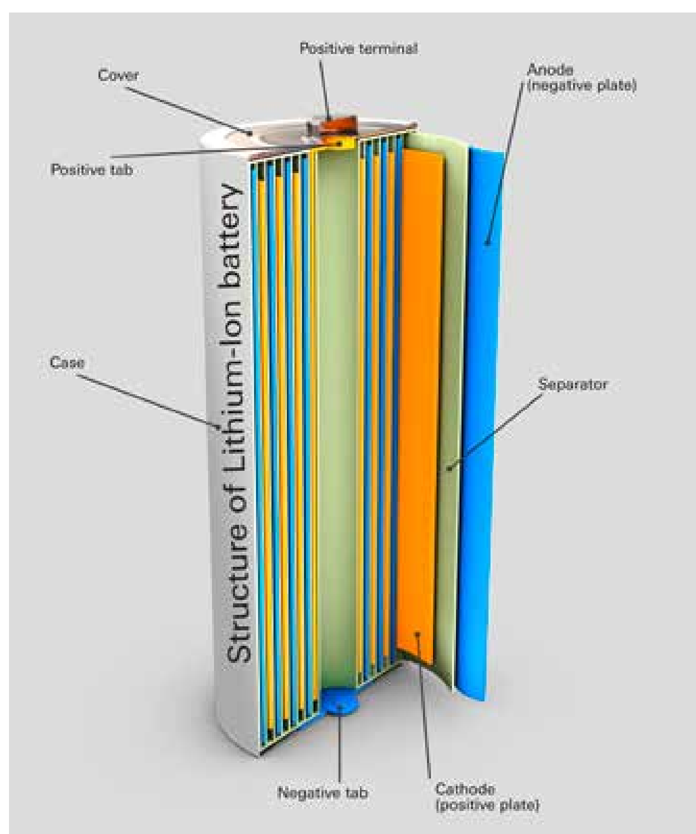
Battery production

Vacuum and leak detection solutions for clean energy

PFEIFFER VACUUM

Battery production

Vacuum and leak detection solutions for clean energy



Content

Battery production

Vacuum and leak detection solutions for clean energy

Today, more than ever, environmentally friendly technology solutions are needed to prevent further global warming. Our world has surpassed the threshold of 400 ppm carbon dioxide in the atmosphere and the average temperature in nearly hundred years has increased by almost 1°C.

Energy storage and electro mobility

The ongoing transition from fossil energies to renewables requires short-term storage media with high efficiency, like batteries and flywheels. One of the most promising future energy storage options are electrical vehicles with grid capability. In addition to environmentally friendly mobility this option increases the flexibility of a power system by making optimum use of the energy whenever it is available from a renewable source like a photovoltaic solar cell installation.

Battery production

Main drivers

General

Batteries, especially Li-Ion batteries, have become an integral part of our everyday life. They are used in mobile devices such as laptops, smartphones and other wearables. Batteries are even powering our cars. But how are these compact power storage devices manufactured and what does it all have to do with vacuum and leak detection?

This brochure will answer these questions by providing a detailed look into production related vacuum applications in the manufacturing of today's most wanted technology: Li-Ion batteries.

Electro mobility

Mobility has to become more climate-friendly in the future. The CO_2 emissions caused by traffic amount to around 24%[1)] of global CO_2 emissions. Thus mobility has a considerable impact on our environment. More and more CO_2 is being released into the atmosphere. The consequence: our earth is getting warmer and warmer. Batteries are having the potential to reduce mobility related CO_2 emissions. Additionally, they can be used for energy storage from renewable energy sources.

Grid stability

Battery storage systems can also be used to stabilize power grids. These are intended to ensure grid stability by stabilizing short-term fluctuations in the grid frequency. In order to compensate for these fluctuations, energy must be supplied to or withdrawn from the grid as quickly as possible, depending on demand. Battery storage systems are perfectly suited to quickly react to these grid fluctuations.

Future technologies

In addition to the Li-Ion battery cell with liquid electrolyte, research is already underway on a new generation of Li-Ion batteries: the so-called „solid state battery", which is characterized by a solid electrolyte. Other vacuum applications compared to the present production processes of Li-Ion batteries will then become relevant. However, it will require some more time before the new battery generation is ready for the market.

[1)] https://de.statista.com/statistik/daten/studie/167957/umfrage/verteilung-der-co-emissionen-weltweit-nach-bereich/

Battery production

Production steps that require vacuum

Fundamental research

Battery components play a major role in battery production. Especially the composition and structure of the electrodes which have a considerable influence on the performance characteristics and lifetime of a battery cell. Furthermore, coating processes under vacuum are crucial to protect the battery from corrosion or negative effects on the boundary layers between the electrodes. The housing of a battery should also not be neglected. A high tightness is mandatory to prevent moisture from penetrating the battery which would cause an unacceptable deterioration in performance. Therefore, fundamental research is of great importance.

Mixing

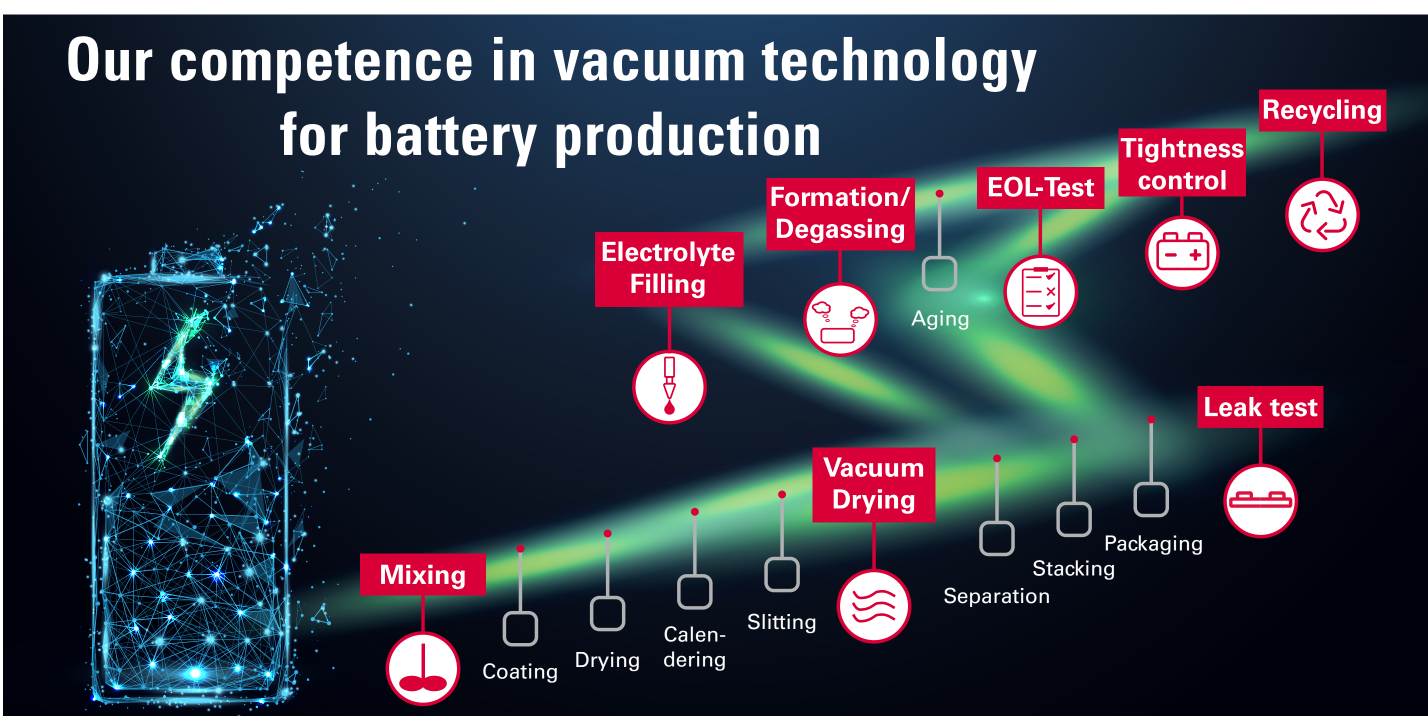
When mixing the slurry for the electrodes of the cell, it is necessary to avoid the introduction of gas bubbles. With the help of vacuum, air pockets inside the slurry can be prevented and thus homogeneous slurry can be produced.

