

Supporting information - TEM measurements of specimen in liquids in a trap chip configuration

Joakim Lajer¹ · Sofie Tidemand-Lichtenberg¹ · Niccolò Bottauscio^{1,2} · Mervan Ramadan² · Emil C. S. Jensen² · Kristian S. Mølhave¹
*Corresponding email: krmo@dtu.dk

Received: date / Accepted: date

1 Height measurement from TEM images

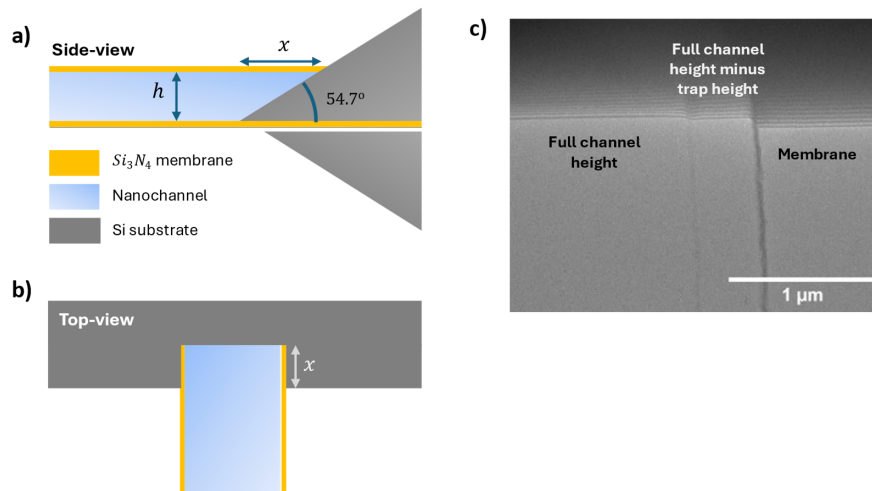


Fig. 1 Nanochannel thickness estimation. a) Schematic of the side views of the nanochannel with a height of h , where the distance x is defined. b) shows a top view of the same principle, which is what will be seen from the TEM image, as shown in c).

¹ National Centre of Nano Fabrication and Characterisation, DTU Nanolab, Technical University of Denmark, Kongens Lyngby, Denmark

² InsightChips Aps, Kongens Lyngby, Denmark

2 Trap chip

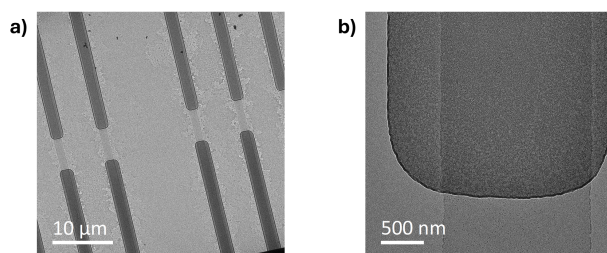


Fig. 2 Overview of the trap chip with ferritin. a) This design of the trap channels, offers a height variation to trap particles in the narrow region. High contrast comes from the solution in the channels. b) Few particles had made it into the trap region.

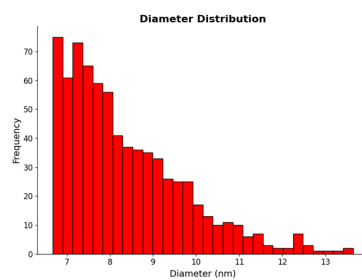


Fig. 3 Size distribution of ferritin particles. The size was found from the particles in Fig. 4.a), with a mean diameter found was: 8.5 ± 1.4 nm - Number of Particles: 745.

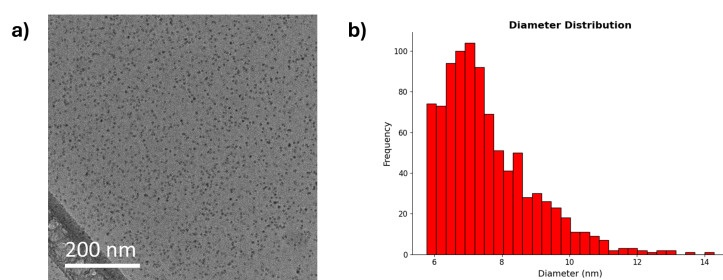


Fig. 4 Cryo TEM imaging of same ferritin sample as used in the trap chip a) Cryo TEM images of ferritin with iron oxide core. b) Particle distribution of (a), with a mean diameter found was: 7.6 ± 1.3 nm - Number of Particles: 928.

