micromeritics®

Advanced Energy Storage Technologies

Lithium-Ion Battery Series

MARKET DEMAND IS INCREASING. RAPID ADVANCEMENT IN TECHNOLOGY CONTINUES.

The requirement to remain competitive is more challenging than ever.

Strong analytical skills and innovative instrumentation help advance the knowledge and understanding of electrochemical performance. As your partner, Micromeritics Instruments will work diligently to ensure your success in this fast paced marketplace.

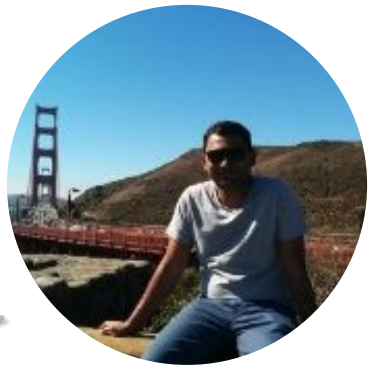
Our customers believe in our products, our people, our technology, and our partnership.

"I've been able to obtain unique instrumentation capable of measuring useful data for my research group. Micromeritics has been fantastic at supporting my unique research directions and helping with technical issues."

PAUL FORSTER
Principal Investigator
University of Nevada, Las Vegas

"We use the surface area machine to check the surface area of activated carbon. The results help us to identify the right carbon that meets the customer's requirements."

Prasad Galhena
Lab Technician
Carbon Activated Corp.



Lithium-ion Battery Technology

The Lithium-ion battery is an energy storage device capable of almost continuous charging and discharging. In comparison to traditional battery technologies, lithium-ion batteries provide a significantly higher performance and efficiency In terms of application. The lithium-ion battery market can be segmented into consumer electronics, automotive, storage grid energy, and industrial use.

Improving global economic conditions, rising disposable income, the need for clean energy and surging demand for quality & uninterrupted power are a few of the major factors anticipated to boost demand for lithium-ion battery. The power density, safety, recharge time, cost, and other aspects of its technology are expected to continue to advance.

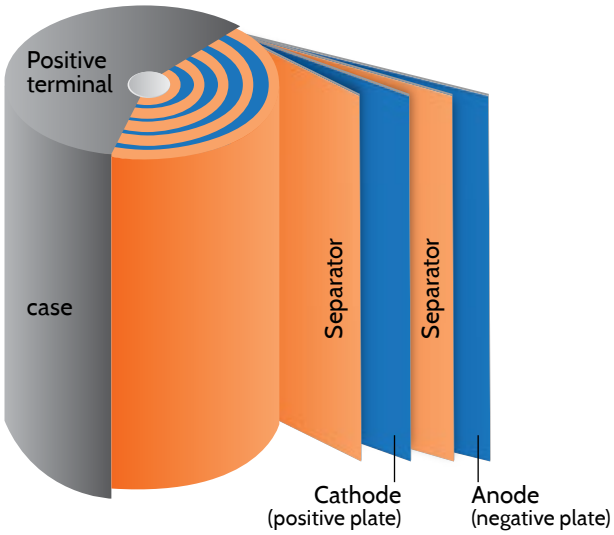
Micromeritics Instruments, experts in Materials Characterization Instrumentation for Electrochemical Analysis

Electrode Analysis

Cathode/Anode

The development of cathode and anode materials for lithium-ion batteries is based on improvement to power and energy density as well as the thermal/chemical stability for enhancements in battery life and charge cycling.

The theoretical capacity of a lithium-ion battery is determined by the materials used. In electrode processing, knowledge of particle morphology–including particle size, shape, powder density, porosity and surface area–have critical affect to manufacturability and the desired performance characteristics of the electrode.



Porosity Measurements

The electrodes porosity structure has a direct influence on particle to particle contact between the active material and the conductive diluent. Porosity is essential for the electrolyte to transport lithium-ions to and from the active materials of the electrode. By controlling porosity, higher intra-electrode conductivity can be achieved to ensure adequate electron exchange as well as sufficient void space for electrolyte access/transport of lithium-ions for intercalation of the cathode. Porosity blocking/clogging during intercalation can lead to capacity fade.

See pages 12, 13, 14, and 15

Particle Size/Particle Shape

Particle size influences capacity, cycling, and coulomb efficiency. Particle size will impact the amount of solid-state diffusion of lithium-ions that intercalate at the electrode. Smaller particles, especially nanoparticles, will lead to smaller volume changes upon cycling. This contributes to less mechanical stress, increased hardness, and greater resistance to fracture.

