Quantitative High-Resolution TEM/STEM and Diffraction

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3D Automated Electron Diffraction Tomography for nanomaterials

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Automated Electron Diffraction Tomography (EDT) is a fast and efficient technique that has been recently developed by us [1]. It can be used for fast three-dimensional (3D) reciprocal space scanning with a given fine step (0.01°-0.1°). We demonstrate how this method can be utilized for characterization of materials that exhibit periodic crystal structure at nano-scale.

In this work two types of nano-crystals were investigated: (i) an individual cubic nano-particle composed of manganese ferrite ($MnFe_2O_4$, cube sizes 14 nm and 20 nm, Figure 1; s.g. *Fd*-3*m*, a = 8.4983Å) and (ii) gadolinium orthophosphate cylindrical nano-rod ($GdPO_4$, diameter ~ 7nm; s.g. $P3_121$, a = 6.9Å, c = 6.35Å).

The crystal structures of the individual nanocube and nano-rod were determined from the EDT data collected in conventional selected area electron diffraction (SAED) mode using EDT-COLLECT software package [2] on JEOL JEM-2100 LaB_6 CTEM equipped with a single ultra-high tilt holder. Two sets of ~600 ($MnFe_2O_4$, exposure 2 sec/frame) and ~700 ($GdPO_4$, exposure 1 sec/frame) unique electron diffraction patterns were recorded from individual single nanocrystals using 3D EDT. Reciprocal space coverage was ~100° for the nano-cube and ~113° for the nano-rod. The recorded frames were processed using the EDT-PROCESS software package [2] and assembled into a corresponding 3D volumetric representation of reciprocal space (Figure 2).

The crystal structures were successfully determined using direct methods from the integrated intensities extracted by EDT-PROCESS program. In case of gadolinium orthophosphate the crystal structure of the hexagonal phase has been determined for the first time (until now it has only been described by an X-ray powder profile [3]).

It is possible to study dynamical scattering effects quantitatively using several 3D EDT frames from each dataset due to relatively small thickness values of the investigated nano-particles. In both cases the *R*-merge values for Friedel pairs were very low (5.7% for $GdPO_4$ and 10.2% for $MnFe_2O_4$). In case of the investigated gadolinium orthophosphate cylindrical nano-rod it was possible to utilize the Bloch wave formalism for the partial dynamical structure refinement using several individual electron diffraction frames.

In this work we show that 3D EDT as a very powerful technique which offers a facile and systematic way to study a wide ranges of materials at different scale levels. Simultaneously EDT studies performed on nano-crystals with almost perfect shape can be utilized for further quantification taking dynamical scattering into account.

1. D.L. Zhang, P. Oleynikov, S. Hovmöller and X.D. Zou, Z. Krist. 225 (2010), p. 94.
2. M. Gemmi and P. Oleynikov, Z. Krist. 228 (2013), p. 51.
3. B. Scheetz in JCPDS (ICDD), file no. 39-0232.
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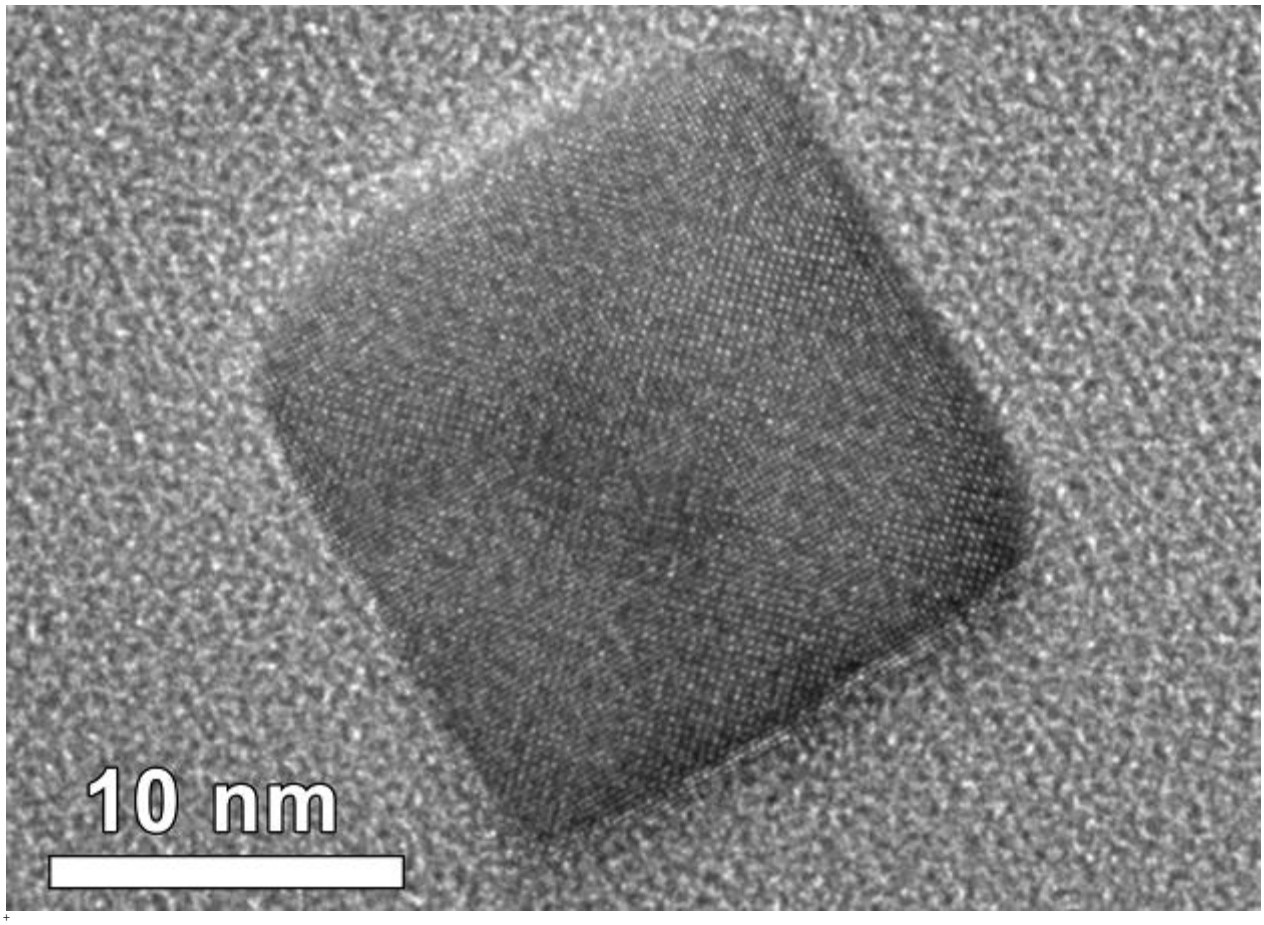

