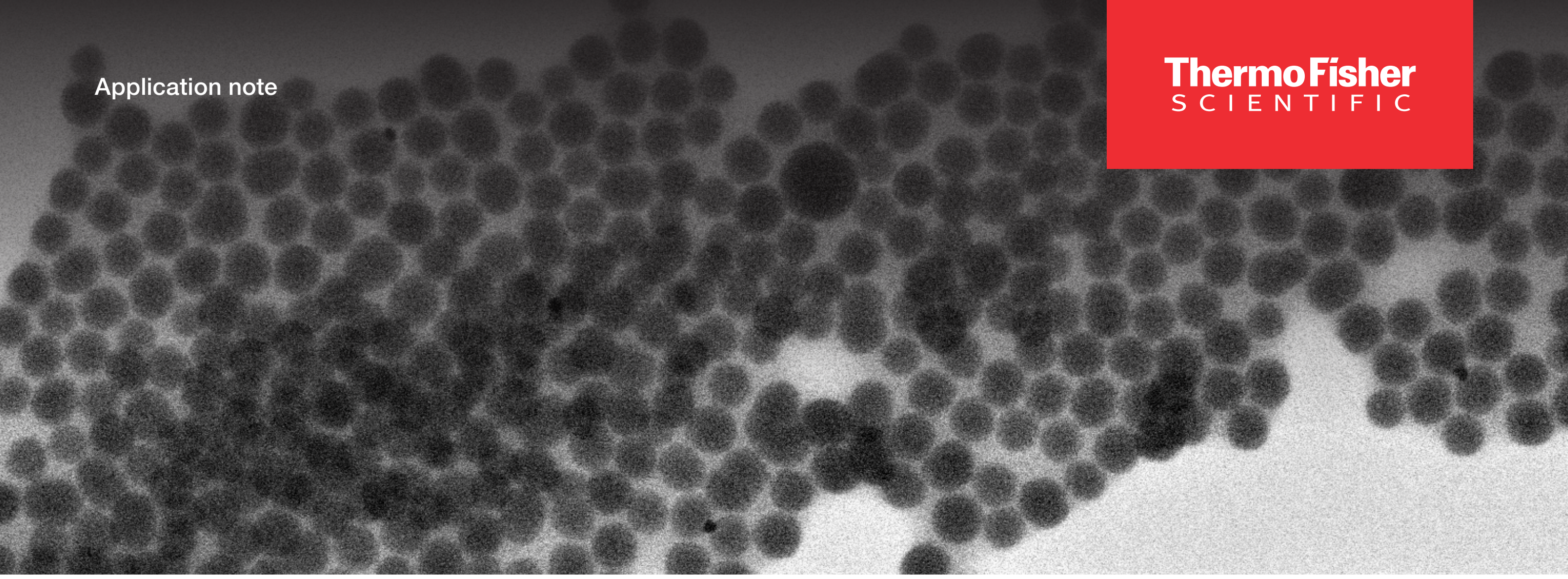

Application note

WetSTEM imaging of liquid silver nanoparticles

Introduction

Traditional scanning electron microscopy (SEM) characterization has been generally linked to the imaging of dry materials due to the conditions imposed using high vacuum in the chamber. This has always prevented direct studies of wet samples and dynamic imaging in a changing sample environment. With increased use of ESEM mode, however, the observation of wet organic and inorganic materials has become a straightforward analysis technique, allowing an increasing variety of users, including those inexperienced with SEM, to run complicated experiments and study their materials in wet environments.

WetSTEM Technology

The ever-growing demand for SEMs with more and more additional functionalities and options has led to the development of Thermo Scientific™ WetSTEM Technology for transmission imaging of suspension; for example, objects from a few nanometers to several millimeters stabilized in a liquid phase.