It has been reported that a broad particle size distribution may increase the energy density more than a mono-dispersed distribution. Controlling and customizing particle size distribution can result in the ability to make available custom tuning that will result in high power (mono-disperse) or high energy density (poly-disperse).

Shape will affect packing density. Spherical shaped particles will pack more densely than fibrous or flake shaped particles. The average strain energy density stored in a particle increases with the increasing sphericality. Fibrous and flake shaped particles are expected to have lower tendency for mechanical degradation than the spherical shaped particles.

See pages 16, 17, and 18

Surface Area

Increasing the surface area of the electrode will result in improvement in the efficiency of the electrochemical reaction and facilitates the ion exchange between electrode and electrolyte, especially within the anode as higher surface area permits short diffusion paths to the lithium-ions between the graphite particles. Lower surface area materials are better suited for improved cycling performance of the cell resulting in longer battery life.

Greater surface area does present some limitations due to the degradation interaction of the electrolyte at the surface and resultant capacity loss along with thermal stability. Nanoparticles hold much promise to increase surface area without capacity loss. For the anode, higher surface area permits shorter diffusion paths for lithium-ions between the graphite particles This facilitates fast charge and more efficient discharge rates and improves the capacity of the battery.

See pages 13 and 15

Density/T.A.P. Density

The density of the graphite anode has an effect on its ability to withstand degradation under challenging load and discharge operations. A higher anode electrode particle density decreases the porosity resulting in a lower active surface area of the electrode. This reduces the electrode/electrolyte contact area.

True/absolute density and envelope density can help by determining electrochemical performance attributed to the electrodes available porosity for intercalation. A clear correlation has been found between irreversible capacity and internal pore volume.

T.A.P. density measurement is an important indicator of volumetric energy density. A low T.A.P. density translates into a low volumetric energy density where the converse indicates a high volumetric density. Higher T.A.P density permits denser electrode films (more active material per unit volume) to be made for coating the electrode.

See page 12

Pore Size, Shape , Distribution, and Tortuosity

The size, shape, and tortuosity of the electrodes pores will significantly affect lithium ion transport rates through the electrolyte retained within this porous structure. Electrode microstructure resulting from the manufacturing process has direct influence on energy density, power, lifetime, and reliability of the lithium-ion cell.

A better understanding of the interconnectivity of adjacent pores, closed pores, and channels that may be created during the manufacturing process helps to ensure optimal electrolyte and electrode interaction. Knowing the tortuosity of a porous electrode and electrolyte interface makes it possible to determine if cell performance limitations are due to its microstructure.

