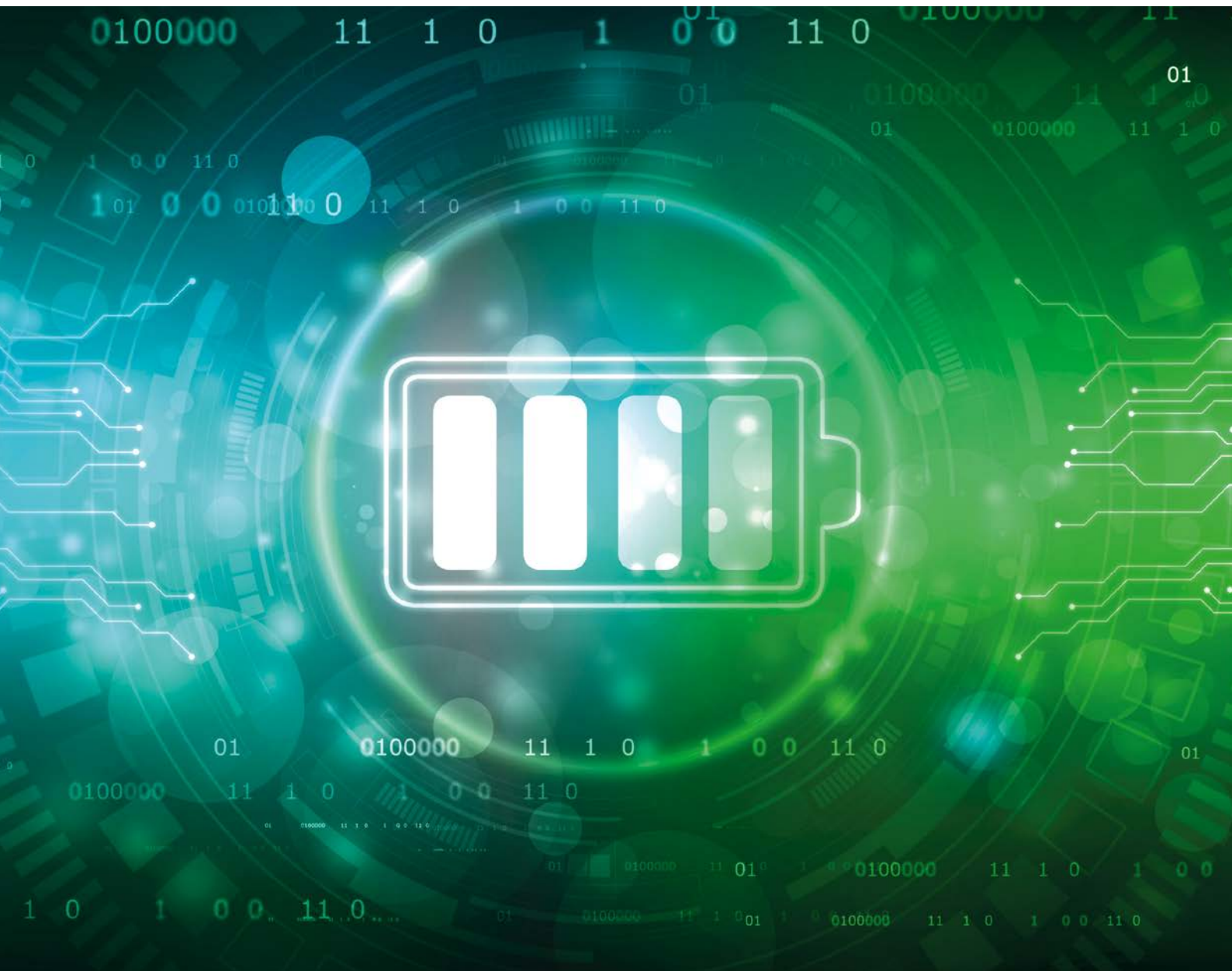


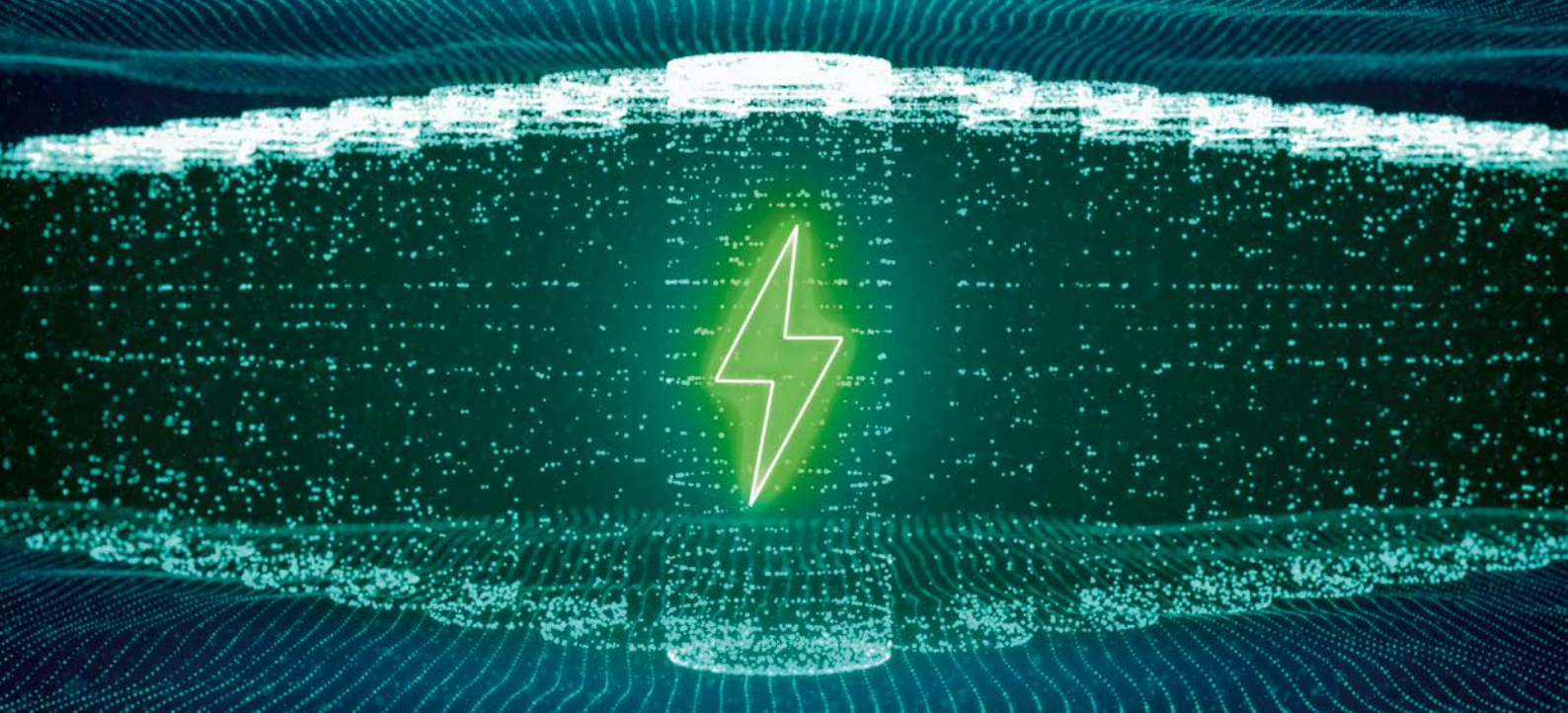
# NETZSCH

Proven Excellence.



## Thermal Analysis and Rheology of Batteries

Analyzing & Testing



# THERMAL ANALYSIS AND RHEOLOGY OF BATTERIES

Engineers often face challenges when designing lithium ion batteries. A thorough thermal management strategy is required to enhance performance, improve battery life, and to keep batteries from going into thermal runaway. Thermal management starts with careful characterizations of battery components for the development of inherently safer batteries. Design parameters, for intended use, manufacturing processes and operating conditions all have an effect upon battery temperature during use.

Manufacturers of battery components must consistently deliver on quality throughout the entire manufacturing process. Continuity of the manufacturing process means that defects or contaminants accumulate at an early stage and have a much greater impact further down the production line. Quality and critical parameters that could affect battery performance should be monitored at every stage – from raw materials to cell assembly.

Today, the development of powerful batteries with increased capacity, a longer lifetime, shorter charging times, lower weight and size becomes even more crucial due to the change in mobility.

Since 1962, NETZSCH has consistently provided our customers with the latest thermal analysis techniques, the broadest range of highest quality products, the most comprehensive technical support and the highest level of service. Whether you are a battery component manufacturer looking for greater process efficiency and better quality control, or a researcher trying to determine the performance parameters of newly emerging battery materials, our solutions will offer you the insight and control needed to power the development and production of superior-quality batteries.