Vacuum drying

An important step in battery production is the in-depth drying of the materials. Residual moisture in the cells leads to rapid loss of performance and premature aging. Drying the coated electrodes of the cell under vacuum guarantees minimum residual moisture and prepares the electrodes for the next production steps in the dry room.

Electrolyte filling

When filling the cell, the electrolyte is introduced via a high-precision dosing lance under vacuum. A defined pressure profile, by alternating evacuation and inert gas purge of the cell, activates the capillary effect. This leads to a homogeneous distribution of the electrolyte. By this optimized wetting process quality and lifetime of the cell is increased.



Formation/Degassing

During formation of battery cells, a strong gas evolution occurs in the first charging process of the cell. Under a protective atmosphere in vacuum, the emitting gases will be extracted. Due to the toxicity and sometimes explosive risk nature of those gases, customer specific requirements on vacuum technology have to be taken into account.

End-of-Line test

At the end of production a battery cell has to fulfill the manufacturers quality level. Electrical safety, leak tightness, and also the ordered specifications of the end-customer are the main reasons to run end-of-line tests. High cycle times have to be met as the current and future demand in terms of quantity and quality needs 100% testing.

Leak detection

In order to ensure long-term performance and safe operation of a battery, leak detection is an essential step in quality control. This applies for battery components, cooling, battery modules and battery packs. The cell has to be protected from moisture ingress in order to ensure the safety of the system.

Battery recycling

To enable the shift from conventional to electrical mobility the availability of resources has to be secured. Like for any other product recycling is a cost efficient and sustainable way to reduce the need for constant flow of freshly mined resources. New promising recycling methods can reach up to 91% recycling rate with utilization of processes under vacuum.

Battery production

Fundamental research – Electrode coatings

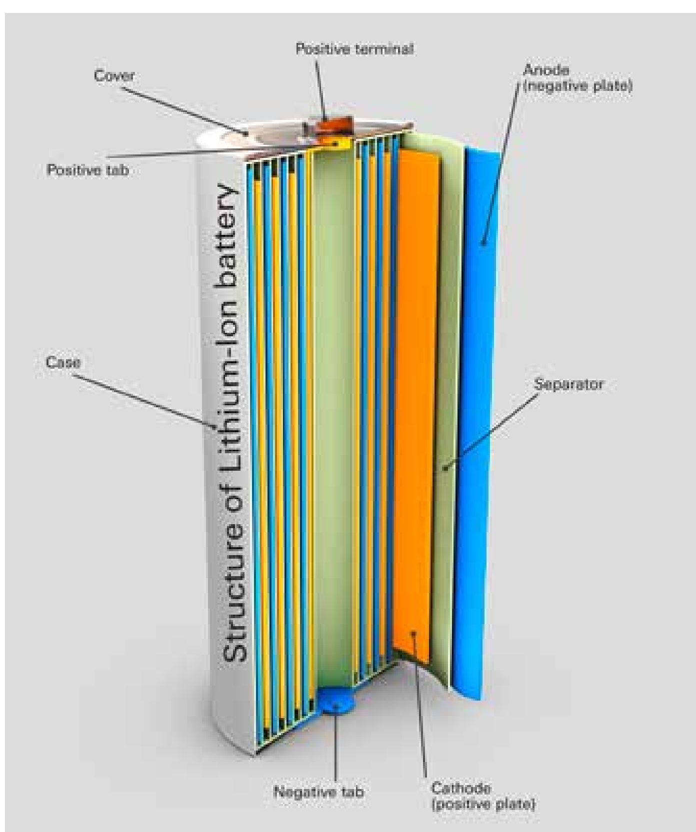
Basics

In principle, each battery is composed of a positive and a negative electrode, a separator and an electrolyte in between them. There are numerous materials for each of the components and even more combinations of them. What they all have in common is that the conductivity of the electrodes is crucial for the overall performance of the battery. This not only applies for conductivity of the bulk material but also for surface and surface boundary phenomena on the electrodes.

Coatings on electron collectors made of Aluminum or Copper are passivating the respective surface of the conducting material and protect the materials against corrosion. Thus the lifetime of the whole battery is prolonged. Furthermore, negative effects on the boundary layers between electrodes and their environment are reduced. This results in improved interfacial conductivity and enhanced battery performance.

Requirements

The technology used to apply this surface modification is physical vapor deposition (PVD), e.g. sputter deposition technology. Material is deposited from a material source, the „target", onto the electrode, the "substrate". Sputter coating is operated at low pressures to prevent contamination of the deposited layer with particles or unwanted elements contained in ambient air. For the former, the substrate surface, i.e. the electrode surface, can be cleaned by different measures, like plasma etching in vacuum, to create a pure surface for best electrical performance. To maintain the inert surface low pressures offer low contamination probability.



Composition of a cylindrical Li-ion cell

Complete solution

Low level vacuum for pre-cleaning the surface to be coated and process vacuum is generated by Pfeiffer Vacuum turbo molecular pumps with perfectly matched backing pumps. The complete Pfeiffer Vacuum product portfolio comprises also vacuum chambers, gauges, valves, fittings, connection pieces and all other accessories needed for construction of a perfect coating solution.

Research on Electrode Coatings

Product overview

Turbopump HiPace	Screw pump HeptaDry L	Roots pump OktaLine
		
■ The HiPace turbopump is a robust yet economical solution ■ High suction speed for light and heavy gases ■ High process capability, robust against particles ■ Maximum operational safety through monitoring of operating data ■ Extensive range of accessories	■ Suction speed up to 950 m^3/h ■ Compact design ■ High pumping speed at atmospheric pressure -> fast pump down, high output ■ Most robust and durable pump design: ■ Low speed, contactless seals, standard industrial motor ■ Low overall cost: Low energy consumption, long maintenance intervals, no cost for oil or filters	■ Suction speeds available: 145 – 27.400 m^3/h ■ Short pump down time through high compression ratio and overflow valve ■ Low operating costs due to air cooling ■ Optional: wear free magnetic bearing increasing maintenance intervals and no unplanned downtime due to oil leaks.

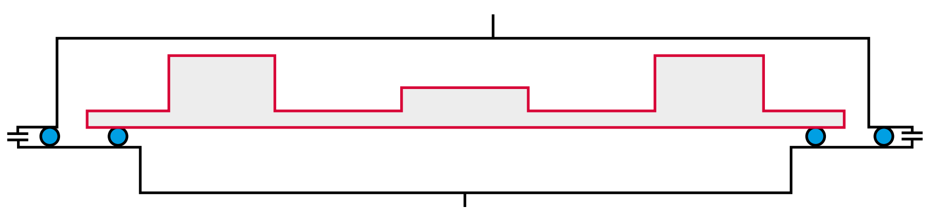
Battery production

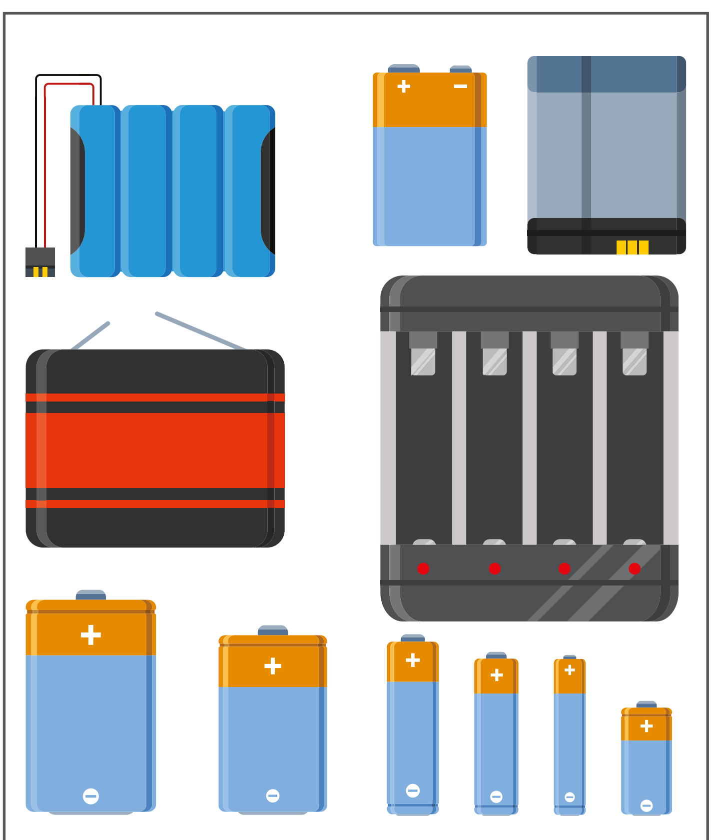
Fundamental research – Battery housings materials

