Microtrac
Total Solutions in Particle Characterization

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Microtrac Company History

1970 – 1974 Leeds and Northrup
Research into new applications of laser industrial products. **Release of first commercially available Particle Size Analyzer using laser diffraction in 1974.**

1974 - 1996
Microtrac commercially produced by Leeds and Northrup until 1996 when **Honeywell** purchased Leeds and Northrup.

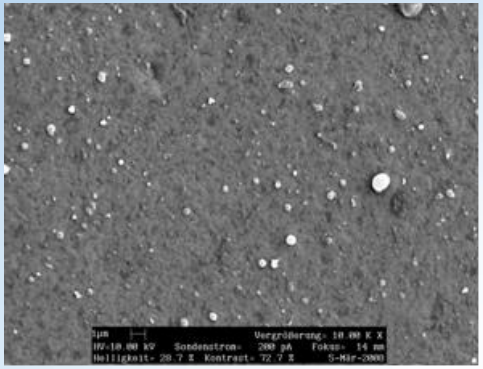
2000
Microtrac product line acquired by **Nikkiso** ($1.2 bn turnover) to form Microtrac Inc.

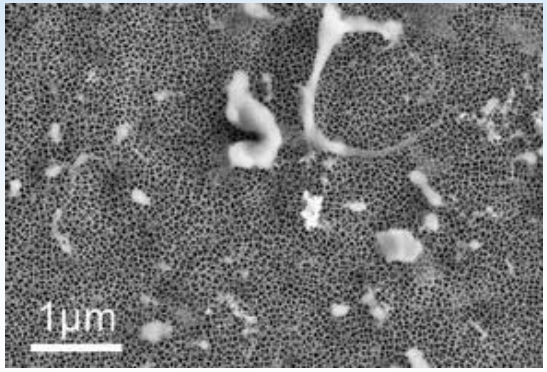
2015
Foundation of **Microtrac GmbH**, Krefeld, Germany, to foster EMEA region.

>45 Years Light Scattering Experience and Technology

Particle characterization in tribology

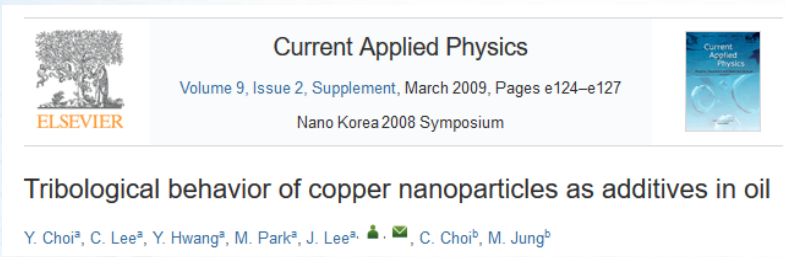
- Measure Particles from abrasion (wear)



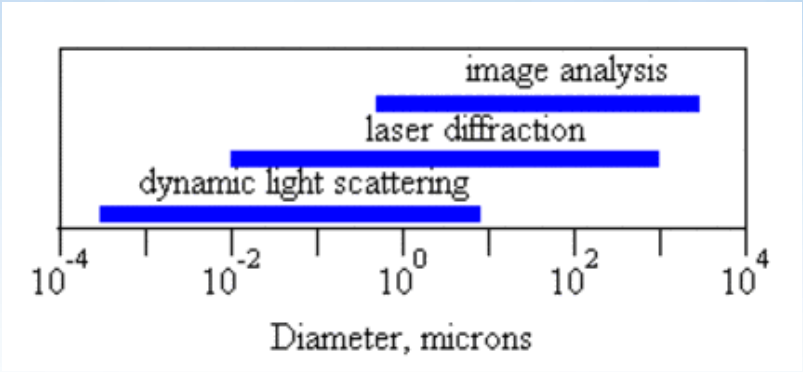


Abrasion particles from orthopaedic implants, REM images,
a) 1-20 µm b) 0.1-3 µm
(source: DGOU / MHH website)

- Measure deliberately added particles (additives)



Some Particle Sizing techniques



- Dynamic Light Scattering (DLS) – Brownian Motion → Doppler Effect (Frequency Shift) of scattered light. ISO recommendation 1nm-1µm

- Laser diffraction granulometry – angle-dependent scattering (a form of Static light scattering). ISO recommends 100 nm-3mm for red lasers and blue lasers to extend <100nm

- Image Analysis – Optical Microscopy (static or Dynamic)

Microtrac Solutions Overview

0.3 nm — Size — 127 mm

ZP and MW

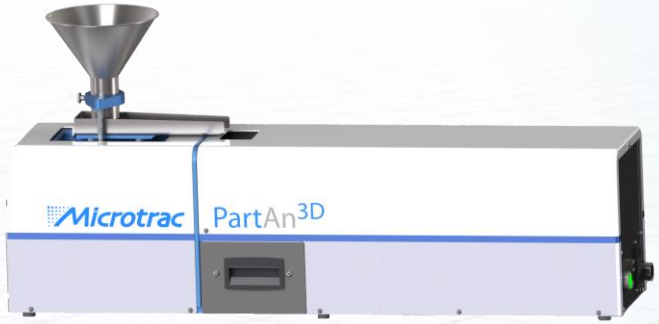
Shape/Morphology

Surface Area/ Porosity

Pioneers of Particle Characterization

Microtrac Technologies

- Dynamic Light Scattering – Nano-Materials 0.3 nm to 10 µm
- Laser Diffraction – Particle Size Analysis 10 nm to 2.8 mm
- Image Analysis – Particle size and shape analysis 1 µm – 127 mm
- Surface Area/Adsorption – Gas and Chemisorption, BEL products



Microtrac/BEL Surface Area Analysis Portfolio

Adsorption Analysis

- Strategic Alliance between Microtrac and BEL Belsorp Mini and Max –Surface Area and Pore Size Analysis
- BELCAT – Catalyst applications
- Custom built Adsorption and Chemisorption solutions
- manufacturing in US and Japan.

