



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.

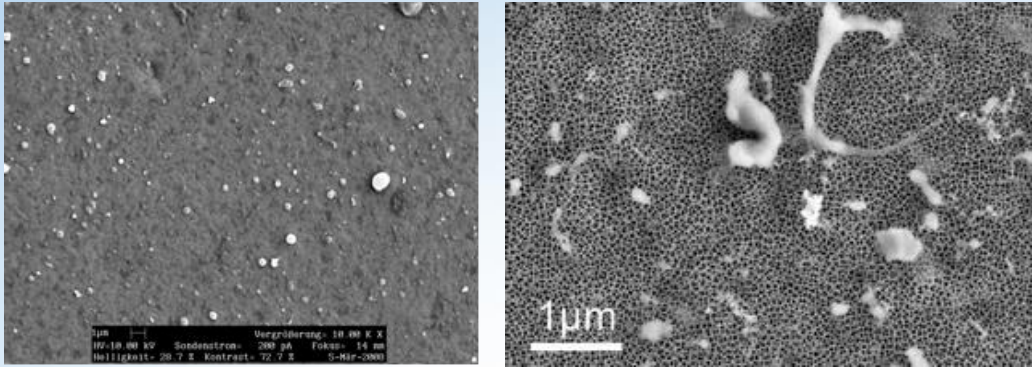
2015

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)



Current Applied Physics
Volume 9, Issue 2, Supplement, March 2009, Pages e124–e127
Nano Korea 2008 Symposium



Tribological behavior of copper nanoparticles as additives in oil
Y. Choi^a, C. Lee^a, Y. Hwang^a, M. Park^a, J. Lee^a, C. Choi^b, M. Jung^b



Procedia Engineering
Volume 102, 2015, Pages 1038–1045



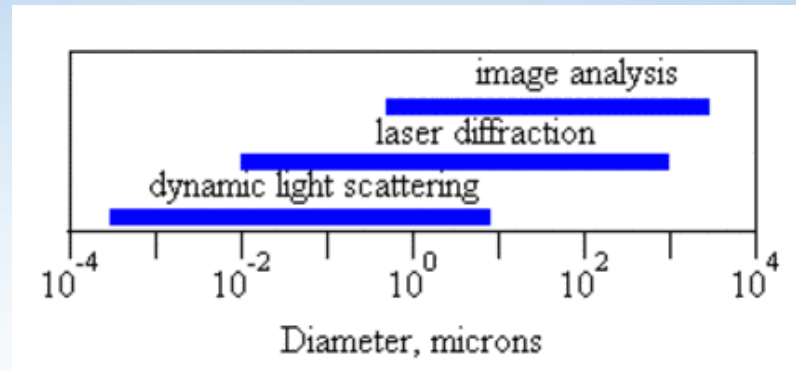
New Paradigm of Particle Science and Technology Proceedings of The 7th World Congress on Particle Technology

Open Access

Tribological Behaviour of a Lubricant Oil Containing Boron Nitride Nanoparticles ☆
Qingming Wan^{a, b, c}, Yi Jin^{a, c}, Pengcheng Sun^a, Yulong Ding^{a, c}



