Development of environmental tomography for sensitive nanomaterials in wet-STEM

J. Xiao[1]*, J. Ferreira[1], A. Malchère[1], F. Dalmas[1], L. Roiban[1], G. Foray[1], K. Masenelli-Varlot[1]
[1]*Université de Lyon, INSA-Lyon, MATEIS, bât. B. Pascal, 7 avenue J. Capelle, 69621 Villeurbanne cedex, France*

*juan.xiao@insa-lyon.fr; Telephone: 33660618820; Fax: 0472437930

1. INTRODUCTION

ESEM (Environmental Scanning Electron Microscopy) allows the observation of materials under controlled relative humidity due to the presence of the gaseous environment and the control of the sample temperature [1]. Under specific conditions of pressure and temperature, the observation of nano-objects in a liquid is made possible in the transmission mode, with images quite similar to those obtained in Transmission Electron Microscopy using a closed cell [2]. Moreover, the specificity of ESEM lies in the fact that hydration and de-hydration cycles can be carried out by varying the environmental pressure.

In parallel, a device has been developed for the characterization of the 3D structure of non-conductive and low-contrast materials [3]. Very recently, it has been shown that the acquisition of image series in wet samples (wet-STEM tomography) before and after in situ condensation of water can be achieved with the implementation of a Peltier stage in the tomographic sample holder [4]. In this work, we will present some improvements on the wet-STEM device, who now permits to characterize liquid suspensions. For that purpose, it is necessary to get the more accurate measurement of the sample temperature for a better controlling of the process of water evaporation or condensation.

2. RESULTATS

2.1 Development of experimental device

The wet-STEM tomography device (Figure 1) is placed in a XL-30 FEG ESEM from FEI and designed according to the literature [4]. It consists in: (a) a tilting system ensured by a rotating piezoelectric system, (b) two-translation piezoelectric systems to position the area of interest at the eucentric position and keep it in the field of view while tilting, (c) a Peltier stage, which can transfer the cold to the sample holder by slight friction. (d) a sample holder, and (e) the detection system. In this device, a thermocouple is inserted in the Peltier stage. The sample temperature is then controlled through the controller of the FEI conventional Peltier stage. In this case, the thermocouple is located far away from the sample and the sample temperature may be different from the measured temperature. Therefore, another thermocouple was placed in the sample holder to get a more accurate sample temperature, which is necessary to control the evaporation and condensation of water. The variations of the different temperatures are displayed in Figure 2.

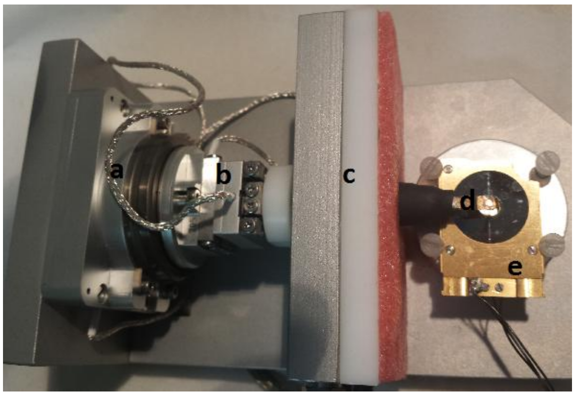

Figure 1. Device for wet-STEM tomography in the ESEM

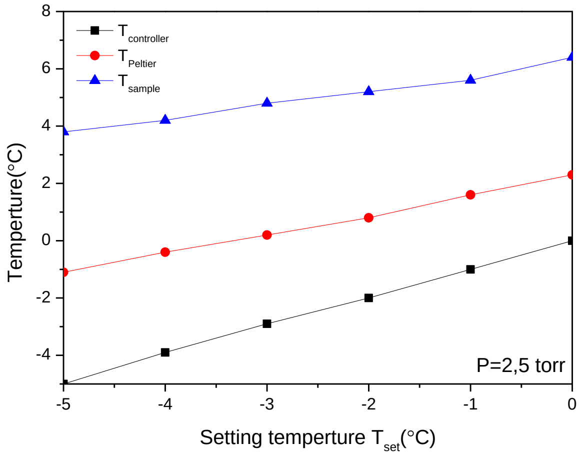

Figure 2. The variations of the different temperatures under the pressure 2.5 torr, $T_{controller}$ - temperature of FEI controller, $T_{Peltier}$ - temperature of Peltier stage, T_{sample} - temperature of sample holder (near to the sample)

2.2 Wet-STEM image of latex suspension

SBA-PMMA is a copolymer derived from styrene and metacrylic acid esters in aqueous solution. In this experiment, the SBA particles already include a 3% PMMA shell, playing the role of steric surfactant. For some applications, another surfactant can be introduced to further stabilize the latex. The image in Figure 3 shows that SBA particles are surrounded by a bright shell, which can be assigned to the surfactant. The stability of the sample during the acquisition of the tilt series will then be discussed. First reconstructions of the 3D film formed during water evaporation will be presented, and the resolution will be discussed in function of the environmental pressure.

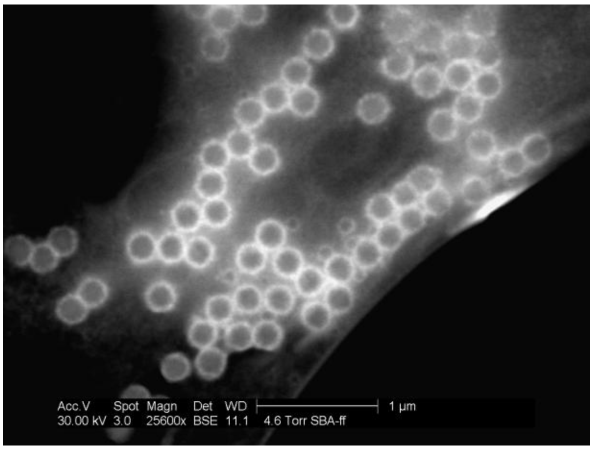

Figure 3. Wet-STEM image of SBA latex suspension

3. CONCLUSION

More accurate sample temperatures have been measured with the development of wet-STEM device. *In situ* hydration / dehydration experiments have been carried out with the control of water condensation or evaporation. Volumes have been reconstructed, showing the potentialities of such technique for the characterization of suspensions from the wet to the dry state.

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