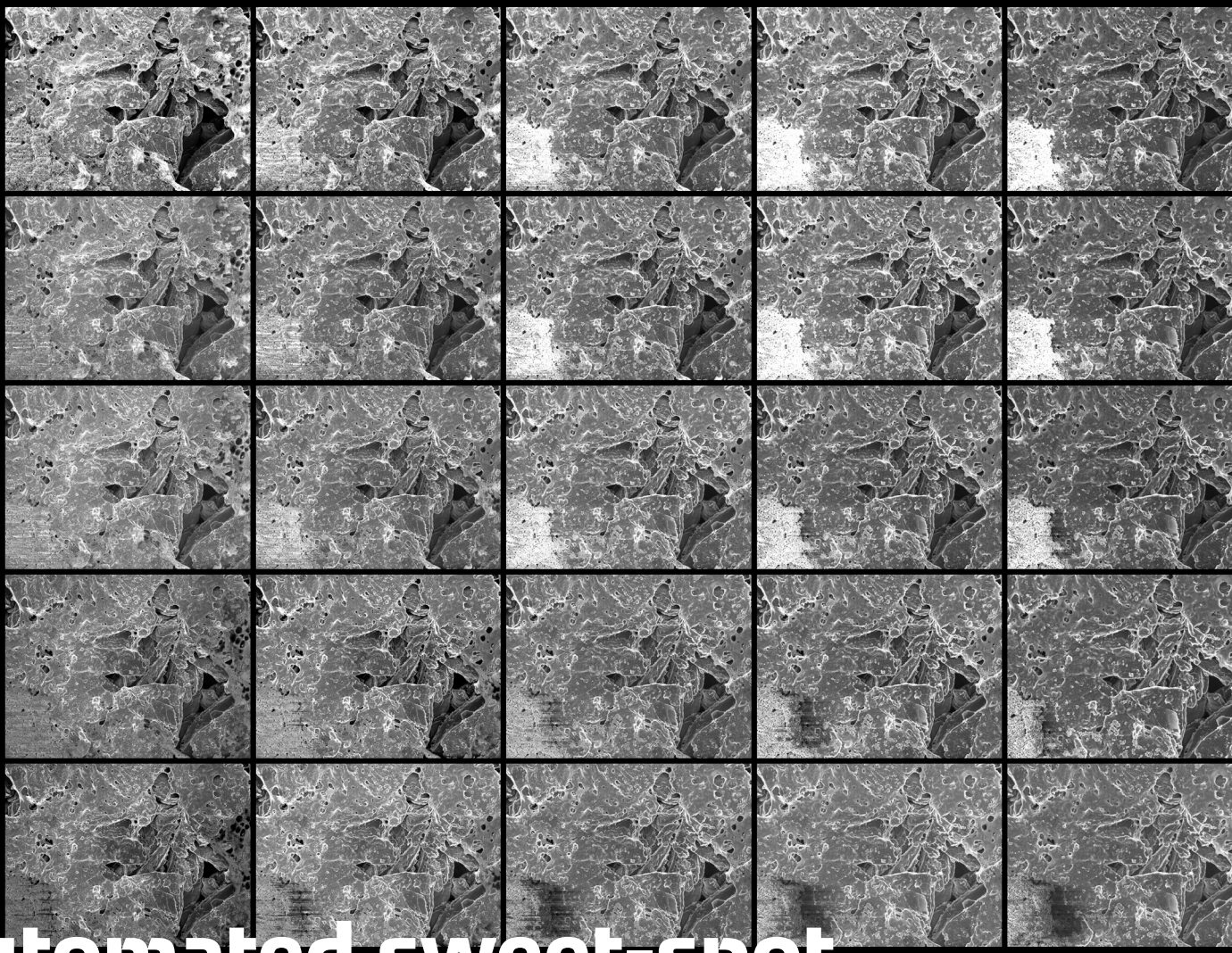
Application note



Automated sweet-spot SEM imaging

Software-based optimization of acquisition parameters using ZEISS Blockwise

Seeing **beyond**

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Abstract

Image quality is fundamental to interpreting SEM (scanning electron microscope) data and, consequently, to understanding the investigated specimens. Since imaging parameters decisively affect SEM image quality, identifying suitable settings is essential yet often time-consuming, as optimal parameters depend on the application and material.

