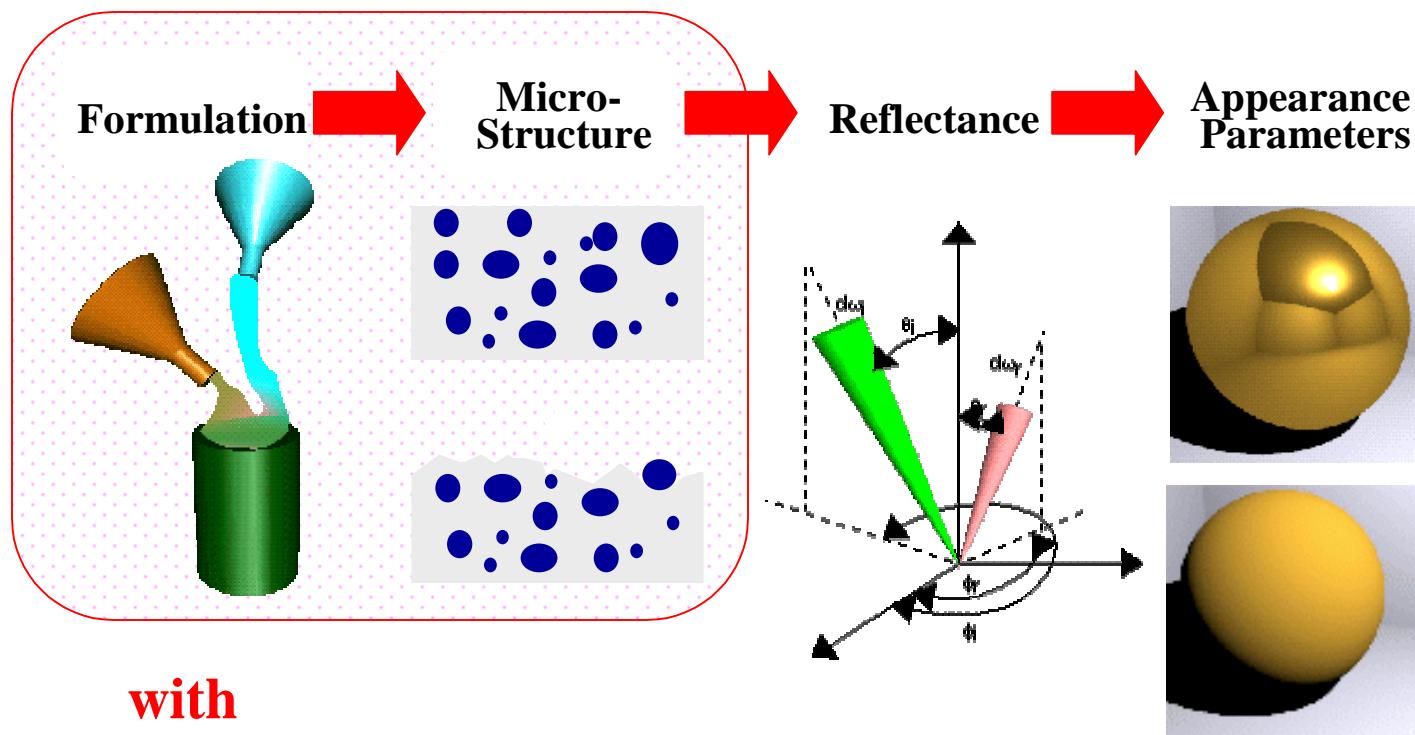


Characterizing Coating Microstructure

Li-Piin Sung



with

Mary McKnight
Paul Stutzman



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Characterizing the Microstructure of Pigmented Coatings and Reflectance Properties of Their Constituents

Microstructural Properties

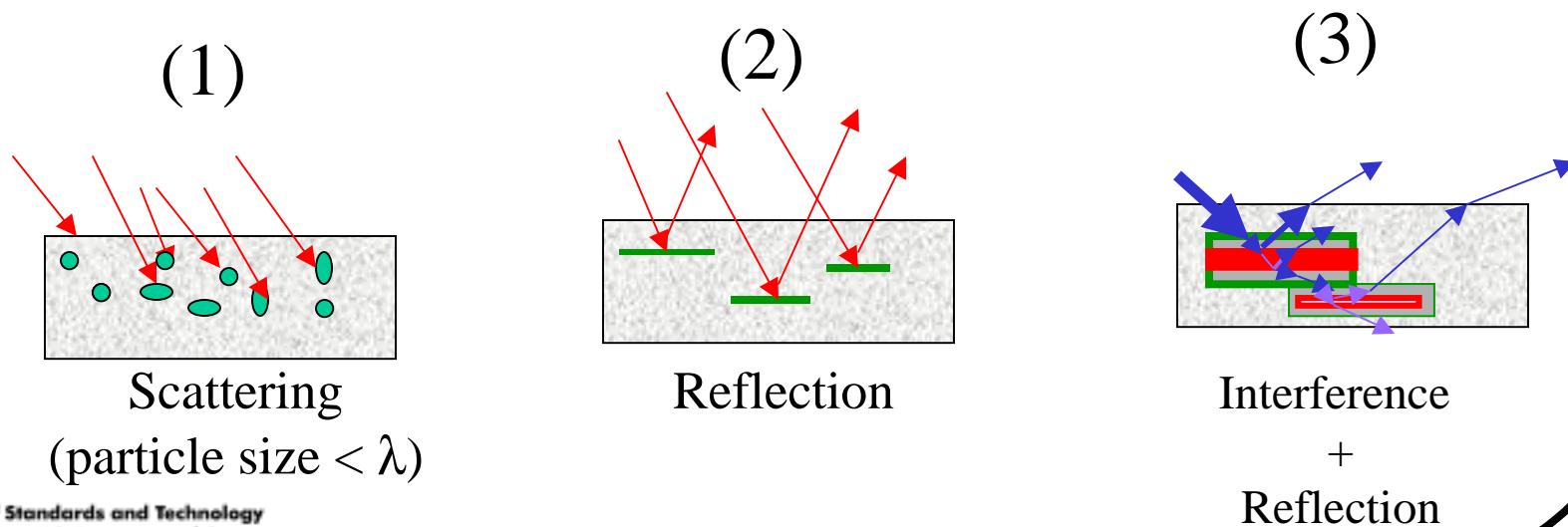
Surface Roughness
Pigment Particle Size
Pigment Size Distribution
Spatial Distribution
(orientation, shape, density)



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Three Major Types of Pigments Investigated:

1. TiO_2 Pigments – prepared in NIST
2. Metallic Flakes – in collaboration with coating industrial partners
3. Light Interference Pigments - in collaboration with coating industrial partners



Techniques:

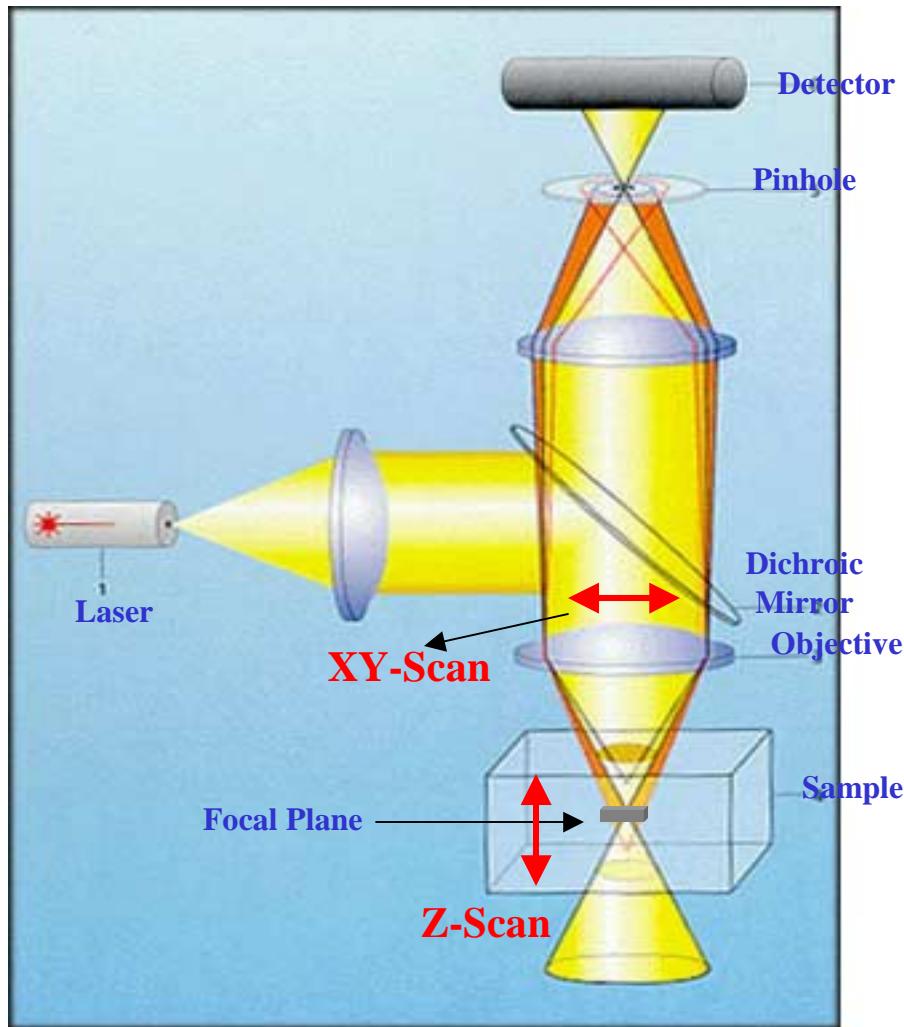
- Laser scanning confocal microscopy (LSCM)
- Scanning electron microscopy (SEM)
- Atomic force microscopy (AFM)
- Scattering metrology
 - reflectance (BRDF)
 - neutron, light scattering (static/time dependent)*
- Advanced laser-base image technique*

*Proposed Laser Goniometric Scattering (LGS) System



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LSCM Basics (I)



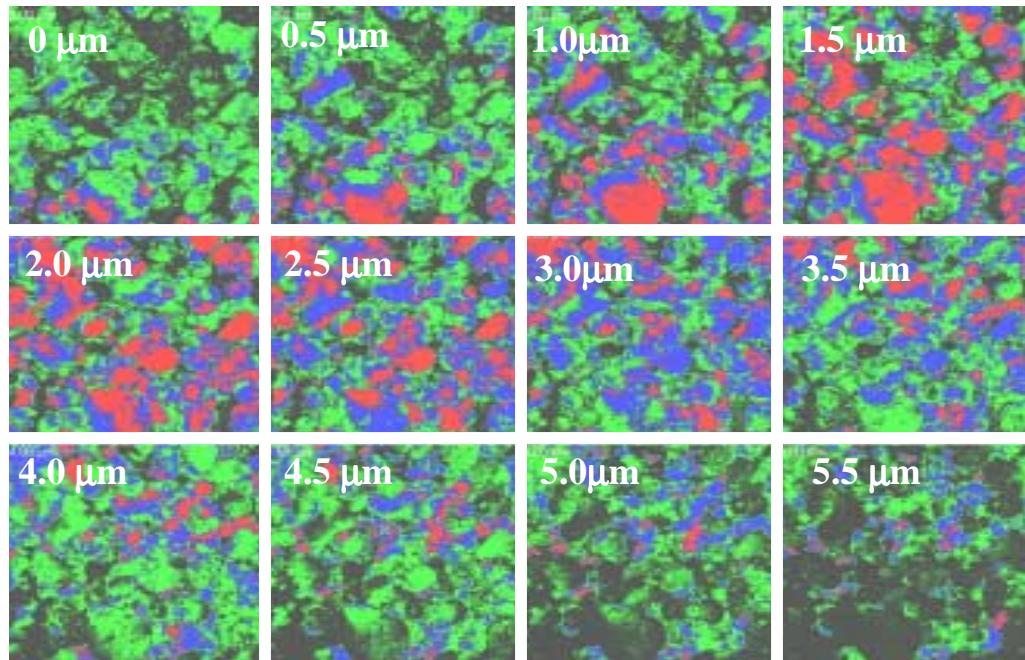
- Collect light exclusively from a single plane
(A pinhole sitting conjugated to the focal plane)
- Scan the sample sequentially point by point, line by line, and assembles the pixel information to one image.

Advantages

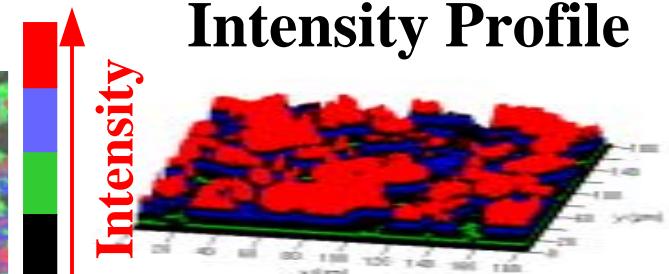
- Non-contact / non-destructive method
- By moving the focus plane, single images (optical slices) can be put together to build up a three dimensional stack that can be digitally processed afterwards.

LSCM Basics (II)

Optical Slices (Z-scan)

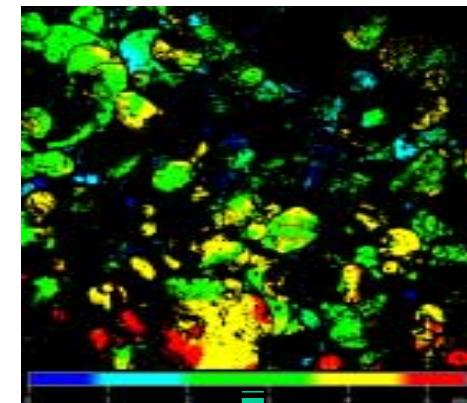


Intensity Profile

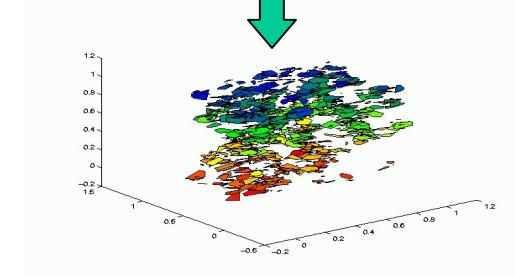
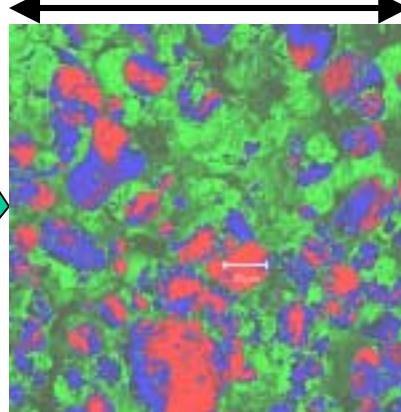
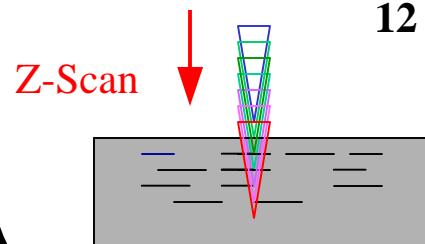