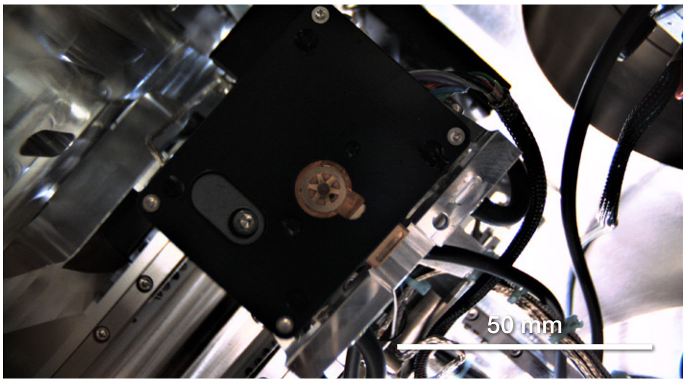

Figure 1. WetSTEM stage with the possibility to load both TEM grids and SEM stubs for ESEM in situ cooling experiments.

The WetSTEM system allows observation of wet samples in transmission mode through thin membranes of water. Compared to TEM, its main advantage is lower acceleration voltages that result in higher contrast. Additionally, EDS analysis is also possible and will provide high spatial resolution results.

Silver nanoparticle STEM imaging in liquid

The material under study is a sample of silver nanoparticles in water. A 1.5 µl drop of the solution was drop-casted onto a carbon-coated holey copper TEM grid. The characterization was carried out on a Thermo Scientific™ Quattro ESEM using the WetSTEM stage.

Water vapor was added to back-fill the chamber, which is essential to accurately control the relative humidity (RH) level of the sample. An initial purging cycle was employed to switch from the room pressure (from the venting cycle) to the fully wet and hydrated condition (RH 100%) without drying the sample. The chamber pressure was then decreased with the aim of removing excess water while keeping the sample fully hydrated. The temperature of the stage was set at 1˚C and maintained at this temperature by the Peltier stage for the entire experiment.

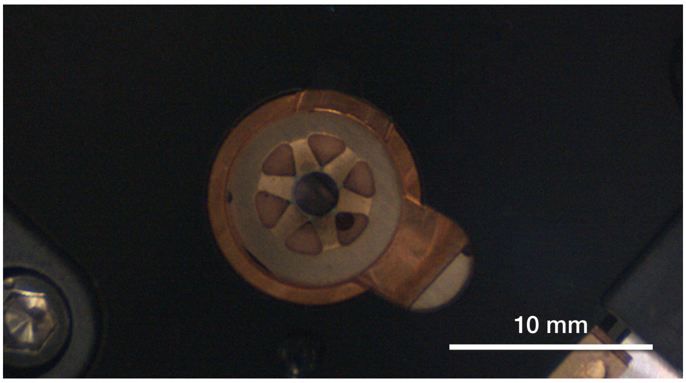

Figure 2. Navigation camera showing the WetSTEM stage with the sample droplet in its center.

The imaging was conducted with the use of the STEM3+ solid-state detector, which, thanks to its configuration with several segments, allows the acquisition of bright field (BF), dark field (DF), and high-angle annular dark-field (HAADF) contrast.

This possibility, together with the SEM user interface design that offers simultaneous detection of up to four signals/detector segments, provided a complete and optimal characterization of the experiment.

