BATTERY TECHNOLOGY

ENABLING PROGRESS IN



Retsch

ELTRA



CONTENT

06 BASIC MATERIALS

- 06 FOREWORD WE ENABLE PROGRESS
- 06 EXPERT INTERVIEW THE ROLE OF LABORATORY ANALYSES
- 06 BASIC MATERIALS METALS, GRAPHITE & POLYMERS
- 10 COMPONENTS ELECTRODE POWDERS, ANODES & CATHODES, ELECTROLYTES & SEPARATORS
- 16 ASSEMBLY BATTERY UNITS & PACKS
- 20 RECYCLING VALUABLE MATERIALS







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BATTERY TECHNOLOGY

ENERGY STORAGE OF THE FUTURE: WE ENABLE PROGRESS

The energy industry and our societies as a whole are facing an epochal change: In the fight against the climate crisis, energy production must be made sustainable within a very short time and we have to abandon fossil fuels. One of the crucial questions for the success of this endeavor is how to store electrical energy and make it available at any time and any place.

Therefore, powerful, efficient and recyclable primary and secondary batteries are a key technology for the future. It comes as no surprise that experts expect global battery demand to grow by roughly 25 percent annually, reaching 2,000 GWh in 2030.

Everywhere around the globe, scientists are working on the technical improvement of existing, and the development of new battery solutions for consumer products as well as industrial applications.

Challenges include the advancement of new technologies, economical use of raw materials, efficient production processes, enhanced battery safety or weight reduction, to name just a few. True to our guiding principle ENABLING PROGRESS, Verder Scientific can assist you in continuously improving battery technology, making more efficient use of resources, ensuring quality and securing maximum sustainability.

Under our umbrella we combine the know-how of five renowned developers and manufacturers of scientific equipment: RETSCH, MICROTRAC MRB, ELTRA, QATM and CARBOLITE GERO are among the leading specialists in their respective fields of activity which are Milling and Sieving, Particle Characterization, Elemental Analysis, Materialography and Hardness Testing, and Heat Treatment.

On the following pages, we present a selection of application-specific solutions for research and development, production and recycling of batteries.

For specific in-depth consultations, our team of experts will be happy to answer your questions at any time.



Dr. Jürgen Pankratz CEO Verder Scientific

ENABLING PROGRESS. TO THE BENEFIT OF MANY.

BASIC MATERIALS METALS, GRAPHITE & POLYMERS

I Sample preparation of ores for analysis

- I Materialographic preparation of lithium metal oxide particles
- I Nitrogen & oxygen analysis of silicon nitrid
- I Process-related particle characterization

COMPONENTS ELECTRODE POWDERS, ANODES & CATHODES, ELECTROLYTES & SEPARATORS

- Thermal processing of carbonaceous material
- I Ball milling to pulverize and mix electrode material
- Particle characterization of electrode material (density, porosity, surface area, pore size distribution, particle size & shape)



RECYCLING VALUABLE MATERIALS

- I Shredding of batteries, fractionizing by sieving
- I Online particle analysis of shredded batterie
- I Thermal treatment to extract reusable components
- I Homogenization of material fractions
- I Elemental analysis of byproducts like slag

ASSEMBLY BATTERY UNITS & PACKS

- I Materialographic sample preparation of Lithium-ion batteries by cutting, grinding and polishing prior to microstructure analysis
- I Carbon & sulfur analysis of lead components

EXPERT INTERVIEW

THE BATTERY OF THE FUTURE NEEDS STATE-OF-THE-ART LABORATORY EQUIPMENT

BATTERIES ARE BECOMING MORE POWERFUL, LIGHTER AND MORE SUSTAINABLE. WHAT ROLE DOES LABORATORY ANALYSIS PLAY IN THE DEVELOPMENT, PRODUCTION & RECYCLING OF ENERGY STORAGE UNITS? IN THIS INTERVIEW, DR. JÜRGEN ADOLPHS, HEAD OF THE APPLICATION CENTER AT MICROTRAC MRB, SUMMARIZES THE CURRENT STATUS.

Dr. Adolphs, in your view, what were the most important developments in battery technology in the past?

More than 200 years ago, Alessandro Volta invented the battery. The physical principle remains unchanged until today, but throughout the years, a great variety of material systems has been developed. Significant progress was achieved when small-size batteries turned from nonchargeable primary to rechargeable secondary batteries. Lithium batteries as power carriers in mobile and stationary power supplies are today's standard in all mobile phones and electric vehicles. For some areas of application, for example space technology, special solutions like Ni-Cad batteries were developed.