Depth Profile

184.3 μm



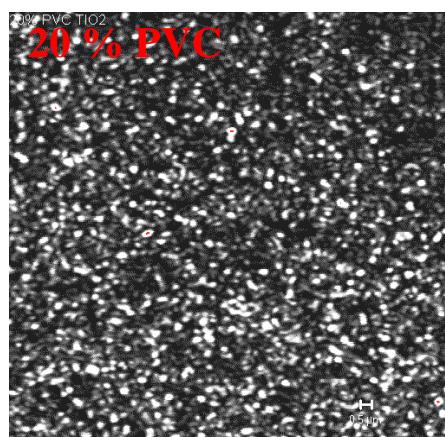
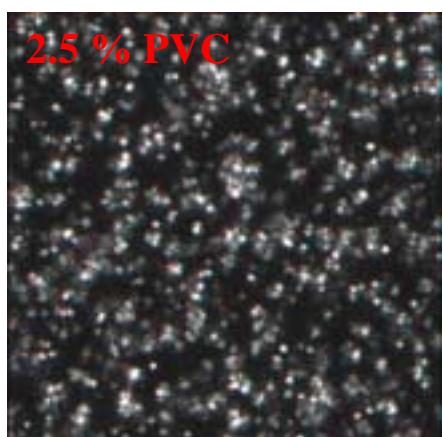
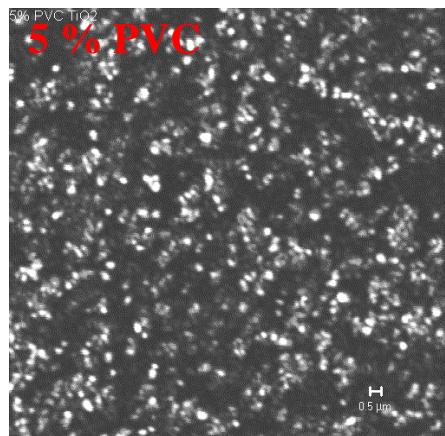
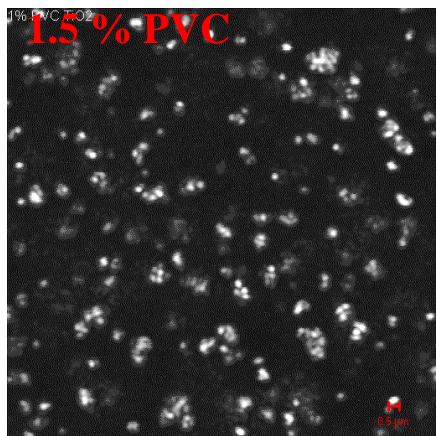
Overlapping
12 Optical Slices



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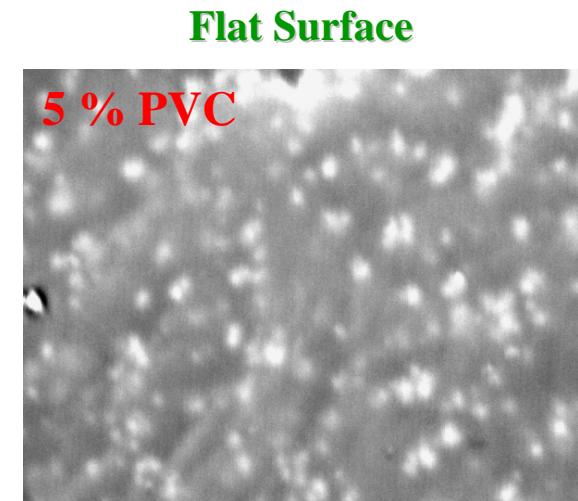
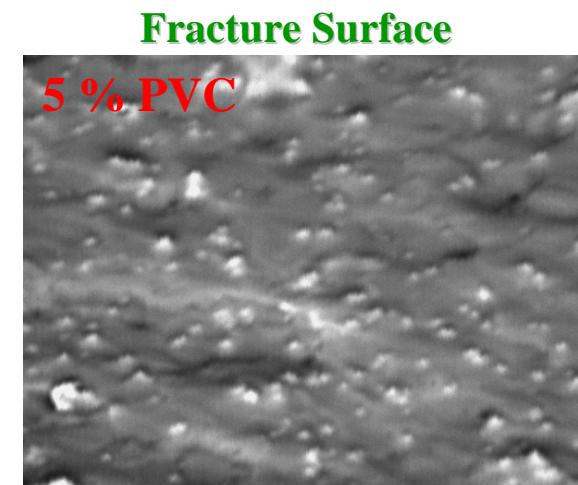
TiO₂ pigment dispersion in Acrylic Urethane Binder



— 2 μm

(LSCM Images)

PVC: pigment volume concentration



— 1 μm (SEM Images)

Pigment Size ~ 300 nm

NIST

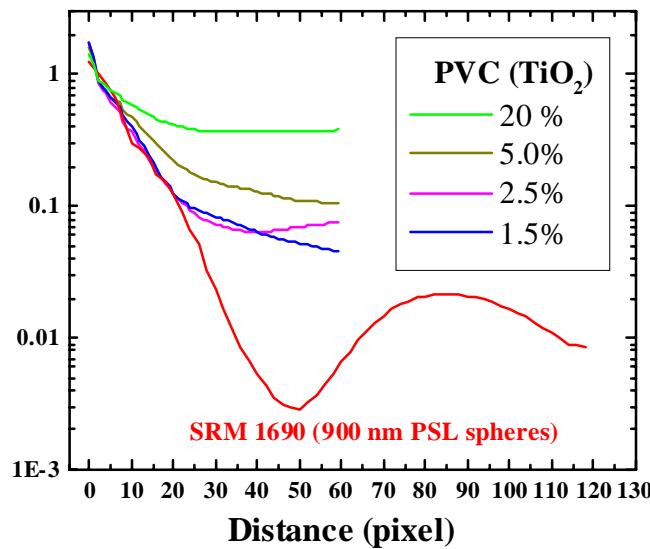
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Characterization of TiO_2 pigments

