Why the University of Washington chose the HORIBA LA-950 Laser Scattering Particle Size Distribution Analyzer

Presented by Michelle Katz and Tiffany Tang

COLLEGE OF ENGINEERING

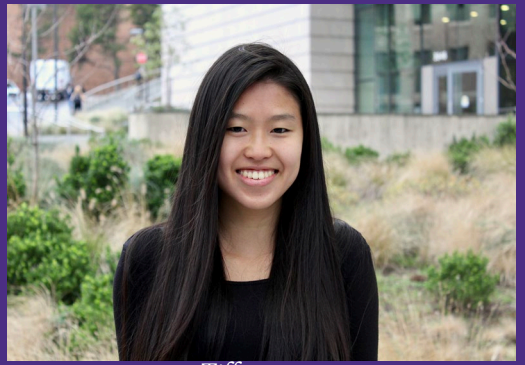
UNIVERSITY of WASHINGTON

AGENDA

- About us
- Why we chose the HORIBA LA-950
- Intro to Laser Scattering Particle Size Distribution Analysis (LSPA)
- How we use the HORIBA LA-950
  - Training users
  - Maintenance and Operation
- Complimentary Instruments
- Example analysis using tin oxide
- Concluding Remarks

OUR ROLE AT UW

- **Graduate students in Materials Science & Engineering (MSE)**
- **Lab Assistants in the MSE User Facility**
  - Instruments and equipment available for research and teaching
  - We maintain and train students/faculty to use these resources
- **Previously taught undergraduate classes involving the HORIBA LA-950**



Tiffany

Michelle

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OUR CRITERIA FOR CHOOSING AN INSTRUMENT

- Analysis software that is free to students
- Technical support after installation
- Representative of current state of the art
- Cost effective

THE HORIBA LA-950

water or suspension fluid

sample holder

drain

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SCIENCE OF LSPA & THE HORIBA LA-950

- Particles in water or solvent are hit with light
- Uses static light scattering (a.k.a. laser diffraction)
- Calculates particle size using angle and intensity of scattered light
  - Large particles: higher intensity and smaller angle
  - This calculation is dependent on refractive index
- HORIBA LA-950 particle size range: nm to mm

IMPORTANCE OF REFRACTIVE INDEX

- **Refractive index of many materials is already in software**
  - If not, can be found in literature
- **Size calculation uses Mie Scattering Theory**
  - Requires user to input refractive index
- **Refractive index is irrelevant for particles larger than ~30 microns**
  - The intensity of refracted light is much smaller than reflected and diffracted light

HOW WE USE THE HORIBA LA-950

- **Part of undergraduate instructional curriculum**
  - Students measure size of polymer spheres they learn to synthesize
  - Students measure size of ceramic materials they process in class
  - Students get hands-on experience from day 1
- **Used by research groups across UW**
  - Example: used by Geology Department to measure rock samples
- **Students can use for independent projects at no cost**

THE HORIBA LA-950 IS FASTER THAN…

- Sieving
- Gravity Settling
- Photo-sedimentation
- Electrical sensing zone techniques

The LA-950 is intuitive to use and has better repeatability.

TRAINING USERS

- **Mandatory 2-part training session & written test**
  - 1st session: we show them how to use instrument & software
  - 2nd session: they test their sample and demonstrate competence
  - Short written test to prove thorough understanding of SOP
- **We test samples for students occasionally**
  - Usually for one-time guests or short-term projects

OPERATION GUIDELINES

- Analysis software available for free download
- Reservation system: CORAL
  - Manages training, scheduling and user fees (for funded-research & industry)
- SOP & step-by-step instructions provided for reference
- Users may not troubleshoot, calibrate, or adjust the instrument in any way
  - Must contact us or our manager for assistance
- Non-hazardous materials only

MANAGING MULTIPLE SAMPLES

- Rinse cycle to clean sample holder
- Change suspension fluid if necessary
- Update the refractive index
- Take a new background measurement

COMMON CAUSES OF BAD DATA

- Surfactants can cause micro-bubbles
- Some materials are very prone to agglomeration
- *Agitation* can break particles into smaller pieces

PREVENTING AGGLOMERATION

- Surfactants increase the surface area of the solvent, but can also cause bubbles
- *Agitation, Circulation,* and *Ultrasonic* settings on LA-950 keep samples moving
- Also a *de-bubble* function