What are the biggest challenges today?

We need smart solutions for a variety of applications. Efficient energy storage is surely one of the most pressing tasks in terms of increased energy and power density, capacity, charging efficiency, cycling stability and size. Sustainability, reusability by recycling and, of course, safe batteries are further challenges.

Which analysis methods help developers and producers?

In battery research solid material development, e.g. for electrodes and separators, is supported by particle size and morphology measurement, as well as density, surface area and porosity analysis.



Dr. Jürgen Adolphs Product Specialist, Microtrac MRB

Elemental analysis of basic materials is also essential. These methods are helpful for quality control, however, any analysis result can only be as good as the analyzed sample. Therefore, representative and reproducible sample preparation through homogenization, or sectioning, grinding, polishing is an indispensable step of the process.

What is important when choosing analytical equipment?

First, analytical equipment must be able to analyze the characteristic features of choice. Moreover, performance stability and accurate results are mandatory. Userfriendly operation and safe processing backed by reliable worldwide service – these are the important points.

What are the benefits of working with VERDER SCIENTIFIC?

VERDER SCIENTIFIC covers different technologies with its five manufacturing companies, namely sample preparation, heat treatment, elemental analysis and particle characterization. This interdisciplinary approach is what distinguishes us from other solution providers who usually only cover one step in the process chain.

Where do you see battery technology in ten years' time?

Enormous global efforts are made in research and development for the entire life-cycle-process, supported by machine learning. The employment of Artificial Intelligence will lead to faster results which will truly advance battery development. The Internet of Things (IoT) may become important as a communication control tool for seamless electric power supply to consumers - from small-scale battery packs to large battery energy plants. The next development step will bring forward light-weight batteries, not only with increased energy density but also highspeed charging capability. Besides high voltage technology, they will clearly affect electromobility.

BASIC MATERIALS

METALS, GRAPHITE & POLYMERS

FROM THE VERY FIRST STEP IN THE BATTERY CYCLE – MINING AND EXTRACTION OF BASIC MATERIALS – TECHNOLOGIES FROM VERDER SCIENTIFIC ENSURE THAT THE REQUIRED HIGH QUALITY STANDARDS ARE MET AND MAINTAINED.

Depending on the type, a wide variety of materials is required to produce a battery. A lithium-ion battery, for example, involves the use of nickel, manganese, cobalt, graphite and carbon black for the electrodes, as well as aluminum, copper, binders and polymers employed as separators.

Whether the battery ultimately delivers the promised quality and properties depends to a significant degree on the grade and purity of these raw materials.

Take metallic basic materials, for example: The very first step is to grind and homogenize the sample in a reproducible way to ensure that all subsequent analysis results are trustworthy. In a next step, precise material analysis and determination of the metal content in a mined mineral, as well as the exact determination of chemical compositions and purities, provide the basis for assessment of suitability and market value.

WHAT WE CAN DO FOR YOU

Laboratory and analysis equipment from VERDER SCIENTIFIC ensures reliable quality control of the raw materials through sample homogenization, metallographic preparation, elemental analysis and particle characterization.

Bearing in mind the limited availability of some raw materials and the high share in the overall production costs, reliable quality control of these materials is an essential step in the process chain.

SAMPLE PULVERIZATION

CRUSHERS AND MILLS TO PREPARE ORES FOR ANALYSIS

Only a few grams of sample are needed to analyze the concentration of valuable metals like lithium, manganese, or cobalt. The preparation of a representative sample for analysis may involve several steps: pre-crushing, fine-grinding, sample dividing and pellet pressing. RETSCH offers a wide range of crushers, mills, accessories and assisting devices to ensure reproducible sample homogenization for subsequent analysis.



Sample preparation of copper ore: A: Original sample, B and C: sample pre-crushed in the Jaw Crusher BB 600 / BB200, D: sample pulverized in the Disc Mill RS 200 and E: sample divided with PT 100 and pelletized using the Tablet Press PP35, for XRF analysis.

MATERIALOGRAPHY

INVESTIGATION OF LITHIUM METAL OXIDE PARTICLES

Lithium metal oxide particles are subjected to metallographic preparation and examined by light microscopy and scanning electron microscopy with EDX detector to investigate their size and chemical composition.

QATM's epoxy-based cold mounting materials (KEM 90 and KEM 92) allow for mounting the oxide particle powders without any gaps, preparing them for grinding and polishing. Thanks to an extensive selection of polishing cloths and polishing diamond suspensions, a wide variety of lithium metal oxide particles can be prepared. The Qdoser system is used for automated dosing during polishing and fine polishing.