Patented AFSM technology

AIR LIQUIDE™

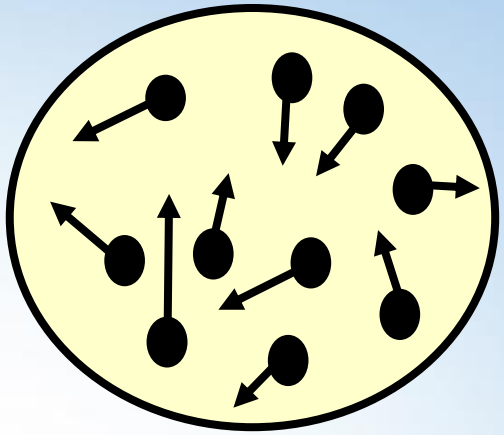
A MEMBER OF NIKKISO

NIST

Microtrac
Total Solutions in Particle Characterization

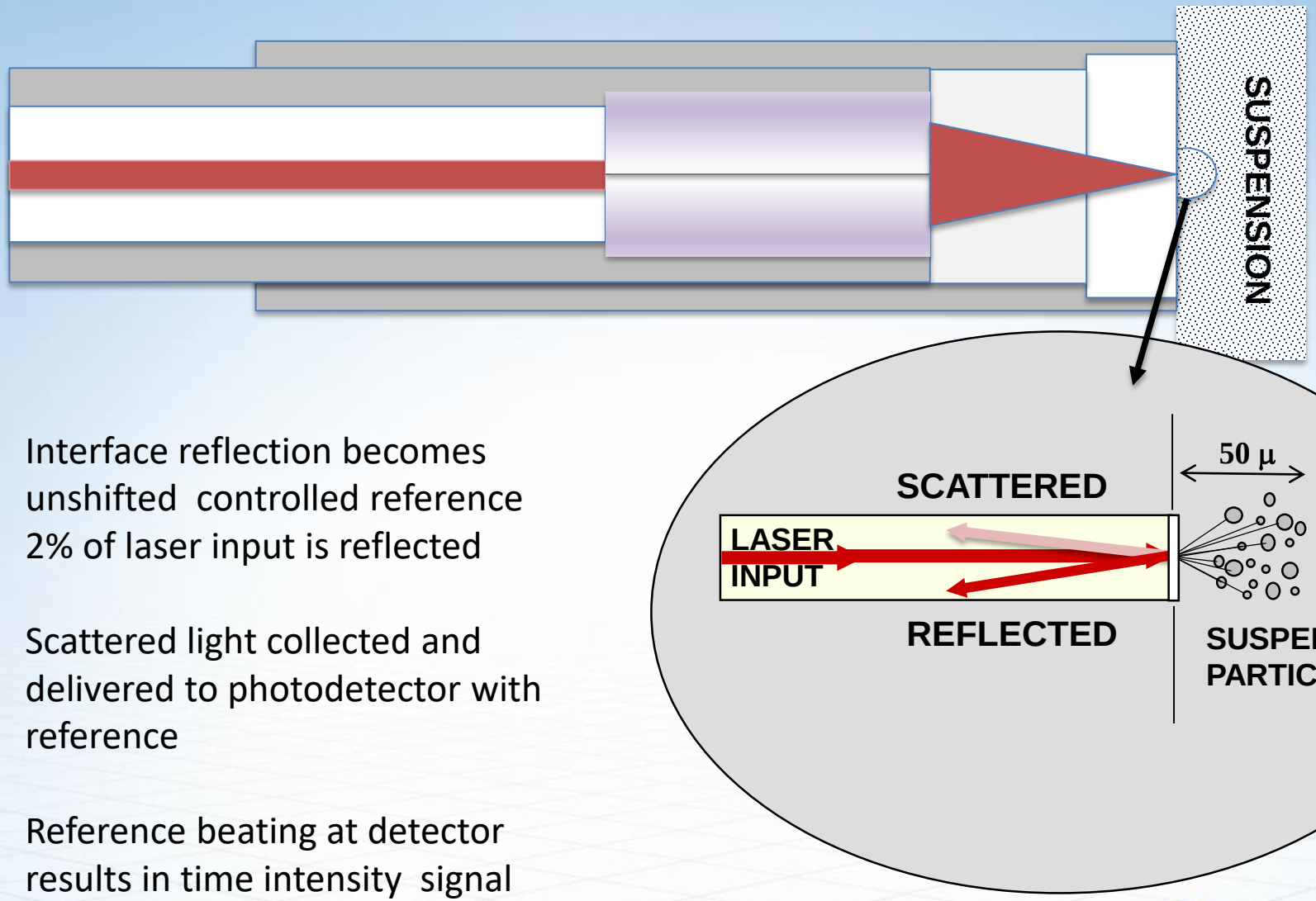
Dynamic Light Scattering – concept

- Suspended particles exhibit brownian motion
- Velocity distribution of particles determined by particle size
- Light scattered from moving particles measures particle velocity distribution



THE SOLUTION FOR NANOMETER PARTICLE SIZING

Controlled Reference – signal amplification for low concentrations

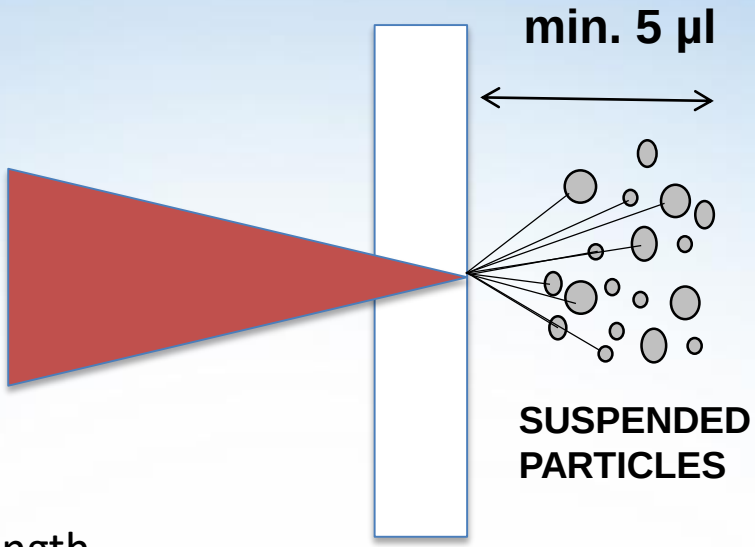


Interface reflection becomes unshifted controlled reference 2% of laser input is reflected

Scattered light collected and delivered to photodetector with reference

Reference beating at detector results in time intensity signal

180° detection angle for High Concentrations



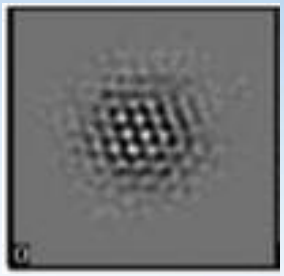
- 180° backscatter
- Minimum laser path length
- Minimum multiple scattering

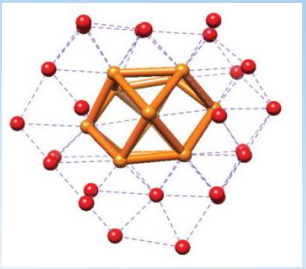
→ Highest possibly concentrations with 180° backscattering

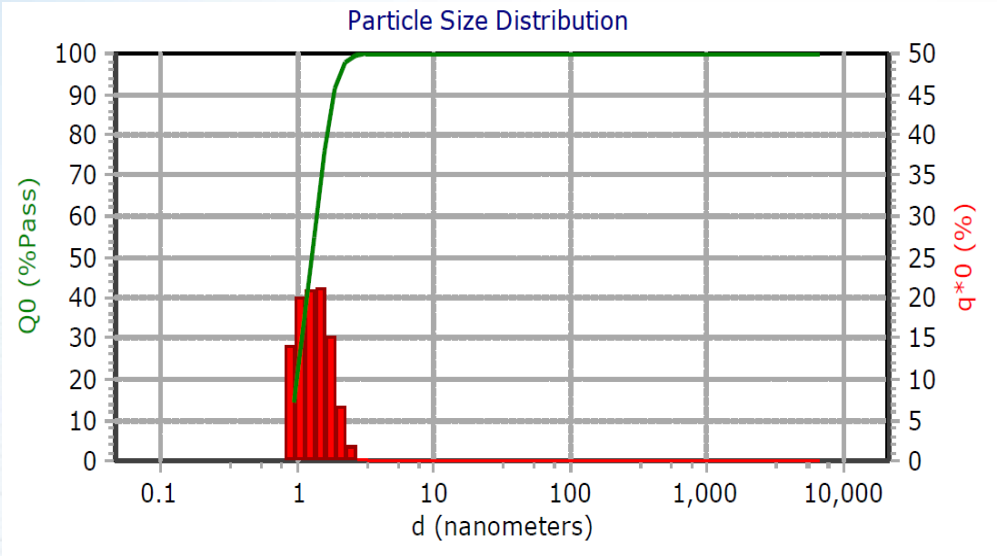
Limit: Free Brownian Motion of particles (beyond 10-40% w/v)

