New generation environmental in situ TEM holder for gas cell studies across multiple platforms

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In situ (scanning) transmission electron microscopy ((S)TEM) experiments in gases became widely possible with the development of sandwiched MEMS-based samples carriers (e.g. Nano-Chips) – gas Nano-Reactors. Various stimuli like heating or biasing in gaseous environments have been employed to study materials synthesis [1], catalyst [2], ferroelectrics [3], resistive switching [4] and more. The majority of the commercial in situ (S)TEM sample holders, however utilize only four electrical contacts that limit the combination of stimuli that the user can simultaneously apply. Real life applications, like proton exchange membranes, fuel cells, and non-volatile memory, etc, on the other hand, require combined application of thermal and electrical stimuli at ambient environmental conditions and thus, higher number of electrical signals.

In this work we present our new platform for in situ and operando environmental (S)TEM experiments. The heart of this platform is a newly designed holder that has eight electrical connections and allows the application of thermal and electrical stimuli in ambient environmental conditions. This is achieved through a newly designed dual chip environmental cell with an increased number of contacts. Additional electrodes are used to characterize the electrical performance of a FIB lamella sample either in 2- or 4-contact mode. The holder has a removable tip that has generic design and fits different brands of TEM, which substantially improves the correlation of the same sample between different TEM platforms. Furthermore, the design of the holder allows to rotate the tip making it suitable to both, TEM and STEM operation.

Using the new holder, it is possible to achieve high resolution at ambient gas pressure as obvious from the TEM image below. When rotating the tip by 180 degrees, the sample will become on top, which is beneficial for the high resolution STEM imaging. Since the tip flipping is done without the Nano-Reactor disassembly, it is possible to observe the same sample in different modes and easily trace the same region of interest. We will also demonstrate the capability of the holder in terms of elemental analysis using EDS and EELS analysis. We will show a few experimental examples of the new environmental operando system and explain how our new platform can be used in correlative studies involving different experimental methods like in situ TEM, SEM and beamlines.

Graphic
During the in-situ experiments, the user has full flexibility to define if the sample should be on top (better for STEM imaging mode) or on the bottom (better for TEM imaging mode). Therefore, the user can simply flip the tip 180 degrees (as seen in Figure a-c). Despite the rotation of the tip, the user can always preserve the same environmental and stimuli conditions constant. Figures d-f) show that the same tip is mounted on Thermo Fisher and JEOL holder bodies. The high resolution image of a nano-particle at ambient pressure in TEM (left) and STEM(right) imaging modes. The inset in the TEM image correspond to the sample place of the sample imaged in the STEM mode.

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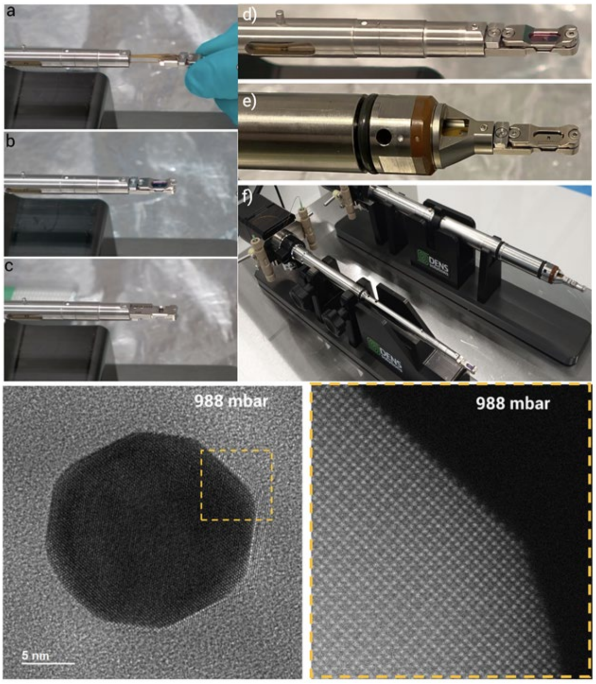
Graphic:



Keywords:

In situ, cross-platform, TEM, beamline

Reference:

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