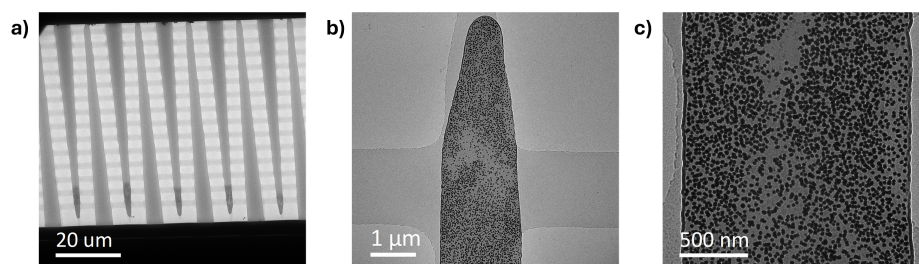


Fig. 5 Metallic nanoparticles in the trap chip. a) Particles distributed in the trap channel and the dense variation. b) Typical example of one of the traps filled with stuck particles. c) Particle size similarity was early observed in TEM images like this.

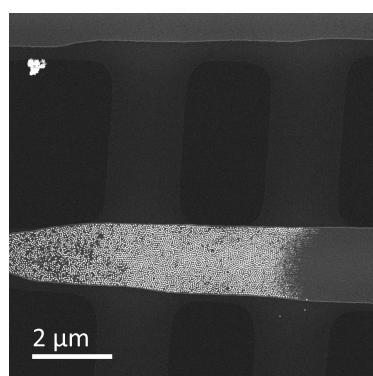


Fig. 6 HAADF-STEM images of particles trapped. Show flow front to the side channels of particles stuck in a trap, with 1000 mbar pressure applied.

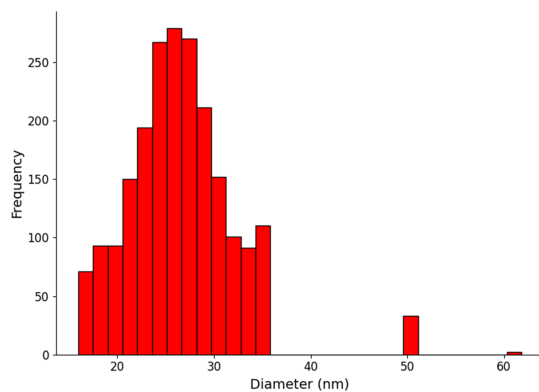


Fig. 7 Particle size distribution. Fig. S3 c) was processed in Fiji [1], with gaussian blur, threshold, fill holes and watershed function to do a particle size analysis. Mean diameter found was: 28 ± 6 nm - Number of Particles: 2386.

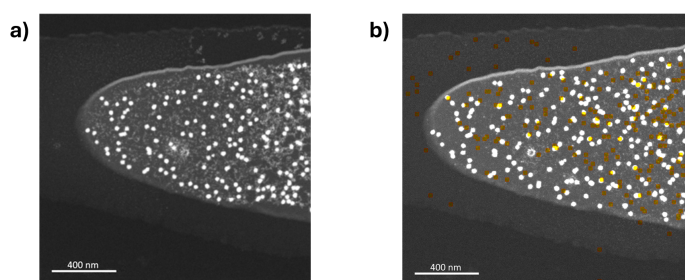


Fig. 8 STEM DF on the area after EDX map. Copper signals from the EDX map are displayed with orange dots. Traces of copper are shown all over the background where the liquid solution is present in the chip.

