The ESEM and water – Prospects and limits

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The specimen chamber of an environmental scanning electron microscope (ESEM) forms a micro-laboratory, where a multitude of dynamic experiments can be performed. The onset and progression of the respective processes can be observed with high magnification and great depth of focus. For example, the investigation of the wetting and drying of porous media like membranes can be carried out on a micro-scale. It provides, amongst others, information about the dependence of the number of dry pores and their size on the time elapsed. This information cannot be gained by conventional test procedures.

But the low pressure in the specimen chamber (between 13 and 1600 Pa) compared to the ambient pressure entails some limitations. If a cup of pure water is placed in the specimen chamber, the water starts boiling below a certain pressure, with the atoms and molecules of the dissolved gas acting as nuclei for bubble formation. But the evaporation of water causes a cooling down to a temperature below the dew point at the respective pressure. As a consequence a lot of bubbles and water splashes are generated (Fig. 1), which can harm the vacuum system of the ESEM. If the cup and the water stay at the same temperature, the boiling stops. Whereas this could actually be observed for a cup made of glass, the boiling went on in a metal cup, even if degassed water was used. The reason may be that, due to its good thermal conductivity and the thermal contact with the surrounding, the cup has always a slightly higher temperature than the water. Thus it would be very elaborate, if not impossible, to perform experiments like the one sketched in Fig. 2, with the aim to investigate the permeability of porous systems for water by pressing it through this system [1]. But this test arrangement may work for other liquids, e.g. oils.

A water reservoir inside the specimen chamber can also be created by condensing water at a Peltier cooling stage. As in this case cup and water have the same temperature, no boiling will happen. Whereas with this setup it will be difficult to press the water through porous media, it can rather easily be adapted for the investigation of the wetting and drying of specimens [2]. Often this process is controlled by changing the pressure between values above (wetting) and below (drying) the dew point. But one has to be aware, that such pressure changes are always connected with temperature changes at the wet specimen, which are due to the occurrence of condensation and evaporation heat. This is demonstrated in Fig. 3, with a copper specimen placed at the Peltier cooling stage. The temperature was measured with thermocouples both at the stage and at the copper specimen. Because of the heat sink between stage and copper, the measured temperature changes at the stage are much smaller than those occurring at the specimen itself.

In the Figs. 4 and 5 the results from the investigation of the drying process of polyethersulfone membranes (Micro PES4F from Membrana) are shown. During the drying process images were recorded in regular time intervals (Fig. 4). With image processing software from every image the number and the size distribution of the already dry pores could be determined as a function of time. Both to minimize specimen damage [3, 4] and to maximize contrast the specimens were coated with a Au/Pd-layer. A comparison of the drying process at coated and uncoated specimens demonstrated, that the coating had no measurable effect on the drying up of the pores (see Fig. 5).

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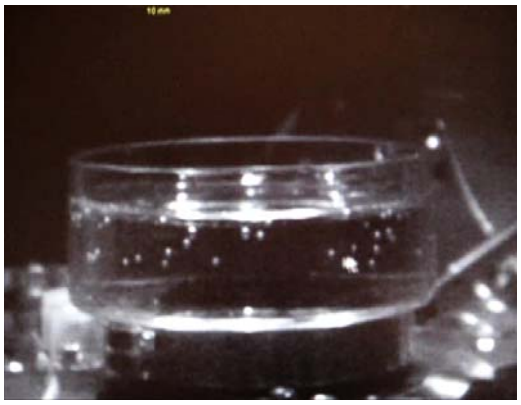

Figure 1: Boiling water (note the bubbles) in a glass cup at vacuum conditions (1000 Pa).

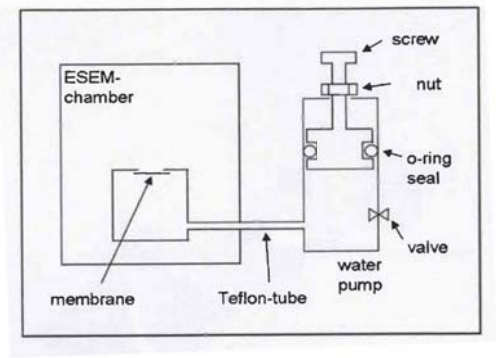

Figure 2: Sketch of an experiment for pressing water trough porous media in an ESEM.

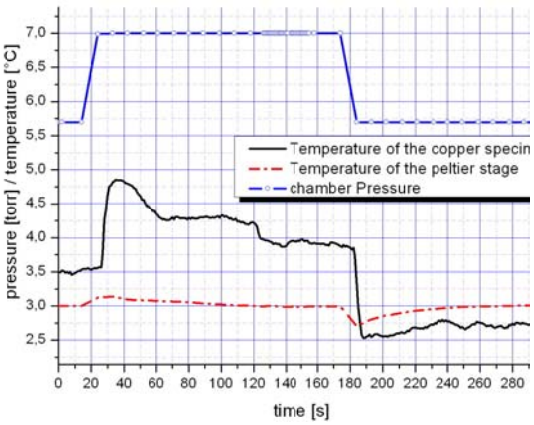

Figure 3: Temperatures measured at the Peltier stage and a cooled copper specimen mounted on it during pressure changes.

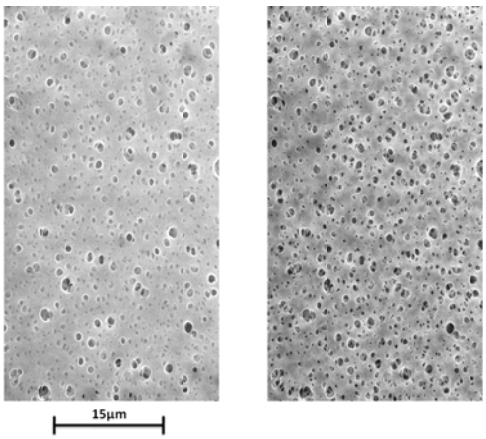

Figure 4: Micro PES 4 F membrane during the drying process, left side partially dried, right side fully dry.

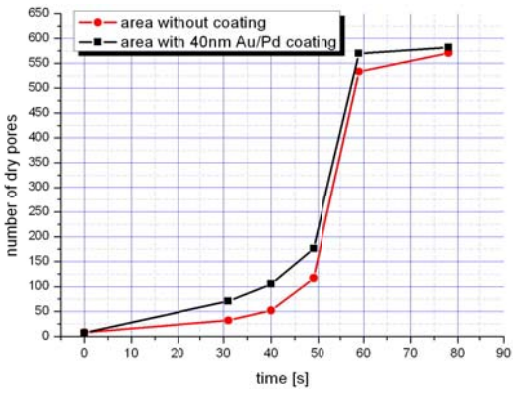

Figure 5: Number of dry pores as a function of drying time.

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