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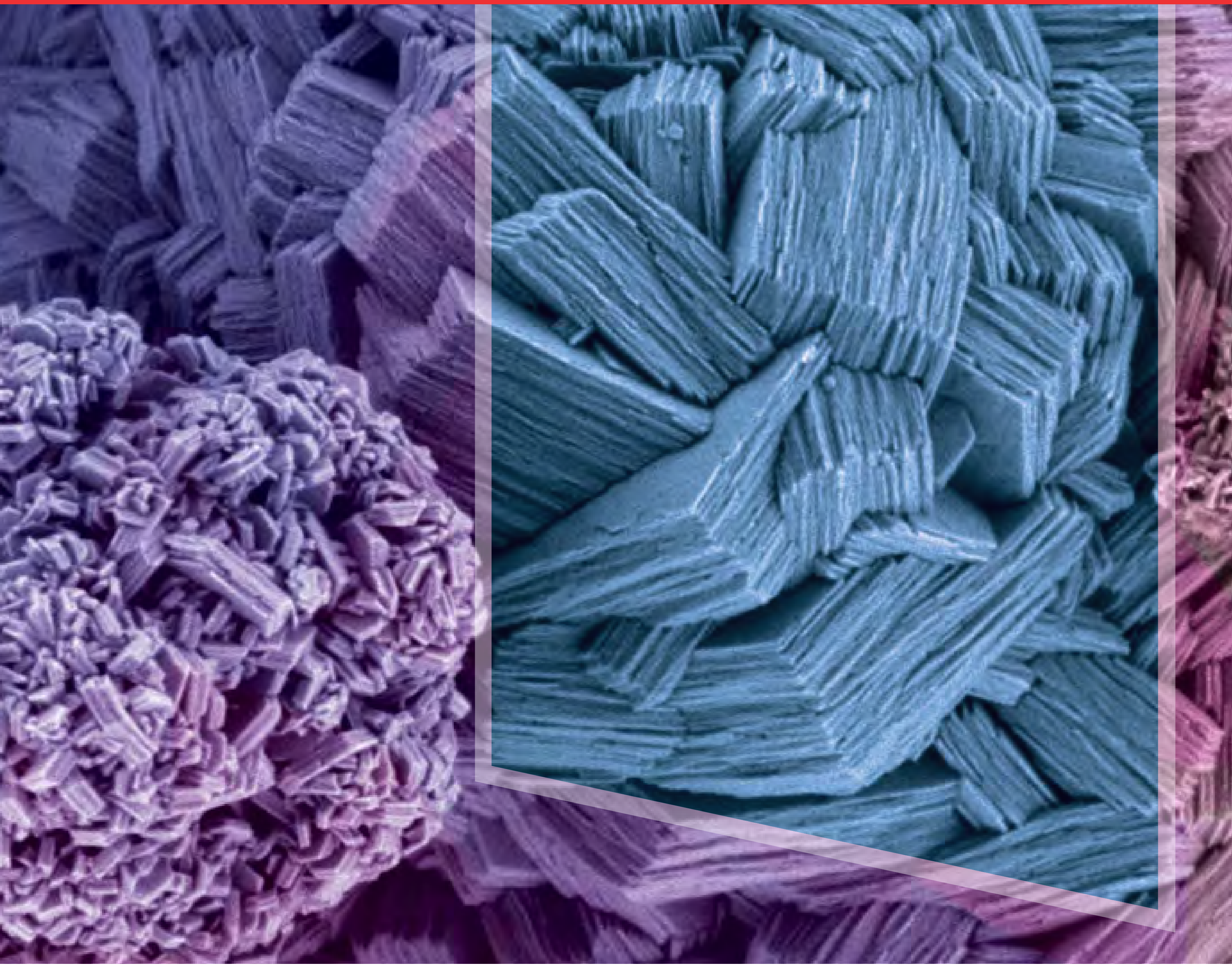


Analytical solutions for improved battery and energy storage products

Thermo Fisher SCIENTIFIC

The global lithium-ion battery market is expected to reach USD 93.1 billion by 2025. This is largely driven by increased usage in electric vehicles, grid storage, and portable consumer electronics where its higher energy density over that of lead-acid batteries is of primary importance.

However, in order to increase performance and to obtain a better understanding of the different degradation mechanisms further research is required. Battery performance is dependent on the active materials used and in the morphology of the battery. While fundamental degradation mechanisms occur inside active cathode materials and are affected by chemical interactions during charging-discharging cycles.

Evaluation of batteries and battery components requires a variety of analytical methods that study bulk materials and component surfaces at various scales. As the world leader in advancing science, Thermo Fisher Scientific provides the widest range of analytical instrumentation for battery analysis and product formulation, including X-ray photoelectron spectroscopy (XPS/ESCA), electron microscopy (SEM, TEM and FIB-SEM), optical spectroscopy (FTIR, Raman, & NIR), mass spectrometry (GC-MS, HPLC, LC-MS, HREMS-MS, TEA), microCT, nuclear magnetic resonance (NMR), X-ray diffraction, X-ray fluorescence, torque rheometry and extrusion, and viscometry.

Imaging techniques such as Raman, microCT, and electron microscopy are mainly used to study the 2D and 3D morphology of battery components at different stages in the lifecycle. These techniques cover the full length scale from the cell level with Raman and microCT down to the atomic scale with TEM. 3D imaging provides complete geometric evolution of the cathode microstructure upon cycling. Geometric parameters such as volume fraction, surface area, particle size distribution and tortuosity are typically assessed using a combination of microCT and FIB–SEM techniques.

Spectroscopy, NMR, X-ray diffraction and mass spectrometry are key to study the evolution of structural changes and the defect formation in battery electrodes. These techniques permit the analysis of electrode materials as they change during the redox reactions; and give information on both crystalline and amorphous phases. Local differences in Raman spectral changes can create a state-of-charge (SOC) distribution map showing the composite electrode. The composition of the Solid Electrolyte Interface (SEI) is commonly studied with *ex situ* XPS and *in situ* FTIR and Raman spectroscopy to monitor the SEI formation.