# MONITORING AT EVERY STAGE – FROM RAW MATERIALS TO COMPLETE CELL TESTING

## Critical Parameters

Battery Materials/ Components	Critical Parameters	Analytical Techniques	Product Solutions
Cathode precursor & electrode materials	Crystal phase	Simultaneous Thermal Analysis coupled to Mass Spectrometer	STA 509 <i>Jupiter</i> ® series coupled to QMS 505 <i>Aëolos</i>
Battery slurry	<ul style="list-style-type: none"> <li>■ Viscosity</li> <li>■ Viscoelasticity</li> <li>■ Stability</li> </ul>	<ul style="list-style-type: none"> <li>■ Rotational Rheometry</li> <li>■ Capillary Rheometry</li> </ul>	<ul style="list-style-type: none"> <li>■ Kinexus Prime Series</li> <li>■ Rosand Series</li> </ul>
Electrode and electrode coating	<ul style="list-style-type: none"> <li>■ Thermal management (thermal diffusivity/ conductivity)</li> <li>■ Specific heat capacity</li> <li>■ Thermal stability</li> <li>■ Reactivity</li> <li>■ Performance changes</li> <li>■ SEI Formation</li> </ul>	<ul style="list-style-type: none"> <li>■ Laser/Light Flash Analysis</li> <li>■ Differential Scanning Calorimetry</li> <li>■ Isothermal Calorimetry</li> <li>■ Accelerating Rate Calorimetry</li> </ul>	<ul style="list-style-type: none"> <li>■ LFA 717 <i>HyperFlash</i>®</li> <li>■ DSC 500 <i>Pegasus</i>®</li> <li>■ MMC 274 <i>Nexus</i>®</li> <li>■ ARC® 244/305</li> </ul>
Electrolyte	<ul style="list-style-type: none"> <li>■ Thermal stability</li> <li>■ Degradation with air/moisture</li> <li>■ Performance changes</li> </ul>	<ul style="list-style-type: none"> <li>■ Simultaneous Thermal Analysis coupled to Evolved Gas Analysis (e.g., QMS) or only TGA</li> <li>■ Differential Scanning Calorimetry</li> <li>■ Isothermal Calorimetry</li> <li>■ Accelerating Rate Calorimetry</li> </ul>	<ul style="list-style-type: none"> <li>■ STA 509 <i>Jupiter</i>® series coupled to QMS 505 <i>Aëolos</i></li> <li>■ TG 309 <i>Libra</i>® <i>Select/Supreme</i></li> <li>■ DSC 500 <i>Pegasus</i>®</li> <li>■ MMC 274 <i>Nexus</i>®</li> <li>■ ARC® 244/305</li> </ul>
Binder, additives	<ul style="list-style-type: none"> <li>■ Thermal behavior</li> <li>■ Viscosity</li> <li>■ Viscoelasticity</li> </ul>	<ul style="list-style-type: none"> <li>■ Simultaneous Thermal Analysis</li> <li>■ Rotational Rheometry</li> <li>■ Capillary Rheometry</li> </ul>	<ul style="list-style-type: none"> <li>■ STA 509 <i>Jupiter</i>® series</li> <li>■ Kinexus Prime series</li> <li>■ Rosand series</li> </ul>
Separator	<ul style="list-style-type: none"> <li>■ Specific heat capacity</li> <li>■ Dimensional change</li> <li>■ QA/QC</li> <li>■ Safety effectiveness</li> </ul>	<ul style="list-style-type: none"> <li>■ Differential Scanning Calorimetry</li> <li>■ Thermomechanical Analysis, Dilatometry</li> <li>■ Isothermal Calorimetry</li> </ul>	<ul style="list-style-type: none"> <li>■ DSC 500 <i>Pegasus</i>®</li> <li>■ TMA 402 <b>F1/F3</b> <i>Hyperion</i>®</li> <li>■ DIL 402 <i>Expedis</i>® <i>Supreme</i></li> <li>■ MMC 274 <i>Nexus</i>®</li> </ul>
Battery cell	<ul style="list-style-type: none"> <li>■ Efficiency of cells</li> <li>■ Thermal stability</li> <li>■ Thermal runaway</li> <li>■ Internal short tests</li> <li>■ Enthalpies</li> <li>■ Thermal conductivity</li> <li>■ Gas production (quantitative and qualitative)</li> <li>■ Vent design</li> <li>■ Safety – CID, PTC</li> <li>■ Kinetic model development</li> <li>■ Isotropic &amp; parasitic reactions</li> </ul>	<ul style="list-style-type: none"> <li>■ Vent design</li> <li>■ EGA coupled to Thermal Analysis</li> <li>■ Isothermal Calorimetry</li> </ul>	<ul style="list-style-type: none"> <li>■ MMC 274 <i>Nexus</i>®</li> <li>■ LFA 717 <i>HyperFlash</i>®/ HFM 446 <i>Lambda Medium</i></li> <li>■ STA 509 <i>Jupiter</i>® series coupled to QMS 505 <i>Aëolos</i>/GC-MS/FT-IR</li> <li>■ PERSEUS STA 509 <i>Jupiter</i>® coupled to GC-MS/MS</li> <li>■ Kinetics NEO</li> </ul>
Material testing	Compatibility between and among the different cell parts	Thermal Analysis in general	

# Thermal Analysis & Rheology

Characterizing each material, measuring compatibility between and among the different components can be achieved by DSC, TGA, STA, MMC and ARC. With this information battery components are engineered to be more thermally stable, produce less heat and react more slowly. The release of toxic, flammable or explosive gases during the decomposition reactions can be studied and mitigated using GC-MS, QMS and FT-IR coupled to the thermal analyzer. The performance of cells can also be measured to determine parameters such as efficiency over the expected operating temperatures and use. For instance, the Coin Cell Module allows users to run performance tests on coin/button cells by isothermal calorimetry over an extended temperature range.

## Understanding Reactions Occurring at Higher Temperatures – Hot Spots!

- How much heat is produced and at what rate?
- How quickly can heat be dissipated inside the cell?
- How likely that the heat triggers additional reactions and additional heat?
- How much gas will be produced and what will the effect be on the cell?

Thermoanalytical methods can help in the design of cells and chemistries that are more resilient to hot spot growth and propagation. Further information can be extracted from the data by creating model kinetics of the chemistries involved. The development of multi-physics models requires information on the physical transport properties of materials being used including the container and the surrounding environment. DSC, LFA, DIL and HFM are appropriate tools to get thermal conductivity, dimensional changes and heat capacity measurements on materials at normal operating and extreme temperatures.



ARC® 305

MMC 274 Nexus®  
with Coin Cell  
Module



HFM 446 Lambda Medium



DIL 402 Expedis® Supreme

LFA 717 HyperFlash®



# PERFORMANCE & SAFETY

Design of Raw Materials



TG 309 *Libra*® Series



STA 509 *Jupiter*® coupled to QMS 505 *Aëolos*

Cell Design



DSC 300 *Caliris*®

STA 509 *Jupiter*®

Life Cycle



Kinexus Prime ultra+

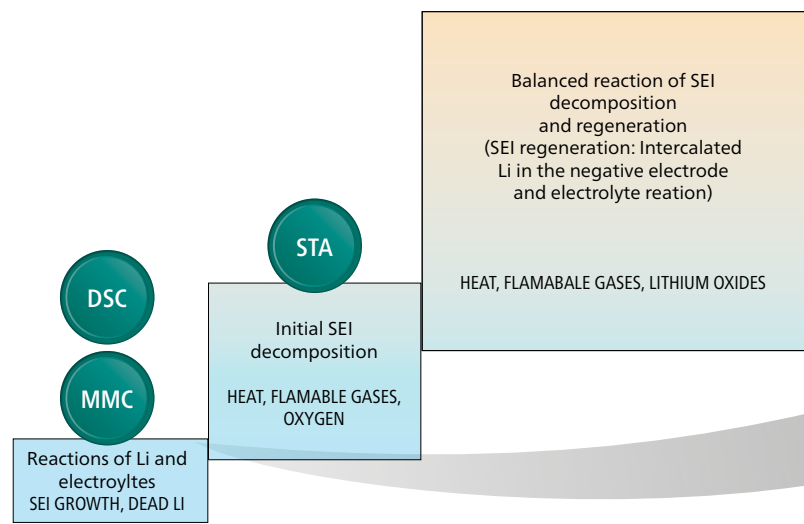
Rosand RH2000

# Thermal Runaway

Li-ion cells are relatively lightweight and have a high energy density. These performance benefits have made them invaluable to the portable energy market. These two benefits also makes them more likely to be involved in thermal runaway. Thermal runaway occurs when the self-heating of cell is greater than the amount of heat that can be removed from the cell. This stored heat in the cell causes the temperature to rise which results in even higher self-heating. Left unchecked, a thermal runaway can cause the cell temperatures to rise rapidly leading to the production of toxic gases, combustion and explosions. Even small cells that go into thermal runaway can lead to physical harm and extensive damage. The impact of these events can be catastrophic.

