# Pushing Correlative Microscopy to the Next Level: Advanced Characterization of Plasmonic Nanostructures

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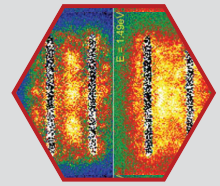
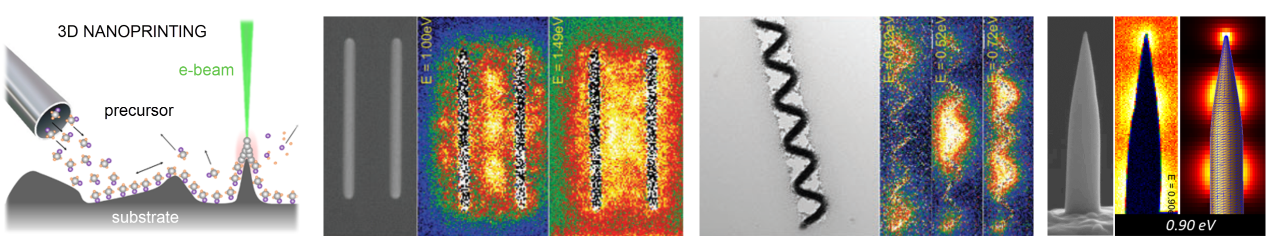



## Relevance of Plasmonic Structures and 3D Nanoscale Fabrication

Plasmonic nanostructures exhibit unique optical properties that enable cutting-edge applications in sensing, optoelectronics, and quantum technologies **[1]**. These structures concentrate light into sub-wavelength dimensions, enhancing electromagnetic fields at nanoscale "hot spots." While planar plasmonics has been extensively studied, fabricating 3D architectures introduces a new realm of possibilities, such as vertical mode coupling and spatially tailored resonances for multifunctional systems. However, the move to 3D plasmonics still remains challenging. At such small scales, even minimal deviations in geometry can strongly alter the targeted plasmonic behavior. To fully harness the potential of 3D plasmonics, precise characterization and subsequent simulations are essential to validate functionality and ensure reliable performance as originally designed.

## FEBID: Unlocking 3D Nanoprinting

Over the past decade, *Focused Electron Beam Induced Deposition* (FEBID) has emerged as a versatile and precise additive manufacturing technology for nanoscale 3D structures. Using a focused electron beam to locally dissociate precursor molecules (Figure 1 (A)), FEBID allows for the additive direct-write fabrication of complex architectures (Figure 1 (B)-(C)) with spatial resolutions down to a few nanometers **[2]**. Its flexibility enables the realization of 3D geometries that traditional lithographic techniques cannot achieve (Figure 1 (D)). Despite its potential, FEBID structures typically contain a high proportion of carbon due to incomplete ligand dissociation. Purification processes are consequently essential to remove this carbon while preserving the structures' geometry and enabling functional properties, such as plasmonic activity **[3]**. This makes the correlation between fabrication, purification, and characterization essential for success.




**(A)** **(B)** **(C)** **(D)**

***Figure 1:*** *3D nanoprinting using focused electron beams (A) facilitates the flexible fabrication of plasmonic structures from simple (B) to intricate geometries (C), and even fully free-standing 3D architectures (D). Following purification, the material transforms into pure gold, enabling distinct plasmonic responses, as illustrated by the colored STEM-EELS maps. The objective of this study was to achieve full optimization to completely align with simulation predictions, as shown right in panel (D).*

## The Challenge of Accurate Characterization

Purified FEBID structures must be accurately characterized to validate their design and optimize their functionality, which is particularly challenging when working on the nanoscale. A key technology to visualize plasmonic activities in a spatially and energetically resolved manner is *Scanning Transmission Electron Microscopy* (STEM) based *Electron Energy Loss Spectroscopy* (EELS). While excellent in these respects, simulations can complement these measurements as key to evaluating whether the designs behave as intended (Figure 1 (D)). For this, precise morphological information is essential at this scale as it has a huge impact on related simulations. While AFM characterization of such nanostructures on bulk substrates would be straightforward, it lacks of comparability as growth and purification on ultra-thin sub-3 nm carbon grids, required for STEM, is drastically different. Hence, the only reliable way for characterization and simulation are initial STEM-EELS measurements, followed by AFM characterization of the very same structures. Without this correlation, simulations based on assumptions rather than real geometries risk introducing significant inaccuracies, compromising the reliability of experimental results and predictions. Although AFM measurements on such membranes are highly challenging, the advantages of this approach are substantial and broadly applicable. This strategy can be extended to any scenario requiring combined TEM and AFM.