The rheometry and viscometry systems measure the dispersiveness and coating capability of battery materials in the electrode slurry. Torque rheometers deliver information about melting behavior, influence of additives on processability and temperature or shear stability; all critical parameters for the production of polymer separators.

Electron microscopy

Imaging techniques such as microCT, SEM, FIBSEM and TEM are mainly used to study the 2D and 3D morphology of battery components at different stages in the lifecycle. These techniques cover the full length scale from the cell level with microCT down to the atomic scale with TEM. 3D imaging is necessary to provide a complete geometric evolution of the electrode microstructure upon cycling. Geometric parameters such as volume fraction, surface area, particle size distribution and tortuosity are typically assessed using a combination of microCT and FIBSEM techniques.

TEM

Thermo Scientific Themis Z Transmission Electron Microscope

FIB

Thermo Scientific Helios G4 PFIB CXe DualBeam Microscope

On the cover:

Cross section of a lithium carbonate (Li_2CO_3) particle and nickel manganese cobalt (NMC) particles which provide the lithium to a battery system.

Electron microscopy at a glance

High resolution 2D Imaging in the SEM

- Structural characterization of electrodes and separator
- Cathode active particle morphology

Quick and easy imaging in the desktop SEM

- For fast analysis, even inside the glovebox.
- Quality control on graphite particles in the anode

3D imaging at the cell level with microCT

- Micrometer resolution, optimized for cylindrical samples
- Quality control of cathode sheets

3D imaging at the electrode level with PlasmaFIB

- Representative volumes for transport property calculations
- A 100 micrometer wide section of a battery cathode as part of a 3D reconstruction

3D Imaging at the active material level with FIBSEM

- Nanometer resolution with high contrast in 3D
- Section through an NMC cathode particle

Atomic scale characterization with TEM

- Structural characterization of the SEI and different active material chemistries
- Direct imaging of Li-atoms in the lithium oxide crystal structure

2D and 3D Image Analysis with the Avizo Software Suite

- Visualization, segmentation and modeling based on 2D and 3D datasets
- Three phase segmentation of a battery cathode

FTIR

Fourier-transform Infrared Spectroscopy (FTIR) is a commonly used vibrational spectroscopy that reveals molecular information about the sample. FTIR spectrometers accommodate an array of sampling accessories. Several third-party companies also offer a number of specialized FTIR sampling accessories useful in battery research.

The Thermo Scientific™ Nicolet™ iS50 FTIR spectrometer accommodates technique modules for FT-Raman, IR-TGA, IR-GC and a dedicated NIR unit. All or our spectrometers use our award-winning Thermo Scientific™ OMNIC™ software that provides fast and easy materials identification, and features designed for time-resolved experiments, kinetics and advanced spectroscopic studies such as phase modulation infrared reflection-absorption (PM-IRRAS) spectroscopy.

Enabling maximum sample ease-of-use on all Thermo Scientific FTIR spectrometers are attenuated total reflectance (ATR) sampling. Simply place the sample over the reflectance crystal (diamond, germanium), clamp down the material and acquire data. The use of Raman and IR spectroscopy as complementary techniques means that Raman can be used to examine structural changes in the electrode material and IR to probe the interface between the lithium and the organic electrolyte.

In developing research for battery materials FTIR has been shown to be useful in providing specific information about chemical bonds and functional groups used to identify transient lithium species. FTIR is non-destructive and supported by a comprehensive library of IR spectra for common lithium species. FTIR supports researchers actively exploring batteries alternatives like Li–S, Li–O2, Na-ion, Mg batteries and different metal–organic batteries.

Due to its versatile sample handling capabilities and large accessory chamber, FTIR is now found in several *in operando* experiments to investigate the decomposition process of the electrolyte solutions. *In situ* FTIR can provide real-time information about the chemical nature of adsorbates and solution species as well as intermediate/product species involved in the electrochemical reactions.

FTIR at a glance

- Acquires infrared spectra of solids, liquids and gases
- Provides information about chemical bonds and functional groups
- Spectral fingerprinting enables rapid identification of unknowns
- ATR sampling provides quick and easy data collection
- Multiple sampling capabilities for specialized studies
- Monitors time-based and dynamic events
- *In operando* and *in situ* sampling capabilities

FTIR

Thermo Scientific Nicolet iS50 FTIR spectrometer

Raman microscopy

[Raman](#) spectroscopy provides a structural fingerprint by which molecules in a sample can be identified. The Raman technique measures the energy shift in light scattered photons that yields molecular and structural information. Combining small spot microscopy with precision stage movement enables chemical mapping across a sample area. Raman spectroscopy is an important technique in analyzing various forms of carbon such as graphene and graphite, and diamond-like materials.

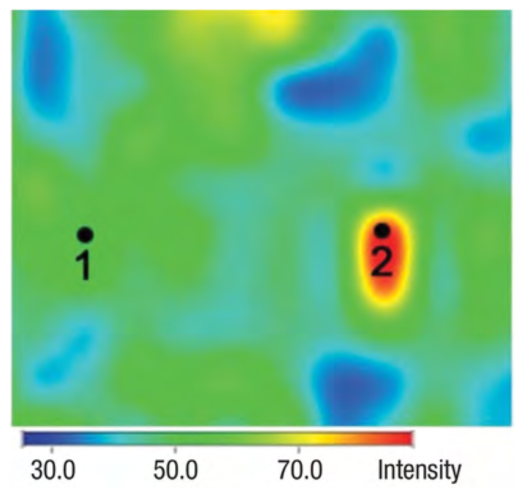
Raman is a useful technique in developing alternatives to lithium cobalt oxide cathodes, such as manganese spinel material. Raman is useful in looking at lithium cobalt doping with transition metals to synthesize materials with different morphologies.

In developing anode materials, Raman has been useful in studying carbon allotropes besides graphite. Raman spectral data can be used to determine the number of sheets of graphene in a stack, provide information on defects and disorder in the structure of graphene, and determine diameters of single wall carbon nanotubes.