Basics

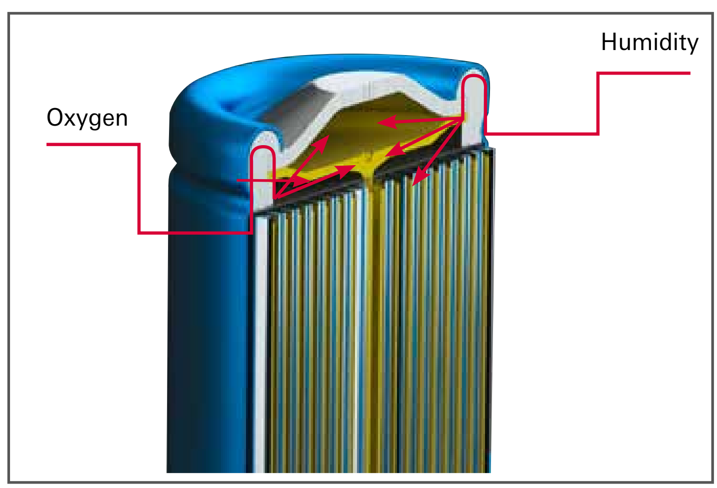
Many important functions of a battery are integrated in the top covers of prismatic and cylindrical battery housings: electrodes and their electrical insulation against the housing, an overpressure safety function and the hole for electrolyte filling. Tightness of each individual functional component plays a crucial role for the overall performance of the battery. If the tightness level does not meet the technical requirements during the product lifetime, ingress of humidity and oxygen may result in inacceptable fast degradation of the battery's electrical performance. On the other hand, the electrolyte which could contain fluorine may react with the incoming gases and form hazardous chemicals like hydrogen fluoride, or could inflame.

Permeation testing – Schematic set-up





Overview about different types of battery housings



Ingress of humidity and oxygen through the housing of a cylindrical cell

Ingress of humidity and oxygen through the housing of a prismatic cell

Requirements

Tightness also means low permeation, i.e. humidity and oxygen transfer through solid materials. The non-conducting materials of the battery cell play an important role in the overall tightness requirement of the battery. Permeation through either elastomer or glass materials must be so low that gas intake can be neglected over the lifetime of the battery.

The lifetime cycle of a consumer type electronics device like a smartphone is roughly two years. The functionality of the mobile device over this average period of time then is determining the tightness specification for a battery, housing, or permeation of materials.

The functionality over this period of time is not sufficient for e-mobility applications where a lifetime of 15 years is required. Consequently, materials used for mobility applications need to meet stricter quality demands.

Complete solution

Pfeiffer Vacuum’s quadrupole mass spectrometers and GSD gas analysis systems offer a perfect solution to measure gas transfer characteristics of the sealing materials with any desired gas. In a second step correlation between gas transfer of the respective materials and the tracer gas used in serial testing can be determined in order to develop an industrial serial testing recipe.

The robustness of the analytical devices and versatility of mechanical interfaces allows for precise integration into existing test benches like climatic chambers for test of temperature change resistance or testing after interval investigations like a salt spray test.
Data interfaces allow for transfer and storage not only of mass spectrometry data but also other sources in a central data base. Access to data and data analysis is massively facilitated for fast time-to-market of the product under development.
Industry 4.0 starts on R&D level already!

Product overview

Gas analyzer
GSD 350

- Qualitative and quantitative gas analysis
- Low detection limit even for condensable gases
- Heated capillary inlet, up to 350 °C
- Bakeable all-metal sealed high vacuum chamber for low backgrounds
- Customized versions possible, e.g. pressure controlled inlet, special inlet systems, special housing designs
- Soft ionization option (15 – 100 eV)
- Compact and portable
- Low gas consumption
- Fast measuring time

Battery production

Mixing

Basics

Mixing involves combining two different starting materials with the aid of a mixer to form a so-called „slurry“. The process is divided into two steps. In the first step, the active material is mixed with additives and binders. In the second step, the solvent is added and the solution is mixed until a homogeneous slurry is formed. The mixing temperature varies between 20 °C and 40 °C. The slurry is prepared for each electrode. This results in a different formulation for anode and cathode. To prevent air inclusions , the mixing step should be performed under vacuum. The larger the slurry batch, the more difficult it is to prevent air inclusions. For large batches, the use of vacuum is therefore recommended. This requires pressures above the vapor pressure of the solvent.

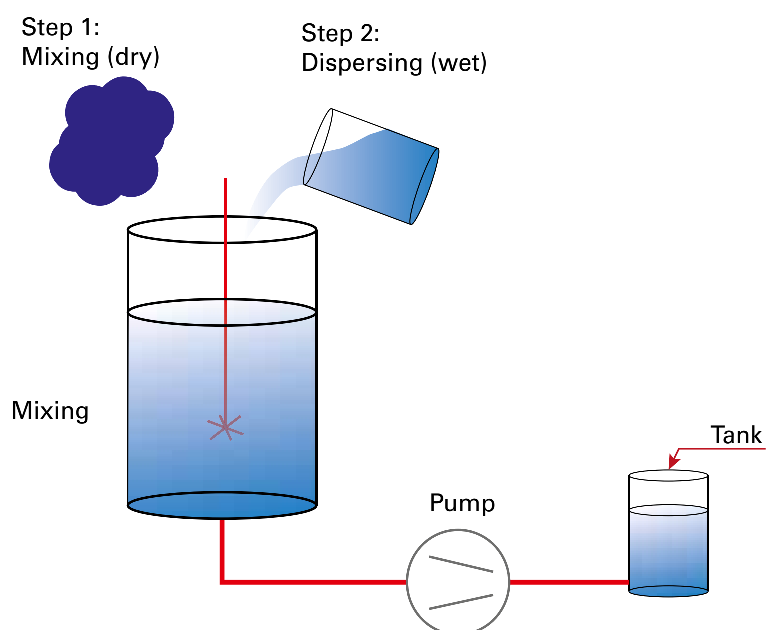
Requirements

If possible, the vacuum should be generated by a dry pump in order to avoid the entry of operating fluids into the process. If an oil-lubricated pump (e.g. rotary vane pump) is to be used, it can be equipped with an oil mist separator, which uses individual filter elements to prevent oil vapors from entering the process.

Mixing of the slurry under vacuum

Complete solution

Pfeiffer Vacuum can provide dry and oil-lubricated pumps to generate the vacuum in order to avoid air inclusions inside the slurry. With our dry screw pump series and our new dry scroll pumps, no operating fluid can enter the process. Also our oil-lubricated single-stage rotary vane pumps come with an equipped oil mist filter to prevent oil vapors for entering the process.



Mixing process - steps

Product overview

Screw pump HeptaDry	Rotary vane pump HenaLine	Scroll pump HiScroll
		
■ Highest pumping speed available ■ Fast evacuation of large chambers ■ Shortest cycle time ■ Superior reliability and robustness	■ Clean exhaust air through integrated oil mist separator ■ High energy efficiency thanks to intelligent design and optional energy recovery through cooling water ■ Maximum reliability as well as process reliability ■ Low maintenance costs thanks to direct drive and long oil change intervals	■ Fast evacuation of small chambers ■ Easy integration ■ Intelligent monitoring ■ Short cycle time ■ Superior bearing lifetime ■ Best cost / performance ratio

Battery production

Vacuum drying

Basics

The materials used to coat the anode/cathode electrodes show a pronounced hygroscopic behavior. The sorption equilibrium curves, e.g. for the anode, show loads of several 1000 ppm depending on the ambient humidity. Processing in the drying room cannot ensure that the moisture loading of the coatings remained below the tolerable values (< 300 ppm). Excessive moisture loading has a negative effect on the number of charging cycles, the loading capacity and, thus, on the lifetime of the cells.