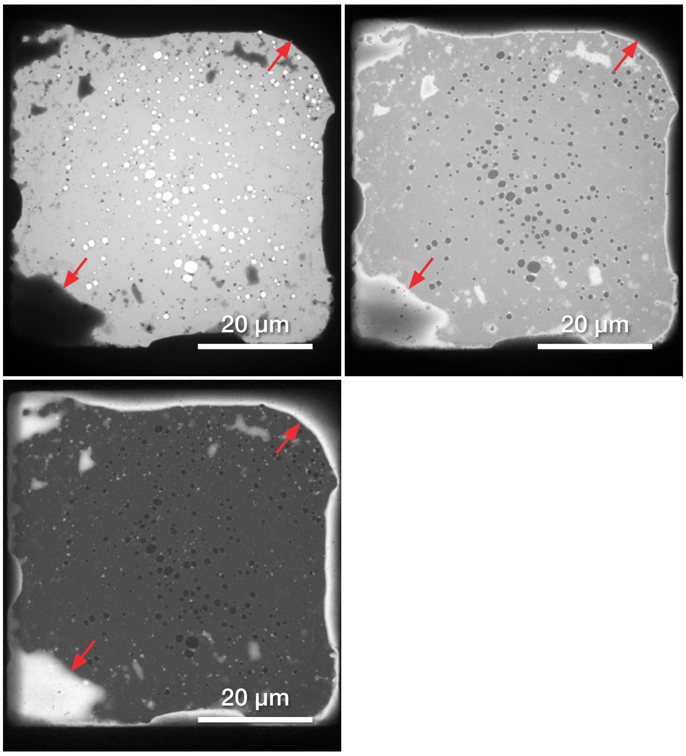

Figure 3. Bright field (BF), dark field (DF), high angle annular dark field (HAADF), images of a large-scale overview of one of the meshes. The images were acquired in wet mode, and liquid water is visible at the edges of the copper mesh.

A first overview of one of the grid's meshes is shown in Figure 3. Liquid water is visible at the edges of the copper mesh, and it appears as either darker or brighter than the sample, depending on the different contrast obtained from the different detectors' segments.

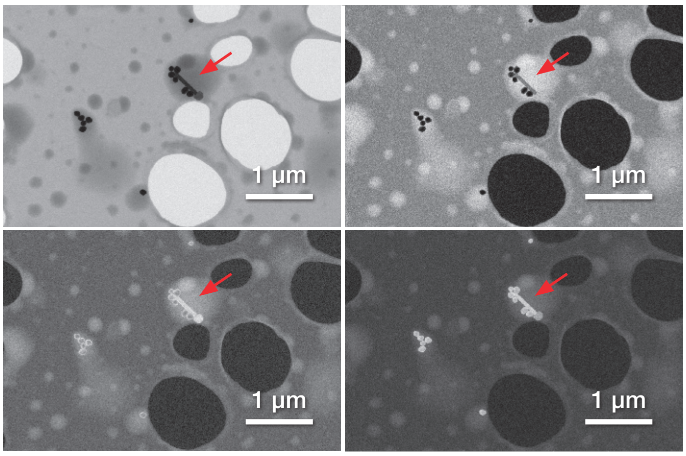

Figure 4. BF, DF2/4, and HAADF imaging of the silver nanoparticles in the water droplets.

Higher magnification images (Figure 4) were acquired to characterize the nanoparticles' shape. Additionally, with an RH of 100%, the WetSTEM stage allowed the inspection of the nanoparticles' arrangement in liquid and how they interacted with each other (examples are shown in Figure 5).

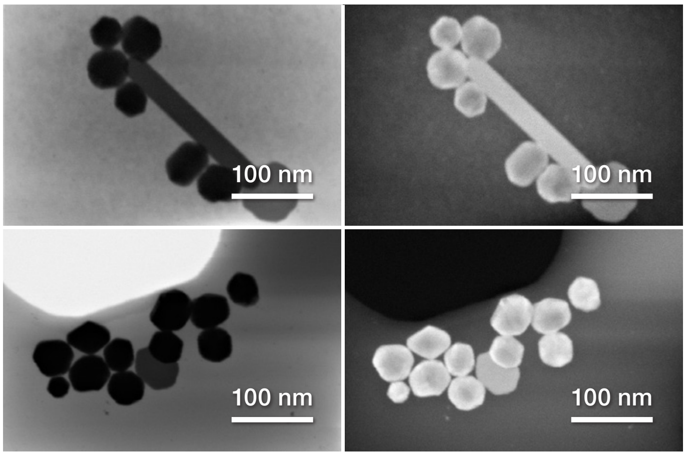

Figure 5. High-magnification BF and HAADF of different nanoparticles' shapes.

Conclusion

The Quattro ESEM is the workhorse SEM for in situ dynamic experiments. It serves a very wide range of needs, including the need to obtain images in transmission mode, with the additional benefit of doing it in a changing environment that allows study of the interaction between materials in suspension.

WetSTEM Technology provides access to several parameters such as particles size and their size distribution, but it can also be used to determine, for example, the stability of objects in a liquid phase or to study how they align with respect to each other when in liquid. With its enhanced contrast, thanks to the low voltages, and the extremely good resolution results, WetSTEM Technology proves to be an easy and effortless transmission technique that also gives access to volume information. This means researchers are no longer limited to imaging only the surface of a water drop.

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