Raman spectroscopy can be used to study the degree of association of electrolyte ions in solutions and in polymer materials. The association of ions has a direct effect on the ion mobility and ion conductivity and thus affects battery performance. Raman can measure the effects of additives used to suppress the crystallinity of the polymer matrix and to improve the mechanical and electrochemical properties of the resulting composite polymer electrolytes.

Raman at a glance

- Visualize chemical compositional distribution on component surfaces
- Profile chemical changes during battery cycling
- Evaluate the spatial distribution of phases in a sample
- Ideal for studying allotropes of carbon and transition metals



A Raman map showing the distribution of the two different spinel phases in a sample. The red-yellow locations (such as location 2) indicate areas of the P4332 phase whereas the blue-green areas (such as location 1) represent areas of the Fd3m phase. Mapping data collected using a DXR2 Raman microscope with a motorized stage and Atlµs software.*

Raman

[Thermo Scientific DXR2xi Raman Imaging microscope](#)

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XPS

X-ray Photoelectron Spectroscopy (XPS) is a surface analysis technique that provides elemental and chemical state information about the top layer(s) of a material. XPS detects photoelectrons emitted from a surface exposed to an X-ray beam. Under typical laboratory conditions, these photoelectrons have relatively low kinetic energies meaning that only the top few nanometers of the sample are observed.

XPS instrumentation provides a quantified composition of not only the elements present, but also their chemical state. Instruments are usually equipped with an ion beam for removing material from the sample so that analysis can be extended deeper into surface. All elements from lithium and above in the periodic table can be detected.

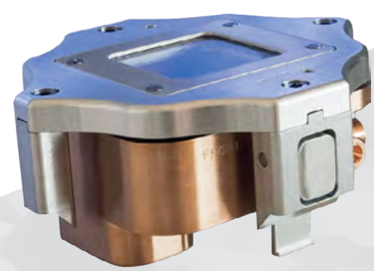
As a surface technique, XPS is essential for understanding the interfaces between electrolytes and electrodes. Cathode and anode materials of Li-ion cells can be investigated to confirm changes in composition following cycling, to understand the oxidation states of the cathode components, and to determine the variation with depth of the solid electrolyte interphase layer as it develops. XPS has been proven useful in studying surface pre-treatment of graphitic electrode materials to significantly reduce the irreversible consumption of material during battery charging.

XPS at a glance

- Detect top ~10 nm of sample
- Quantified chemical state analysis
- Depth profiling of hard and soft materials
- Vacuum Transfer Module for air-sensitive materials

XPS

Thermo Scientific Nexsa Surface Analysis System



The vacuum transfer module allows samples that have been prepared in an inert environment to be transferred *ex situ* into the spectrometer chamber without exposure to air.

XRF

X-ray Fluorescence (XRF) spectroscopy is an elemental analysis technique that identifies and quantifies the composition of a broad range of materials. XRF measures characteristic X-rays emitted from atoms in the sample that have been energized.

Energy-dispersive XRF (EDXRF) is a rapid screening technique that captures X-ray energies across the spectral range from fluorine to uranium, identifying specific elements or unknowns.

Wavelength-dispersive XRF (WDXRF) uses crystals to disperse the fluorescence spectrum into individual wavelengths of each element (beryllium to americium), providing high resolution and low background spectra for accurate determination of elemental concentrations.

Quickly identifies elements and their concentrations within battery/energy storage product materials. XRF can also perform elemental mapping and small spot analysis for identifying defects, inclusions or inhomogeneities down to 0.5 mm. XRF determines the thickness of layers; both conducting and insulating films, non-destructively and consistently.

EDXRF at a glance

- Analyzes bulk solids, granules, powders, thin films and liquids
- Qualitative or quantitative elemental analysis
- Identifies major, minor and trace elements
- Simultaneous identification of elements from F to U.

WDXRF at a glance

- Bulk elemental analysis with mapping and small spot analysis
- Analyze materials in varied sizes, coatings, layers, heterogeneities and inclusions
- Covers wide concentration ranges and varied samples matrices

XRD

X-ray Diffraction (XRD) is a structural analysis technique that is typically used to identify and quantify mineral composition, phases or compounds in powder materials or thin films and layers.

X-ray diffraction systems can be used to do *in situ* and *in operandi* measurements to optimize formulations with suitable structural forms of the lithium and related materials such as polymers and/or graphite based materials.

XRD at a glance

- Real-time rapid determination of polymorphic structures
- Measure percentage of crystallinity and study phase transitions
- Observe the stability of the materials at high temperatures, reactivity, texture and stress

Thermo Scientific ARL PERFORM'X

Thermo Scientific ARL QUANT'X

Thermo Scientific EQUINOX 100

Rheology

Rheology is the study of flow and deformation of materials. The techniques investigates properties that correlate strongly with the microstructure of a material and are an indicator for any structural changes.

Rheometers are high performance instruments used for extended quality control applications as well as for research purposes.

The electrodes of lithium batteries are made by coating concentrated solid suspensions (slurries) onto metal foils, forming a thin porous layer. The electrochemical performance of a lithium ion battery is determined by how homogeneously the active components are distributed within the slurry and how uniform the slurry is coated onto the substrate. To optimize this performance, the rheometer provides critical information about the complex viscoelastic properties of battery slurries.

Rheology at a glance

- Investigate viscosity, yield stress and the viscoelastic moduli in formulations
- Study the complex viscoelastic properties of battery slurries
- Determine flow properties in a coating process that produce a continuous and even coating

Twin-screw extrusion

Extrusion is a formulation and manufacturing process that melts and compounds viscous materials with additives and fillers. The extruder transports materials through specialized screws and heating zones to mix ingredients or create new compounds. The Thermo Scientific Process 11 Twin-screw Extruder is a bench-top instrument capable of supporting the end-to-end process of battery/fuel cell manufacturing.

An important topic in lithium-ion battery (LIB) technology is the use of solid-state polymer electrolytes (SPE). Twin-screw extrusion methods can play an essential role in developing a molecular dispersion of the polymer matrix with lithium salts and other additives, and thus help the search for improved formulations.

