NETZSCH
Proven Excellence.

NETZSCH Energy Solutions

Batteries – Characterization & Process Technologies

Trust NETZSCH Solutions for Battery Materials

Engineers face challenges when designing lithium ion batteries. A proper thermal management strategy is required to ensure performance and the life span of the battery. The main concern with the thermal behavior of room temperature batteries is the possible significant temperature increase that may cause thermal runaway. Battery design parameters, manufacturing process and operating conditions have an effect on temperature rise/profile during battery operation.

Manufacturers of battery components must deliver consistent overall 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.

Chemical composition, shape and particle size distribution of the active materials, homogeneity and absence of defects in the coatings on the conductor foils of the electrodes, transport and dosing of difficult, aggressive media, feeding during mixing and grinding and dosing of cathode and anode slurry influence the manufacturing process – and thus in the battery performance.

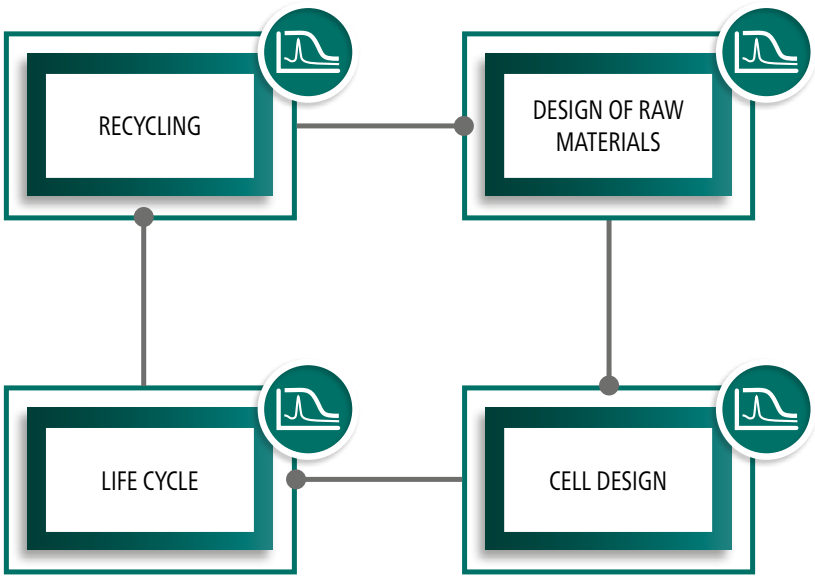
NETZSCH offers a broad portfolio of machines and equipment accompanying the overall manufacturing process as well as research and development of Li-ion batteries. The product portfolio includes for: dry and wet grinding, mixing and homogenization, dispersion, delamination, separation, deaeration, classification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

Proven Excellence.

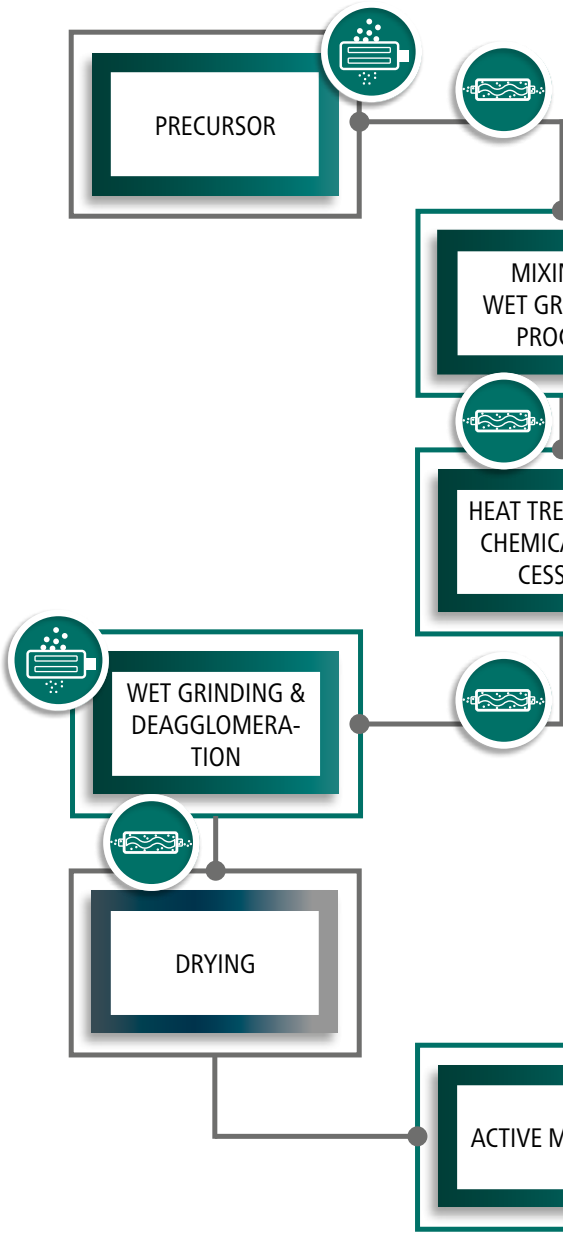
NETZSCH TECHNOLOGIES IN LI-ION

We offer Tailor-Made Solutions for Every Spe

PRODUCTION STEP
Characterization of Components Accompanying the R&D Process



PRODUCTION STEP
Raw Materials for Anode & Cathode



Business Unit
Analyzing & Testing

Business Unit
Pumps & Systems

Business Unit
Grinding & Dispersing

BATTERIES PRODUCTION PROCESS

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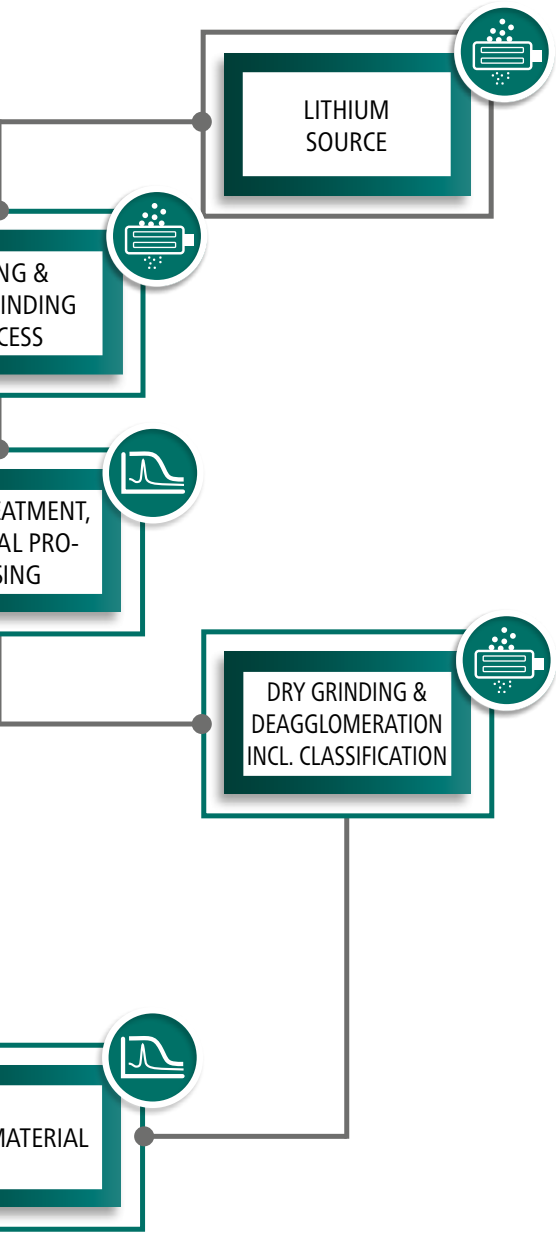
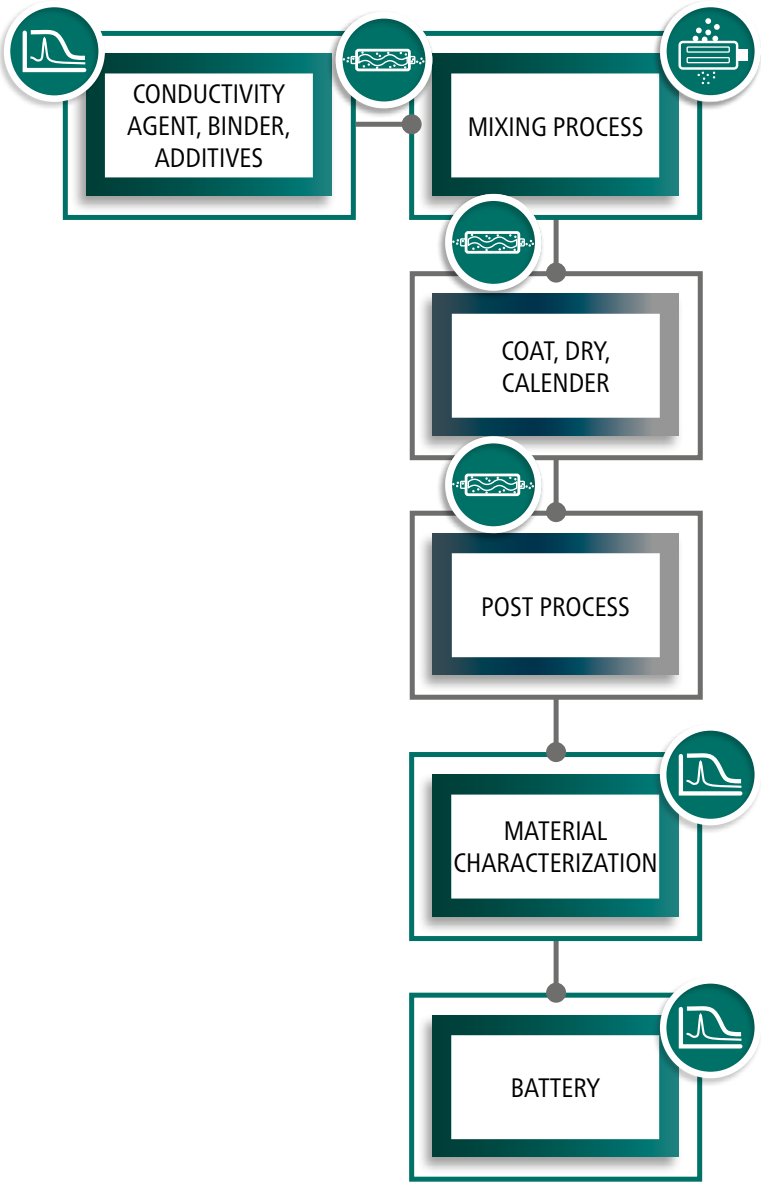