Lower limit of detection: Practical test

Au_{68} gold nanocluster of 68 gold atoms
d=1.6nm







Peaks Summary		
d(nm)	Vol%	Width
1.36	100	0.7

Measurable down to 0.1% w/v

Microtrac's Dynamic Light Scattering Portfolio

Dynamic Light Scattering

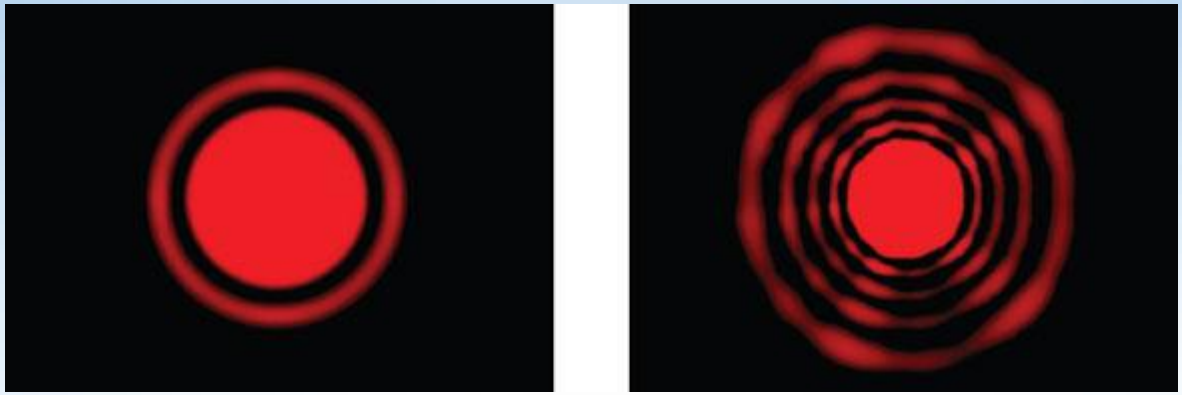
Particle Size Distribution Analysis – 0.3 to 6500 nm

- Nanotrac Wave II
- Nanotrac Wave II Q
- NANO-flex
  - Zetrator

- Patented Controlled Reference Method
- Unique Probe Technology
- Heterodyne signal processing (Frequency Shift Analysis instead of PCS
- wide concentration measurements
- Zeta Potential measurements with titration (up to 5 pumps)
- Molecular Weight – Hydrodynamic and Debye plot
- Concentration determination
- Temperature Control
- Auto sampling option

Particle Size integration into reactors - in-line

Laser diffraction - concept



- Large particles result in a high scattering intensity at relatively narrow angles to the incident beam
- smaller particles produce a lower intensity signal at much wider angles
- Laser diffraction analyzers determine particle size from the recorded angular dependence of the intensity of light scattered by a sample, using an appropriate theory of light behavior

