In situ investigations of porous membranes in a wet environment in the ESEM

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Introduction

The environmental scanning electron microscope (ESEM) enables the investigation of specimens like porous or biological samples even in wet environment. This work presents first results of the wetting and drying behavior of porous micro filtration membranes (Micro PES 4F from Membrana) with an asymmetric porosity profile.

Porous micro filtration membranes

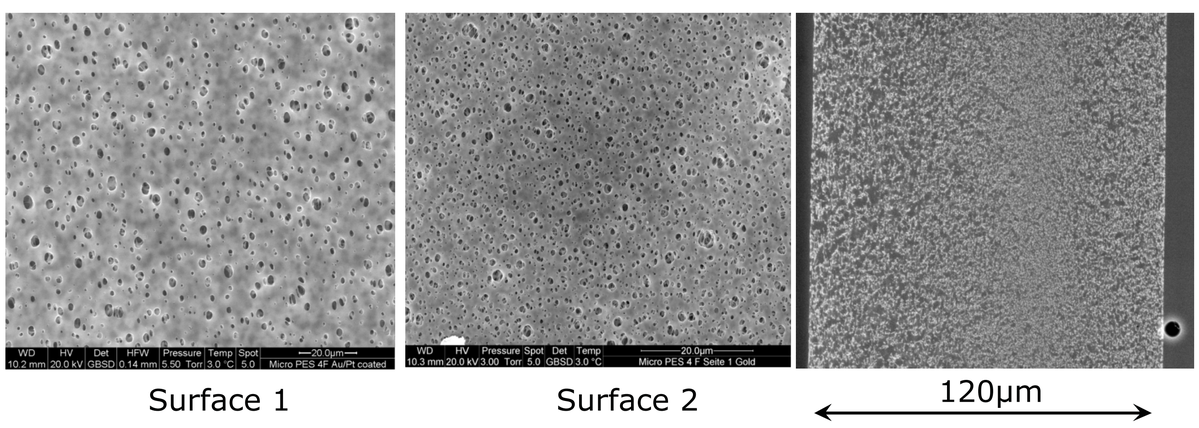

Fig.1: Images of the membrane surfaces and the asymmetric cross section. The pore density varies from 500 (Surface 1) to 6000 (Surface 2) pores per (100 x 100) µm.

The investigated membranes (Micro PES 4F) have an asymmetric cross section (porosity profile) [2]. Surface 2 shows smaller pores with a higher pore density than surface 1 (Fig.1).

Experimental and Instrumentation

This work was carried out using an ESEM Quanta 600 FEG from FEI (Eindhoven).

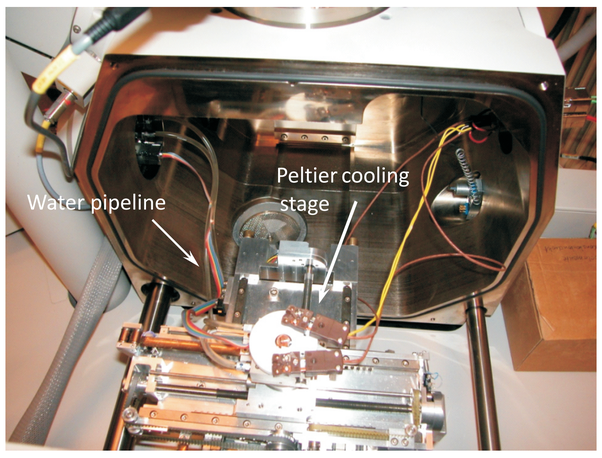

Fig.2: Sample chamber of the Quanta 600 FEG ESEM with mounted peltier cooling stage

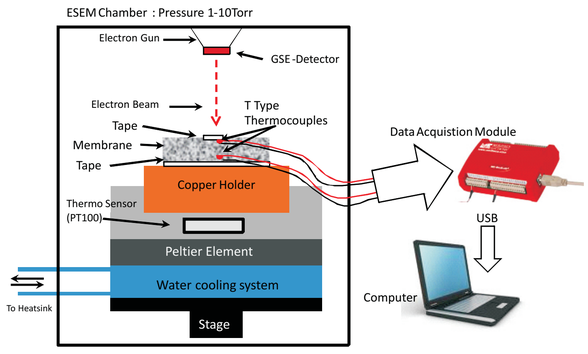

Fig.3: Sketch of the experimental Setup: 2 micro T type thermocouples were attached at the membrane surface

