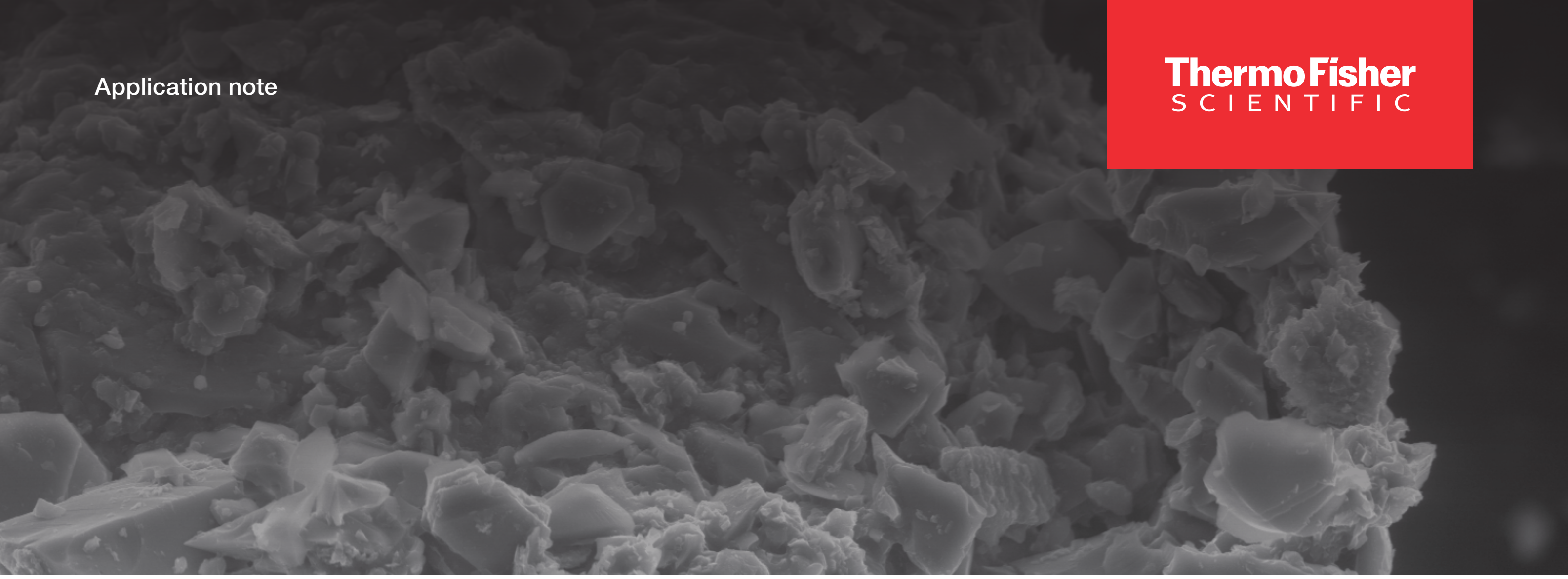

In situ characterization of sodium sulfate exposed to water vapor: a Peltier application example

Introduction

Scanning electron microscopy (SEM) has been known generally in the past as an in-vacuum imaging technique. However, industries in many fields, such as wood products, chemicals, pharmaceuticals, cotton, and polymers, make use of the hygroscopic characteristics of these products to control the humidity in production and to understand how long-term storage of such materials can be affected by changes in the water content. In this perspective, environmental scanning electron microscopy (ESEM) has expanded the boundaries of traditional SEM to deliver deeper insights into a wider variability of sample types by providing a way to observe and record the abovementioned changes at high resolution.

The Thermo Scientific™ Quattro ESEM offers the possibility to pair ESEM with a Peltier cooling stage for full control of sample hydration by varying pressure and temperature (the allowed temperature range is between -20°C and +50°C). The Peltier cooling stage allows you to study and inspect how humidity changes a material and how water interacts at the surface of the samples. Additionally, it offers unique methods for testing of materials that were commonly obtained with other tools and techniques, such as wettability measurements by contact angle, measurements of size, and shape changes during water absorption and desorption. Dedicated detectors both for secondary electrons (SE) and backscattered electrons (BSE) detection allow you to obtain the ultimate resolution, whether you need to work at low or high pressures. Furthermore, the SEM user interface integrates different options to capture movies and TIFF image series during the experiment or after data collection.

In situ characterization of sandstone

An example of an in situ cooling experiment has been conducted on a sandstone sample with crystals of sodium sulphate. The target of the experiment was the exposition of salt crystals to the water vapor in the SEM to study their behavior and observe their dissolution and recrystallization.

