Accessing the Internal Morphology of Nano-Granular FEBID Materials in 3D Space by Electron Tomography

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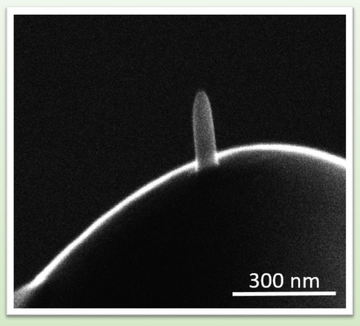
Introduction

Focused electron beam induced deposition (FEBID) is an additive, direct-write fabrication technique that enables controlled material deposition of nanoscale structures. Typically, organometallic precursors are used, e.g. $MeCpPtMe_3$, resulting in nanogranular materials composed of metallic nanograins (e.g. Pt) spatially embedded in carbonaceous matrix [1]. The distances between individual grains, as well as the chemistry of the carbon matrix significantly influence electrical [2] and mechanical [3] properties, which form the basis for potential applications of FEBID structures, such as gas sensing via nanoscale resonators. To find the connection between morphology, chemistry and materials' properties, scanning transmission electron microscopy (STEM) tomography is used for the characterization of an AD (as deposited) and EBC (electron beam cured) FEBID nanostructure.

Experiment

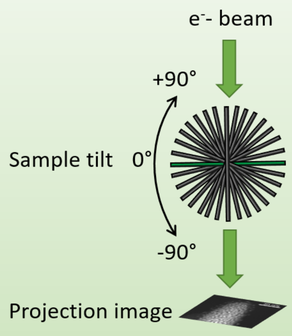
Sample Preparation

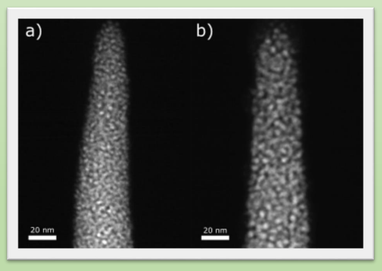


Fabrication
- As deposited (AD)
  Primary energy: 30 keV
  Beam Current: 21 pA

Post-growth electron irradiation
- Curing (EBC)
  Primary energy: 30 keV
  Beam Current: 150 pA
  Electron Dose of 20 µC/µm²

