3D Nanoprinting of advanced AFM nanoprobes

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Introduction

Atomic Force Microscopy has become a standard technique in many fields of research due to its high resolution and versatility. Employing functionalized nanoprobes allows to measure materials properties at highest lateral and vertical resolution. AFM nanoprobes have to meet certain requirements to ensure high-performance reliably. High-resolution topography scanning is one of AFM's key advantages demanding ultra-sharp nanoprobe apices. At the same time, mechanical stability is mandatory for stable operation minimizing nanoprobe flex and related distortions. Another key property of nanoprobes is wear resistance, improving measurement reliability and lifetime. 3D nanoprinting via Focused Electron Beam Induced Deposition **(FEBID)** is ideally suited to fabricate advanced fully functionalized AFM nanoprobes[1].

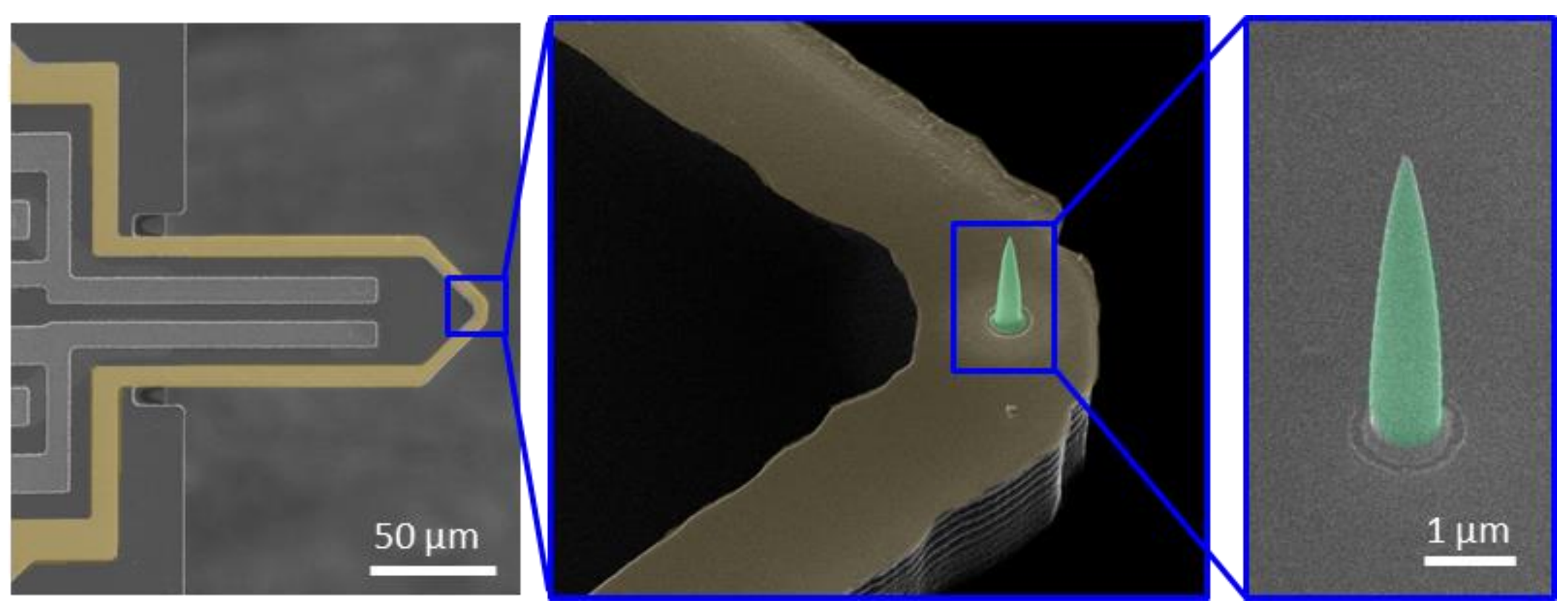

Figure 1: Pre-structured self-sensing cantilever with FEBID-based CAFM nanoprobe.