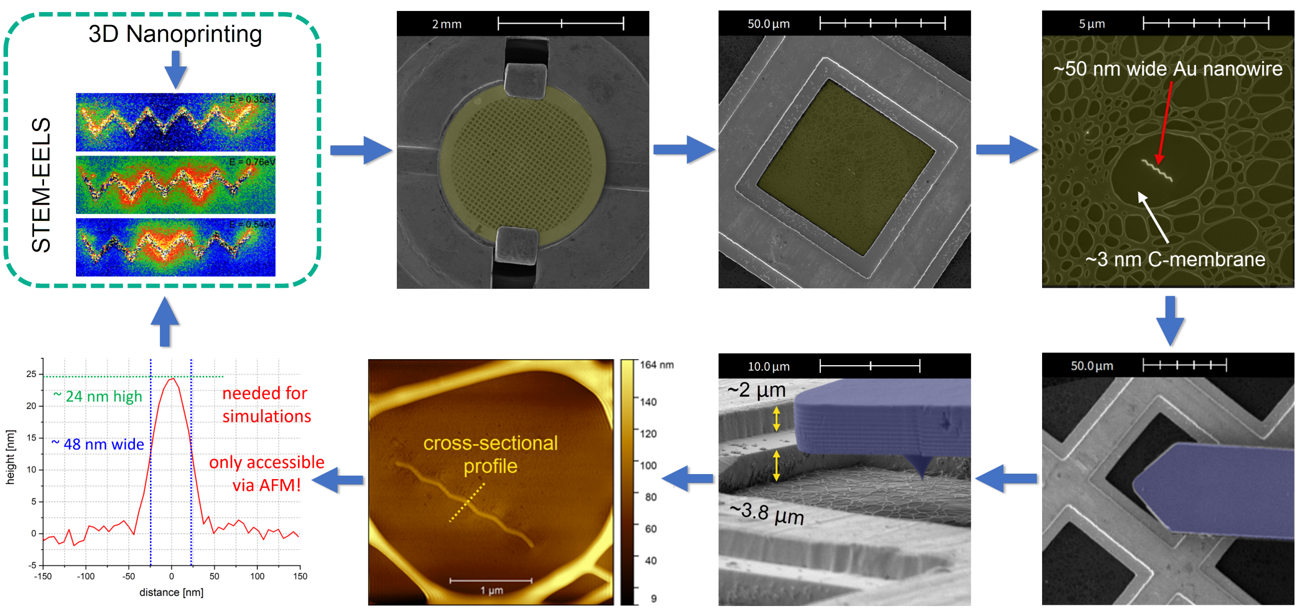



***Figure 2:*** *Comprehensive workflow for 3D plasmonic nanostructure analysis. After 3D nanoprinting, STEM-EELS characterization is performed to evaluate the plasmonic functionality (top left box). The FusionScope then begins with SEM overviews to locate the region of interest and identify the specific nanowires of interest (red, top right). Next, the SEM guides the precise approach of the AFM cantilever (blue, bottom row) for high-resolution morphological characterization on ultra-thin membranes. The resulting cross-sectional profiles (bottom left) are used as input for simulations, enabling optimization of the fabrication process* **[4]**.

## Strategy: Bridging the Morphology Gap with the FusionScope®

To address the challenges of accurate characterization and simulation, we implemented a comprehensive strategy that integrates morphological characterization into the entire workflow. At the core of this approach is the FusionScope (**[5]**) by Quantum Design, which combines the imaging capabilities of *Scanning Electron Microscopy* (SEM) for nanoscale feature identification with the precise surface analysis capabilities of *Atomic Force Microscopy* (AFM). The workflow (see Figure 2) follows these steps:

1. **Fabrication and Initial Characterization**: After 3D nanoprinting on sub-3 nm membranes, STEM-EELS was employed for detailed functional characterization of the plasmonic nanowires.
2. **Correlative Morphology**: The FusionScope was used to locate and analyze the same nanowires. SEM-guided positioning enabled the nanowire identification and enables accurate AFM approach.
3. **Simulation Validation**: Quantitative AFM cross-sectional data served as input parameters for plasmonic simulations, ensuring that the modeled geometries matched the real situation. This improved the predictive accuracy of the simulations significantly.