Twin-screw extrusion at a glance

- Develop separator films using either a dry or wet compounding process
- Produce graphene and lithium cobalt oxide ($LiCoO_2$) slurries
- Flexible setup and low sample volumes
- Screw geometries comparable with larger equipment for efficient scale-up to manufacturing

Rheology

Thermo Scientific HAAKE MARS Rheometer

Extrusion

Thermo Scientific Process 11 Twin-screw Extruder

If you'd like to speak to an application specialist please visit **thermofisher.com/specinquiry**

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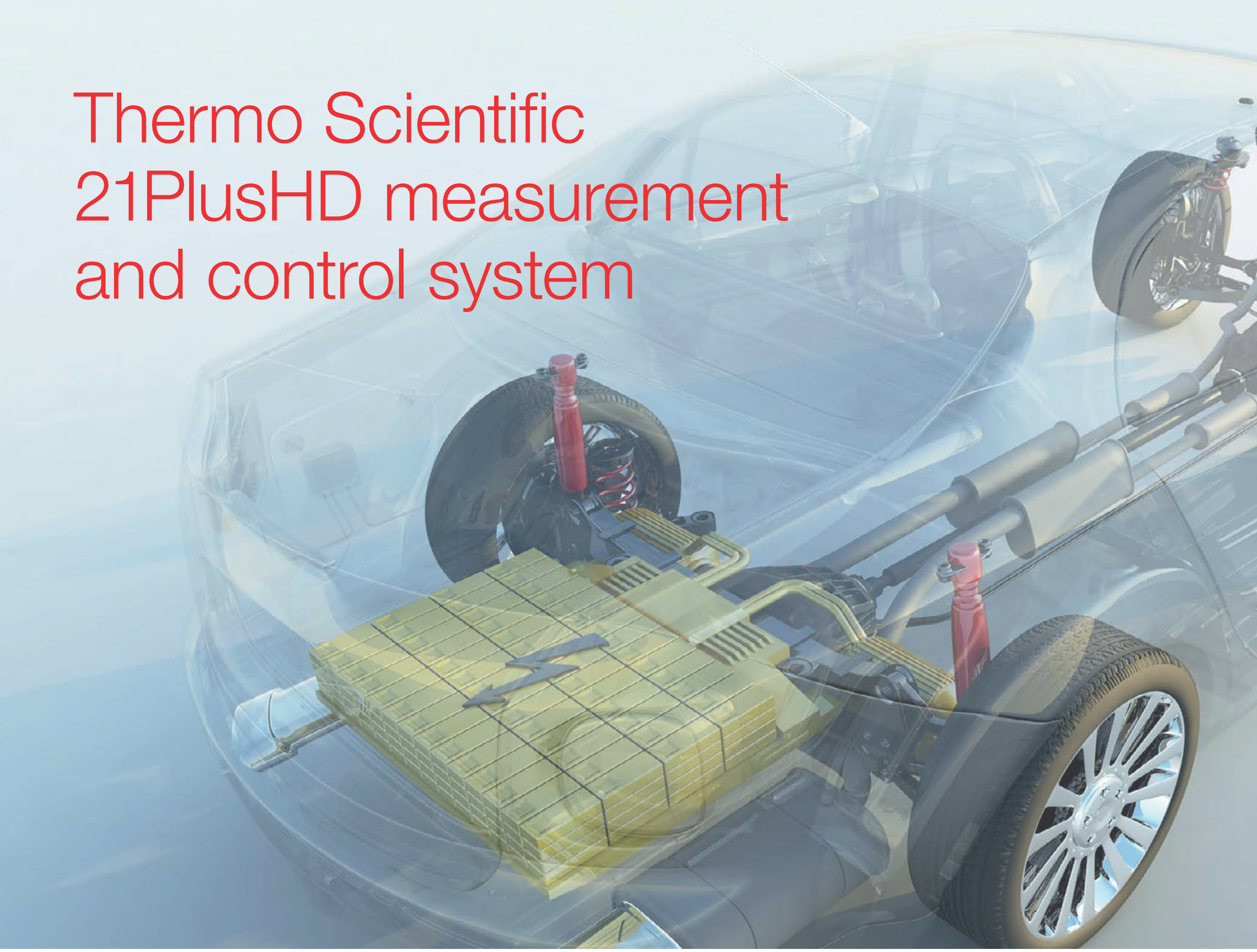
Thermo Scientific 21PlusHD measurement and control system



Solutions for lithium-ion battery

ThermoFisher SCIENTIFIC

Thermo Scientific 21PlusHD measurement and control system

Solutions for lithium-ion battery

Application overview

From small hand-held electronics to medium-sized electric vehicles such as cars, buses and trucks, to larger marine vessels and smart-grid energy storage systems, lithium-ion battery technology is changing our lives. Critical to the advancement of the battery is the emergence of higher quality separator film, the coating of separator film for higher efficiency, and the coating of the anode and cathode. Discover how the Thermo Scientific™ 21PlusHD measurement and control system from Thermo Fisher Scientific is helping the industry improve the quality, consistency and productivity of lithium-ion batteries.