See pages 12 and 15

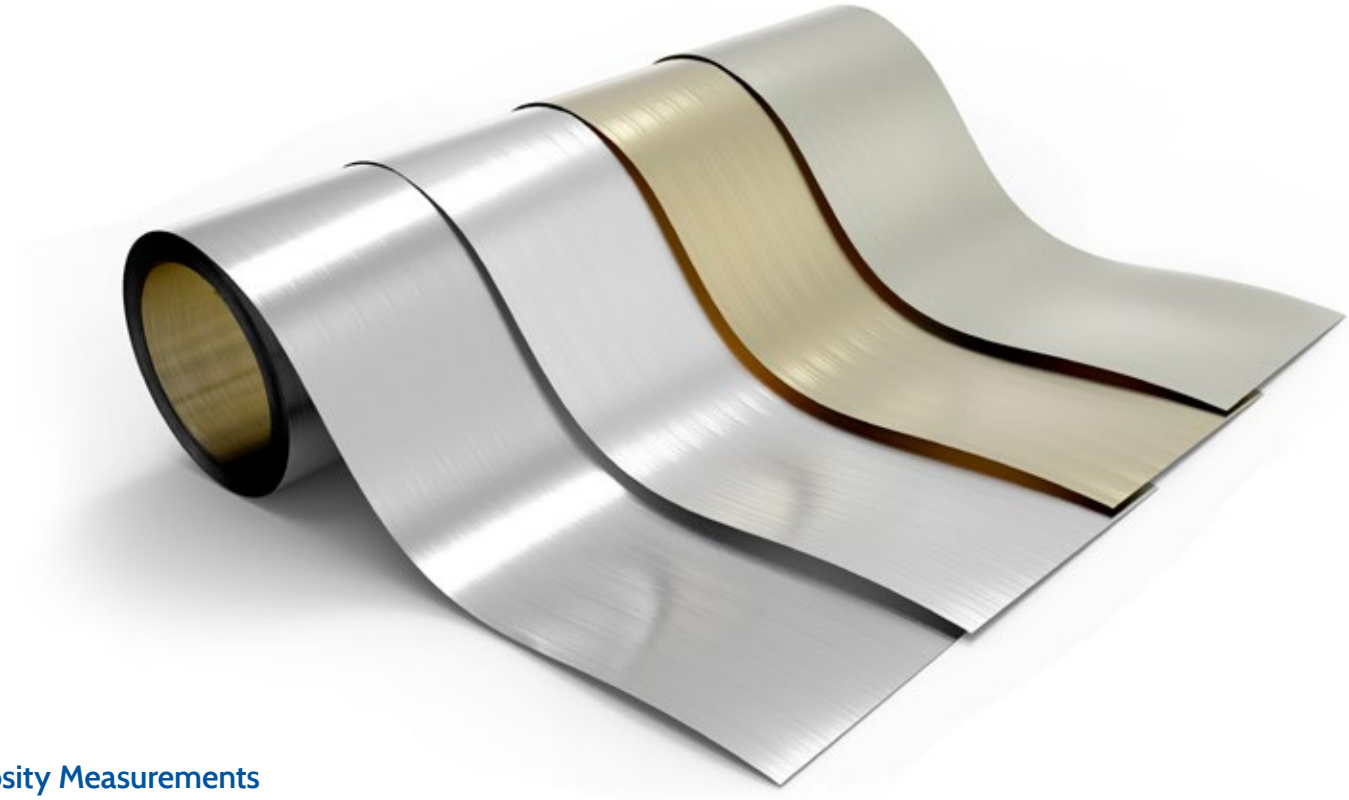
Separator/Binder Evaluation

The separator permits ion flow from one electrode to the other while preventing any electron flow, essentially separating the anode from the cathode.

The typical separator is made up of polyolefins, usually polypropylene and/or polyethylene, along with other polymers, ceramics, and ceramic/polymer blends.

Separators are highly porous, typically >40% porosity, approximately 25 µm thick and exhibit low ionic resistivity. Layered or composite separators are used as safety devices to prevent thermal runaway of the cell.

Binder materials are used to hold the active electrode material particles together and in contact with the current collectors, i.e. the Aluminum Foil of the cathode or the Copper Foil of the anode.



Porosity Measurements

Specification of percent porosity is an important parameter in the acceptance criteria for the separator. The separator must have sufficient pore density to hold the liquid electrolyte that supports ionic movement between the anode and cathode. Higher porosity means less heat generated in the cell and greater energy density.

Uniform porosity is essential to avoid variations in ion flow. The more variation in ionic flow within the separator, the greater the effect at the surface of the electrode and the quicker it will fail with a significantly decreased cycle life. Excessive porosity hinders the ability of the pores to close, which is vital to allow the separator to shut down an overheating battery.

See pages 12, 13, 14, and 15

Pore Size, Shape , Distribution, and Tortuosity

The separator pore size must be smaller than the particle size of the electrode components, i.e. the electrode active materials and any conducting additives. Most separator membranes contain sub-micron pore sizes that block the penetration of particles.

Uniform distribution and a tortuous structure of the pores are also a requirement. Uniform distribution prevents uneven current distribution throughout the separator and tortuosity suppresses the growth of dendritic lithium.

See pages 14 and 15

Zeta Potential

To further understand the transport mechanisms of the separator membrane, zeta potential can indicate the membranes electrolyte affinity. This can permit fine tuning of battery performance to improve cycle life. Cycle life is extended when the separator has a low electrolytic resistance but high aqueous permeability. Zeta potential can also provide needed information about the membranes affinity with electrolyte additives.

See page 19

Electrolyte Analysis

Liquid electrolyte plays a key role in commercial lithium-ion batteries to allow conduction of the lithium-ions between cathode and anode. The most commonly used electrolyte is comprised of lithium salt, such as LiPF6 in an organic solution.