Microtrac's Laser Diffraction Portfolio

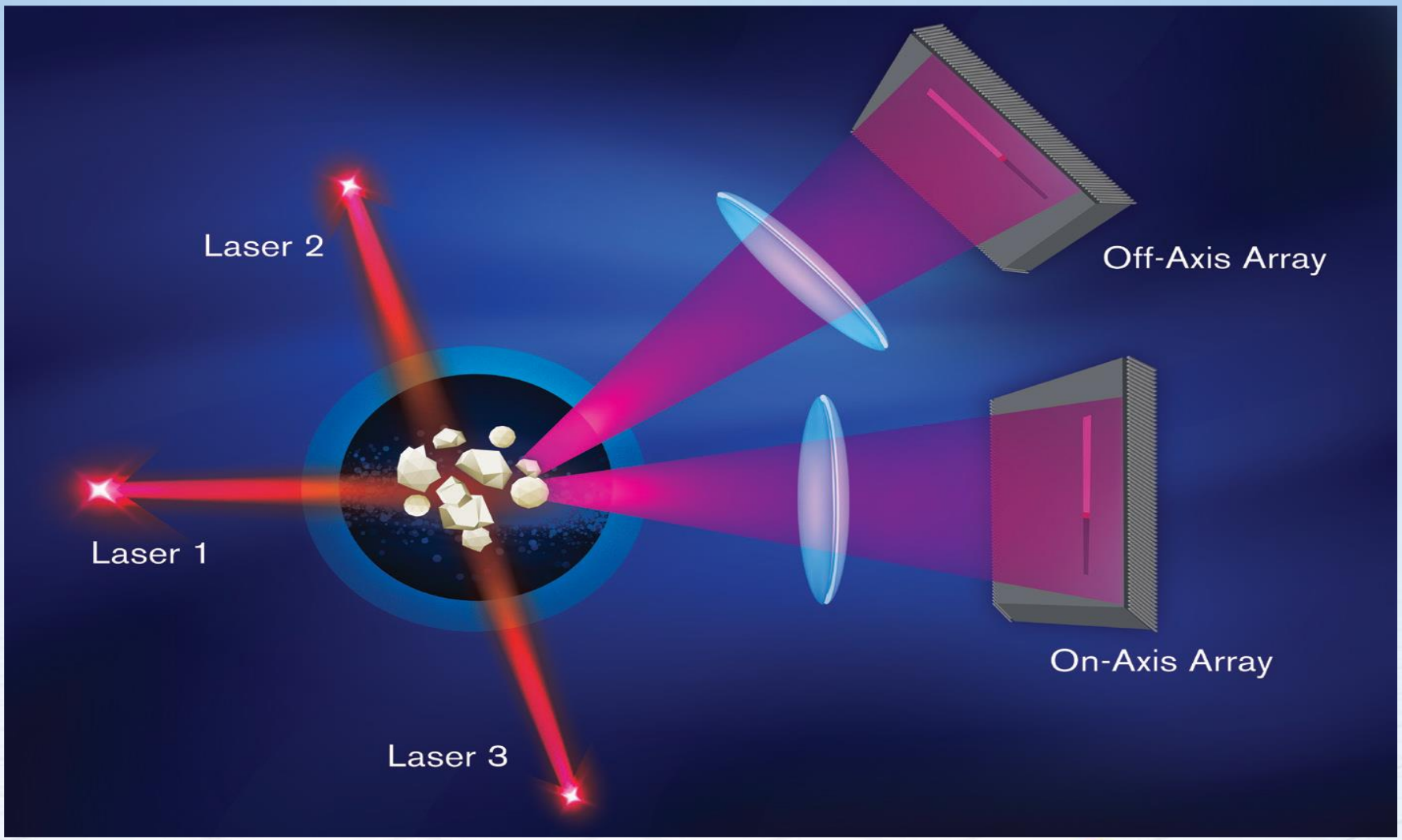
Laser Diffraction

Particle Size Distribution Analysis – 0.01 to 2800 microns

- S3500
- Bluewave
- TRI-BLUE
  - Turbotrac
  - SDC
  - USVR

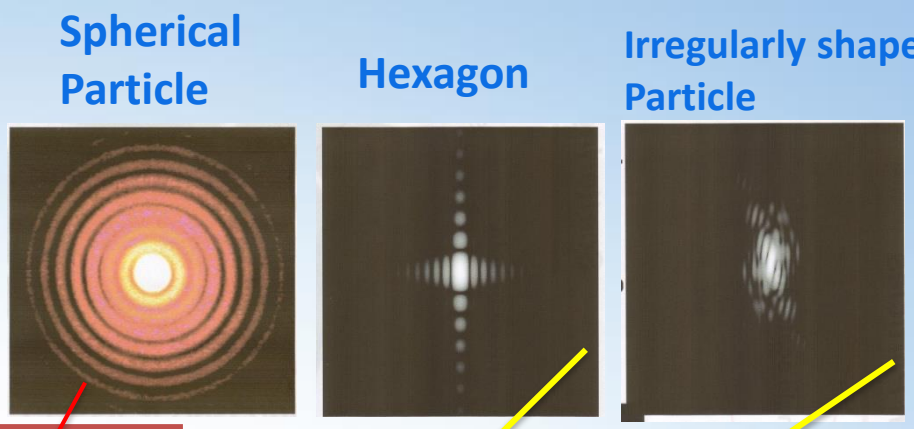
- Wet and Dry mode
- Patented Tri Laser Systems
- Use of coherent lasers (red and blue)
- Unique non-spherical measurement algorithm
- Integrated Image Analysis – size and shape

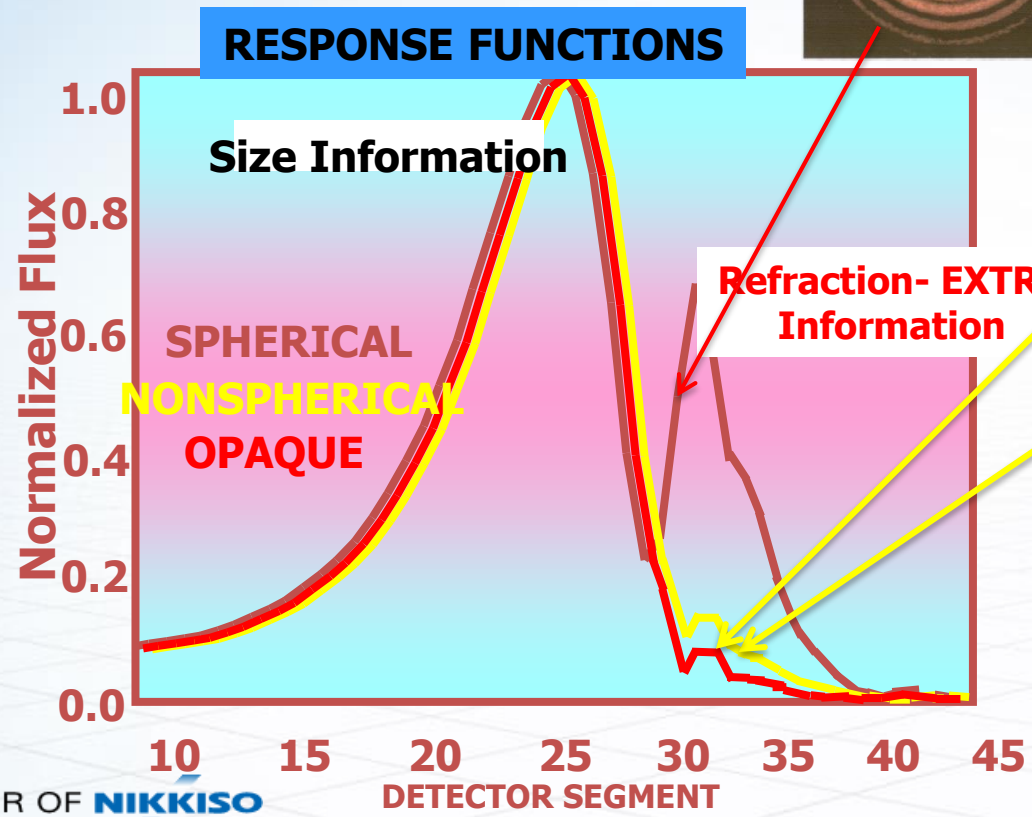
Microtrac's Patented 3-Laser Design – Detection 0.02-163°



Modified Mie – Patented By Microtrac

More than 95% of all particles are NOT spherical





Diffraction lower size limit – Bluewave&Triblue

405 nm Blue Lasers Provide for . . . .

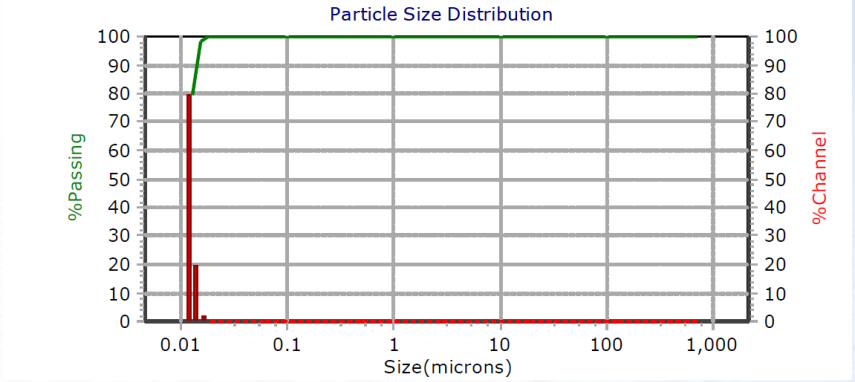
- High Intensity → High Signal Processing
- Coherent Light Sourdce → Accuracy
- 10X Greater Submicron → ttering Efficiency
  - → High Sensitivity
- Detection up to 163°
  - → Accuracy well below 100 nm

12 nm Ludox sample

Summary	
Data	Value
MV(um):	0.01210
MN(um):	0.01190
MA(um):	0.01200
CS:	498.1
SD:	0.00095
Mz:	0.01205
σ1:	0.00098
Ski:	0.2727
Kg:	1.052

