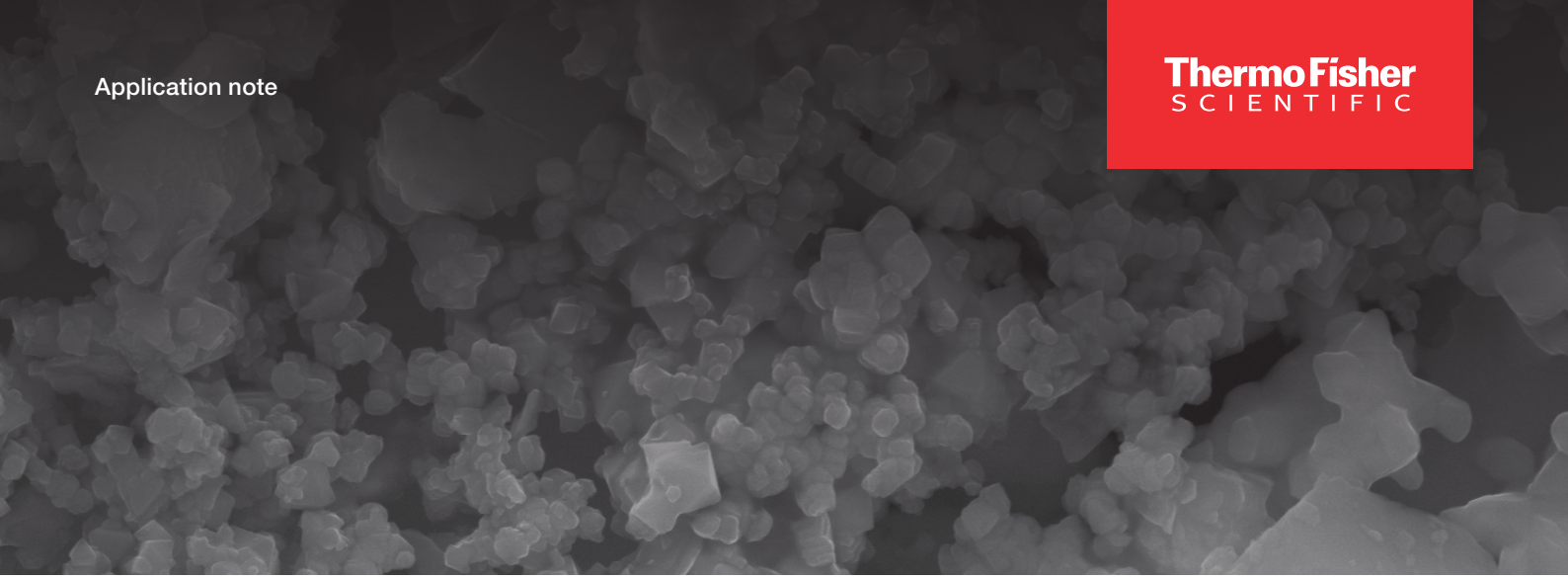
Application note



Nanoparticle in situ heating with the µHeater Holder

Scanning electron microscopy and EDS characterization of the particles' microstructural changes

Introduction

The characterization of high-temperature phenomena, such as particle melting and recrystallization, grain growth, or phase changes, is important in various fields both from a research perspective and from an industrial point of view. QA/QC laboratories may need to perform thermal stability tests on final products, or research facilities may need to assess the usability of a specific material depending on its behavior and characteristics when submitted to rapid heating treatments.

In the past, such studies were typically performed via scanning electron microscopy (SEM) on room-temperature characterization of materials before and after the heat treatment. However, this approach has limitations; for instance, it does not allow you to directly observe the microstructural evolution of the material of interest during the heating or cooling ramp. In addition, the need to remove the sample from the SEM to perform the heating experiment increases the risk of losing the area of interest.

µHeater Holder

The possibility of running in situ dynamic experiments has led to a completely different way of employing SEMs. SEMs are no longer simply a means to characterize previously run researches but are now an active part of the research itself.

The Thermo Scientific™ Quattro ESEM, as the most versatile and flexible high-resolution SEM with in situ capabilities, provides a dedicated solution for in situ high-resolution imaging at high temperatures, up to 1,200°C. The Thermo Scientific µHeater Holder is a high-vacuum-compatible, ultra-fast heating stage for in situ sample heating up to 1,200°C. Thanks to the tiny thermal mass of the holder, the MEMS device of the µHeater Holder delivers consistent, reproducible, and uniform temperature distribution over a heated area of 100 µm.