In this study, the optimal imaging parameters for three different SEM detectors were determined for a lithium-ion battery (LIB) anode used as a reference sample. To determine these parameters, a fully automated workflow was implemented using the software ZEISS Blockwise. The workflow performed automated image acquisition over a broad parameter space, enabling systematic exploration of the parameter space without continuous operator input. Automated acquisition proved reliable within the selected parameter range, and the resulting images enabled the identification of detector-specific parameter combinations. The results revealed that the parameter ranges yielding the highest image quality differed between the detectors and showed little to no overlap, indicating that detector-specific settings are required to maximize the information obtained from the SEM images.

Introduction

A wide range of parameters influence the quality of SEM images. To capture the best images with good contrast and low noise, contrast and signal optimization is crucial. In addition to the sample properties, important parameters are working distance, accelerating voltage, probe current, detector geometry, and detector-specific settings. Some of these parameters can have conflicting effects on image quality. To obtain an optimal image, it is always necessary to find the “sweet spot” for these imaging parameters—or at least a satisfactory compromise. Theoretical considerations, such as those derived from trajectory simulations or experience, can serve as a guide for determining the appropriate parameters; however, finding a satisfactory solution often requires time-consuming trial-and-error experimentation under different imaging conditions at the SEM.

This study presents an automated workflow for determining a set of suitable parameters for imaging a reference sample using Blockwise. This automation software uses a visual drag-and-drop interface to simplify EM workflows. It systematically and automatically varies the imaging parameters to generate a collection of images. The user then selects those with the best imaging conditions for their sample.

Experimental

A graphite anode taken from a cycled lithium-ion battery was investigated. The battery was opened inside a glovebox, and the electrode was subsequently transferred to the electron microscope under an argon atmosphere using an inert transfer shuttle (ZEISS Uniport protect) to prevent exposure to ambient air.

Imaging was performed using a ZEISS Crossbeam 550 FIB-SEM equipped with a Gemini 2 electron column. Images were acquired simultaneously using a backscattered electron detector (EsB, energy selective backscatter in-column detector) and two secondary electron detectors: the chamber detector (SESI, secondary electrons secondary ions) and the in-column Inlens detector for low-energy secondary electrons.

For demonstration purposes, this study focused on the influence of working distance (WD) and accelerating voltage (EHT, electric high tension). Accelerating voltages ranging from 1.0 to 10.0 kV (1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0, 6.0, 7.0, and 10.0 kV) and working distances between 1 and 6 mm (1, 2, 3, 4, 5, 5.5, and 6 mm) were systematically investigated. At very low accelerating voltages, the accessible working distance range was restricted by the electron optical system. Therefore, a Python script integrated into the workflow automatically identified and excluded parameter combinations that exceed the instrument's operating limits or lie at the practical operating limit where stable imaging cannot be ensured.

Accelerating voltages ranging from 1.0 to 10.0 kV (as stated above) and working distances between 1 and 6 mm (as stated above) were systematically investigated, resulting in a total of 77 unique parameter combinations. Exhaustively testing all 77 possible parameter combinations manually would have taken a significant amount of time for the operator.

Software

Blockwise enables the creation of customized automated workflows through a graphical, block-based programming environment. Each block represents a single operation, such as moving the microscope stage, performing an autofocus routine, or acquiring and saving an image. Blocks are arranged vertically and executed sequentially from top to bottom (Figure 1). Control structures, including loops and conditional (if–then) branches, are represented by rightward branching block structures, providing an intuitive visualization of the workflow logic. By enabling standardized workflow recipes, reducing operator-dependent variability, and supporting the integration of advanced tools such as AI-based analysis, Blockwise facilitates reproducible, efficient, and high-throughput automated microscopy workflows.