High purity is required to prevent oxidation at the electrode and to promote good cycle life. In addition to lithium salt, various additives are also included in the final electrolyte solution. These additives are mixed with the LiPF6

solution to prevent lithium dendritic formation and degradation of the solution.

Zeta Potential

There are electro kinetic phenomena caused by charge separation at the separator-electrolyte interface. Diffusion of charged electrolyte solution through the pores of the separator has to undergo the influence of the zeta potential at the interface. The zeta potential at that interface may impede or aid the passage of the electrolyte across the separator.

The zeta value gives an indication of the potential stability of a system: the larger the value (positive or negative) the more stability of the solution.

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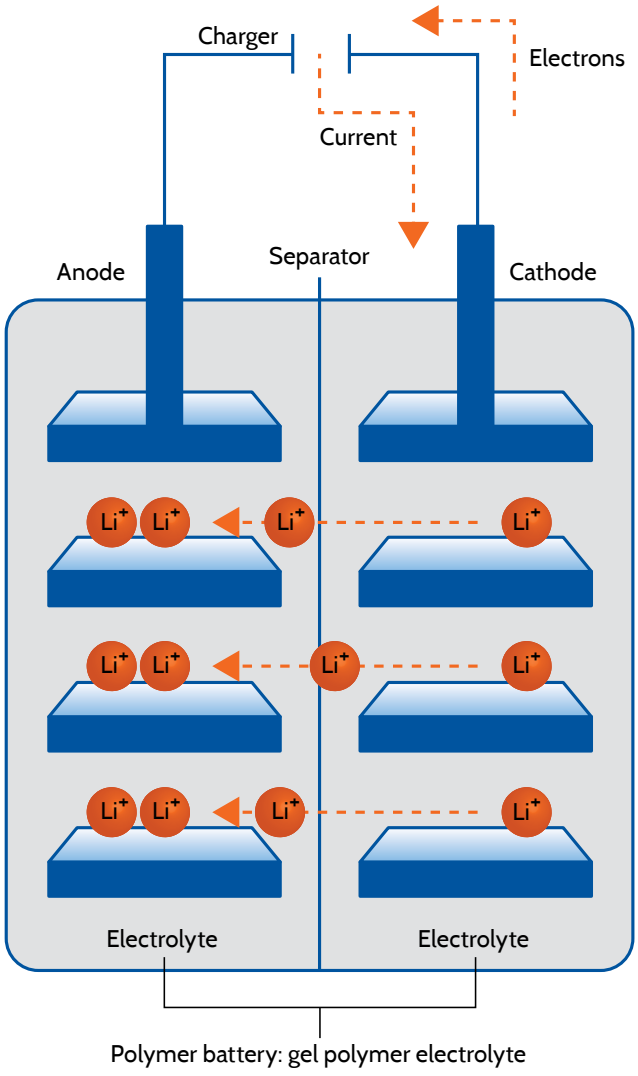


Manufacturing and Failure Analysis

Materials characterization during and prior to manufacturing is a critical control parameter to ensure the optimal operation of cell components and the final assembled battery.

From raw materials to component manufacture and the assembled battery itself, material characterization plays a vital role in determining the desired electrochemical performance, safety, cell cycling and other important parameters.

Particle Size/Particle Shape-Raw Materials

Particle size and shape influences packing density which in turn affects electrode thickness and therefore energy density. It has been shown that the particle size distribution of graphite, as well as particle orientation in the coated foil affects the electrochemical performance of graphite anodes. Purity is also an important issue and low levels of metallic impurities must be maintained in all powders and additives used in electrode manufacture.

See pages 16, 17, and 18

Calendering/Solid Fraction Determination

Calendering is the most critical step in the production of high performance electrodes. Porosity and thickness of the electrode film will decrease with increasing calendering. Calendering would also be expected to change the pore structure of the electrode, which would thereby impact the wetting behavior of the film by the electrolyte.

Calendering beyond the optimum level causes reductions in porosity and average pore diameter which can result in irreversible capacity loss, high rate cycling, and poor longevity in cycle performance.

Solid Fraction is a control parameter used in roller compaction operations. This control parameter assists in determining the optimal setting for speed, compression and nip angle in the roller compactor. Using the the solid fraction as a critical quality attribute will ensure consistent product batch to batch, along with the end product having the designed and desired electrochemical performance.