Controlled heating of a mix of magnetite and hematite nanoparticles

A mix of magnetite and hematite nanoparticles had been diluted in ethanol and sonicated for a few minutes to avoid large aggregates. 3 µl of the solution had been drop casted onto the µHeater Holder's MEMS chip and allowed to air dry. The sample was then mounted, and the stage's heating turned on to 40°C while the SEM was still vented. This ensured complete drying of the substrate before the system was pumped down. Figure 1 shows the secondary electrons (SE) and backscattered electrons (BSE) images of the area of interest.

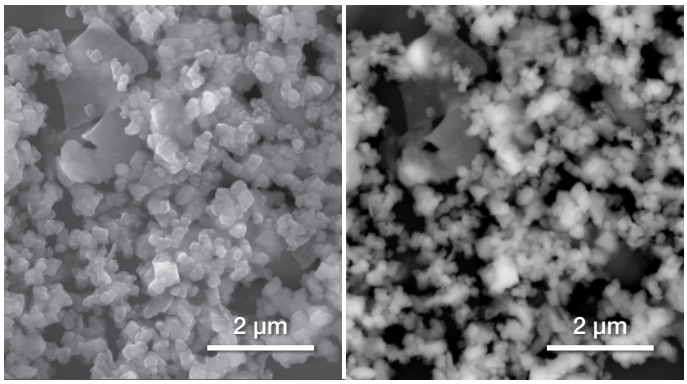


Figure 1. SE and BSE images acquired at the beginning of the experiment, with the temperature set at 40°C. Acc voltage 20 keV, beam current 0.13 nA.

The heating had been executed in different steps, as shown in the graph of Figure 2, to allow the sample to stabilize and be able to acquire additional images and EDS maps. To be noted, the initial steps, up to 800°C, had been executed more quickly and with higher heating rates (°C/s), as the material did not show sensitive changes. The rates had been decreased up to 1°C/s from 800°C up to 1,100°C/s to more accurately monitor the process.

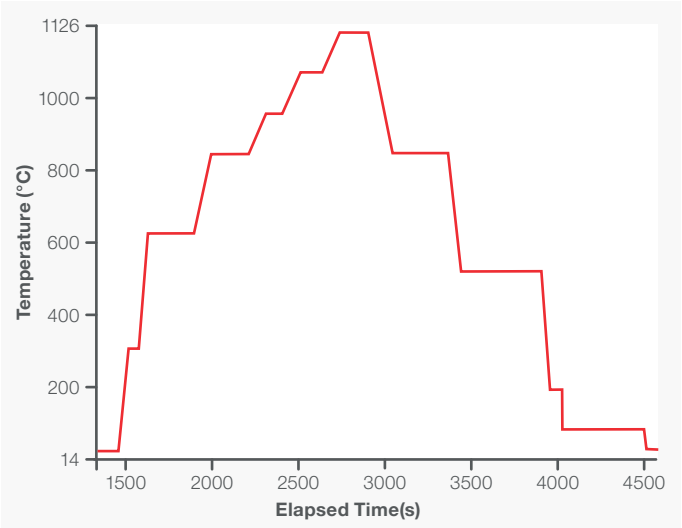


Figure 2. Temperature change in time. The first half of the graph shows the heating, in steps, up to 1,100°C. The second half shows fewer steps during cooling, as it was performed more quickly than the heating.

The evolution of the particles' microstructure had been followed with both SE and BSE detectors, and each acquired frame had been recorded and then converted to a movie.

Some of the frames acquired are shown in Figure 3. Both particles' populations melted and changed, from both a microstructural and compositional point of view. Toward the end of the process, larger square-edged particles were formed, and they appeared to be laid on top of a uniform layer.