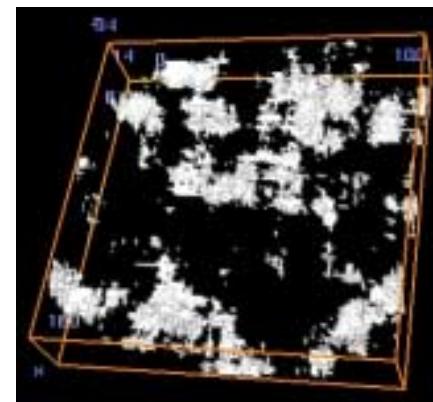
3-D Reconstruction

Concentration
Flocculation
Size Distribution

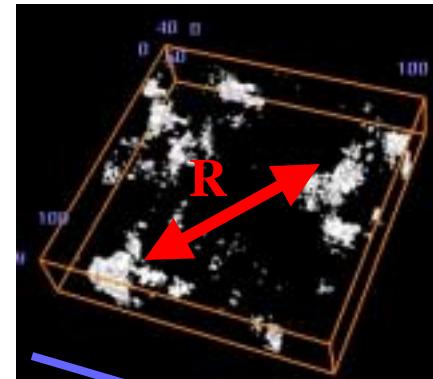
Autocorrelation Function



5 % PVC



2.5 % PVC

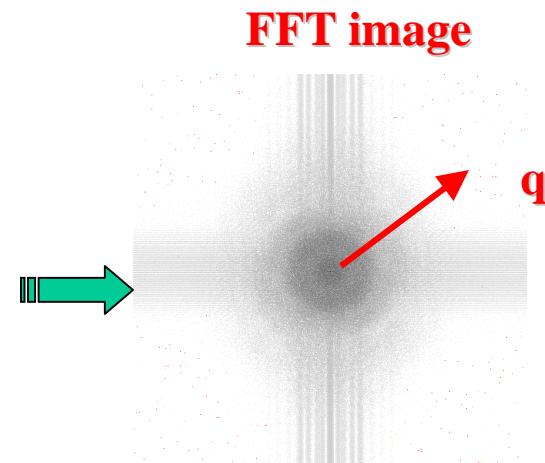
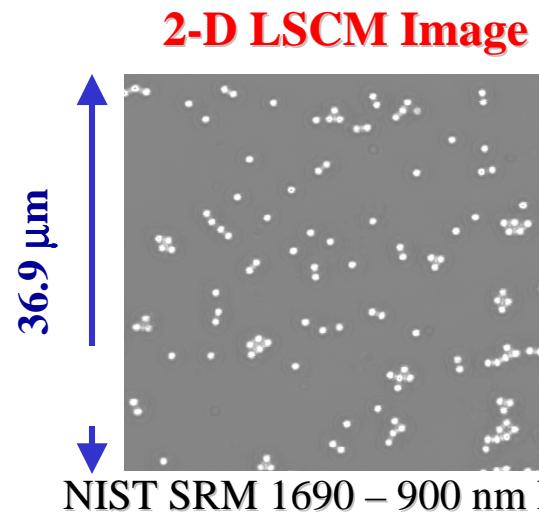


$R \rightarrow$ smaller as PVC \rightarrow larger

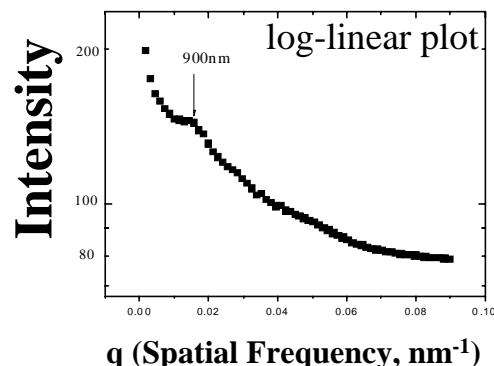
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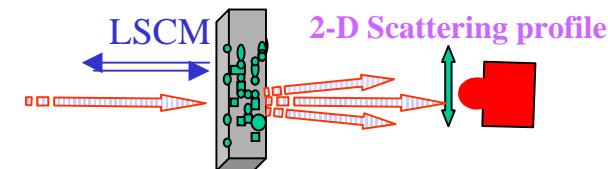
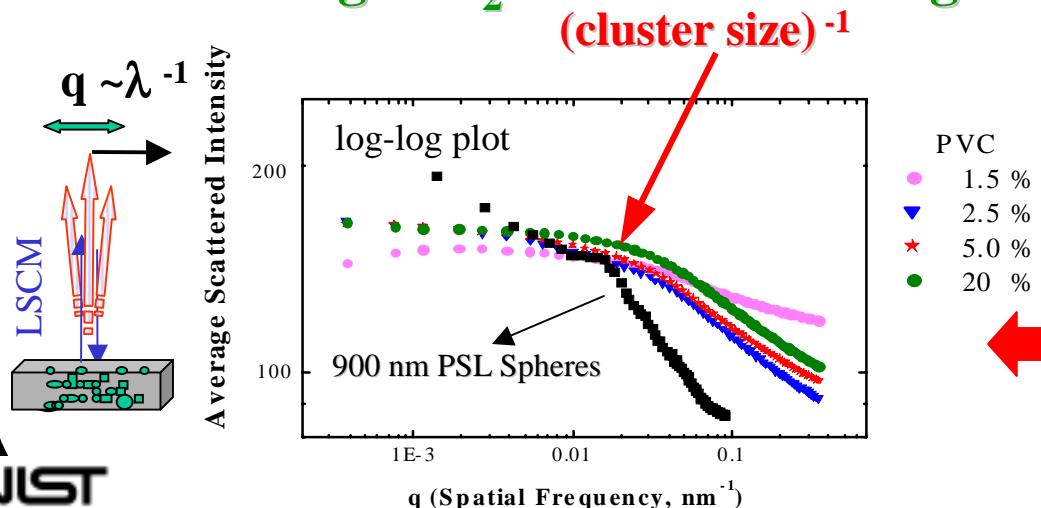
Particle Sizing and Particle-Particle Distribution



Circularly Averaged Intensity Profile



Characterizing TiO_2 cluster sizes using FFT images of LSCM images

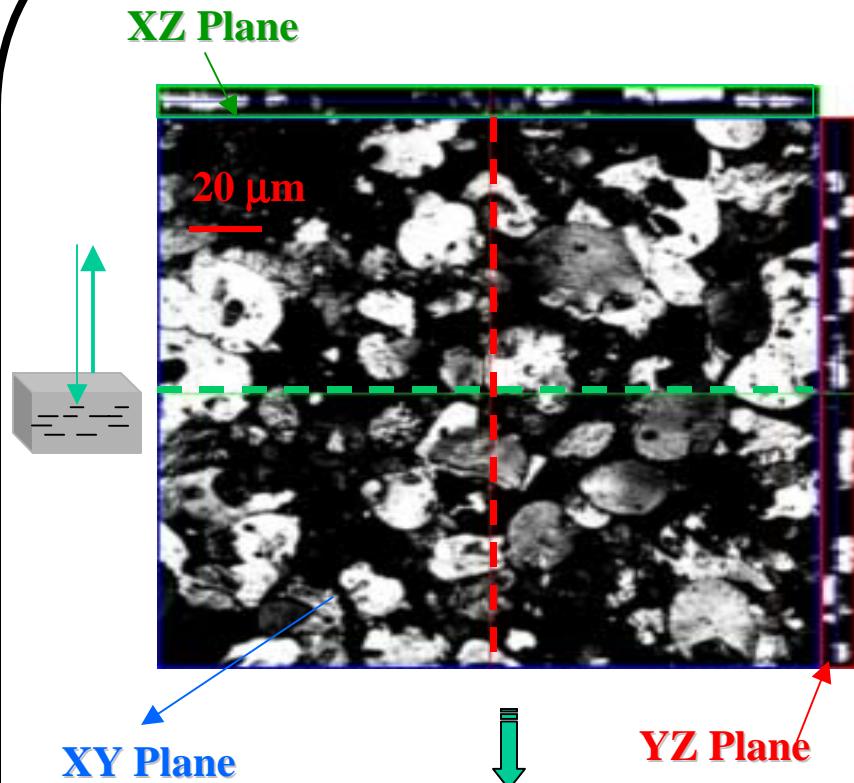


Light Scattering and Neutron Scattering Experiments



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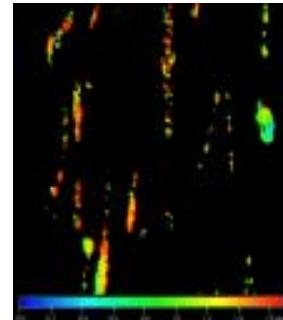
Microstructure of Metallic-flaked Coatings