This strategy highlighted that direct morphological correlation is not just advantageous but essential for achieving reliable and reproducible results as a gateway toward full process optimization.

## Experimental Success: FusionScope in Action

The FusionScope proved to be the tool-of-choice without any alternative for this high-precision workflow (Figure 2). Its integrated SEM-AFM capabilities allowed us to locate and characterize nanowires on fragile, ultra-thin sub-3 nm films – a significant achievement showcasing the instrument's sensitivity and stability. SEM-guided navigation was critical, enabling the AFM probe to approach structures precisely without causing damage. As shown in Figure 3, AFM measurements revealed excellent agreement with TEM data, confirming the reliability and accuracy of the FusionScope for nanoscale imaging. This direct correlation between TEM and AFM data enabled us to refine simulation inputs and optimize the fabrication and purification processes, paving the way for predictable 3D plasmonic structures. The high degree of predictability achieved allowed the entire workflow to transition to a deductive design process. By starting with simulations and progressing to fabrication, the longstanding promise of FEBID as a reliable method for creating 3D plasmonics was finally realized.

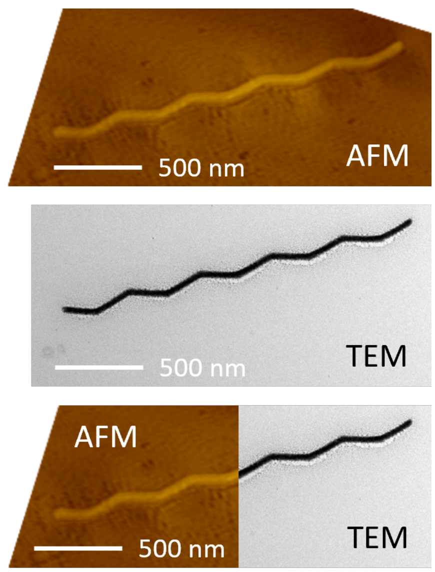



***Figure 3:*** *TEM-validated AFM data, demonstrating very good agreement in morphological characterization. This comparison highlights the exceptional sensitivity, stability, and reliability of the FusionScope, even when analyzing structures on ultra-thin sub-3 nm membranes, reinforcing its credibility for high-precision nanoscale analysis.*

## Conclusion: A New Standard in Correlative Microscopy

The FusionScope has revolutionized applicable workflows for our activities in 3D plasmonic nanostructures (**[6]**, **[7]**, **[8]**) by bridging the critical gap between fabrication and simulation for the first time in a consistent and reliable way. In a field where nanoscale precision is paramount, this instrument sets a new benchmark for next-generation correlative microscopy, offering capabilities unmatched by alternative systems.

Beyond this specific application, the FusionScope has redefined the potential of correlated microscopy. Its seamless integration of SEM-guided AFM with advanced operational modes makes it indispensable for pre- and post-characterization workflows even on highly exposed surface regions **[9]**. The instrument excels in analyzing challenging surfaces, as demonstrated on sub-3 nm membranes, and consistently delivers exceptional sensitivity, precision, and stability with an intuitive operation philosophy.

For researchers at the forefront of nanotechnology, the FusionScope offers unparalleled flexibility and reliability, representing a significant technological advancement in nanoscale characterization.

## About the Author

Harald Plank is a professor at Graz University of Technology and Director of the "Christian Doppler Laboratory for Direct-Write Fabrication of 3D Nanoprobes". His focus lies in research & development around "Functional Nanofabrication" with the aspiration of transferring basic research into industrially relevant applications. In addition, Professor Plank coordinates activities related to advanced microscopy for industrial services to support clients with complex material problems, time-sensitive challenges, or forensic analysis of patent infringements.

## References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**[9]** See reference **[5]**.