m15.p02

Structure of $Cs_xNb_{2.54}W_{2.46}O_{14}$ determined by exit wave reconstruction

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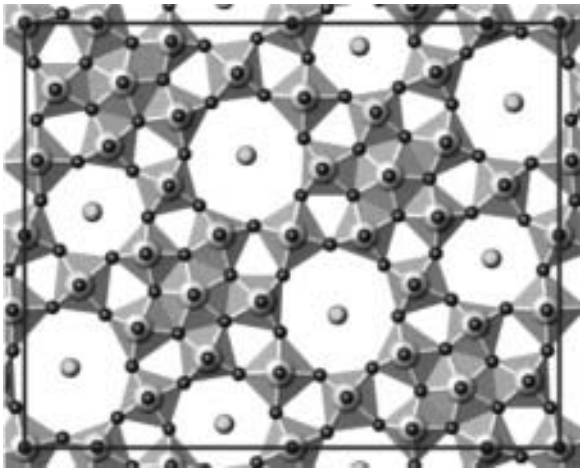
A severe drawback of standard X-ray powder diffraction for structure determination are the extreme difficulties in detecting light atoms in the presence of heavier ones. Due to this obstacle previous X-ray and TEM studies on the polycrystalline title compound failed to locate the positions of the oxygen atoms. This prompted us to employ exit-wave reconstruction on HRTEM images obtained with a spherical-aberration corrected TEM, because both methods have shown to reveal the oxygen columns in perovskite ceramics. Thus combination of the former with the established crystallographic image processing method of HRTEM images is most promising to reach the so far unmatched goal of determining the all atom positions in $Cs_x(Nb, W)_5O_{14}$. To reach this a series of 18 high-resolution images with defocus values between +23.0 and -35.4 nm were recorded along the short 0.39 nm crystal axis of the structure (C_S = -75μm). Subsequent application of numerical exit-plane wave function reconstruction (EPWFR) on this series yielded a phase image that served to determine the projected atomic structure of the crystal. Averaging of nine projected unit cells close to the crystal edge and subsequent application of crystallographic image processing enabled to obtain a set of 2D co-ordinates for all 29 oxygen, two caesium and 11 niobium/tungsten atoms in the asymmetric unit of the structure. The herefrom derived 3D structure model was compared with the result from first-principles calculations on the underlying M_5O_{14} framework to confirm the correctness of the structure. Numerical comparison of the atom co-ordinates from experiment and calculation shows for the heavy atoms an agreement better than 0.09 Å, whereas the average displacement for the oxygen is somewhat larger within 0.13 Å.



Projected structure model of $Cs_x(Nb, W)_5O_{14}$ along [001] as derived from the EPWFR image. Crystal data: a = 27.145(2), b = 21.603(2), c = 3.9463(3) Å, Space group *Pbam* (no. 55).

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On distortion of Kikuchi lines

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As know extended crystalline defects lead to the enlargement of the Kikuchi lines [1]. In accordance with [2] the limited crystalline defects lead to the displacement of the diffraction spots. The goal of the present work is the investigation of the limited defects influence on the transmission Kikuchi patterns of silicon. In the present work Kikuchi patterns of silicon with curvilinear Kikuchi lines were obtained. The distortion both of only the excess Kikuchi lines and of the defect-excess pair lines were observed in the diffraction patterns. In the last case the distortion occurs both to the same side and to the opposite sides. The distortion of the Kikuchi lines increases in sections, which are more distant from the track of the primary electron beam. In the spot diffraction patterns, obtained after an additional chemical etching of the same samples and under the same conditions, the displacement of the spot reflection from their normal position was observed. It is concluded, that the distortion of Kikuchi lines is also caused by the presence of the limited defects in the silicon investigated samples. Thus, if extended defects lead to the Kikuchi lines enlargement [1], then the limited defects lead to the distortion of the Kikuchi lines.

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