

Towards customized in situ TEM Chips for “device-like” geometries

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Functional nanostructures and thin films have applications within the next generation of memory storage, energy storage, quantum computers and sensor technology [1,2]. Their characteristic properties originate from the nano or even atomic scale, necessitating the combination of high-resolution techniques and imaging of functional properties to better understand their structure-property relationships. Scanning transmission electron microscopy (STEM) can be used to capture the structure, chemical composition, and functional properties of these materials.

For in situ STEM characterization, the material of interest must be transferred to an electron-transparent region of a TEM chip. For thin films, this means depositing it on the membrane of a commercial TEM chip, or alternatively, transferring it by FIB lift-out or similar techniques. However, for larger systems consisting of nano- or micro-sized structures relevant for prototyping of new device concepts, this quickly becomes challenging, as the field of view required for such geometries may be many times larger than what is feasibly prepared by plan-view FIB lift-out. An alternative approach would be to fabricate these “device-like” structures directly on the TEM membrane. However, performing advanced lithography steps on a fragile, pre-made, electron-transparent window involves a non-negligible probability of fracturing it. We aim to solve this by fabricating TEM membranes directly from the substrate, etching electron-transparent windows from its back side after processing the front side. Similar work has been performed on customizable TEM chips for nanowires [3], but here we aim to utilize this approach for specimens requiring a larger field of view. The outlook for a simple, reproducible, fabrication process is many, as the advantages stretch across customizable window sizes and tailored chip designs. Additionally, it may expand the type of material systems that can be studied in situ, as-grown and in-plane without the need FIB lift-out.

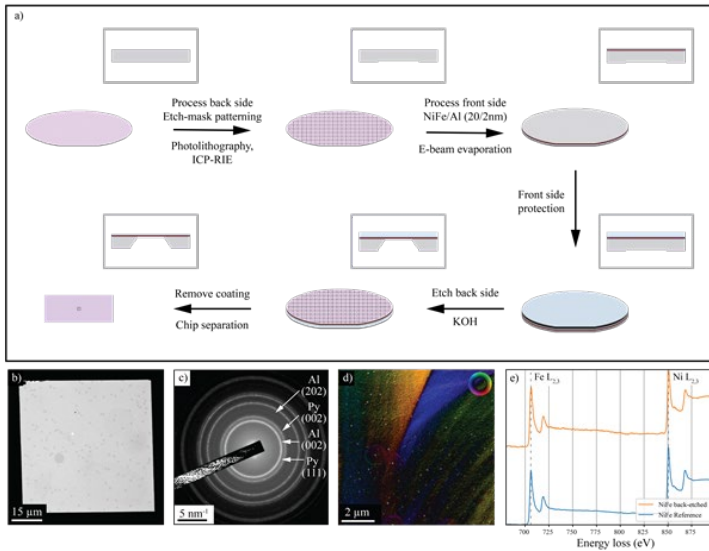
The first step in achieving a robust protocol for the fabrication of customizable TEM chips is to optimize the back-etch to release electron-transparent membranes. As proof of concept, back-etched TEM chips were fabricated for a simple ferromagnetic Ni₈₀Fe₂₀ thin film (Fig 1.). Starting with a double-side polished silicon wafer coated with 30 nm Si₃N₄, the etch-mask was fabricated by patterning the Si₃N₄ on the back side of the wafer by photolithography and inductively coupled plasma reactive-ion etching (ICP-

RIE). A 20 nm $\text{Ni}_{80}\text{Fe}_{20}$ thin film was deposited on the front side by electron beam evaporation, and thereafter protected by AR-PC 5040 protective coating. The electron-transparent windows were released by immersing the wafer in a KOH bath, and consecutive removal of the protective coating. TEM characterization of the windows shows successful fabrication, with the $\text{Ni}_{80}\text{Fe}_{20}$ retaining its ferromagnetic properties. This provides further evidence that the sample preparation was successful and that the front side was sufficiently protected from the KOH etch.

The next steps will focus on fabricating $\text{Ni}_{80}\text{Fe}_{20}$ nanomagnet arrays by electron beam lithography on a similar $\text{Si}/\text{Si}_3\text{N}_4$ substrate with wires for in situ biasing. As such, this presentation will also show the next steps towards doing in operando studies of magnetic device concepts. [4]

Figure 1: a) Schematic of the TEM membrane fabrication steps. The insets show the cross-section of the wafer. b-d) Low-magnification STEM image, SAED pattern, and STEM-differential phase contrast (DPC) image of the back-etched $\text{Ni}_{80}\text{Fe}_{20}$ TEM window. Some particle contamination can be observed on the back-etched samples. e) EELS spectrum of the back-etched $\text{Ni}_{80}\text{Fe}_{20}$ thin film against a reference $\text{Ni}_{80}\text{Fe}_{20}$ specimen.

Graphic:



Keywords:

STEM, in-situ TEM chip, nanofabrication

Reference:

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