Separator film

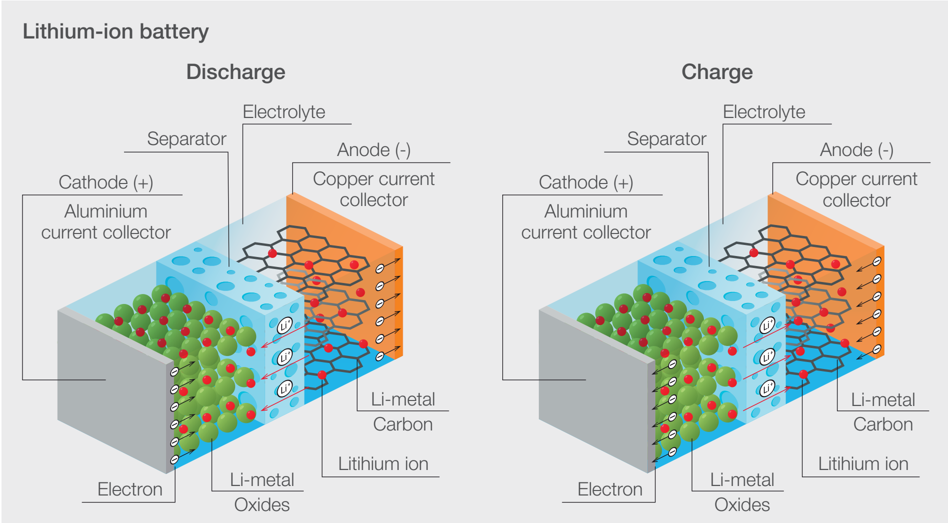
Separator film is one of the key components of a lithium-ion battery. It is a thin but permeable layer of film used to separate the anode from the cathode and prevent short-circuiting while facilitating the flow of charged ions. Separator films are produced either by a dry or wet process to create the required micro porous structure. Therefore, consistent thickness of the film and homogenous distribution of the pores are necessary to optimize the performance and life of the battery. Additionally, separator film can be coated with ceramic or another material to improve efficiency.

Anode and cathode coating

Continuous or patch coating on an aluminum substrate for the cathode or on a copper substrate for the anode is an expensive and challenging operation. Uneven coating of the cathode or anode will result in poor construction of the battery. Even worse, it could create a hotspot lowering the efficiency of the battery, shortening its lifespan, increasing charge time and the risk of thermal runaway. Furthermore, coated products are not recoverable. Therefore, an accurate measurement of the coat weight on both sides of the substrate is paramount to controlling and perfecting the process, and improving yield and quality.

Anode and cathode calendering (press line)

After the electrode coating is controlled to the proper coat weight, the electrode material is calendered for homogeneous thickness and particle size. Pressing the ingredients of a composite electrode improves electrical contact and adhesiveness, and assures the desired geometric and electrochemical characteristics. Proper coating thickness measurement and calender control is critical not only to avoid excessive calender pressing, which could destroy the porous nature of the electrode, but also to provide final dimensional accuracy and essential product attributes in this important process step.



Direct thickness measurement of separator film

Leveraging over 20 years of experience with on-line infrared measurement technology, the Thermo Scientific™ PROSIS™ thickness sensor is a new breed of advanced non-nuclear measurement technology and in combination with the 21PlusHD measurement and control system, is the ideal solution for measuring the thickness of separator film. Designed to provide the highest accuracy and measurement resolution possible, the PROSIS thickness sensor has a wider spectral coverage range that allows it to measure more materials than ever before. Unlike any other IR sensing technology available on the market, the PROSIS thickness sensor analyzes the full spectral response of the separator film to infrared IR energy and provides the most accurate thickness measurement possible.

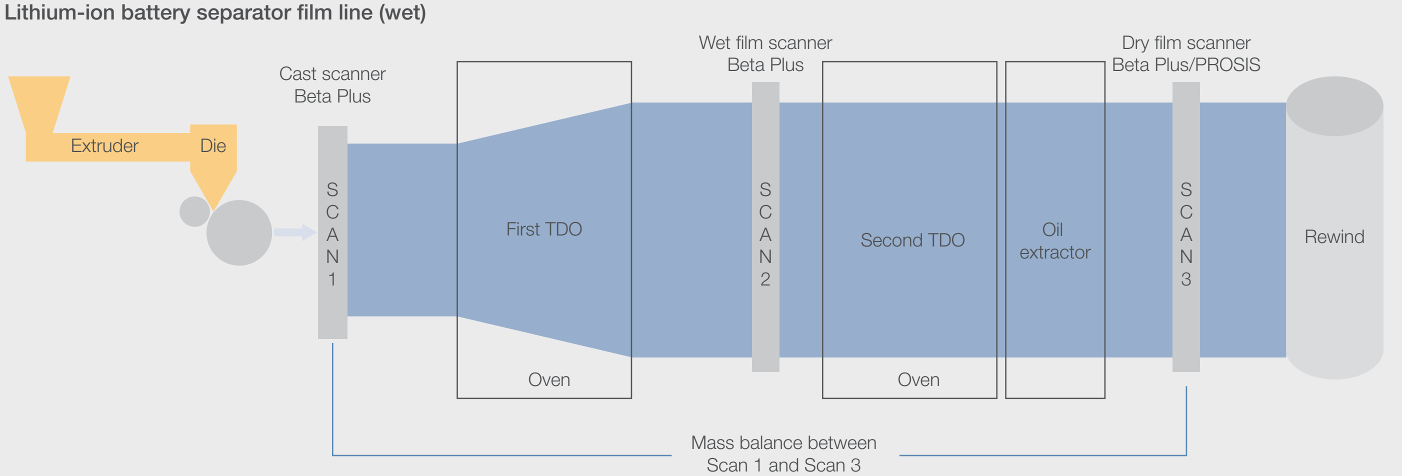
Coupled with options like Automatic Profile Control (APC) and Machine Direction Control (MD), the 21PlusHD measurement and control system will provide measureable return on investment through higher quality production, increased yield and decreased scrap.

The Thermo Scientific™ Beta Plus basis weight sensor can also be used for the measurement and control of separator film and is especially suitable for film coated with ceramics or other similar inorganic material.

