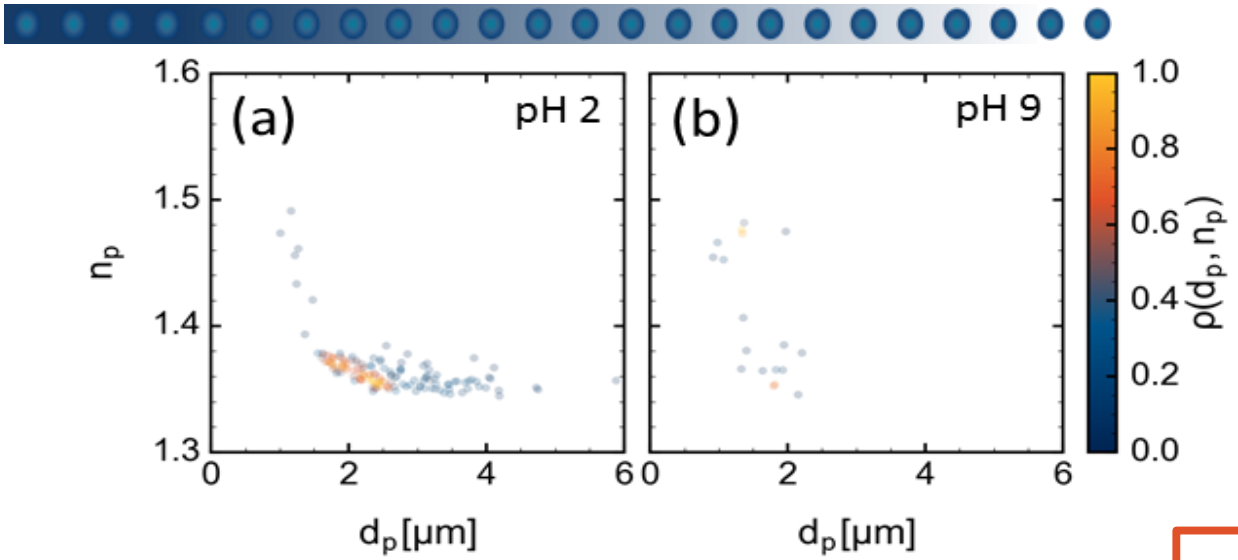
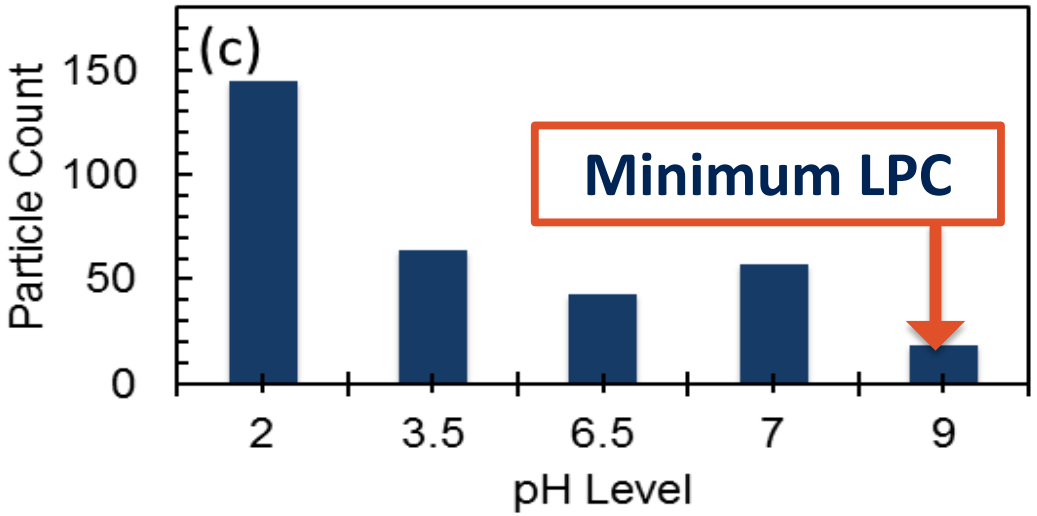
Silica Slurries: pH Studies



pH affects the agglomeration of silica slurry



Varying pH induced different number and structures of LPC

Summary

- Spheryx's Total Holographic Characterization® (THC) provides new insights into slurry agglomeration
- THC provides size, refractive index, composition and morphology
  - Temperature changes induce LPC
  - Dilution changes agglomeration behavior
  - Mechanical stress affects LPC
  - LPC increases above critical salt concentration
  - LPC changes with pH
- THC is a valuable tool for characterizing and detecting LPC during slurry formulation, application, storage and transportation

Acknowledgement

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NSF

NYU

GENERAL ENGINEERING AND RESEARCH