Conductive Nanoprobes

Link to paper

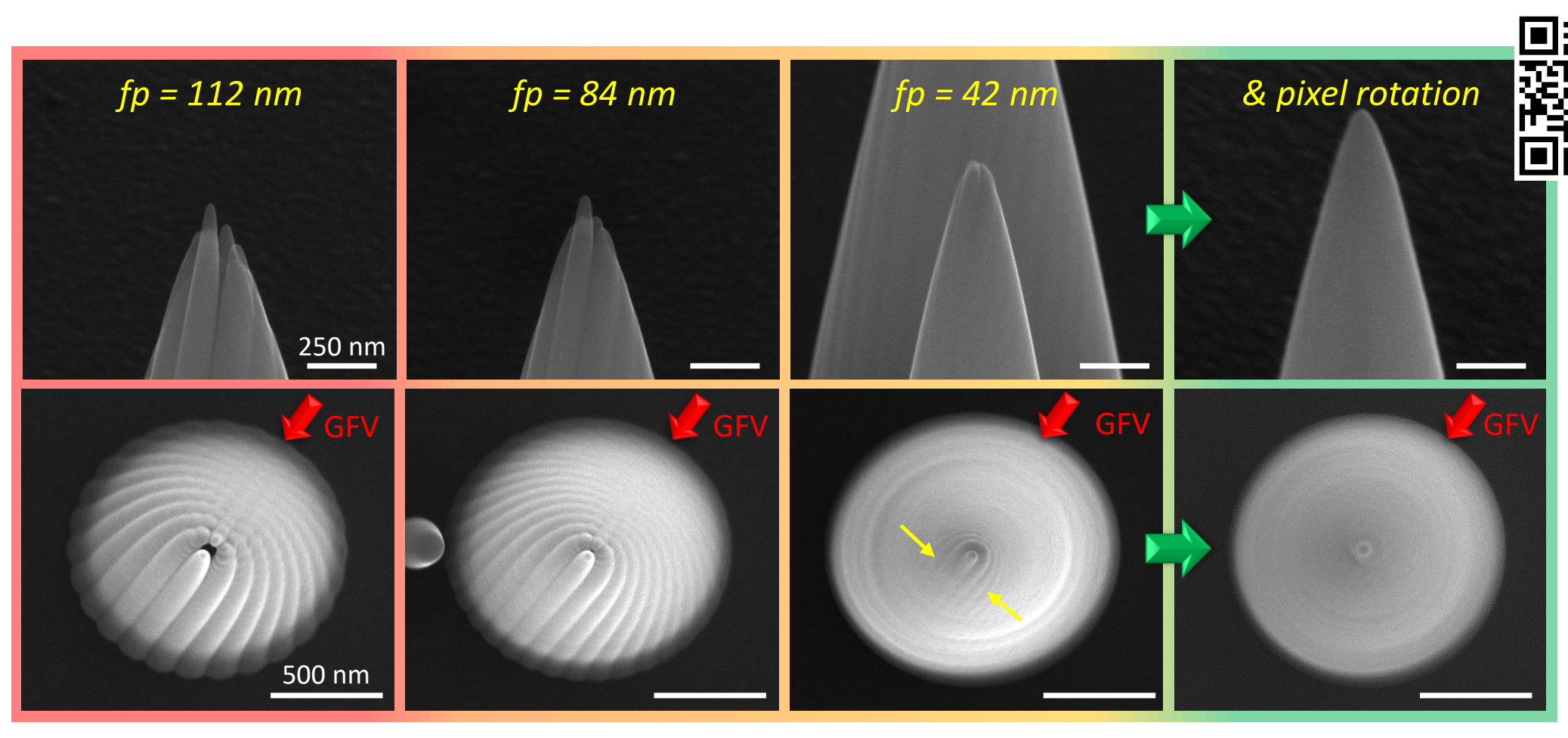

Figure 4: Parameter variations and design adoptions towards a controlled shape.