Some Particle Sizing techniques



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

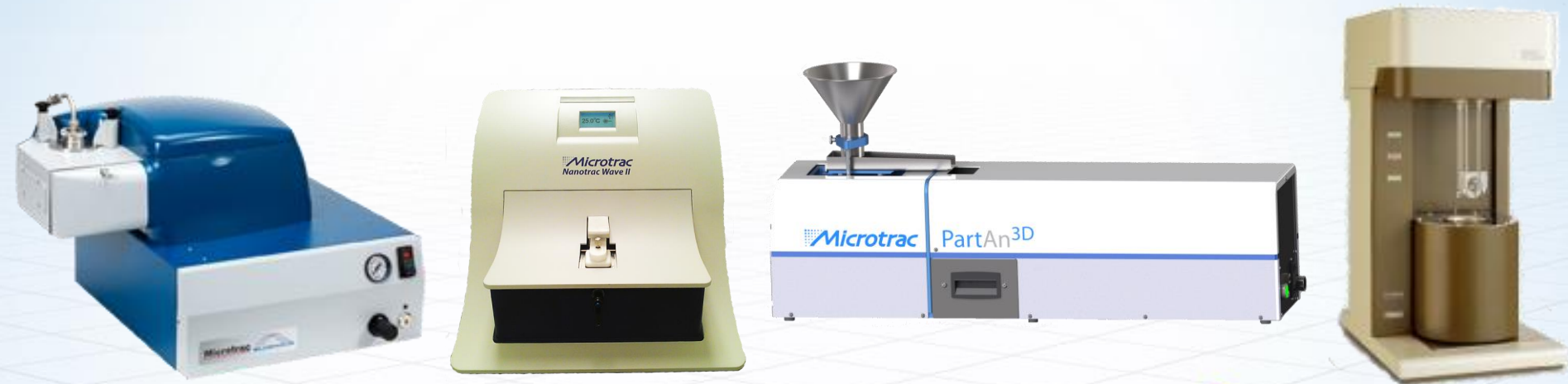
Surface Area/ Porosity



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

™

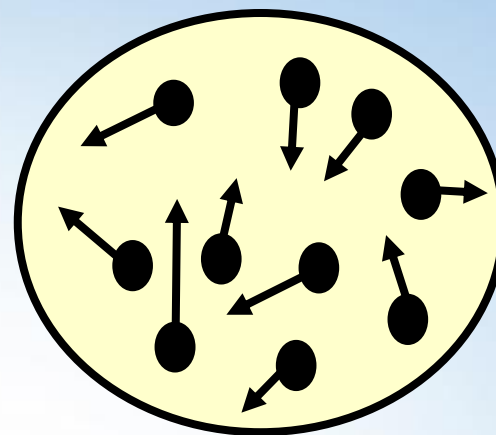
NIST

A MEMBER OF **NIKKISO**

Microtrac
Total Solutions in Particle Characterization

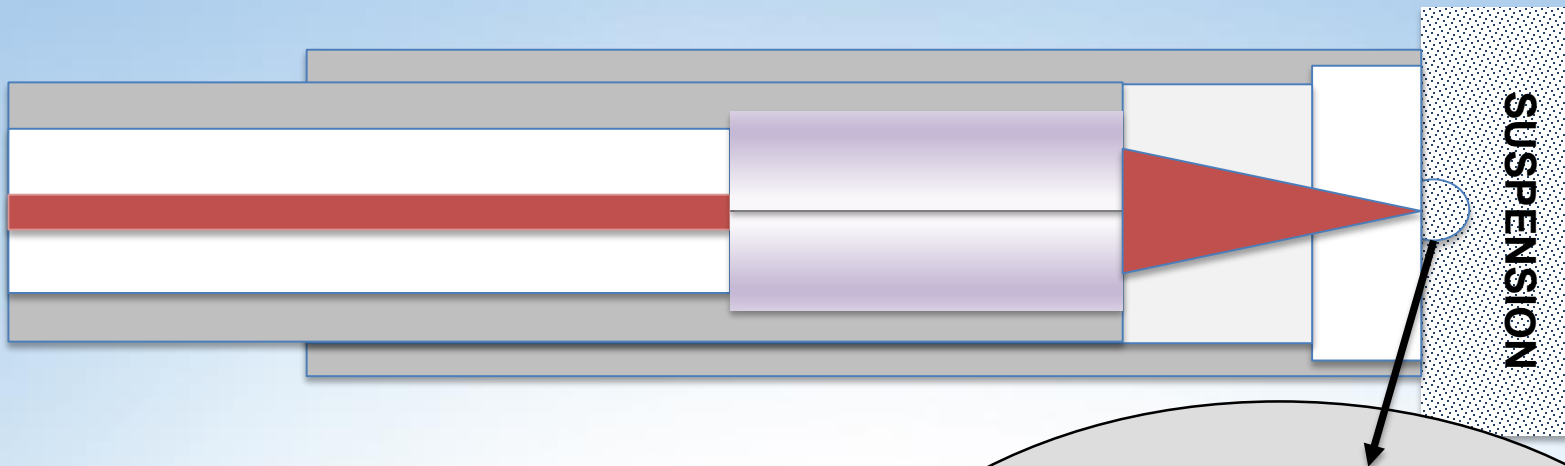
Dynamic Light Scattering – concept

- 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

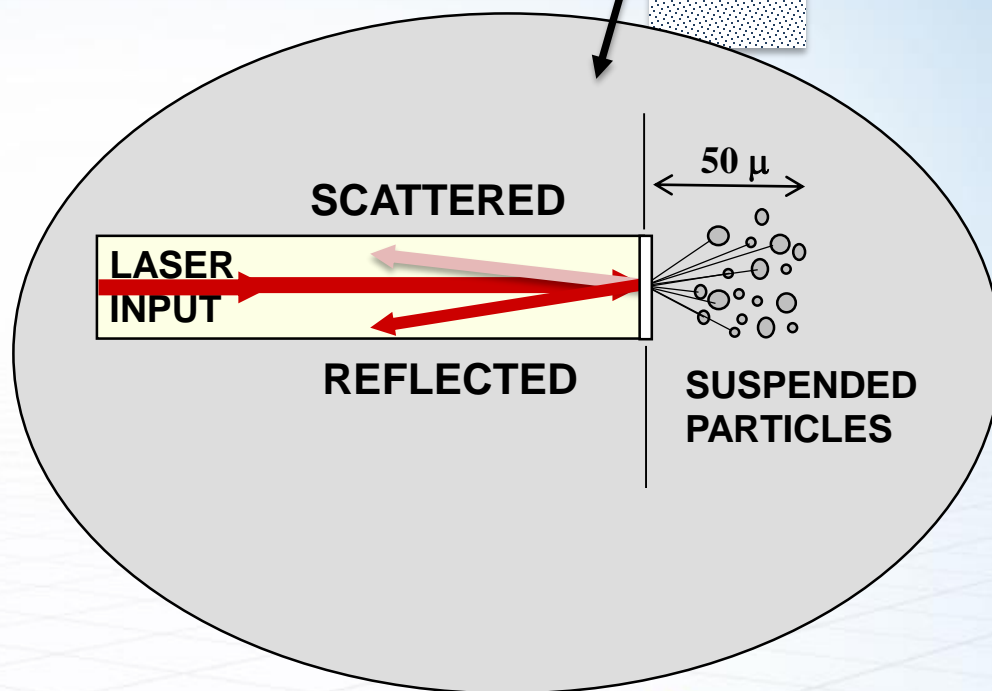
Controlled Reference – signal amplification for low concentrations



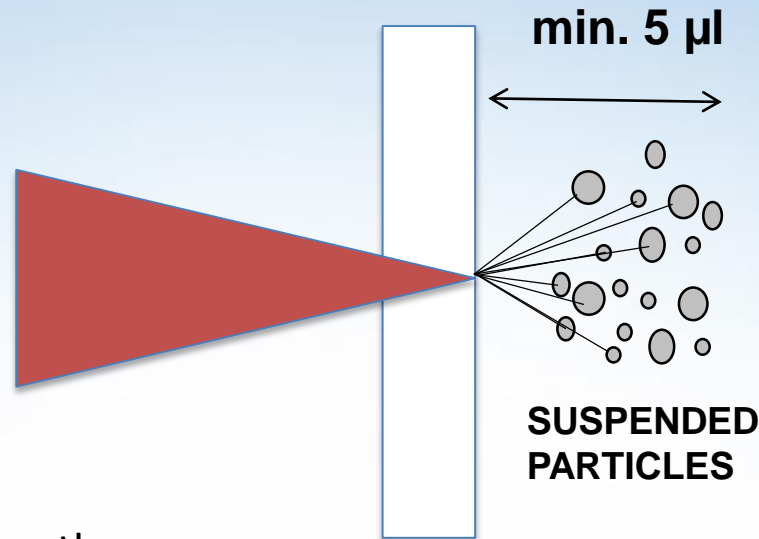
Interface reflection becomes unshifted controlled reference
2% of laser input is reflected