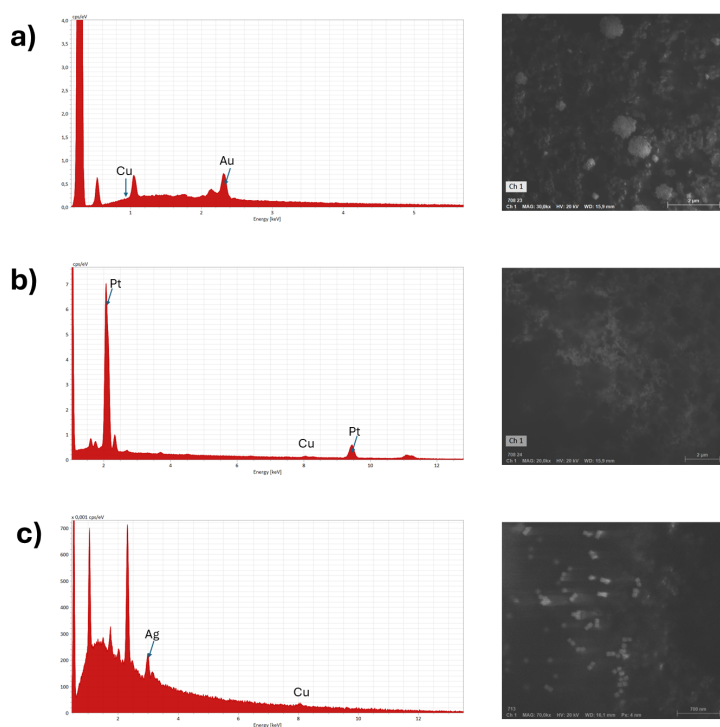


Fig. 9 SEM EDX spectrum of the three metallic solutions. All metallic components were analyzed in the SEM to investigate the copper source. The gold, platinum and silver signal was detected in their respective solution, but no copper signal was present.

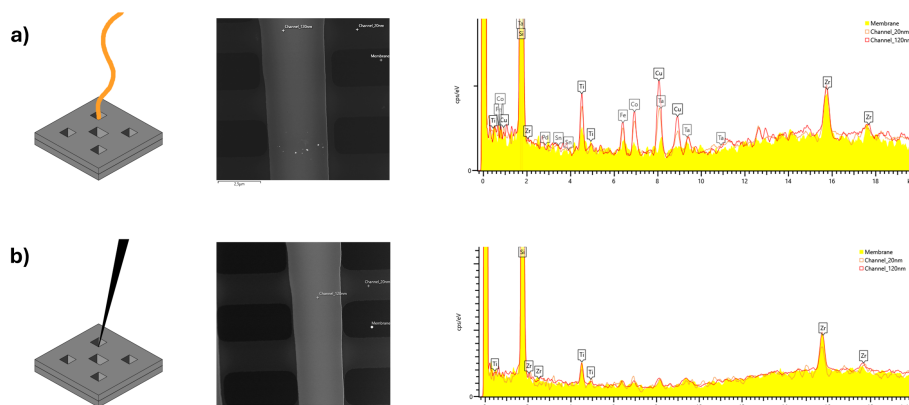


Fig. 10 STEM-EDX point measurements of the same type of chip, the membranes were punctured differently followed by the same dropcasting of Milli-Q H₂O. a) The membrane in the inlets was punctured with a thin copper wire, the same wire as used to puncture the membrane in the experiments conducted in this study. **b)** A hypodermic needle used to puncture the membrane on the inlets.

3 Diffraction

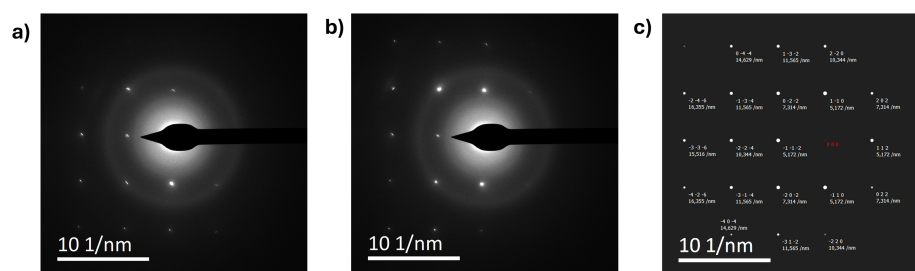


Fig. 11 Diffraction pattern of Si(111) rotated to ensure perpendicular beam to chip. a) Diffraction at 0° α tilt. **b)** Aligned diffraction pattern to the Si wedge at 0.8° α tilt. **c)** Simulated diffraction pattern of 0° tilt Si(111) from Recipro [2].

