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The structure of cdse and au nanoparticle ensembles on carbon substrates

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Metal-containing nanoparticles is one of the most studied classes of nanoparticles nowadays. CdSe and Au nanoparticles attract much attention from scientific and practical point of view due to their properties. CdSe nanoparticles form optical active quantum dots materials displaying high brightness with a narrow emitting spectrum, which is perspective for application in optoelectronics, photocatalysis and biology as markers and sensors. Au nanoparticles are of interest for creation of fuel cells, nanosensors, catalysts etc.

New techniques of synthesis of CdSe and Au nanoparticles have been developed in this study. Ordered ensembles of nanoparticles formed via self-organization process have been fabricated and characterized as well. CdSe nanoparticles were synthesized by interaction of cadmium stearate with triphenylphosphine in heptadecan mediumin at the presence of oleamin and triphenylphosphine oxide as surface active agents (SAA). Au nanoparticles were synthesized by reduction of $HAuCl_4$ in hexane through boron hydride at the presence of SAA. A new preparation technique for electron-microscopic studies of nanoparticles has been developed. The technique provides a minimal substrate influence and high stability of image observation.

X-ray and electron diffraction patterns indicate that prepared samples of CdSe and Au nanoparticles are single-phase. CdSe nanoparticles have a wurzite structure (the $P6_3mc$ space group). Crystal structure of Au nanoparticles is described in terms of face centered cubic lattice (the Fm3m space group). High resolution electron microscopy and X-ray small angle scattering evidence that the prepared samples contain nanoparticles of spherical form with a narrow size distribution. The average size of CdSe nanoparticles is 12 nm, and that of Au nanoparticles is 10 nm.

Self-assembly of CdSe nanoparticles into close-packed two-dimensional (2D) ensembles with the 6-order symmetry was observed on carbon substrate in the regions of high density of nanoparticles. The pronounced texture was present in CdSe ensembles when all nanoparticles were orientated with the [001] direction perpendicular to carbon substrate. A new type of ordered ensembles formed of associates of CdSe nanoparticles (several joined nanoparticles) has been found.

No texture in the form of preferred crystallographic orientation of nanoparticles in relation to each other or to the substrate has been found in close packed 2D ensembles of Au nanoparticles. The three-dimensional (3D) structures of Au nanoparticles have been found, where the second layer of nanoparticles resided in voids of the first layer forming a close-packed bulk.

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Accurate structure factors of skutterudites: electron diffraction techniques

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The utility of the precession technique in electron diffraction has been demonstrated by a number of applications to structure solution [1]. A basic advantage is the suppression of dynamic diffraction, allowing kinematical interpretation of intensity data. Next task should be to develop procedures whereby the remaining dynamical scattering effect can be included in practical way in structure solution and refinement. Extensive dynamical computations along the precession circle, based on either multislice or Bloch wave theory may be cumbersome. We propose therefore to develop methods based on approximate, two-beam–like expressions for intensity profiles pertaining to multiple beam situations, along the precession circle. These profiles can then be integrated according to the Blackman formula [2], [3]:

$$\int_{-\infty}^{\infty} I_g(s_g,t)ds_g = U_g \int_0^{U_g t} J_0(x)dx$$

This implies that an *effective potential*, U_g^{eff} can be inserted. Various alternatives for calculation of effective potentials will be presented and compared with complete Bloch wave computations.

Applications are to modified binary skutterudites with interesting thermoelectric properties. Voids in the basic TX3 structure (Co_8P_{24}) have been filled with different "rattle" atoms (La,Ce). Skutterudites and "filled skutterudites", Co:P:Sn and Ce(La):Fe:P:Sn, have been prepared by mixing and melting the starting materials in the atomic ratios 1:3:25 and 1:4:20:50 respectively in evacuated silica tube and heat treated. HCl was used to dissolve away the tin from skutterudites. Phase identification and characterization of the material has been done by powder XRD and TEM.

The structural aim is to determine accurate Debye-Waller factors over a range of s-values. Intensity data collected by precession electron diffraction and rotation electron diffraction techniques will be used in conjunction with X-ray and neutron powder diffraction.

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Large-angle rocking-beam electron diffraction (LARBED)

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Large-angle rocking-beam electron diffraction (LARBED) [1], [2] is an alternative approach to diffraction tomography for recording 3D electron diffraction data [3], [4] which does not require the sample to be tilted or moved. An implementation of this technique which tilts the beam but keeps the illuminating spot on the same position on the sample by compensating for all 42 aberration coefficients of the illumination system up to 7th order is commercially available as a plug-in to DigitalMicrograph™ (Gatan Inc., Pleasanton, CA). Since the orientation of the sample is kept fixed, the tilt range available by this technique is determined by the quality of the electron optical setup of the microscope and is commonly less than ±10° in both θ_x- and θ_y-