Scattered light collected and delivered to photodetector with reference

Reference beating at detector results in time intensity signal



180° detection angle for High Concentrations



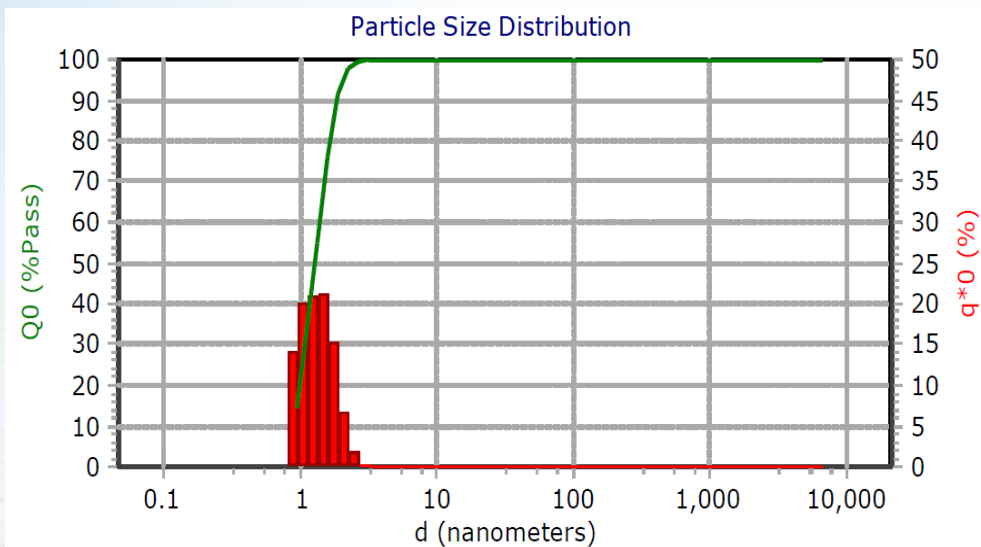
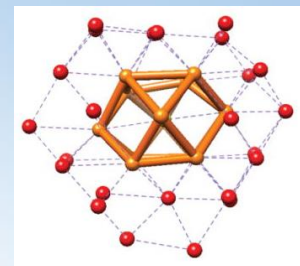
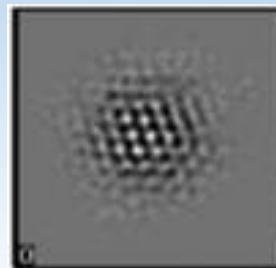
- 180° backscatter
- Minimum laser path length
- Minimum multiple scattering

→ Highest possible concentrations with 180° backscattering

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

Lower limit of detection: Practical test

Au₆₈ 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

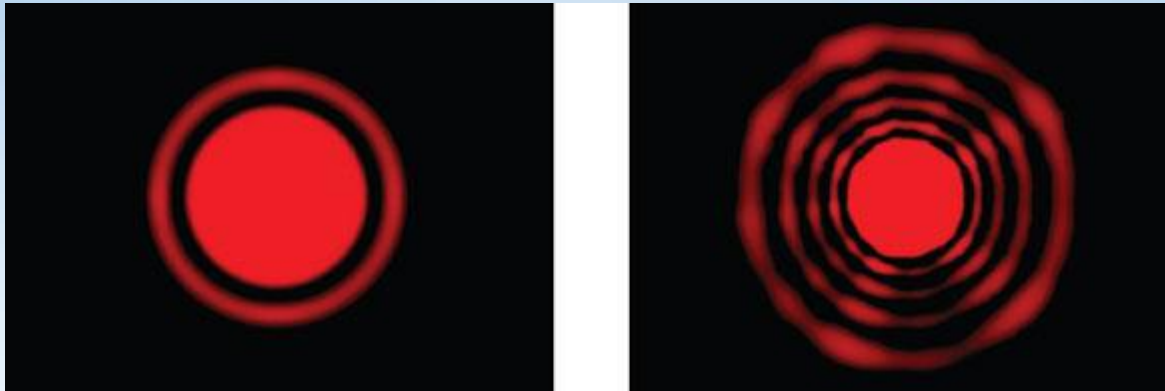
- Nanotracs Wave II
- Nanotracs Wave II Q
- NANO-flex
 - Zetrator
- Patented Controlled Reference Method
- Unique Probe Technology
- Heterodyne signal processing (Frequency Shift Analysis instead of PCS)
- wide concentration measurements
- Zeta Potential measurements with titration (up to 5 pumps)
- 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

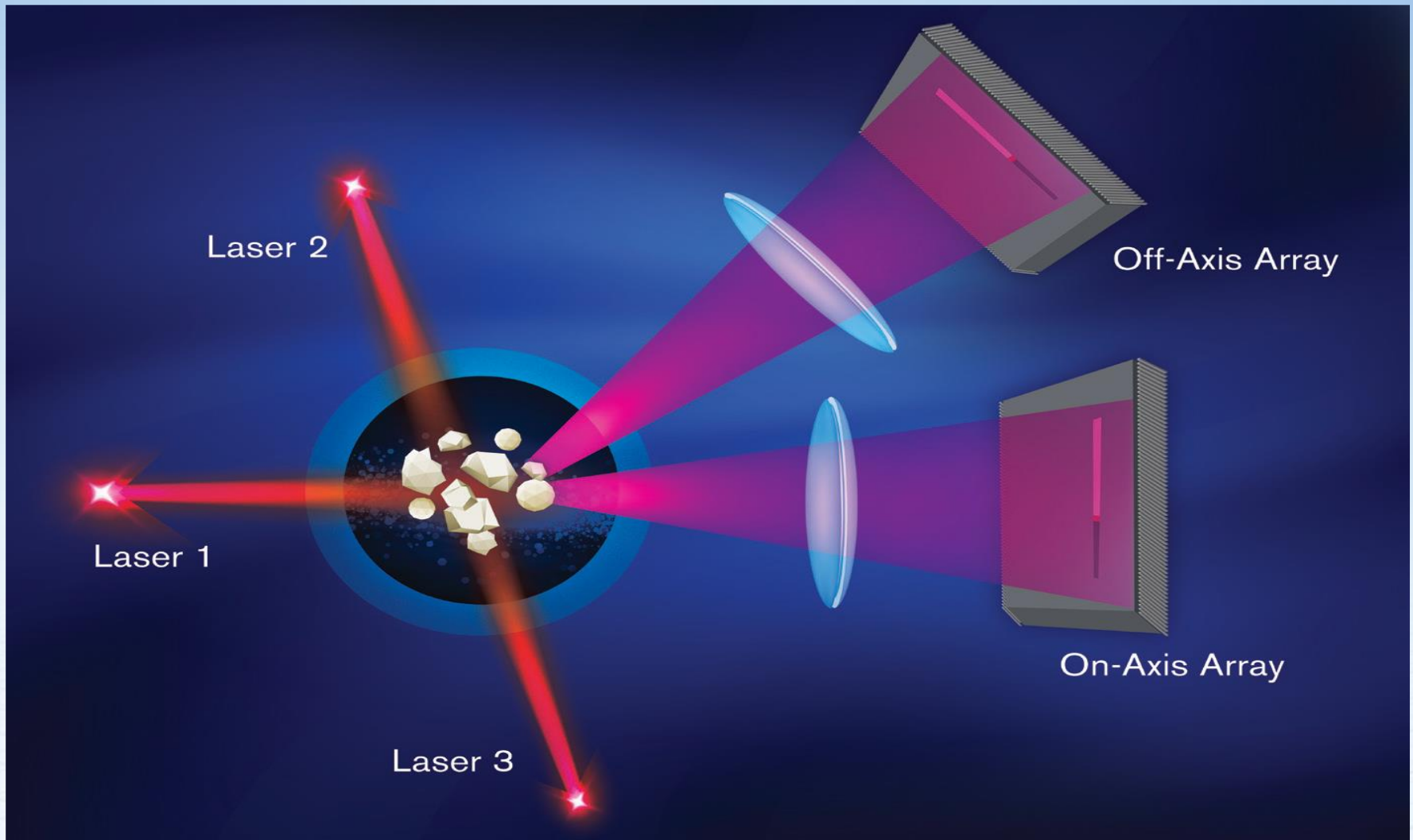
Laser Diffraction

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



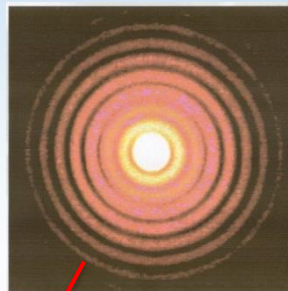
Microtrac's Patented 3-Laser Design – Detection 0.02-163°