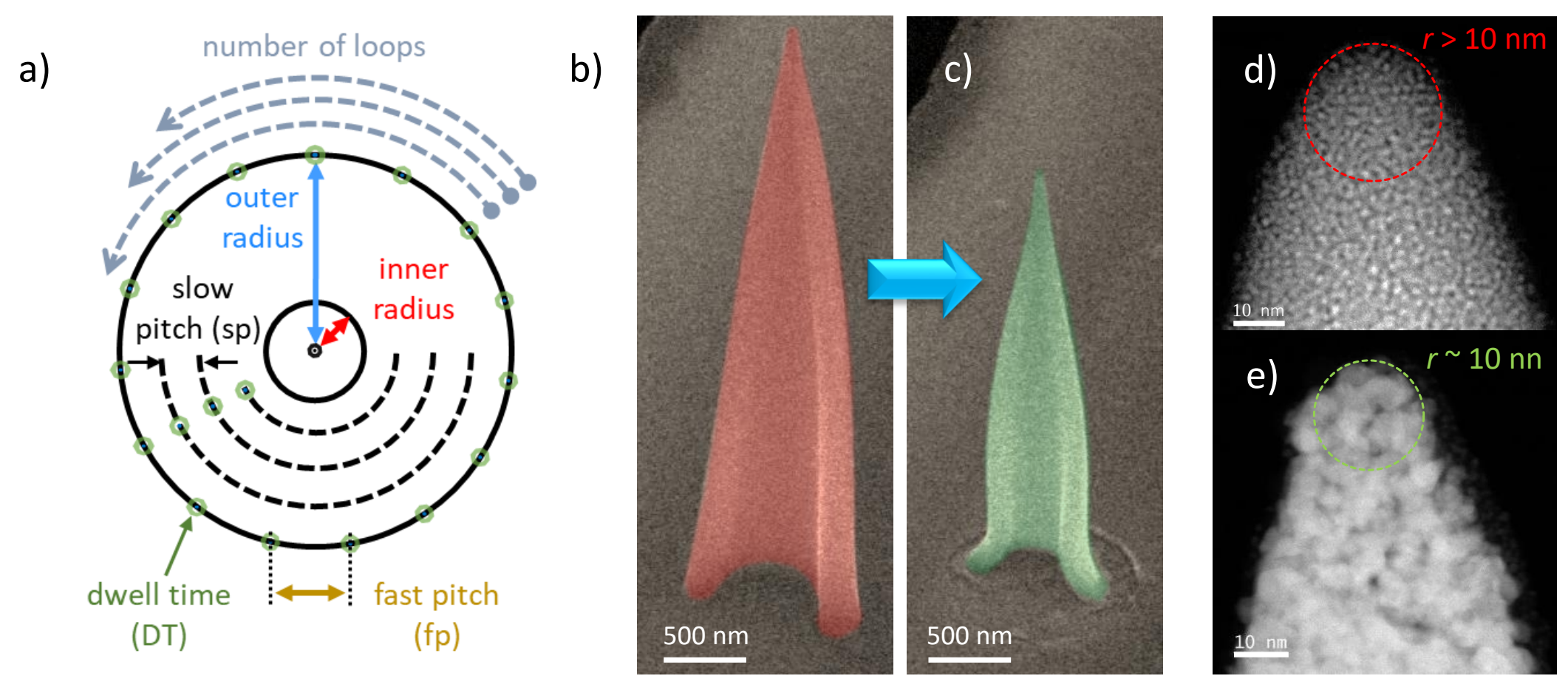

Figure 5: Hollow cone patterning design (a). Volume loss during purification for the example of a half hollow cone made on purpose for visualization (b, c). TEM images of the hollow cone apex before (d) and after (e) purification.

Conductive AFM is performed in contact mode, i.e. the nanoprobe is constantly in touch with the investigated surface. Hence mechanical stability and high wear resistance along low electrical resistance are targeted. To meet the desired constraints, the conductive nanoprobe exhibits a hollow cone design, made up of stacked concentric circles. Parameter studies and design adaptions were conducted aiming at a smooth surface and controlled transition towards a sharp apex. Due to the hollow character less material and thus deposition time is required, but more importantly, the entire structure can be penetrated be the electron beam for complete purification. The carbon removal is accompanied by a significant volume loss (Fig. 5 b,c) yielding even sharper apices terminated by individual crystallites (Fig. 5 d,e). This then results in high-resolution capabilities on par with commercial high-res probes, while providing excellent capabilities for CAFM, both mapping (Fig. 6 a-c) as well as spectroscopy (Fig. 6 d-f).