LIMITATIONS TO LSPA

- **Some materials do not work very well with LSPA**
  - Unstable nanoparticles
  - Magnetic particles
- **There is a minimum amount of material needed**
  - Especially a concern for small-batch synthesis
  - Specific amount of sample dependent on material properties; can be tricky to predict in advance

TECHNIQUES TO COMPLIPENT LSPA

- **X-ray Diffraction (XRD)**
  - Measures crystallite size (not particle size)
  - Provides insight into crystal structure
- **Optical Microscopy**
  - Provides insight into shape of large particles
- **Scanning electron microscopy (SEM)**
  - Provides insight into shape of small particles
  - Can confirm elemental composition

XRD AND SEM

← XRD

SEM →

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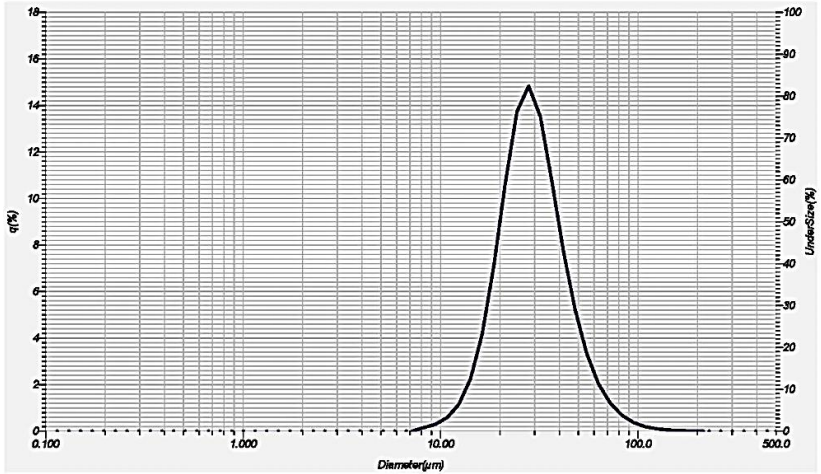
EXAMPLE: TIN (II) OXIDE WITH LSPA

2013.03.19 14:56:45

University of Washington-MSE

Laser Scattering Particle Size Distribution Analyzer LA-950

Sample Name	: Tin Oxide	Median Size	: 28.60883(μm)	Form of Distribution	: Auto
Data Name	: Average of 15	Mean Size	: 31.67864(μm)	Refractive Index (R)	: SnO[Stannous Oxide(1.840 - 0.000i),SnO
Transmittance(R)	: 95.3(%)	Std.Dev.	: 14.3838(μm)	Refractive Index (B)	: SnO[Stannous Oxide(1.840 - 0.000i),SnO
Transmittance(B)	: 96.5(%)	Geo.Mean Size	: 29.1207(μm)	Distribution Base	: Volume
Circulation Speed	: 15	Geo.Std.Dev.	: 1.4945(μm)	Span	: YES (10.00 - 90.00)
Agitation Speed	: 5	Span	: 1.0832		
Ultra Sonic	: 00:30 (7)	Diameter on Cumulative %	: (2)10.00 (%)- 17.8674(μr		
			: (9)90.00 (%)- 48.8557(μr		



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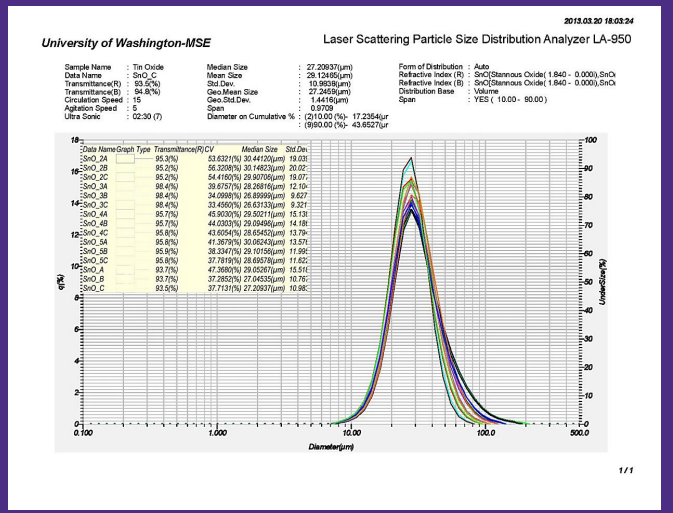