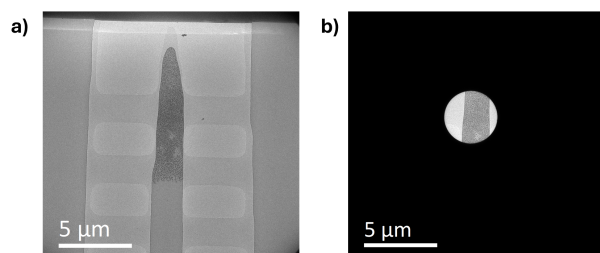


Fig. 12 TEM images from the SAD. a) Particles in trap, a dense area was chosen for diffraction. b) The particles in SAD used for diffraction.

4 ePDF

The ePDF is derived through an inverse sine Fourier transformation of the reduced scattering function, converting reciprocal space data into real space information. It represents the variation in atomic density as a function of distance from a reference atom, directly correlating to the probability of finding an atom at a given distance \mathbf{r} . The ePDF analysis followed the method described in [3]. The measured total scattering intensity, $I(Q)$, includes both elastic and inelastic contributions, accounting for the total scattering. First, $I(Q)$ is clipped to match the experimental Q -range (1.5 – 12.5 Q). Electron scattering factors are then computed using values from the International Tables for Crystallography [4] and used to normalize the scattering intensity. Further corrections, including polynomial background subtraction, smoothing, and additional data clipping to reduce Fourier ripples, are applied. Finally, the inverse sine Fourier transform is performed to obtain the ePDF. The various processes is shown below in Fig. S8.

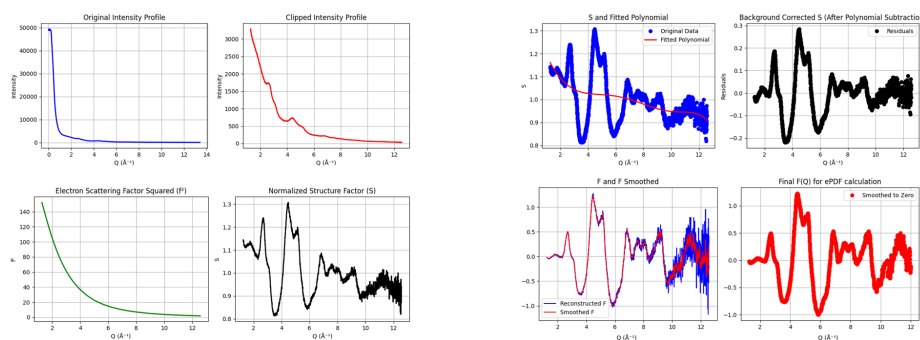


Fig. 13 Steps in ePDF analysis plotted.

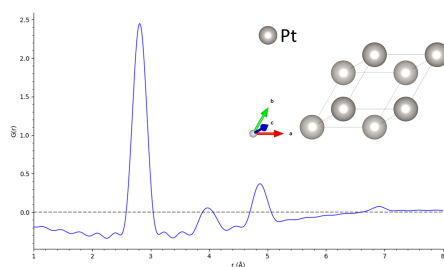


Fig. 14 Simulated PDF of Pt FCC. The simulated diffraction pattern is with similar Q-range as observed in the electron diffraction data. The simulation is done with the python package DebyeSimulator [5].

References

- [1] Johannes Schindelin et al. “Fiji: an open-source platform for biological-image analysis”. In: *Nature methods* 9.7 (2012), pp. 676–682.
- [2] Yusuke Seto and Masahiro Ohtsuka. “ReciPro: free and open-source multi-purpose crystallographic software integrating a crystal model database and viewer, diffraction and microscopy simulators, and diffraction data analysis tools”. In: *Journal of Applied Crystallography* 55.2 (2022), pp. 397–410.
- [3] Tatiana E Gorelik et al. “Towards quantitative treatment of electron pair distribution function”. In: *Acta Crystallographica Section B: Structural Science, Crystal Engineering and Materials* 75.4 (2019), pp. 532–549.
- [4] Edward Prince. *International Tables for Crystallography, Volume C: Mathematical, physical and chemical tables*. Springer Science & Business Media, 2004.
- [5] Frederik Lizak Johansen et al. “A GPU-Accelerated Open-Source Python Package for Calculating Powder Diffraction, Small-Angle-, and Total Scattering with the Debye Scattering Equation”. In: (2023).