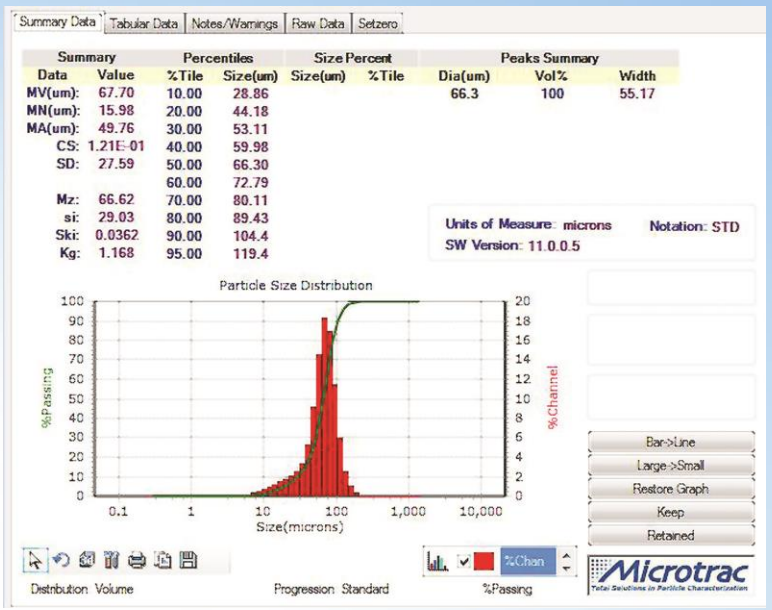
Percentiles	
%Tile	Size(um)
10.00	0.01100
20.00	0.01130
30.00	0.01150
40.00	0.01170
50.00	0.01190
60.00	0.01220
70.00	0.01250
80.00	0.01280
90.00	0.01350
95.00	0.01420

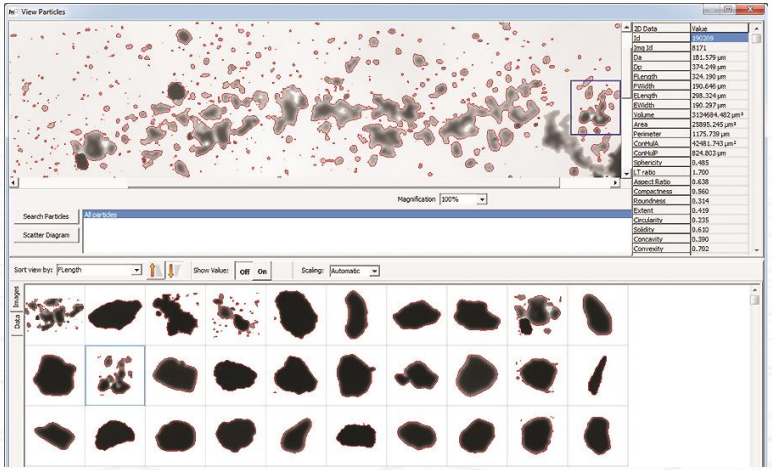
Peaks Summary		
Dia(um)	Vol%	Width
0.0119	100	0.00191



Integrated Laser Diffraction With Dynamic Image Analysis

- Visually validate your material
- Identify variations = proactive
- >25 morphological parameters
- LD – Size 10 nm to 2800µm
- DIA – Morphology – 3 to 2000µm





Concept in Image Analysis

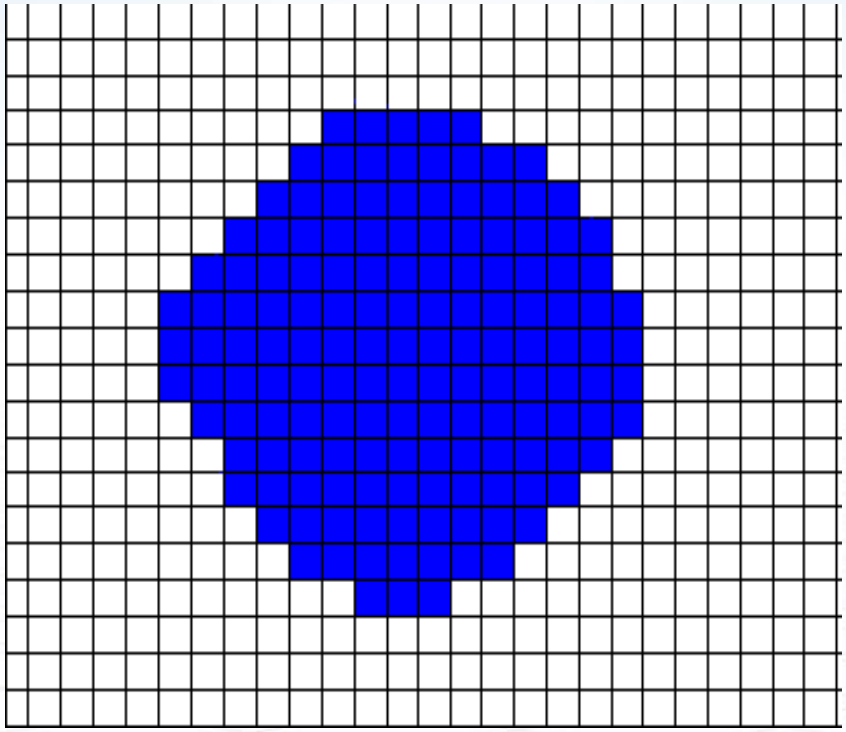
- *Photos* of individual particles are digitized, stored in a viewable image file
- Based on the size of a pixel, dimensions are measured
- Further calculations are performed for characterization
- Static IA – Microscopy
- Dynamic IA – Particles in motion

How Does It Work?

Particle detection – 1 micron pixel

Area = Count the pixels or portions covered by the particle shadow.

Perimeter = Measure along the sides



Microtrac's Morphological Parameters

Size

Da
Dp
FLength
FWidth
FThickness
ELength
EWidth
EThickness
Area
Volume
Perimeter
Surface Area
CHull Area
CHull Perimeter
CHull Surface Area

Form

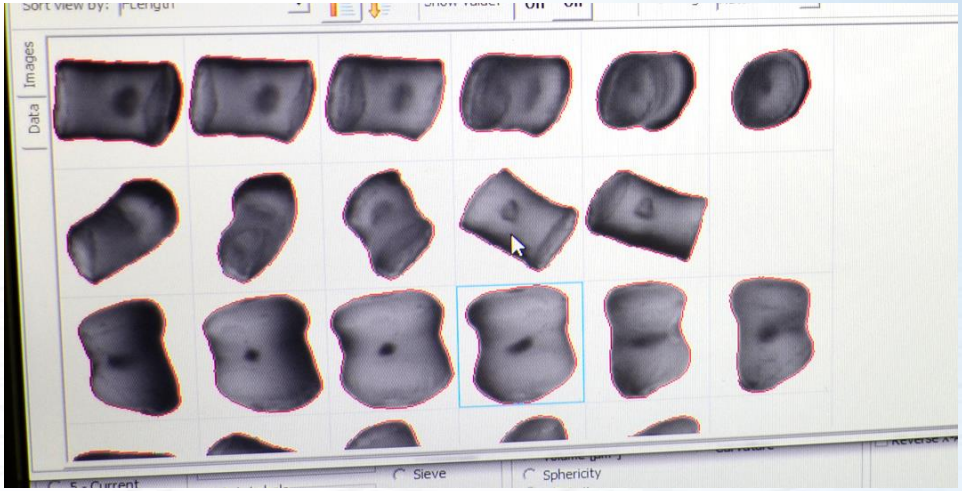
Sphericity
Circularity
Angularity
Roundness
Compactness
Extent
Ellipse Ratio
W/L Aspect Ratio
T/L Aspect Ratio
L/T Ratio
L/W Ratio
T/W Ratio
W/T Ratio