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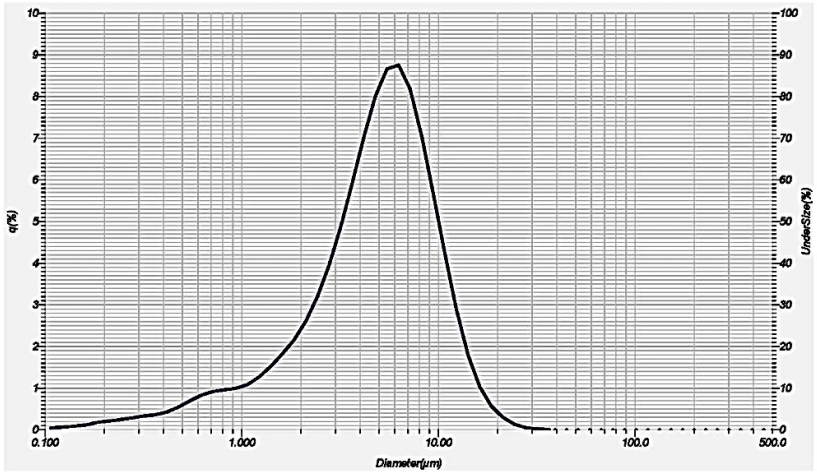
EXAMPLE: TIN (IV) OXIDE WITH LSPA

2013.03.19 14:59:28

University of Washington-MSE

Laser Scattering Particle Size Distribution Analyzer LA-950

Sample Name	: MSE 312 2013 Sr	Median Size	: 5.05755(μm)	Form of Distribution	: Auto
Data Name	: SnO2_Average of	Mean Size	: 5.62856(μm)	Refractive Index (R)	: Tin(IV) Oxide[Tin Dioxide(2.006 - 0.000i),V
Transmittance(R)	: 96.3(%)	Std.Dev.	: 3.7272(μm)	Refractive Index (B)	: Tin(IV) Oxide[Tin Dioxide(2.006 - 0.000i),V
Transmittance(B)	: 93.3(%)	Geo.Mean Size	: 4.2857(μm)	Distribution Base	: Volume
Circulation Speed	: 10	Geo.Std.Dev.	: 2.3371(μm)	Span	: YES (10.00 - 90.00)
Agitation Speed	: 5	Span	: 1.7905		
Ultra Sonic	: 00:48 (7)	Diameter on Cumulative %	: (2)10.00 (%)- 1.3602(μm		
			: (3)20.00 (%)- 2.4923(μm		



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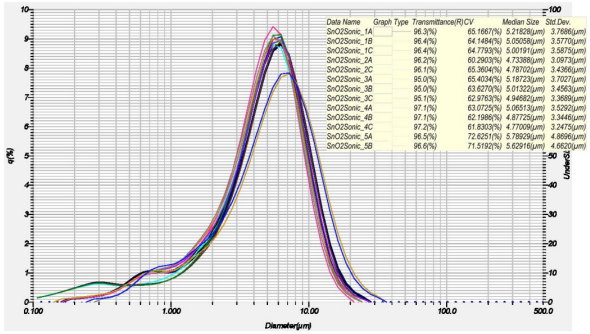
Laser Scattering Particle Size Distribution Analyzer LA-950

Sample Name	: MSE 312 2013 Sr	Median Size	: 5.62916(μm)	Form of Distribution	: Auto
Data Name	: SnO2Sonic_5B	Mean Size	: 6.51856(μm)	Refractive Index (R)	: Tin(IV) Oxide[Tin Dioxide(2.006 - 0.000i),V
Transmittance(R)	: 96.6(%)	Std.Dev.	: 4.6620(μm)	Refractive Index (B)	: Tin(IV) Oxide[Tin Dioxide(2.006 - 0.000i),V
Transmittance(B)	: 94.2(%)	Geo.Mean Size	: 4.8805(μm)	Distribution Base	: Volume
Circulation Speed	: 10	Geo.Std.Dev.	: 2.3161(μm)	Span	: YES (10.00 - 90.00)
Agitation Speed	: 5	Span	: 1.9734		
Ultra Sonic	: 02:48 (7)	Diameter on Cumulative %	: (2)10.00 (%)- 1.4312(μm		
			: (3)20.00 (%)- 2.5815(μm		



