doi:10.1017/S1431927616010072
Microsc. Microanal. 22 (Suppl 3), 2016

Electrospray Deposition of Nanoparticles on TEM Grids

Johannes Mielke[1], Pavla Dohányosová[2], Silvia López[2] and Vasile-Dan Hodoroaba[1]

[1.] Federal Institute for Materials Research and Testing (BAM), Berlin, Germany.
[2.] RAMEM S. A., Madrid, Spain.

Although there are many experimental techniques for measuring particle sizes and size distributions, electron microscopy (EM) is still considered as the gold standard in this field, especially, when it comes to particles in the nano range (1 nm – 100 nm). This is the case, because EM cannot only resolve the size of individual particles accurately, but also the particles shape and morphology and can – to a limited extend – also address constituent particles in aggregates and agglomerates.

To be able to extract particle size distributions from the EM micrographs that are representative for the material under investigation, one needs to assure the representativity of the particles as sampled on the substrate and their homogeneous spatial distribution, to avoid operator bias when selecting the imaged area. Furthermore, agglomeration should be avoided as far as possible.

Preparing such an EM sample for generic particulate materials is a difficult task. Several sample preparation techniques exist since a long time, the most common way being suspending the particles in a liquid and depositing them on the grid. However, this procedure includes the drying of larger solvent amounts on the substrate itself, which can affect the spatial distribution of the deposited particles. One possibility to overcome this problem is the use of an electrospray system, where the suspension of particles is sprayed onto the substrate in charged droplets that are so small that they either dry off on the substrate immediately without affecting the position of particles, or even already during their flight time to the substrate (Figure 1). Additionally, the charging of the particles minimizes their agglomeration and aggregation, maximizing the collection of the EM grids that acts as counter-electrode.

The authors have tested the prototype of an electrospray deposition system developed by the company RAMEM under its trademark IONER (www.ioner.eu). Electrospray is theoretically described since a long time [1] and is quite an established technique in many areas of research and manufacturing like thin film deposition [2], deposition of single molecules under UHV conditions [3] and for the spraying of particle suspensions into aerosol analysis experiments [4]. No dedicated commercial instruments are available for the preparation of TEM grids yet, only electrostatic deposition of aerosols on TEM grids has been reported so far [5].

To test the prototype and assess its performance, several materials have been sprayed onto TEM grids and the resulting particle distributions were compared to more traditional sample preparation strategies like the “drop on grid” method (Figure 2). Operation parameters such as the sample flow-rate, capillary – substrate distance, electric field strength and sampling period length have been optimized. It was found that the particles deposited by electrospray generally show a much more homogeneous spatial distribution on the substrate and a substantial increase of the number of single particles, which are much better suited to an automatic image evaluation procedure than the agglomerated particles observed otherwise. The applicability of the technique to a broad range of materials is demonstrated by various examples, but also the influence of the substrate, the choice of the particular TEM grid, on the obtained spatial particle distribution is assessed [6].

References:

[1] G Taylor, Proceedings of the Royal Society of London. Series A, Mathematical and Physical Sciences, **280** (1964), p. 383.
[2] A Jaworek, Journal of Materials Science, **42** (2006), p. 266.
[3] C J Satterley *et al*, Nanotechnology, **18** (2007), p. 455304.
[4] L F Pease *et al*, Langmuir, **26** (2010), p. 11384.
[5] M Fierz *et al*, Aerosol Science and Technology, **41** (2007), p. 520.
[6] The research leading to these results has received funding from the European Union's Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 604347. Dr. K Löschner and Dr. M Correia (Technical University of Denmark) are thanked for their help to optimize the dispersion of the various materials in suspension.

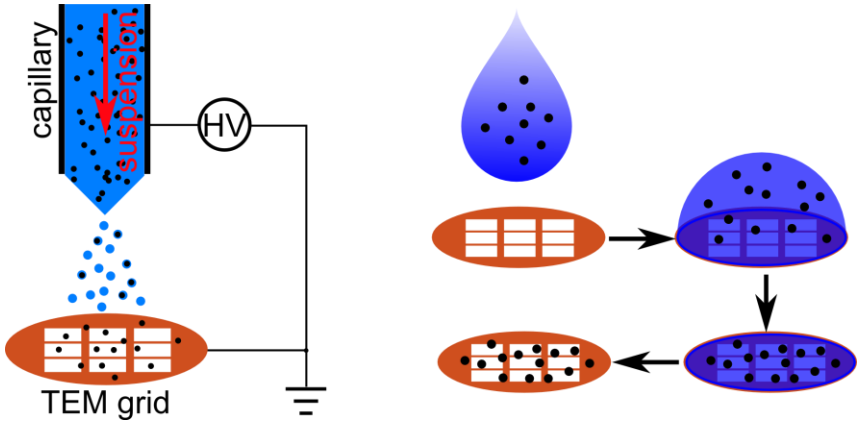

Figure 1. The principle of electrospray (left) versus the common “drop on grid” technique (right)

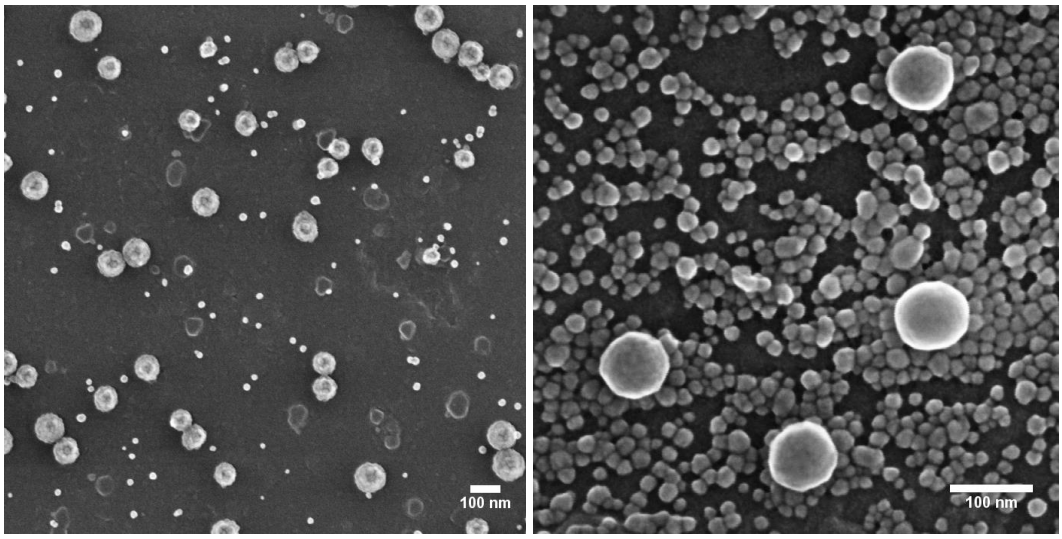

Figure 2. Comparison of SEM images of the same bimodal silica suspension (ERM-FD102), prepared with the electrospray deposition system (left, concentration as received, deposition time: 5 s) and the drop on grid method (right, dilution 1:100). In both cases, a TEM grid with a carbon film is used as substrate. Both images were taken at 20 kV, with an *In-Lens* detector.