Applications

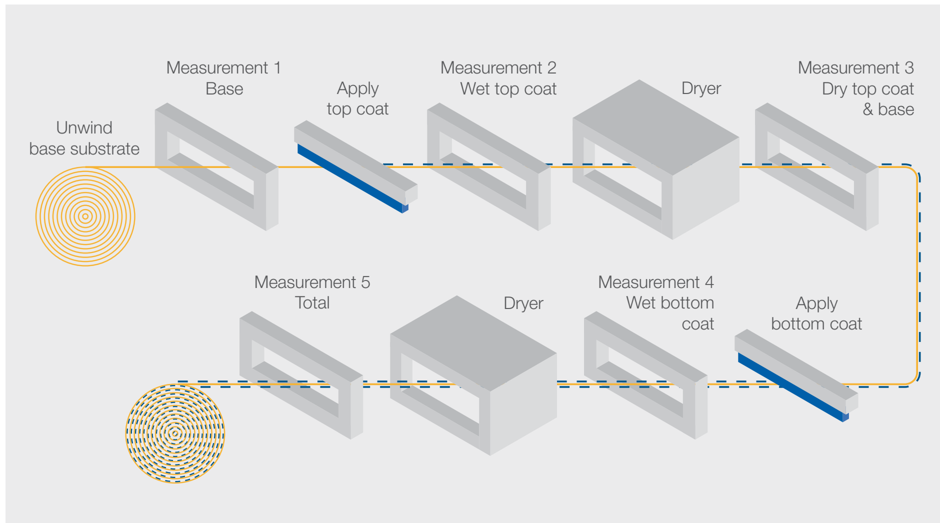
- Film thickness
- Wet and dry weight
- Oil
- Density
- Integrated mass balance die mapping
- Coated and uncoated films

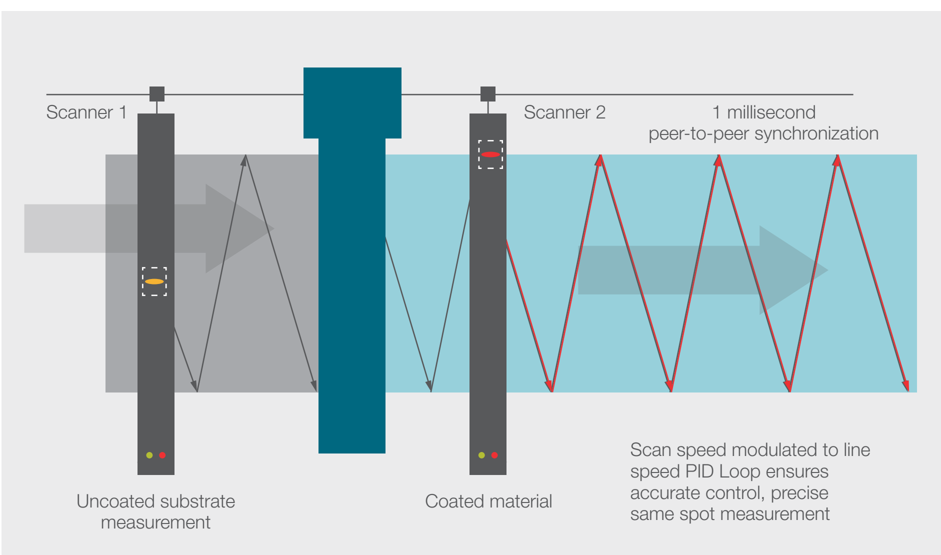
Lithium-ion battery separator film line (wet)



Coat weight measurement of anode and cathode

Anode / cathode coating line
The Beta Plus basis weight sensor is the ideal sensing technology for measuring the coatings on the cathode and anode. The Beta Plus basis weight sensor features exceptionally high signal with extremely low noise and a unique isotope geometry that results in the most accurate coating weight measurement possible and highest edge resolution. With a dynamic scanning repeatability of ±0.025% (Krypton-85 model), the performance of the Beta Plus basis weight sensor in combination with the 21PlusHD measurement and control system is unparalleled.



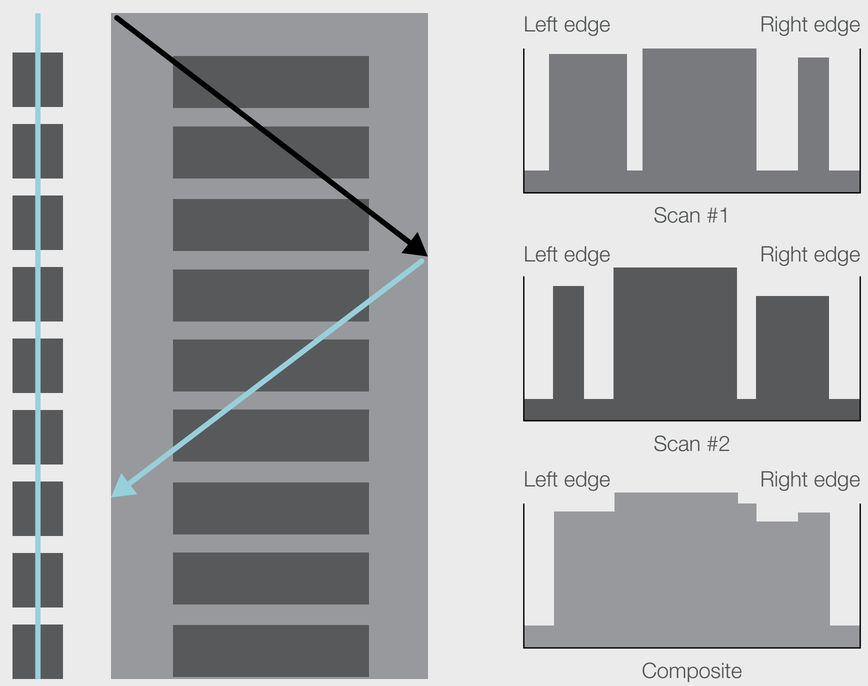


Thermo Scientific Exactrax same spot measurement
Combining the Thermo Scientific™ Exactrax™ same spot measurement synchronization with the lithium-ion battery coating application package available on the 21PlusHD measurement and control system, it is possible to intelligently measure the correct coat weight by recognizing both coated and uncoated sections of the anode or cathode. This will ensure high quality and in-spec production and help reduce the risk of non-uniformity and other downstream battery production issues.