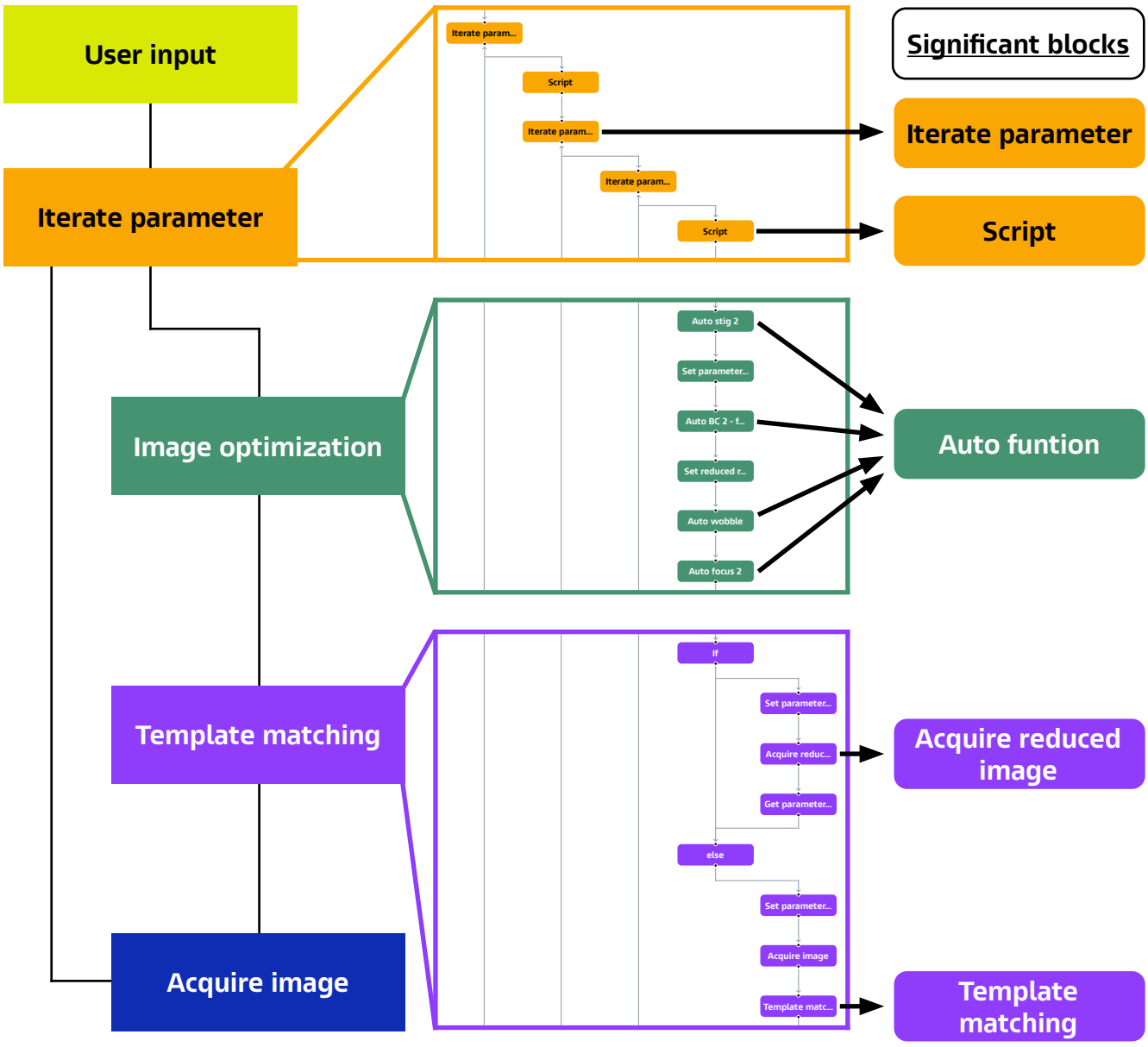


Figure 1: User workflow (far left), color-coded into the following sections: User input (yellow), iteration parameters (orange), image optimization (green), drift correction / template matching (purple), and image acquisition (blue). It also highlights the most significant blocks or groups of blocks.

Workflow

Figure 1 is a graphical representation of the workflow implemented in this study. In the initial section (blue), the user is guided through the required setup steps. Only minimal manual input is necessary: the image is brought into approximate focus, the desired magnification is selected, and the output directory can be specified if required.

In the next part (orange) a series of consecutive “iterate parameter” blocks are used to sequentially loop through the predefined combinations of imaging parameters. To further increase the flexibility and complexity of automated workflows, the standard modules can be supplemented with custom Python scripts. In this workflow, for example, a Python script calculates all subsequent stage positions based on the initial stage coordinates and the working distance defined during the initial setup.