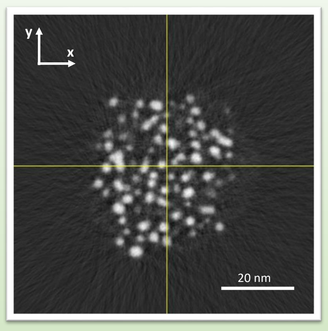
Electron Tomography

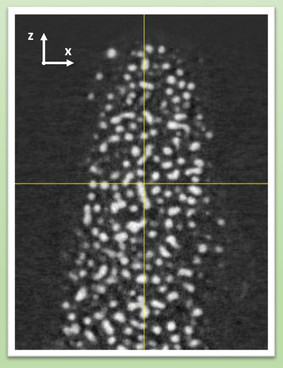




HAADF STEM
Tilt series are acquired
Tilt range: 180° & Step size: 1°

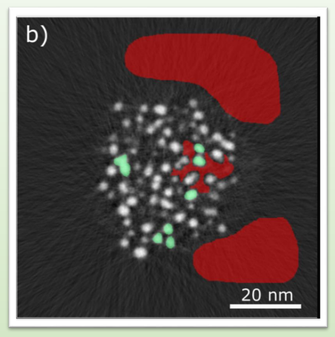
Reconstruction

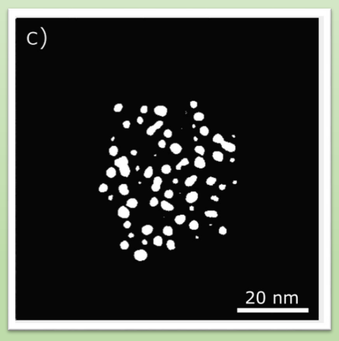




SIRT-Algorithm
Simultaneous Iterative Reconstruction Technique

Segmentation

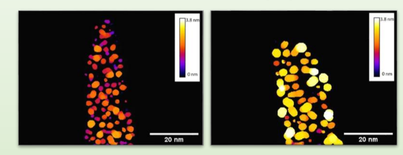




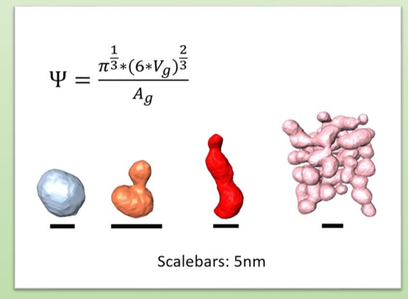
Trainable Weka Segmentation
Combination of machine learning algorithm and user input

Measurements

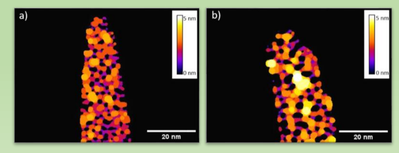
Grainsize (1)



Sphericity (2)



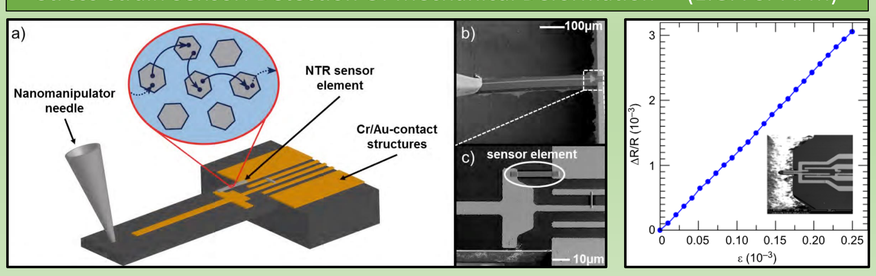
Grain Distances (3)



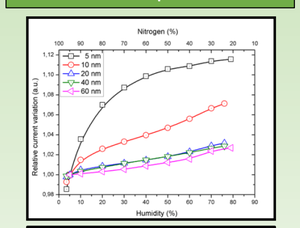
Applications & Results

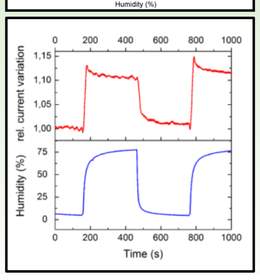
Variable grain-to-grain distances modulate the macroscopic, electric current as basis for sensing applications. This is used in different concepts as shown below.

Stress-strain Sensor: Detection Of Mechanical Deformation [1] (E.G. For AFM)

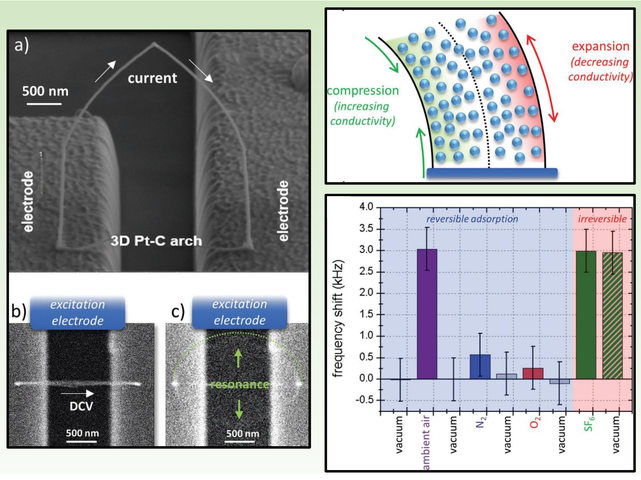


Chemical Sensing: Modulation of Dielectric Matrix Properties [1]