This makes it necessary to dry them several times during their production process.
Typical examples are:

- Anode/cathode/separator coils are dried before cutting to sheets.
- Prismatic cells receive a post-drying before the actual electrolyte filling

Requirements

Not only the residual moisture content, but also a gentle after-drying with low structural changes of the layers at the micro level are essential for good electrochemical performance. Especially the binder used for coating the electrode bands (CMC at the anode and PVDV at the cathode) show brittleness and thermal creep at too high drying temperatures, which leads to deterioration of the coating’s structure.

Therefore, the use of vacuum technologies allows optimum drying at low temperatures (80 to 100°C) and at base pressures below 1 mbar to achieve the highest quality of the cells.

Drying processes

The electrode bands are cut into several small bands and afterwards rolled up to coils. These are stored and dried in tunnel ovens acting simultaneously as transfer chambers between the clean room of electrode band production and the dry room of cell assembly.

A typical process consists of alternation between the following steps:

1. Heating up the coils with hot air
2. Intermediate vacuum pressure reduction
3. Fine vacuum phase with infrared heating of the coils providing the necessary evaporation heat under vacuum conditions.

The final drying of prismatic cells, before electrolyte filling, is carried out in a three-stage inline drying chamber: lock-in and preheating chamber, final drying chamber, and cooling down and lock-out chamber. The drying process is similar to the aforementioned drying process, mainly consisting of heating up with hot nitrogen (up to 100°C) with alternating intermediate pressure reduction.

Complete solution

Pfeiffer Vacuum can supply vacuum pumps for evacuation to intermediate and fine vacuum levels, pressure measurement gauges to control the pressure within the oven as well as valves, flanges and components to connect the different vacuum components.

Suitable for these processes are two stage vacuum pump units consisting of Roots pump and dry screw pump. The standardized multi-stage Roots pumping stations consisting of these pumps are the CombiLine series WH (OktaLine) and RH (HiLobe) with a wide range of suction capabilities and final vacuum down to $5 \cdot 10^{-3}$ hPa.

In case the right pumping station is not within the standard portfolio, Pfeiffer Vacuum can assist with customer-specific vacuum solutions adapted specifically to a customer's production process.

For pressure control, Pfeiffer Vacuum's portfolio offers a wide range of analogous and digital gauges (DigiLine, ActiveLine, CenterLine and ModulLine) with varying measurement principles.

Product overview

Pumping station CombiLine RH

- Oil-free pumping system:
- HiLobe Roots pump and HeptaDry pump
- Fast evacuation of medium and big chambers
- Superior performance in heavy cycling
- High uptime and long maintenance intervals

Pumping station CombiLine WH

- Oil-free pumping system: OktaLine Roots pump and HeptaDry pump
- Mounted and piped together on a robust common framework
- Optionally, each pumping unit is also available with switch cabinet and PLC

Vacuum gauges ActiveLine PCR 280 / DigiLine RPT 200

- Measurement range
- $1 \cdot 10^{-4}$ – 1.200 hPa
- $5 \cdot 10^{-5}$ – 1.500 hPa
- Two different sensors in one housing
- Independent from gas

Battery production

Electrolyte filling

Basics

The liquid electrolyte enables Li-ion transport between the electrodes. The wetting degree has a major influence on battery performance since non-wetted sections of electrodes and separator do not contribute to electrochemical reactions. In addition, on un-wetted sections needle-shaped structures can be formed on the electrodes which penetrate the insulating materials and may trigger safety issues due to short-circuiting adjacent electrodes.

Requirements

In production, the electrolyte filling process dispenses a defined amount of electrolyte into the battery within a cycle time determined by the overall process. During dispensing of the electrolyte the cavities of the stacked or wound cell are not completely filled. The optimum distribution of the electrolyte during the filling process has a major influence on the following step of "wetting" and requires a fast effective penetration of electrolyte into the porous media of the battery as it represents a very costly bottleneck in cell production.

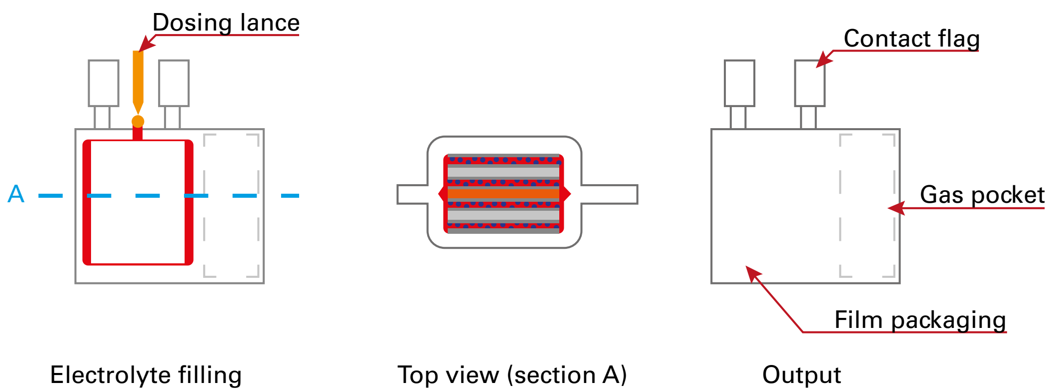
The duration of the wetting procedure can be influenced by the electrolyte filling step. In many cases the amount of gas inside the cell is reduced by evacuation prior to filling. The evacuation process can be optimized by consecutive evacuation / venting sequences (pressure swing cycling) and the venting gas can be used to tailor residual humidity inside the cell. Thus an optimized filling process has the potential to significantly improve product quality and at the same time reduce energy cost and overall process time.

There is no universal best practice for an electrolyte filling process, i.e. a broad range of process requirements and equipment specifications exists. For the evacuation and refill steps either precise timing recipes or pressure-dependent processes may be run. The geometry of the filling holes in the respective cells and cell types defines a conductance limitation to the evacuation process. This influences the available base pressure regime and, thus, the selection of the pumping solution.

It is common to seal pouch cells inside the filling apparatus at low pressure. In contradiction, prismatic and cylindrical cells usually are sealed after the wetting procedure at ambient pressure.

Complete solution

Pfeiffer Vacuum can supply single vacuum pumps or pumping stations, both able to reach desired pressure levels in the required time. All product ranges are available as dry solutions to prohibit the introduction of undesirable hydrocarbons into the battery housing. Suction capability, base pressure, timing requirements, media tolerance and the ability of alternating pressure at elevated levels determine the selection of the appropriate product family.



Electrolyte filling of a pouch cell under vacuum

Product overview

Mult-stage Roots pumps ACP / A 200 L	Scroll pump HiScroll	Screw pump HeptaDry
		
■ Fast evacuation of small and medium chambers ■ Superior performance in heavy cycling ■ Multiple gas ballast options ■ High uptime and long maintenance intervals	■ Fast evacuation of small chambers ■ Easy integration ■ Intelligent monitoring ■ Short cycle time ■ Superior bearing lifetime ■ Best cost / performance ratio	■ Highest pumping speed available ■ Fast evacuation of large chambers ■ Shortest cycle time ■ Superior reliability and robustness

Battery production

Formation / Degassing

Formation

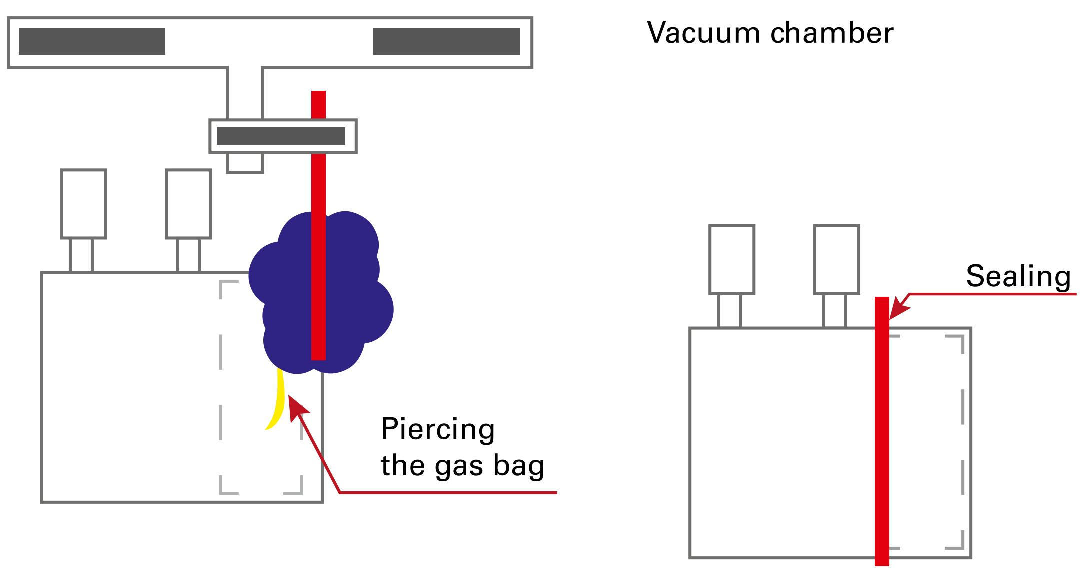
During formation, the charging and discharging process takes place for the first time. For this purpose, the cells are clamped in devices and contacted by means of spring contact probes. Once contact has been made, the cell is charged and discharged according to precisely defined current and voltage curves. This process step represents the core knowledge of battery manufacturers. The individual parameters for the charging and discharging process largely determine the subsequent cell performance.

