QUALITY ASSURED

APPLICATION NOTE

Hardness testing and analysis of Lithium-Ion batteries

Manual micro hardness tester “Qness 60 M EVO”

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

ATM Qness GmbH

Reitbauernweg 26 | 5440 Golling | Austria
www.qness.com | sales.at@qatm.com

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 manual Qness 60 M 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.

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.

Manual micro hardness tester: Qness 60 M EVO

The QATM Qness 60 M 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, automatic 8-fold turret with Plug & Play test modules, high-resolution optical system and optimized motion sequences. In combination with the digital X/Y slide and position feedback and QpixControl2 software, there are no limits when it comes to mapping complex applications. This perfectly coordinated system impresses with its overall package.

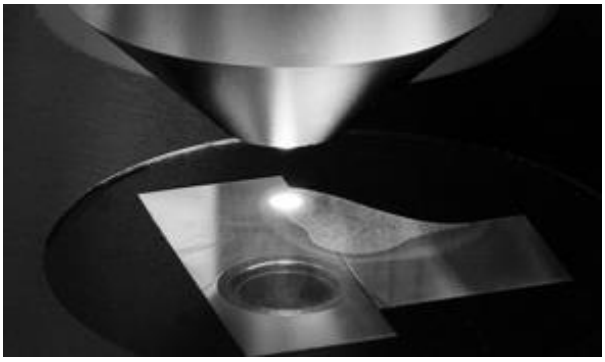
Key facts:

- 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 (patented)
- Köhler illumination with motorized aperture diaphragm
- X/Y stage with digital spindle and position feedback, e.g. for CHD
- Inspection analysis functions directly integrated, Grain, Layer, Phase
- Working area illumination
- Modern "State of the Art" operating software Qpix Control2
    - Management of projects/specimens/test series
    - Reporting, export functions, QDAS standard
    - Cloud IoT connection, digital lab management

Highlights Qness 60 M 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.

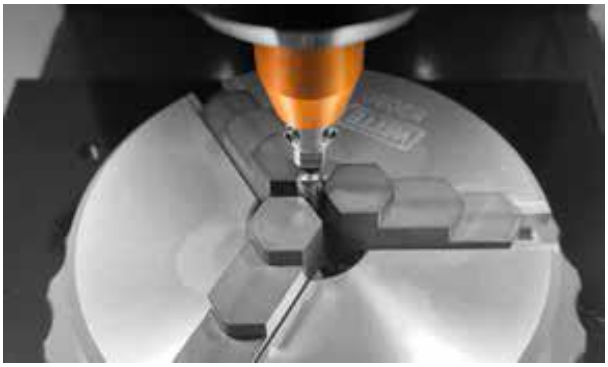


DIGITAL CROSS SLIDE WITH DATA FEEDBACK

Enables serial predefinition of test programs with fixed numbers of test points. If required, also with a manual slide, digital micrometer spindle and positional return – as used for manual CHD progressions.

COMPREHENSIVE RANGE OF BASIC FUNCTIONS

The base model offers several labor-saving features, including an optimized autofocus system, automatic brightness regulation, automatic image evaluation for hardness testing with multiple evaluation modes, and a built-in protocol generator.



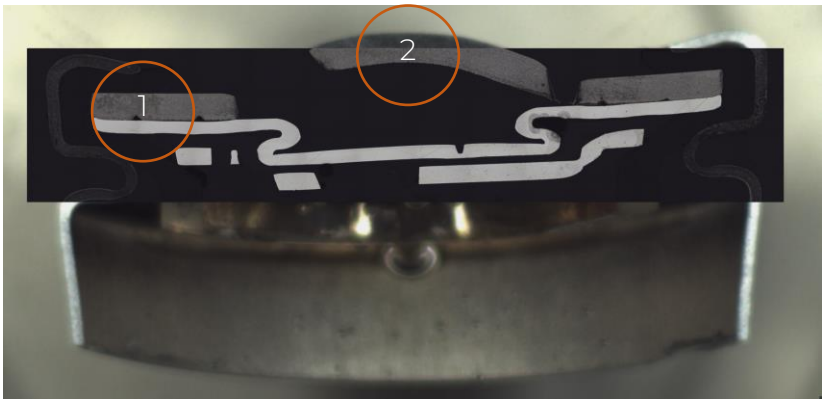
MAXIMUM VERSATILITY

Unsurpassed in single-piece testing and limited series tests on items of all sizes: Simple operability and optional additions for microscopy make QATM Qness 60 M EVO a unique, high-quality, all-round package.

