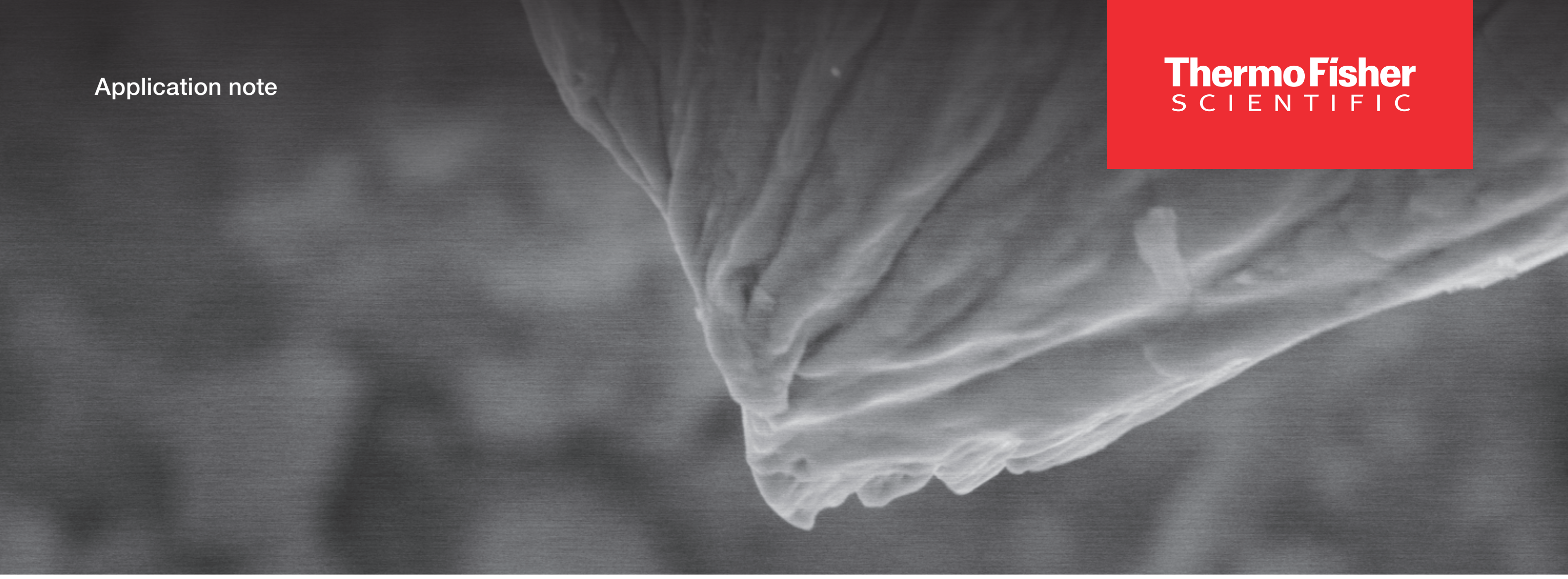

Application note

Stable SEM imaging during long *in situ* dynamic experiments: Image tracking with AutoScript 4 Software

Introduction

Material researchers make deeply intense use of scanning electron microscopes (SEMs). SEMs' resolution performance, analytical capabilities, and high throughput make them the tool of choice to characterize a wide variety of materials for various applications.

As materials research continues to advance, though, it is becoming increasingly important to not only observe materials in their initial and final states but also throughout their various applications. This might include the characterization of hygroscopic characteristics by wetting and drying specific materials or imaging product changes during a heating procedure.

Understanding the material's behavior in real-world conditions is crucial for the development of new products. With the Thermo Scientific Quattro ESEM, materials and life scientists are now able to observe material interactions with water, with the possibility to conduct *in situ* experiments by taking advantage of the introduction of gasses to characterize dynamic changes.

Dynamic changes, however, can cause the area of interest in a material to move or shift during the experiment. This might happen due to a sudden change in the humidity or an expansion of the sample after a temperature increase. If this happens, researchers might lose the correct view or the development of the sample during the *in situ* experiment.

To avoid such problems, Thermo Scientific AutoScript™ 4 Software offers control of the Quattro ESEM and other Thermo Scientific systems to implement advanced functions for powerful automation, which can include imaging, interfacing, patterning, and data display.

AutoScript 4 Software

Thermo Scientific AutoScript™ 4 Software is a Python-based application programming interface (API) that supports unattended, automated analysis through scripting. Being a Python 3.5-based scripting environment makes it extremely flexible and versatile. Python, in fact, is the most popular programming language available, and it is the standard in scientific computing, providing access to a vast collection of pre-installed libraries for scientific computing, data analysis, data visualization, image processing, documentation, and machine learning.

Use case example of an ESEM heating experiment

AutoScript 4 Software can be important for *in situ* experiments using either heating or cooling, especially for data recording and analysis or even process optimization. Specifically, when applied to dynamic experiments, it can be extremely beneficial; for example, in case of image movements, it can help optimize the experiment's results by reducing the movement during heating. It can be also used to record images at fixed intervals and to monitor and log time, stage position, temperature, and pressure during an experiment.