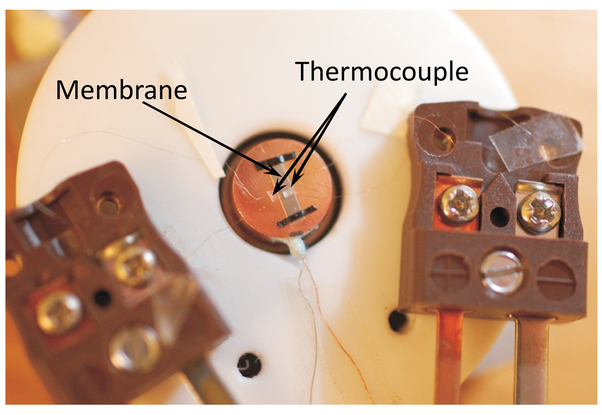

Fig.4 Membrane with attached micro thermocouples mounted at the peltier stage

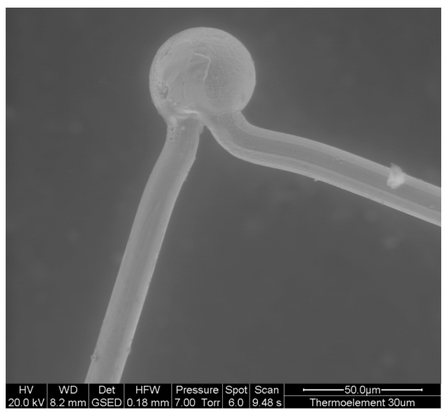

Fig.5: Micro T type thermocouple (Diameter ca. 30 µm) from Omega

Using a peltier cooling stage the environment of the specimen, e.g. the relative humidity, can be controlled by choosing the appropriate values of pressure and temperature. By crossing the dew point even water can condense and experiments with wet or wetted specimens can be performed. This was used to investigate the wetting and drying behaviour of porous membranes *in situ* [1].
Several thermocouples were used to monitor the temperature change at different points of the experimental setup (Fig.3).

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Results and Discussion

During the drying process pictures were recorded in regular time intervals to document the process. Dark areas represent the dry pores (Fig. 6). With the image histogram it is possible to separate dark pores and the bright membrane material[3]

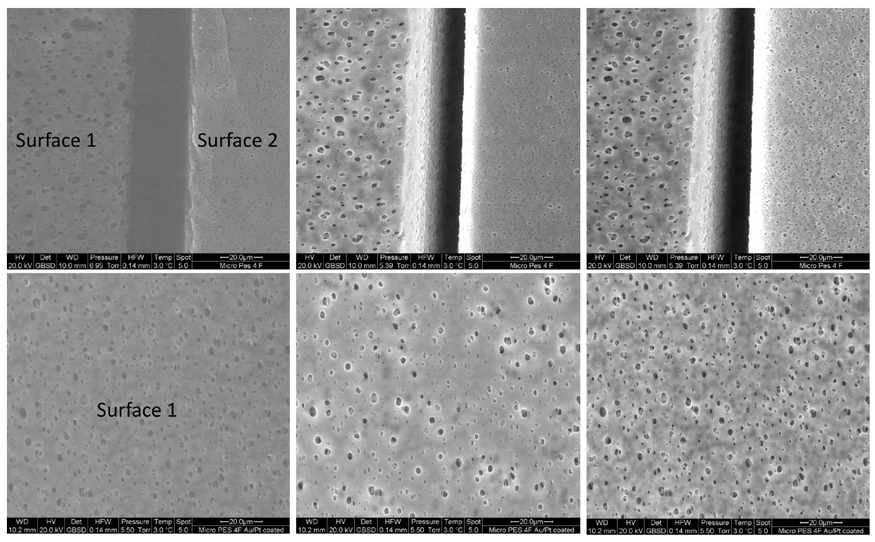

Fig.6: Image sequence of the membrane during drying. At the beginning of the drying process mainly the big pores are drying, subsequently drying of the small pores starts (Surface1-bottom). The top sequence shows both surfaces. Surface 1 dries fist, followed by surface 2.