Solid Fraction, Envelope, and True Density

SF= Solid Fraction (relative density)
Pe = envelope density of the coated foil
Po = true density of the granules

$$SF = \frac{P_e}{P_o}$$

See page 12

Performance Degradation

Over the life of a cell, physical and electrochemical occurrences contribute to degradation in performance. This drop in performance is most notably recognized through capacity fade during charge and discharge cycling or by reduced shelf life.

Expansion and contraction may cause interfacial stress that adversely effects the electrode performance, to the point that delamination may occur causing a reduction in contact between the electrode material and the current collector. Pore size changes can occur from this mechanical failure resulting in reduction in electrolyte contact and poor cycling behavior.

See pages 12, 13, 14, and 15

AccuPyc 1340 and GeoPyc 1365 Instrument Porosity Bundle

AccuPyc 1340 Gas Displacement Pycnometry System

Gas pycnometry is recognized as one of the most reliable techniques for obtaining true, absolute, skeletal, and apparent volume and density. This technique is non-destructive as it uses the gas displacement method to measure volume. Inert gases, such as helium or nitrogen, are used as the displacement medium. The AccuPyc features speed of analysis, accuracy, repeatability, and reproducibility.

GeoPyc 1365 Envelope and Tap Density Analyzer

The GeoPyc 1365 Pycnometer determines the envelope volume and density of monolithic samples as well as the bulk volume and density of powdered materials by a unique displacement technique. To determine envelope volume the GeoPyc uses a quasi-fluid displacement medium composed of microspheres having a high degree of flowability that do not wet the sample or fill its external or internal pores. The GeoPyc collects the displacement data, performs the calculations, and displays or prints the results.

When equipped with the T.A.P. Density option, the GeoPyc measures the bulk volume and calculates the bulk density of granular and powdered samples.

Combining measurements from both these instrument permits the user to accurately calculate percent porosity and total pore volume of a substance.

Density Type	Definition	Material Volume	Open-Pore Volume	Closed-Pore Volume	Inter Particle Volume	External Void Volume	Addressed by
True (Absolute)	The mass of a substance divided by its volume, excluding open and closed (or blind) pores	◎					AccuPyc II
Skeletal (Apparent)	The ratio of the mass of the solid material to the sum of the volume including closed (or blind) pores	◎		◎			AccuPyc II
Envelope	The ratio of the mass of a substance to the envelope volume (imaginary boundary surrounding the particle)	◎	◎	◎	◎	◎	GeoPyc
Bulk	Mass of the material divided by the volume occupied that includes interstitial space	◎	◎	◎	◎		GeoPyc
Tap	Apparent powder density obtained under statedconditions of tapping	◎	◎	◎	◎		GeoPyc with T.A.P. function

TriStar II Plus

Analytical Versatility/ High Throughput/Small Footprint

The TriStar II Plus is an automated, three-station, surface area and porosity analyzer that delivers excellent performance and speed of analysis. With three available analysis ports, the TriStar II Plus provides high sample through-put and advanced data analysis features to the user. The unique stainless steel analysis manifold features very low leak rates and is designed for highly accurate gas management for confident and repeatable results.

The TriStar II Plus features MicroActive software for data analysis with its powerful suite of tools to permit the user to easily obtain important information about their sample.

MicroActive also includes a powerful utility that allows the user to overlay a mercury porosimetry pore size distribution with a pore size distribution calculated from gas adsorption isotherms. This new import function allows users to rapidly view micropore, mesopore, and macropore distributions in one easy-to-use application.

Specific Surface Area
From 0.01 m^2/g, nitrogen unit
From 0.001 m^2/g, krypton unit

Total Surface Area
From 0.1 m^2, nitrogen unit
From 0.01 m^2, krypton unit

Pore Volume
From 4 × 10-6 cm^3/g

Some of the available tabular and Graphical Reports:

- Single and multipoint BET surface area
- Total pore volume
- Langmuir surface area and Isotherm reports
- t-Plot
- BJH adsorption and desorption
- F-ratio
- Mesopore
- Volume and area distributions by pore size
- DFT pore size
- DFT surface energy
- Summary Report
- Custom reports using Python interface

AutoPore V

Mercury Intrusion Porosimetry

Mercury porosimetry analysis is based on the intrusion of mercury into a porous structure under stringently controlled pressures. Besides offering speed, accuracy, and a wide measurement range, mercury porosimetry permits you to calculate numerous sample properties such as pore size distributions, total pore volume, total pore surface area, median pore diameter and sample densities (bulk and skeletal).