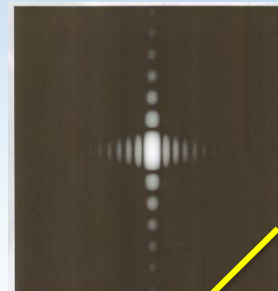
Modified Mie – Patented By Microtrac

More than 95% of all particles are NOT spherical

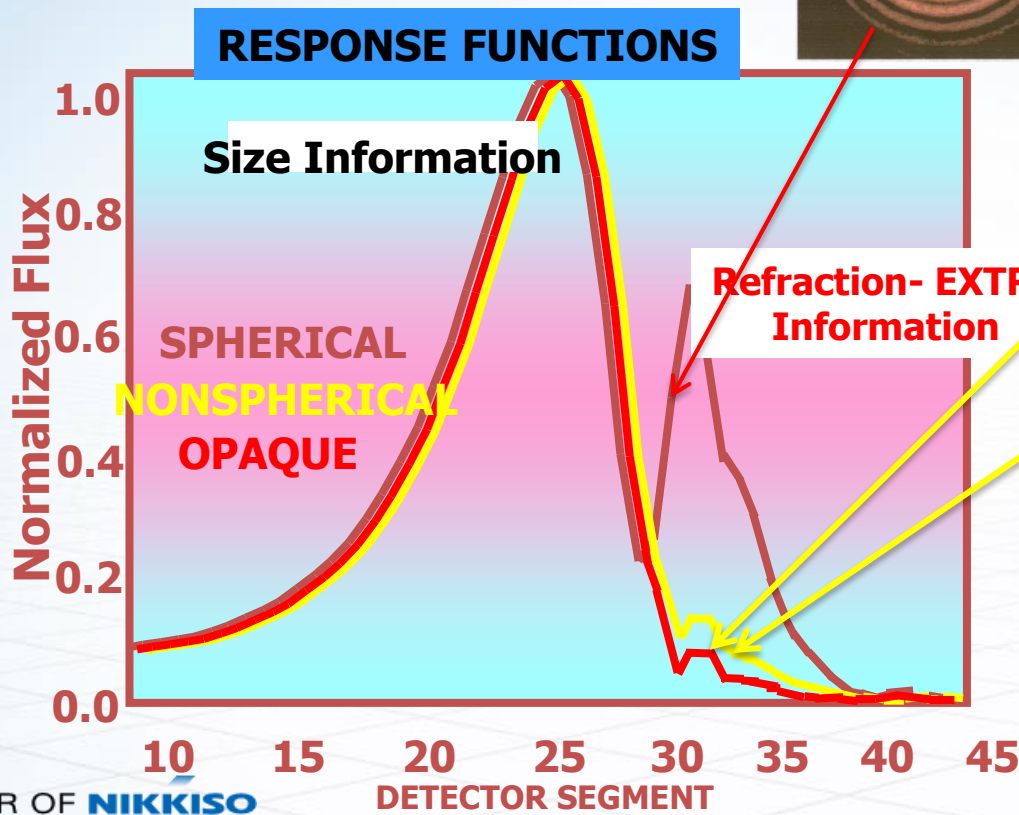
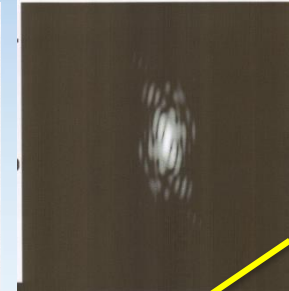
Spherical Particle



Hexagon



Irregularly shaped Particle



Modified Mie

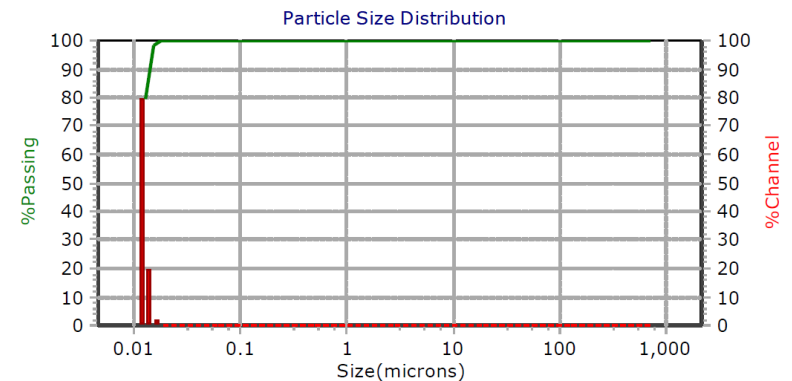
Diffraction lower size limit – Bluewave&Tribblue

