

A new approach for the synthesis of layered niobium sulfide and restacking route of NbS₂ nanosheet

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We have developed a new process for the synthesis of a layered niobium sulfide that involves heating $K_4Nb_6O_{17} \cdot 3H_2O$ with a H_2S/N_2 gas mixture. It was confirmed that heating the starting layered oxide at 750 °C for 10 h under the gas flow yielded a highly crystalline, single-phase $K_{0.34}(H_2O)_{0.7}NbS_2$. The layered sulfide slabs had a large plate-like shape. Potassium ions in the interlayer of $K_{0.34}(H_2O)_{0.7}NbS_2$ could be exchanged with protons by stirring in 2M H_2SO_4 . It was found that the proton in the proton-exchanged form can be easily exchanged with other cations. The proton-exchanged form was exfoliated into NbS₂ nanosheets by ultrasonication in water. According to the atomic force microscopy (AFM) images, NbS₂ nanosheets had a thickness of around 4 Å, which roughly corresponded to the thickness of a single NbS₂ host layer. NbS₂ nanosheets could be restacked with the intercalation of Eu^{3+} or tetrabutylammonium ions by an electrostatic self-assembly deposition (ESD) technique.

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