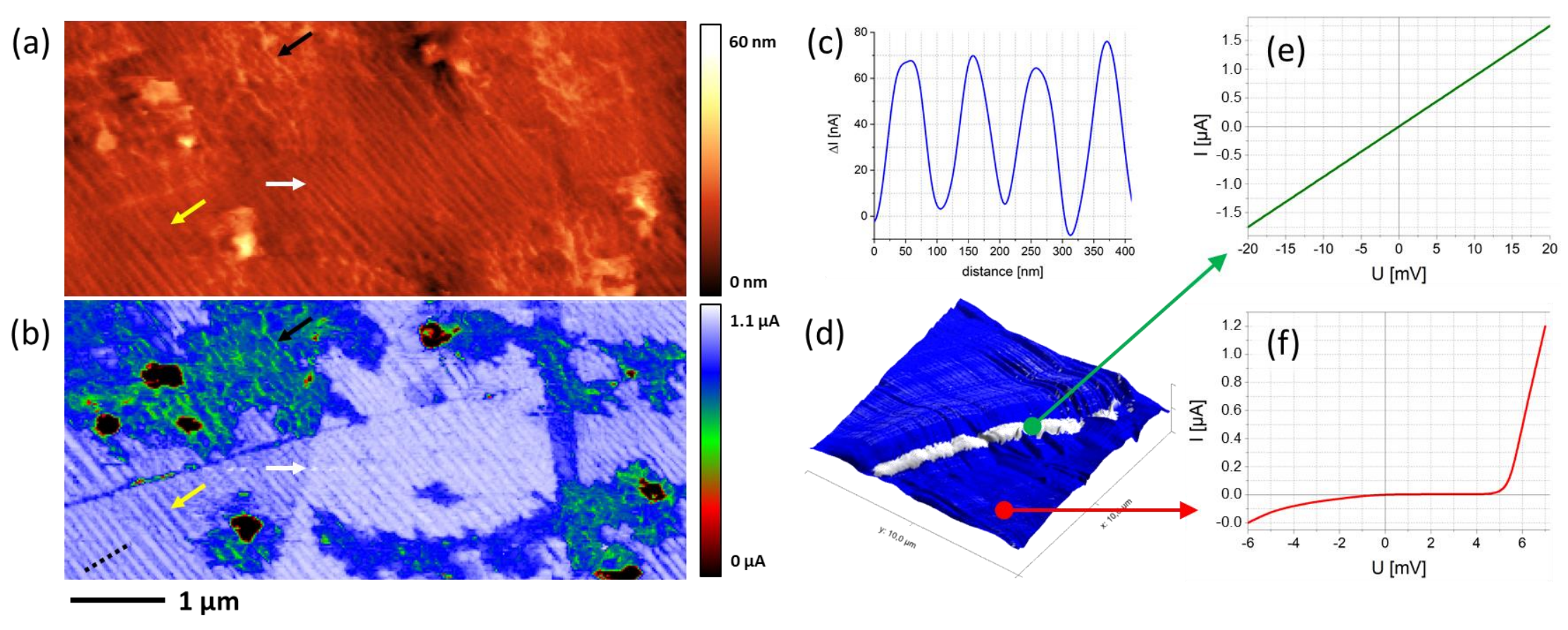

Figure 6: CAFM investigation of a thermally grown Cu surface, modified by CVD graphene layers and decorated with Sb particles (a-c). Copper fibrils are present in both topography (a) and CAFM map (b), graphene multilayers are only visible in (b) indicated by white arrows, insulating Sb particles appear black in (b). The line section (c) corresponding to the black dashed line illustrates the resolution performance. (d) depicts a Cu substrate (white) partially covered by MoS_2 (blue), with I-V spectra for both Cu (e) and MoS_2 (f) areas.

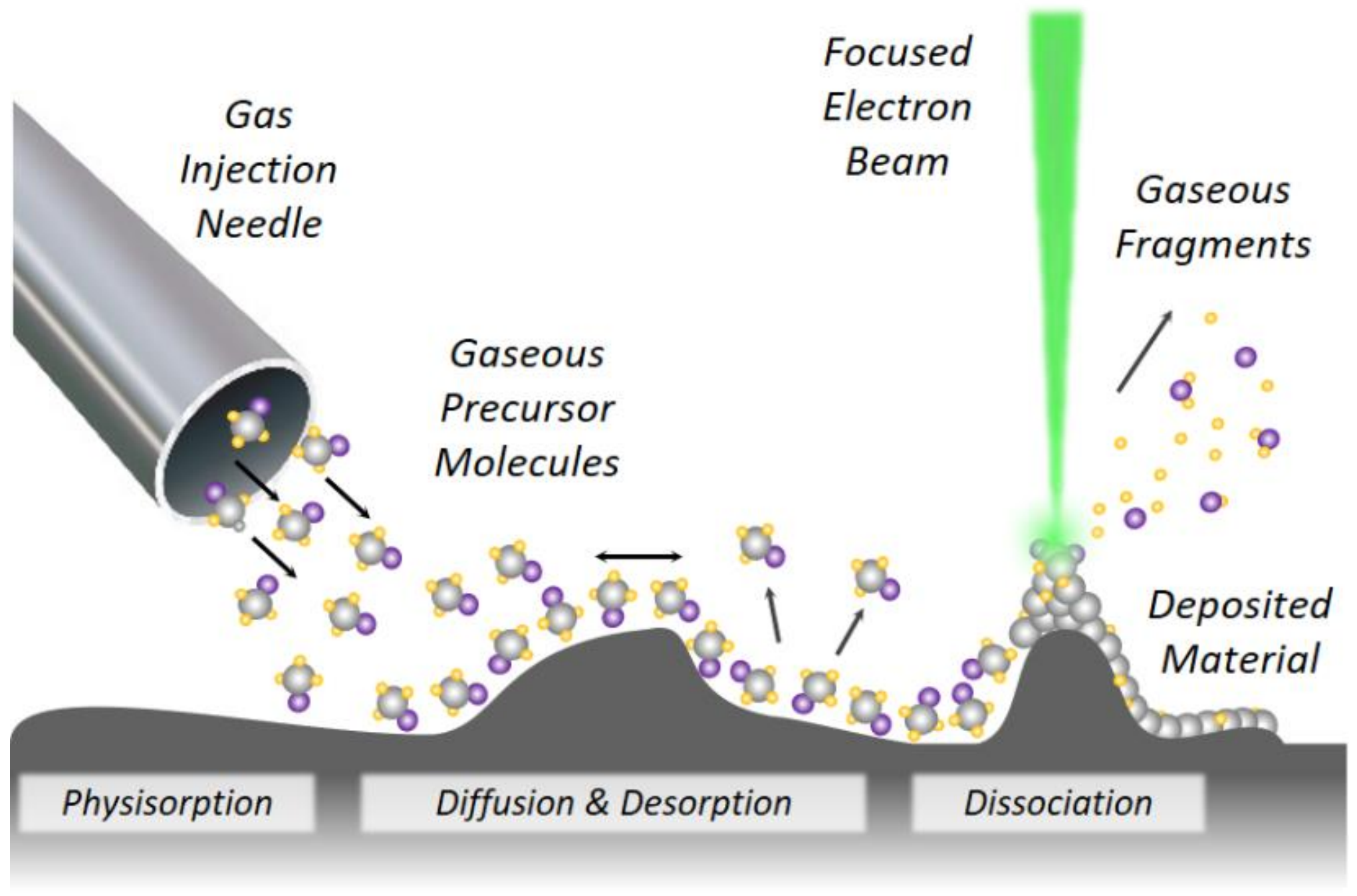

Figure 2: Basic principle of 3D-FEBID

FEBID is an additive manufacturing process at the nanoscale. A gaseous precursor, directed towards any arbitrary substrate, is decomposed upon interaction with the electron beam. Accurate control of dwell time and beam position allows direct deposition of desired nanostructures not requiring any masking routines. FEBID based 3D-nanoprinting allows high shape flexibility ranging from complex and delicate framework structures[2,3] to arbitrary 3D shaped objects[4].