Changes in imaging parameters, such as WD or EHT, typically require re-optimization of the image quality (green). Therefore, after each parameter update, the workflow performs a stepwise image optimization using auto functions. To minimize acquisition time, image optimization is performed in three successive steps. The initial optimization is carried out at low magnification and high scan speed, followed by progressively finer adjustments. During this process, brightness and contrast, astigmatism correction, aperture alignment, and focus are repeatedly optimized.

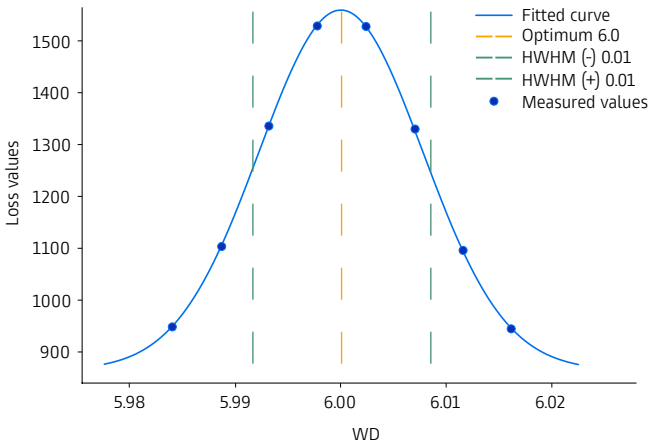


Figure 2 Visual representation of the results of an "auto-focus block." An optimal working distance is determined based on an adjustable number of points. The half-width at half-maximum (HWHM) values of the curve can be temporarily stored as variables to be used as the focus range for an auto-wobble function.

During the image optimization process, the intermediate results of the automated adjustment routines are displayed graphically (Figure 2), allowing the user to monitor the optimization in real time. In addition, the current values of predefined workflow variables can be viewed throughout the execution, providing continuous feedback on the status of the automated process.

Since these optimization procedures may introduce small lateral image shifts, the region of interest is verified before image acquisition. To compare the different SEM working conditions, it is important to always image the same region.

A drift correction based on template matching is therefore performed. The reference template acquired during the first iteration is compared with the current image, and any detected displacement is compensated either by beam shift or, if necessary, by stage movement, ensuring that all images are acquired from the same sample location throughout the automated workflow. For the fine-tuning of the selected automated functions, a single iteration from parameter adjustment to image acquisition requires approximately 5–7 minutes. The processing time varies depending on the contrast conditions, as the automated brightness and contrast adjustment takes longer when the image exhibits more challenging contrast characteristics.