The AutoPore V Series Mercury Porosimeters can determine a broader pore size distribution more quickly and accurately than other methods. This instrument also features enhanced safety features and offers new data reduction and reporting choices that provide more information about pore geometry and the fluid transport characteristics of your material.

Operational Advantages

- Ability to measure pore diameters from 0.003 to 1100 µm*
- Controlled pressure can increase in increments as small as 0.05 psi from 0.2 to 50 psia. This allows detailed macropore data to be collected
- High-resolution (sub-microliter) measurement of intrusion/extrusion volumes produces high resolution data allowing the development of tighter sample specifications, improved production processes, and high-quality research data
- Operates in scanning and time- or rate-of-intrusion equilibrated modes
- Real-time diagnostics provide knowledge of an issue before it becomes critical or impairs your analytical results
- Collects extremely high-resolution data; better than 0.1 µL for mercury intrusion and extrusion volume
- Improved linear motion for high-pressure chamber closure

Pro X Scanning Electron Microscope

Powerful Magnification in a Desk Top Foot Print

The Phenom ProX desktop scanning electron microscope (SEM) is the ultimate all-in-one imaging and X-ray analysis bench top system. With the Phenom ProX , sample structures can be visually examined and their elemental composition determined.

Viewing three-dimensional images of microscopic structures only solves half the problem when analyzing samples. It is often necessary to collect more than optical data to be able to identify the different elements in a specimen. This is accomplished in the Phenom ProX with a fully integrated and specifically designed EDS detector.

EDS is a technique that analyzes X-rays generated by the bombardment of the sample with an electron beam. EDS elemental analysis is fully embedded into the Phenom ProX system. X-ray detector and control software are combined in one package.

Specifications

Light optical magnification	20 - 135x
Electron optical magnification range	80 - 130,000x
Resolution	≤ 10 nm
Digital zoom	Max 12x
Light optical navigation camera	Color
Acceleration voltages	Default: 5 kV, 10 kV and 15 kV

Advanced mode: adjustable range between 4,8 kV and 15 kV imaging and analysis mode

Sub-Sieve AutoSizer

Air Permeability Particle Sizing and Surface Area Instrument

The air-permeability technique is well established for measurement of the aerodynamic Specific Surface Area (SSA) of a sample powder. The SSA measured by this technique has been found to be a useful parameter in various industries such as, metal coatings, metal powders etc.

The SAS utilizes dual pressure transducers to measure pressure drop across a packed bed of powder by using the principle of pressure drop across a packed powder bed. By varying the sample height, and hence the "porosity" of the bed, average surface area and particle size can be determined as a function of pressure drop in accordance with the Carmen equation.

The SAS measures particle size in the range of 0.2 µm to 75 µm and has a compression accuracy of less than 0.05 mm.

Quick and Easy Set-up
Simple step-by-step, easy to follow, ensuring that no parameters are overlooked

Superior Software
The SAS Software sets a world-wide standard for instrument operation, data acquisition and handling, reporting and systems integration

Real Time Data Display
Data can be viewed as it is acquired simplifying method development