The pore size distribution of dry membranes follows a log normal distribution. For each picture recorded during the drying process, the pore size distribution of the dry pores was analyzed. The distribution changes from a gaussian form at the beginning of the drying process into the log normal form (Fig.7).

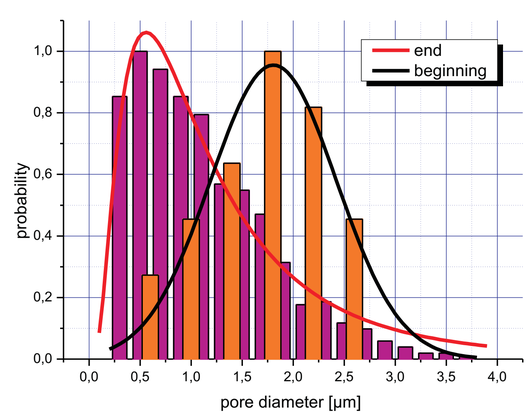

Fig.7: Pore size distribution of dried pores at the beginnig and at the end of the drying process via surface 1

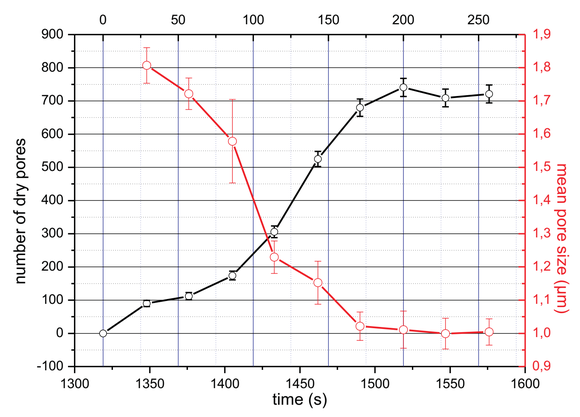

Fig.8: Number of dried pores and their mean pore size during the drying process via surface1 (Fig. 6 bottom).

The drying process of the membrane, for the two surfaces, shows a strongly different behaviour (Fig.6 top). At surface 1 (greater pores) drying starts earlier than at surface 2 (smaller pores). Also the recorded temperature profiles are different. The wetting and drying process via surface 2 is associated with a strong temperature change at the membrane surface (Fig.9 right).

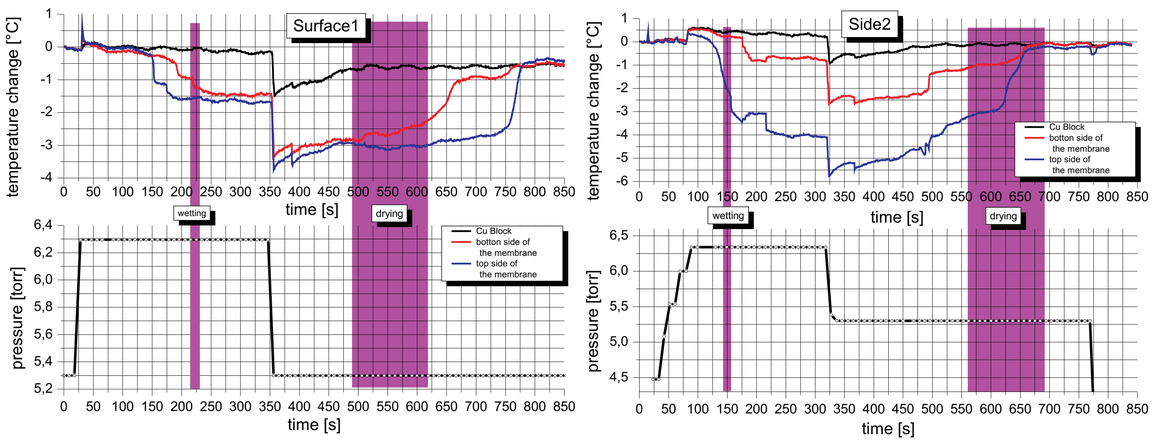

Fig.9: Recorded temperature profiles during the drying process via surface 1 (left) and via surface 2 (right). The wetting and drying process is marked in the diagram.

At surface 1 (grater pores) drying started earlier than at surface 2 (smaller pores). Evaporation and condensation heat couses jumps In the temperature when pressure is changed. The pressure change is connected with the change from the condensation of water to the evaporation of water.

References

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