QUALITY ASSURED

APPLICATION NOTE

Hardness testing and analysis of Lithium-Ion batteries

Automatic micro hardness tester “Qness 60 A+ EVO”

A complete test for the performance of quality controls on different parts of a Lithium-Ion batteries (LIB).

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part of VERDER scientific

Introduction

Li-ion battery technologies have evolved significantly over the last three decades, from the first Li metal anodes to today's commercial Li-ion batteries. The history of Li-ion batteries is rich with breakthroughs and historic developments.

Technology is becoming more efficient, reusable primary and secondary batteries will be a key technology in the future. A corresponding inspection of the components is essential, where we as QATM would like to offer the best possible support for quality control.

A test scheme of analysis methods, e.g. grain size determination, layer thickness measurement and hardness test evaluation at different positions of a LIB sample, can be performed.

The test was carried out using a fully automatic Qness 60 A+ EVO micro hardness tester. With this device, the test cycles for the analysis and the hardness test can be carried out one after the other in the same workflow.

This method of working makes daily checks much easier for the operators, resulting in high-quality work.

Preparation of Lithium-Ion batteries

The preparation was carried out based on the steps listed below.

EQUIPMENT	SAMPLE HOLDER	PRESSURE MODE				
Qpol 300 A1 ECO+		Individual				
STEP	**MEDIUM**		**rpm**		**Single Pressure N**	**min**
Planar grinding	VEGA 54 µm	H_2O	SH: 120 WP: 200	►► Synchronous Rotation	25	Till flat surface (≈1:00)
Pre-polishing	BETA	Dia suspension Poly, 9 µm + Lubricant (alcohol-base)	SH: 120 WP: 150	►► Counter Rotation	25	5:00
Polishing	SIGMA	Dia suspension Poly, 3 µm + Lubricant (alcohol-base)	SH: 120 WP: 150	►► Synchronous Rotation	25	4:00
Final polishing	OMEGA	Eposil F 0.1 µm	SH: 80 WP: 100	►► Counter Rotation	30	1:30 (H_2O during final 0:30)
Etching (electrolyt.)	Klemm I 3% alcoholic nitric acid					40 s 5 s
Notes	SH: The rotational speed of the sample holder WP: The rotational speed of the working plate Pre-dosing time for diamond suspension 9 µm and 3 µm and lubricant: 3 s The dosing interval for dia. suspension 3 µm and 1 µm: each 30 s for 1.5 s The dosing interval for lubricant 3 µm and 1µm polishing: each 60 s for 1.5 s					

In conjunction with the application example of the preparation of Lithium-Ion batteries, the hardness test was subsequently carried out with corresponding optical analysis of the material.

Automatic micro hardness tester: Qness 60 A+ EVO

The QATM Qness 60 A+ EVO micro hardness tester is a combination of a classic micro hardness tester and a microscope for metallographic analysis. With a new design and high-end components, such as glass scales in all axes, high-resolution optical system and optimized motion sequences. In combination with the QpixControl2 software, there are no limits when it comes to mapping complex applications. This perfectly coordinated system impresses with its overall package and high level of automation.

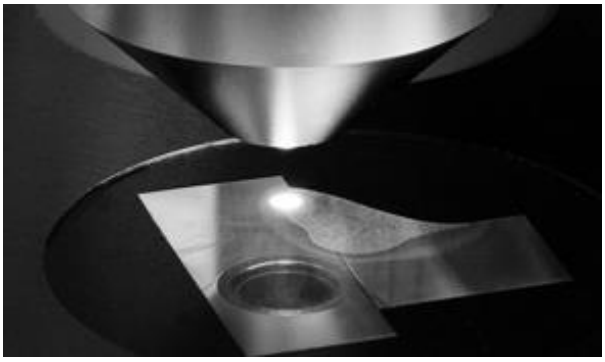
Key facts:

- Test load range 0.25g - 62.5 kg
- Automatic 8-fold tool changer
- Vickers, Brinell, Knoop test modules, easy to retrofit (Plug & Play)
- Motorized dynamic Z-axis height adjustment and automatic positioning system CAS (patented)
- Rotatable Knoop indenter - IPC (patented)
- Köhler illumination with motorized aperture diaphragm
- Inspection analysis functions directly integrated, Grain, Layer, Phase
- Working area and overview camera illumination
- Modern "State of the Art" operating software Qpix Control2
    - Auto-Snap, automatic alignment to component edge
    - Management of projects/specimens/test series
    - Reporting, export functions, QDAS standard
    - Cloud IoT connection, digital lab management

Highlights Qness 60 A+ EVO

REVOLUTIONARY OPTIC SYSTEM

Clear images for hardness testing and structural analysis. The Koehler illumination and color-corrected lenses provide ideal contrast, even at high magnification. The image quality compares to sophisticated microscopes, and the device meets strict test requirements according to DIN EN ISO6507-1/2.