Results

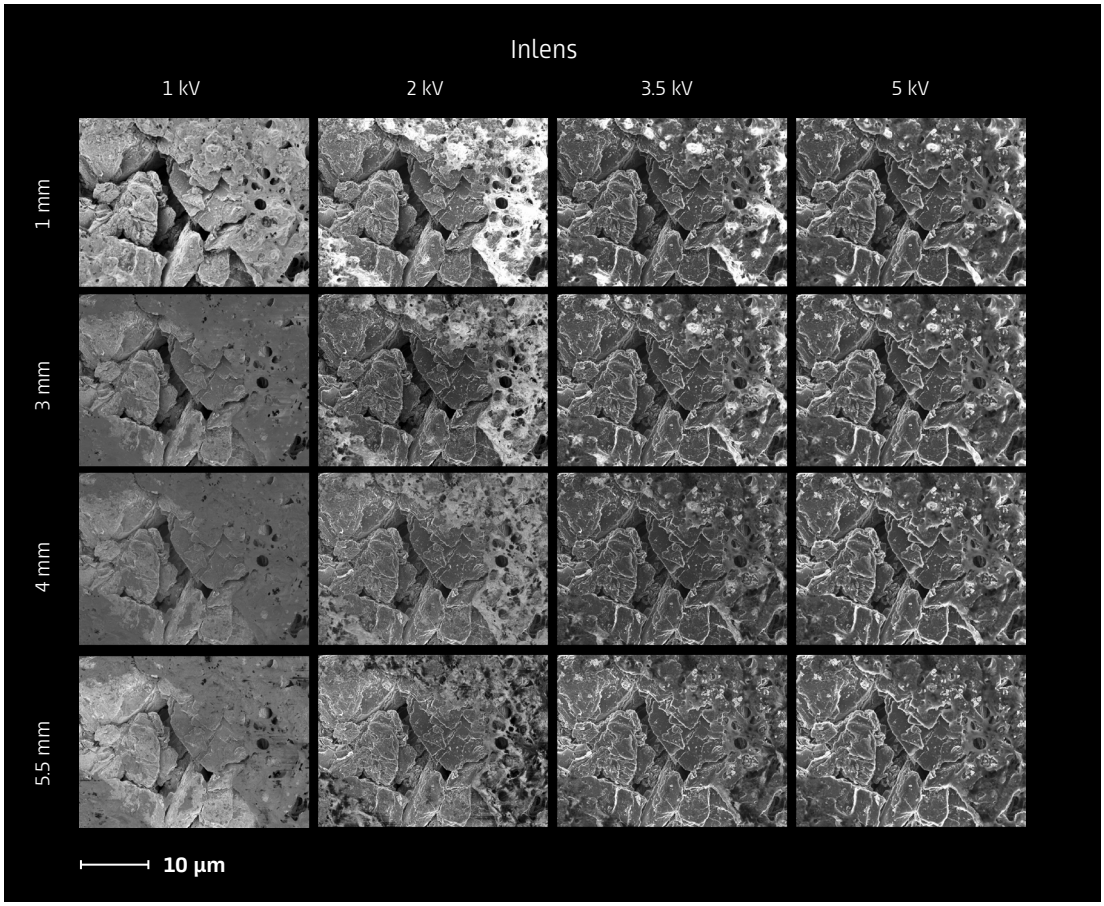


Figure 3 Selection of the most relevant Inlens images acquired at different accelerating voltages (EHT). At an EHT of 1 kV, a dark surface layer is visible. With increasing EHT, edge effects and localized charging of the poorly conductive aluminum particles become more pronounced.

