



Enabling Tailored In Situ Electron Microscopy Studies at Low Temperature

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Addendum

Enabling Tailored In Situ Electron Microscopy Studies at Low Temperature

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In situ transmission electron microscopy (TEM) in combination with high-resolution imaging, diffraction, and spectroscopy enables real-time observation of dynamic processes at the atomic scale and the correlation between site-specific atomic structure and properties. The in situ techniques include the application of external stimuli such as temperature, electrical biasing, mechanical strain, and reactive environments. Traditionally, these experiments rely on specialized TEM holders, making the integration of multiple excitations challenging. Recent advancements in micro-electromechanical systems (MEMS) have enabled the development of in situ TEM chips giving access to experiments in both solid and liquid environments. By harnessing MEMS technology, we can design and fabricate chips that not only overcome the limitations of commercial solutions but also enable novel excitations, expanding the scope of in situ experiments.

This work presents the principles of our MEMS chip design, fabrication and use of 2D materials emphasizing customizability to expand experimental possibilities. By leveraging nanofabrication techniques, we develop MEMS chips that allow innovative, multi-modal in situ studies while allowing scalable wafer-level fabrication. 2D materials exhibit intriguing electrical and optical properties that can be tuned through strain engineering. In situ biasing and straining experiments in scanning electron microscopes (SEM) and transmission electron microscopes (TEM) can reveal the mechanisms that are responsible for the changes in properties. The SEM and TEM studies require careful and advanced specimen preparation and manipulation of 2D flakes that are a few nanometers in thickness and extend a few micrometers. In addition, the 2D materials can be used to design the function of MEMS chips used for tailored in situ experiments.

We have developed a computer-controlled transfer station for assembling 2D material devices tailored for in situ studies. The assembly process follows the method described in [1] using heating to control flake release. To enable the chip heating during transfer, a custom heating base of aluminum and PEEK was designed, based on COMSOL heat simulations. To secure the chips without disrupting the transfer of the flakes, a vacuum system applies downforce on the underside of the chips. The base features two mounting sites: one for standard 3mm TEM grids and another adaptable to various chip geometries. A rotation stage is mounted beneath the heated stage to enable high-precision alignment for Moiré pattern assembly. A control system, implemented in LabView, manages a Piezo motor for precise Z-movement. High-accuracy X and Y movements of the slab with the 2D flake enable precise flake placement with respect to the chip within an area smaller than 3 microns. Combined with controlled flake release, this ensures consistent and precise device assembly.

Our approach of tailoring the MEMS chips and utilizing 2D materials not only enhances experimental efficiency but also reduces overall costs through batch processing. We will demonstrate our first attempts at custom MEMS chip fabrication, provide insights into the process flow, and discuss future directions for in situ TEM experiments including studies at low temperature and methods to determine local specimen temperature at the position of electron beam exposure [2].

References

1. Diez-Merida J *et al. Newton* (2025) 1 100007.
2. The authors acknowledge the funding from the Knut and Alice Wallenberg Foundation. Authors would like to acknowledge Dr. Wan-Gyu Lee and his team at National Nano-fab Center, Republic of Korea for support in the nanofabrication process. This work was performed in part at the Myfab Chalmers and the Chalmers Materials Analysis Laboratory (CMAL).