

Ultra-High Vacuum Aberration-Corrected STEM for in-situ studies

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Aberration-corrected scanning transmission electron microscopes (ACSTEMs) can resolve all the individual atoms, including impurities, in graphene and other 2D materials [1]. They can also record energy-dispersive X-ray spectra (EDXS) and fine-structure electron energy loss spectra (EELS) from individual atoms [2-4], and can operate at beam energies below the knock-on displacement threshold, thus minimizing or altogether avoiding radiation damage. These capabilities are allowing the structural, chemical, electronic and magnetic properties of matter to be examined in an unprecedented level of detail. They will become even more powerful if we can precisely control the sample environment and modify the sample structure in-situ.

Achieving these goals requires a flexible micro-laboratory inside the microscope, operating in ultra-high vacuum (UHV). We have therefore developed a new sample chamber, shown in a side section in Fig. 1, for the Nion UltraSTEM [5]. The chamber is 120 mm high, a 60 mm increase relative to the regular sample chamber. The gap of its condenser-objective lens (OL) is 4 mm, and the polepiece shape has been optimized for low chromatic aberration C_c rather than low spherical aberration C_s (which is corrected by the UltraSTEM's C_3/C_5 aberration corrector), with $C_c = 1.0$ mm and $C_s = 1.1$ mm. The outer magnetic circuit was “bulked up” to allow for the extra magnetic flux due to longer polepiece pedestals, and the resultant configuration is capable of operating in a double crossover regime (with lowered OL aberrations coefficients) at 40 keV.

There are 3 new ports entering from the lower half of the chamber, at 25° to the horizontal plane, with an unobstructed view of the entrance (lower) side of the sample. This means that they can be used for deposition or ion bombardment/etching of an untilted sample in the observation position at the center of the polepiece gap. The lower half of the chamber also has a custom-made Ti-sublimation pump of about 500 liters/s pumping capacity, and it provides space for a secondary electron detector (not shown). When the sample chamber has been baked at the maximum temperature of 140 °C, the Ti-sub pump should permit the vacuum at the sample to reach the 10⁻¹¹ Torr range. A leak valve has also been provided, for immersing the sample in different gas atmospheres. The sample chamber is separated from the CFE gun of the microscope by two intermediate pumping volumes, which give about 10⁷:1 differential pumping isolation. This arrangement readily allows operation with a sample chamber pressure of around 1x10⁻⁵ torr. The upper half of the chamber mainly houses the microscope's 5-axis sample stage. It has 4 line-of-sight ports leading to the sample, which however cannot “see” an untilted sample. Six of the chamber's principal ports terminate in CF40 conflat flanges, which makes it possible to use conflat UHV windows and other standard vacuum attachments.

The sample stage can accept two very different types of sample holders (Fig. 2): the regular range of Nion side-insertion sample holders, which includes fixed tilt, double tilt and electrical/heating cartridges, or a 32x15x1 mm sample puck, which is inserted at 90° to the regular sample port. A magnetically coupled UHV arm with a grabber allows the sample puck to be removed from the

microscope and transferred to an auxiliary vacuum chamber. The auxiliary chamber, separate from the ACSTEM, provides facilities such as scanning tunneling microscopy (STM) [6], sample deposition, and other techniques of surface science.

The new system has recently been installed at the University of Vienna and is being tested. Results from the system will be reported at the meeting.

References

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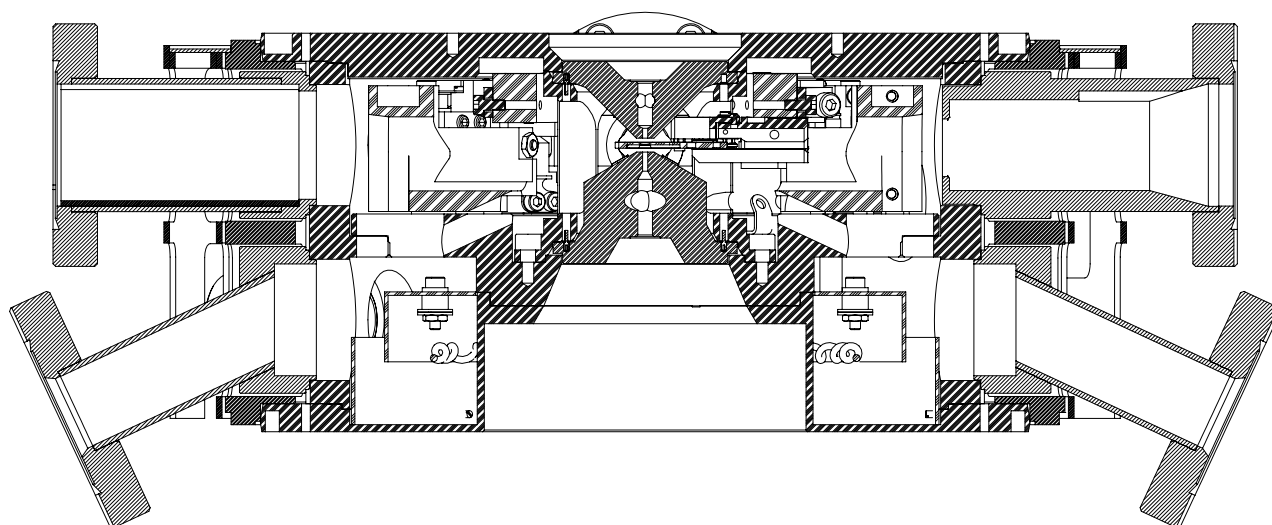


Figure 1. Cross-section of the UHV sample chamber, with an STM puck in the OL polepiece.

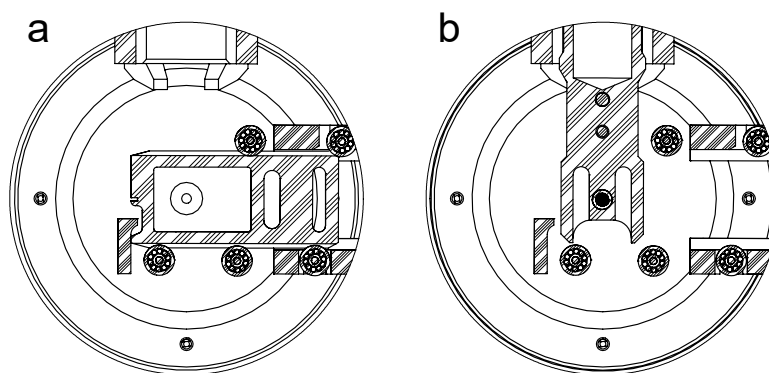


Figure 2. Horizontal cross-sections showing the OL region of the microscope operating with a) scanning tunneling microscope (STM) puck, b) regular Nion sample holder.