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A Materials Scientist's CANVAS: A System for Controlled Alteration of Nanomaterials in Vacuum Down to the Atomic Scale

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To get a true understanding of materials' properties, one has to assess their structure down to the atomic level. The next step is to enhance these properties by controlled and atomically precise alterations, such as the introduction of dopands into semiconductors. While investigations of materials are usually carried out in high-resolution electron microscopes, their alteration typically has to be done in separate devices in controlled atmospheres. This leads to the problem of transferring the samples between various devices, which is troublesome for numerous reasons. A major issue is the matter of contamination of samples during the transfer, which is especially critical for low-dimensional materials due to their high surface-to-volume ratio.

To mitigate contamination and provide a controlled environment, ultra-high vacuum (UHV) systems are widely used in various scientific fields as well as in many technical and industrial facilities. The setup of larger UHV systems usually involves heavy large-scale constructions. This is especially true for transfer systems, which are often realized using magnetically coupled arms or wobble sticks. While this is feasible for transfer distances up to some meters, for longer distances or situations where a straight linear transfer is not possible, one needs to resort to detachable transfer chambers (vacuum suitcases). The hassle of handling these suitcases involving attachment, pump-down, optional bake, transfer, venting and detachment is perfectly acceptable for transfers between different sites, perhaps even in different cities or countries. On the other hand, for transfers between UHV systems in the same building, this is often overly complicated and time-consuming.

To overcome this drawback, we have designed and built a novel arbitrary-length UHV transfer system that allows direct connection of different devices even located at different floors of a building. To allow for a seamless interchange of samples between the devices, we use a new sample-holder design which can be accommodated by all devices in the system. For interfacing with our UHV scanning transmission electron microscope (Nion UltraSTEM 100), a new stage module fitting both the standard and the new holders was designed and installed [1]. For inter-device connection, the sample holder "pucks" are loaded into small "cars" that can be moved inside standard UHV pipes by hand using magnetic coupling. Each car can transport up to 3 pucks, which can then be loaded and unloaded via standard UHV linear transfer arms. The magnetic coupling of the cars and their puck fastening mechanism were optimized to allow driving through most of the standard UHV components including flexible bellows and gate-valves (cf. Fig. 1a). While these connections are one-dimensional, it is also possible to branch off using turnouts consisting of two UHV pipes joining at a small angle (10–20 degrees (cf. Fig. 1b)).

This design allows the construction of a vast UHV system with a variety of integrated devices enabling the controlled alteration of nanomaterials in vacuum down to the atomic scale (CANVAS). The CANVAS-system spans over two floors and consists of the following parts: An aberration-corrected UHV scanning transmission electron microscope NION UltraSTEM 100 [1,2] equipped with additional pumps and a leakvalve system at the objective stage. This allows to control the atmosphere at the sample while imaging at atomic resolution. The typical base pressure at the sample is in the low 10^{-10} torr range and can be increased by leaking in high-purity gases up to the low 10^{-6} torr range. Further devices in the system are: an atomic force microscope GETec AFSEM [3], a plasma source SPECS MPS-ECR [4], a vacuum thermal evaporation (VTE) source RIBER S40 [5], multiple electron beam evaporators Focus EFM3 [6] and a 6W 445 nm-wavelength diode laser [7], all mounted at a UHV chamber. The system is also equipped with a storage option for up to 72 samples, a heating stage and an integrated glovebox MBRAUN LabStar under argon atmosphere (residual H_2O and 0_2 typically < 10 ppm) [8]. The glovebox is connected to the UHV system via a loadlock and is equipped with devices for sample preparation by mechanical exfoliation. The loadlock allows the insertion of samples into the UHV-system via an entry or via the glovebox and is fully computer-controlled to automatically handle venting, pump-down and bake procedures. Figure 2 shows an overview of the system.

The CANVAS-system allows the controlled alteration of materials [9-13] and even the growth of new ones [14] by utilizing various parts of the system and quickly moving samples between them. Typical workflows include experimental steps like the alteration of a materials by thermal evaporation or plasma irradiation augmented by quick checks at atomic resolution at the STEM. The integrated storage system is kept at pressures in the low 10^{-9} to 10^{-10} torr range, which is practical for the controlled long-term storage of samples.

The system has been installed at the University of Vienna over the past years and is now in full operation. One of the advantages of the design is its versatility allowing the easy extension or remodelling of the current system, by adding or changing UHV components respectively. While we tried to highlight a few typical use-cases here, a comprehensive description of the CANVAS-system would be beyond the scope of this publication. Moreover, the combination of existing as well as the easy addition of new devices allows for the design and implementation of an unlimited number of future experimental setups [15].