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EXAMPLE: TIN (IV) OXIDE WITH LSPA

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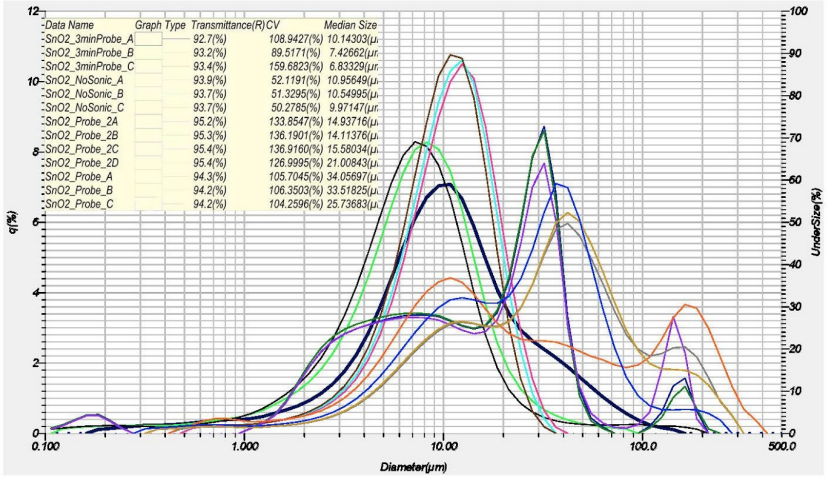
Laser Scattering Particle Size Distribution Analyzer LA-950

Sample Name	: MSE 312 2013 Sr	Median Size	: 25.73683(μm)	Form of Distribution	: Auto
Data Name	: SnO2_Probe_C	Mean Size	: 33.98009(μm)	Refractive Index (R)	: SnO2[SnO2 G10R2(2.000 - 0.000i),Water
Transmittance(R)	: 94.2(%)	Std.Dev.	: 35.4379(μm)	Refractive Index (B)	: SnO2[SnO2 G10R2(2.000 - 0.000i),Water
Transmittance(B)	: 91.7(%)	Geo.Mean Size	: 21.0276(μm)	Distribution Base	: Volume
Circulation Speed	: 10	Geo.Std.Dev.	: 2.9733(μm)	Span	: YES (10.00 - 90.00)
Agitation Speed	: 5	Span	: 2.3651		
Ultra Sonic	: 04:00 (7)	Diameter on Cumulative %	: (2)10.00 (%)- 5.4098(μm		
			: (9)90.00 (%)- 66.2801(μm		