405 nm Blue Lasers Provide for

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

12 nm Ludox sample

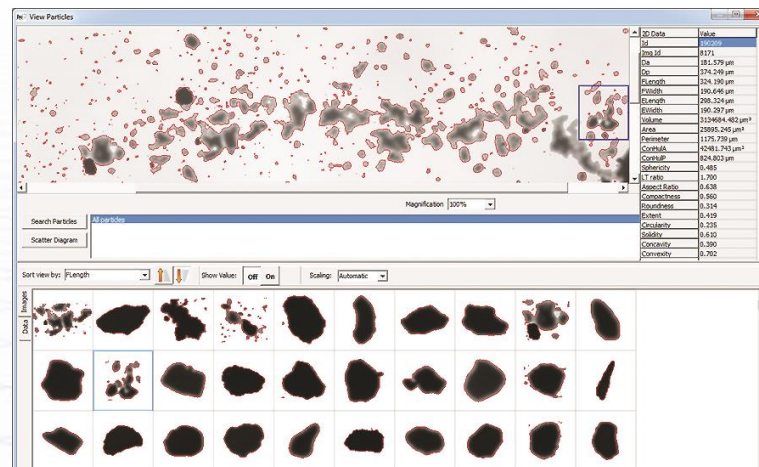
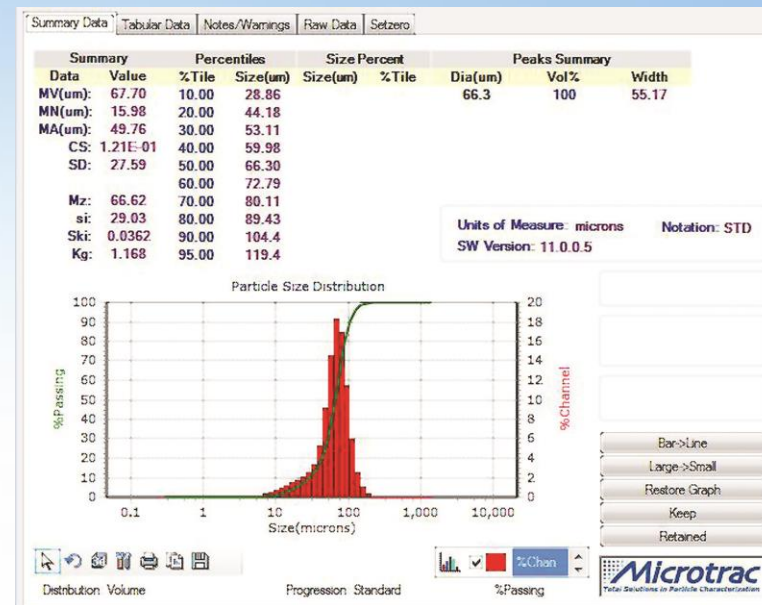
| Summary | | Percentiles | | Peaks Summary | | |
|----------|---------|---------------|---------|---------------|------|---------|
| Data | Value | %TileSize(um) | | Dia(um) | Vol% | Width |
| MV(um) | 0.01210 | 10.00 | 0.01100 | 0.0119 | 100 | 0.00191 |
| MN(um) | 0.01190 | 20.00 | 0.01130 | | | |
| MA(um) | 0.01200 | 30.00 | 0.01150 | | | |
| CS | 498.1 | 40.00 | 0.01170 | | | |
| SD | 0.00095 | 50.00 | 0.01190 | | | |
| Mz | 0.01205 | 60.00 | 0.01220 | | | |
| σ | 0.00098 | 70.00 | 0.01250 | | | |
| Ski | 0.2727 | 80.00 | 0.01280 | | | |
| Kg | 1.052 | 90.00 | 0.01350 | | | |
| | | 95.00 | 0.01420 | | | |





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

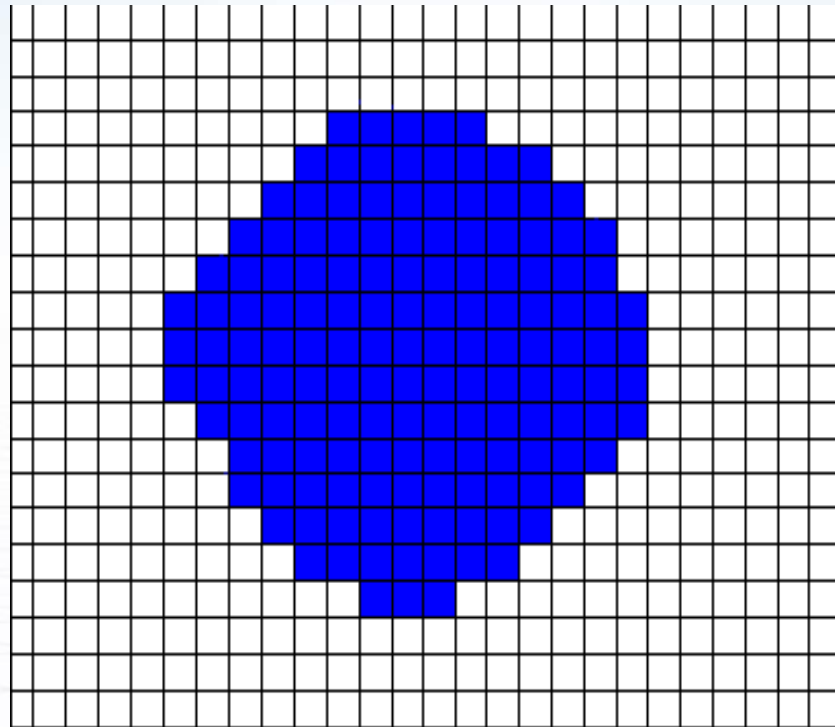


How Does It Work?

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

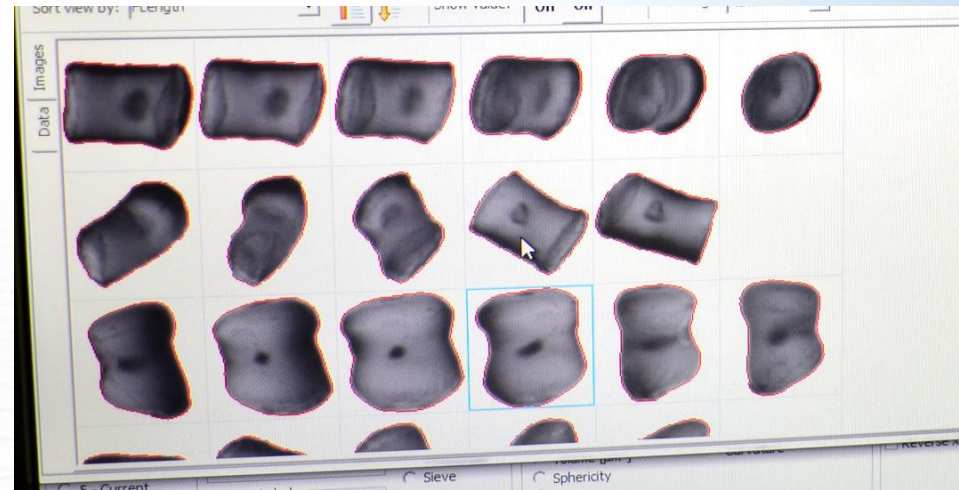
Sphericity
Circularity
Angularity
Roundness
Compactness
Extent
Ellipse Ratio
W/L Aspect Ratio
T/L Aspect Ratio
L/T Ratio
L/W Ratio
T/W Ratio
W/T Ratio