PRODUCTION STEP

Conductive Additives & Battery Slurry

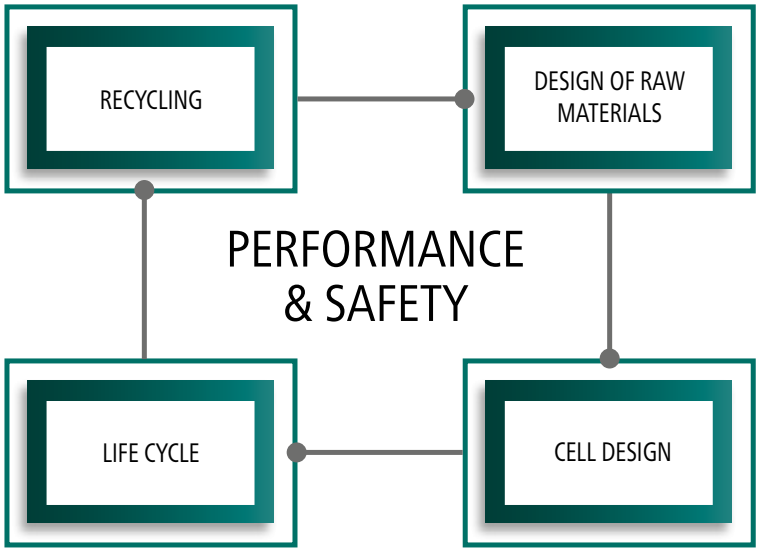


CHARACTER-IZATION OF COMPONENTS

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 new levels of insight and control needed to power the development and production of superior-quality batteries.

Manufacturers of battery components must not only deliver consistent overall quality, but also throughout the entire manufacturing process. Performance and safety aspects play a significant role and influence the selection of raw materials, the cell design, the life cycle and subsequent recycling.

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.



THERMAL ANALYSIS & RHEOLOGY OF BATTERIES

Battery Materials/ Components	Critical Parameters	Analytical Techniques	Product Solutions
Cathode precursor & electrode materials	Crystal phase	Simultaneous Thermal Analysis coupled to Mass Spectrometer	STA 509 *Jupiter*® series coupled to QMS 505 *Aëolos*
Battery slurry	▪ Viscosity ▪ Viscoelasticity ▪ Stability	▪ Rotational Rheometry ▪ Capillary Rheometry	Kinexus Prime Series Rosand Series
Electrode and electrode coating	▪ Thermal management (thermal diffusivity/ conductivity) ▪ Specific heat capacity ▪ Thermal stability ▪ Reactivity ▪ Performance changes ▪ SEI Formation	▪ Laser/Light Flash Analysis ▪ Differential Scanning Calorimetry ▪ Isothermal Calorimetry ▪ Accelerating Rate Calorimetry	▪ LFA 717 *HyperFlash*® ▪ DSC 500 *Pegasus*® ▪ MMC 274 *Nexus*® ▪ ARC® 244/305
Electrolyte	▪ Thermal stability ▪ Degradation with air/moisture ▪ Performance changes	▪ Simultaneous Thermal Analysis coupled to Evolved Gas Analysis (e.g., QMS) or only TGA ▪ Differential Scanning Calorimetry ▪ Isothermal Calorimetry ▪ Accelerating Rate Calorimetry	▪ STA 509 *Jupiter*® series coupled to QMS 505 *Aëolos* ▪ TG 309 *Libra*® *Select/ Supreme* ▪ DSC 500 *Pegasus*® ▪ MMC 274 *Nexus*® ▪ ARC® 244/305
Binder, additives	▪ Thermal behavior ▪ Viscosity ▪ Viscoelasticity	▪ Simultaneous Thermal Analysis ▪ Rotational Rheometry	▪ STA 509 *Jupiter*® series ▪ Kinexus Prime series ▪ Rosand series
Separator	▪ Specific heat capacity ▪ Dimensional change ▪ QA/QC ▪ Safety effectiveness	▪ Differential Scanning Calorimetry ▪ Thermomechanical Analysis, Dilatometry ▪ Isothermal Calorimetry	▪ DSC 500 *Pegasus*® ▪ TMA 402 ***F1/F3*** *Hyperion*® ▪ DIL 402 *Expedis*® *Supreme* ▪ MMC 274 *Nexus*®
Battery cell	▪ Efficiency of cells ▪ Thermal stability ▪ Thermal runaway ▪ Internal short tests ▪ Enthalpies ▪ Thermal conductivity ▪ Gas production (quantitative and qualitative) ▪ Vent design ▪ Safety – CID, PTC ▪ Kinetic model development ▪ Isotropic & parasitic reactions	▪ Vent design ▪ EGA coupled to Thermal Analysis ▪ Isothermal Calorimetry	▪ MMC 274 *Nexus*® ▪ LFA 717 *HyperFlash*®/ HFM 446 *Lambda Medium* ▪ STA 509 *Jupiter*® series coupled to QMS 505 *Aëolos*/GC-MS/FT-IR ▪ PERSEUS STA 509 *Jupiter*® coupled to GC-MS/MS ▪ Kinetics NEO
Material testing	Compatibility between and among the different cell parts	Thermal Analysis in general	

ANODE & CATHODE MATERIALS

One option for the synthesis of cathode and anode materials is the so-called solid-state process. In this process route, the active material is created from the raw materials through a chemical transformation in suitable furnaces. Depending on the raw materials used, a wet grinding stage with dry pre-grinding processes can be required for the raw materials prior to the synthesis.

The CGS and *s-Jet*® fluidized bed jet mills can be used for the dry grinding. For the wet grinding, the *Zeta*®, *Neos* or *Zeta*® *RS* agitator bead mills are used, depending on the required target fineness. Synthesis via a furnace process can result in the formation of undesired agglomerates. In order to separate these, a dry dispersion with a CSM classifier mill or a CGS fluidized bed jet mill is usually carried out after the synthesis furnace, without change of the original size and shape of primary particles.

Typical compounds for cathode materials are:

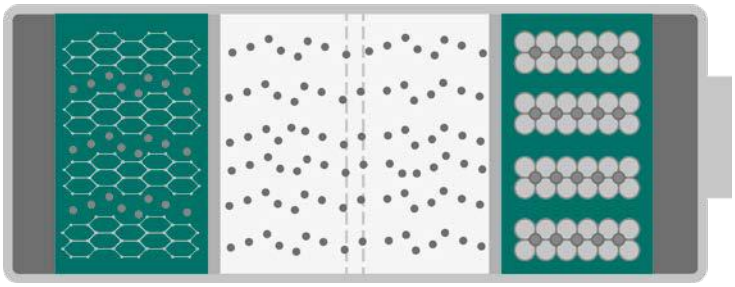
- LCO (Lithium Cobalt Oxide, $LiCoO_2$)
- NCA (Lithium Nickel Cobalt Aluminum Oxide, $LiNiCoAlO_2$)
- NCM (Lithium Nickel Manganese Cobalt Oxide, $Li[NiCoMn]O_2$)
- LMO (Lithium Manganese Oxide, $LiMn_2O_4$)
- LFP (Lithium Iron Phosphate, $LiFePO_4$)

The conductor foil is usually made of aluminum.