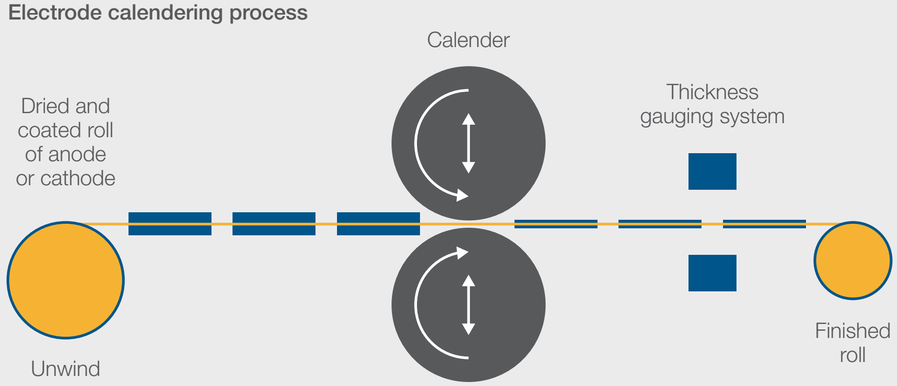
Thickness measurement of anode and cathode

The battery calendering application package completes the Thermo Scientific suite of solutions for lithium-ion battery. It uses a dual displacement laser C-Frame to accurately measure, control, and provide key dimensions of calendered electrode materials. This robust solution is available for electrode thicknesses from 0.2 to 20mm, with an accuracy of ±1.0 µm. Its high-speed scanning capability maximizes measurement coverage in patch and strip coating applications, while its hardened design can withstand temperatures of up to 50°C with an available cooling option.

Patch coating example



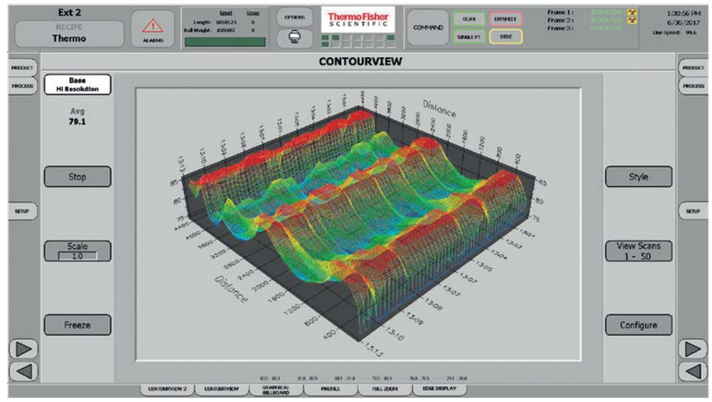
Electrode calendering process



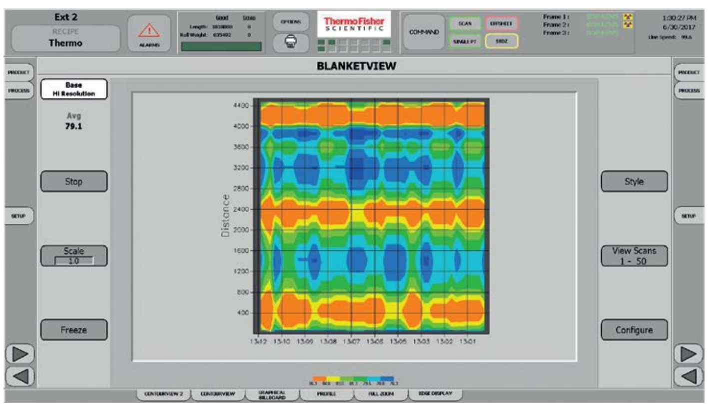
The combination of advanced control strategies, analytics and reporting capabilities (roll, shift, and production reports, Contourview 3D profile displays, zoom displays, SPC/QC control charts, and defect mapping), make the 21PlusHD measurement and control system a high performance platform, specifically designed to increase the quality, consistency and performance efficiency of lithium-ion battery separator film, electrode coating, and electrode calendering production lines.

21PlusHD measurement and control system example displays

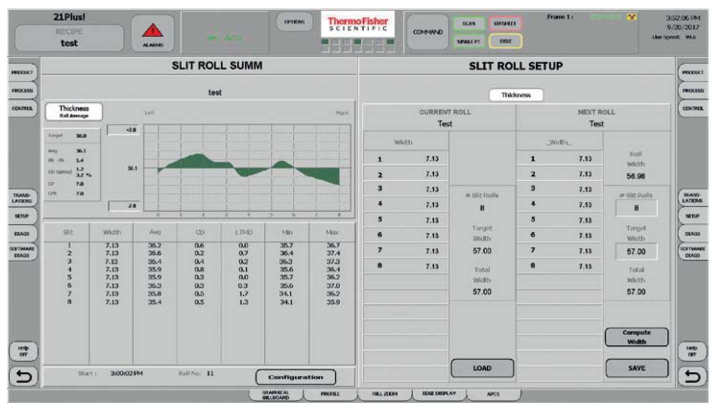
Profile with Edge Zoom



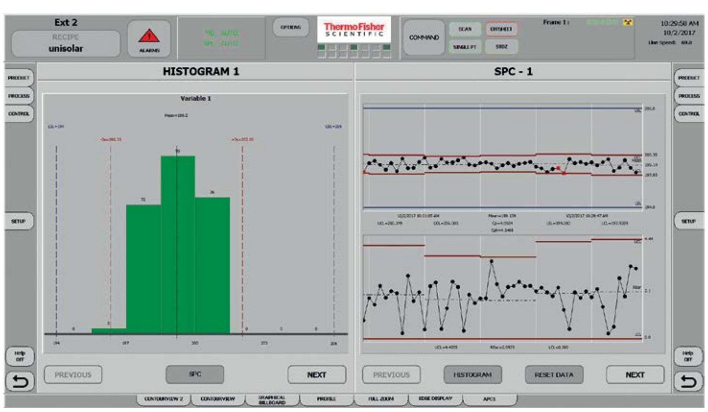
3D ContourView



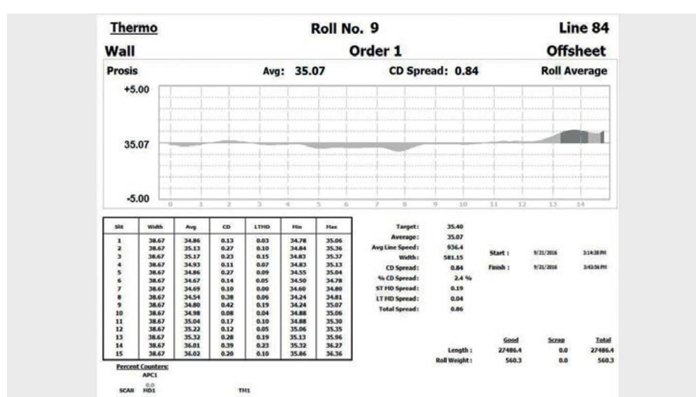
2D HeatMap



Slit Roll Summary



SPC Control Charts



Roll Report

Support you can depend on

Thermo Scientific products are supported by our extensive network of qualified application engineers who will work closely with you to understand and evaluate your specific production parameters. Our experts will help you choose the right instruments for your application, then keep them performing to spec. Their goal is to optimize your process today, and also lay the foundation for easy upgrades in the future.