Surface Roughness

Convexity
Solidity
Concavity

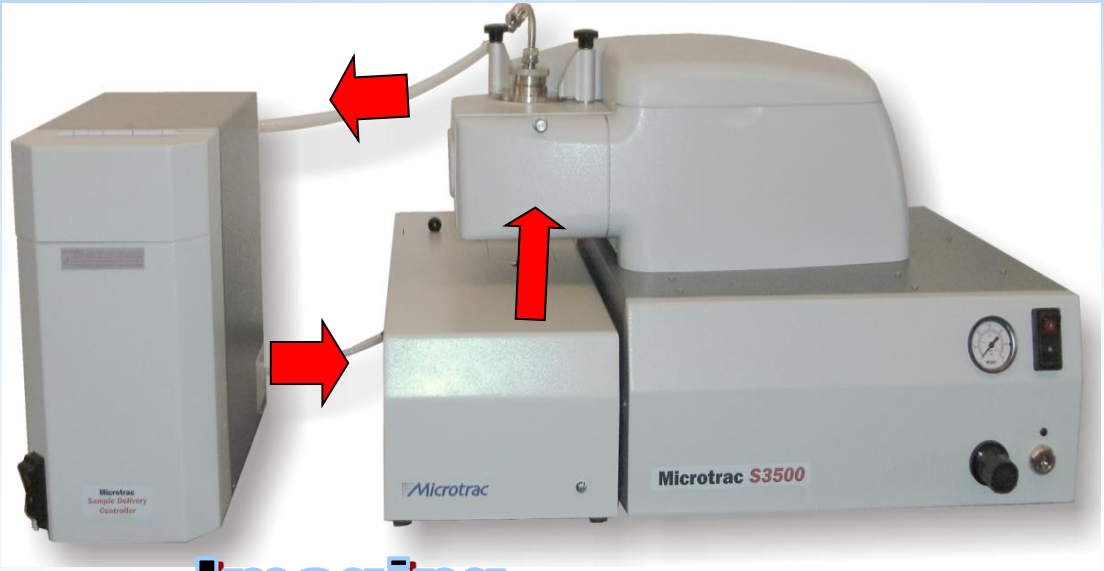
Other

Transparency
Curvature
ID
Img ID



Microtrac's PartAn-SI For Wet Measurements

SDC

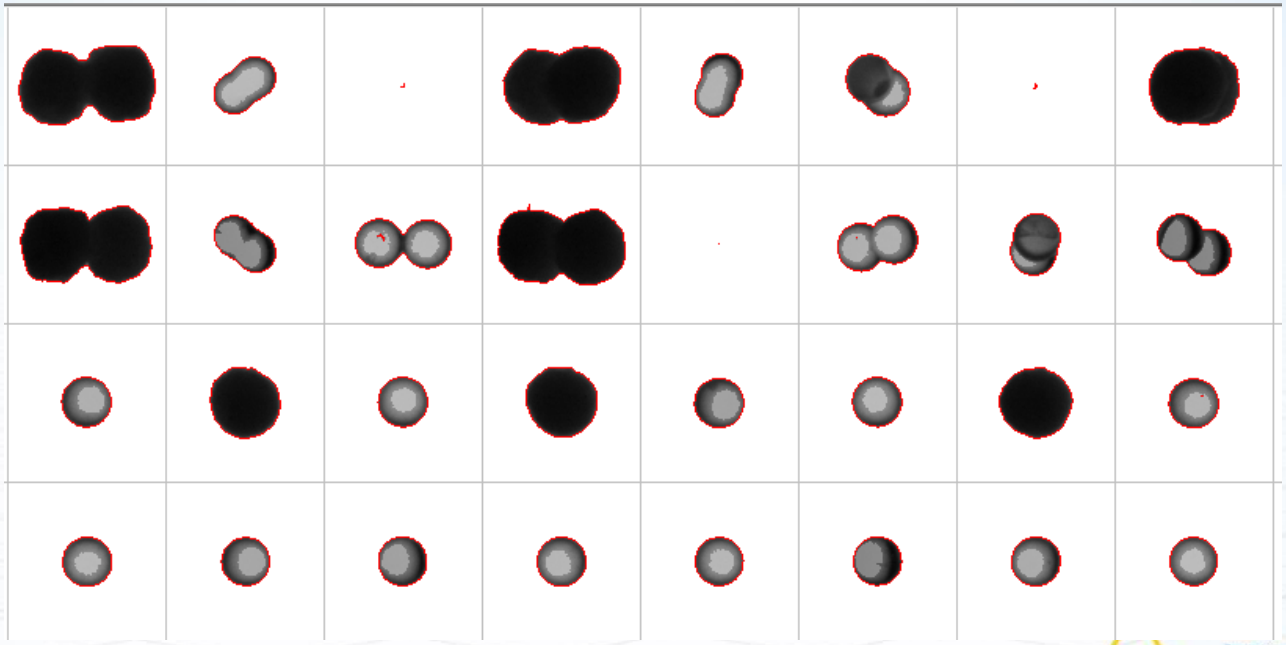


S3500 or Bluewave

Imaging SI

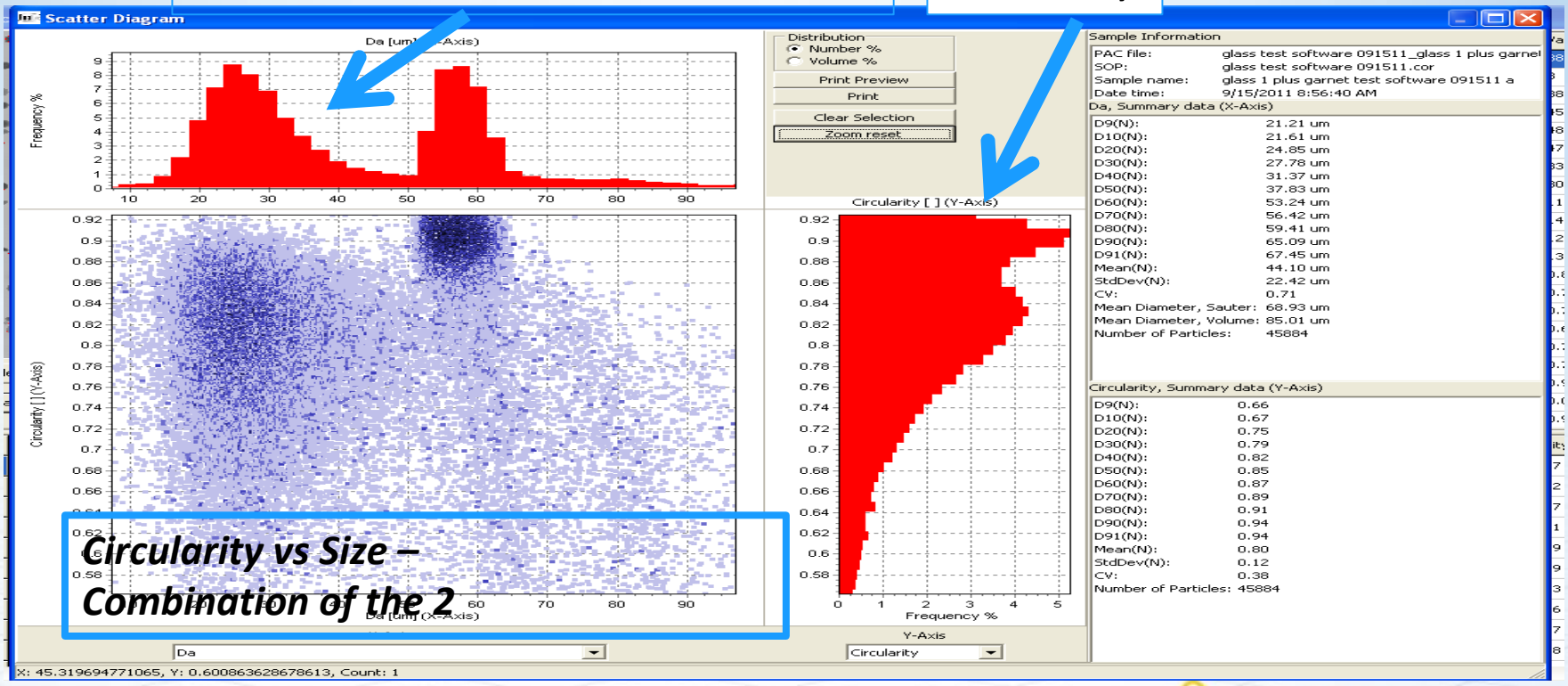
PartAn SI modular design allows connection to any S3500, Bluewave or TRI-BLUE