Some examples of active materials for anodes are:

- Amorphous Carbon
- Graphite
- Lithium Titanate (LTO, $Li_4Ti_5O_{12}$)
- Metallic Anode Materials (Silicon, Tin)

The conductor foil is usually made of copper.



APPLICATION TASKS,

which we have successfully mastered

DRY PROCESSING

Product	Technology	Machine	Working capacity [kg_{solid}/h]	Fineness [µm]
Lithium Iron Phosphate (LFP)	Fluidized Bed Jet Mill	CGS 100	500	d_{50} = 2.4 µm
Silicon powder (Si)	Fluidized Bed Jet Mill	CGS 16	up to 5	d_{50} = 2 - 7 µm
Lithiumhydroxid (LiOH)	Fluidized Bed Jet Mill	CGS 71	400 - 600	d_{99} = 50 - 80 µm
Lithium Carbonate (Li_2CO_3)	Fluidized Bed Jet Mill	CGS 100	1 500 - 1 700	d_{50} = 4 - 5 µm
Lithium Nickel Cobalt Mangan Oxide (NCM)	Fluidized Bed Jet Mill	CGS 71	800 - 1 000	d_{50} = 10.5 µm
Lithium Nickel Cobalt Aluminum Oxide (NCA)	Fluidized Bed Jet Mill	CGS 71	1 000	d_{50} = 12 µm
Lithium Cobalt Oxide (LCO)	Classifier Mill	CSM 165	400	d_{50} = 11 - 12 µm
Lithium Nickel Cobalt Aluminum Oxide (NCA)	Classifier Mill	CSM 260	300 - 350	d_{50} = 11 µm
Lithium Nickel Cobalt Mangan Oxide (NCM)	Classifier Mill	CSM 260	500 - 600	d_{50} = 11 - 12 µm
Lithium Nickel Cobalt Mangan Oxide (NCM)	Classifier Mill	CSM 360	1 000	d_{50} = 11.5 µm
NCM single cristal	Fluidized Bed Jet Mill	CGS 71	650	d_{50} = 6 µm

Working capacity depends on required fineness and product characteristics.

WET PROCESSING

Product	Technology	Machine	Working capacity [kg_{solid}/h]	Fineness [µm]
Lithium Manganese Oxide (LMO)	Agitator Bead Mill	*ZETA*® 25	110	d_{50} = 0.15
Lithium Iron Phosphate (LFP)	Agitator Bead Mill	*NEOS* 150	200 - 300	d_{50} < 0.2 - 0.4
Lithium Titanate (LTO)	Agitator Bead Mill	*ZETA*® 25	20	d_{50} = 0.10
Metal Silicon	Agitator Bead Mill + Nanomill	*ZETA*® 25 + *ZETA*® *RS* 60	3 - 4.5	d_{50} = 0.09
Multi-Walled Carbon NanoTube	Agitator Bead Mill	*ZETA*® 10	3 - 10	d_{50} ≤ 5 - 10

Working capacity depends on required fineness and product characteristics.

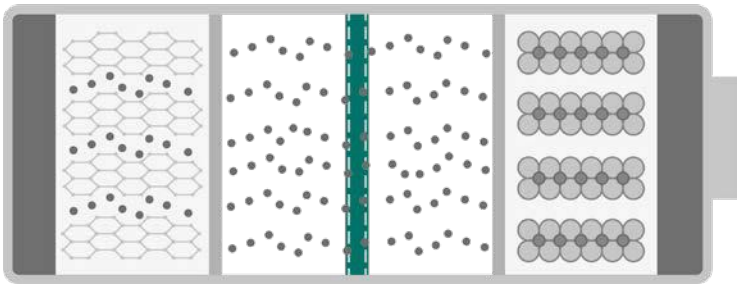
SEPARATORS

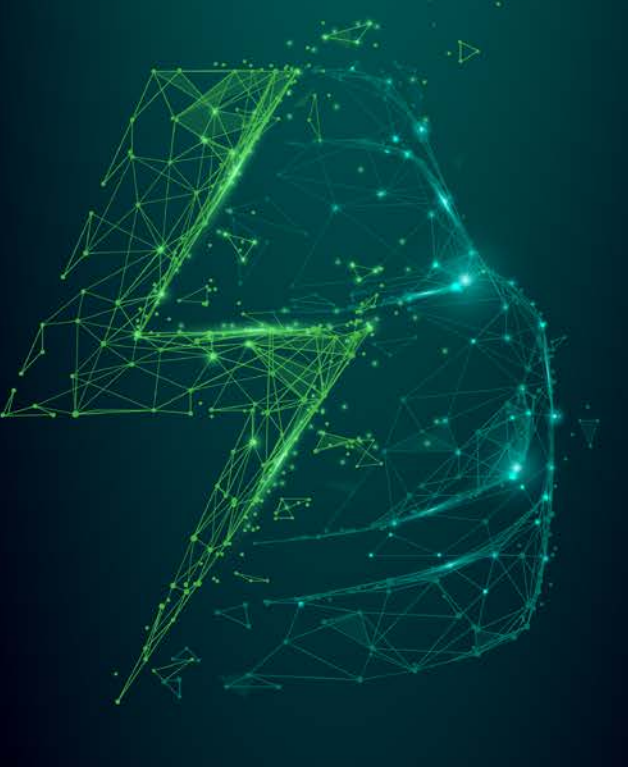
Wet operated agitator bead mills are the most suitable technology to grind abrasive ceramic materials down to the required submicron particle size range.
Different grinding systems are used depending on the required material hardness and the associated required energy density and particle size.
To guarantee a total avoidance of metal contamination all machine parts with contact to the material can be made from ceramic.

In order to prevent an internal short-circuit in the cells, a separator is inserted between the electrodes. The separators, which are typically made of polymer (e.g. PE, PP), are perforated to allow the ions to pass through.

For increased thermal stability and the prevention of shrinkage, so-called inorganic composite separators (ceramic-coated foils) are more and more used.

The ceramic suspensions used here typically have particle sizes in the submicron range. Wet mills, such as the *Discus, Zeta®*, *Neos* or the *Zeta® RS* are used for the production of these coating systems.





APPLICATION TASKS,
which we have successfully mastered

WET PROCESSING

Product	Technology	Machine	Working capacity [kg_{slurry}/h]	Fineness [µm]
Alumina (Al_2O_3)	Agitator Bead Mill	*ZETA*® 10	50	$d_{95} = 1.0$
Alumina (Al_2O_3)	Agitator Bead Mill	*DISCUS* 20	580	$d_{50} \leq 1.0$

Working capacity depends on required fineness and product characteristics.

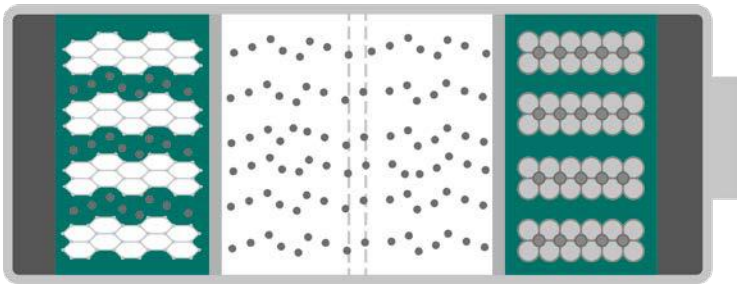
CONDUCTIVE ADDITIVES & BATTERY SLURRIES

For the production of high quality battery materials and components like active materials, additives, cathode or anode slurries and ceramic suspensions for inorganic composite separators a lot of different process steps and equipment are required.

Conductive Additives

The majority of active materials are in the electrode with limited electrical conductivity additionally the polymer matrix in the electrode needs to be electrical conductive. That is why additives are used also to reduce the required charging time of the battery when beeing added to the battery slurries. Here it is primarily the carbon-based raw materials that are used, such as carbon black, graphite and carbon nano tubes (CNTs), which should be characterized by the highest possible aspect ratio.

The materials must first be carefully dispersed or delaminated in preliminary stages. Here the size of the primary particles, the properties of the bulk solids and the purity requirements usually present particular challenges, which NETZSCH solves with the *Epsilon*, *Omega*®, *s-Jet*® and the grinding systems *Zeta*® and *Discus*.



