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# Water Condensation / Evaporation Experiments in ETEM using a Thermoelectric Microcooler

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Efficient closed-cell specimen holders for Liquid Cell Transmission Electron Microscopy (TEM) has led to many advances over the last two decades but still suffer limitations due to their sealing membranes which hamper comfortable tomography approach, optimized chemical analysis and signal-to-noise ratio. 'Windowless' approaches in a dedicated Environmental TEM can not only overcome these limitations but provide opportunities to study water (liquid) condensation-evaporation experiments as required for example to study the water uptake of aerosols in the context of climate and atmospheric chemistry [1]. We present here a development and operationalization of such a windowless Wet ETEM method using a Peltier based MEMS chip compatible with a DENSsolutions Wildfire holder. The environmental conditions for the evaporation and condensation cycles require a pressure of 6-15 mbar and a temperature within a 0-11oC typical range. As for Environmental Scanning EM (ESEM) [2], these conditions can be achieved using a FEI-Titan ETEM instrument installed at CLYM Lyon, where a gas pressure of up to about 20 mbar can be maintained around a cooled specimen in the pole pieces gap. Adequate temperatures were obtained by placing at the tip of the Wildfire holder a thermoelectric cooler which can produce a temperature difference of ~40 K between the hot and the cold junctions. A 'micro' Peltier stage with adequate dimensions for being safely introduced in the ETEM was then connected to a custom printed circuit board (PCB) compatible with the DENSsolutions holder. The sample is deposited on a classical TEM carbon grid mounted in direct contact with the cold side of the Peltier device insuring its efficient cooling. Figure 1 shows the tip of the Wildfire holder when equipped with the Peltier system.

Initial experiments have been conducted on NaCl crystals used as model aerosols [1]. Owing to the fast cooling when a current is applied to the Peltier, a very efficient and easily controlled condensation was observed with a vapor pressure typically around 7 mbar in the column as illustrated by figure 2. These preliminary results will be discussed in comparison to similar experiments conducted with a classical cryo-holder (Elsa model from Gatan) cooled down by a liquid nitrogen reservoir as previously performed [1, 3]. As a first statement it appears that a much better and faster stability, resulting in a much better knowledge of the exact relative humidity (RH), is obtained with the proposed solution. Improvements of the setup are still possible. These results offer new perspectives for the study of the deliquescence or efflorescence of aerosols through condensation and evaporation cycles which can then be easily performed and reproduced [4].

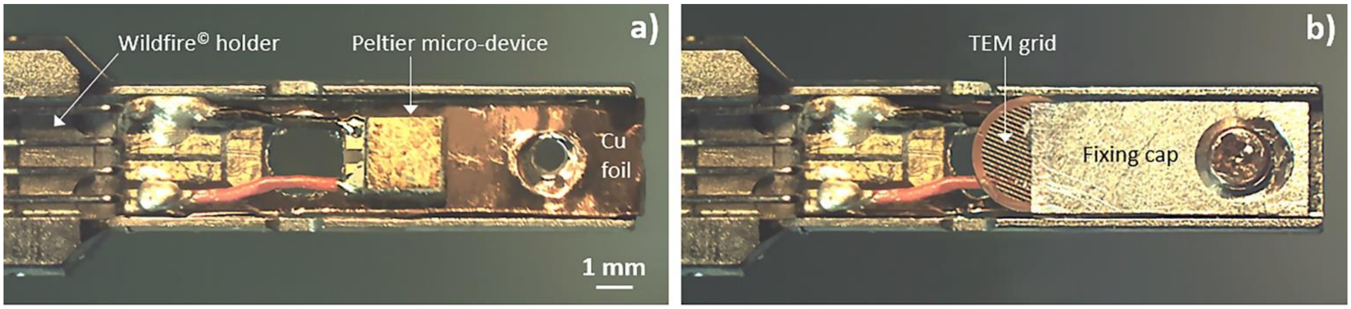


**Figure 1.** Montage of the micro Peltier system, including a Cu support, a PCB (a) and a fixing cap maintaining the TEM grid (b) on the tip of a Wildfire heating holder from DENSsolutions.

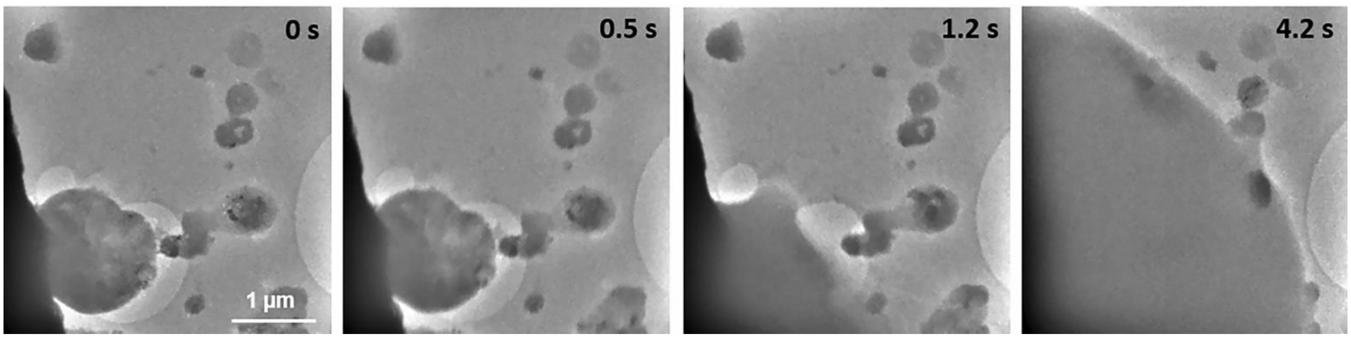


**Figure 2.** sequence of low mag bright field images (Gatan OneView camera) showing the fast condensation of water on a TEM carbon grid with NaCl and some other crystals (Titan ETEM, 300 kV, 7 mbar of water vapor; the temperature is estimated to about 6°C). The Peltier stage is powered at 0.5 s (image slightly out of focus); the droplet appears within less than one second from the grid corner (dark area on the left); the final image shows the extension of the water drop just before shutting the current down.

References:

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