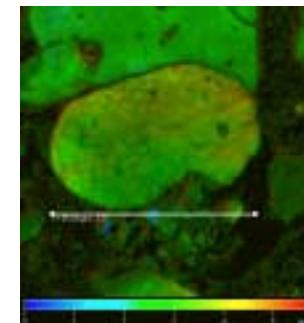
Characterization of Metallic Flakes

- Size Distribution
- Spatial Distribution
(Density, Orientation)

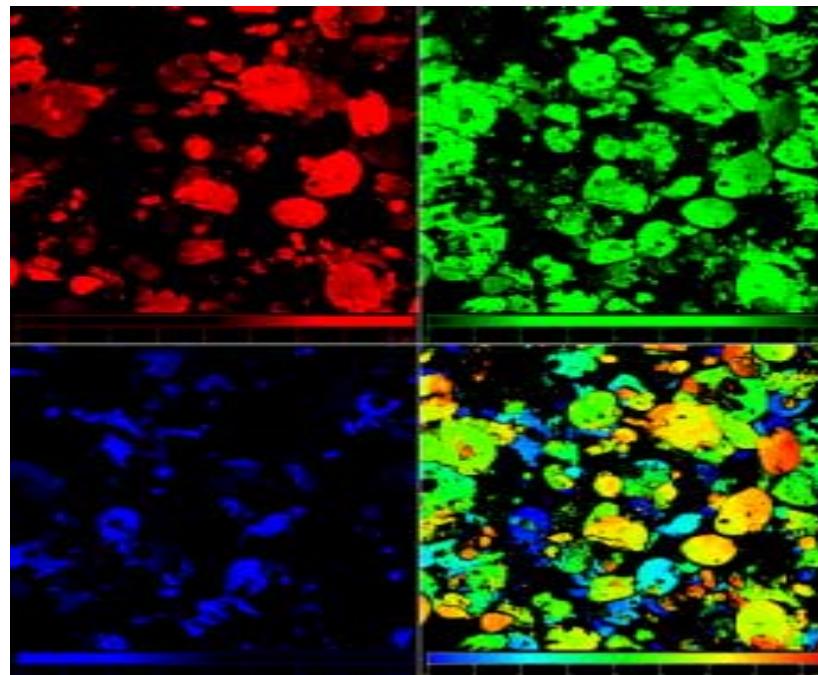
Cross Section



Single Pigment



Depth Profile

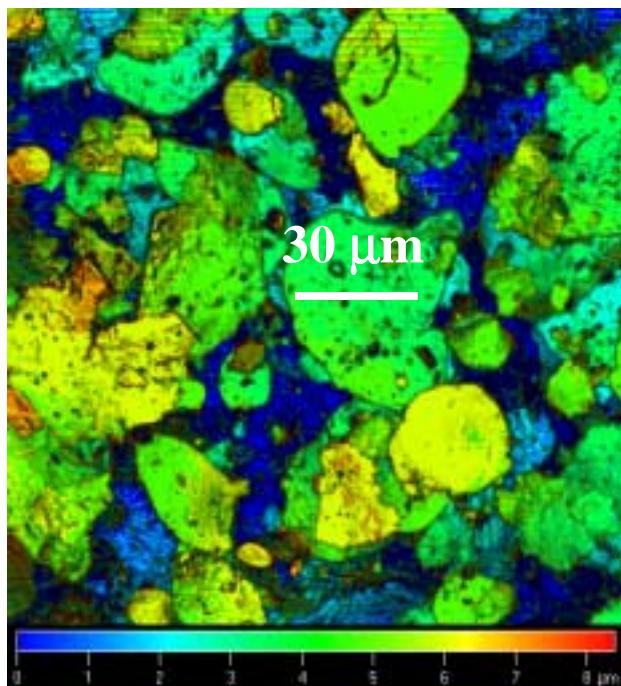


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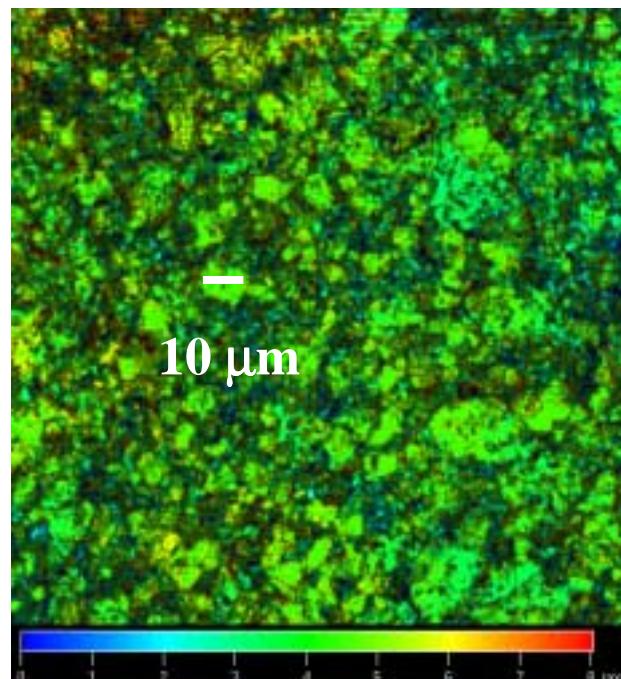
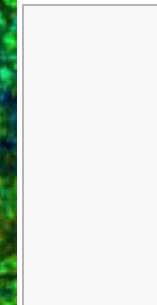
Aluminum Flake-Pigmented Coatings

Same PVC, different pigment sizes

Coarse



Fine



Grainy texture

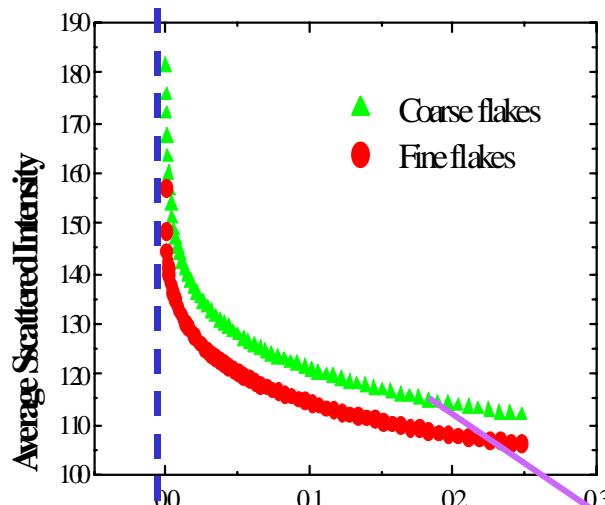
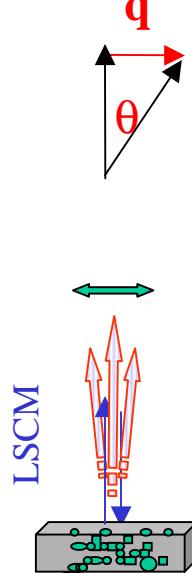
Finer finish

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Intensity Profile
FFT of LSCM image