Battery Slurries

PMH / PML planetary mixers have established themselves as state of the art for the production of so-called battery slurries. With PMH / PML planetary mixers a combination of different processes like dissolution of binders, the premixing and the alloying of the dry components, the mixing, kneading and homogenization as well as the degassing of the high viscose pasty slurries can be realized with an excellent temperature control.

However, depending on the material system used, the binder can also be dissolved first and very gently mixed after addition of the active materials. To prevent defects in the coating due to gas inclusions, the mixing process is typically carried out under a vacuum. This way, pockets of air that have entered via the raw materials can be removed from the product.

The key aspects for selection of the PMH / PML planetary mixer are the excellent temperature control and the broad viscosity range for which this machine is suited. Feeding the planetary mixer and pumping the mixed fluids to the storage tank is handled reliably and effectively by our positive displacement pumps.

APPLICATION TASKS,

which we have successfully mastered

WET PROCESSING

Product	Technology	Machine	Working capacity [kg_{slurry}/h]	Process
MWCNTs in NMP	Economic Dispersionizer	*Omega*® 500	50	Pass operation
SWCNTs in Water	Economic Dispersionizer	*Omega*® 500	15	Re-circulation operation
SWCNTs in Water	Economic Dispersionizer	*Omega*® 500	25	Re-circulation operation
SWCNTs in NMP	Economic Dispersionizer	*Omega*® 500	25	Re-circulation operation
Acetylene black in Water	Economic Dispersionizer	*Omega*® 500	90	Pass operation
Acetylene black in NMP	Economic Disperionizer	*Omega*® 500	90	Pass operation

Working capacity depends on required fineness and product characteristics.

PUMPING

Pump Type	Process	
NEMO® Progressing Cavity Pumps	▪ Vacuum defoaming ▪ Feeding of stirring and storage tanks	▪ Coating ▪ Mixing und wet grinding
TORNADO® Rotary Lobe Pumps	▪ Feeding of stirring and storage tanks	▪ Mixing und wet grinding
PERIPRO® Peristaltic Pumps	▪ Vacuum defoaming ▪ Mixing and wet grinding	▪ Feeding of stirring storage tanks ▪ Adding binders and adhesive

BATTERY MATERIALS

Applicable Instruments, Pumps & Machines

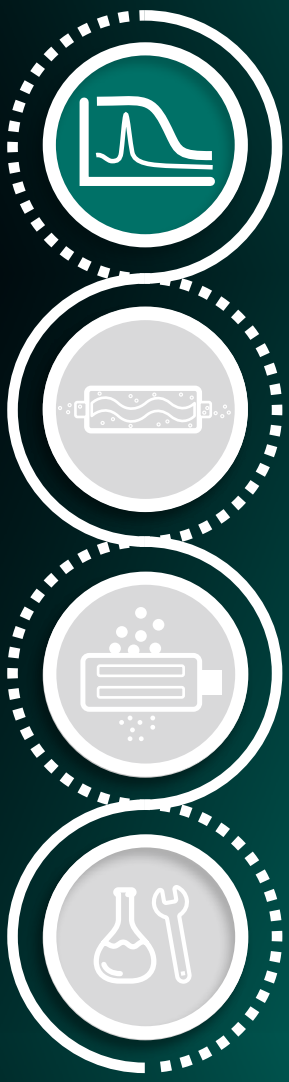
	Anode & Cathode Materials	Separators	Conductive Additives & Battery Slurries	Battery Cells	Electrolyte
Material Characterization					
Differential Scanning Calorimetry DSC 500 *Pegasus®* DSC 300 *Caliris® Select/Supreme*		■			■
Simultaneous Thermal Analysis STA 509 *Jupiter®* series Quadrupole Mass Spectrometer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

	Anode & Cathode Materials	Separators	Conductive Additives & Battery Slurries	Battery Cells	Electrolyte
Pumping					
NEMO® Progressing Cavity Pumps	■		■		■
TORNADO® Rotary Lobe Pumps	■		■		
PERIPRO® Peristaltic Pumps	■		■		
Mixing & Emulsifying					
Omega® Dispersionizer			■		
PMH/PML Planetary Mixing & Kneading Machines			■		
Epsilon Inline Disperser			■		
PMD Intensive Mixer			■		
Wet Grinding					
LabStar Laboratory Mill	■	■	■		
Discus Grinding System	■	■			
Zeta® Grinding System	■	■	■		
Neos Grinding System			■		
Zeta® *RS* Nano Mill	■	■			
Dry Grinding					
CSM Classifier Mill	■				
CGS Fluidized Bed Jet Mill	■		■		
GyRho Rounding Unit	■				
CFS/HD-S High-efficiency Fine Classifier	■				

PROVEN SOLUTIONS

in Characterization of Components

Proper battery thermal management ensures longer lifespan by keeping the cells within a limited temperature range during storage, operation and charging. 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.



Thermal Diffusivity and Thermal Conductivity by Laser Flash Analysis

The Laser/Light Flash technique has proven itself a fast, versatile and absolute method for measurement of the thermal diffusivity. A reliable thermal conductivity of the battery is significant for the accurate prediction of battery thermal characteristics during the charging/discharging process. During use (or abnormal use), considerable heat is generated inside the battery. Dissipating these heat as soon as possible is the key to ensure safety.

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 Ø 10 mm)
- Special sample holders: liquids, pastes and powder, fibers, etc.
- Various calculation models, corrections and mathematical operations
- Model wizard

THERMAL MANAGEMENT

Controlling Battery Thermal Characteristics – Separator, Electrode and Electrode Coating

Thermomechanical Analysis and Dilatometry

Each time a material is exposed to temperature changes – it shows a variation in its dimension. Whether it is in the course of its regular thermal expansion or by passing a phase transition, the substance will either be shrunk or elongated. 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. This is achieved by Thermomechanical Analysis (TMA) and Dilatometry (DIL), expressed by the coefficient of thermal expansion (CTE).

- 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

TMA 402 ***F1*** *Hyperion®*

DIL 402 *Expedis® Supreme*

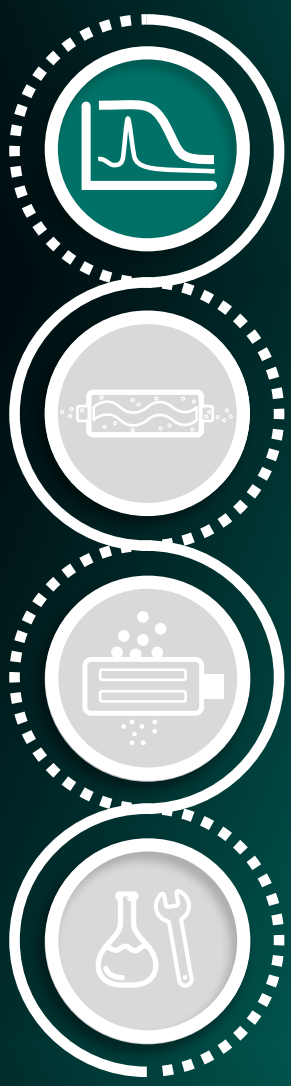
- Temperature range: -150°C to 1550°C (three interchangeable furnaces)
- Measurement of length change and corresponding force
- Vacuum-tight thermostatic measuring system
- Easily interchangeable sample holders made of fused silica or alumina
- Max. sample length 30 mm
- High resolution: 0.125 nm/digit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

- Temperature range: -180°C to 2800°C, various furnaces
- Single or double dilatometer
- Measuring range: 25 mm/50 mm
- *NanoEye*
- Δl 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 mm to 52 mm
- Vacuum-tightness: $\approx 10^{-5}$ mbar

PROVEN SOLUTIONS

in Characterization of Components

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. Due to overcharging, batteries can overheat to the point that they catch fire. Besides various metals (e.g., Co, Al, Mg, etc.), the cathode material of Li-ion batteries contains nickel. There is a positive correlation between the nickel content and the battery capacity. However, nickel reduces the stability as it reacts easily to the external environment.



Differential Scanning Calorimetry

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. There is a correlation between the energy release and the increase in cell temperature. The heat of reaction can be one of the factors that can play a role in battery formulation.