Surface Roughness

Convexity
Solidity
Concavity

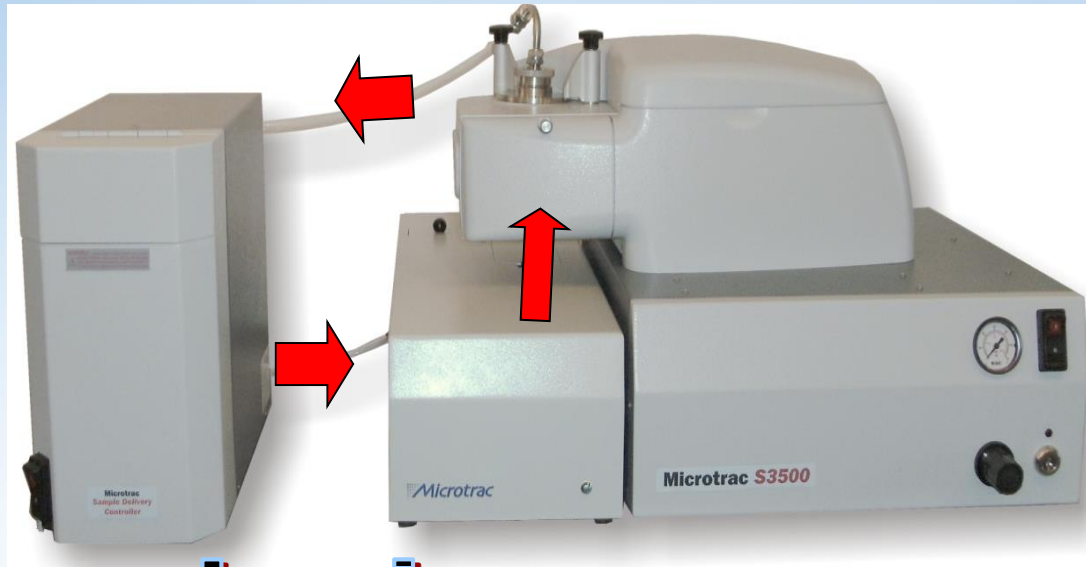
Other

Transparency
Curvature
ID
Img ID



Microtrac's PartAn-SI For Wet Measurements

SDC

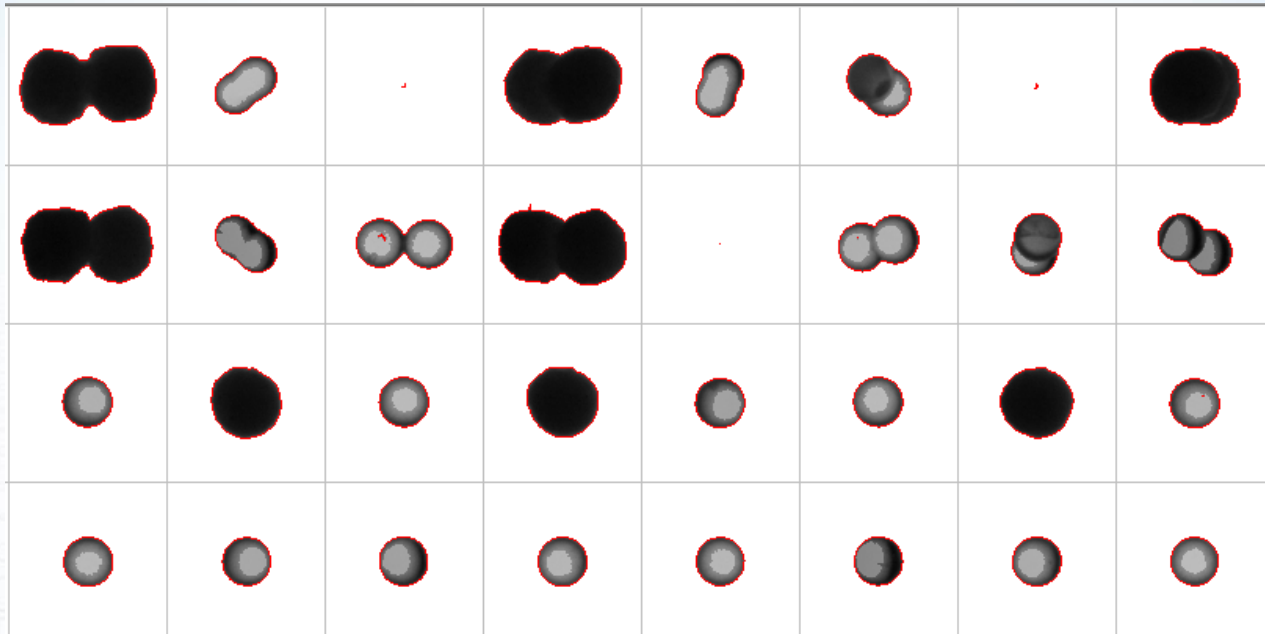
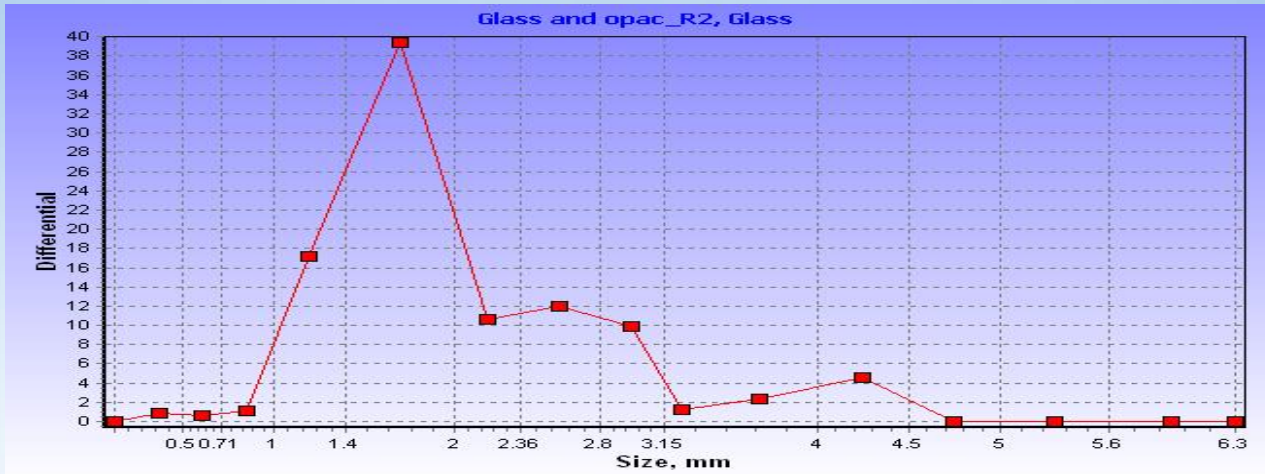