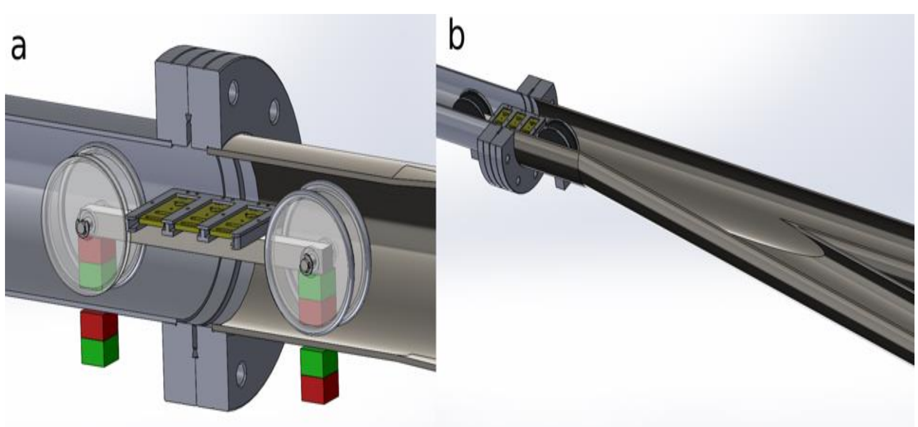

Figure 1. Renderings of the car used in the CANVAS transfer system. (a) Car driving through a flange. (b) Cut-out rendering of a turnout branching of a UHV pipe.

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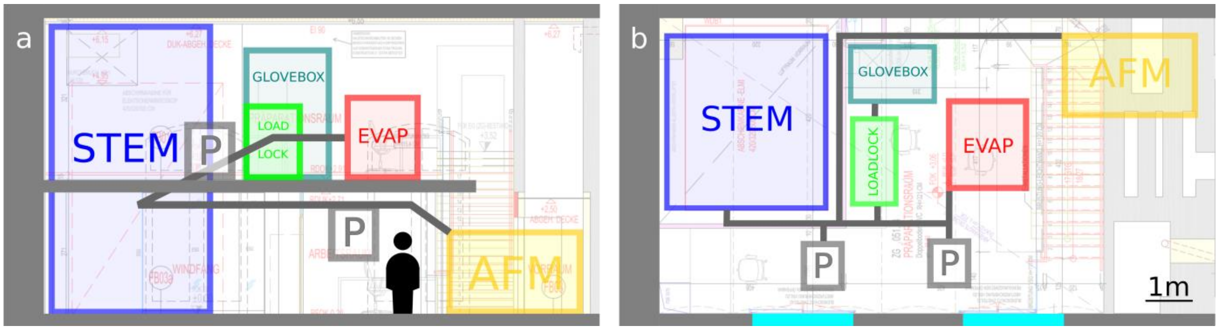

Figure 2. Cross-section (a) and top-view (b) of the Vienna lab showing the CANVAS-system: scanning transmission electron microscope (STEM, blue), loadlock and glovebox (green/teal), evaporation chamber (EVAP, red), atomic force microscope (AFM, yellow) and sample storage (P, grey).

References:

[1] MT Hotz et al., Microscopy and Microanalysis **22**(S3) (2016), p. 34. doi:10.1017/S1431927616001021
[2] OL Krivanek et al., Ultramicroscopy **108**(3) (2008), p. 179. doi:10.1016/j.ultramic.2007.07.010
[3] GETec Microscopy, https://www.getec-afm.com/afsem/ (accessed Feb. 18, 2022)
[4] SPECS, https://www.specs-group.com/nc/specs/products/detail/mps-ecr-an/ (accessed Feb. 18, 2022)
[5] Riber products, https://www.riber.com/product/s40-st-hl-cl-dz-series/ (accessed Feb. 18, 2022)
[6] FOCUS EFM3, https://www.focus-gmbh.com/efm-evaporators/efm-3/ (accessed Feb. 18, 2022)
[7] Lastertack, https://lasertack.com/en/6w-445nm-laser-diode-nubm44 (accessed Feb. 18, 2022)
[8] MBRAUN Glovebox Workstations, https://www.mbraun.com/en/products/glovebox-workstations.html (accessed Feb. 18, 2022)
[9] H Inani et al., The Journal of Physical Chemistry C **123**(20) (2019), p. 13136. doi:10.1021/acs.jpcc.9b01894b
[10] A Trentino et al., Nano Letters **21**(12) (2021), p. 5179. doi:10.1021/acs.nanolett.1c01214
[11] GT Leuthner et al., 2D Materials **8**(3) (2021), p. 035023. doi:10.1088/2053-1583/abf624
[12] K Mustonen et al., Advanced Materials (2022), p. 2106922. doi:10.1002/adma.202106922
[13] C Hofer et al., Nature Communications **10** (2019), p. 4570. doi:10.1038/s41467-019-12537-3
[14] G Zagler et al., 2D Materials **7**(4) (2020), p. 045017. doi:10.1088/2053-1583/ab9c39

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