- 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

DSC 300 *Caliris® Select/Supreme*

DSC 500 *Pegasus®*

- Temperature range: depending on instrument -180°C to 1750°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
- Choice of sensor-furnace modules or furnaces

THERMAL STABILITY

Safety is Ensured when Deterioration of Stability is Avoided

Evolved Gas Analysis with Simultaneous Thermal Analysis (STA)/Thermogravimetry (TGA)

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

- DSC Results (see left page)
- Temperature stability
- Oxidation/reduction behavior
- Decomposition
- Corrosion studies
- *Thermokinetics*

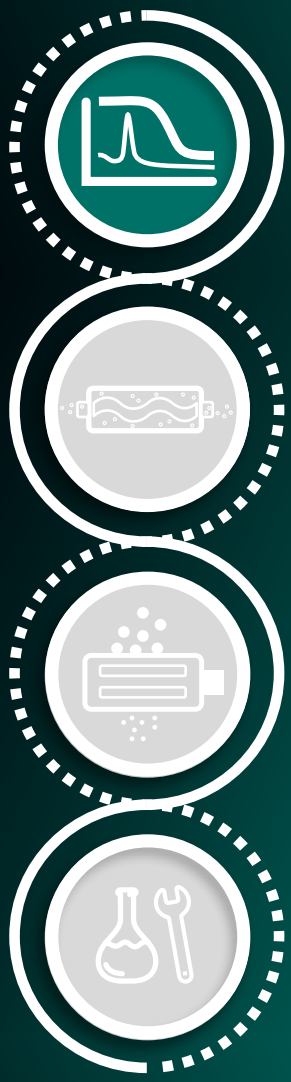
- Temperature range: -150°C to 2400°C
- Furnaces: 10, incl. water-vapor, high-speed
- Sample weight, weighing range: 5 g, 35 g
- TGA resolution: 0.025 µg, 0.1 µg
- Vacuum: up to 10^{-4} mbar
- Sensors: TGA, TGA-DSC, TGA-DSC-c_p, TGA-DTA
- Temperature-modulation
- Automatic sample changer: 20 samples
- Glove box version
- 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)

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

PROVEN SOLUTIONS

in Characterization of Components

Specifically, understanding of the 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. 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.



Multiple Mode Calorimeter

The MMC 274 *Nexus*® offers three interchangeable calorimeter modules:

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

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: 100 bar
  - Container volume: 0.1 to 8.5 ml

CHARACTERIZATION OF COIN CELLS AS A WHOLE

Heat Signature as Key Information for Battery Development & Testing

Accelerating Rate Calorimetry

Accelerating Rate Calorimeters help secure safe and cost-effective operations in industry. As highly versatile miniature chemical reactors, they measure thermal and pressure properties of exothermic chemical reactions. The resulting information helps engineers and scientists to identify potential hazards and address key elements of process safety design including emergency relief systems, process optimization, and thermal stability. For all NETZSCH Accelerating Rate Calorimeters with internal heaters, the patented *VariPhi*® option enables measurements at constant heating rates (exo/endo). Monitoring of pressure data with the ARC systems is possible.

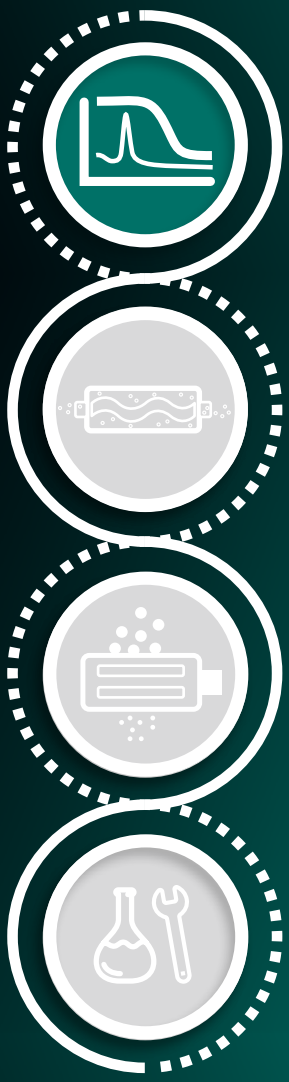
ARC® 305

- 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

PROVEN SOLUTIONS

in Characterization of Components

A battery slurry is typically processed by blade coating or slot die coating. During these processes, the slurry is undergoing mid-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.



Kinexus Prime Series

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 10 000 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

Kinexus Prime Rotational Rheometers

- Temperature Range: -40°C to 450°C
- Torque Range: 0.5 nNm to 250 mNm, depending on instrument version
- Torque resolution: 0.05 nNm to 0.1 mNm, depending on instrument version
- Force Range: 0.001 N to 50 N

Rosand Capillary Rheometers

- Temperature range: RT - 400°C (Optionally up to 500°C) +5°C to 300°C (Cooling coil option)
- Force range: 12kN, 20kN, 50kN, and 100 kN
- Pressure transducer ragen: 250 psi - 30000 psi (17 bar - 2000 bar)

PROCESSING PROPERTIES & LEVELING CHARACTERISTICS

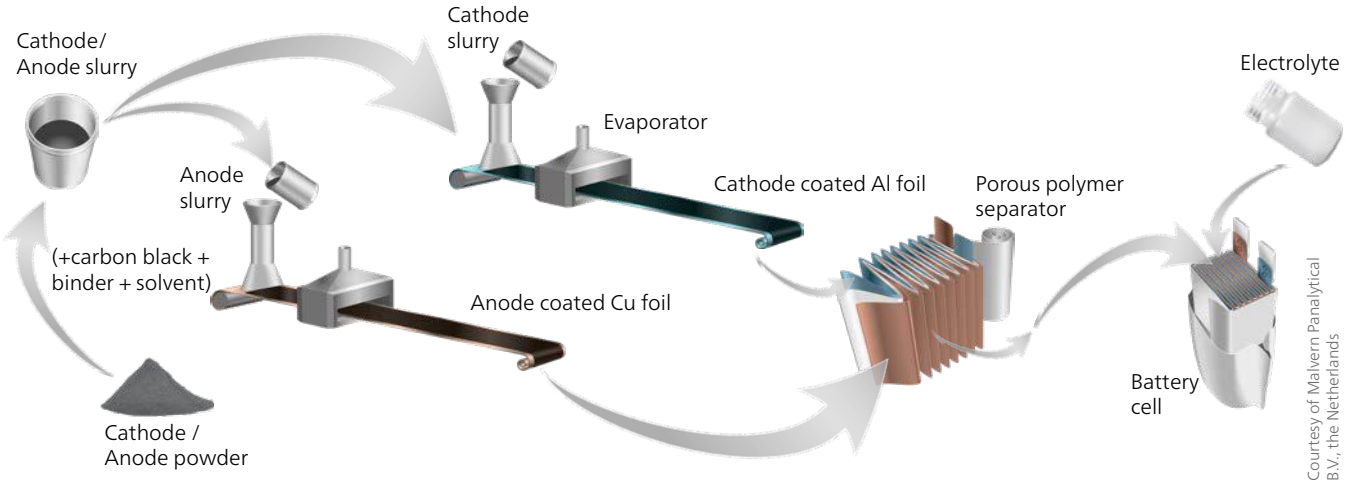
How to Apply Battery Slurry onto the Electrode

Critical Factors Influencing Rheology of the Slurry:

- Volume fraction
- Particle size and distribution
- 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



Courtesy of Malvern Panalytical B.V., the Netherlands

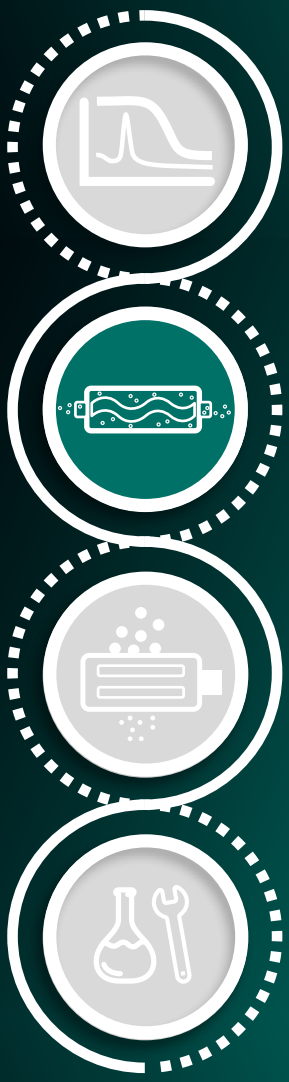
PROVEN SOLUTIONS

in Pumping Technologies

Aggressive and abrasive media such as lithium are pumped safely and gently by our positive displacement pumps. These pumps are used in almost all industry sectors for transporting and dosing difficult media, such as solids-bearing fluids having low to very high viscosities or even aggressive media which are used in battery production.

NETZSCH Pumps & Systems has consistently aligned its product portfolio to support operators with exactly the right pump technology. NETZSCH offers three types of positive displacement pumps for battery production: TORNADO® rotary lobe pumps, NEMO® progressing cavity pumps and PERIPRO® hose pumps. Depending on the application, each technology offers specific advantages from which users can benefit. These pumps are available in the industrial and in the hygienic version.

