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The structure of pseudo-cubic Mn2O3 solved by electron crystallography

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The emerging electron crystallography is a powerful tool for the determination of the atomic structures of crystals. Many examples have been shown in which electron crystallography has been able to solve even complex structures correctly. However, compared to X-ray crystallography it remains a delicate and time consuming method. Therefore it finds its real application in cases where X-rays are not sufficient to solve the structures. Prominent examples are multi-phase powders constituted of nanometre sized grains. In this work we present one of these "real" cases. The sample studied is a nano-sized powder of β-MnO_2, which is interesting for applications in batteries, but which contained a few percent of an unexpected phase. Powder X-ray diffraction only showed a few weak peaks in the β-MnO_2 spectrum, which were insufficient for a phase determination. Using electron diffraction in a Philips CM300ST transmission electron microscope, we identified the minority phase to be pseudo-cubic α-Mn_2O_3. The structure determination was done using the diffracted intensities of 16 different zone axes. Since the diffracted intensities are known to be affected by the effects of dynamic diffraction it is not possible to employ the standard methods of crystallography to solve the structure. We have therefore acquired high resolution electron micrographs (HREMs) of the main zone axes of the cubic structure. The Fourier Transform of the HREMs yields the phases of the structure factors, which are much more sensitive to the atomic positions than the intensities. The combination of the measured diffracted intensities and the phases obtained from the HREM allow solving the structure.

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Synchrotron X-ray diffraction for art history, archaeology and heritage conservation

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From the identification of minute pigment samples to the use of small angle X-ray diffraction to characterise the ageing of ancient Human and animal hairs, the application of synchrotron diffraction techniques covers a wide range of heritage and archaeology needs. Synchrotron beams can now be focused to sub-micrometric spots while maintaining a reasonably low divergence, thus allowing very precise mapping of structural information. Indeed, working at such a length scale is absolutely essential to cope with the strong heterogeneity of cultural heritage materials.

The development of these applications at the new SOLEIL synchrotron source takes place in the context of the setting up of a dedicated liaison office for Heritage and Archaeology research.