Figure 1. 20 nm cubic nanoparticle of $MnFe_2O_4$.

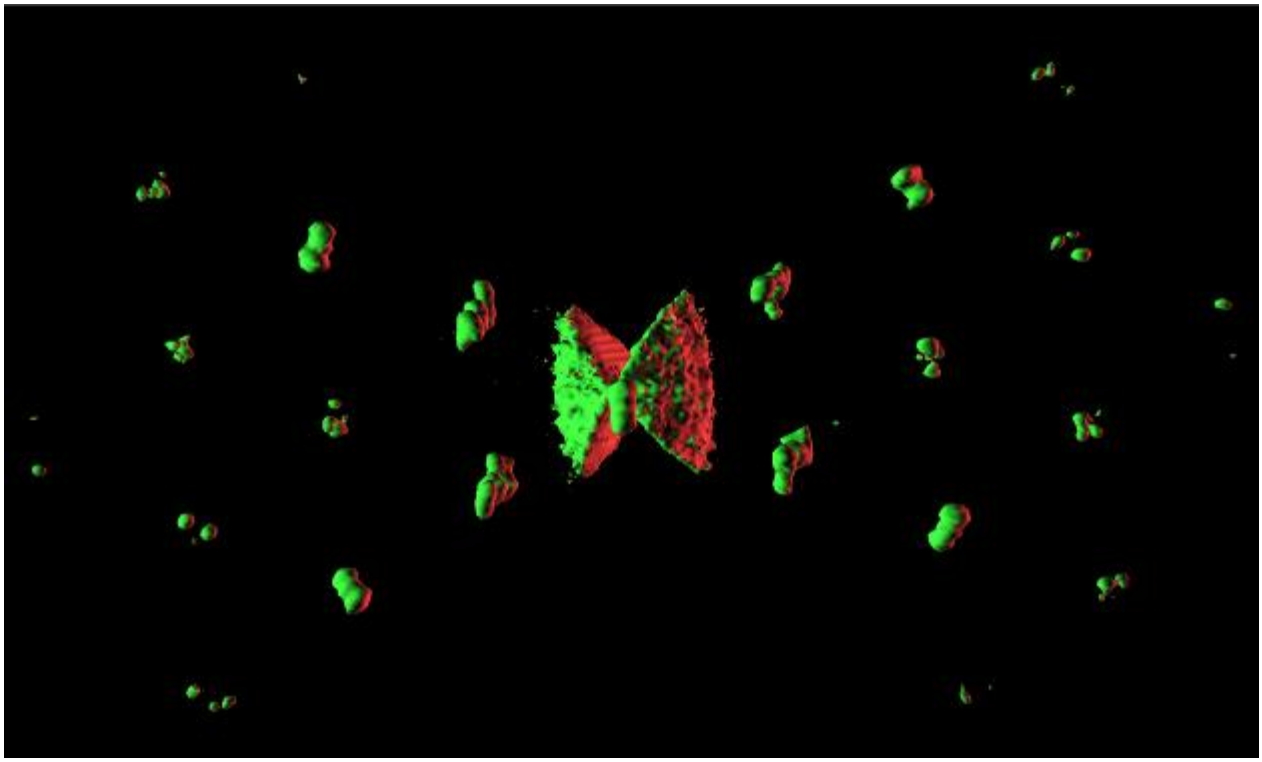

Figure 2. Reconstructed 3D volume of reciprocal space (113° coverage) using 700 unique electron diffraction frames recorded from the individual $GdPO_4$ nano-rod (view along c* axis).