We are a reliable partner for our customers during the whole life cycle of every NETZSCH product. Through this NETZSCH ensures fast and professional help for technical problems on site worldwide. We will always find a solution, even for unusual challenges. That is helping you to save costs and time.



TORNADO® Rotary Lobe Pumps

- Flow rates from just a few ml/h up to 1000 m^3/h
- Pressures up to 8/10 bar
- In industrial or hygienic version available, with polished or non polished surfaces
- Temperatures up to 100 °C
- Tooth belt drive, without any use of oil

Your Benefits of Displacement Pumps:

- Self priming
- Can be serviced without disconnecting pipework
- For nearly every media suitable: with or without solids, low to high viscosity, thixotropic and dilatant, shear, sensitive, abrasive, non lubricating and lubricating, aggressive, adhesive, toxic

TORNADO® rotary lobe pump in stainless steel

In production processes like feeding during mixing and grinding or dosing of anode and cathode slurry NETZSCH pumps are very helpful and reliable companions.

These pumps are in two different versions available: the industrial version with metal and elastomer and the hygienic version with stainless steel. The surfaces of the hygienic version, which are in contact with the medium are polished to prevent product adhesion and facilitate

PERIPRO® Peristaltic Pumps

- Flow rates from 200 l/h to 17,000 l/h, by the use of a double-head pump it can be expanded to up to 34,000 l/h
- For pressures up to 10 bar
- Temperatures up to 80 °C
- Tolerance of dry running

PERIPRO® Peristaltic Pumps in the industrial version and in the hygienic version

NEMO® Progressing Cavity Pumps

- Flow rates from just a few ml/h up to 1000 m^3/h
- Number of stages ranging from 1 to 8 for pressures from 6 to 48 bar (standard)
- In industrial or hygienic version available, with polished or non polished surfaces
- Temperatures form 20 °C to 200 °C
- Hardly any pulsation

NEMO® MY Magnetically Coupled Pumps

- Completely hermetically sealed pump
- Specifically designed for slurries with viscosities of up to 20,000 cps
- Automated cleaning processes possible

NEMO® MY magnetically coupled pump

NEMO® progressing cavity pump

PROVEN SOLUTIONS

in Wet Grinding Technologies

The bundling of process-related know-how and the extensive machine program from laboratory to production machines to complete production lines is our strength. The wetting and homogenization of solids in liquids, deagglomeration and dispersion, all the way to true comminution of primary particles make the agitator bead mill a universal wet processing machine.

Nano Mill *ZETA® RS*

The highly efficient centrifugal separation system enables the use of smallest grinding media from a diameter of 30 µm - 300 µm in reliable continuous operation.

It is specially designed for wet grinding and dispersing processes especially "gentle dispersing" in the nanometer range. Working with the machines is a pleasure due to its easy operation.

Nano Mill *ZETA®* RS 60

NARROW PARTICLE SIZE DISTRIBUTION

Effective Process for Battery Materials

Machine frame

On the standard machine frame *Alpha* different grinding systems can be mounted, depending on the product to be processed. Whereas the disk grinding system *Discus*® is mainly used in single and multi-pass operation, for the peg grinding systems *Zeta*® or *Neos*® recirculation mode operation is preferred. Because of the system-wide standardization a high level of spare parts availability can be guaranteed. The clear and straightforward design with integrated hoses and pipes also results in simplified operation, prevents contamination and, at the same time, means considerably less effort is required for cleaning.

Grinding System *Discus*

Activation of the grinding media with high intensity through the entire grinding chamber by the disk agitator shaft. Use of grinding media from approx. 0.5 mm to 5.0 mm.

Grinding System *Zeta*®

The closed horizontal agitator bead mill is designed for the highest product throughput rates and has a peg grinding system with extremely high grinding intensity.

Suitable for a wide variety of every viscosity and almost any product, with this technology you will achieve the highest product qualities and finenesses into the nanometer range using a wide variety of grinding media from 0.3 mm to 3 mm in diameter.

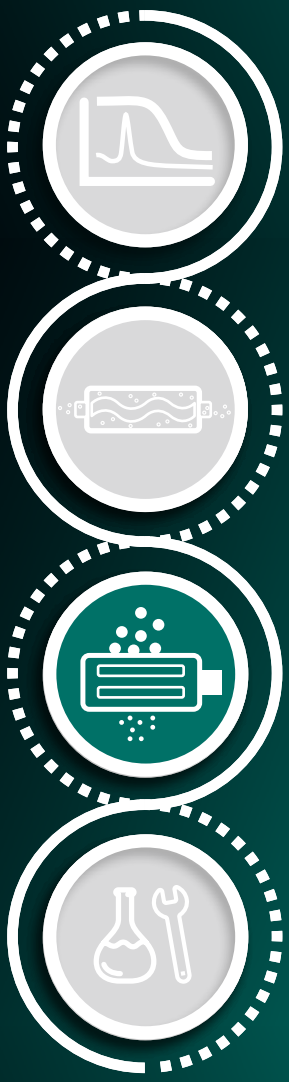
- Highest productivity
  - Low specific energy requirement
  - Significantly narrower residence time distribution
  - Highly-efficient grinding media separation system
  - Reliable scale-up
- Optimum product cooling with an inner pipe of grinding tank made of NETZSCH-*Ceram* C
- Efficient recirculation is possible

Agitator Bead Mill *Alpha*® *Zeta*® 10

PROVEN SOLUTIONS

in Mixing Technologies

Our mixing equipment is used for manufacturing a suspension of solids/powders in a fluid. Solid particles usually appear in agglomerate form due to the high surface forces that occur. To choose the right machine to de-bundle the powder, first we have to check the properties such as diameter, length and type (bundled or entangled). NETZSCH offers a new, compact solution for producing homogeneous dispersions with reproducible quality in an inline process.



Inline Disperser *Epsilon*

The liquid flow in the atmospherically sealed process chamber of the *Epsilon* creates an under-pressure leading to an air expansion from the center of the fed agglomerates.

After the contact between solids and liquids the rotor of the *Epsilon* pumps the suspension to the outlet of the process chamber. At this point the pressure is higher than the atmospheric pressure leading to an air compression as well as to a core wetting. Therefore, the required energy input for the dispersion process is low, reducing product heating to a minimum. Depending on the bulk material properties, the *Epsilon* inline disperser can be operated with different feeding concepts for powders or solids. The Inline Disperser *Epsilon* works dust and emission free and highly energy efficient.

Inline Disperser *Epsilon* 30

SOLUTION FOR CONDUCTIVE SLURRY

De-Bundling Powder

Planetary Mixer PMH / PML

In a planetary mixer the mixing elements rotate on a central axis in a fixed tank, with each element rotating on its own axis as well, thus passing through the entire mixture. One advantage of this machine is, that an exact and fully automatic dosing of different components like binders, additives, active materials and solvents according to a defined time profile is possible to the closed system without emission and contamination of the environment.

Furthermore, all necessary process steps for the production of a high quality battery slurry such as dissolution of binders, premixing and mechanical alloying of dry components, mixing, homogenization, kneading of high viscose pasty slurries and degassing of the slurry can be realized in only one machine under the conditions of an excellent temperature control.

Planetary Mixer PML 90

Intensive Mixer PMD

The PMD-VC intensive mixers are large-volume stationary mixing and dispersing units for processing medium- to high-viscosity product batches.
They are successfully employed primarily in the coatings, printing inks, wall paints, pigments and construction industries.
The special conical geometry of the mixing tank allows the processing of partial batches from approx. 25% of the stated effective volume, and thus guarantees great flexibility.

Intensive Mixer PMD 500

PROVEN SOLUTIONS

in Dispersing Technologies

With the NETZSCH dispersing technologies, product quality is improved and production expense is minimized. In particular, the exceptionally short processing times lead to considerable cost advantages. The use of an Economic Dispersionizer is especially economical compared to standard technologies.

Economic Dispersionizer *Omega*®

The Economic Dispersionizer *Omega*® is the most suitable machine to realize smooth de-bundling or defibrillation of Carbon Nano Tubes (CNT) without cutting of the fiber or tube length or for delamination of Graphite keeping the best possible aspect ratio.

Successful dispersion requires targeted force in order to separate agglomerated particles. The *Omega*® Economic Disperser applies dispersive forces then and there, where they are especially effective: in the *Omega*® disperser body, energy is transformed into very high speeds under pressure. Turbulence and cavitation in perfect combination with specifically-applied shearing forces ensure maximum dispersion results. In addition, the system can be easily adapted to different operating conditions or formulations thanks to the *Omega*® disperser body, which consists of a nozzle with infinitely adjustable flow properties.