Apex Quality

Mechanical Stability

Wear Resistance

Functionality

Thermal Nanoprobes

Link to paper

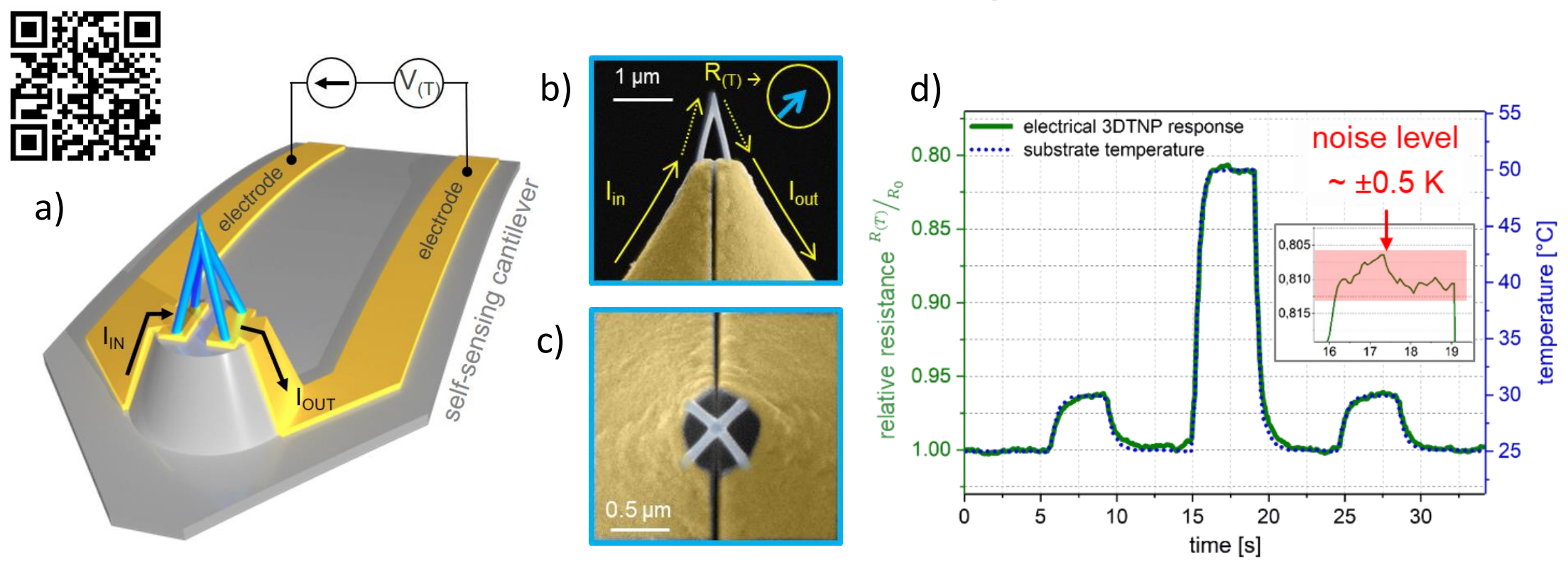

Figure 7: a) Functional scheme of the thermal nanoprobe, the thermistor is placed on top of a truncated, FIB cut Au covered cantilever. b) and c) show coloured SEM images of the fabricated nanoprobes. The graph in d) shows the temperature response to a heating chip.

The thermal nanoprobe is a thermistor type sensor which is designed as a tetrapod, fabricated on top of a truncated and FIB cut Au covered AFM cantilever. Design rules were derived from an FEM assisted design study enabling fabrication of thermal nanoprobes with vertical stiffness up to 50 N m^{-1}. Yet the legs of this structure are only around 100 nm wide and the overall small volume allows the nanoprobe to quickly adopt its temperature yielding a sensing rate of 30 ms K^{-1}. To improve the wear resistance and mechanical stability, a curing procedure was developed. The cured thermal nanoprobes were tested in contact mode AFM for up to 4 hours showing minor apex broadening (Fig. 8).