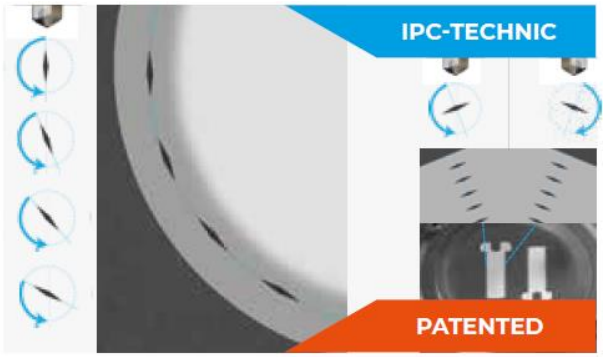


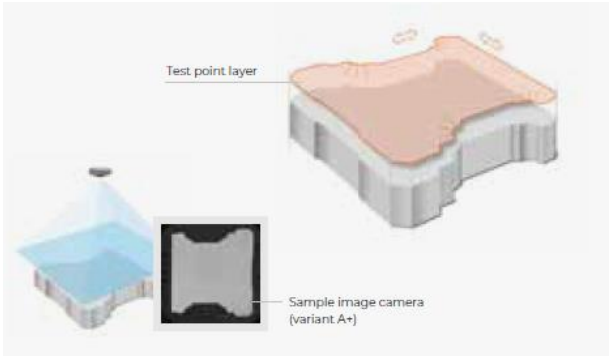
SAMPLE IMAGE CAMERA

Customers prefer QATM's 'A+' version with a built-in sample image camera. The camera captures the sample image quickly, aiding navigation in the software and enhancing documentation in the test report when combined with DOUBLE-VIEW TECHNOLOGY.

KNOOP IPC TECHNOLOGY

'Indenter Parallel to Contour', allows the operator to choose the route and points for the Knoop indenter along each contour fully automatically. The compact indenter unit has a built-in rotation drive, facilitating fully automated hardness testing in layers or along the edge of the workpiece.





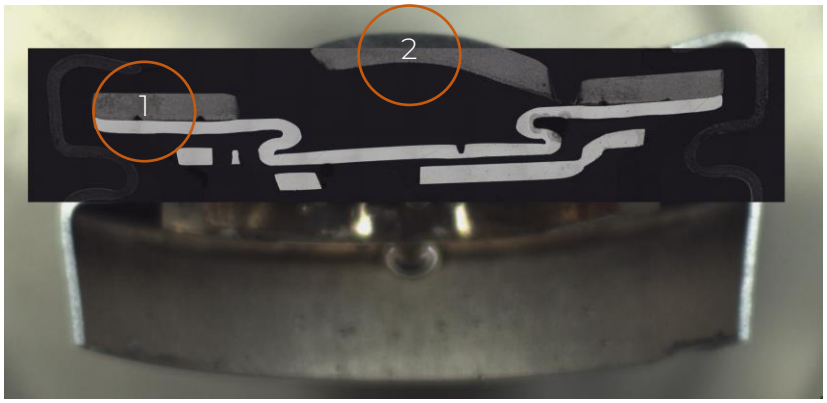
TEMPLATE FUNCTION

Is ideal for repeated tests on components. It aligns test point mappings on the workpiece with reference lines and benchmarks, without requiring a fixed stop or sample holder. The sample image can be included in the test report.

LIB - Sample A

A necessary quality control that can be carried out on the cap and housing of the LIBs is optical analysis of the material. In this example, the microstructure of sample A was analyzed at two different positions.