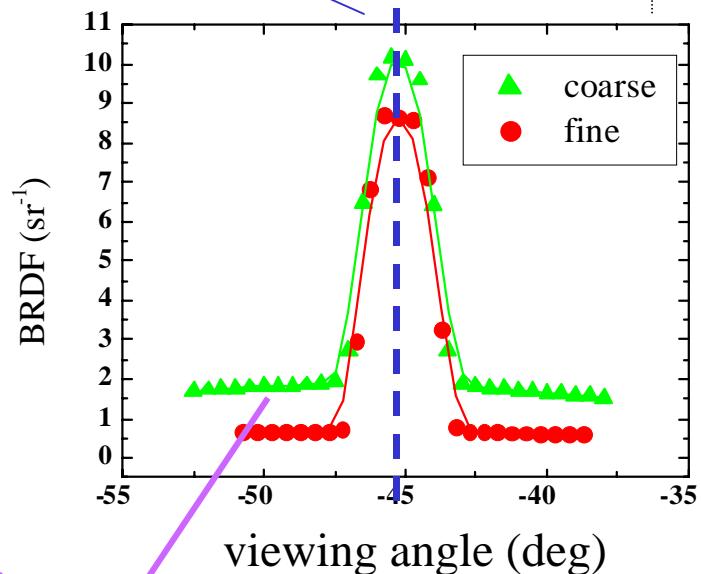
Optical Reflectance
(Specular Data- STARR)



q (spatial frequency) $\sim \sin\theta$
 \sim away from specular angle ($\theta = 0$)

Specular intensity

Incident angle = 45 degree



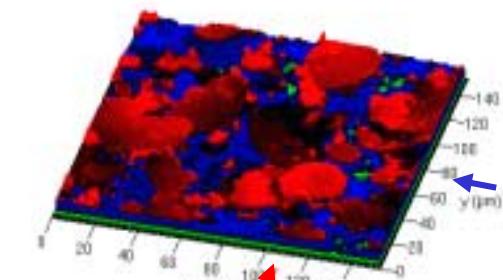
off-specular intensity

$$\text{BRDF} = \frac{\text{Scattered Intensity}}{(\text{Detected Solid Angle}) \times (\cos \theta_i) \times \text{Incident Intensity}}$$

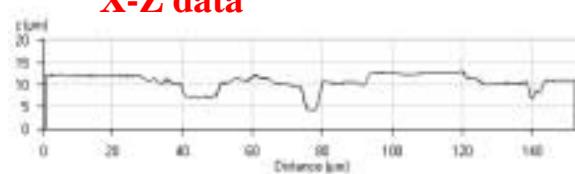
LSCM Topographic Measurements

(X, Y, Z) Data

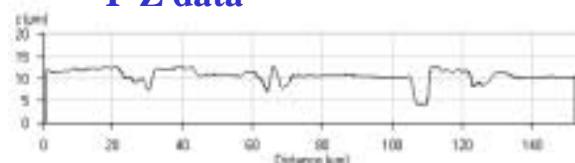
Coarse flakes



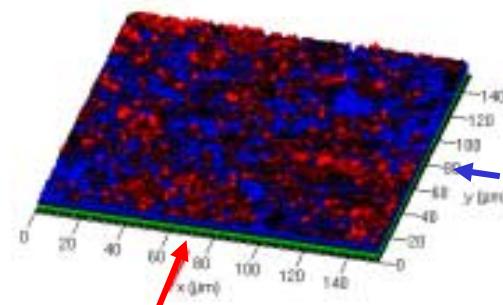
X-Z data



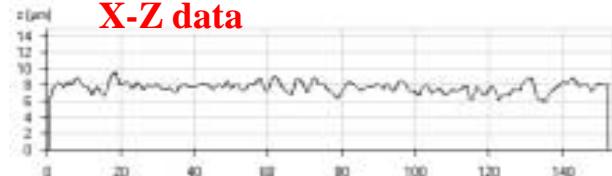
Y-Z data



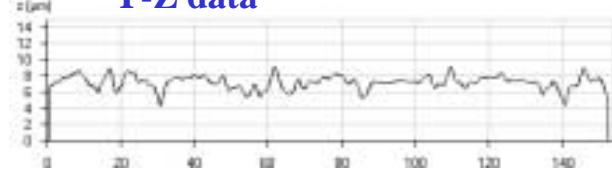
fine flakes



X-Z data



Y-Z data



pigment size
location (x,y,z)

Ray Approximation

BRDF Data

Measured BRDF Data

Computer Rendering

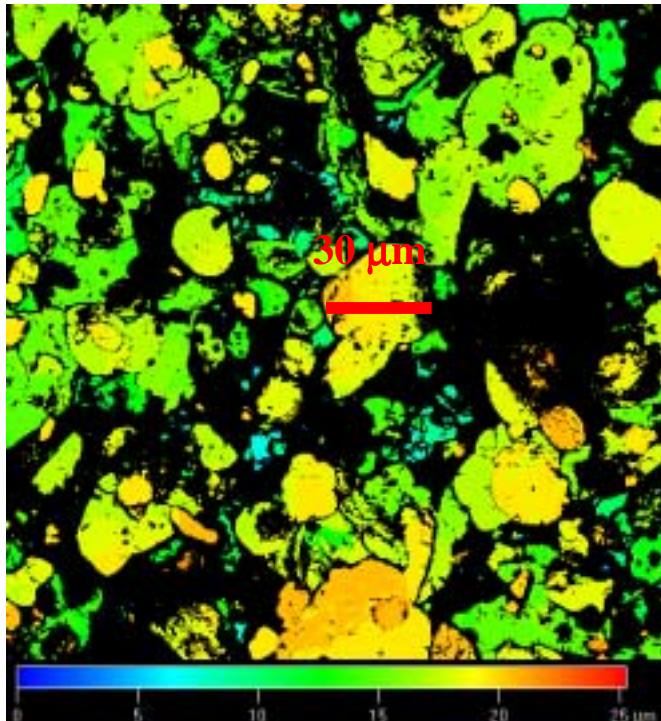


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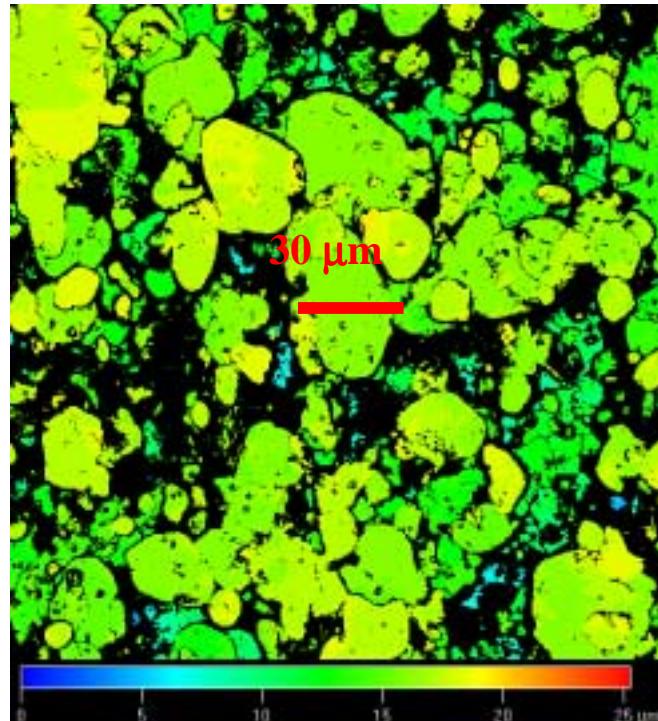
Aluminum Flake-Pigmented Coatings

