Technical Bulletin

sciTEM – A loading method for toluene-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 sample loading and TEM measurements

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. Small drop volumes and the low surface tensions of solvents such as toluene cause a wide spreading of the drop and a fast solvent evaporation on the support films is then observed. It has been shown, that toluene-based nanoparticle suspensions can be handeled with the sciTEM to dispense 190 pL droplets onto various support films.

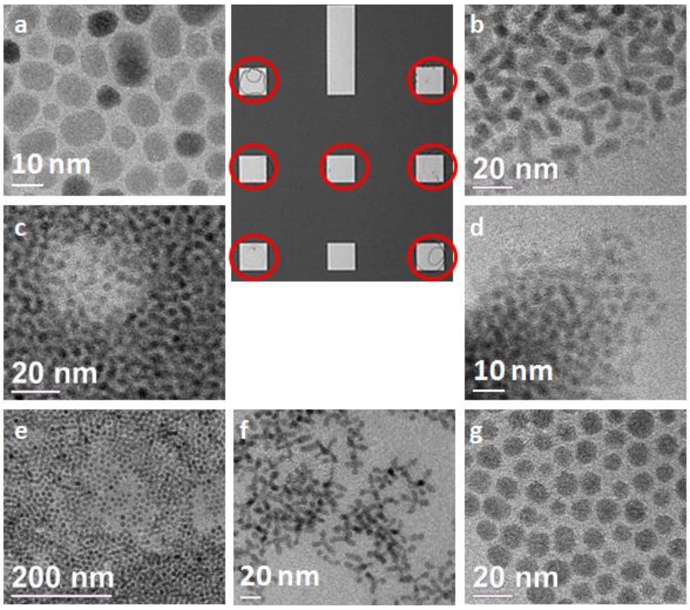

Figure 1. TEM images of different samples on a sample grid with silicon nitride membrane (SIMPore TEM grid) a) $NaYF_4$ <100 nm b) CdTe 3x10 nm; c) CdSe 4 nm, diluted d) CdSe 3x10 nm; e) CdSe 4 nm undiluted f) $NaYF_4$ 9 nm; g) $NaYF_4$ < 100 nm.

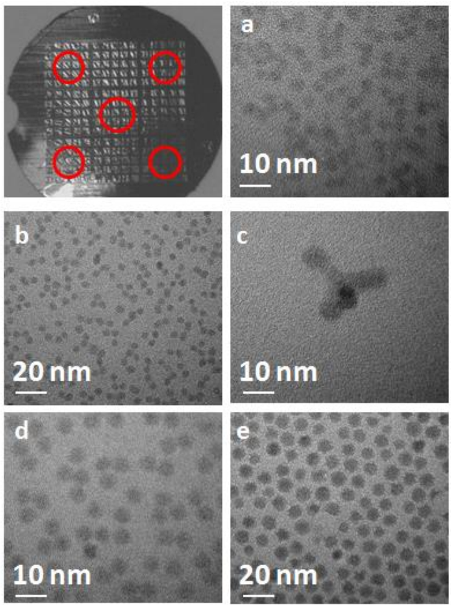

Figure 2. TEM images of different samples on a copper grid with carbon film (Ted Pella Inc.) a)CdSe 4 nm; b) CdSe 6 nm; c) CdTe 3x10 nm d) $NaYF_4$ 9 nm; e) $NaYF_4$ 6 nm.

Spotting was done on a silicon nitride TEM support with one 100x350 µm and eight 100x100 µm silicon nitride membrane windows (50 nm thickness), Figure 1. The spotting routine includes the automatic detection of the geometry, substructure and orientation of the TEM support. Droplets of 190 pL volume have been placed in the centre of the silicon nitride membranes. The deposited sample spreads over the whole window area before drying. The best results of the TEM measurements have been obtained for CdTe tetrapods and $NaYF_4$ nanospheres as shown in Figure 1 e.g. Spotting has also been perfomed with the sciTEM system of five different nanoparticle samples on a copper pinpointer grid with carbon film (10-25 nm thickness), Figure 2. The TEM images show a homogeneous distributions of the nanoparticles.

Summary

It has been shown that the sciTEM can be successfuly used as a loading technology for toluene-based nanoparticle dispersions on TEM and SEM supports. Up to nine different samples have been printed on one support grid. A homogeneous distribution of the nanomaterials within the spots formed from picoliter drops can be achieved to measure nanoparticle size distribution, shape and purity.

Acknowledgement

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