An example of powder particles mounted in KEM 90.



JAW CRUSHER BB 200

SHORT FACTS

- I Sample preparation of battery raw materials for analysis
- I Pre-crushing, fine grinding, sample dividing and pellet pressing
- FIELD OF USE

 Quality Control

Retsch





AUTOMATIC DOSING SYSTEM QDOSER ECO & CONSUMABLES

SHORT FACTS

- Metallographic preparation for optical microscopy investigation
 Corresponding consumables for
- battery casing preparation

FIELD OF USE

I Research & Quality Control





ELEMENTAL ANALYSIS

NITROGEN & OXYGEN ANALYSIS OF SILICON NITRIDE

Lithium-based batteries can incorporate silicon nitride as part of an electrode. The nitrogen content is measured to indicate the purity of the silicon nitride, while the oxygen content is determined to evaluate electrical properties. The ELEMENTRAC ONH-p 2 is perfectly suited for precise measurements of both elements. The highly sensitive detectors used in ELTRA Elemental Analyzers accurately determine element concentrations ranging from low parts per million content to high percentages.



Measurement graph ELEMENTRAC ONH-p-2 The red curve shows the release of nitrogen, the black curve the release of oxygen.

ANALYSIS RESULTS

Silicon nitride (Si_3N_4) (reference material from BAM: ED 101) Oxygen content: 2,06% +- 0,05; Nitrogen content: 43,54% +- 0,19



OXYGEN / NITROGEN / HYDROGEN ANALYZER ELEMENTRAC ONH-p 2

SHORT FACTS

- I Wide range measurement of O/N/H for research and production control
- I Inert gas fusion analyzer with TCD and IR detection

FIELD OF USE





ENABLING PROGRESS.

HEAT TREATMENT ELEMENTAL ANALYSIS MATERIALOGRAPHY & HARDNESS TESTING MILLING & SIEVING PARTICLE CHARACTERIZATION Under the roof of VERDER SCIENTIFIC we support thousands of customers worldwide in realizing the ambition we share. As their technology partner behind the scenes, we deliver the solutions they need to make progress and to improve the everyday lives of countless people. Together, we make the world a healthier, safer and more sustainable place.

www.verder-scientific.com

PARTICLE SIZE & SHAPE ANALYSIS

PROCESS-RELATED PARTICLE MEASUREMENT

The particle size distribution of crushed ore material is important to control the various process steps in battery cell manufacturing. For this purpose, a complete recording of important size parameters is crucial. The CAMSIZER 3D provides relevant size and shape parameters within a very short time and can be used in harsh production environments. This means that deviations in the processes can be detected, and adjustments can be made almost immediately. The CAMSIZER 3D is also available in a version for online measurement and in an XL version for particle sizes up to 135 mm.



CAMSIZER 3D analysis of different grades of an ore sample (note the logarithmic scaling of the x-axis)



PARTICLE SIZE & SHAPE ANALYZER CAMSIZER 3D

SHORT FACTS

- I Dynamic Image Analysis for particle size & shape analysis
- particle size & shape analysis
 3-dimensional characterization of particles

FIELD OF USE

I Research & Quality Control





COMPONENTS

ELECTRODE POWDERS, ANODES & CATHODES, ELECTROLYTES & SEPARATORS

ELECTRODES, SEPARATORS, AND ELECTROLYTES ARE ESSENTIAL BATTERY COMPONENTS. THEIR MATERIAL FORMULATION AND PRODUCT QUALITY DEFINE THE PERFORMANCE OF THE BATTERY AND ALSO THE SUSTAINABILITY AND OVERALL COST OF THE PRODUCTION PROCESS.

CHEMICAL FORMULATION

Different types of batteries require different basic materials. The chemical formulation and quality of the electrode materials have an impact on the battery's key characteristics like energy density, power density, cycle life, charging rate or temperature stability. In Lithium-ionbatteries, for example, multi-metal oxides of cobalt, nickel and manganese are currently the most common cathode materials whereas anodes are mainly based on graphite.

To improve the performance of batteries, researchers focus on the development of new chemical compositions of the electrode powders, separators and electrolytes.

MATERIAL QUALITY & PROCESS CONTROL

The manufacture of electrode components ready for assembly involves various steps. To produce electrode powders and electrode foils for Lithium-ion-batteries, particle size reduction, heat treatment, slurry mixing, coating, drying and calendaring are just a few of the required procedures.