Degassing

Degassing is mainly used with pouch cells, as strong gas generation often occurs during the first charging process. These gases are pressed by product carriers into the so-called „gas pocket" provided for this purpose. Afterwards, the gas pocket is pierced under vacuum and the escaping gases are extracted at a pressure of approx. 100 mbar. The gas pocket is then separated by sealing the area between cell and pocket, and disposed of as hazardous waste. Before the extracted gases can be fed into an exhaust air system, they have to be treated in accordance with occupational safety and environmental regulations.

Requirements

To create the vacuum, dry screw pumps in particular, as well as dry Roots pumps or rotary vane pumps can be used. The pumps should only be resistant to toxic vapors.



Degassing of a pouch cell under vacuum

Complete solution

Pfeiffer Vacuum can supply single vacuum pumps or pumping stations, which are able to reach desired pressure levels to guarantee degassing. In case the right pumping station is not included in the standard portfolio Pfeiffer Vacuum offers customer-specific vacuum solutions that are specially adapted to the customer's production process.

Smartphone battery

Product overview

Pumping station CombiLine RH

- Oil-free pumping system:
- HiLobe Roots pump and HeptaDry pump
- Fast evacuation of medium and big chambers
- Superior performance in heavy cycling
- High uptime and long maintenance intervals

Pumping station CombiLine WH

- Oil-free pumping system: OktaLine Roots pump and HeptaDry pump
- Mounted and piped together on a robust common framework
- Optionally, each pumping unit is also available with switch cabinet and PLC

Screw pump HeptaDry

- Highest pumping speed available
- Fast evacuation of large chambers
- Shortest cycle time
- Superior reliability and robustness

Rotary vane pump HenaLine

- Clean exhaust air through integrated oil mist separator
- High energy efficiency thanks to intelligent design and optional energy recovery through cooling water
- Maximum reliability as well as process reliability
- Low maintenance costs thanks to direct drive and long oil change intervals

Battery production

Leak detection – End-of-line test

Basics

After final assembly of all individually manufactured, assembled and tested parts the outcoming product has to pass a scope of final tests – the end-of-line tests. Exemplary, for the car manufacturer this means to check whether the car is working and no potential safety issues exist as well as controlling that all customer features are installed according to order acceptance.

In battery production, the end-of-line test can be placed at various sub-part production line ends, like battery cell or battery housing, or on the final battery module manufacturing line.
In case of battery cells the end-of-line tests take place after the last production step, the aging of the cell. Afterwards, these are classified and have to withstand the end-of-line tests which are mandatory for quality assurance as all functionalities are thoroughly tested, including electrical testing and leak detection process.

Requirements

The goal of the end-of-line test is to secure a distinct level of quality for the tested products to guarantee a nominal life time of the battery pack in its final operating system, i.e. an electric vehicle or a stationary power storage. But the even more important goal is to prevent fatal accidents to protect the product users lives.

The test processes developed during prototyping are optimized to achieve very short cycle times. Further, it is (often) not possible anymore to alter the product, e.g. by connecting tracer gas inlet/outlet or to apply elevated differential pressures, as the product has to be tested in its final condition without receiving any damage.

Time is the key parameter in serial production in order to save costs and also to increase throughput of produced parts. Consequently, the accumulated duration of all tests has to be as short as possible and as long as necessary for the product to achieve the right cost-effort-ratio. One possibility to not only save time but to reduce probability occurrence of operating errors are automatable test systems.

Complete solution

Pfeiffer Vacuum's GSD gas analysis systems offer a perfect solution to measure gas transfer characteristics, e.g. from a leaking electrolyte, from the interior of the finalized product. In a second step correlation between gas transfer of the respective materials and the tracer gas used in serial testing can be determined in order to define the maximum tolerable gas transfer.

The capability to measure from atmospheric pressure down to single digit mbar range allows for various differential pressures and, thus, to utilize one end-of-line testing station for different products to be tested.

Data interfaces allow for transfer and storage not only of mass spectrometry data but also other sources into a central data base. This allows tracking results of uniquely identifiable parts and the gained results which is mandatory later for warranty issues and in case the final product experiences a malfunction.

Full-automated End-of-Line Test

Product overview

Gas analyzer
GSD 350

- Qualitative and quantitative gas analysis
- Low detection limit even for condensable gases
- Heated capillary inlet, up to 350 °C
- Bakeable all-metal sealed high vacuum chamber for low backgrounds
- Customized versions possible, e.g. pressure controlled inlet, special inlet systems, special housing designs
- Soft ionization option (15 – 100 eV)
- Compact and portable
- Low gas consumption
- Fast measuring time

Battery production

Leak detection – Battery cell housings

Basics

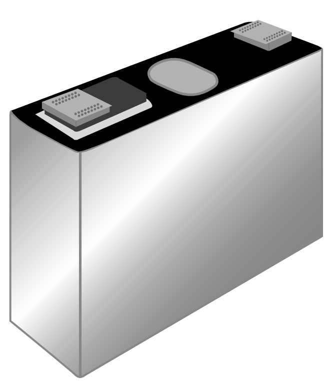
All types of battery cells require tightness on a level which allows for safe and reliable operation over the projected lifetime of the product. Battery covers are the assemblies in the battery which have the most joints which need to be leak tight.

Depending on the housing design and material, i.e. rigid cell or a flexible pouch, the available process parameters for thorough leak detection vary. Exemplary, a prismatic cell can be tested prior to final filling with pressurization of a tracer gas, e.g. Helium. On the other hand, a pouch cell will be sealed once the battery cell is manufactured and no inlets remain for tracer gas filling. Sometimes, during sealing of the pouch the surrounding atmosphere contains a strongly diluted tracer gas which can be detected afterwards. However, this unnecessary ingredient has unwanted influences on the cell chemistry and, thus, is more and more avoided in cell manufacturing.

Requirements

Consequently, the requirements on the equipment (and process) have to be differentiated between rigid and pouch cells. While the former allow for higher differential pressures during measurement the latter is very sensitive to pressure differences and will inflate and, finally, irreversibly become damaged.

Helium leak detection is the technology of choice for quantitative integral test of top covers of any kind. This is true for small consumer-type batteries as well as large batteries filling half the width of a battery pack in an electrical vehicle. Test equipment allows for fast cycle times and high sensitivity.
However, tracer gas may accumulate in insulating materials of battery covers which may result in an inacceptable high signal background during leak testing. Leak testing with air can be used in such applications compromising with respect to sensitivity but allowing higher flexibility of the process.



Prismatic cell

Cylindrical cell

Pouch (coffee-bag) cell

Detection of escaping materials from a pouch cell can be achieved by total flow measurements or quadrupole mass spectrometry. Both concepts first need the material to exit the unit under test which can be accelerated by application of an external driving force, like a pressure difference. On the one hand, the total flow measurements do not differentiate between the particles, e.g. one humidity atom will have the same influence like a solvent atom, and thus requires less knowledge about the total system situation.

On the other hand, mass spectrometry requires deeper knowledge about the composition of escaping material and of the system parameters during detection as the gained mass spectrum changes fast with changing atmosphere. Both techniques offer different advantages and at the same time challenges

Complete solution

Pfeiffer Vacuum's line of products offer the full range of described detector technologies. The modular leak detector ASI 35 offers optimum integration into industrial leak detection systems as well does the Micro-Flow based air leak tester E-PDQ. Fast response times allow for short cycle times and high throughput. Accuracy and reproducibility of test results ensures high quality production.

