

Transfer of Nanomaterials for in-situ TEM with electrical Currents

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Background incl. aims

In-situ transmission electron microscopy (TEM) has evolved to be a crucial tool for studies of dynamic processes in a large variety of specimens. While numerous examples exist that use gaseous/liquid environments, heating, biasing or the application of a mechanical force, studies in which the electrical current (and not bias) has been used as external stimulus are scarce. [1,2] A possible reason is the necessary controlled contacting of the specimen, which is challenging, especially for nanomaterials that need to be kept free from any source of damage and contamination. Here we present a preparation technique that allows a clean and almost damage-free preparation to focus on the materials during the application of electrical currents inside the electron microscope. [3]

Methods

The key element of the process is a holey silicon nitride membrane, which supports the nanomaterial during the transfer with the focused ion beam (FIB) technique using Ga⁺ ions. In brief, the specimen is first prepared conventionally on a TEM grid made of such a holey silicon nitride membrane (PELCO). In a TEM analysis, a suitable individual nanomaterial can be selected, as exemplarily shown in Figure A for a nanotube (NT) and a flake made of the misfit-layered compound LaS-TaS₂. [4] The grid is then loaded into a dual-beam (SEM-FIB) instrument and the nanomaterial with the supporting membrane is transferred to a microchip using a microneedle and FIB induced deposition (FIBID) of Pt/C (Figures B and C). The properties of the silicon nitride facilitate easy contacting of the membrane to the contact pads of the chip by FIBID. Finally, the membrane is removed in the central part to leave the nanomaterial as only conductive bridge.

Results

Using the above-described transfer technique, we have prepared a range of different devices based on nanomaterials and evaluated in detail the transfer quality. Figure D shows a HRTEM image of the transferred LaS-TaS₂ (NT and flake) sample revealing no contamination or charging. The limits of the methods were tested with a monolayer of WS₂, which was successfully transferred and revealed that Pt/C contamination during FIBID is kept well below a monolayer. For 1D nanomaterials, Ga implantation can be kept to zero, while for 2D materials or thin films, a small amount of Ga is implanted at

the edge of the specimen in the final step of removing the membrane in the central part. We will present exemplary experiments conducted on different prepared devices.

Conclusion

The development of a support-based transfer and contacting procedure of individual nanomaterials to microchips, e.g. used for in-situ TEM investigations, allows an almost artifact-free preparation of nanomaterial specimens for electrical in-situ TEM. Suitable nanomaterials can be selected by previous TEM analysis and correlative microscopy techniques. The resulting devices can be used to electrically characterize the nanomaterials and to dynamically study the effect of an electrical current on the sample. This includes Joule heating, which can be used to heat samples to extremely high temperatures, not reachable by standard heating in-situ TEM studies. Further possible investigations concern electromigration, electrical failure analysis or EBIC. In addition to in-situ TEM, the preparation method can equally be used to prepare devices on related microchips.

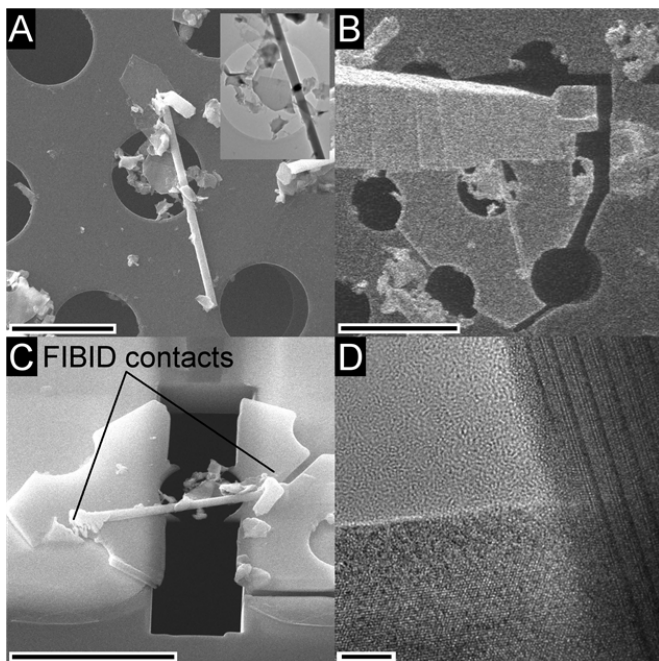
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Figure caption:

(A) SEM image of LaS-TaS₂ nanotube and flake. Inset shows a low-magnification TEM image of identical sample position. (B) SEM image of micro-needle holding membrane and nanomaterials after contacting and cutting free. (C) SEM image after milling of membrane in hole area of the chip with nanomaterials forming the only bridge between contacts. (D) HRTEM image of a flake and a nanotube (shown in (C)) after transfer. Scale bars are (A) 2 μm, (B,C) 5 μm, and (D) 5 nm.

Graphic:



Keywords:

in-situ-TEM, nanomaterials, sample preparation, focused-ion-beam

Reference:

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