Environmental and In Situ SEM/TEM

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In situ characterization of microfiltration membranes in the ESEM - Results and limitations

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Polymeric microfiltration membranes consist generally of several different layers and are used in a great variety of applications such as waste water treatment or filtration of colloids and particles in the beverage industry. The structures of such membranes are of microscopic size, but up to now most methods used for the characterization of such membranes yield only macroscopic parameters [1]. But *in situ* experiments in the environmental scanning electron microscope (ESEM) enable the simultaneous measurement of both microscopic and macroscopic parameters and their correlation may provide maximum insight into the system [2]. And with such experiments not only the investigation of the structure of layered membranes, but also the interaction of their pore walls with water is possible.

The investigation of wetting and drying of cooled membranes in the ESEM gives on the one hand results about the number of dry surface pores and of their mean diameter as a function of time (microscopic parameters). On the other hand simultaneously the change of the temperature at both membrane surfaces can be measured (macroscopic parameter). The former provides information about the membrane structure at the surface, the latter information about the wetting and drying process in the interior of the membrane and also about its inner structure. The crucial point is the extremely poor heat conductivity of the membrane. Therefore the temperature of the individual layers is mainly dependent on their water content.

But accurate temperature measurements at the membrane surfaces are a great challenge. The membranes have a thickness of around 150 µm (see Figure 1.), therefore the head of the thermocouple should be as small as possible. Two types of thermocouples were tested; a T type (copper, constantan) and a K type (chromel, alumel). Their diameter was around 60 µm. Both are suitable for the respective temperature range. Fixing them with a thermally conductive adhesive to the membrane surface did not prove to be successful, because the adhesive penetrated too deep into the membrane. Therefore, thermal contact was realized by only pressing the micro thermocouples against the membrane surface by use of their own flexible contact wires (see Figure 2.).

Figure 3. shows temperature characteristics recorded with both types of thermocouples. It is immediately obvious that all measurements show the same temperature profile, but the absolute values measured differ for the two types. The reason is the different heat conductivity of the contact wires of the two types of micro thermocouple. The values measured with the T type are higher due to the excellent heat conductivity of copper, which causes a heat flux from the much warmer surrounding via the contact wires to the thermocouple head. This could also be confirmed by finite element simulations [3]. Therefore, the heat conductivity of contact wires should be as low as possible. Damage of the membrane material caused by the electron irradiation of the membrane poses another problem. The size of smallest pores that can be imaged is not limited by the resolution of the microscope, but by irradiation damage. Irradiation damage is much stronger in the presence of water, because the free radicals that are generated attack the material. Additionally also contamination of the specimen surface caused by irradiation can influence the wetting and drying process, because it changes the surface properties and thus the interaction of the surface with water.

Thus, the accuracy of the results gained by *in situ* experiments in the ESEM is not only limited by microscope parameters like the resolution, but to a much higher degree by the buildup of the experiment itself and also the type of material investigated.

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3. H. Reingruber, thesis, Graz University of Technology (2012), p. 48.

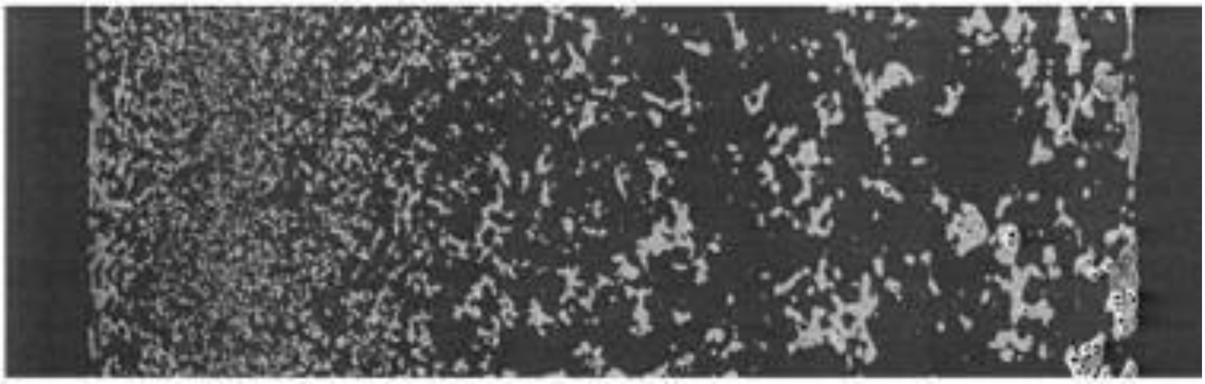

Figure 1. Cross-section of the membrane DuraPES®600 (image width: 174 µm).