## Thermal Runaway Occurs Due to

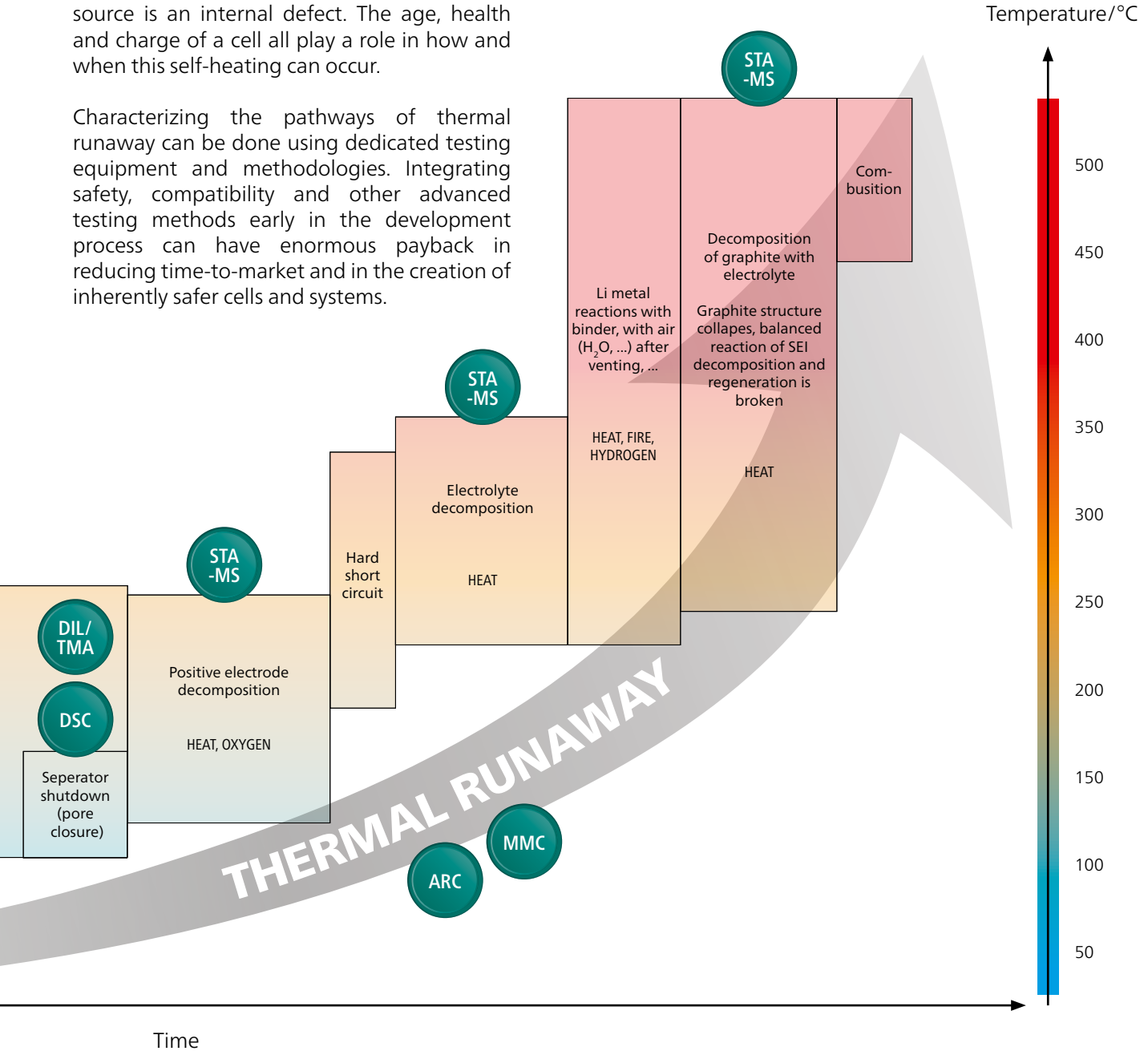
- Environmental conditions/external influences (external or internal short)
- Excessive heat
- Mechanical damage
- Improper charging or discharging of the cell
- Creation of hot spots in the cell due to latent defects within the cell itself
- Heat produced by nearby electronics
- Thermal runaway of an adjacent cell
- Parasitic reactions



Source:  
Swapnil Kanvinde Oct, 18, 2020, Blog on Thermal Management Technologies for Batteries  
<https://skill-lync.com/student-projects/week-10-thermal-management-21>

Where it may be possible to design battery management and safety systems to reduce the frequency and consequence from external sources, this is a bit harder to do when the source is an internal defect. The age, health and charge of a cell all play a role in how and when this self-heating can occur.

Characterizing the pathways of thermal runaway can be done using dedicated testing equipment and methodologies. Integrating safety, compatibility and other advanced testing methods early in the development process can have enormous payback in reducing time-to-market and in the creation of inherently safer cells and systems.



## UNDERSTANDING THE PATHWAYS TO THERMAL RUNAWAY – THERMAL STABILITY

# THERMAL MANAGEMENT

## Controlling Battery Thermal Characteristics – Separator, Electrode and Electrode Coating

Like the human body, the operating temperature of a battery should always be monitored, protected and kept at an optimal level. If the ambient temperature is too low, it will not deliver its full power and reduce the lifetime of the cell. If the ambient temperature rises too high promote the increase of parasitic reactions, reducing efficiency and lifetime. The battery can even swell, catch fire, explode and release toxic gases.

Proper battery thermal management ensures longer lifespan by keeping the cells within a limited temperature range during storage, operation and charging. The State-of-Charge(SOC)-State of Health (SOH) and operating temperature are all interrelated to performance. Understanding how much heat can be dissipated by the cells requires understanding of the basic heat transfer properties of the cell design.

Measuring the thermal diffusivity and thermal conductivity along with the specific heat capacity form the basis for comprehensive understanding. To investigate these thermophysical properties, NETZSCH offers Laser/Light Flash Analysis systems (LFA) as well as systems for Differential Scanning Calorimetry (DSC). Thermal management system failures can be avoided.



### LFA 717 HyperFlash®

- Temperature range:  
-100°C to 500°C
- Various cooling devices
- Thermal conductivity range:  
0.1 W/(m·K) to 3000 W/(m·K)
- Light source: Xenon flash lamp
- Data acquisition: up to 2 MHz
- Automatic sample changer  
for up to 16 samples  
(4 x Ø 25.4 mm, 6 x Ø 12.7,  
16 x □ 10 mm)
- Special sample holders: liquids,  
pastes and powder, fibers, etc.
- Various calculation models,  
corrections and mathematical  
operations
- Model wizard

LFA 717 HyperFlash®

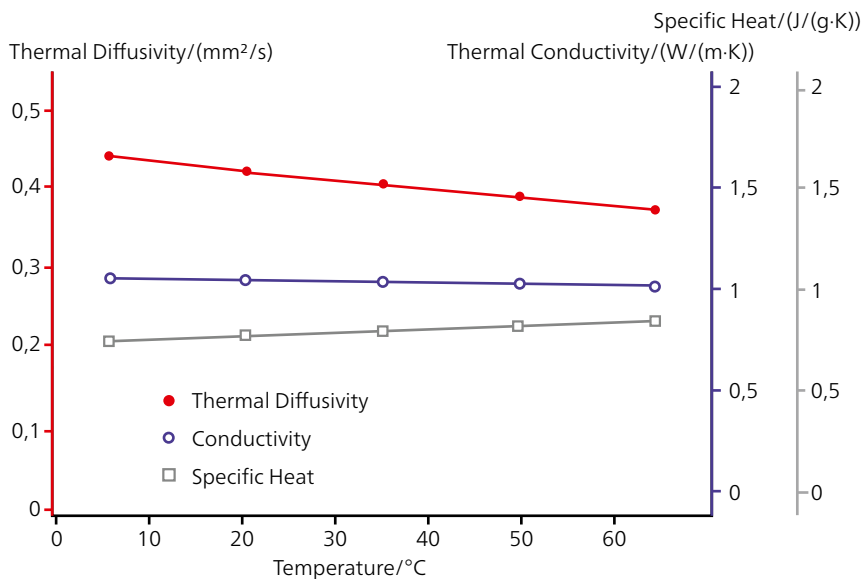


## Laser Flash Analysis

Thermal conductivity and diffusivity are the most important thermophysical parameters for the description of the heat transport properties of a material or a component. The Laser/Light Flash technique has proven itself a fast, versatile and absolute method for measurement of the thermal diffusivity. NETZSCH offers three models, covering the widest temperature range for the broadest spectrum of materials.

