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Nano-Particle TEM Sample Preparation Primer

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As nanotechnology continues to drive scientific research, it becomes increasingly difficult to characterize the "nano" structures. Transmission electron microscopy (TEM) has proven to be a significant technique to provide a range of data for most nanomaterials. Point resolutions at 0.24 nm or better with the aberration corrected TEMs can allow for direct imaging of the atomic structure of a sample. [1] It can be a daunting task to determine which TEM grid and or support film to use to obtain the best results for a specific sample type. The old adage of "garbage in, garbage out" is never truer than with electron microscopy sample preparation. Nanomaterial samples seem to magnify this issue at a geometric rate! Often times there is a small amount of the sample with which to work, and when working with "unseen samples" one can sometimes be unsure of adequate sample preparation. To compound these issues is the decision as to which sample substrate should be utilized to provide the best analytical characterization of nanomaterials.

To determine an elementary "go to" protocol for grid and support film selection for TEM examination of nano-particles, copper grids with different support films were evaluated. The support films were lacey carbon (LC), thin holey carbon (HC), holey carbon/Formvar (HCF) and lacey silicon monoxide/Formvar (SiO/F). Carbon black (CB), Cabot Black Pearl 120, and colloidal gold solution (Au), Sigma, were used as the test materials. The CB simulated the lower atomic weight nano-particles while the Au simulated the higher atomic weight nano-particles. The size of the particles are in the 5-100 nm range with the CB at the larger end of the range. Dilution of the CB was at 0.02g/ml and for the Au it was 6.5 x 10^{-5} g/ml in 100 ml of CH_3OH.

Two methods of dispersing the CB and Au on the TEM grids were evaluated. One method involved the dispersion of the solid in CH_3OH by ultrasound and manually dropping 10 µl of the solution on a TEM grid. The grids were then allowed to air dry. The second method was by using an ASP-1000 Automated Specimen Processor by Microscopy Innovations. TEM grids were placed in the mPrep/g capsules and attached to the reagent lines of the ASP-1000. Initially the suspension liquid was drawn into the loaded capsules and the grids were allowed to dry. After loading, the grids some were rinsed for 5 minutes, with CH_3OH, for up to 3 times, Table 1. The grids were allowed to dry in the mPrep capsules. Evaluation of the grids from both sample protocols was completed on a Tecnai F20 at a low and a high magnification, Figure 1. ImageJ as used to determine the percent coverage and percent agglomeration of the grids, Table 1. Other characteristics evaluated were, degree of aggregation and negative support film interference, Table 1.

Single loading with 10 µl and automated loading with no rinsing often resulted in total coverage of the grid. The automated loading with rinsing showed less material on the grid and fewer particles in the agglomerations, Figure 2. Lacey SiO with Formvar film proved to be the best support film, with multiple single particles on the film edges with little or no background artifacts in the TEM images. Automation uses less initial volume and with controlled rinses reduces the amount of agglomerations thus allowing for single particle imaging. As most labs will not have automation capabilities the use of the mPrep capsule fitted with a microliter pipet can have the same affect for sample prep. A basic primer for TEM sample preparation for nano-particles includes a support film with multiple openings, minimal material for loading and multiple rinses for better single particle imaging.

References:

[1] J. C. H. Spence, *et al*, Philos. Mag. 86, (2006), p. 4781.

Table 1 Grids and Characteristics (Number of rinses in brackets preceding value)

Grid	Loading*	# of Rinses	% Coverage	Agglomeration % (CB)	Film Background
SiO/F	D	0	80	Not determined	Multiple over open
SiO/F	A	0, 1, 2, 3	[1]15	[1]70, [2]50, [3]20	Multiple over open
HN	D	0	50	Not determined	Few over open
HN	A	0, 1, 2, 3	[1]13	[1]85, [2]50, [3] 25	Few over open
HCF	D	0	100	Not determined	Few over open
HCF	A	0, 1, 2, 3	[1]26	[1]90, [20]50, [1]30	Few over open
LC	D	0	14	Not determined	Few over open
LC	A	0, 1, 2, 3	[1]3	[1]82, [2]60, [3]20	Few over open