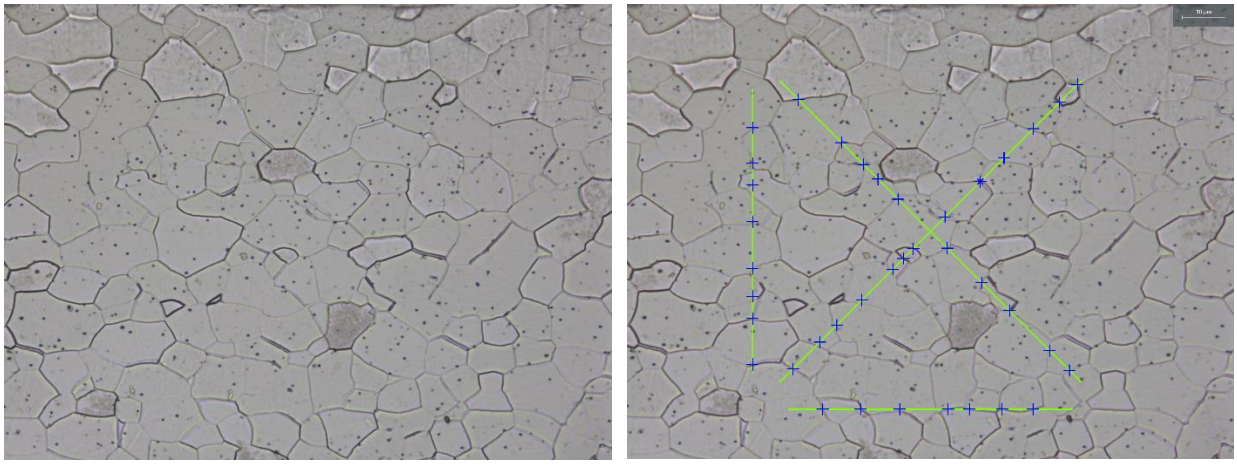
Overview image:



Grain size measurement:

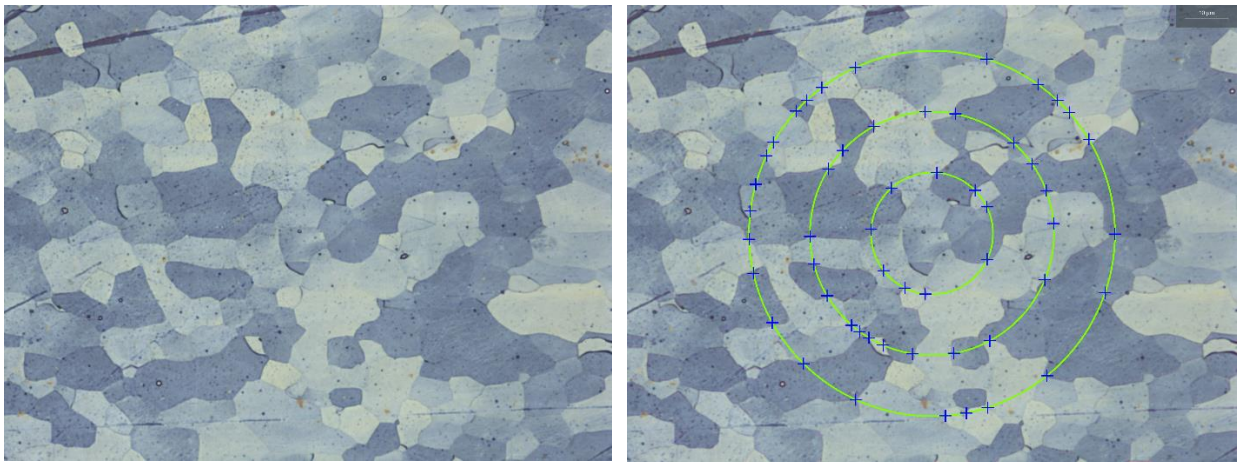
The grain size determination describes the size of the individual grains in a mixture. The grain size distribution has a significant influence on the material properties. Various methods can be selected to determine the composition of a batch depending on the grain size, which ultimately always determine an equivalent value. Which method is suitable depends on the grain size range or the regulations. A distinction is made between Heyn lineals and Abrams circles.