Product maintenance

Our comprehensive service offering is based on corrective and preventative maintenance that not only reduces downtime, but also helps you improve your process. We offer multiple levels of support agreements, with varying degrees of access and response, including:

- System commissioning
- System calibration
- Preventative maintenance
- On-site repair
- Depot repair

Some options feature complete cost predictability, with all travel, labor, spare parts, and consumables included.

Education and training

We offer multiple training options to help you increase productivity by optimizing the use of your instruments and expanding the skills of your operators. You can receive hands-on instruction in your plant or at one of our training facilities in the USA, Europe and Asia. Our range of courses covers:

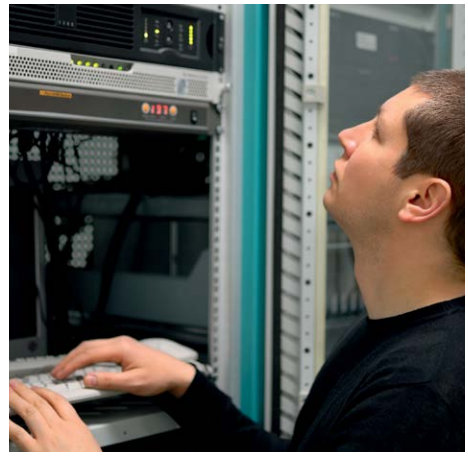
- Basic operation
- Calibration
- Routine maintenance
- Troubleshooting
- Certification

We will also work with you to develop a custom program that meets your specific training objectives, often incorporating your own operating procedures.

Professional services

Our certified engineers are available to review your process, perform benefit analysis and recommend improvements to help you meet your best-practice goals. We will develop an implementation plan that integrates all Thermo Scientific systems, as well as third-party components including:

- System layout and connectivity
- Software implementation, configuration and support
- Site modifications



You can rely on us to manage the entire installation and start-up if you choose, including serving as a liaison with licensing agencies where necessary.

Parts and upgrades

Our spare parts are designed specifically for your Thermo Scientific system, and we make it easy for you to secure high-quality, low-cost replacements by maintaining offices around the world that respond quickly to your phone or online requests. You can also extend the lifetime of your older instruments with our add-on system enhancement and retrofit packages, which adapt your instruments for new uses and eliminate the time and cost to retrain operators on new equipment.

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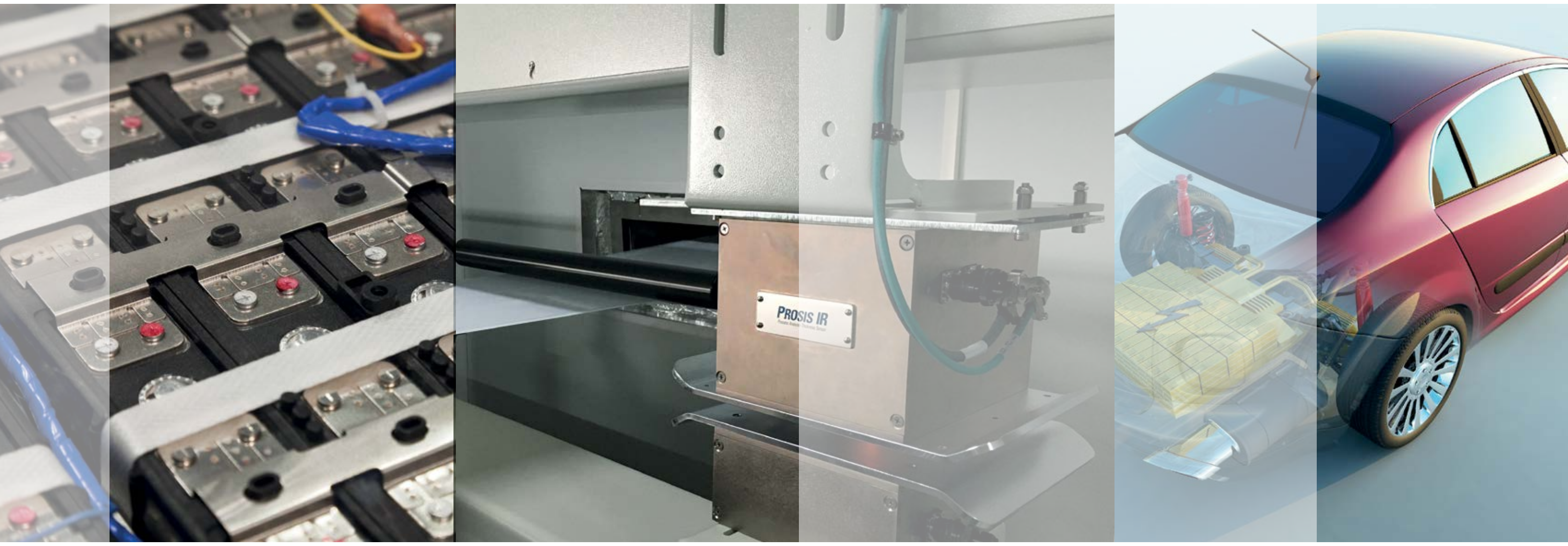
21PlusHD measurement and control system

Solutions for lithium-ion battery



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