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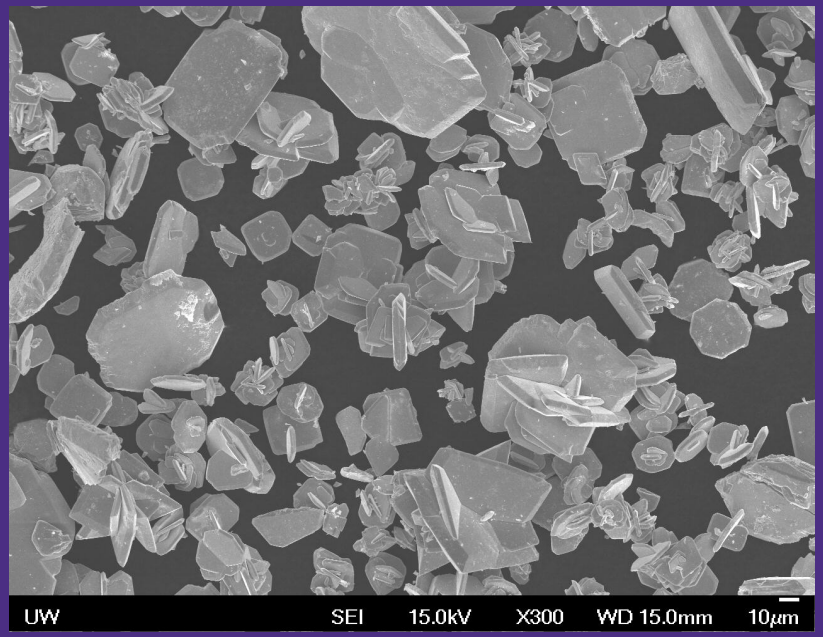
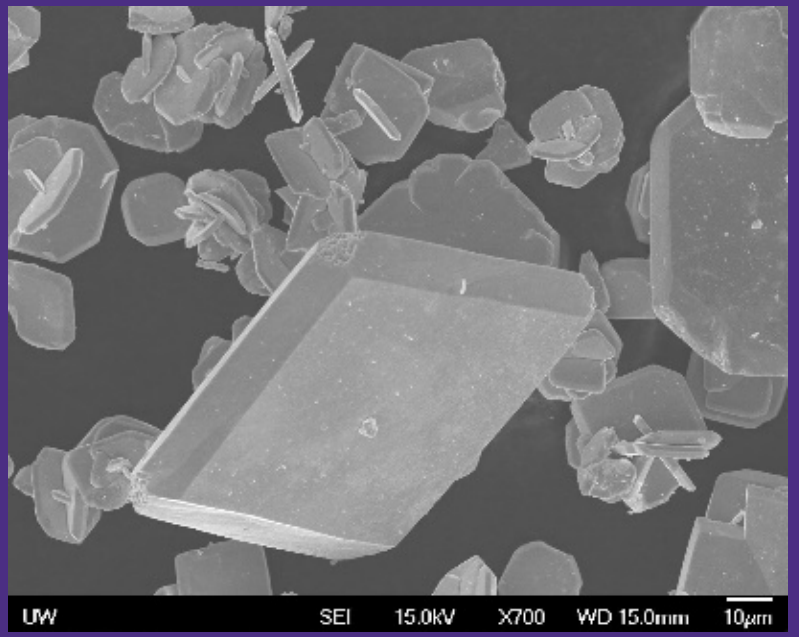
TIN (II) OXIDE SEEN WITH SEM





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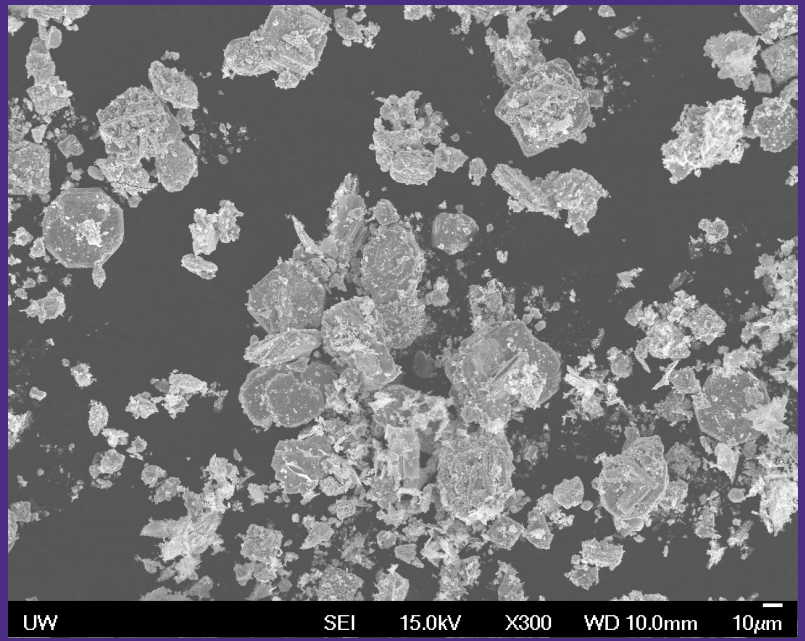
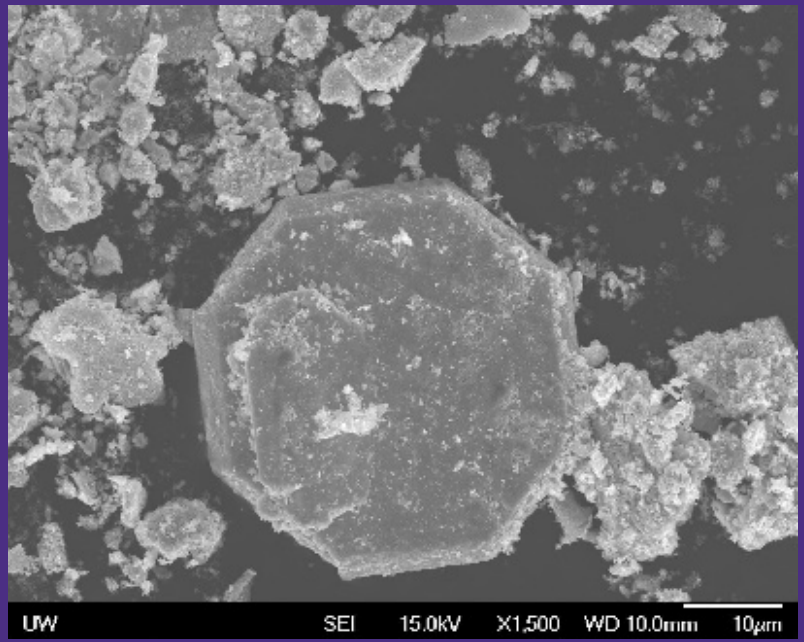
TIN (IV) OXIDE SEEN WITH SEM