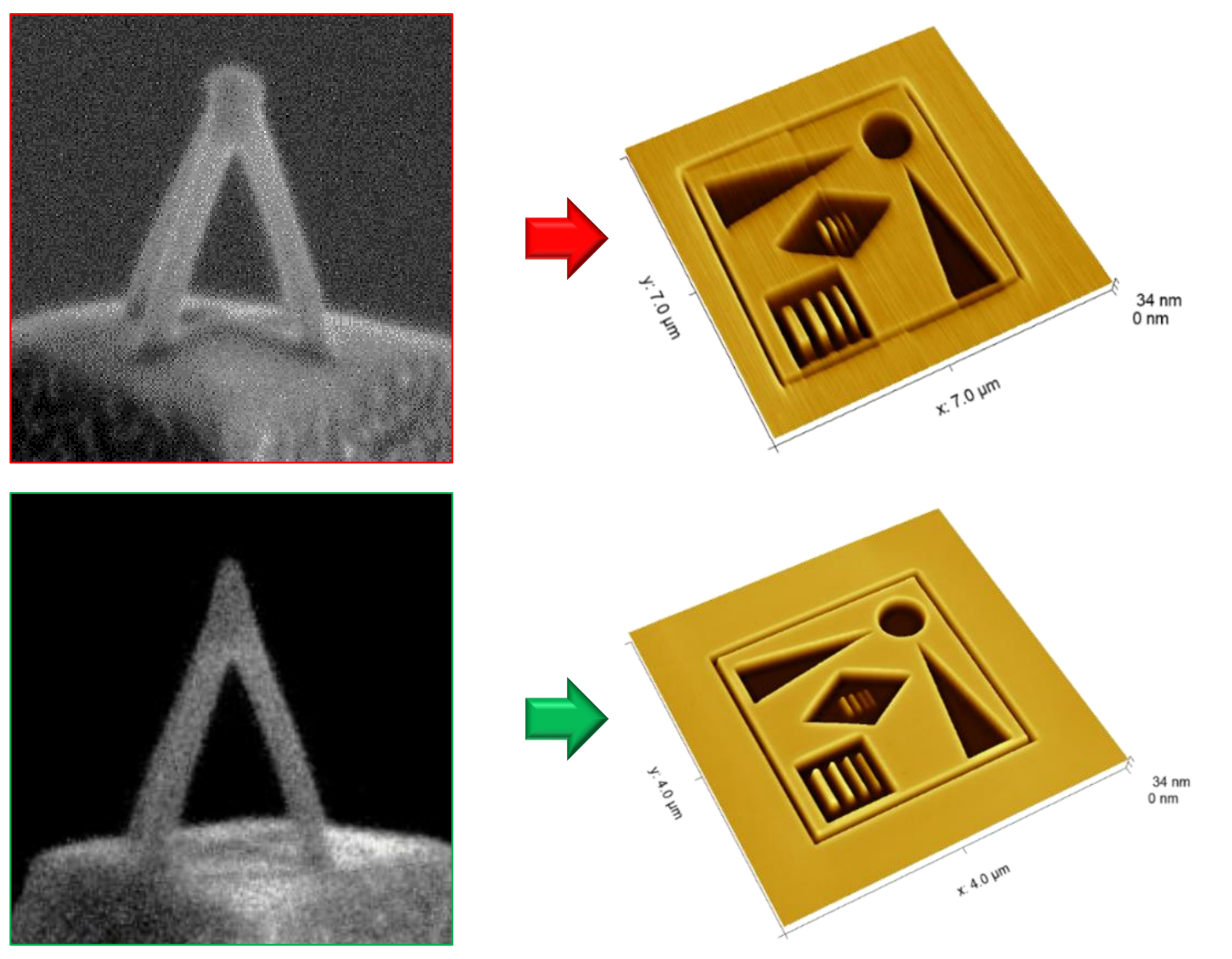

Figure 8: Comparison of uncured vs. cured thermal nanoprobes. SEM images and corresponding AFM images illustrate the effect of wear on the uncured nanoprobe, whereas the cured structure only shows minor wear.

Structures deposited from commonly used organometallic precursors exhibit a nanocrystalline microstructure. Depending on precursor dissociation efficiency, organic ligands can remain in the deposit forming a carbonaceous matrix hosting the metallic nanocrystals. This material configuration yields properties attractive for sensing applications since conductivity is based on hopping transport between individual crystallites, mediated by the matrix material. Further variability in materials properties is achieved via post-deposition irradiation in UHV or other atmospheres. Electron Beam Curing **(EBC)** takes place in UHV and aims at completion of precursor dissociation. Accompanied by slight grain growth, it enables precise control over electric conductivity and mechanical properties, e.g. stiffness and wear resistance. Irradiation in water atmospheres allows to entirely remove remaining carbon residues from the deposit, therefore termed **Purification**[5].