Manufacturers not only focus on a high degree of purity of the purchased graphite and metals, but also on clean production and transport processes. Any material contamination with foreign substances, impurities or moisture induced by machinery, humans or environmental influences should be avoided, to guarantee consistent high quality of the battery components. The chemical composition, particle size, specific surface area or pH-value of individual batches are constantly analyzed throughout production to ensure quality control without gaps.

OUR CONTRIBUTION

VERDER SCIENTIFIC supports manufacturers of active materials and battery components in all these challenges. Our solutions help you to further develop your products and production processes and to ensure consistently high quality.

On a laboratory scale, mills from RETSCH are used to reduce the particle size of electrode materials and produce homogenoeus mixtures. High temperature furnaces from CARBOLITE GERO allow you to thermally process cathode and anode materials. With analyzers from MICROTRAC MRB you can determine material density, porosity, pore size distribution, homogeneity, coating quality, particle size and shape, and much more, to check the material properties or the exact physical or chemical structure.

And that's just the beginning: Our application specialists will be happy to introduce you to further solutions from our group's portfolio.



HEAT TREATMENT

THERMAL PROCESSING OF CARBONACEOUS MATERIAL AT UP TO 3000°C TO PRODUCE HIGH-QUALITY ELECTRODE MATERIAL

Carbonaceous material is converted into uniform, stacked layers by subjecting it to high-temperature processing. The resulting nanostructures are held together by Van der Waals forces, which are weak intermolecular forces that occur between molecules or atoms. The HTK and GLO furnace series are specifically designed to optimize temperature control for the production of consistent and uniform materials. They feature off-gas handling systems to ensure safety and compliance with environmental regulations. The uniformity of the stacked layers enhances the movement of electrons and ions through the material, an essential feature for high-performance applications such as batteries.



CHAMBER FURNACES FOR GRAPHITISATION

I Developing high quality graphite for

I Off gas management system, safe, environmentally friendly

I Research & Quality Control

SHORT FACTS

FIELD OF USE

electrode material I Thermal processing at 3000°C under inert atmosphere



Phase changes in carbon to produce amorphous carbon structures.

SAMPLE PULVERIZATION

LABORATORY BALL MILLS WITH TEMPERATURE CONTROL TO DEVELOP ALL-SOLID-STATE BATTERY COMPONENTS

The physical arrangement of active materials is one of the key characteristics of all-solid- state batteries. RETSCH's high energy laboratory ball mills are used to pulverize, mix and coat electrode materials on a laboratory scale. Ball mills with automatic temperature control are ideally suited, for example, to prepare specific particle size distributions of temperature-sensitive electrode materials, or to execute mechanochemical reactions under controlled temperatures and atmospheres. The MM 500 control allows to prepare battery materials at temperatures between -100°C and + 100°C.



Picture of a thiophosphate powder with 3 mm grinding balls in a 125 ml Screw-Lock jar. The sample was pulverized in the MM 500 control at -100 °C under inert atmosphere; the jar was opened in a glove box.



MIXER MILL MM 500 control

SHORT FACTS

- I Pulverizing, mixing and coating of battery active materials
- I Material development for all-solidstate-batteries
- I Temperature controlled grinding

FIELD OF USE

Research





SAMPLE PULVERIZATION

BALL MILLING TO PULVERIZE AND MIX ELECTRODE MATERIAL

RETSCH ball mills are used to pulverize and mix battery active materials on a laboratory scale. A typical area of application is the preparation of powders and slurries of electrode materials. The grinding, coating and mixing process can be carried out in planetary ball mills and mixer mills. High energy laboratory ball mills such as the MM 500 nano or Emax pulverize particles down to nanometer size.



Particle size distribution of silicon powder obtained by wet grinding with the Mixer Mill MM 500 nano using zirconium oxide grinding tools (green curve) and with the Emax (yellow curve) for comparison. Measurement performed with Microtrac SYNC analyzer.

PARTICLE SIZE & SHAPE ANALYSIS

LASER DIFFRACTION AND DYNAMIC IMAGE ANALYSIS TO MEASURE THE PARTICLE SIZE AND SHAPE OF ELECTRODE MATERIALS, ELECTROLYTES AND SEPERATORS

NMC (Nickel Manganese Cobalt) and Lithium are the main active cathode components in LIB (Lithium Ion Batteries), while graphite is a typical material for the anode. Both the particle size distribution and the shape of the electrode materials are important criteria for battery quality. Size and shape can affect the kinetics and reactivity of the battery materials, determining the diffusion rate, power density, anode layer porosity and the discharge rate. A typical particle size for electrode materials is approximately 6 microns. Particle size distribution and shape are reliably measured using a combination of Laser Diffraction and Dynamic Image Analysis.