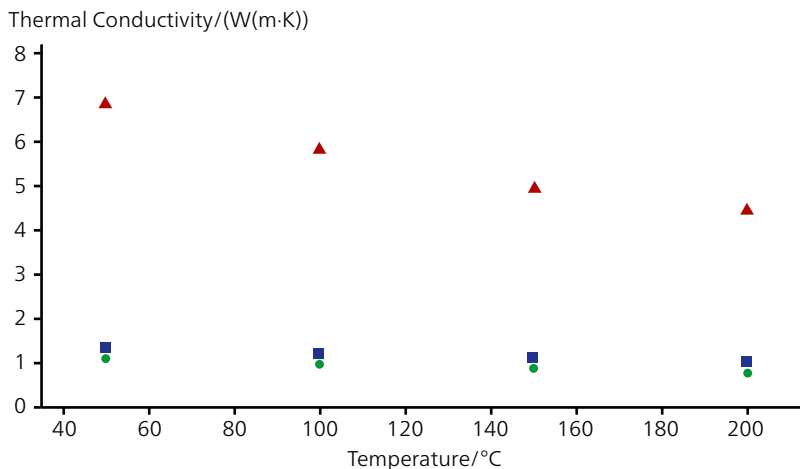
For the investigation of electrodes, electrode coating, separators, the LFA 717 *HyperFlash*® is the right instrument. The patented *ZoomOptics* allows the detector's field of view to be adjusted until the only temperature increase registered is that of the sample and no influences by the surroundings may have any effect.

The ability to get accurate thermophysical property data at the component level is a requirement for detailed modeling. Understanding these same properties for a battery in bulk is necessary for thermal management of packs and modules. During use (or abnormal use), considerable heat can be generated inside the battery. Dissipating these heat as soon as possible is the key to ensure safety.



### Coated Aluminum Electrode

Aluminum electrode coated on both sides: The thermal conductivity (blue curve) is determined by measuring the thermal diffusivity with the NETZSCH LFA *HyperFlash*® (red curve) and specific heat capacity,  $c_p$  (gray curve). In this example,  $c_p$  was determined using DSC.



### Using High Conductive Materials for Heat Dissipation

The integration of a washer with high thermal conductivity can effectively improve the thermal accumulation inside the battery modules and reduce the chance of thermal runaway. The plot shows the thermal conductivity of three different washer materials.

## Thermomechanical Analysis/Dilatometry

Temperature changes the physical properties of materials. Lithiation/Delithiation can also change physical properties. These changes can include dimensional and volumetric changes, changes in strength, flexibility and durability. In a cell, these changes are not uniform and can add mechanical stress and affect material performance. For example, polymer separators can shrink significantly at elevated temperatures which affects the battery performance. To predict the deformation and stresses in the separator in battery cells, it is necessary to measure the expansion/shrinkage behavior.

Thermomechanical Analysis (TMA) determines dimensional changes of solids, liquids or pasty materials as a function of temperature and/or time under a defined mechanical force (DIN 51 005, ASTM E831, ASTM D696, ASTM D3386, ISO 11359 – Parts 1 to 3). It is closely related to Dilatometry (vertical dilatometer), which determines the length change of samples under negligible load (e.g. DIN 51 045). The length change phenomena can be studied revealing information regarding their thermal behavior and about process parameters or curing kinetics.

## Typical DIL/TMA Applications

- Linear thermal expansion
- Coefficient of thermal expansion (CTE)
- Volumetric expansion
- Shrinkage steps
- Softening point
- Glass transition temperature
- Phase transitions
- Sintering temperature and step
- Density change
- Influence of additives and raw materials
- Anisotropic behavior
- etc.



TMA 402 **F1** Hyperion®

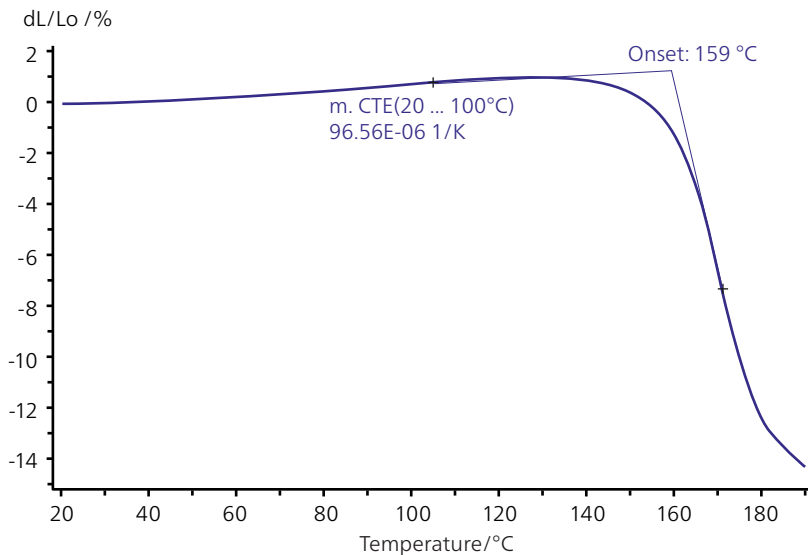
DIL 402 Expedis® Supreme

## TMA 402 F1/F3 Hyperion®

- Temperature range: -150°C to 1550°C (three interchangeable furnaces)
- Measurement of length change and corresponding force
- Vacuum-tight thermostatic measuring system
- Easily interchangeable sample holders made of fused silica or alumina
- Max. sample length 30 mm
- High resolution: 0.125 nm/digit
- Force range: 1 mN to 4 N (only for **F1**, 3 N for **F3**, **F3 Polymer Edition**)
- Modulated force (only for **F1**)

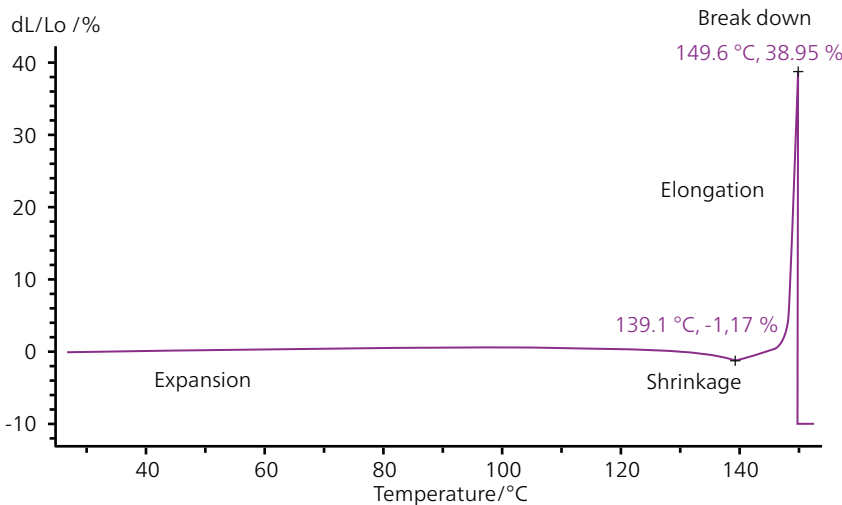
## DIL 402 Expedis Select/Supreme

- Temperature range: -180°C to 2800°C, various furnaces
- Single or double dilatometer
- Measuring range: 25 mm/50 mm
- *NanoEye*
- $\Delta l$  resolution: 1 nm/0.1 nm/digit
- Automatic sample length detection
- Controlled contact
- Force range: 0.01 N to 3 N
- Force modulation
- Sample length: 0 to 52 mm
- Vacuum-tightness:  $\approx 10^{-5}$  mbar



### Thermal Expansion of a Polymer Film

TMA measurement on a polymer film between RT and 190°C at a tensile force of 5 mN. Shrinkage starts at 159°C (extrapolated onset).



### Thermal Stability of a Separator in Tension Mode

Temperature increase may cause melting or breaking of the separator, which can result in a short circuit. In this example, the temperature increases rapidly and leads to shrinkage followed by a strong elongation of the material. This eventually causes thermal runaway.