Not only a detailed analysis of the escaping species is possible with the quadrupole mass spectrometer Prisma Pro but in combination with a calibrated leak also the test if the specified leak rate is passed or not.

Product overview

Integral leak detector E-PDQ	Helium leak detector ASI 35	Mass spectrometer PrismaPro
		
■ Quantitative measurement ■ No specific tracer gas needed	■ Modular leak detector for vacuum and sniffer testing. ■ Test gas: helium or hydrogen ■ High sensitivtiy, reliabilit and reproducibility ■ Small and compact dimensions	■ Quadrupole mass spectrometer ■ High speed, stability and resolution ■ Wide mass range ■ (1-300 amu)

Battery production

Leak detection – Battery cooling

Basics

Cooling is vital for the functionality and long-term operation of power electronics, battery modules, and battery packs. Both air cooling and liquid cooling systems are used in battery applications. Leak tightness of the liquid cooling system prevents short circuits and ensures environmental protection in case a refrigerant is used as cooling medium in combination with the AC system of the vehicle.

Requirements

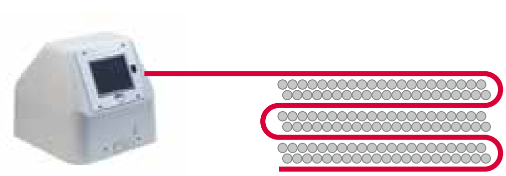
The cooling method used in the respective component of the battery determines the technology used for leak detection. In case of water-glycol cooling mainly leak testing with air is used. This quantitative integral method can be complemented by a localizing technique with tracer gas if the leak shall be found for a corrective action.

For water cooling, specifications can be transferred from mature and field-proven test recipes from water cooling in other automotive applications. Often the PASS / FAIL criterion is in the range of 0.1 … 1 sccm.

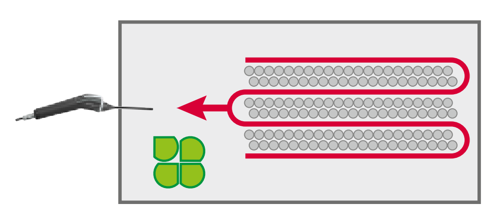
Specifications for refrigerants as a coolant are driven by environmental regulations. The requested sensitivity makes it necessary to use a method with a lower detection limit than leak testing with air. A tracer gas method has to be applied. The tracer gas can be Hydrogen (diluted to 5% as “forming gas”) or Helium. Methods for quantitative measurement are e.g. accumulation under atmospheric conditions or integral measurement in vacuum, depending on the required sensitivity and cycle time. Again, the quantitative measurement can be complemented by a localizing sniffing technique.

Refrigerants are classified with respect to their greenhouse potential. An allowed mass loss rate according to regulations is converted into a pV loss rate and often is in the range of $5 \cdot 10^{-6}$ … $5 \cdot 10^{-5}$ mbarl/s. The exact reject limit also depends on the process recipe including type of tracer gas, concentration of tracer gas, and test pressure.

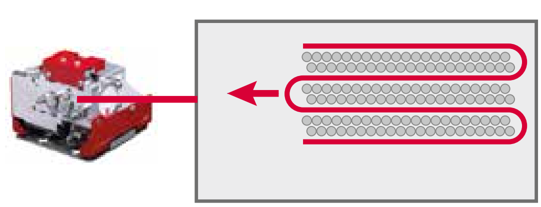
Cooling systems – test methods



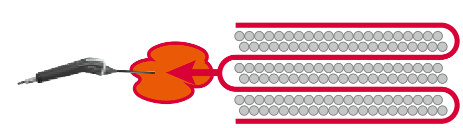
Micro-flow leak testing



Accumulation



High vacuum



Tracer gas sniffing

Complete solution

Pfeiffer Vacuum is the only supplier in the world who manufactures test equipment for both leak testing with air and with specific tracer gases. So whatever the application is, our application team will select the optimum test method, whether it be micro flow, tracer gas leak testing, or a combination of both.

Battery pack with cooling system

Product overview

Integral leak detector E-PDQ	Helium leak detector ASM 340	Helium leak detector ASM 306 S
		
■ Quantitative measurement ■ No specific tracer gas needed	■ Universal leak detector for vacuum and sniffing mode ■ Leak location and measurement ■ Tracer gases Helium and Hydrogen	■ Leak detector for sniffing and accumulation testing ■ Leak location and measurement ■ Tracer gases Helium and Hydrogen ■ High flow allows fast detection of leakages

Battery production

Leak detection – Battery pack

Basics

The battery pack incorporates several components which have gone through leak testing in the previous assembly process. This is true for battery cells and modules as well as cooling channels. In the final battery pack testing, often cooling channels are tested again in order to detect any potential damage during the assembly process.

Additionally, the battery pack housing is tested. The specification for this component often is derived from a phenomenological ingress protection class which ranges from temporary soaking under water simulating a defect car in a tunnel full of water up to exposure to a high pressure water jet simulating a cleaning process. But not only battery cells dedicated for automotive applications are tested with respect to ingress protection class. Exemplary, batteries are also part of smart wearables which partly are promised to endure several minutes under water to cope for swimming, rainy weather or the simple fall into a sink.

Requirements

Experimental work has been made in order to evaluate a correlation between the respective ingress protection class (often IP X7 up to IPX9K) and a leak rate in a defined test recipe. The model used for determination of the correlation must represent the real geometry of an assumed leak scenario. The geometry of a defined leak must be representative for the most probable scenario of a real life defect. This means that material combination, ingress direction, and most probable leak geometry should be taken into account in the model in the ideal case. Unfortunately, many calibrated leaks cannot simulate the real defect since they are not available with the real material combination. For that reason glass capillaries are the best compromise available for channel-shaped geometries. For orifice-shaped geometries so-called "equivalent diameter" leaks are available

Complete solution

A complete solution often is a compromise between technical and economical parameters. Any leak test should be performed with a pressure gradient simulating the real life operation of the part to be tested. For the battery pack this means an outside-in test method.

Leak testing with air offers the pressure gradient as in real life use of a battery pack. However, this often is a compromise with respect to detection limit.

Method	Sniffing	Accumulation	Vacuum test outside-in	Vacuum test inside-out	Micro flow test outside-in
Test direction	inside → out	inside → out	outside → in	inside → out	outside → in
Detection limit of $1 \cdot 10^{-6}$ mbarl/s	Challenging at ambient pressure, needs tracer gas discipline	In general yes Not achievable in given cycle time	Yes	Yes	No
Cycle time	Depending on detection limit, velocity and distance	Long	OK, depending on pump technology	OK, depending on pump technology	Depending on sample and environmental influences
Quantitative	No; needs calibration and correlation	Yes	Yes	Yes	Yes
Integral	Needs validation	Yes	Yes	Yes	Yes
Leak localization	Yes	No	No	No	No
Temperature influence	No	No	No	No	Yes
Volume influence	No	No	No	No	Yes
Cost					
■ Invest	Medium	Medium	High	High	Medium
■ Tracer gas	Medium	Medium	High	Medium	Low
■ Life time	Medium	Medium	Medium	Medium	Medium

Quantitative methods in vacuum with short cycle times often cannot be applied due to battery pack components like a pressure equalization element.

Tracer gas sniffing is used therefore transferring a method which is qualitative by definition to a quantitatively measuring application. Great care has to be taken with respect to tracer gas charging and a correlation of the measured signal with the real tracer gas emanation from the defect. In addition, tracer gas distribution inside the battery pack has to be performed carefully. Sniffing methods can be quantified using the accumulation technique. However, often this method does not meet cycle time requirements in production.

A comparison between the potential methods is given in the table above. Whatever the application is, Pfeiffer Vacuum can advise selecting the optimum method for individual battery packs.