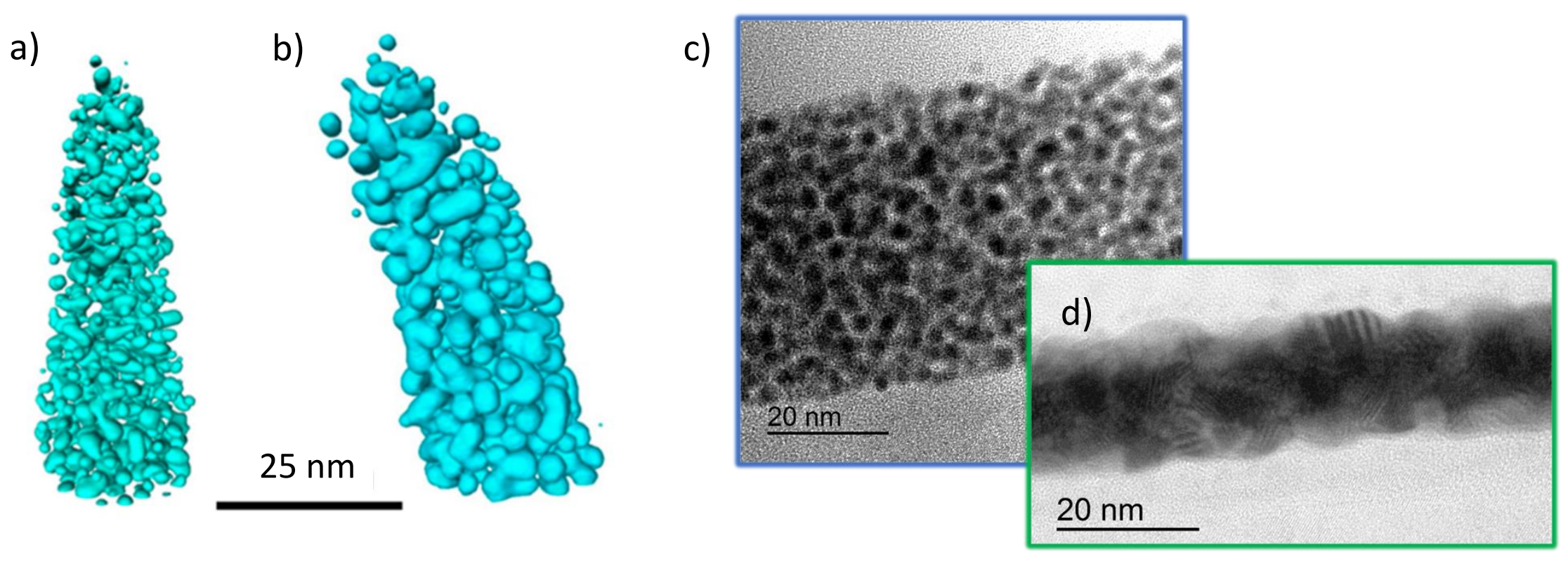

Figure 3: Electron tomography of nanocrystallites before (a) and after (b) EBC treatment. Microstructure before (c) and after (d) Purification process.

Magnetic Nanoprobes

Link to paper

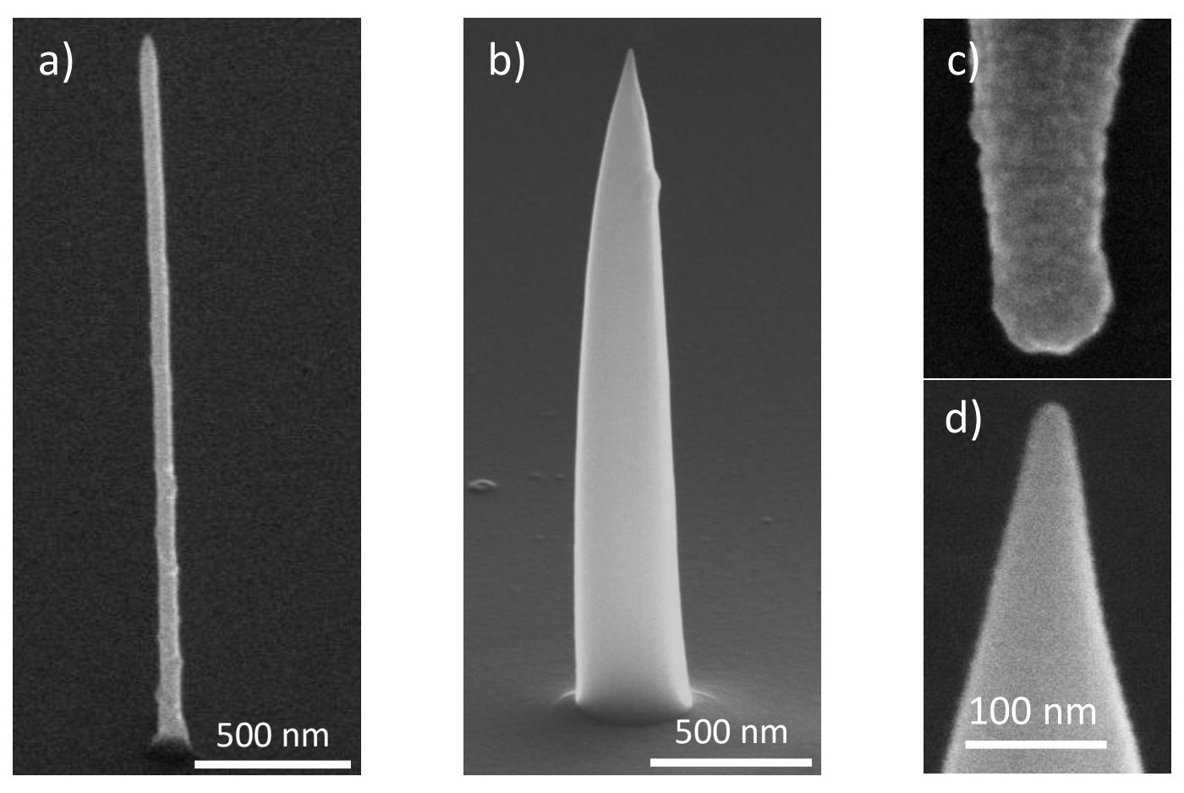

Figure 9: SEM images of a *single* pillar grown with a stationary beam (a) and the optimized α-Pillar (b), which is fabricated using a defocused electron beam that is successively brought back to focus resulting in a defined and sharp apex (d) as compared to coated nanoprobes (c).