# THERMAL STABILITY

## Designing Inherently Safer Batteries Which Meet Application Challenges

Electrolytes are characterized by high conductivity, good electrochemical stability and the ability to perform at low temperatures. However, the thermal stability of many electrolyte solutions is restricted even at moderate temperatures where side reactions can begin to limit the lifetime and performance of cells.

Thermal stability is one of several important criteria in battery design. The trick is to find the right design that meets the application criteria. For example, there is a positive correlation between the nickel content in the cathode and the battery capacity. Since nickel is relatively cheaper than cobalt, adding more nickel lowers the unit cost as well.

The presence of nickel, however, reduces the stability of the battery as it reacts easily to the external environment. The nickel content in batteries can increase to 80% or higher, and the consequent deterioration in stability must be improved to ensure safety.

The thermal stability can be investigated by means of Differential Scanning Calorimetry (DSC) and Thermogravimetry (TG or Thermogravimetric Analysis, TGA). A combination of both methods represents the Simultaneous Thermal Analyzer (STA).

### Differential Scanning Calorimetry

Specific heat capacity ( $c_p$ ) is determined by means of Laser Flash Analysis (LFA) or Differential Scanning Calorimetry (DSC).

DSC is the most employed Thermal Analysis method. It allows for the precise detection of thermal characteristics such as melting/crystallization temperatures and enthalpies, glass transition temperatures, reaction temperatures and enthalpies, cross-linking reactions (curing), and more.

The DSC method is suitable for the investigation of electrodes, electrolytes, separators, slurries, etc. For example, it can be used to investigate the energy released during a reaction between electrolyte and electrode.

### Typical DSC Applications

- Specific heat capacity
- Melting, crystallization
- Glass transition
- Oxidative stability
- Crystallinity of semi-crystalline materials
- Decomposition onset
- Solid-solid transition
- Solid-liquid ratio
- Polymorphism
- Phase diagrams
- Eutectic purity
- Compatibility

## Technical DSC Data

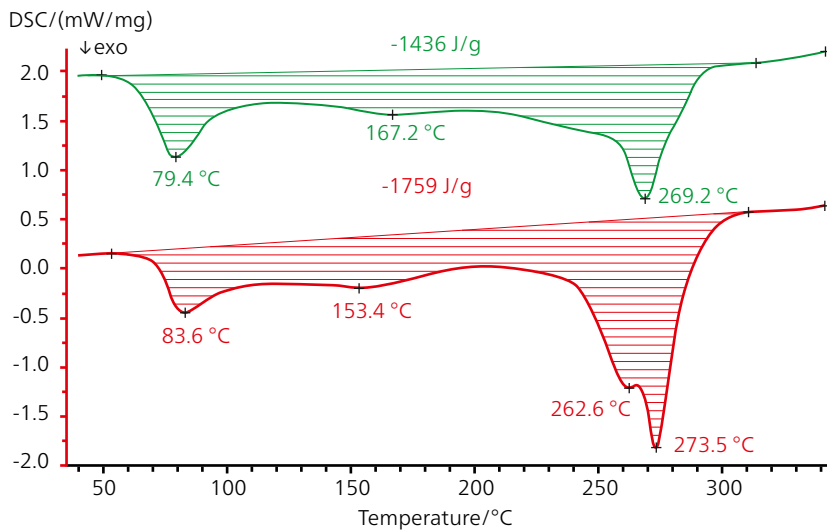
- Temperature range: depending on instrument -180°C to 2000°C
- Heating/cooling rates up to 500 K/min
- Determination of the specific heat capacity ( $c_p$ )
- Automatic sample changer (optional)
- AutoEvaluation* and *Identify* for curve evaluation and interpretation
- Temperature-modulation
- Gas- and vacuum-tight systems



DSC 300 Caliris®



DSC 500 Pegasus®



## The Reaction of a Cathode Material in two Different Electrolytes

A cathode sample was measured in two different electrolyte formulations. In both cases, the reaction between cathode material and electrolyte starts at approximately 60°C and releases a high amount of energy (exothermic reaction). The total reaction enthalpy of sample 2 amounts to -1759 J/g which is higher compared to sample 1 (-1436 J/g peak enthalpy). The exothermic peak at 273°C is higher and indicates a shoulder at 262°C which results from the different electrolyte formulation. The heat of reaction for undesired reactions occurring in the cell needs to be mitigated to make inherently safer cells.

## Evolved Gas Analysis with Simultaneous Thermal Analysis (STA)/ Thermogravimetric Analysis (TGA)

The STA 509 *Jupiter*<sup>®</sup> series combines configuration flexibility and unmatched performance in just one instrument. Pluggable interchangeable DSC and TGA sensors and various furnaces allow for precise thermo-analytical measurements. By supplementary MS coupling, even more comprehensive analyses are possible.

The NETZSCH QMS 505 *Aëolos* is a compact mass spectrometer with a heated capillary inlet system for routine analysis of gases and, in particular, volatile decomposition products. The software is unique: fully integrated MS software for measurement and analysis allows for direct correlation between the mass-loss steps and gases released. This coupling is suitable for studying the thermal degradation of electrolytes.

### STA 509 *Jupiter*<sup>®</sup> Series

- Temperature range: -150°C to 2000°C
- Furnaces: 9, incl. water-vapor, high-speed
- Sample weight, weighing range: 5 g, 35 g
- TGA resolution: 0.025 µg, 0.1 µg
- Vacuum: 10<sup>-4</sup> mbar
- Sensors: TGA, TGA-DSC, TGA-DSC-c<sub>p</sub>, TGA-DTA
- Temperature-modulation
- Automatic sample changer: 20 samples
- Glove box version

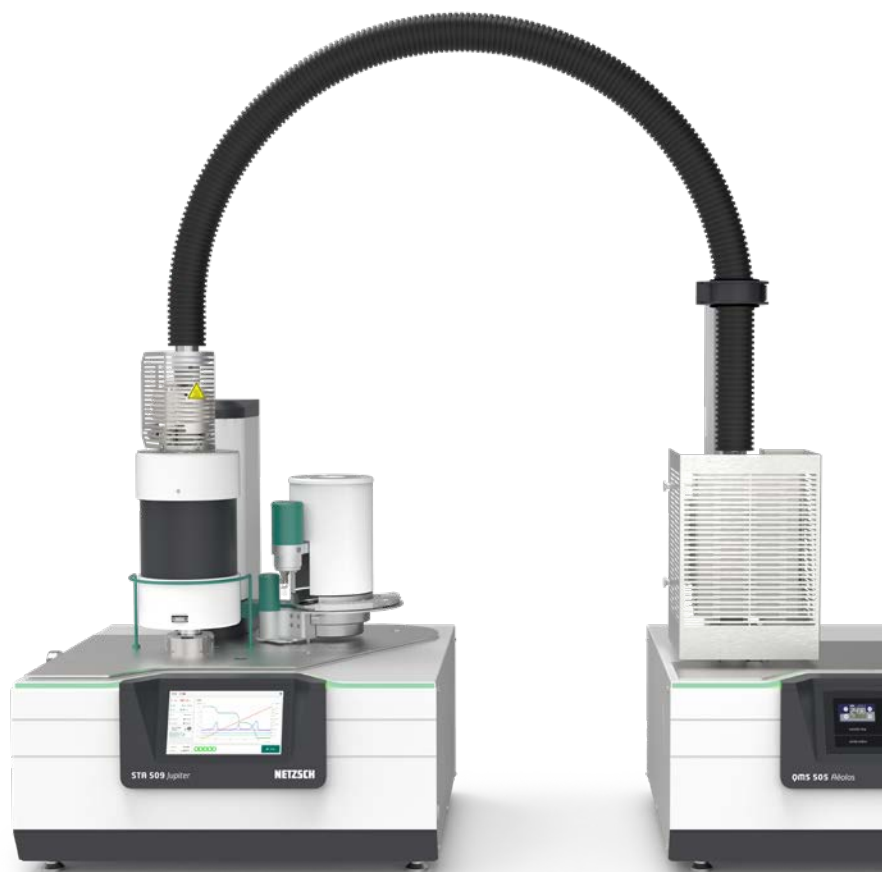
### STA 509 *Jupiter*<sup>®</sup> coupled to QMS 505 *Aëolos*

The STA systems offer fast atmosphere adjustment, especially with very pure and non-oxidizing sample atmospheres.