Similar pigment size and PVC,
but different densities near the surface

Sample #1: low density



Sample #2: high density

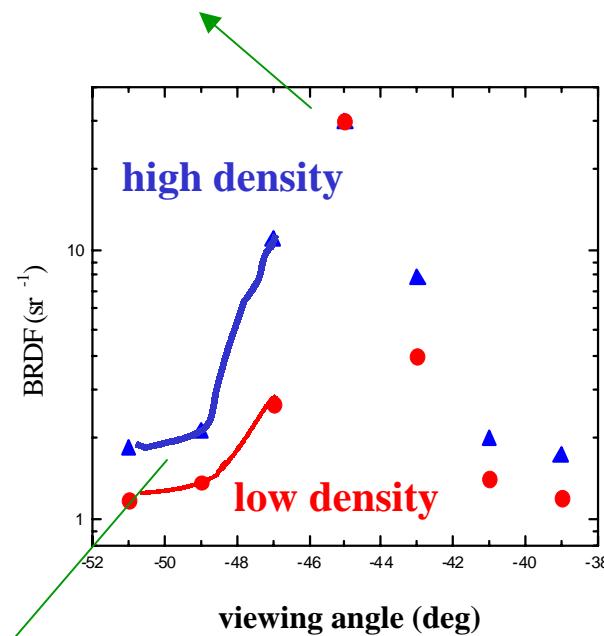
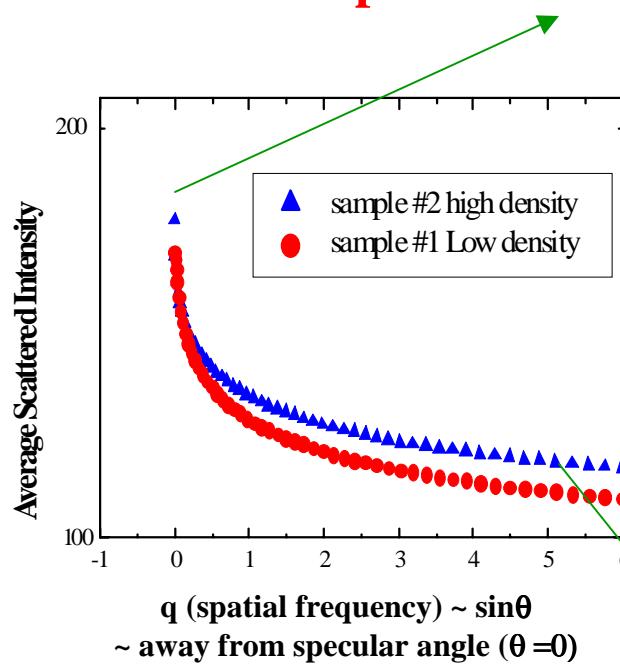


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Intensity Profile
FFT of LSCM image

Optical Reflectance
(Specular Data- STARR)

specular intensity – no significant difference



Difference in the off-specular feature

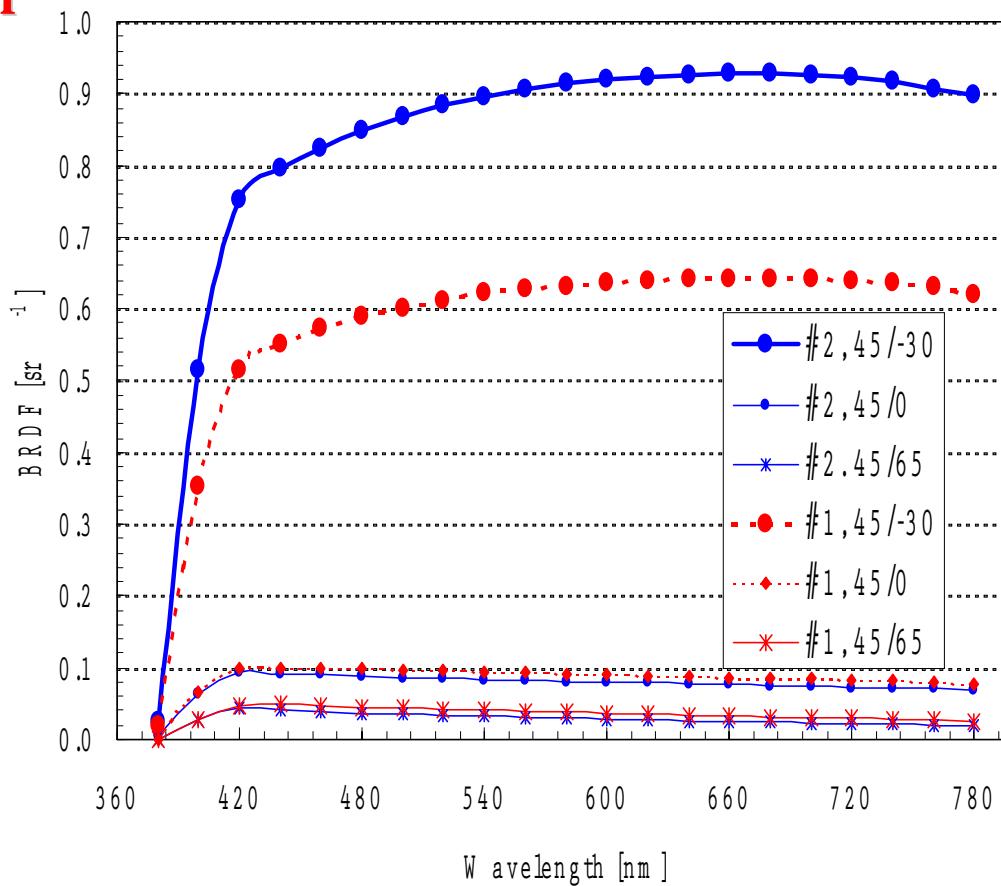
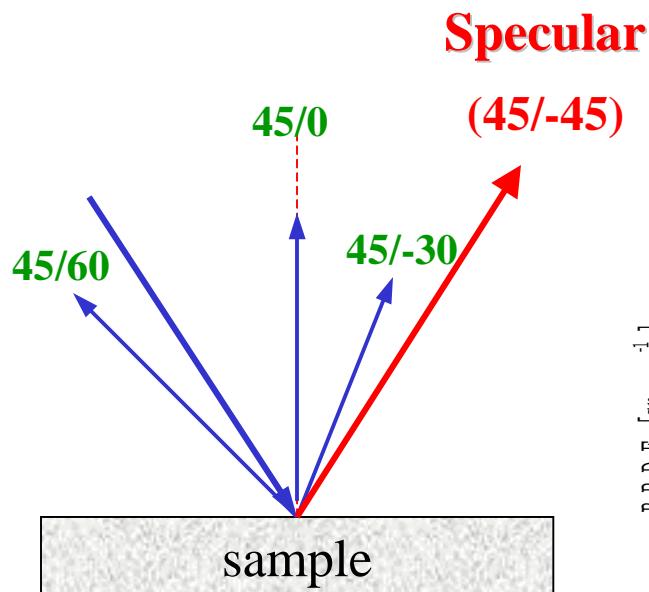


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Optical Reflectance Data – Using STARR (Maria Nadel)

Flop Angle Sample (Metallic Flake)

(varying degree of flop)



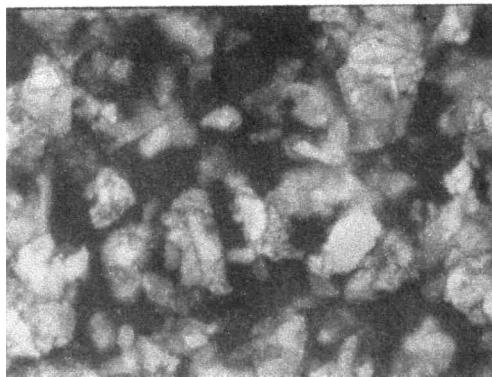
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Need to build a title stage for LSCM