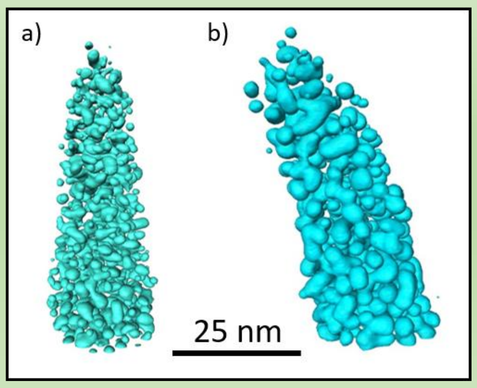


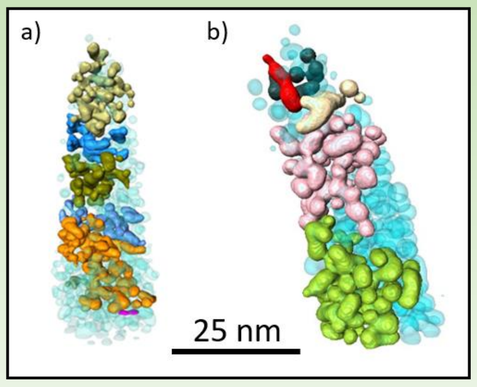
Resonant High-resolution Sensing: Surface Adsorption Changes Resonance Frequency & Stress-Strain Modulation Enables External Readout [3]



Applications

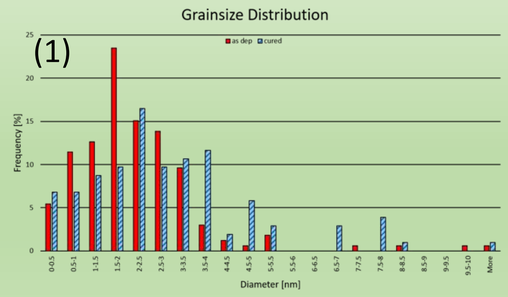
Results

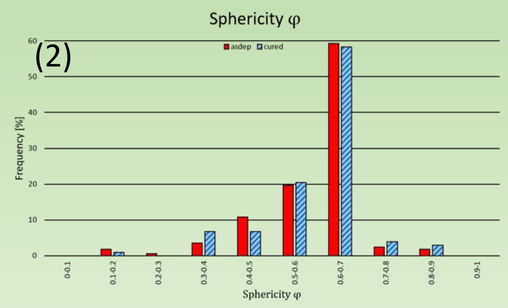


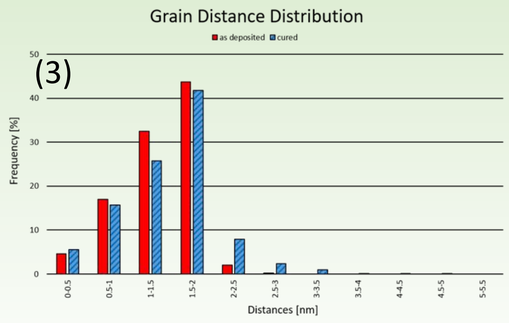


Aligned and reconstructed samples : a)AD b)EBC (Visualization: Avizo)

Electrical conductivity is influenced by the formation of long grain paths through the deposits.







References

[1] Huth, M., Porrati, F., Schwalb, C., Winhold, M., Sachser, R., Dukic, M., Fantner, G., Focused electron beam induced deposition: A perspective. Beilstein Journal of Nanotechnology, 3, (2012), 597–619.

[2] Plank, H.; Kothleitner, G.; Hofer, F.; Michelitsch, S. G.; Gspan, C.; Hohenau, A.; Krenn, J,. Optimization of Postgrowth Electron-Beam Curing for Focused Electron-Beam-Induced Pt Deposits. J. Vac. Sci. Technol. B 29 (2011), 51801–51807.

[3] Arnold, G., Winkler, R., Stermitz, M., Orthacker, A., Noh, J., Fowlkes, J., Kothleitner, G., Huth, M., Rack, P., Plank, H., Tunable 3D Nano-Resonators for Gas-Sensing Applications. Advanced Functional Materials (2018), 1-16.

Conclusion

We compared the morphology of an AD and EBC FEBID nanostructure. The cured sample has bigger and less spherical grains and greater distances between the grains in comparison to the AD sample. The grain growth is caused by the fact that they are merging during the curing step, resulting in long grain paths through the deposit, influencing the electrical conductivity. Our work will help in understanding the processes involved in deposition and post-processing of FEBID deposits.

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