Visual Validation – Glass Beads Application



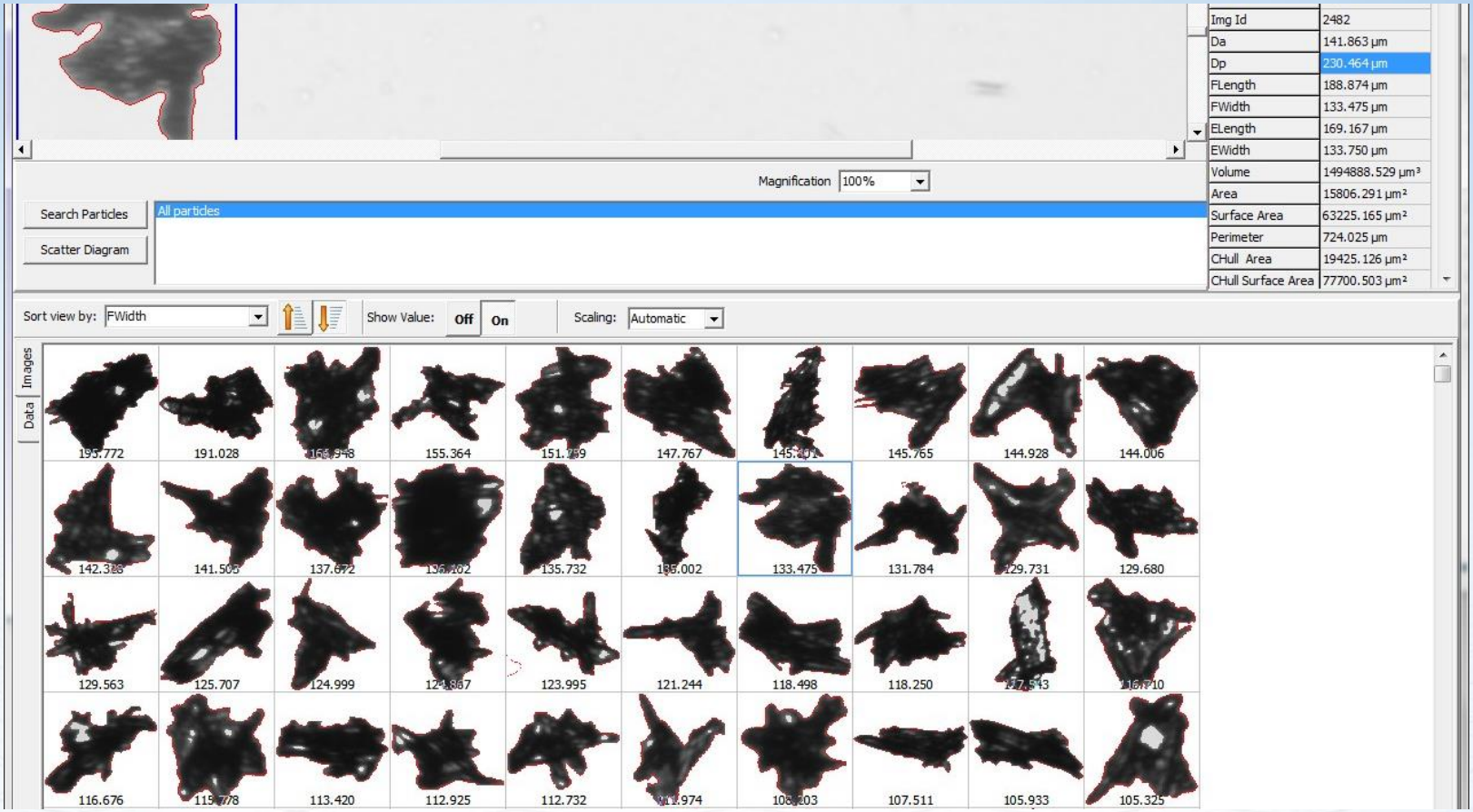
Scattergram And Other Graphs

1. Diffraction data show change in size or distribution.
2. Start SI Unit to evaluate for particle shape changes – aspect ratio, circularity., maximum length, minimum length, Legendre dimensions, etc.