*D-Drop, A-Automated

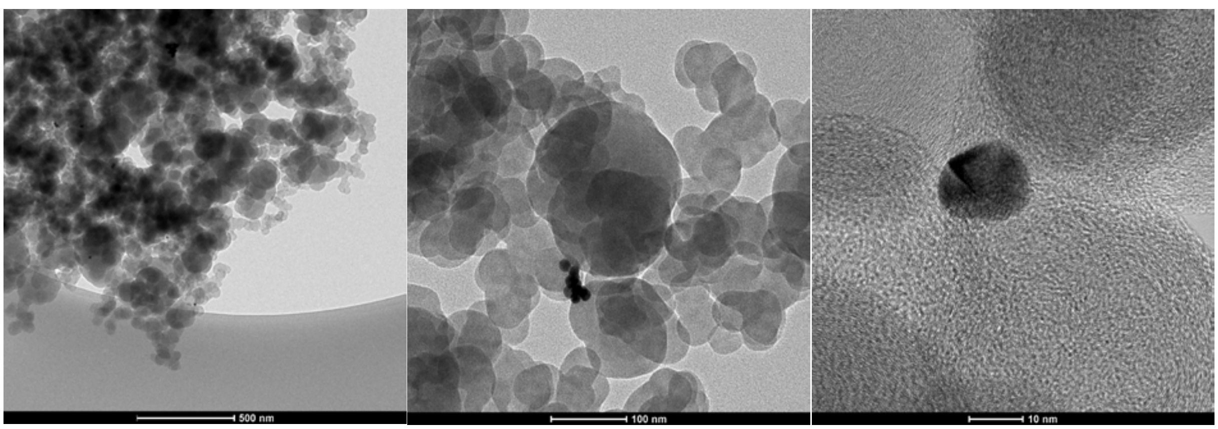

Figure 1 – CB and Au particles on a HCF support film, a. 9,900x, b. 38,000x, c. 285,000x

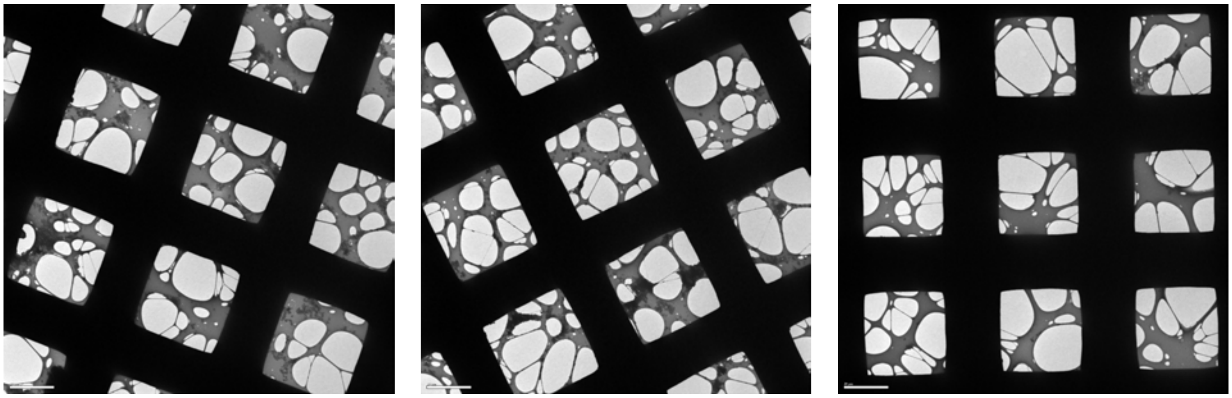

Figure 2 – CB and Au particles on a LC support film at 300x; a, 1 rinse, b. 2 rinses, c. 3 rinses

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