Scatter Diagram of Area Equivalent Diameter and Sphericity of the Anode Material



HIGH ENERGY BALL MILL EMAX

SHORT FACTS

- I High energy laboratory ball mills for grinding of battery active materials
- Pulverization down to nanometer scale

FIELD OF USE

I Research & Quality Control







PARTICLE SIZE AND SHAPE ANALYZER SYNC

SHORT FACTS

- I Laser Diffraction and Dynamic Image Analysis for particle size analysis
- I Particle size distribution and shape of active battery materials

FIELD OF USE

I Research & Quality Control



SIEVE ANALYSIS PARTICLE SIZE ANALYSIS OF BATTERY MATERIALS BY SIEVING

For quality control of electrode materials RETSCH sieve shakers are used to characterize the particle size distribution. The portfolio includes shakers for various sieve diameters based on different movement patterns, with sieve apertures ranging from 10 µm to 125 mm. The Air Jet Sieving Machine AS 200 jet is particularly suitable for sieving fine powders, like graphite, which tend to agglomerate. To divide the sample into representative fractions before sieving, the sample divider PT 100 is used.



Sample remainder on a 20 μ m sieve after sieving fine graphite powder with particle sizes < 300 μ m. Sieving was performed with the "Swiss method", using the Sample Divider PT 100 and the Air Jet Sieving Machine AS 200 jet.

DENSITY MEASUREMENT

GAS PYCNOMETERS TO MEASURE THE DENSITY OF ELECTRODE MATERIALS

The main active cathode components in a Lithium Ion Battery (LIB) are nickel, manganese, cobalt (NMC) and lithium, while graphite is commonly used as the anode material.

The density (g/cm³) is a crucial factor in characterizing and evaluating battery active materials. A gas pycnometer determines the density of electrode materials by measuring the amount of displaced gas (helium).



Graphite powder measured with BELPYCNO L, density = 2.223g/cm³



AIR JET SIEVING MACHINE AS 200 JET

SHORT FACTS

- I Sieving machines for particle size analysis
- I Sieve shakers with various movement patterns and different sieve diameters
- I Sample divider for representative division of bulk materials

FIELD OF USE

Research







GAS PYCNOMETER BELPYCNO L

SHORT FACTS

- I Temperature-controlled gas pycnometer for density measurement
- I Characterization of the skeletal density of battery active material
- I Standard method for R&D and QC

FIELD OF USE

I Research & Quality Control



GAS ADSORPTION MEASUREMENT

GAS ADSORPTION TO SPECIFY THE SURFACE AREA AND PORE SIZE DISTRIBUTION OF ELECTRODE MATERIALS

The specific surface area and a pore size distribution of electrode materials can be derived from the measured gas sorption isotherm. The surface area related to the mass as specific surface area (m²/g) is an important parameter in the characterization and evaluation of battery active materials, as their morphology has a direct impact on the battery performance.



BET Specific Surface Area (SSA) of NMC with 0.34m²/g derived with gas adsorption devices Belsorp Mini X

POROSIMETRY MERCURY POROSIMETRY TO DETERMINE THE POROSITY OF ELECTRODE AND SEPARATOR MATERIALS

Mercury Intrusion Porosimetry (MIP) is used to assess the porosity of electrode and separator materials. Graphite is commonly used as the anode material, while variations of porous polymers form the basis of separators. The pore volume and distribution of pore size are crucial parameters in characterizing and evaluating batteries. Other obtainable results include the specific surface area, bulk and apparent density, and bulk modulus of electrodes.



BELPORE pore size distribution of a bimodal separator membrane



SURFACE AREA & PORE SIZE DISTRIBUTION ANALYZER BELSORP MINI X

SHORT FACTS

- I Gas sorption to characterize the specific surface area and pore size distribution (<1nm up to >300nm)
- I Characterization of the porosity of battery active material
- I Standard method for R&D and QC

FIELD OF USE

I Research & Quality Control





MERCURY POROSIMETER BELPORE SERIES

SHORT FACTS

- I Mercury intrusion for porosity measurement in a range from 3.6 nm to more than 1 mm
- I Characterization of porosity of electrodes and porous separators
- I Standard method for R&D and QC

FIELD OF USE

I Research & Quality Control



ASSEMBLY

BATTERY UNITS & PACKS

THE WHOLE IS MORE THAN THE SUM OF ITS PARTS: THIS IDIOM ALSO APPLIES TO BATTERIES. THE ASSEMBLY OF THE VARIOUS COMPONENTS INTO A FUNCTIONING BATTERY CELL IS COMPLEX. SEAMLESS QUALITY CONTROL AND PERFORMANCE ANALYSIS ARE ESSENTIAL FOR CONTINUOUS PRODUCT AND PROCESS IMPROVEMENT.

