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High quality Structural refinement of nanoparticle atomic arrangement from Pair distribution Function derived from Precession Electron diffraction in a TEM.

Nanoparticles (NPs) attract a great deal of interest in science and technology, due to the possibility of controlling their properties by changing size and structure. The atomic arrangement in NPs may be quite different from bulk materials, however the access to reliable and quantitative structural characterization tools for this size regime is still an open issue. Bulk crystals display long-range atomic order, and x-ray diffraction analysis fulfils requirements of precision and easy use. Nanomaterials are in between amorphous systems (short-range order) and bulk crystals, then diffraction patterns contains less information due to peak broadening, low signal, high background and higher thermal vibrations. This leads, leads to a lack of sufficient constrains to the required fitting free variables (atomic positions). Transmission Electron Microscopy (TEM) is the most popular NP characterization approach, but analysis are mostly qualitative and many times lack of the number of repetitions to confirm the reliable assessment from the statistical point of view. In this work, we report a structural refinement approach to gather quantitive information on NP atomic arrangement using pair distribution function (PDF) calculated from precession electron diffraction (PED). The application of PED reduces the contribution of multiple electron scattering (dynamical regime) and, the diffraction intensities can be described by simple kinematical calculation. Using this approach, we can study the direct study of atomic order at different scales (local, medium and long range) in real space for an ensemble of particles. We have applied the developed methodology to complex polycrystalline noble metal NPs (few nm in diameter) generated by a gas aggregation source; usually less than a picogr of sample mass is necessary, and electron doses may be quite low < 10 e-/A2. We have obtained a high quality structural refinement whose residue values (~15%) are similar to synchrotron-based nanosystem studies. We have used a low-profile non-corrected 200 KV TEM and a CCD detection camera, then we expect that the refinement quality can still be much improved with modern instrumentation.

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