When needed, it can also easily extract parameters from metadata, with the possibility to run the script as a post-processing analysis as the metadata are automatically saved in each image.

The following shows an example of the previously mentioned advantages applied to a heating experiment of a silver wire. The 1,000°C ESEM heating stage was used for the experiment; a silver wire was heated from 310°C to 522°C to understand the material behavior and change during melting.

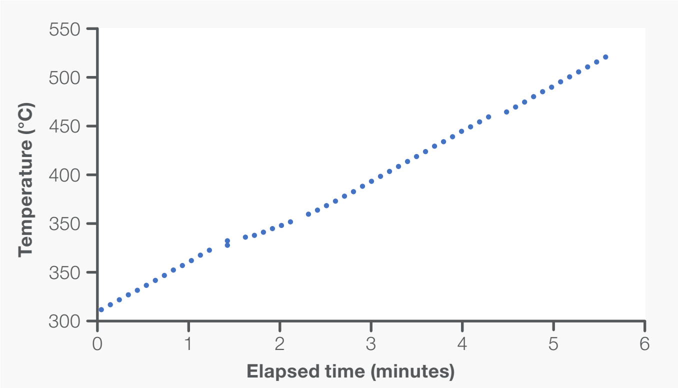

Figure 1. The experiment's temperature profile (˚C) versus the elapsed time (s).

A series of secondary electron (SE) images (Figure 2) was automatically captured during heating. The function is available within the microscope user interface and is extremely useful in cases of dynamic experiments to allow you to focus on the experiment rather than collecting the images.

The acquisition of an entire set of similar images additionally provides a complete set of metadata embedded in them that will give information about the temperature, pressure, and humidity changes. In this specific application example, the most important information to be extracted is the beam shift or stage movements.

Environment changes, in fact, generated sample movements, and the area of interest was not always in the same position. The generation of a movie from the acquired set of SE images would have generated a series of frames with a non-centered area of interest. It also would have posed a risk of losing the area of interest.

Thanks to a Python script, the feature of interest was tracked

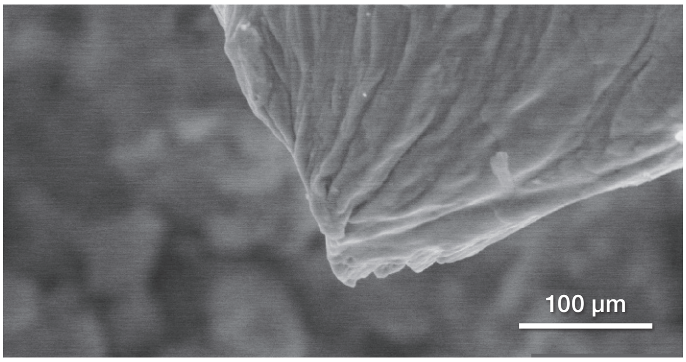

Figure 2. Secondary electron image captured during the heating of the silver wire.

down, and live drift compensation was applied through a combination of beam shift and stage moves. The result is a stable image of the silver wire for the entire duration of the movie, even when the wire changes location over the substrate.

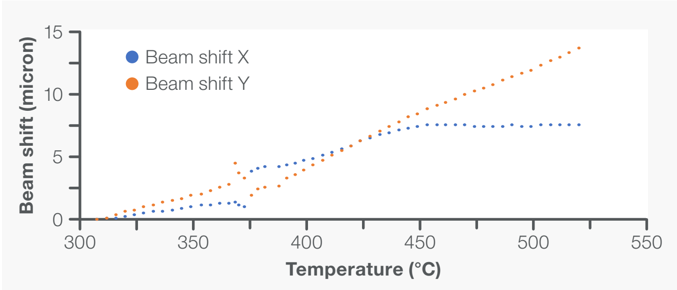

Figure 3. Beam shift variation profiles (X and Y) in time.

Conclusion

In situ experiments are extremely important for a complete understanding of a material. They allow you to not only characterize a sample before or after use, but they can provide information related to the material's behavior during its use in a real-life product. This causes a critical impact in several industrial environments, such as clean energy, transportation, catalysis, nano-electronics, and even human health.

However, depending on the application, many heating or cooling experiments, such as annealing or sintering, can generate structural changes, phase transformations, and segregation/diffusion of specific elements, altering the surface of the material. The ability to follow the development of the sample during the entire duration of the process is crucial to understand its behavior under specific conditions. With AutoScript 4 Software, this is now possible.

Running a Python script for the entire duration of the experiment allowed the area of interest in the field of view to be maintained through live drift compensation. Additionally, AutoScript 4 Software allowed the extraction of several bits of information from the images' metadata, such as the beam shift and stage moves done to compensate the drift of the silver wire, giving the possibility to evaluate the displacement of the area of interest.