The design and manufacturing of batteries have a significant impact on performance. Battery manufacturers continuously need to ensure the quality of their production process, the foundations for which are already laid in the laboratory. They are facing the challenge of constantly evolving their formulations and procedures to provide answers to new market demands.

MANUFACTURING PROCESS

To a certain extent, the production steps are the same for all battery types; nonetheless, each individual battery system also requires specific steps and process control. The assembly is largely automated, but for prototyping and research and development semi-automated or manual assembly is also common practice.

Taking a Lithium-ion-battery as an example, the assembly is based on 5 steps:

- 1. Anode and cathode coils are cut to the required format.
- Electrodes and separators are stacked, wound or folded together to form a cylindrical, prismatic or pouch cell.
- Contacts for positive and negative electrodes are created, e.g. by welding together the uncoated tabs of a pouch cell.
- 4. After housing, the stacks are filled with electrolyte and degassed, if necessary. The battery unit is finally sealed.
- The LIB is then initiated under defined conditions in a first charging and discharging circle, to form the interface layer between electrolyte and electrode.

Maturing of the battery units, functionality proof and performance testing are also part of the process. The final testing step focuses on the stability of the electrochemical performance and battery safety. Capacity, internal resistance and self-discharge rate are checked to ascertain the properties of the freshly produced unit. If all tests are successful, the battery is ready for use or for the integration into a battery pack.

SOLUTIONS TO ACCOMMODATE YOUR NEEDS

VERDER SCIENTIFIC equipment supports manufacturers throughout the production and quality assurance process. In each of the three phases - assembly, aging and testing - the sample preparation, heat treatment and analysis solutions developed under our group's umbrella help achieve key quality parameters.

For example, they are used by manufacturers to ensure that the electrode coating has a uniform thickness and no cracks, adheres optimally to the substrate, and shows high cut quality and geometric alignment accuracy.

Only if all this is achieved can cathodes and anodes with equal capacitance, durable electrical insulation and reliable package seal be produced. Elemental analyzers from ELTRA and devices for metallographic sample preparation from QATM are used in this context.

MATERIALOGRAPHY

CUTTING OF LITHIUM-ION BATTERIES

LIB cutting can vary depending on the desired outcome. Cut-off machines can be used for sectioning the battery casing to take the whole jelly roll out, or for cutting the whole battery with casing and electrodes, while a scalpel is suitable for sectioning only the electrode foils.

A rotating clamping tool is used to cut the casing and take the jelly roll out. QATM cut-off machines offer a wide range of clamping tools for sectioning entire batteries. The image below demonstrates an LIB cutting process.



Sectioning of a complete LIB with QATM cut-off machine

MATERIALOGRAPHY MOUNTING OF LITHIUM-ION BATTERIES

Mounting Lithium-Ion batteries involves different techniques depending on the components being handled. The casing and spot weld can be hot mounted in some cases, but the electrodes require cold mounting due to their sensitivity to temperature and pressure. Mounting can be performed before cutting to ensure foils and other components remain in the sample. It can also be done after cutting to prepare the sample for grinding and polishing. To prevent gaps between the mounting materials and the sample during cold mounting, it is recommended to use mounting materials with minimal volume shrinkage and polymerization temperature. For mounting LIBs, the epoxy-based cold mounting materials QATM KEM 90 and KEM 92 are the best choices due to their low polymerization temperature of 60°C and 35°C, respectively, and minimal gap formation.



Cold mounted LIB samples



CUT-OFF MACHINE QCUT 350 A

SHORT FACTS

- I Saving time by cutting the sample near to the target surface.
- I Use of rotation device to clamp the samples.

FIELD OF USE

I Research & Quality Control







HOT MOUNTING PRESS QPRESS 50

SHORT FACTS

- I Using epoxy-base cold mounting materials with minimum polymerization temperature and shrinkage.
- I High flexibility by hot mounting due to preheating function, cooling modes, maintenance tasks and user account management feature.

FIELD OF USE

I Research & Quality Control





MATERIALOGRAPHY

GRINDING AND POLISHING OF LITHIUM-ION BATTERIES

Grinding and polishing can be done on different parts of LIBs. Sometimes the casing should be prepared and examined under light microscopy or SEM (also with EBSD detector), in other cases the electrodes or spot welds should be prepared. The cap of the LIB is in many cases low carbon steel with Ni-coating. The image shows the cap of an LIB after 1 µm polishing and etching with Klemm I. For powering certain devices, large battery packs consisting of multiple lithium-ion cells connected in parallel-series configurations are required. A robust and flawless spot weld is a critical concern for many industries, like aerospace. The other important parts of LIB are electrodes. The anode is often a copper foil coated on one or both sides with graphite, and the cathode is in many cases an aluminum foil coated on one or both sides with metal oxide particles (for example LiNiMnCoO, particles).



