Technical Bulletin

sciTEM – A loading method for water-based nanoparticle dispersions onto TEM and SEM supports for high-throughput characterization

Introduction

TEM and SEM measurements are commonly applied as methods for characterization of nanomaterials. However, by nature of electron microscope instrument design placing sample supports under high vacuum, the number of samples that can be measured in a given time period is very limited. Having the capability to deposit multiple samples on one TEM/SEM support and then acquire all images without breaking vacuum for each one, results in a significant increase in the number of samples that can be analyzed per measurement cycle. A piezoelectric nanoliter dispensing technology, sciTEM, is applied for high precision non-contact printing of spots onto the supports.

Results of dispensing of water-based nanoparticles on SEM supports

Samples of nanoparticles are commonly prepared in organic solvents or aqueous solutions. Due to their different surface tensions both systems require different handling when spotting the solutions onto a TEM/SEM support. Using water-based nanoparticle dispersions can cause agglomerations during the drop drying on the sample support. Surface functionalization of the SEM/TEM supports allows adjustment of surface parameters such as wettability and surface charges. Agglomeration effects of water-based nanoparticles can be minimized by application of support films with optimized surface characteristics. Different samples of water-based nanoparticles have been spotted onto a surface functionalized silicon nitride support, Figure 1.

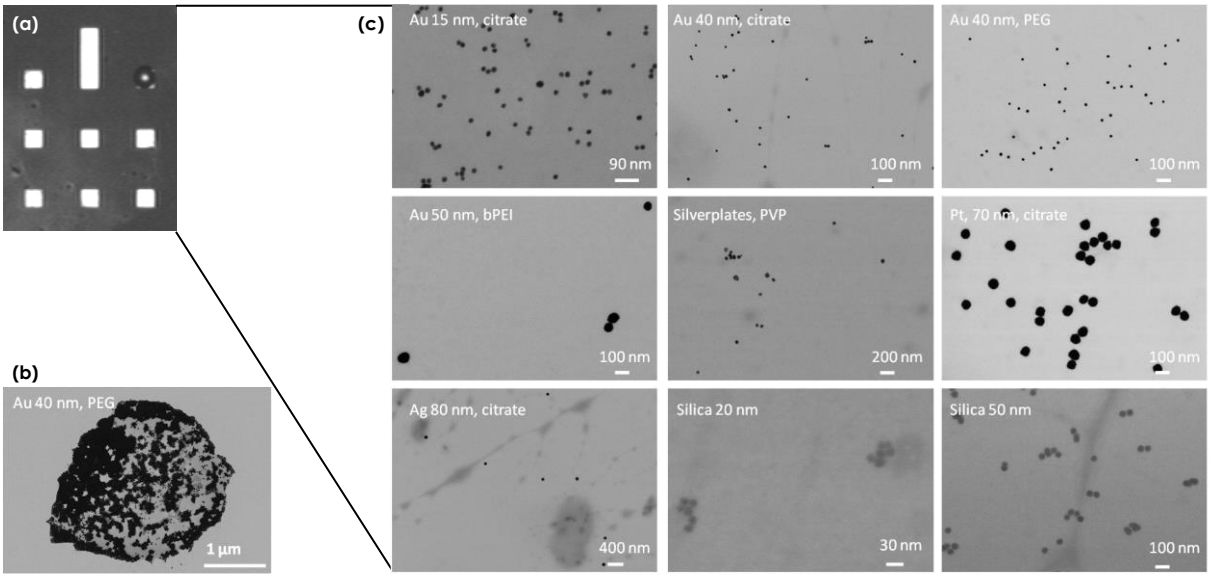

Figure 1. SEM images (transmission mode) of different nanoparticle samples on a surface functionalized silicon nitride TEM support (eight windows 100x100 µm and one window 100x350 µm with a membrane thickness of 50 nm; SIMPore).

The sciTEM was used to deposit nine different water-based nanoparticle samples precisely onto each of the nine windows of the silicon nitride membrane support. Spotting of small volumes of 200 pL onto untreated silicon nitride films and subsequent drying results in the formation of smaller spots with most of the nanomaterial agglomarated within the spots, Figure 1b. Surface functionalization increases the hydrophilicity of the silicon nitride films. Deposited drops of nanopartciles samples spread over the entire area of the 100x100 µm windows. Samples show good particle distribution after evaporation of water. The SEM images show the results after spotting and drying of nine different nanoparticle dispensions, Figure 1c. Gold nanoparticles stabilized by citrate, PEG or bPEI show homogeneous distributions. Minimized agglomeration has also be observed for the spotted silver, silica and platinum nanoparticles.

Summary

It has been shown that sciTEM is a suitable loading method of different water-based nanoparticle dispersions onto one SEM supports for high-throughput characterization. In combination with support films of optimized wettabilitiies nanoparticles distributions with a low degree of agglomeration within the spots have been obained resulting in high qulity SEM images of the nanomaterials.

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