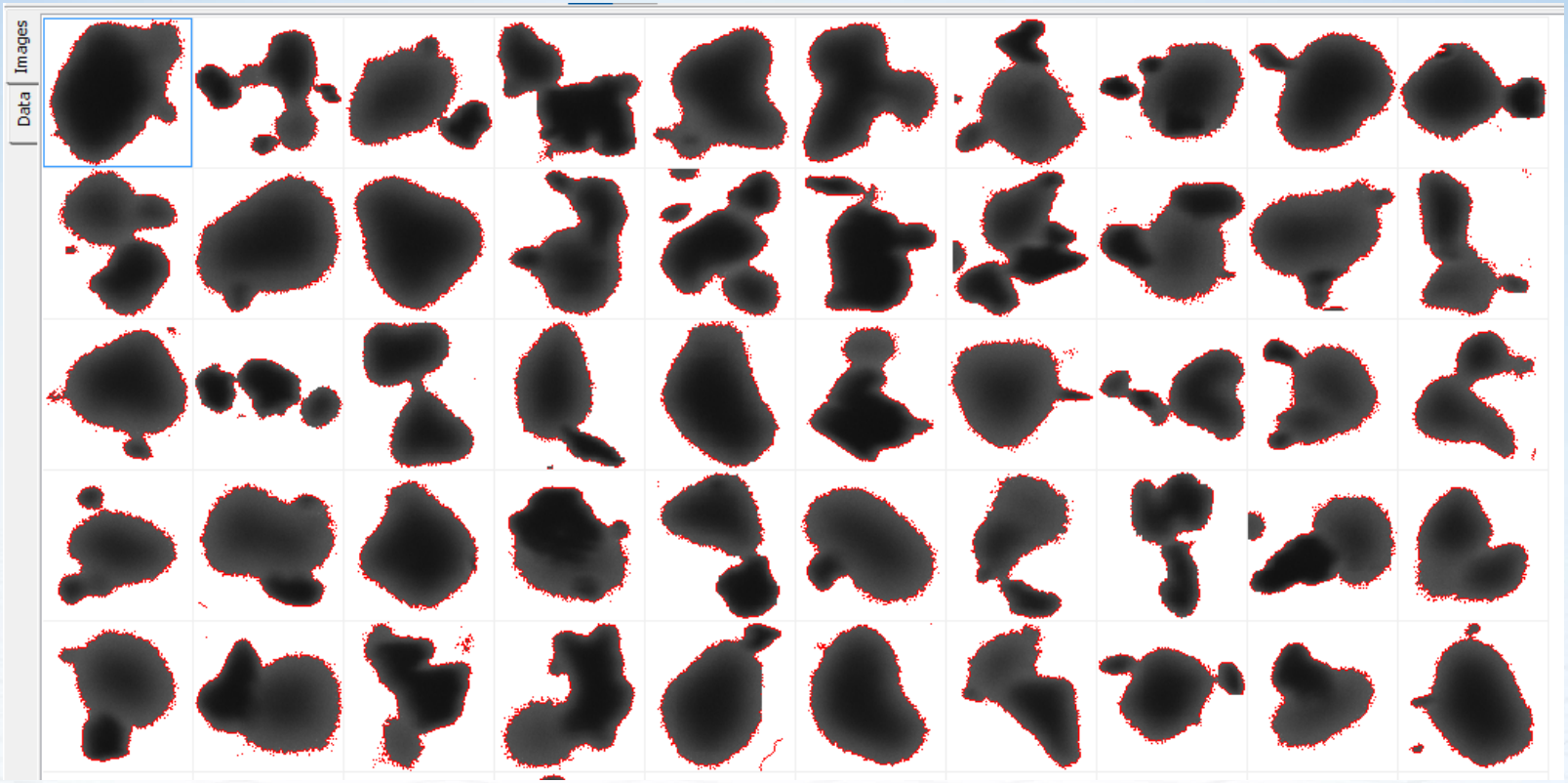
Benefits of Integrated Dynamic Image Analysis

- Crystals



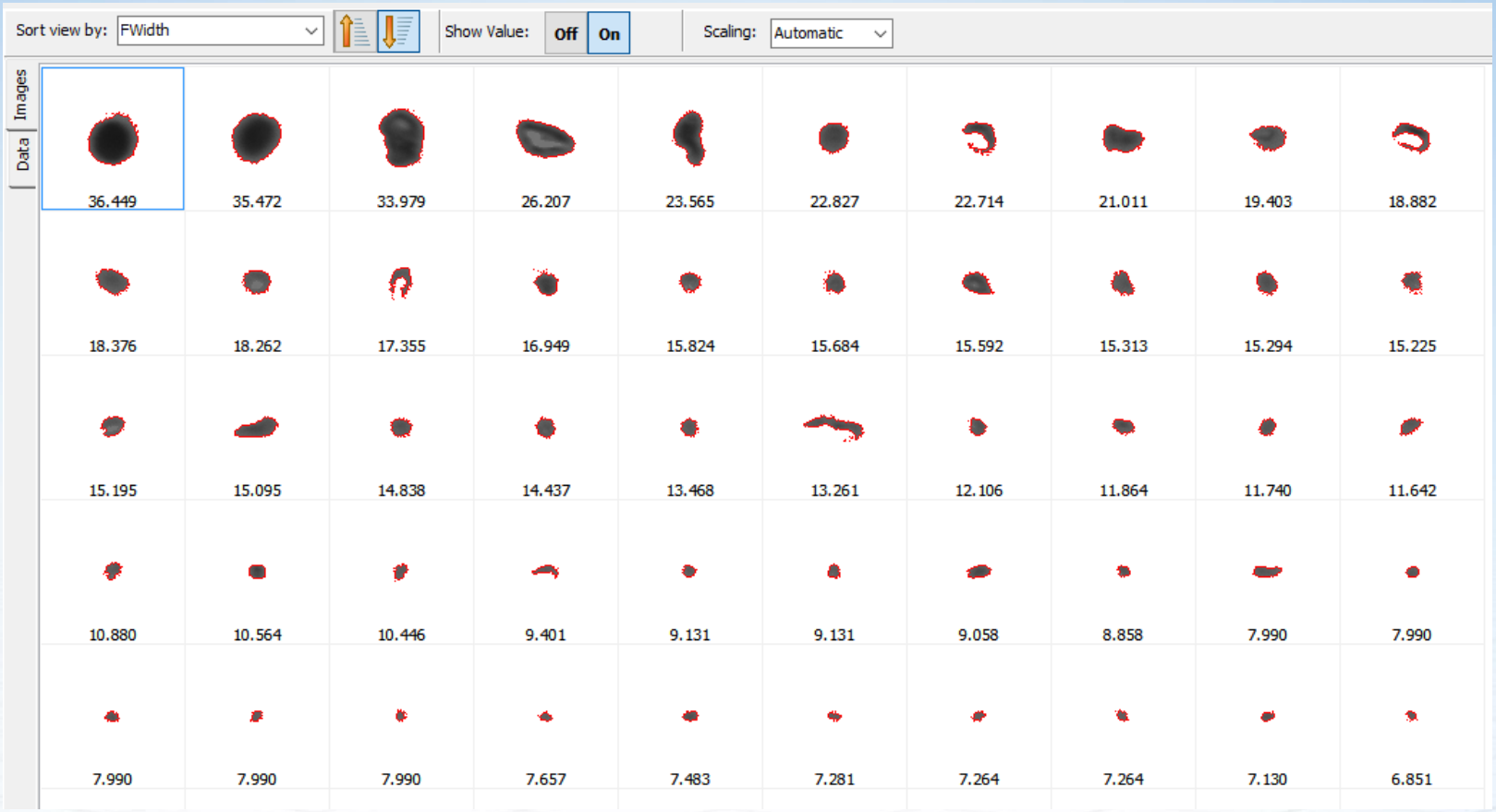
Benefits of Integrated Dynamic Image Analysis

- Agglomerates



Benefits of Integrated Dynamic Image Analysis

- Over-Grains



Microtrac PartAn SI PRO Online

Selected users

Microtrac's unique technologies

- Nanometer: Superior DLS technology (Frequency Power Spectrum) in 180° with Reference Beating instead of PCS. In-line / On-line capability.

- Nano-/Micrometer: Simultaneous combination of laser diffraction with quantitative Dynamic Imaging

- Micro-/Millimeter: Patented 3D imaging with >30 morphological parameters

- High-end BET analysis for surface analysis ("BEL")

Microtrac business fields

- Instrument sales
- Contract analysis
- Training in particle characterization
- Partner in F&E projects / grants

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