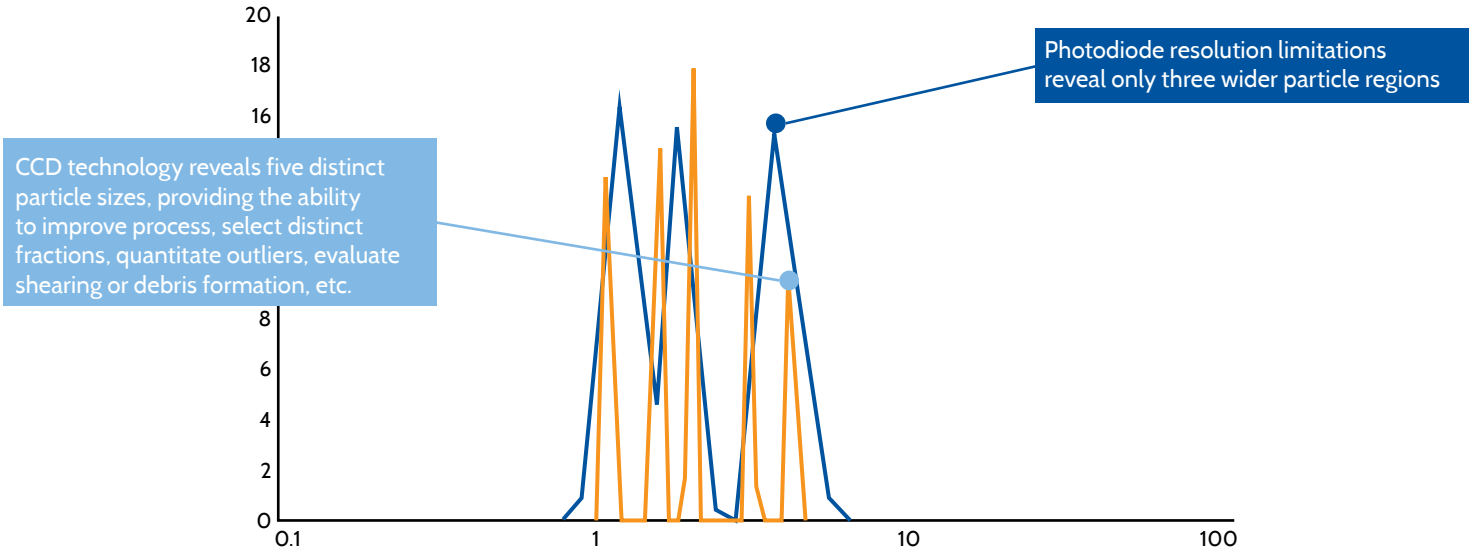
Saturn DigiSizer II

High-Definition Digital Particle Size Analyzer

The Saturn DigiSizer®II is the first commercially available particle sizing instrument to employ the light scattering analysis technique that utilizes advanced digital detection technology to deliver exceptionally high levels of resolution, accuracy, repeatability, and reproducibility.

A state-of-the-art CCD detector containing over three million detector elements enables the completely automated Saturn DigiSizer II to capture a high resolution, digital representation of the pattern produced as a result of laser light scattered from a sample. The resulting information is then processed using data reduction based on Mie or Fraunhofer theory. The Saturn DigiSizer II gives users an extremely high level of resolution and sensitivity not available with other laser particle sizing systems.

The instrument produces fast, detailed results that are repeatable on and reproducible between every Saturn DigiSizer.



Particle Insight

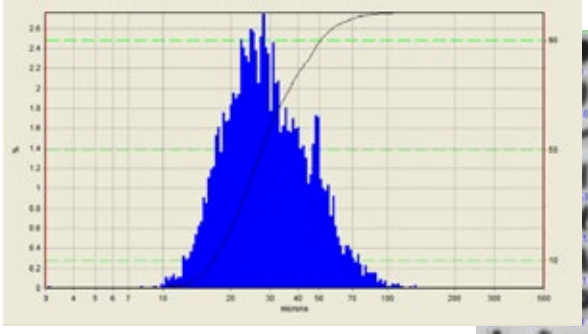
Particle Shape Measurement

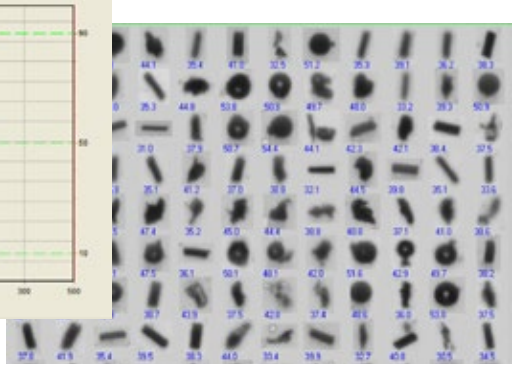
The Particle Insight is a state-of-the art dynamic image analyzer which is ideal for applications where the shape of raw materials, not just the diameter, is critical to performance. The Particle Insight offers up to 28 different shape parameters analyzed and reported in real-time for samples in either aqueous or organic solvent suspensions.

The system operates in a range suitable for a wide variety of industrial specimens from 3 µm up to 300 µm in its standard configuration. Its unique recirculating sample module and precision optics are designed to acquire and report statistically valid measurements quickly, an essential quality control capability in many manufacturing processes. The Particle Insight also has an automated fill and rinse feature allowing for true walk-away operation.

View Collected Data and Thumbnail Images of Each Sub-Component

The classification window allows users to view statistics and statistical listings for all particles or a user defined sub-set of them, filtering by shape type or statistical descriptors for the class of interest. Importantly the user can view each and every particle captured in the analysis.