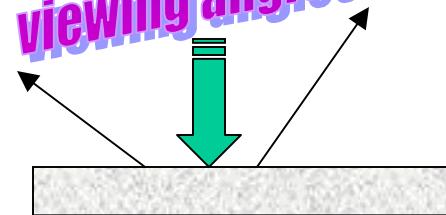
Microstructure of Pearlescent Coatings

LSCM Images of Green-Gold Bicolor Pearlcoat at Different Incident Laser Wavelengths

Optical Image

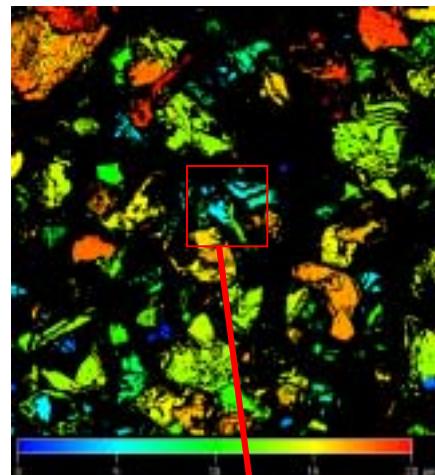


different lights
viewing angles

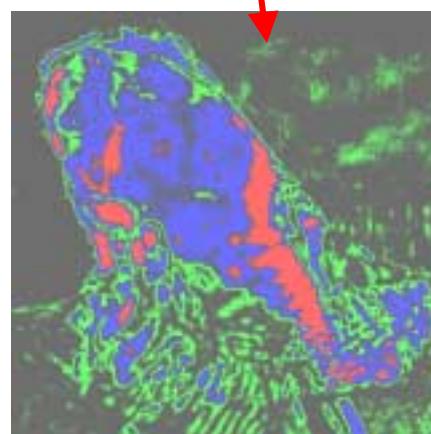


Optical Data will be
presented by
Maria Nadal

Green light (543 nm)

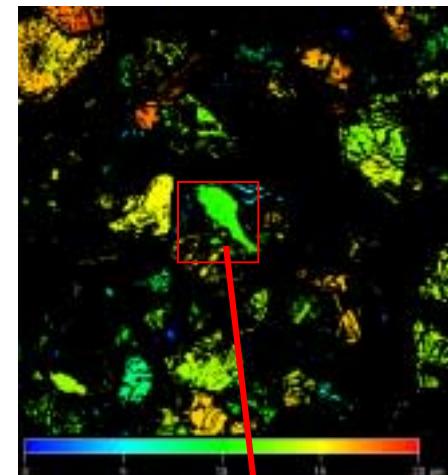


50 μm

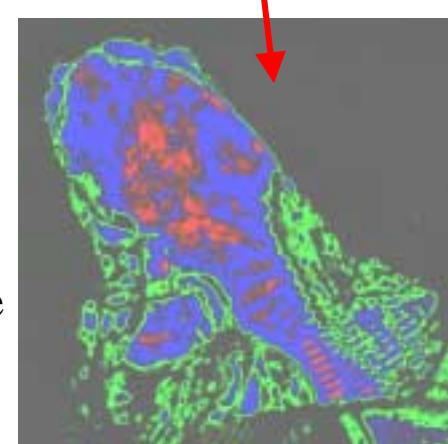


10 μm

Blue light (488 nm)



see more
pigments
under
green light

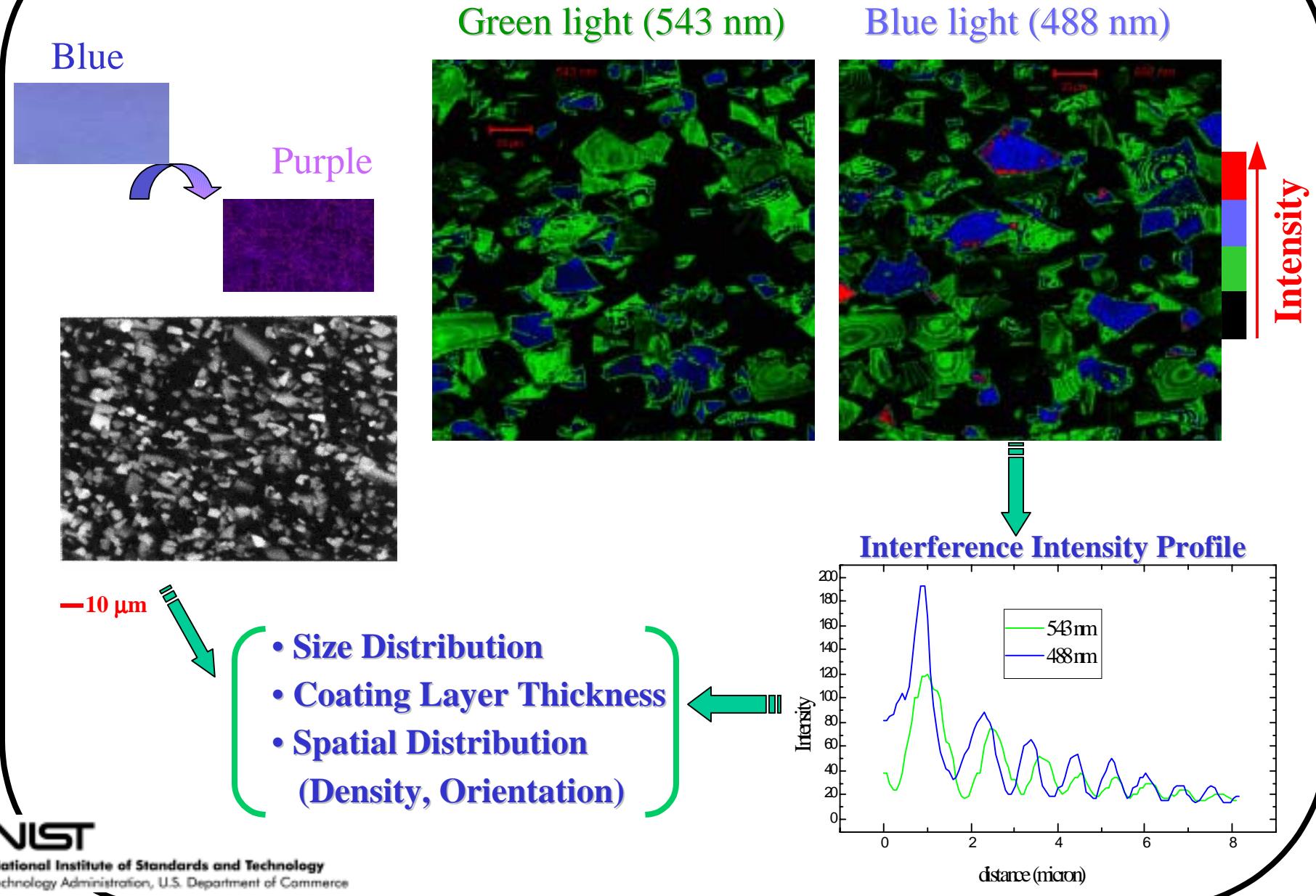


different
interference
fringes

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Microstructure of Light Interference Pigments



SUMMARY

- LSCM is powerful tool for characterizing coating microstructure
- Microstructure-reflectance links for modeling