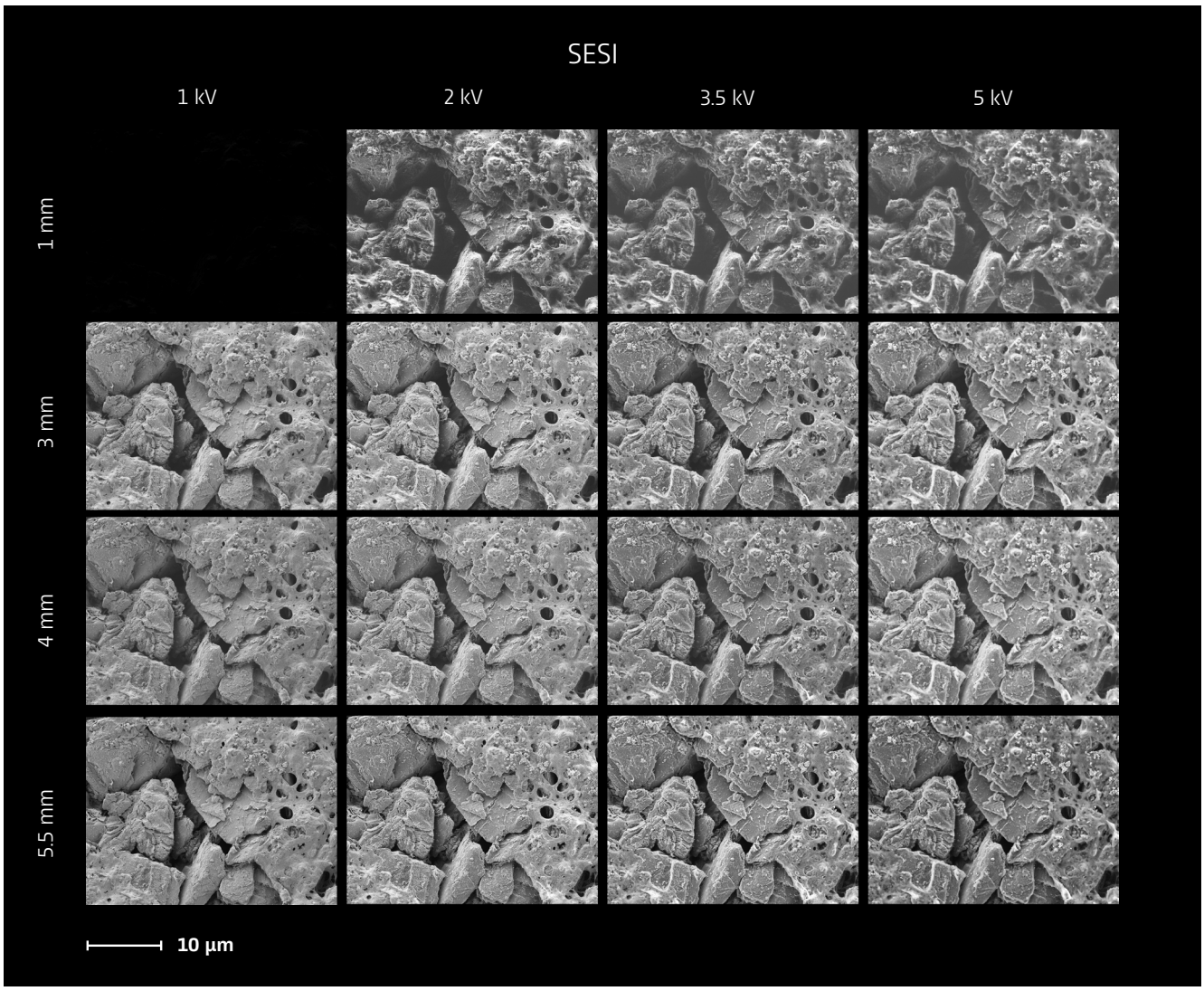


Figure 4 Representative SESI images acquired using the same parameter combinations as those previously applied for the Inlens detector. At a working distance (WD) of 1 mm, shadowing artifacts are observed due to the detector geometry. At accelerating voltages of 5 kV and above, edge effects become increasingly pronounced, accompanied by a loss of surface information.

At an EHT ≥2 kV, edge effects and depth information beneath the surface deposits become increasingly visible. Beneath the surface coating, aluminum oxide particles, originating from the separator's coating, which detached during electrode removal and remained on the surface, are now visible (Figure 3).

Figure 4 shows the same imaging positions using a SESI detector. When the working distance is too small (<2 mm), only an insufficient proportion of the secondary electrons reach the detector due to shadowing. This can lead to partial or complete impairment of the imaging. In addition, the signal-to-noise ratio deteriorates significantly. At an acceleration voltage of ≥5 kV, charge-up and edge effects also increase significantly in this case.

The images captured with the EsB detector (Figure 5) exhibit very low contrast in the range of EHT ≥3 kV in combination with WD ≥3 mm because the images are more surface sensitive and the aluminum particles are not located directly on the surface; therefore, the automatic brightness and contrast adjustments do not function reliably in this range.

The material contrast between the carbon-based graphite and the fluorine-rich surface deposits originating from the electrolyte is weak due to the small difference in atomic number. Consequently, the deposits are only faintly visible at accelerating voltages of 3.5 kV and above.