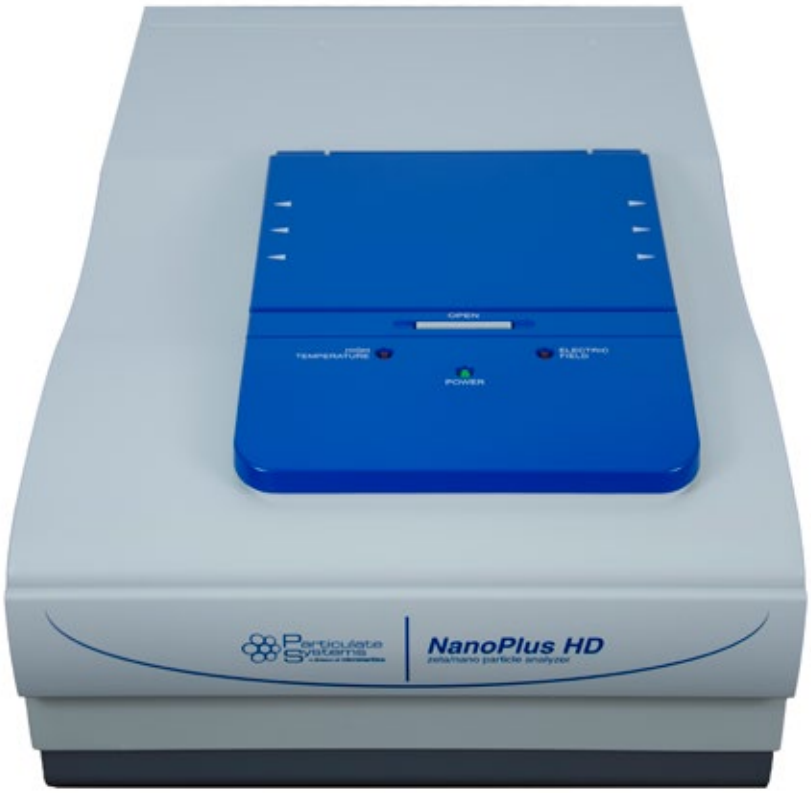
Features:
Real time results: Shape analysis and results in real time; images are analyzed instantly as they are captured

Obtain unprecedented results: Offers 28 Size/Shape parameters, plus the ability to correlate multiple shape measurements

Multi-run sample trending: Statistical Process Control capability to track shape changes over user-defined time intervals

Real-time backup: Data mirroring allows simultaneous storing of all analysis data in multiple locations, allowing remote monitoring

NanoPlus HD



Zeta Potential and Nano Particle Size Analyzer

Outstanding performance, sensitivity, compact bench-top footprint, and intuitive software make the NanoPlus series of products the preferred choice in determining particle size and zeta potential on a wide variety of materials. The NanoPlus HD is a unique instrument that utilizes dynamic light scattering (DLS) and electrophoretic light scattering techniques to determine particle size, zeta potential, and molecular weight. The instrument is compact and easy to use with an extended analysis range, intuitive software, and multiple sample cells to fit the user's application.

The NanoPlus HD provides:

- Highly Accurate Zeta Potential Measurements of Concentrated Solutions and a unique cell for flat surface and membrane studies
- True Determination of Electrophoretic Mobility
- Broad Particle Sizing Range with Increased Sensitivity
- Wide Range of Measuring Cells

Specifications

Principle	Photon Correlation Spectroscopy (PCS), Dynamic Light Scattering (DLS)
Scattering Angle	165° Back-scattering
Minimum Sample Volume	Standard Cell: 0.9 mL, Micro Volume Cell: 20 µL
Concentration	0.00001 to 40%
Measurement Range	0.1 nm to 12,300 nm Size Distribution Range
Light Source	Semiconductor Laser Diode
Detector	HD Avalanche Photodiode
Laser Wavelength	660 nm
Laser Power	70 mW
Correlator	Includes both Time-Domain and Time-Interval correlators. Max of 1,000,000 equivalent channels.

**Due to continuous improvements, specifications are subject to change without notice.*

Micromeritics Instrument Corporation

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To request a quote or additional product information, visit

micromeritics.com

Contact your local Micromeritics sales representative
or our Customer Service Department at

770-662-3636

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