GRINDING & POLISHING MACHINE QPOL 250 A2-ECO



The microstructure of the LIB-casing after etching with Klemm I for 40 seconds

ELEMENTAL ANALYSIS

CARBON & SULFUR ANALYSIS OF LEAD COMPONENTS

Sulfur measurement by combustion analysis is used for final quality control of charged leadbased batteries. The electrodes consist of lead and lead oxide and need to be free of sulfur. The properties of the battery paste have an impact on the performance and life span of the battery and the contained lead sulfate determines its qualities. ELTRA's C/S analyzers provide rapid and reliable measurement of carbon and sulfur concentration from the low ppm range up to 100%.



Measuring graph ELEMENTRAC CS-i: PbSO₄ Measurement The red curve shows the release of sulfur, the blue curve the release of carbon.

ANALYSIS RESULTS

Lead sulfate – Carbon content: 0,15 % +- 0,01 ; Sulfur content: 5,78 % +- 0,21 Lead carbonate – Carbon content: 3,23 % +- 0,07 ; Sulfur content: 1,15 % +- 0,04



- I Water-free polishing suspensions and lubricants
- I High reproducibility because of automatic preparation

FIELD OF USE

I Research, Quality Control & Production







CARBON / SULFUR ANALYZER ELEMENTRAC CS-i

SHORT FACTS

- I Fast and reliable C/S analysis in all relevant battery components
- I Compliant to all relevant standards like ASTM E 1019

FIELD OF USE

I Production





RECYCLING

VALUABLE MATERIALS

10,00

DEMAND FOR ENERGY STORAGE IS GROWING. THE NUMBER OF MOBILE DEVICES INCREASES, THE MOBILITY SECTOR IS ON THE WAY TO ELECTRIFIED VEHICLES AND THE OVERALL ENERGY TRANSITION PROCESS COUNTS ON HIGH PERFORMANCE BATTERIES. BUT LIFE OF A BATTERY IS LIMITED. DEPENDING ON BATTERY TYPE AND OPERATION, SOONER OR LATER A BATTERY IS EXHAUSTED AND MUST BE REPLACED.

For reasons of cost efficiency, supply chain stabilization and environmental protection, it is essential to design battery production in line with the circular economy and to recover basic materials from exhausted batteries for reuse.

The achievable recovery rate strongly depends on the battery system. The recycling rate of lead-acid or alkaline-manganese batteries, for example, is notably high, thanks to well-established return routes of the exhausted batteries and well-developed recycling process technologies.

COMPLEX PROCESSES DEMAND FOR SEAMLESS QUALITY CONTROL

A recycling process starts with health testing, discharging and dismantling of battery packs. This is followed by mechanical, thermal and chemical treatments to separate, extract and refine valuable materials. For quality control, the chemical composition of intermediate or final products is continuously analyzed. After all, quality standards for secondary and primary raw materials are the same.

Recovery and processing of Lithium-ion batteries is extremely complex, as their chemical composition is not standardized, and various geometric forms are available on the market. Currently, different recycling technologies are employed to best recover steel and plastic from the casing and valuable metals from the battery active material. The sustainable separation and extraction of valuable metals like lithium, cobalt, manganese, nickel or copper from the composite fractions is a challenging task. The recovery of graphite becomes increasingly important, however, no common solution has been established yet.

WE WORK TO ENABLE CIRCULAR ECONOMY

To support specialized companies and battery producers in quality control and process optimization of recycling, VERDER SCIENTIFIC companies offer various solutions for sample preparation and solids analysis.

ELTRA elemental analyzers, for example, help with degradation and end-of-life analysis. Laboratory mills and sieving machines from RETSCH are used for shredding, homogenization and mechanical sorting. Real time data on particle size and shape of the shredded materials is continuously monitored with MICROTRAC's online particle analyzers. Thus, our solutions help to develop cost- and energyefficient, as well as environmentally friendly recycling processes.

MILLING & SIEVING

SHREDDING AND SIEVING OF BATTERY MATERIAL FOR SEGREGATION

In the recycling process, shredding of dismantled or complete batteries is one of the initial steps. RETSCH cutting mills are used to shred batteries or components on a laboratory scale which helps researchers to develop new recycling routes. RETSCH sieving machines are employed to separate the different material fractions, for example black mass from polymeric and metallic parts.