**S3500 or
Bluewave**

**Imaging
SI**

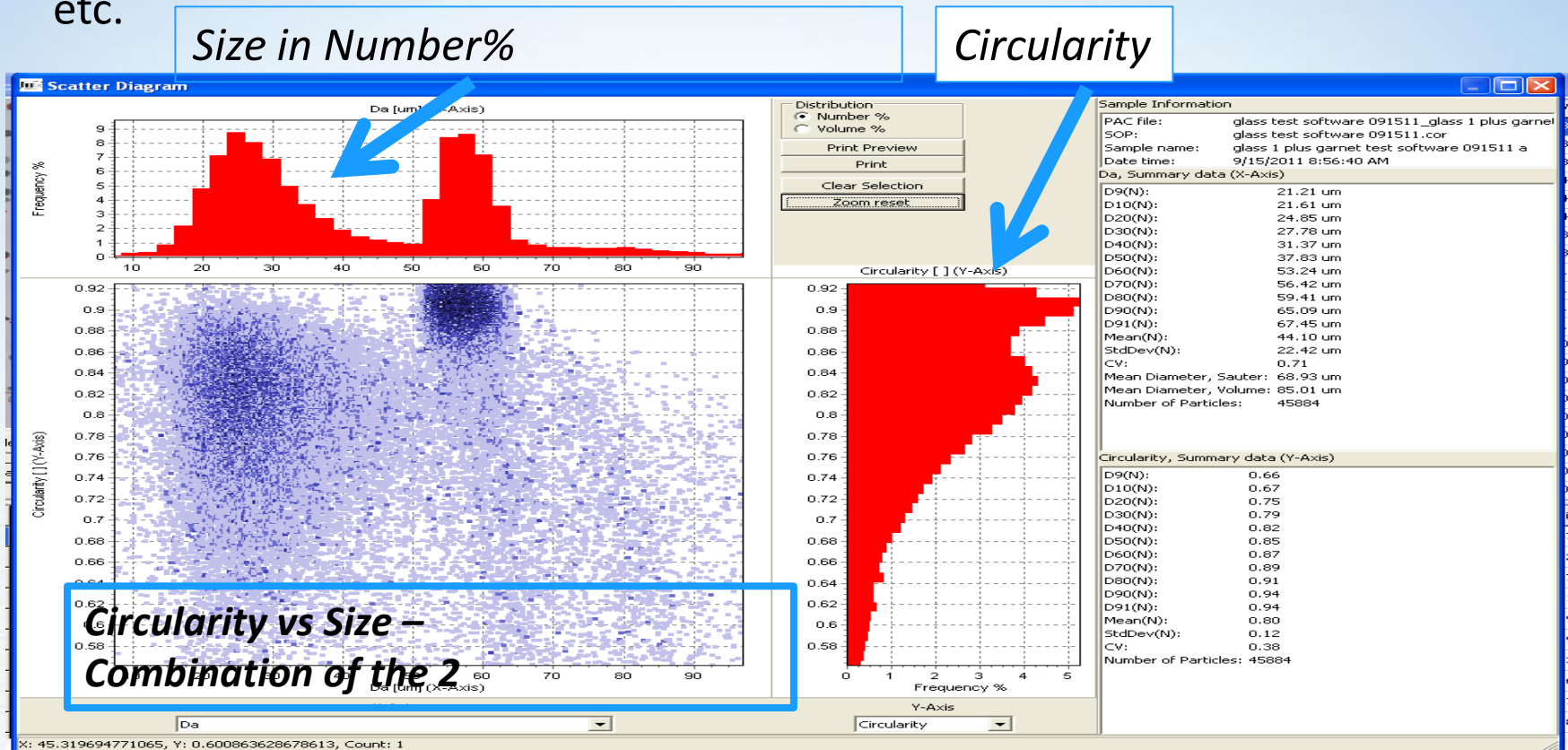
PartAn SI modular design allows connection to any
S3500, Bluewave or TRI-BLUE

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

The screenshot displays a software interface for particle analysis. At the top, a large image shows a single crystal with a red outline. Below this, a search bar contains 'All particles' and a 'Magnification' dropdown is set to '100%'. A table on the right lists various parameters for the selected particle:

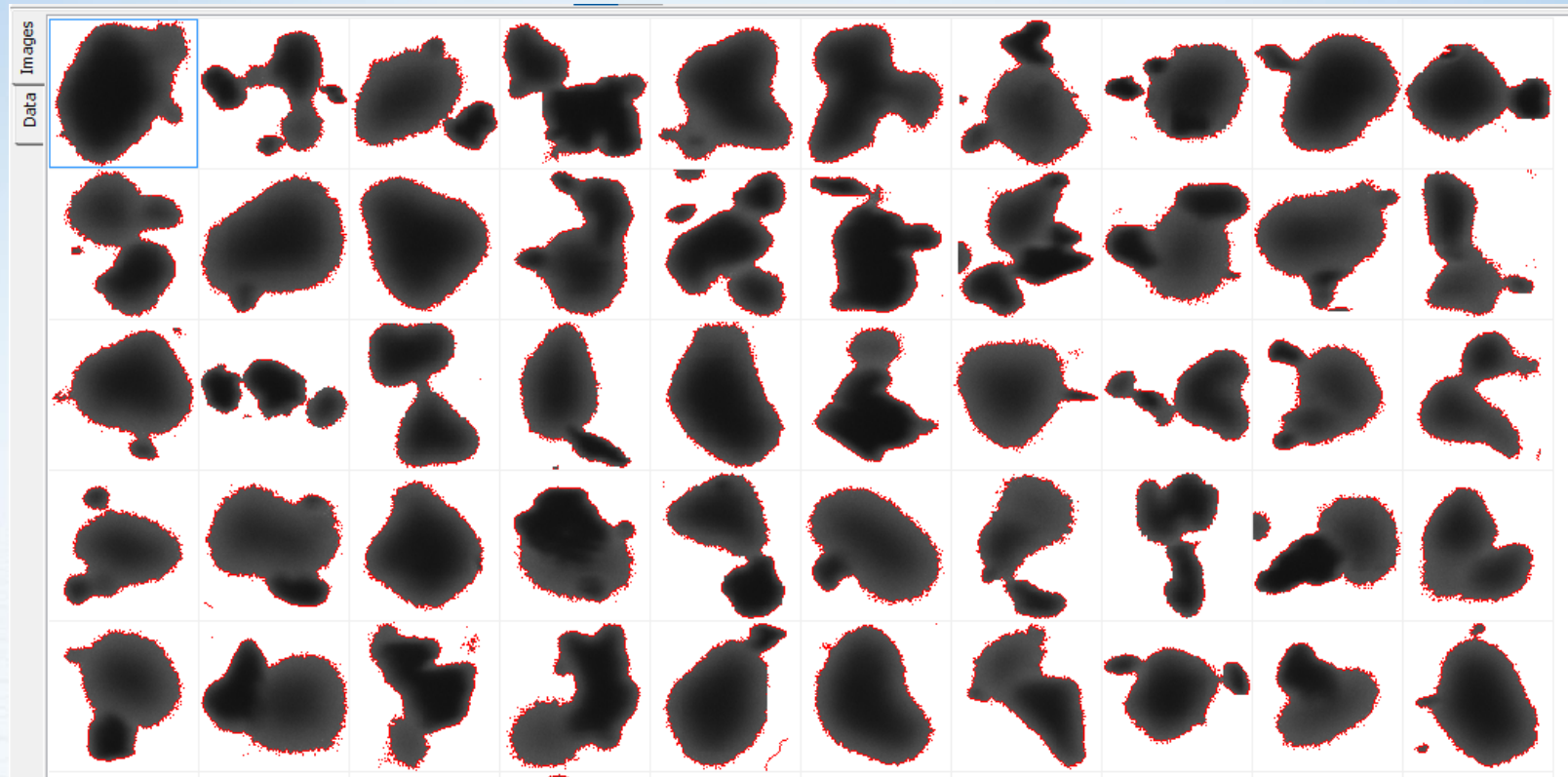
| | |
|--------------------|-----------------------------|
| Img Id | 2482 |
| Da | 141.863 μm |
| Dp | 230.464 μm |
| FLength | 188.874 μm |
| FWidth | 133.475 μm |
| ELength | 169.167 μm |
| EWidth | 133.750 μm |
| Volume | 1494888.529 μm^3 |
| Area | 15806.291 μm^2 |
| Surface Area | 63225.165 μm^2 |
| Perimeter | 724.025 μm |
| CHull Area | 19425.126 μm^2 |
| CHull Surface Area | 77700.503 μm^2 |