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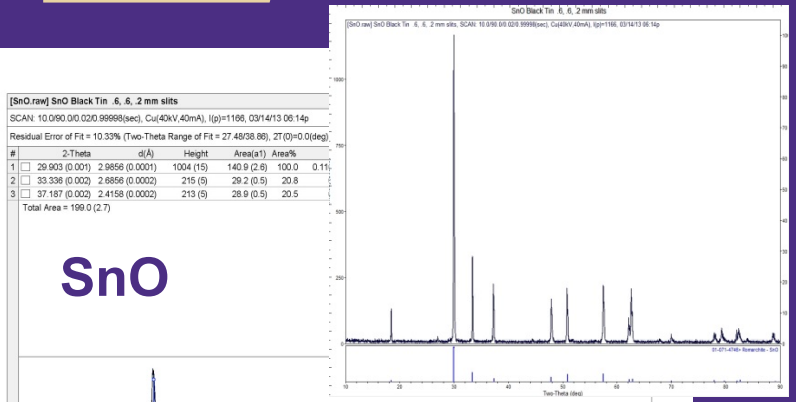
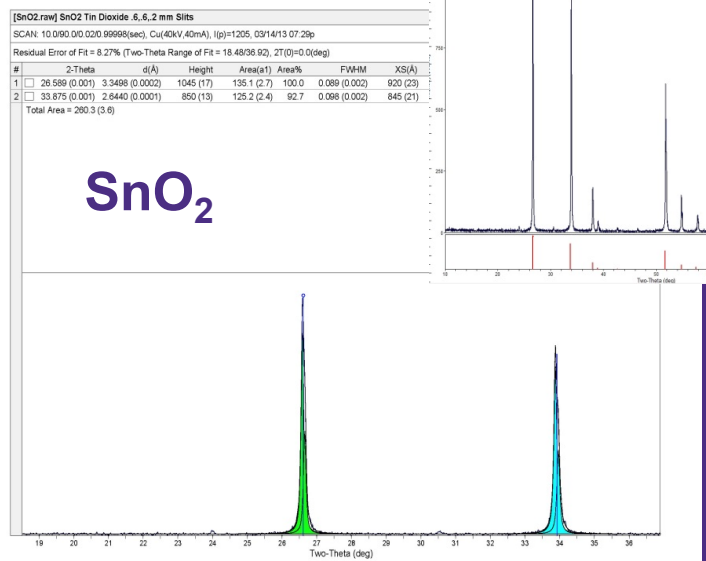
CONFIRMATION WITH XRD

- XRD can determine crystal structure
- Scherrer's equation estimates crystallite size
  - Uses full-width half-max of spectra peaks
- Crystallite size is different than particle size

CONFIRMATION WITH XRD





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FINAL REMARKS

- HORIBA LA-950 is user-friendly for all experience levels
- It is a multi-purpose tool to enhance education and research
  - Much faster and more reliable than many other methods
- Pairs well with other characterization methods
  - XRD , OM, SEM

HORIBA LA-950 vs. LA-960

Sept. 2007 – Dec. 2014
0.01 - 3000μm for wet
0.1-3000μm for dry

July, 2013
0.01 - 3000μm for wet
0.1-5000μm for dry
Improved user interface

CELL OPTIONS

Standard cell:

up to 3000µm

Cell option:

up to 5000µm

Specific Dry cell for size higher than 3mm

Free fall nozzle inside, for larger opening

Labinfo@horiba.com

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