The optimum adaptation and increased efficiency not only lead to reduced energy consumption in the production process and thereby less heat development, but also to less wear as compared to traditional technologies.

Economic Dispersionizer
Omega® 500

PLANT & PROCESS ENGINEERING

Your Product - Our Solution

Based on our know-how in machine technology and plant engineering, we are able to offer you complete solutions (turnkey plants) – direct from the NETZSCH laboratory to your production hall.

Plant Engineering

Whether its turnkey solution you need or help on a particular aspect of plant engineering, we have the know-how. NETZSCH has been engineering manufacturing plants for over 100 years, using state-of-the-art production equipment and manufacturing techniques. We undertake projects for companies around the world, including:

- Plant design
- Process monitoring, control and automation
- Software development and real time application programming
- Mechanical engineering
- Electrical engineering
- Steelwork design
- Abatement systems
- Machinery and vessel manufacturing
- Extraction systems

Project Management

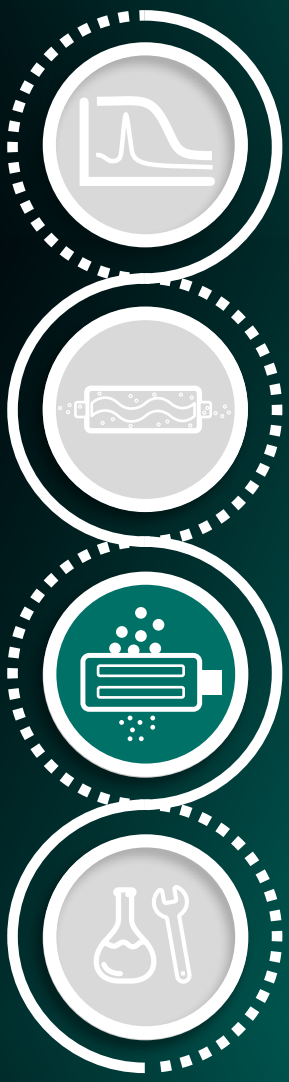
From start to finish, you can rely on professional and exacting project management. At NETZSCH we have the resources and capability to oversee the entire project, even the most complex. All projects are approached with a total commitment to maintaining the highest standards in all areas, including:

- Project planning
- Health and safety expertise including, full working experience of ATEX 94/9 and ATEX 100a
- Construction site supervision and management
- Machinery installation
- Installation and testing of instrumentation and control systems
- Machinery and plant commissioning
- Employee training
- Support throughout production ramp-up

PROVEN SOLUTIONS
in Dry Grinding Technologies

Different technologies of dry comminution and classification are used to grind highly abrasive materials without contamination or to deagglomerate active battery materials gently after the synthesis without changing their original particle shape.

By using the latest patented classifiers, defined particle size distributions can be set or material systems can be dedusted.



Fluidized Bed Jet Mill CGS

A fluidized bed jet mill is a combination of an air jet mill with an integrated dynamic air classifier. In a fluid bed fluidized by the gas jets, particles are accelerated in the free stream and, on the way to the center as well as in the center itself, they collide with other, slower particles and are crushed. Grinding is completely autogenic, so there is no wear on the grinding tools and therefore no contamination of the ground materials. With the air rising in the center, the particles are transported to the classifier wheel, which deflects the particles that are too coarse and only allows particles to pass that meet the set conditions.

- Precise control of the fineness through integrated dynamic air classifier
- Significantly lower compressor capacity as opposed to comparable jet mills (*E-JET*®)
- Highest finenesses and maximum throughput achievable with just one classifier wheel
- Close and steep particle size distribution

Fluidized Bed Jet Mill
CGS 500

VARIOUS APPROACHES TO IMPROVE THE PERFORMANCE

Improved Cathode & Anode Materials in LIB

Classifier Mill CSM *Ceramic*

The classifier mill CSM is a combination of a mechanical impact mill with an integrated dynamic air classifier for steep particle size distributions. In the field of battery materials the CSM is mostly used just for a very gentle deagglomeration with low speed of the impact rotor and high flow rates of the grinding and classifying air, like shown in this example for deagglomeration of active materials without particle breakage and without change of the shape of primary particles.

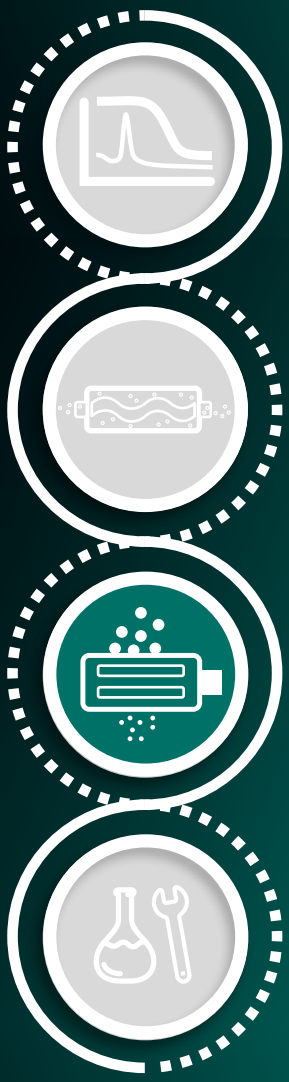
- Highest product fineness without metal contamination
- Closed-loop system to maintain low moisture content (as an option)
- Fast and easy cleaning and maintenance due to optimal access of the grinding chamber and classifier
- Application-oriented product feeding via gravimetric dosing or injector system
- No nonferrous metals (Copper, Zinc) or nonferrous alloys for product- and process gas contacted parts

Classifier Mill CSM 260 *Ceramic*

PROVEN SOLUTIONS

for the Rounding of Graphite

Globally, graphite occurs most commonly as so-called flake graphite, which are graphite flakes containing only a small proportion (maximum 20 %) of actual graphite finely distributed in stone. This natural graphite must be processed using various methods in order to obtain the final product, which is used for battery applications and has a purity of over 99.95 %. As well as the chemical purity, the morphology of the graphite also plays a decisive role. Spherical graphite (SPG) is ideal for the application as a raw material for anodes. Its smooth, small surface prevents flaking and means low irreversible capacity loss and long service life. Thanks to the high tamped density, high charging is achieved and consequently a higher energy density.



High-efficiency Fine Classifier CFS/HD-S

In order to obtain a high-quality product with a precisely defined particle size, the NETZSCH High-efficiency Fine Classifier CFS/HD-S works very efficiently due to its closely defined sharpness of cut and its very specific product flow. Mixing of the classified coarse material and feed material inside the classifier cannot occur.
The optimized classifier wheel geometry produces the finest cut points and high yields that have not been possible with production scale conventional air classifiers with only one classifier wheel. The special dispersion zone directly in close proximity to the classifier wheel uses clean gas to efficiently disperse the material to be classified. Accessibility and easy cleaning of the machine are guaranteed by the hinged housing door and the removable guide vane basket.

High-efficiency Fine Classifier
CFS 30 HD-S

SPHEROIDIZATION OF GRAPHITE

Efficient & Economical Process with the *GyRho* Rounding Unit

Rounding Unit *GyRho*

The system developed from NETZSCH overcomes all disadvantages of the standard technology and delivers an elegant solution for an efficient, process-technology optimized graphite rounding. In a first step, the flake graphite is pre-ground to the optimum initial particle size for spheroidization in a classifier mill or fluidized bed jet mill. The actual particle rounding takes place directly downstream in the newly designed NETZSCH *GyRho* Rounding Unit, which is available in various construction sizes and can be specially designed to suit the necessary output quantity. For larger throughput volumes two (or more) machines can be operated simultaneously and replace a train of 40 machines or more.