- Optimized gas transfer
- No dead volume
- Low dilution – high sensitivity
- Up to 300 u/512 u
- 3D presentation of results
- Detection limit: > 100 ppb (gas dependent)

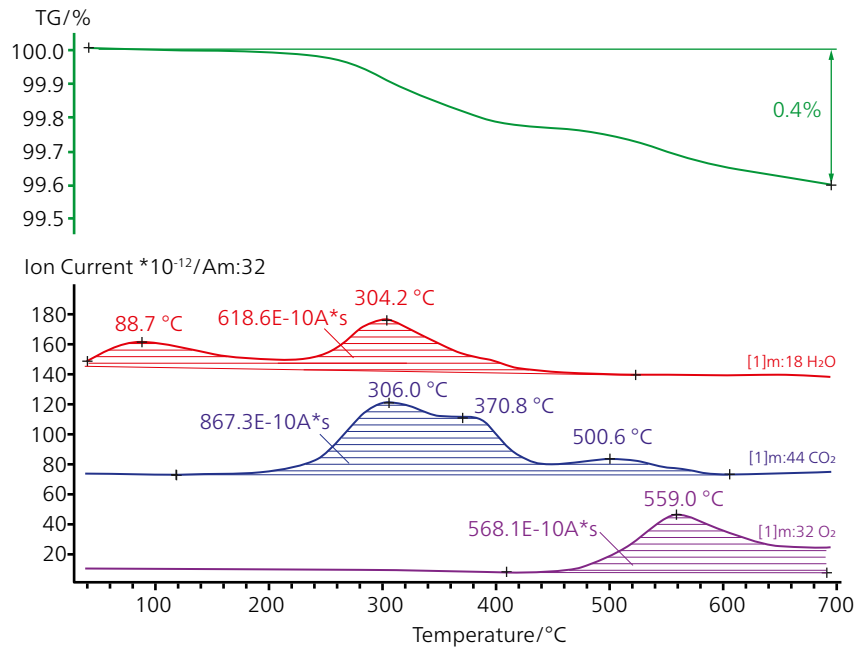
### Typical STA Applications

- Melting/crystallization behavior
- Solid-solid transitions
- Polymorphism
- Degree of crystallinity
- Glass transitions
- Oxidative stability
- Specific heat capacity
- Mass changes
- Temperature stability
- Oxidation/reduction behavior
- Decomposition
- Corrosion studies
- Kinetics Neo



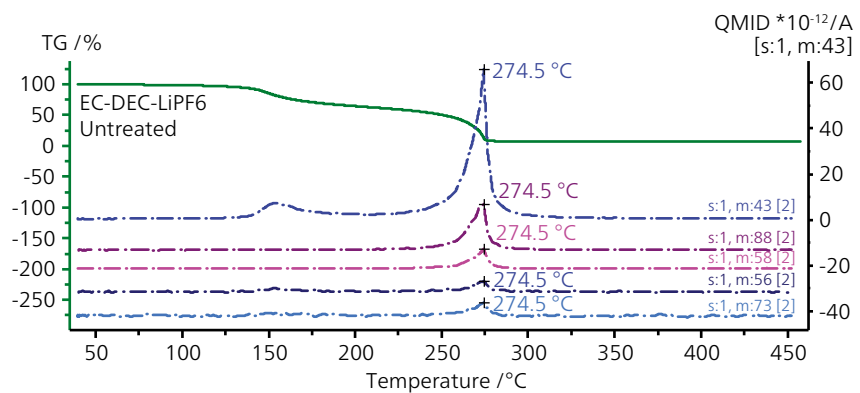
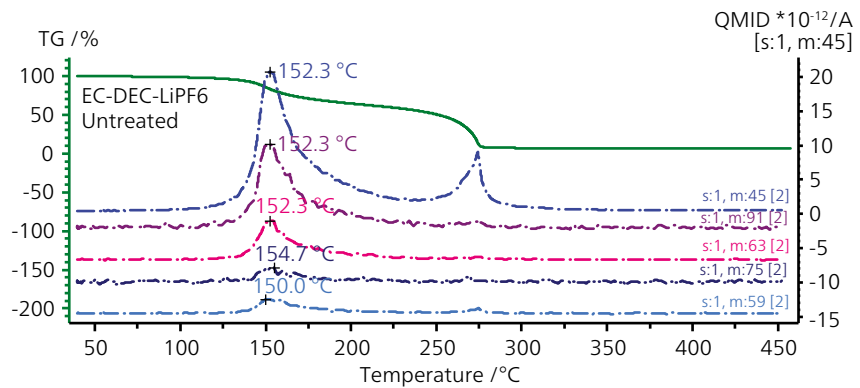
## Water Content and Detection of Released Cathode Gases

The combination of STA with QMS gives a clear insight into the water content and release of further gases ( $\text{CO}_2$ ,  $\text{O}_2$ ). Water leads to the degradation of the electrolyte during the electrochemical reaction. QMS provides the needed identification of what materials are contributing to the weight loss measured. In this example, water release begins before  $100^\circ\text{C}$  and extends to about  $420^\circ\text{C}$ .



## Thermal Stability of an Electrolyte

STA-MS measurement on 8 to 10 mg 1.0 M LiPF<sub>6</sub> in EC/DEC=50/50 (v/v). The upper plot indicates that the 1<sup>st</sup> mass-loss step is most likely due to the evaporation of DEC (45, 59, 63, 75 and 91); the lower plot shows mass numbers 43, 56, 58, 73 and 88 attributed to ethylene carbonate indicating the evaporation of EC during the 2<sup>nd</sup> mass-loss step.



STA 509 Jupiter® coupled to QMS 505 Aëolos

# PROCESSING PROPERTIES AND LEVELING CHARACTERISTICS

## How to Apply Battery Slurry onto the Electrode

A battery slurry is typically processed by blade coating or slot die coating. During these processes, the slurry is undergoing mid or high-level shear rates for a short period of time, followed by gravitational stresses acting on the slurry after the coating process. This has impact on the leveling behavior at low shear rates and film uniformity.

Rheology provides access to optimizing these flow properties of battery slurries. Different low-shear behavior can influence the leveling. With high solid content, shear thickening might occur in processing causing difficulties during the coating process. In addition to the shear viscosity function, the elasticity of the slurry under shear flow is important. It can lead to flow instabilities, such as film rupture or uneven edges of the coating layer. Elasticity of the slurry, however, helps prevent sedimentation of the particles at rest and needs to be balanced against the negative impact of elastic normal stresses during processing. Furthermore, industrial Li-ion electrode manufacturing employs e.g. the slot die coating method, which results in very high shear rates, up to  $10000 \text{ s}^{-1}$ . Therefore, shear rate analysis close to processing conditions, using high-pressure capillary rheometers, conveys further insights and better understanding of the flow behavior of battery slurries (process optimization).



Kinexus Prime ultra+

Rosand RH2000

### Critical Factors Influencing Rheology of the Slurry

- Volume fraction
- Particle size and distribution
- Particle shape
- Electrostatic interactions
- Molecular weight
- Intrinsic viscosity

### Critical Factors when Processing Battery Slurries

- Slurry stability
- Flow properties (viscosity and elasticity) during coating
- Flow instability
- Film structure/leveling behavior

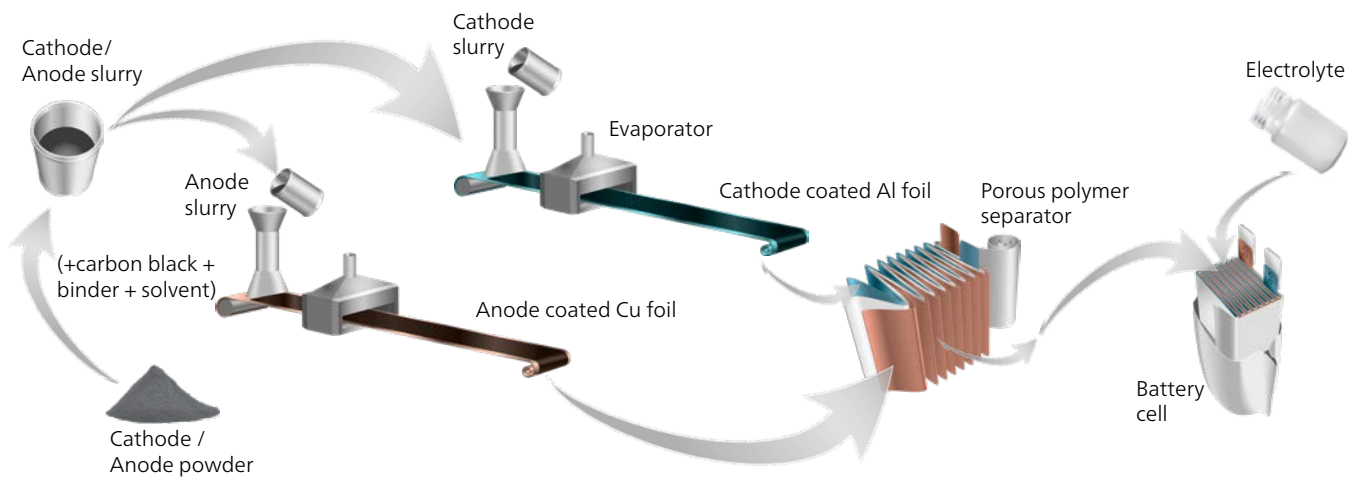
### Kinexus Prime Rotational Rheometers

- Temperature range:  $-40^{\circ}\text{C}$  to  $450^{\circ}\text{C}$
- Torque range: 0.5 nNm to 250 mNm, depending on instrument model
- Torque resolution: 0.05 nNm to 0.1 mNm, depending on instrument model
- Force range: 0.001 N to 50 N

