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Low voltage TEM - new tool for nanoparticle characterization

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The characterization of the morphology of nanoparticles and nanotubes is becoming a growing task in last few years. The two well established techniques of electron microscopy (EM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are suitable for imaging and measuring the morphology of materials including nanoparticles [1]. All the main problems involving imaging nanoparticles are well addressed. Some of the issues concerning the applicability of EM are particle-vacuum compatibility, core-shell nanoparticles, and/or low Z-contrast materials.

An electron microscopy technique using low energy electron beam in TEM and STEM modes to improve low Z materials contrast is available. Decreased working energy leads to higher scattering of electrons in the sample and thus to increased contrast [2, 3, 4, 5].

As light elements scatter imaging electrons very little, weak contrast at low Z materials is one of the EM applicability issues for biological and polymer samples as well as nanomaterials. Stronger scattering to increase contrast can only be achieved in two ways:

- Staining techniques that result in an increased contrast, thus improving the image, however also create artefacts, structures that do not belong to the object of interest.
- Decreasing the electron energy.

DELONG has designed a TEM (LVEM5) with markedly reduced electron beam energy (5 keV) [6, 7, 8]. Recently, this microscope can be equipped with a double projector lens system that fully exploits the resolution power of the objective lens. The parameters of the basic model (magnification of 202,000×, resolution of 2 nm) have thus been improved to magnification of 700,000× and resolution of 1.2 nm.

The sample thickness for this technology shall be as low as 10 – 50 nm, which makes it suitable for nanoparticles or nanotubes characterisation. On the other hand the sample thickness might be an important consideration limit due to the reduced transparency of slow electrons through the object.

We used LVTEM for characterisation of nanoparticles and nanotubes (shape, size and size distribution). The critical step proved to be specimen preparation due to the tendency of nanoparticle suspensions to create aggregates. We used the method of the indirect carbon evaporation for preparing of supporting films with the thickness about 3 nm. As proved Figure 1., thus prepared carbon film shows smooth, no granular structure in LVTEM with clearly visible nanoparticles or nanotubes. The great advantage is the possibility to visualize the interaction of nanoparticles with e.g. organic ligands, macromolecular complexes, antibodies, without adding of staining agents (Figure 1b.).

In this study it was demonstrated that LVTEM is capable to provide a quick and reliable characterization of the nanoparticles and nanotubes morphology. Due to the high image contrast, nanoparticles and nanotubes made of light elements can be examined. The small size of LVTEM, which is able to operate without any cooling, allows its exploitation as a screening tool for a quick check of prepared nanoparticles close to the site of their production and as a metrological tool for the evaluation of shape and size distribution.

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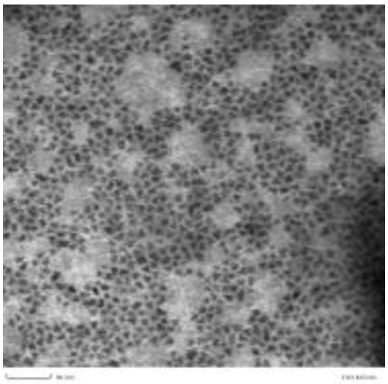
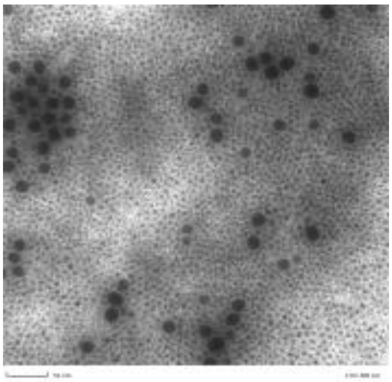


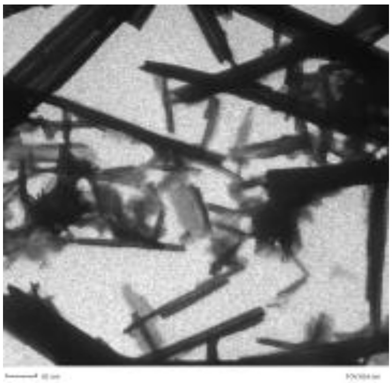
a.) 6nm CdSE NPs on a thin C film.



b.) Fe NP with Oleylamine ligands on a thin C film

c.) Multiwall CNTs

d.) Pd nanocubes on a thin C film



e.) Modified TiO_2 nanotubes