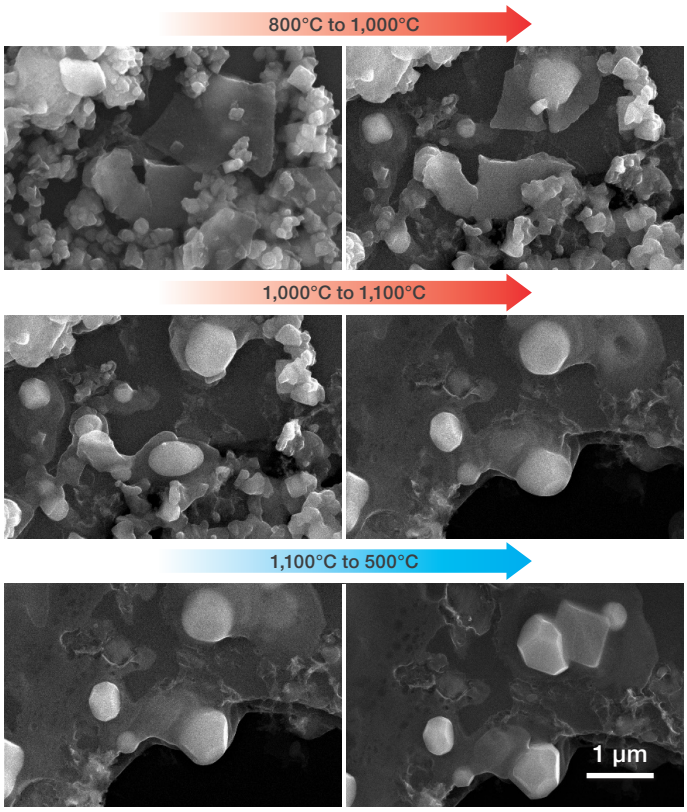


Figure 3. Microstructural changes of the area of interest during the heating and cooling processes. Acc voltage 20 keV, beam current 0.13 nA.

To clarify the compositional changes achieved with the heating experiment, an EDS mapping was performed during cooling at 500°C. This specific temperature was chosen to run the EDS because the area of interest showed a recrystallization of the melted sample into two different populations of particles.

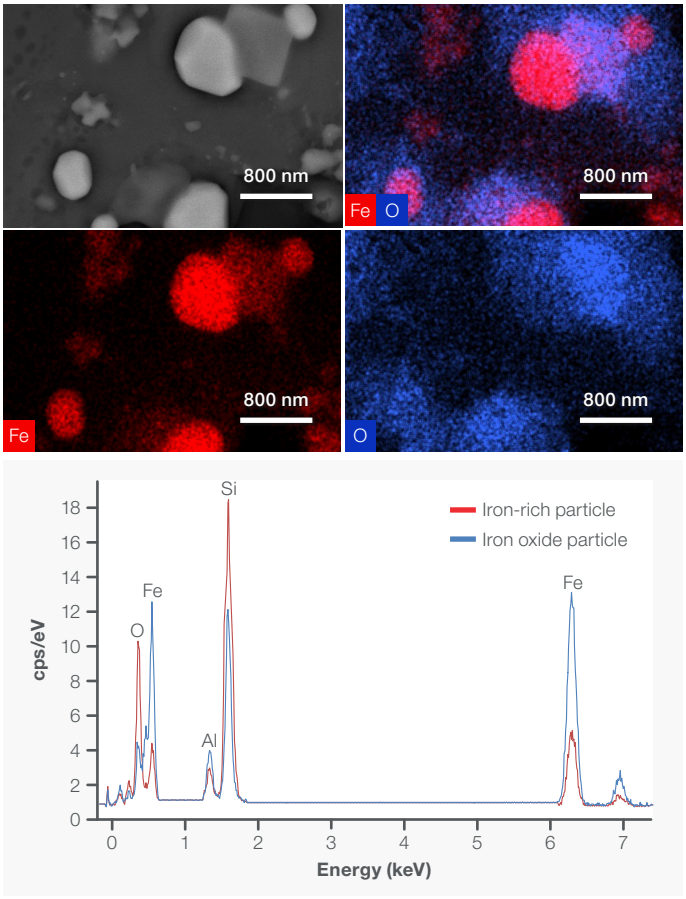


Figure 4. EDS mapping acquired on the heated area. The iron and oxygen maps show an inhomogeneous distribution of the iron in the three particles on the top right area of the image. The two spectra come from two particles showing different compositions.

The ability to utilize the EDS detector and run elemental mapping at high temperature allowed us to understand that the heating experiment had led to the formation of two populations of particles, of which the brightest (shown in the BSE image in Figure 4) are most likely iron with a coating of an oxygen-rich layer. The squared particle on the top right of the image, shows a much higher content of oxygen and lower content of iron.

Conclusion

The SEM and EDS characterization of in situ changes of any material is crucial for a complete understanding of the material's physical and chemical properties. With the µHeater Holder, it is now possible to heat the material of interest up to 1,200°C. Thanks to its design, it allows for precise and rapid heating in high vacuum, reducing the risk for oxidation and providing a stable solution for high-resolution imaging. With its low thermal radiation, several detectors are available during the entire duration of the heating experiments.

In this application note, the heating experiment of a mix of nanoparticles was shown. The microstructural and compositional changes had been monitored during both the heating and cooling of the area of interest with the Everhart Thornley detector (ETD), with the retractable backscattered detector (DBS), and by running EDS analyses at different temperatures.

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