### Rosand Capillary Rheometers

- Temperature range:
  - RT to  $400^{\circ}\text{C}$  (optionally up to  $500^{\circ}\text{C}$ )
  - $+5^{\circ}\text{C}$  to  $300^{\circ}\text{C}$  (cooling coil option)
- Force range: 12 kN, 20 kN, 50 kN, and 100 kN
- Pressure transducer range: 250 psi to 30000 psi (17 bar to 2000 bar)

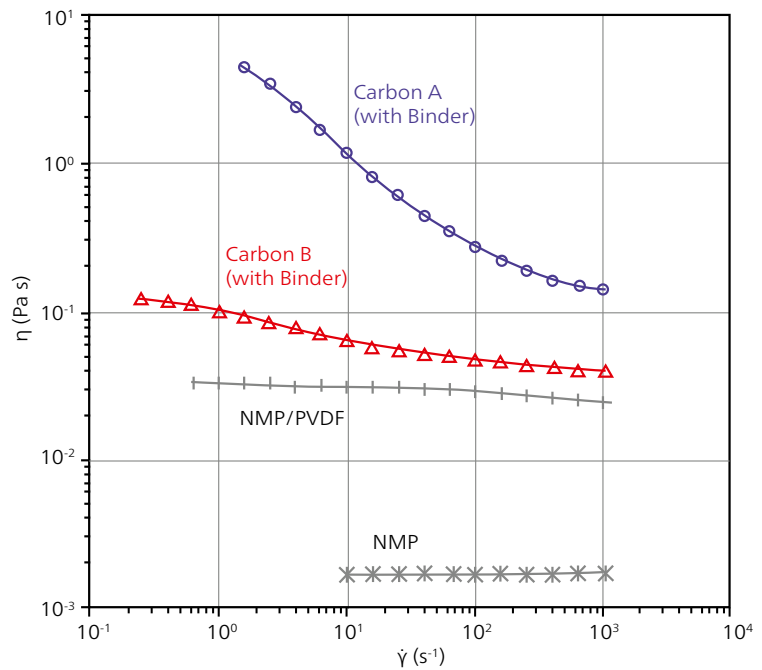




Courtesy of Malvern Panalytical B.V., the Netherlands

## Viscosity and Visco-Elasticity Control of the Coating Process

The rheometer measurement with the NETZSCH Kinexus Prime shows that the addition of PVDF to NMP drastically increases its viscosity whilst remaining largely independent of the shear rate (Newtonian behavior). Adding carbon black further increases viscosity and exhibits shear rate dependence (non-Newtonian behavior). The slurry made with Carbon A yielded a much higher viscosity than Carbon B across all shear rates, which would likely increase the resistance to sedimentation on standing (low shear process) and result in a thicker electrode film on coating (high shear process).



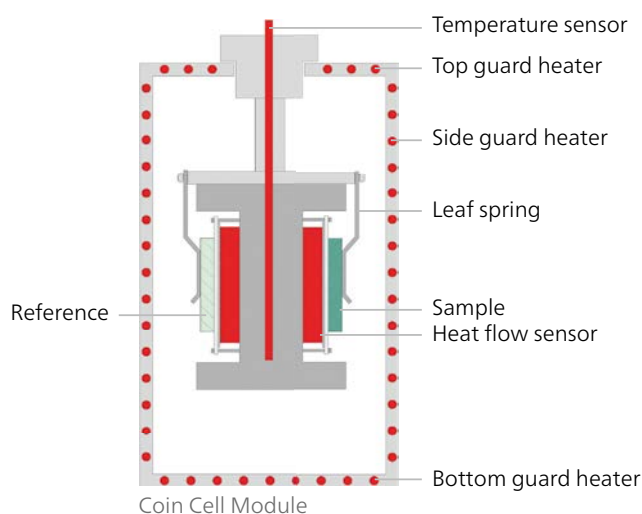
To avoid sedimentation, the slurry structure at rest needs to be dominated by elasticity. This can be investigated by oscillatory shear experiments, which characterizes the viscoelastic properties and thus enables a stability forecast for the slurry before it is coated.

The higher viscosity may also make the coating process more difficult to control, potentially leading to uneven coating and variable layer density which, in turn, results in a variable ion transfer rate and hence battery lifetime (and recharge cycle time).

# CHARACTERIZATION OF COIN CELLS

## Determination of the Heat Signature Provides Key Information for Battery Development

Understanding heat generation during charging/discharging cycles is crucial for improving the cell efficiency, performance and lifetime of batteries. Measuring the heat signature of coin cells during cycling provides insight into the underlying processes and provides a quantitative way of comparing changes in chemistry above and beyond current and voltage measurements. Some of these processes are reversible, some occur during the initial few cycles, and others take place over a period of weeks, months, or even years. The amount of heat released or absorbed during all these physicochemical changes and the rate of energy change within the coin cell provide additional pieces of the puzzle and can accelerate the development process.



### Typical MMC Applications

- Coin cell module for testing a complete coin cell, amount of heat released or absorbed during chemical changes, charging/discharging, rate of energy change, efficiency
- ARC module for Heat-Wait-Search, storage and transport studies
- Scanning module for sample screening, isothermal and constant temperature, ramp test



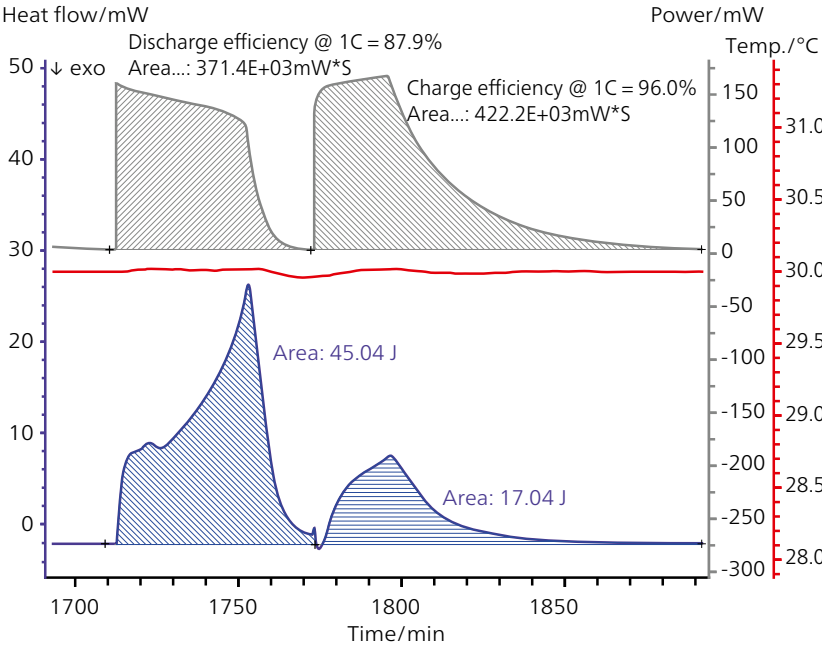
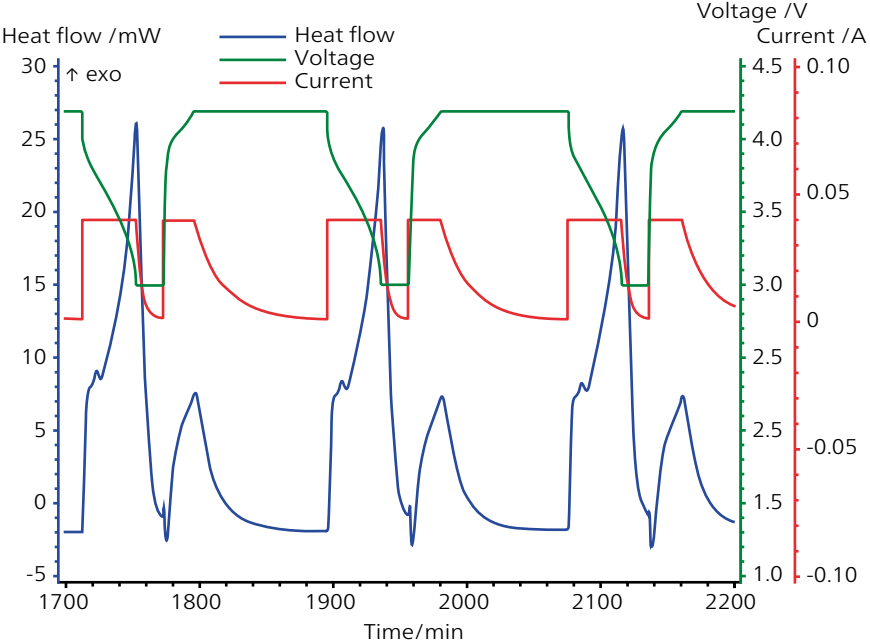
The MMC 274 Nexus® offers three interchangeable calorimeter modules