A novel precursor comprising Co and Fe[6] enabled the fabrication of magnetic nanoprobes, based on an advanced patterning approach. Starting from a defocused beam that is successively refocused results in a mechanically stable structure terminated by a very sharp and controlled apex. MFM investigations on a magnetic heterostructure illustrate the enhanced resolution capabilities (Fig. 10 a) and the improved signal-to-noise ratio of the MFM phase signal (Fig. 10 b). Wear and aging tests of nanoprobes proved their wear resistance (Fig. 10 c) as well as long-time stability. Even storing at ambient conditions for one year resulted in virtually no loss in signal strength (Fig. 10 d).

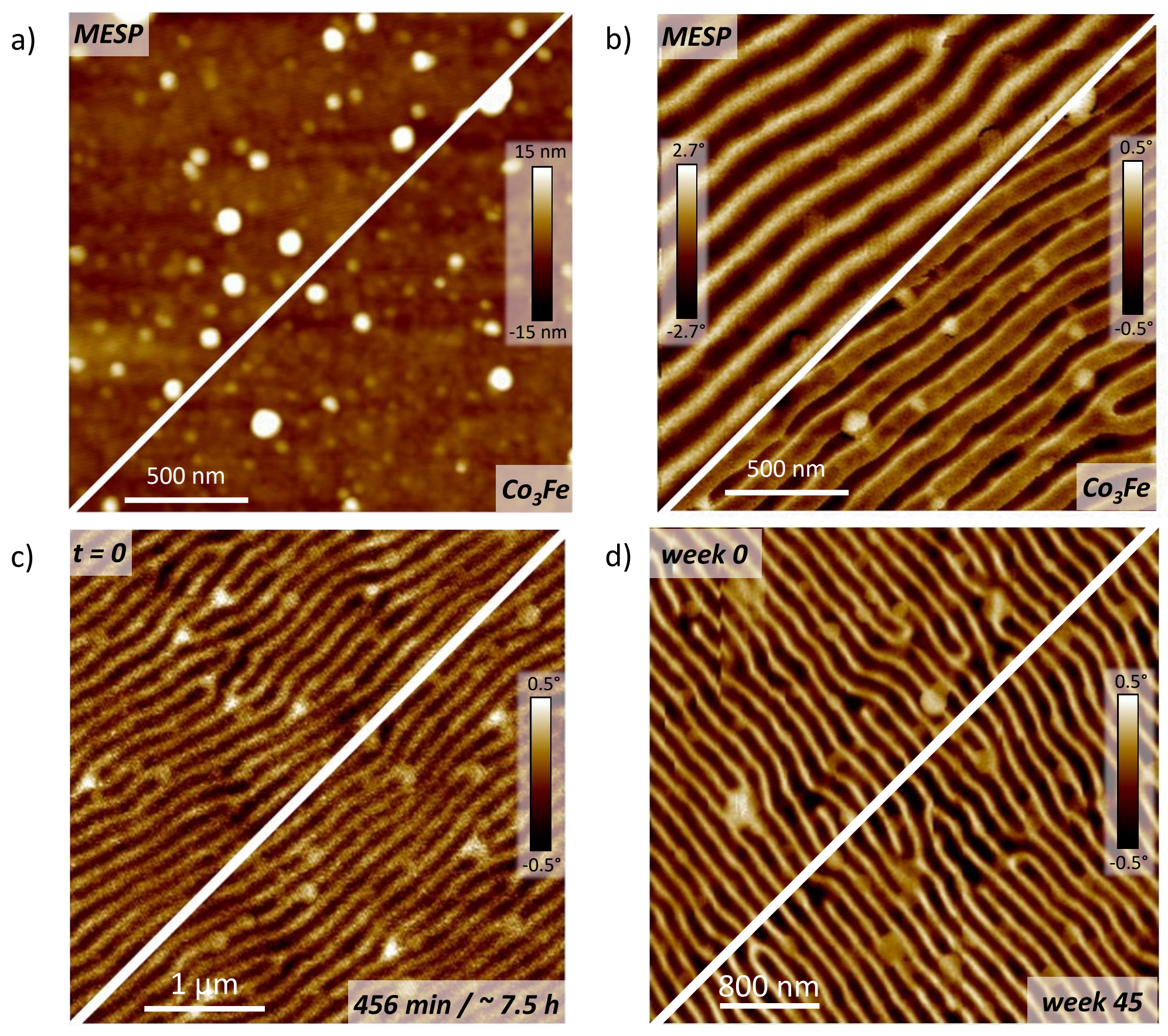

Figure 10: Comparing the α-Pillar to a conventional MFM probe: Topography (a) and MFM phase signal (b). Wear tests over several hours (c) and storing the probe for almost a year (d) had virtually no impact on MFM performance.

Conclusions

FEBID based 3D-nanoprinting is a capable technique for the fabrication of delicate and complex structures at the nanoscale. AFM probes in particular benefit from the design flexibility in terms of geometry and tenability of material properties. With the ongoing research in precursor chemistry novel nanoprobe designs are yet to be realized.

References

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