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Direct refinement of the atomic structure of MoS$_2$ layered compounds from PXRD

I. Bushmarinov[1], A. Goloveshkin[1], A. Golub[1]

[1]*Institute of Organoelement Compounds RAS, X-ray Structural Centre, Moscow, Russia*

The graphene analog MoS$_2$ can be exfoliated into single-layer dispersion of MoS$_2$ crystals by reduction of starting material to LiMoS$_2$ followed by detachment of its layers in aqueous solvents. This dispersion reacts with organic salts in solution, producing precipitates of layered MoS$_2$ intercalated with their cations [1]. Most of these compounds have distinct chemical composition and powder X-ray diffraction (PXRD) patterns. The analysis of published data, however, indicates that these diffraction patterns were used only for fingerprinting and estimation of cation size and orientation by checking the positions of the basal (00l) reflections. The low-intensity hkl zone of the diffraction patterns was effectively ignored. In the current work, we report that the diffraction patterns of MoS$_2$ intercalation compounds contain enough information to refine their atomic structure directly. We found that the patterns of (NR$_4$)x MoS$_2$ (R=alkyl, H) compounds correspond to turbostratic disorder of MoS$_2$-organic layers. The patterns were indexed with a common orthorhombic cell. The cell content was Rietveld refined using a modification of a "supercell approach" [2,3] developed for full-pattern modeling of disordered clays. We determined that the Mo atoms in MoS$_2$ layers in the intercalation compounds change the coordination from prismatic to octahedral and that the S atoms form "nanorunnels" containing organic cations. The geometry of the Mo-Mo chains forming in the layer was consistent with the EXAFS data. The ordering of the organic layer depended on the nature of the cation, with (NEt$_4$)x MoS$_2$ notably ordered enough to have specific stoichiometric composition (x=1/6) defined by the intralayer packing. The PW-DFT-d calculations based on our model confirmed our results. The developed approach allows determination of previously inaccessible atomic structures of a wide class of MoS$_2$-based layered compounds using commercially available software and laboratory X-ray diffraction data.

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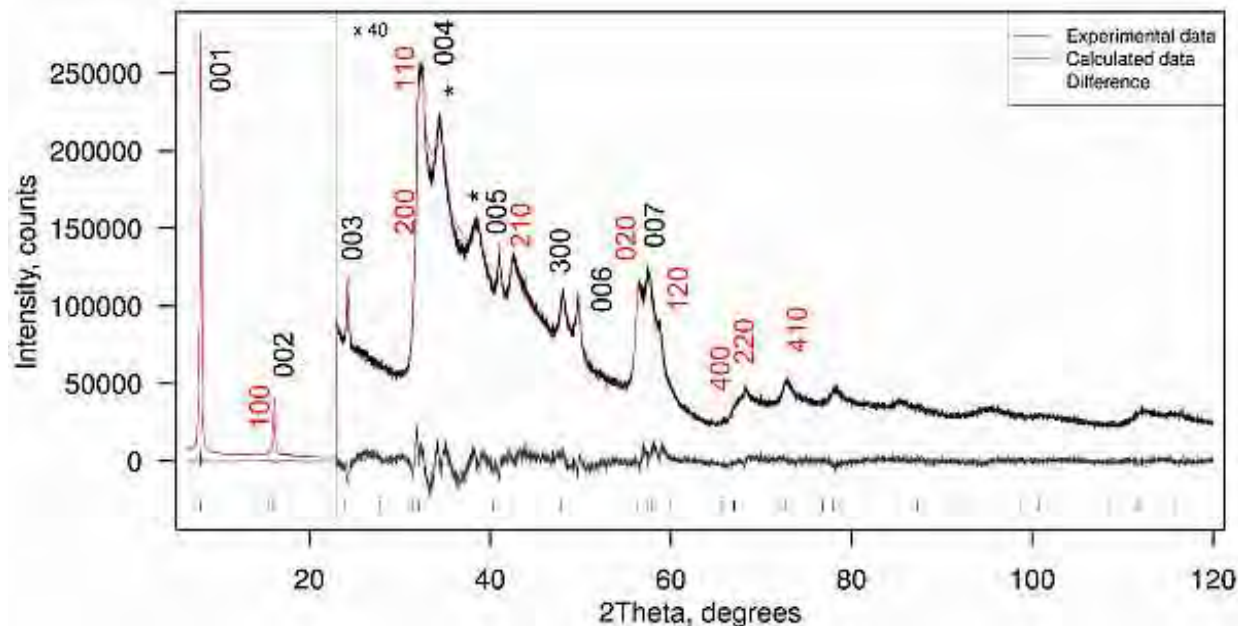
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