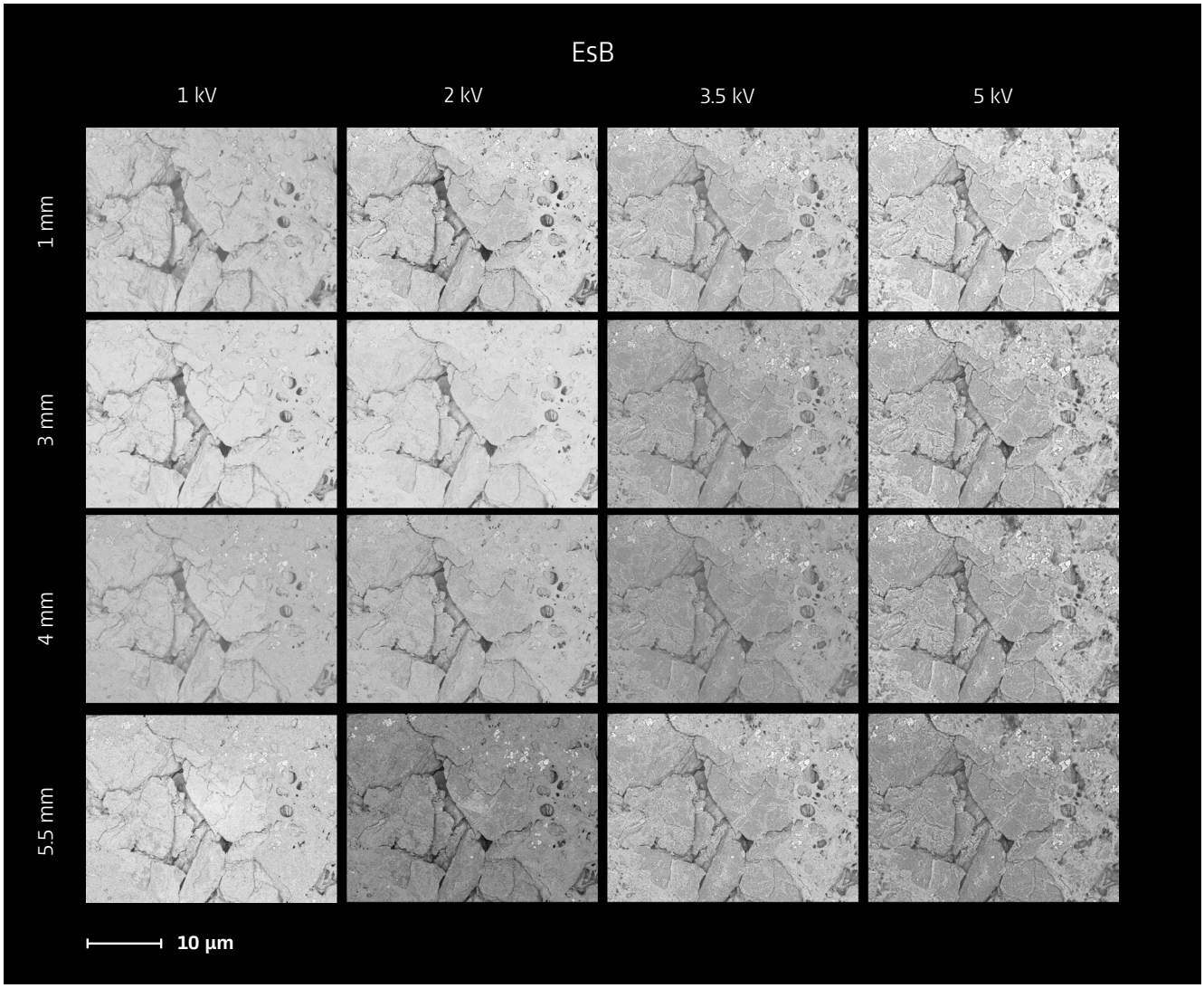


Figure 5 EsB images acquired using the selected parameter combinations. At very low accelerating voltages (EHT) in combination with short working distances (WD ≤ 3 mm), the images exhibit very weak contrast, limiting the visibility of microstructural features.

Conclusion

The automated workflow performed with Blockwise enabled the identification of the optimal imaging parameters easily and efficiently within the investigated parameter range (Figure 6). The results demonstrate that each of the three detectors exhibits a distinct, non-overlapping parameter region that provides the highest image quality.

For the Inlens detector, imaging of the investigated LIB anode with high surface sensitivity and strong contrast is achieved using a very low accelerating voltage (EHT ≤1 kV) combined with a medium to high working distance (WD = 3–5 mm).

For the SESI detector, the geometric arrangement as a chamber detector can result in shadowing effects at very short working distances (WD = 1 mm). However, apart from this limitation, satisfactory images of the investigated specimen can be obtained over a wide range of lower accelerating voltages.

For the EsB detector, material contrast directly at the specimen surface is relatively weak. To obtain sufficient contrast from the subsurface aluminum oxide, higher accelerating voltages (EHT ≥3.5 kV) in combination with a working distance of at least 3 mm (WD ≥3 mm) are required. Within the investigated parameter range, the subjective optimum was found at an accelerating voltage of 5 kV and a working distance of 4 mm.

The optimal parameter combinations determined in this study are 5.5 mm and 1 kV for the Inlens detector, 4 mm and 2 kV for the SESI detector, and 5.5 mm and 5 kV for the EsB detector (Figure 6).