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.

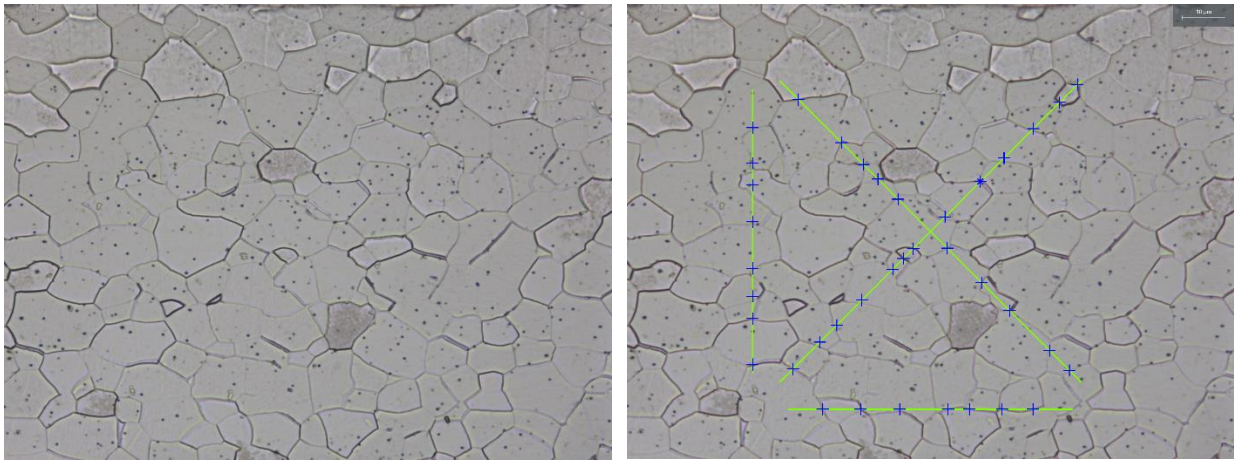
Sample image:



Grain size measurement:

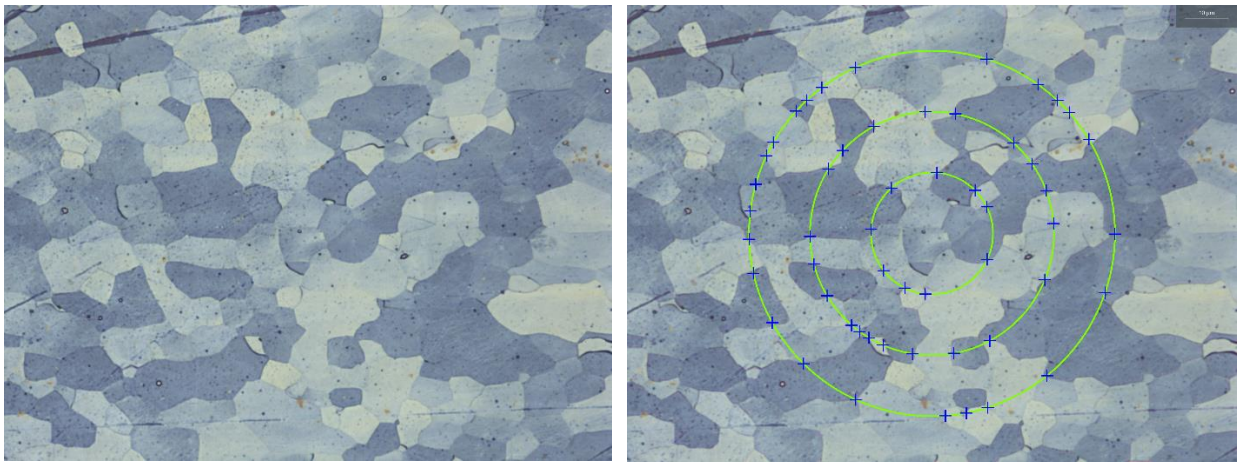
The grain size determination describes the size of the individual grains in a mixture. The grain size distribution has a significant influence on the material properties. Various methods can be selected to determine the composition of a batch depending on the grain size, which ultimately always determine 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:



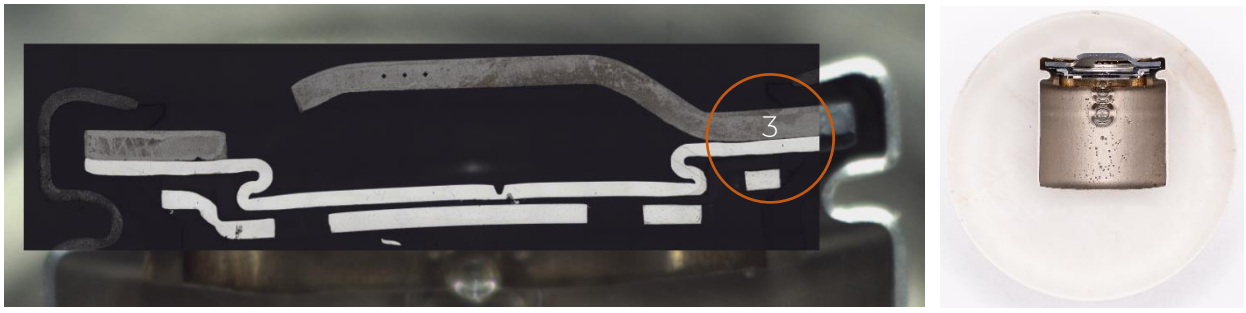
Grain size: 10, evaluation with a 50x HQ lens

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.

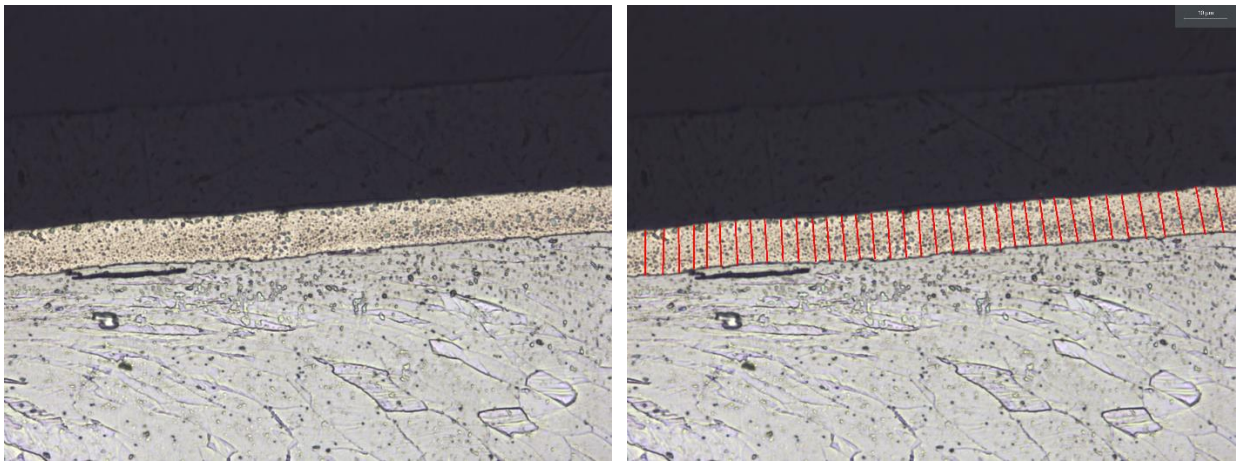
Sample image:



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:

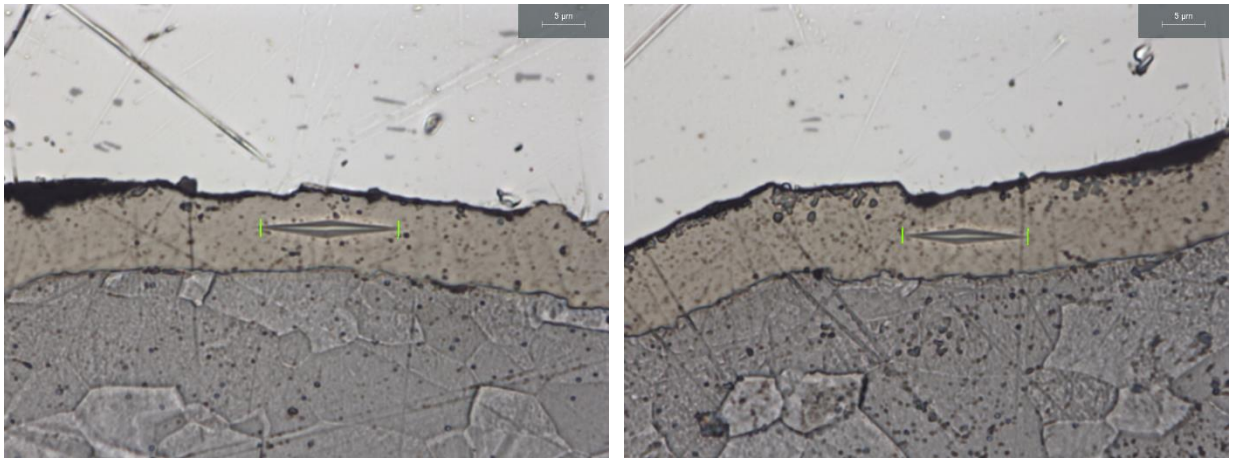


10.28µm layer, evaluation with a 50x HQ lens

Hardness testing:

Determination of the hardness in the layer, here the impressions were made with the Knoop module. The sample must be aligned manually so that the Knoop indenter hits the layer in parallel.

Hardness test points directly in the layer, position 3:

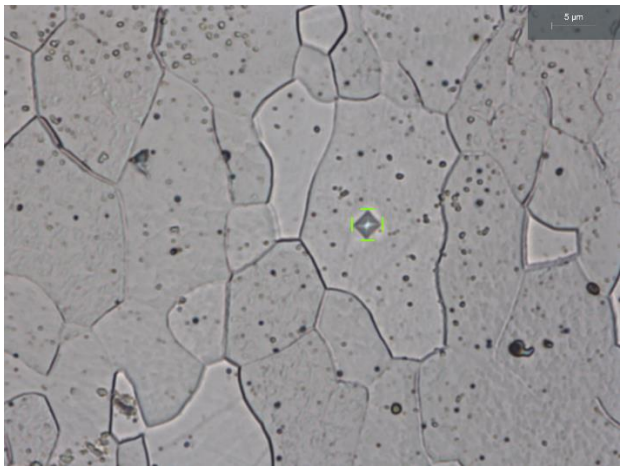


572 HK0.01 (10gf)

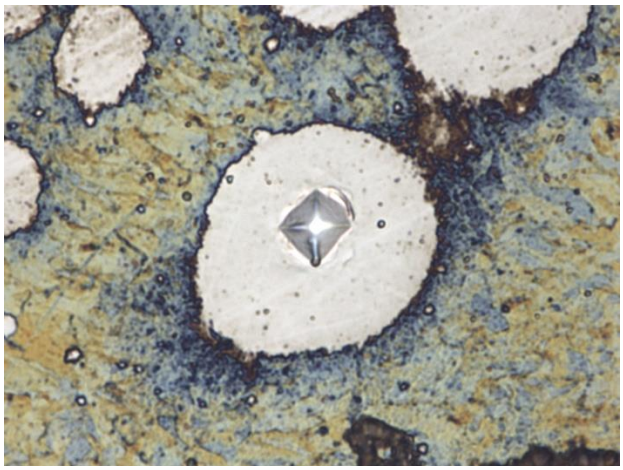
692 HK0.01 (10gf)

Evaluation with a 100x HQ lens

Individual hardness test points in a grain:



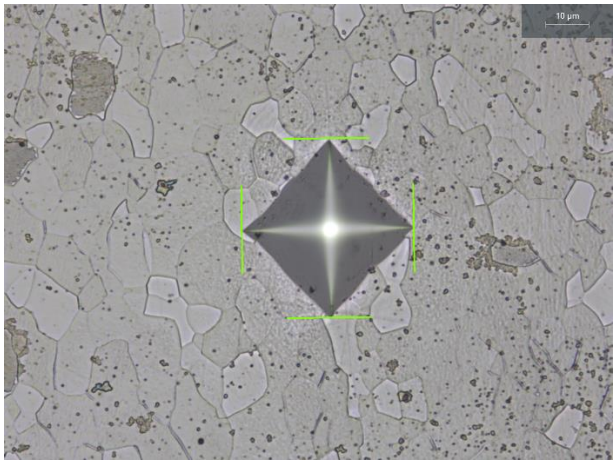
166 HV0.001 (1gf)



245 HV0.01 (10gf)

Evaluation with a 100x HQ lens

Surface hardness test:



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