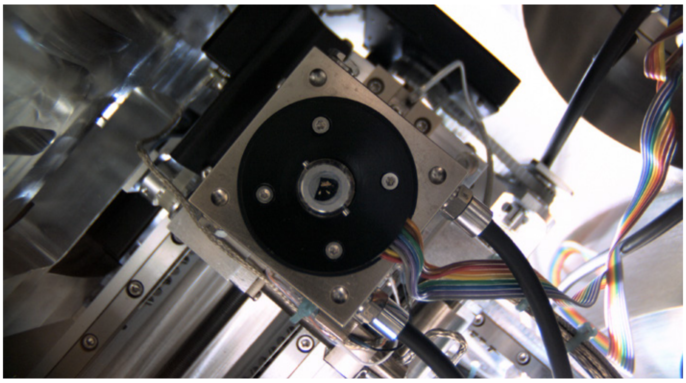

Figure 1. Navigation camera view of the Peltier cooling stage mounted in the SEM chamber.

For this experiment, the Peltier cooling stage was used (Figure 1), and the sample was attached to the stub using a small piece of carbon tape.

At the beginning of the experiment, the temperature of the stage was set to 2˚C, and the system was pumped directly to ESEM while applying an optimized purge cycle. The purging allows you to cycle between a minimum and a maximum pressure (in the range of the desired final pressure) to progressively replace the ambient humidity with the desired pressure of water vapor.

With the stage set at 2˚C and with low relative humidity (RH of 70%, around 470–500 Pa), an EDS map was first acquired at 10 keV to identify the sodium sulfate crystals. The Quattro ESEM, in fact, allows you to perform EDS in ESEM mode. Elemental maps are presented in Figure 2, showing a clear concentration of sodium and sulfur in the object in the center of the characterized area.

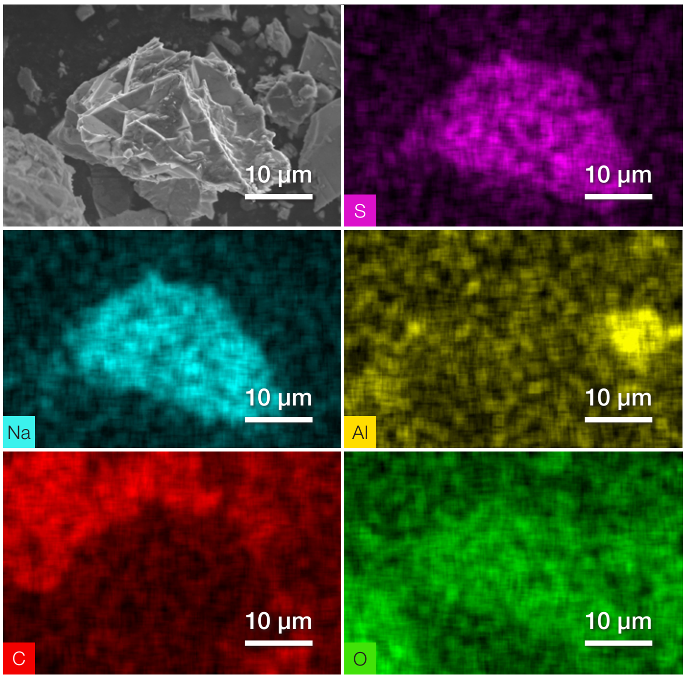

Figure 2. Elemental mapping acquired in ESEM mode, with a pressure of around 500 Pa and a temperature of 2°C.

The crystal shown in Figure 2 was selected and imaged live during the entire experiment. From a starting pressure of around 480 Pa (RH of 70%), the chamber pressure was gradually increased to raise the RH up to 100%. The temperature was kept stable at 2˚C, and, with the increase of humidity up to an RH of 100% (700 Pa), water can be observed on the surface of the sample (Figure 3, third image). Finally, the chamber pressure was gradually decreased (returning to around 400 Pa) to remove water and induce recrystallization of the sample (Figure 3 last image).

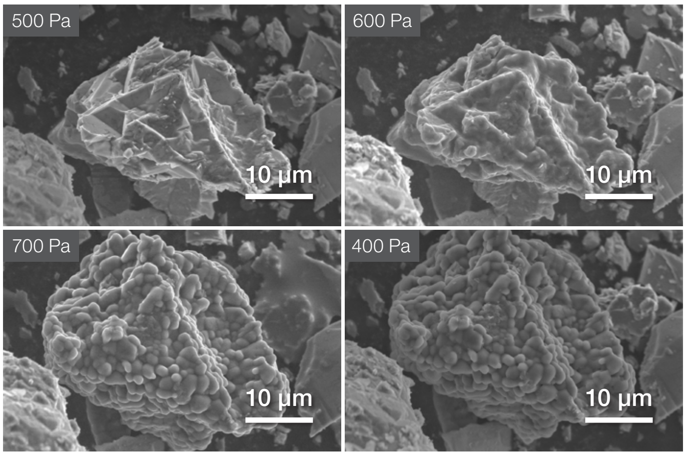

Figure 3. Selection of images showing the progression of the salt's dissolution and recrystallization with the chamber pressure changes.

Conclusion

Thanks to the wide range of additional stages and options for in situ characterization provided by the Quattro ESEM, materials and life scientists are now able to observe real-time material interactions in solution, with the possibility to conduct experiments in ESEM mode by taking advantage of the introduction of gasses to characterize dynamic changes.

Learn more at **thermofisher.com/quattro-esem**

thermo scientific