### MMC 274 Nexus®

- Module for testing coin cells (e.g., CR2032):
  - Temperature range: RT to 300°C
  - Isothermal calorimetry
  - "Large-volume DSC"
- ARC and scanning module:
  - Tracking rate of 50 K/min
  - Temperature range: RT to 500°C
  - Max. pressure: 150 bar
  - Container volume: 1 to 8.5 ml

## Efficiency Tests for Coin Cells

The key component of the coin cell module is the sensor. It features an innovative differential measurement design based on thermopiles for improved sensitivity and stability in heat-flow measurements. The calorimetric block is equipped with a Resistance Temperature Device (RTD) in order to precisely monitor and control its temperature using the surrounding furnace. When the coin cell releases or absorbs any heat due to charging/discharging or internal physical changes, the sensor detects this heat. To nullify the effect of external disturbances, the signal coming from the reference heat flow meter is subtracted from the signal detected by the sample heat flow meter, thus yielding a true differential measurement. This technique is much more accurate and sensitive than that of a single-sensor system.



## Accelerating Rate Calorimetry

Accelerating Rate Calorimeters help secure safe and cost-effective operations in industry. Adiabatic Calorimetry is the most common method for measuring self-heating and thermal runaway of chemicals and reaction mixtures. As highly versatile miniature chemical reactors, they measure thermal and pressure properties of exothermic chemical reactions. ARC measurement of cell components can help researchers understand the stability of different combination of materials. For all NETZSCH Accelerating Rate Calorimeters with internal heaters, the patented *VariPhi*<sup>®</sup> option enables measurements at constant heating rates (exo/endo). Monitoring of pressure data with the ARC systems is possible.

### ARC<sup>®</sup> 244/305

- Temperature range: RT to 500°C
- Max. pressure: 150 bar
- Max. tracking rate:
  - ARC 244 20 K/min
  - ARC 305 200 K/min
- Sample volume: 1 ml to 130 ml



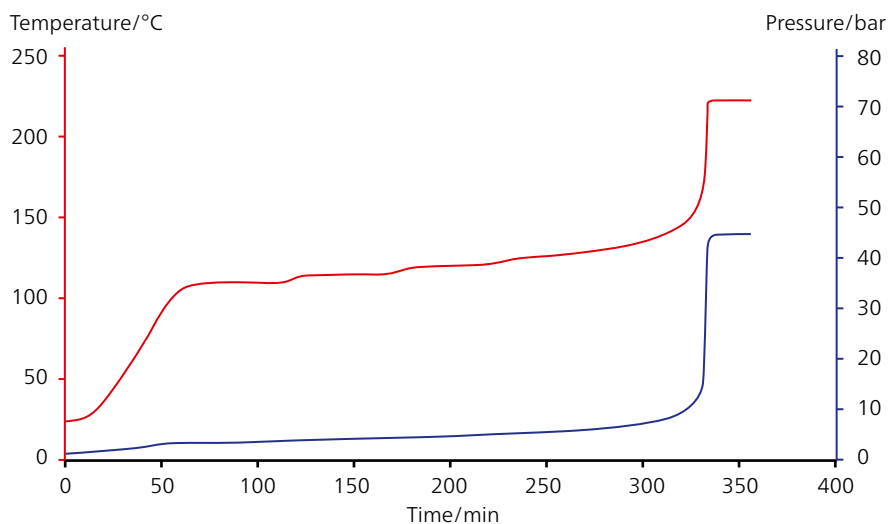
ARC<sup>®</sup> 305

## Understanding Accelerating Rate Calorimetry

Heat-Wait-Search mode (HWS) represents a careful approach to the reaction temperature:

- Heating the sample material to a certain temperature → Heat
- Isothermal stabilization → Wait
- Change to the adiabatic mode → Search (as soon the self-heating rate of the sample extends a customer-defined threshold (usually 0.02 K/min))

A thermal runaway reaction is usually investigated with the HWS mode. Once self-heating is detected by the calorimeter, the calorimeter "tracks" the cell temperature – maintaining a quasi-adiabatic environment.



High tracking rate capability, like in the ARC<sup>®</sup> 305, is required to get appropriate data. Slower calorimeters will only underestimate the dangers.

# Expertise in Service




## Proven Excellence in Lab & Services

NETZSCH Analyzing & Testing offers a broad spectrum of services for your thermal analysis instruments – from the basics to complete value retention of your instrument, including various training programs and application support.

# NETZSCH Energy Solutions

## BATTERIES

Analyzing & Testing, Pumping and Dry & Wet Processing

	Anode & Cathode Materials	Separators	Conductive Additives & Battery Slurries	Battery Cells	Electrolyte
<b>Material Characterization</b> 					
Differential Scanning Calorimetry DSC 500 <i>Pegasus</i> <sup>®</sup> DSC 300 <i>Caliris</i> <sup>®</sup> <i>Supreme/Select</i>		■			■
Simultaneous Thermal Analysis STA 509 <i>Jupiter</i> <sup>®</sup> series	■	■			■
Quadrupole Mass Spectrometer QMS 505 <i>Aëolos</i>	■	■			■
Thermogravimetry TG 309 <i>Libra</i> <sup>®</sup> <i>Select/Supreme</i>	■	■			■
Thermomechanical Analysis/Dilatometry TMA 402 <b>F1/F3</b> <i>Hyperion</i> <sup>®</sup> DIL 402 <i>Expedis</i> <sup>®</sup>		■			
Calorimetry Multiple Module Calorimeter MMC 274 <i>Nexus</i> <sup>®</sup>	■		■	■	■
Accelerating Rate Calorimetry ARC <sup>®</sup> 244/305	■		■		■
Laser/Light Flash Analysis LFA 717 <i>HyperFlash</i> <sup>®</sup>	■			■	■
Heat Flow Meter HFM 446 <i>Lambda</i>	■			■	■
Rheology Kinexus Prime Series Rosand Series			■		



Analyzing & Testing







Pumps & Systems



Grinding & Dispersing

NETZSCH offers a broad portfolio of machines and instruments accompanying the overall manufacturing process as well as research and development of Li-ion batteries. The product portfolio includes for: dry and wet grinding, mixing and homogenization, dispersion, delamination, separation, deaeration, pumping, thermal analysis and rheology.

	Anode & Cathode Materials	Separators	Conductive Additives & Battery Slurries	Battery Cells	Electrolyte
<b>Pumping</b> 					
NEMO® Progressing Cavity Pumps			■		
TORNADO® Rotary Lobe Pumps			■		
<b>Mixing &amp; Emulsifying</b> 					
OMEGA® Dispersionizer			■		
PMH/PML Planetary Mixing & Kneading Machines			■		
EPSILON Inline Dispenser			■		
PMD Intensive Mixer					
<b>Wet Grinding</b> 					
ALPHA® LAB Laboratory Mill	■	■	■		
DISCUS Grinding System	■	■			
ZETA® Grinding System	■	■	■		
NEOS Grinding System			■		
ZETA® RS Nano Mill	■	■			
<b>Dry Grinding</b> 					
CSM Classifier Mill	■				
s-JET® Steam Jet Mill	■		■		
CGS Fluidized Bed Jet Mill	■	■	■		

The owner-managed NETZSCH Group is a leading global technology company specializing in mechanical, plant and instrument engineering.

Under the management of Erich NETZSCH B.V. & Co. Holding KG, the company consists of the three business units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems, which are geared towards specific industries and products. A worldwide sales and service network has guaranteed customer proximity and competent service since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, NETZSCH has it covered. Our 60 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

## Proven Excellence. ■

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