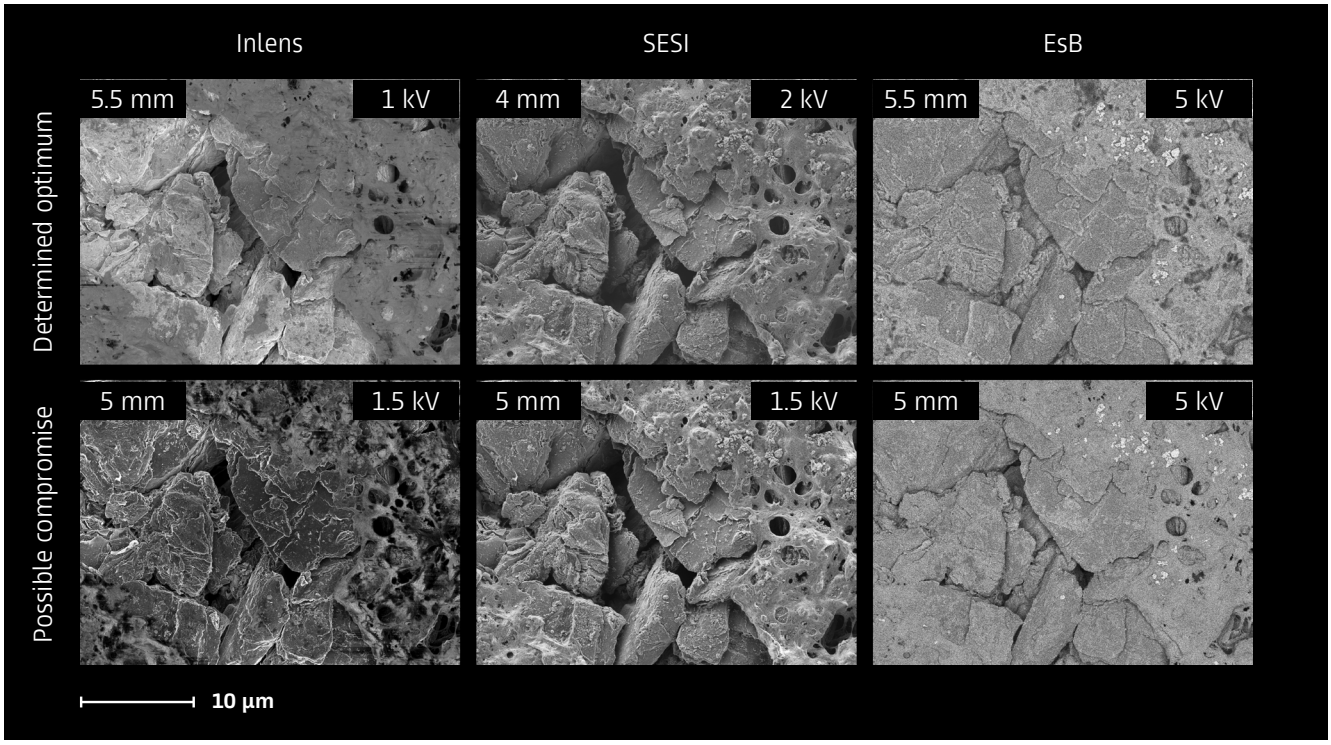


Figure 6 Top: Images acquired under the optimum imaging conditions for each of the three detectors. The corresponding working distance (WD, mm) and accelerating voltage (EHT, kV) are indicated on the left and right, respectively.

Bottom: Images acquired using a possible compromise parameter set for simultaneous image acquisition with all three detectors. While this compromise enables concurrent acquisition, it results in a significant loss of image quality and information content compared with the optimum imaging conditions.

Overall, each detector exhibits a distinct, non-overlapping sweet spot within the investigated parameter space. If time-efficient acquisition of all detector signals in a single imaging process is required, a compromise in the selection of imaging parameters can be made, resulting in a reduction of image quality and/or information content for one or more detector signals.

In addition to enabling the identification of optimal imaging parameters, the image galleries (Figures 3–5) containing the images generated by the automated workflow provide a compact and information-rich overview of the investigated sample.

Summary

Developing application-specific workflows rapidly, executing them without user interaction, and being able to reproduce the identical workflow in future experiments are now possible using the software Blockwise. Through the automated control of imaging and analysis processes, both repetitive and complex measurement procedures can be carried out efficiently and reproducibly. The approach presented here provides a foundation for creating customized automation solutions tailored to specific research questions and helps reduce the time required for this. It has been clearly demonstrated how different materials and material composites benefit from using the sweet spot during image acquisition to reveal material microstructure.

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