Modeling and quantification of intergrowth in γ-MnO_2 by laboratory PDF analysis

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Supplementary material

Validation of the equipment setup

Our setup implying the use of a molybdenum anode in a Bragg-Brentano geometry leads to higher penetration of the X Ray beam in the sample, as compared to that of copper radiation. A too thin specimen should lead to additional signal coming from the sample holder, the amount of this signal being 2θ dependant (the sample thickness as "seen" by the X Ray beam decreases as 2θ gets close to 180°). As a consequence, the participation of the sample holder has to be carefully checked since its signal will not be as easy to detect in a G(r) function (extra inter atomic distances indiscernible from expected ones) as in a classical XRD experiments (additional peaks or bumps depending on the nature of the sample holder).

To avoid this problem without to have to take into account an angle dependence of the sample holder contribution, we chose to use a sample thick enough. The worse situation being obtained for light material, a sulphur sample was considered to be the limit, in terms of lightness, we could envision for checking our experimental setup (for inorganic compounds). Sulphur absorption coefficient μ being 2.084 mm^{-1}, we calculated that, for a 2θ angle of 180°, only 6×10^{-4}% of the incident beam reached the sample holder for a sample thickness of 6 mm.

Figure 3 shows the PDF of sulphur refined using the PDFgui software.

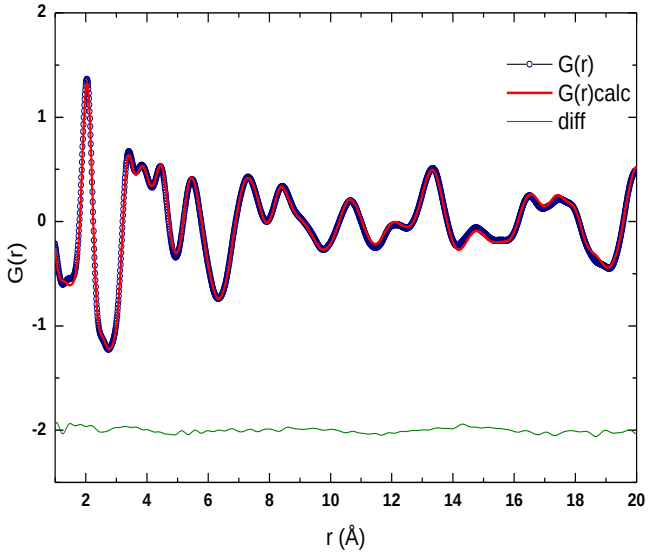

Figure 1 PDF refinement of sulphur with a 6 mm sample thickness (the difference curve is offset by -2). *Rw* = 0.06.

The difference curve between the experimental and the calculated PDF shows that the collected signal is not affected at all by the sample holder. This result clearly validates our experimental setup.

For γ-MnO_2 (μ = 12 mm^{-1}), the same beam transmission to the sample holder as for sulphur compound is achieved for a 1mm thick sample. Thus classical Bruker PMMA specimen holder was used.