Product overview

Integral leak detector VE2	Helium leak detector ASM 340	Helium leak detector ASM 306 S
		
■ Quantitative measurement ■ No specific tracer gas needed	■ Universal leak detector for vacuum and sniffing mode ■ Leak location and measurement ■ Tracer gases Helium and Hydrogen	■ Leak detector for sniffing and accumulation testing ■ Leak location and measurement ■ Tracer gases Helium and Hydrogen ■ High flow allows fast detection of leakages

Battery production

Recycling

Basics

To enable the shift from todays' mobility to electrical propulsion, resources like Lithium, Nickel and Cobalt have to be regained by recycling to serve the market demands in 2035. With an efficient recycling concept for circular economy the majority of material demand can be served by recycled resources. Today, conventional Li-ion battery recycling relies mainly on pyrometallurgical processes. But also mechanical, hydrometallurgical or combinations with pyrometallurgical processes rise in importance and technology maturity level.

Requirements

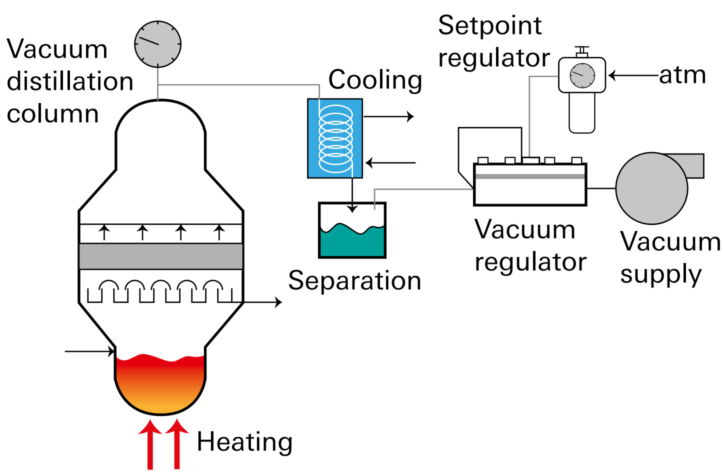
Pyrometallurgical processes on the one hand consume a lot of energy, e.g. furnace operation at 1500°C, which results in a unhealthy CO_2 footprint. On the other hand not all elements can be recovered, i.e. with an efficiency of 80% for Cobalt, Nickel and Copper.
The high temperature treatment of battery waste results in irreversible changes of organic components of the electrolyte. Toxic gaseous fluorinated chemicals and hydrogen fluoride are by-products of high temperature waste treatment. These components need to be removed from exhaust gases by elaborate methods like burning or absorption.
The hydrometallurgical process combines the mechanical separation of the battery components after shredding with the dissolution of the metals in acids and alkalis and their precipitation in the form of salts. The recovery rate of the raw materials used is approx. 30%.

Recycling rates of > 90°C are necessary for a successful circular economy. The first promising developments are based on vacuum technologies. In one approach, the batteries are shredded under an inert gas atmosphere. The resulting electrolytes are fed into a vacuum distillation system and recovered. The remaining solids are separated and processed using mechanical processes (magnets, screens, etc.). The recycling rate is 91%.

Transport of battery waste may be not desirable due to the need for special safety precautions during transportation. Mobile recycling stations which are transported to battery collecting centers are a safe and effective point-of-use alternative. The pre-requisite for such mobile recycling centers are pumping stations which are compact and require little energy.

Complete solution

With the HiLobe and Okta Roots pumps, Pfeiffer Vacuum offers an unrivalled compact solution for applications demanding high pumping speed and process tolerance. With 50 % space saving compared to traditional Okta Roots pumps, flexible installation, low weight and energy-efficient motors this range of Roots pumps is the ideal choice for pumping technology focused on environmentally friendly applications.



Scheme vacuum distillation with Roots pump

Product overview

Roots pump HiLobe	**Roots pump OktaLine**	**Rotary vane pump HenaLine**
		
■ Adjustable pumping speed allows for optimum adaptation to the process ■ Ultra-compact design, vertically and horizontally mountable ■ -> optimum integration ■ Intelligent condition monitoring – highest operational safety and energy cost saving ■ Leakage rate < $1 \cdot 10^{-6}$ Pa m^3/s – highest safety ■ Air cooling – no water media supply and cooling costs ■ Extended maintenance interval of 4 years – lowest operational cost	■ Suction speeds available: 145 – 27.400 m^3/h ■ Short pump down time through high compression ratio and overflow valve ■ Low operating costs due to air cooling ■ Optional: wear free magnetic bearing increasing maintenance intervals and no unplanned downtime due to oil leaks. ■ Optional: ATEX version	■ Clean exhaust air through integrated oil mist separator ■ High energy efficiency thanks to intelligent design and optional energy recovery through cooling water ■ Maximum reliability as well as process reliability ■ Low maintenance costs thanks to direct drive and long oil change intervals ■ Optional: ATEX version

Leak testing services

Individual services in leak testing technology for your applications

We provide you the following solutions

Pfeiffer Vacuum is providing complete vacuum solutions.
We offer products and technical assistance over the entire process. Further, we offer test procedure development and test implementation.
In close cooperation with or customers, we create customized solutions for your product. If you have components or final products, which should be leak tested, we can provide you feasibility studies or cycle-time studies as an all-in-one service in our application laboratory.

- Assistance in determining your leak rate requirements and conversion into a test recipe
- Technical support in selecting the optimal and safest leak detection solution for your application
- Wide range of solutions: Leak detection using tracer gas or air with special solutions for hermetically sealed products
- Integrated all-in-one solutions, including consulting and a network of partners for automated systems

Feasibility / cycle-time studies

In the course of a feasibility study we gain an understanding of the technical requirements of your product. This enables us to develop and apply a leak test procedure in industrial tightness control. This applies for new products prior to the market introduction or existing products with the need for cycle-time optimization. We work out an individual optimum solution for you to meet tightness requirements and respective regulations in detail.

Feasibility study for Container Closure Integrity Testing (CCIT)

Quality and effectiveness of drugs depends significantly on their proper packaging to maintain sterility throughout their lifetime. With a feasibility study we gain an understanding of your packaging and the CCIT capabilities. With our three CCIT-technologies we will confirm achievable detection limit and cycle time on your packaging.

Contract leak testing

When investment in leak testing instruments is not an economically meaningful expense, we are your choice by offering contract leak testing as a service.

Residual gas analysis

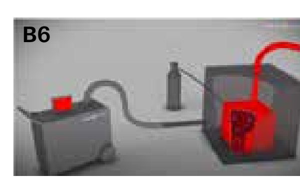
Residual gas analysis in vacuum systems and tightness control with a wide range of tracer gases like air components or electrolytes. The results are transferred to you in a detailed report.

We cover **15 different test methods** according to DIN EN 1779 / DIN EN ISO 20485 and we meet the requirements inside the USP 1207 with our 4 technologies.

Air leak testing	Tracer gas leak testing	Multi gas analysis/ Optical emission spectroscopy	Quadrupole mass spectrometry
Fast and reliable leak testing with air for packaging and electronics as well as for industrial and medical applications	**Highest sensitivity and fast leak testing for high end applications such as automotive, medical & semiconductor industry**	**Integrity testing for highly demanding pharmaceutical packages and advanced sealed devices**	**Residual gas analysis in vacuum systems and tightness control with a wide range of tracer gases like air components or electrolytes**
Pfeiffer Vacuum products			
 E-PDQ	 ASM 340	 AMI 1000	 PrismaPro
Test methods per technology			
D1: Pressure decay test, manually via gauge **D2:** Pressure rise test, manually via gauge **D3:** Pressure change test, bell pressure change, with overpressure, manually via gauge **D3:** Pressure change test, bell pressure change, with vacuum, manually via gauge **D4:** Flow measurement, **outside/in;** from higher pressure to vacuum; Mass Extraction / **inside/out;** from higher pressure to atmospheric; Micro-Flow	**A1–A3:** Helium leak testing, **A1** total vacuum technique, **A2** partial vacuum technique, **A3** local vacuum technique, **B2:** Vacuum box **B3:** Pressure technique by accumulation **B4:** Sniffing test **B5:** Pressurization – evacuation test, "bombing test" **B6:** Sealed objects by external vacuum technique, integral vacuum test **B7:** Carrier gas technique	**B6:** Sealed objects by external vacuum technique, integral vacuum test Detection of e. g. Air, Nitrogen, Argon, Helium, and many other gases	**B6:** Sealed objects by external vacuum technique, integral vacuum test Detection of any gases (1–200 u)

Examples of test methods

Integral test of parts under vacuum in helium



Integral test of helium filled parts in vacuum

Sniffing test

Micro-Flow (over pressure test)