Grain size determination position 1, Heyn lineals:



Grain size: 10, evaluation with a 50x HQ lens

Grain size determination position 2, Abrams circles:



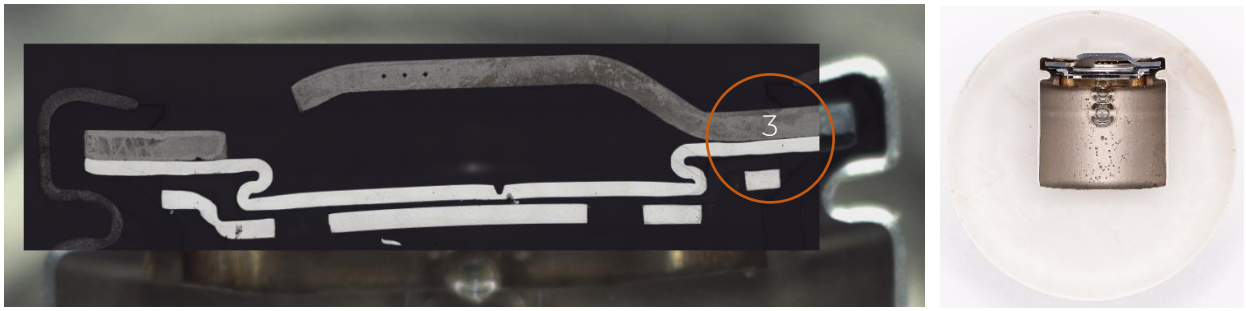
Grain size: 10, evaluation with a 50x HQ lens

LIB - Sample B

Other quality checks that can be carried out on the cap and housing of the LIBs include optical analysis of the material, layer thickness measurement and hardness testing with various options.

In this case, the indentations were applied parallel to the contour in a layer of <15µm. In addition to the minimum hardness range, indentations were made in the micro-hardness range.

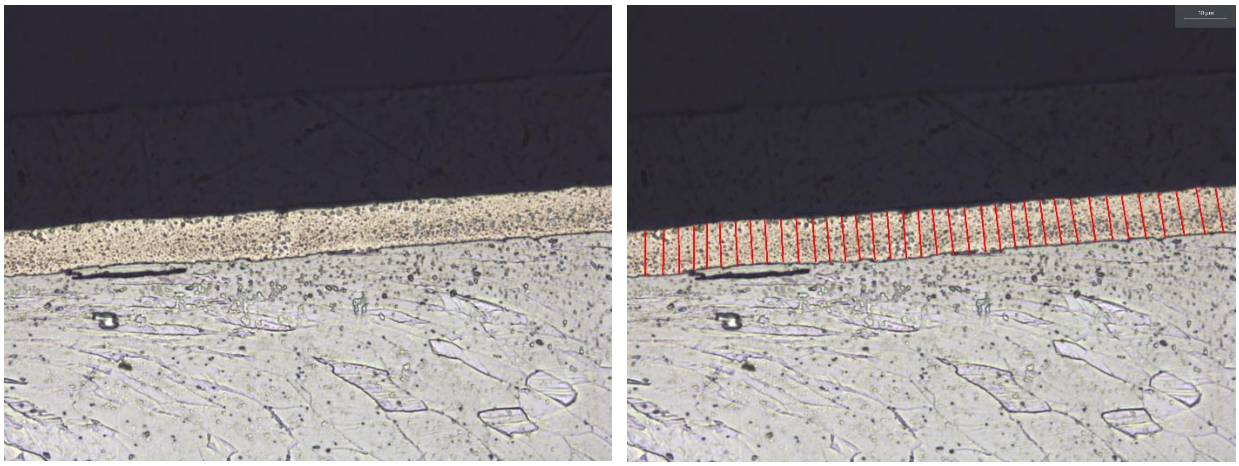
Overview image:



Layer thickness measurement:

Determination of layer thickness in accordance with DIN EN ISO 1463. Semi-automatic measurement of horizontal, vertical and radial layers.

Layer thickness measurement position 3:

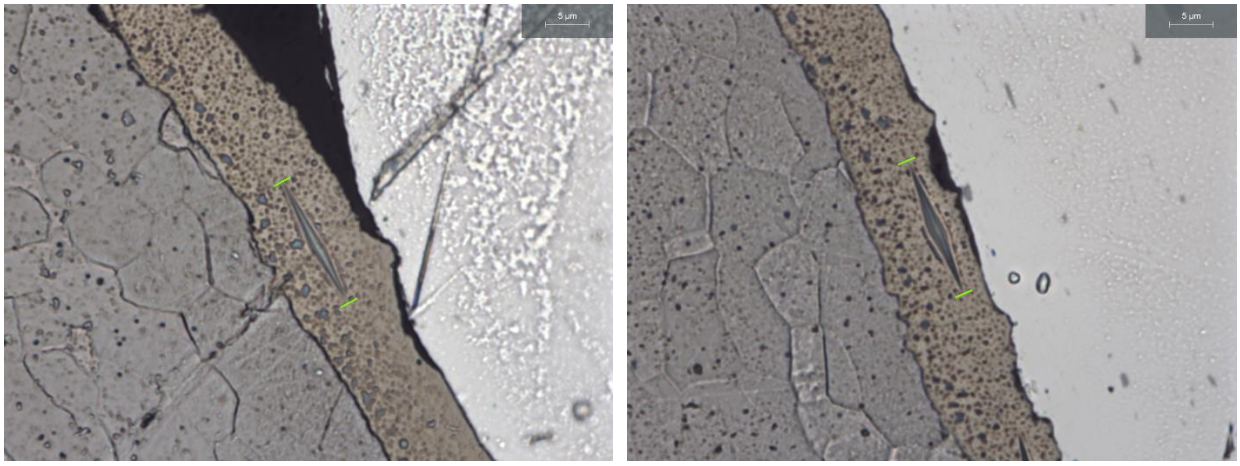


10.28µm layer, evaluation with a 50x HQ lens

Hardness testing:

Determination of the hardness in the layer, here the impressions were made with the Knoop IPC module. The alignment of the Knoop indentations was automatically aligned parallel to the contour.

Hardness test points directly in the layer, position 3:

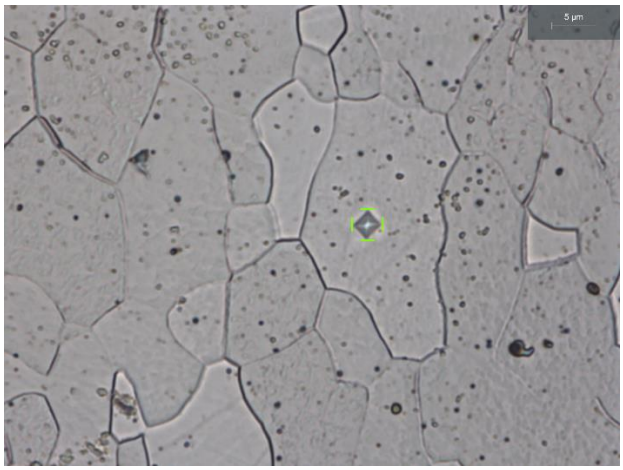


572 HK0.01 (10gf)

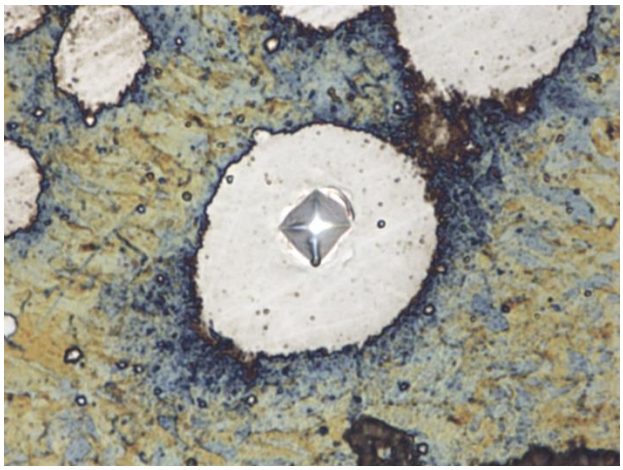
570 HK0.01 (10gf)

Evaluation with a 100x HQ lens

Individual hardness test points in a grain:



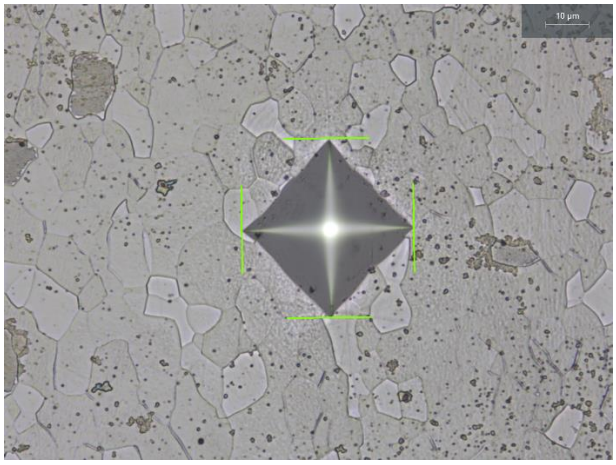
166 HV0.001 (1gf)



245 HV0.01 (10gf)

Evaluation with a 100x HQ lens

Surface hardness test:



114 HV0.1 (100gf), Evaluation with a 50x HQ lens