Rounding Unit *GyRho*

Plant data for the NETZSCH *GyRho* System compared to competing systems:

30 % higher plant availability in comparison to previously used processes

60 % smaller surfaces at identical production capacity thanks to compact setup

35 % lower operating costs* compared to standard plant trains

* Opportunity-, working-, spare part-, electricity costs, depreciation and costs for graphite concentrate

90 % lower maintenance costs thanks to a significantly lower number of machines

65 % total yield* thanks to the innovative plant concept, optimized grinding chamber- and classifier design

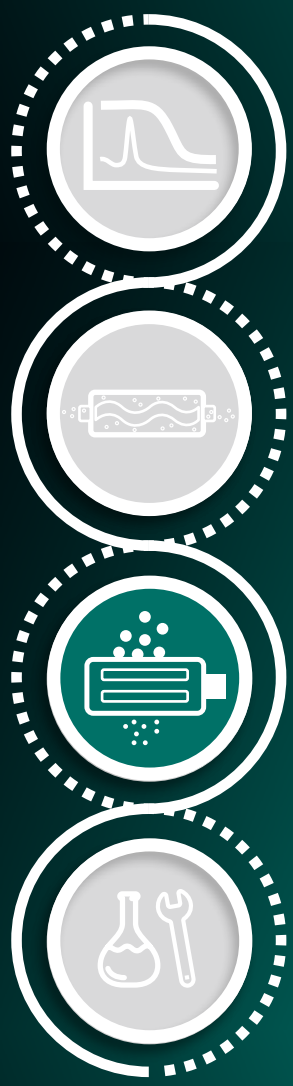
* up to 65 % total yield based on amount of raw material and depending on its origin and type

60 % less energy for identical production capacity (half of installed power and optimized operation mode)

PROVEN SOLUTIONS

for the Rounding of Graphite

The spheroidization process is divided into three phases: Filling, rounding and discharging. During the filling phase, the maximum product volume is conveyed into the processing chamber. During the rounding phase, the particles are stressed until rounded by optimum geometry and process parameters. In the discharging phase, the processing chamber is emptied by a suction unit with cyclone and separated.



Efficient Rounding

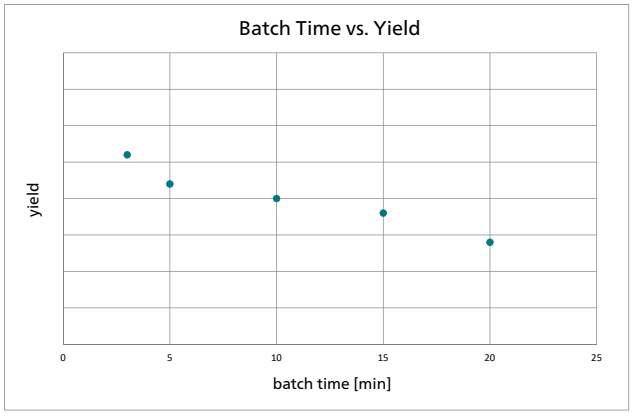
For the manufacture of lithium-ion-batteries spherical graphite is ideally required. Natural graphite and graphite manufactured by synthesis are both plate-shaped and have the typical layered structure. Therefore, the further processing- and size-reduction task consists of the manufacture of a rounded final product with a narrow particle size distribution, a high yield and tap density.

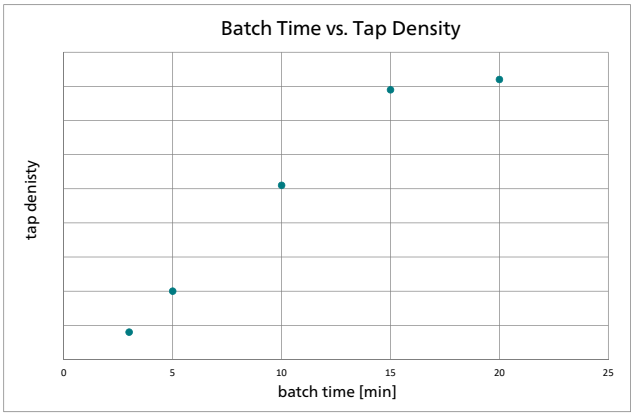
With specific settings, it is possible to achieve the maximum yield of final product with the desired quality. Quality criteria such as distribution width, tap density and particle size can be influenced by varying the process parameters.

In this way, using the newly developed *GyRho* system, all qualities required by the market, such as spherical graphite with a tap density of 963 g/l and a d_{50} value of 16.8 µm (yield based on a raw material volume of 60.7 %) are achievable.

SPHEROIDIZATION OF GRAPHITE

Efficient & Economical Process with the *GyRho* Rounding Unit





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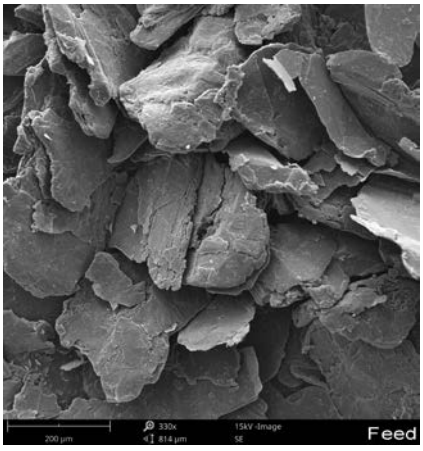
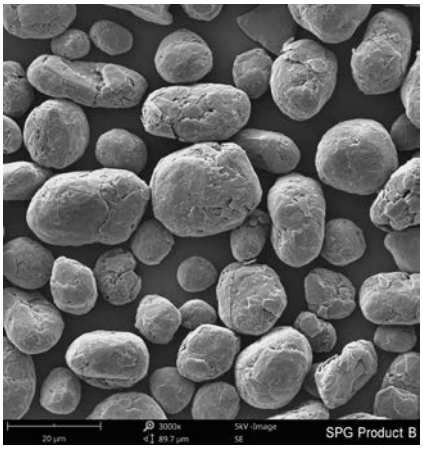


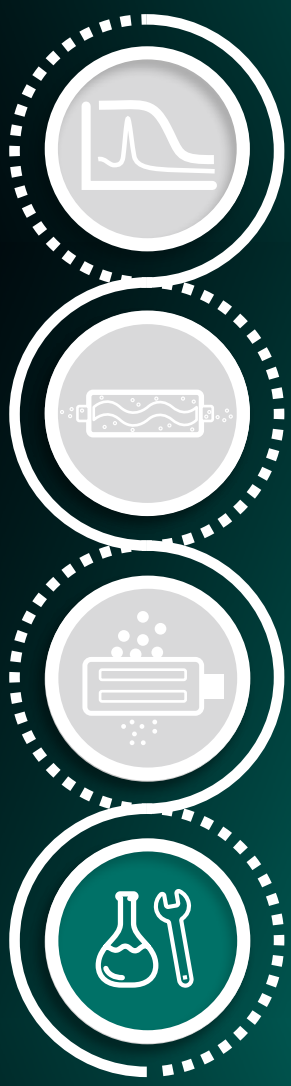
Product A: Yield = 60.7 wt.-%,
d_{50} = 16.8 µm, tap density = 963 g/l



Product B: Yield = 49.3 wt.-%,
d_{50} = 18.4 µm, tap density = 1019 g/l

LABORATORY & SERVICE

NETZSCH applications laboratories are equipped with state-of-the-art technology and are part of our comprehensive service program. NETZSCH laboratories apply the highest quality standards and allow us to precisely test customer products.



Grinding Tests

Grinding tests can be ran in both laboratory scale and production sized machines. After testing is complete, a comprehensive test report, including a sample of the final product, is prepared and sent to the customer.

Customers are welcome to take part in the testing of their product, guaranteeing that all tests are runned exactly according to their requirements. During the trials, customers will also learn more about our company, its manufacturing abilities and our specialized technicians.

Thermal Analysis and Rheology

Within the realm of thermal analysis and thermophysical properties, we offer you a comprehensive line of the most diverse thermal analysis techniques for the characterization of materials (solids, powders and liquids). Measurements can be carried out on samples of the most varied of geometries and configurations.

For the optimization of your chemical processes (e.g., curing behavior of a resin, optimization of the sintering process), we offer a comprehensive service package including test measurements with kinetic evaluation and various predictions for different temperature conditions.

Consult the Experts

Consult with the experts in our applications laboratories to choose the best-suited measuring method for your specific needs.

You will be working with scientists (physicists, chemists, materials scientists) possessing consolidated knowledge about the most varied of methods and materials spectra.

You can rest assured that your matter will be handled confidentially.

PROVEN PERFORMANCE FOR MATERIALS

Highest Quality Standards

Our global service expertise gives you peace of mind for your production

Technical assistance must arrive quickly and work perfectly. That's why we offer an extraordinary range of services, with the assurance that highly-qualified NETZSCH personnel perform these services all over the world. Our specialists provide quick and reliable assistance. We advise you in your own language, wherever you are.

The NETZSCH service network extends to all corners of the globe. As a result, we strengthen the competitive capacity of our customers, facilitate trouble-free, efficient processes and ensure maximum machine and instrument availability.

TECHNICAL SERVICE

- Maintenance, Modifications and Repair
- Procedural commissioning
- Exchange Service
- Spare Part Assistance
- IQ/OQ Documents
- Moving Service
- Software Updates
- Calibration Service
- Process Optimization

TRAINING

- Basic Seminars
- NETZSCH Online Academy
- Comprehensive Instrument and Method Training

LABORATORY

- Application Service and Contract Testing
- Worldwide Locations

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.

Proven Excellence.

customer portal

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