 $\odot BLB$

Left: Shredded LIB pouch cells. The cells are processed with the SM 300 on a laboratory scale. Right: Recycling fractions of LIB pouch cell. Sieving is one method to separate the black mass from the metallic and polymeric foils.

PARTICLE SIZE & SHAPE ANALYSIS

ONLINE PARTICLE ANALYSIS OF SHREDDED BATTERY MATERIAL

During a battery recycling process real time data on particle size and shape is continuously monitored. The results are transferred to a process control system where the recycling process can be largely optimized and automated. MICROTRAC uses patented online 3D analysis technology with the CAMSIZER ONLINE and CAMSIZER ONLINE XL. We have over 25 years of experience helping production departments improve their product quality with Online Dynamic Image Analysis.

- I Characterizes 32 morphological parameters of your material including 3D measurements
- I Non-contact measurement of dry particles from 160 μ m 135 mm
- I Explosion-proof
- I Optimized for control room use
- I Customizable sample conditioning



MICROTRAC Innovation: Patented 3D Measurement Technique



CUTTING MILL SM 300

SHORT FACTS

- I Development of new recycling routesI Powerful cutting devices with
- variable speed I Sieving machines for sieve diameters up to 450 mm

FIELD OF USE

I Research & Quality Control





PARTICLE SIZE & SHAPE ANALYZER CAMSIZER ONLINE XL

SHORT FACTS

- I Dynamic Image Analysis for
- particle size & shape analysis I Online measurement provides
- real time data

FIELD OF USE

I Quality Control





HEAT TREATMENT

1000

900

THERMAL PROCESSING OF RECYCLED BATTERY MATERIALS TO EXTRACT REUSABLE ELEMENTS

Thermal processing is one process step that can be used in research applications for processing batches of material to recover recyclable elements and precious metals under modified atmosphere and in air. CARBOLITE GERO batch furnaces can incorporate a balance to measure weight loss over time and against temperature. Off gas handling systems are available to ensure environmental impact is minimized. Under European Directive 2013/56/EU 50% by mass of battery materials need to be recycled.

6600

5580



ASHING FURNACE AAF - BAL





Loss on ignition measurement of material to ascertain mass loss, indicating furnace temperature, thermal oxidizer temperature and mass change of the sample.

MILLING & SIEVING LABORATORY MILLS TO HOMOGENIZE BATTERY RECYCLING FRACTIONS

In a battery recycling process, the exhausted batteries are separated into different material fractions. To evaluate the efficiency of a recycling process and to investigate the purity of each fraction, samples are homogenized and analyzed. The market value of the black mass, for example, depends on its content of valuable metals, like lithium or cobalt. Black mass can be homogenized in a ball mill. To avoid cross contamination, metallic or ceramic grinding tools should be chosen, respectively. The polymeric material fraction and metallic foils are first pre-cut with a cutting mill and then pulverized, usually at cryogenic temperatures, for example with



Various material fractions of a recycling process, before and after homogenization for subsequent analysis to determine purity and market value.

SHORT FACTS

- I Thermal extraction of desirable materials from recycled battery components
- I Loss on ignition testing

FIELD OF USE

I Research & Quality Control







SHORT FACTS

- I Sample preparation to analyze the chemical composition of recycling fractions
- I Laboratory ball mills for the homogenization of brittle and tough recycling fractions, like black mass, polymers or metallic foils

FIELD OF USE

I Research & Quality Control





ELEMENTAL ANALYSIS

C/S DETERMINATION IN BYPRODUCTS LIKE SLAG

The lead content of batteries can be recycled for use in new batteries in an environmentally friendly way. Lead is present as lead sulfate in exhausted batteries, and also in slags which are a byproduct of battery production and recycling. Sulfate can be precisely measured using ELTRA's C/S combustion analyzers, allowing fast and easy determination of the present lead.



Measuring graph ELEMENTRAC CS-i The red curve shows the release of sulfur, the black curve the release of carbon.

ANALYSIS RESULTS (LEAD SLAG) Carbon content: 9,7 +- 3,4 % ; Sulfur content: 10,9 +- 1,9 %



CARBON / SULFUR ANALYZER ELEMENTRAC CS-i

SHORT FACTS

- I C/S measurement via combustion analysis with IR detection
- Wide C/S measuring range from ppm up to 100%

FIELD OF USE





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ENABLING PROGRESS IN BATTERY TECHNOLOGY