Below the search bar, a 'Sort view by' dropdown is set to 'FWidth'. A 'Show Value' toggle is set to 'On', and 'Scaling' is set to 'Automatic'. The main area contains a 4x10 grid of 40 small images of crystals, each with a numerical ID below it. The ID '133.475' is highlighted in blue.

| | | | | | | | | | |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| 195.772 | 191.028 | 168.948 | 155.364 | 151.889 | 147.767 | 145.401 | 145.765 | 144.928 | 144.006 |
| 142.388 | 141.503 | 137.692 | 136.782 | 135.732 | 135.002 | 133.475 | 131.784 | 129.731 | 129.680 |
| 129.563 | 125.707 | 124.999 | 124.887 | 123.995 | 121.244 | 118.498 | 118.250 | 117.543 | 116.710 |
| 116.676 | 119.078 | 113.420 | 112.925 | 112.732 | 112.974 | 108.403 | 107.511 | 105.933 | 105.325 |

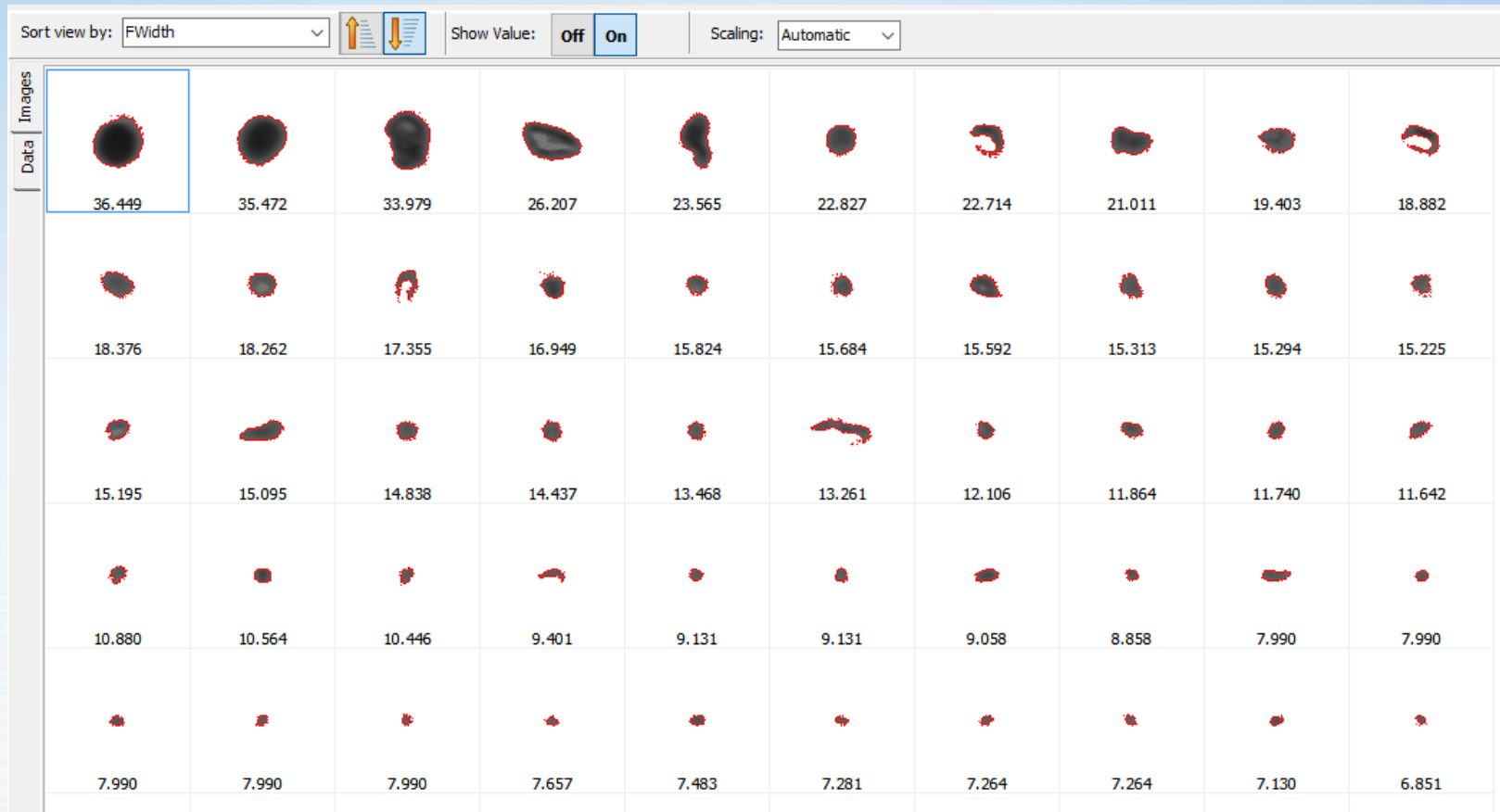
Benefits of Integrated Dynamic Image Analysis

- Agglomerates



Benefits of Integrated Dynamic Image Analysis

- Over-Grains



Microtrac PartAn SI PRO Online



Selected users



Statoil



THE UNIVERSITY OF
SOUTHERN
MISSISSIPPI.



Massachusetts Institute of Technology

ExxonMobil



americhem.

PRAXAIR



KENNAMETAL®

AEROJET
ROCKETDYNE



SANDVIK

A MEMBER OF NIKKISO

Microtrac
Total Solutions in Particle Characterization

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

Contact

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