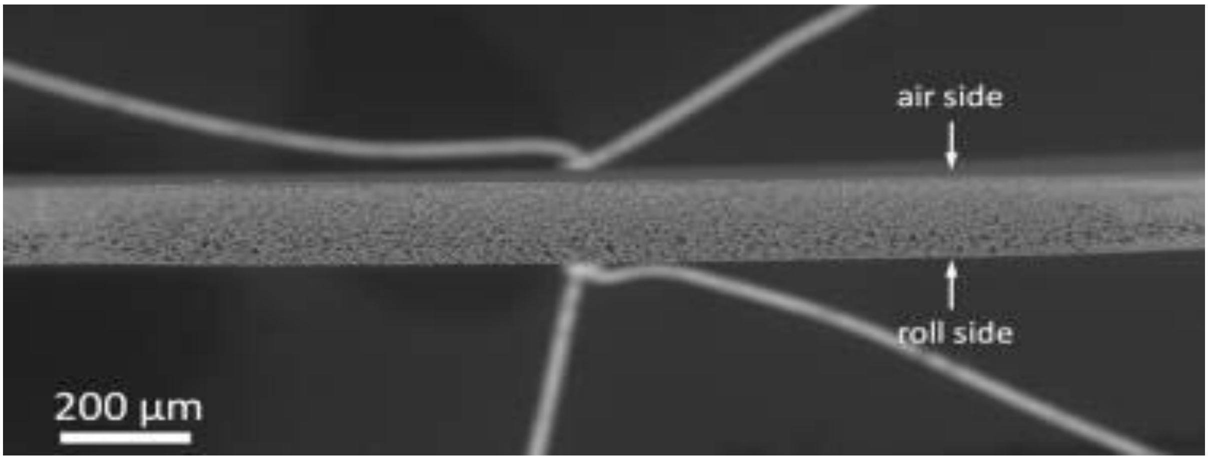

Figure 2. Cross-section of the membrane DuraPES®600 (image width: 174 µm); b: cross-section of the DuraPES®450 membrane with two micro thermocouples (CHAL 0005) contacting the membrane at both surfaces.

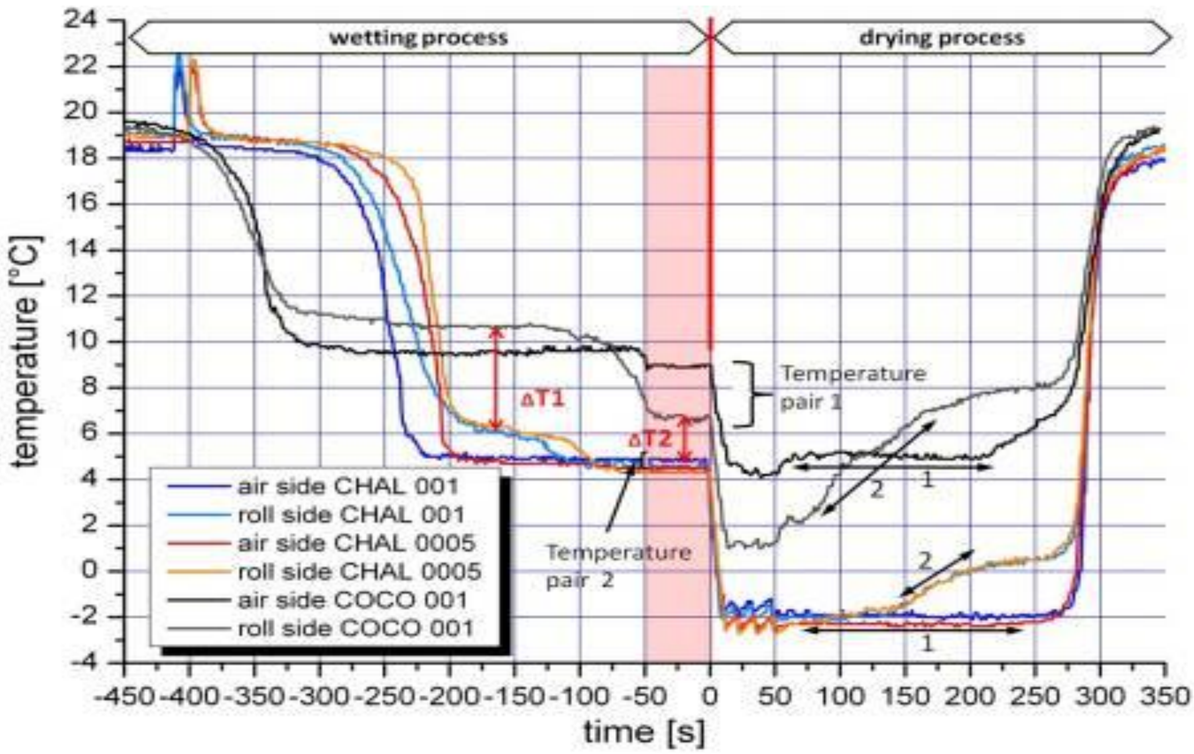

Figure 3. Temperature profiles recorded during the wetting and drying of the DuraPES®200 membrane in the ESEM using K type (CHAL 001, CHAL 005)) and T type (COCO 001) thermocouples.