Hint: **A1, B2, C3, D4...** = Abbreviation for test methods according to DIN EN 1779 / DIN EN ISO 20485

Pfeiffer Vacuum Service

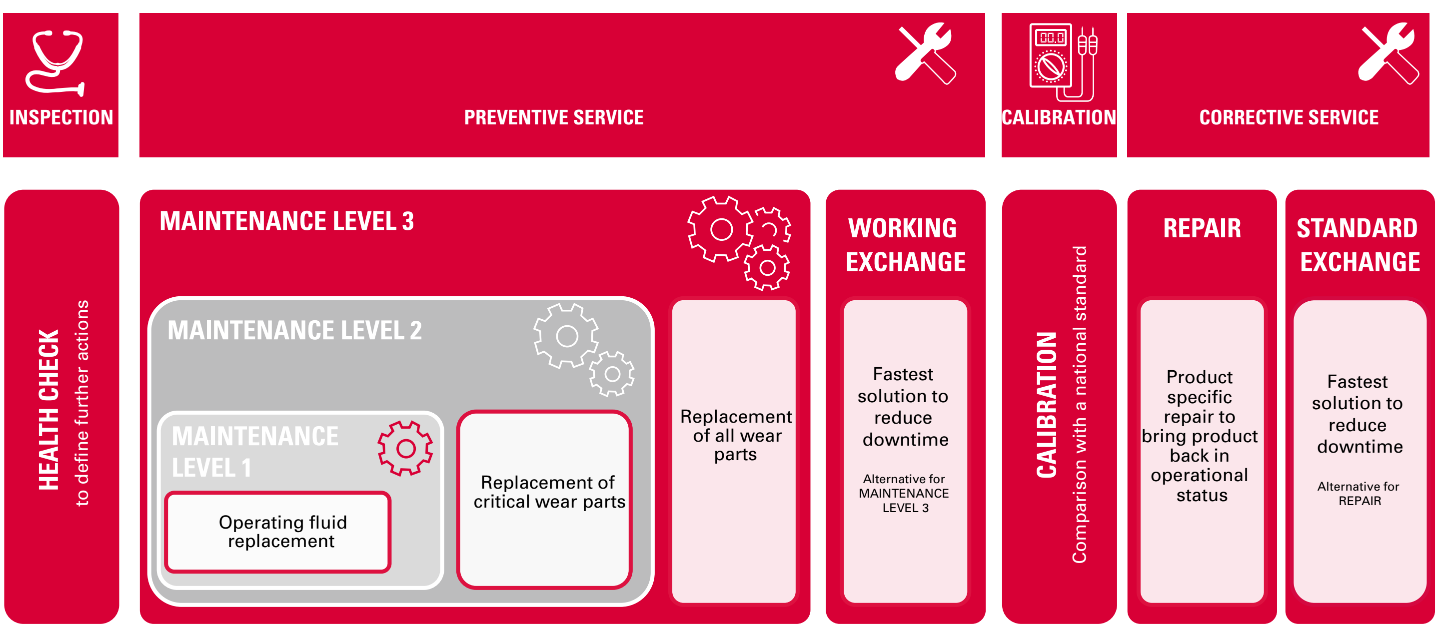
Our services – your advantages

Each customer places its own particular demands on its products, and these may also be influenced by application-specific parameters. Our flexible service concept, with a focus on preventive services, offers just the right solution for you.

Preventive maintenance – avoid downtimes

With our preventive service concept, we can recommend service intervals tailored to each product. The aim is to avoid failures and to carry out planned and predictable servicing.

Maintenance level 1 includes fluid changes and contributes significantly to the good working order of the product. **Maintenance level 2** also includes replacement of all wear and tear parts. In **maintenance level 3**, all wear and tear parts of the product are replaced and the product is overhauled. In order to keep downtimes to a minimum, we offer temporary replacements for many of our products for the duration of maintenance. We provide an equivalent **replacement product** that our customers can start using immediately.



Services at a glance

- User training and product training
- Pfeiffer Vacuum original spare parts and tools
- Troubleshooting and advice from our technical support team
- Comprehensive on-site service by our service technicians
- Maintenance and repair in our service centers worldwide
- Individual service agreements
- Replacement products
- Calibration service for measuring devices and helium test leaks

Spare parts – original parts increase life expectancy

Pfeiffer Vacuum's spare parts and tools are defined as early as at the product development stage. This ensures their tailor-made fit and quality.

Every improvement to our serial products is also transferred to our spare parts. This means products are brought up to state of the art status after undergoing maintenance level 3 or a repair.

Advice – to assist you with any questions you may have

In addition to our individual concepts and the quality of our replacement parts, it is our employees and personal contact that give our service its special touch.

Technical support – competent advice from the experts

Since not everything about our products is self-explanatory and questions can arise both before and after purchase, Pfeiffer Vacuum's Technical Support is available to assist our customers.

Each member of our team specializes in a specific area of our portfolio to enable them to assist our customers competently with technical questions relating to our products. Our team also works closely with our developers and application experts.

Field service technicians on site

From commissioning new vacuum components and systems to troubleshooting, and from maintenance to repairs, we offer our customers a comprehensive range of on-site services. Our service locations ensure customer proximity and short-term assistance in emergencies.

Service agreements – individually tailored to your project

We offer project-specific service agreements so that our customers can plan maintenance or service interventions over a long term. These agreements can be made at a later date or as early as during the project planning stage. In order to take our customers' differing needs into account, agreements may include all or just some of the services we offer.

Components and valves

The connection in your vacuum system

A vacuum system is made up of a variety of individual parts which are combined to form a single unit. Pfeiffer Vacuum also offers more than standard solutions. Components can be modified to meet your requirements or a customized solution can be produced to fit your needs perfectly.

Your advantages and benefits

- A direct contact for you and your projects
- Proactive support and competent advice
- Make ordering more convenient

- Short delivery times
- High delivery reliability
- High security of supply

- More than half a million parts in stock
- High uptime
- Cost saving - no own stock keeping necessary

www.vacuum-shop.com

- Vacuum components available in online shop
- Convenient online ordering at any time
- Information about your prices, delivery times and terms

Components

Valves

Feedthroughs

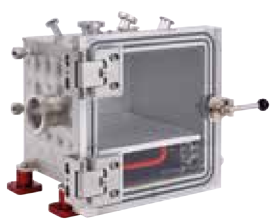
Manipulators

Custom vacuum chambers

Individually designed chambers for your vacuum applications

Due to our many years of experience we are familiar with almost all possible tasks and can provide professional guidance for system specifications, design and engineering.

Our physicists, designers, project managers and production specialists have extensive experience in many applications from all market segments. The tasks are based on your requirements: our starting point on the path to a finished product can range from a rough sketch to a complete set of blueprints.

High vacuum chambers	Advantages	Benefits
	■ Preconfigured design	■ Cost and time savings due to lower design expenses
	■ Proven, tough design	■ Reliable and safe
	■ Customized ports	■ Individual adaptation to your processes

Medium vacuum chambers	Advantages	Benefits
	■ Preconfigured design	■ Cost and time savings due to lower design expenses
	■ Proven, tough design	■ Reliable and safe
	■ Customized ports	■ Individual adaptation to your processes

Modular vacuum chambers	Advantages	Benefits
	■ Preconfigured design	■ Cost and time savings due to lower design expenses
	■ Modularly expandable	■ Maximum flexibility at all times
	■ Customized ports	■ Adaptable individually to your application

Custom vacuum chambers	Advantages	Benefits
	■ Individual design	■ Can be adapted optimally to your process
	■ High quality materials	■ Best quality and long life
	■ Robust design	■ Reliable and safe
	■ Project engineering and construction by qualified and experienced project managers	■ Time saving

VACUUM SOLUTIONS FROM A SINGLE SOURCE

Pfeiffer Vacuum stands for innovative and custom vacuum solutions worldwide, technological perfection, competent advice and reliable service.

COMPLETE RANGE OF PRODUCTS

From a single component to complex systems:
We are the only supplier of vacuum technology that provides a complete product portfolio.

COMPETENCE IN THEORY AND PRACTICE

Benefit from our know-how and our portfolio of training opportunities!
We support you with your plant layout and provide first-class on-site service worldwide.

Are you looking for a perfect vacuum solution? Please contact us:

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www.pfeiffer-vacuum.com

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