

A New Approach for the Preparation, Exfoliation, and Intercalation Properties of Niobium Sulfide Material

大学院自然科学研究科	後期課程	井澤 一 欽
〃	助 教	伊田進太郎
〃	博士研究員	ウナー・ウグー
〃	前期課程	山口 朋 紀
〃	教 授	松本 泰 道

Some layered titanates and Niobates were delaminated to the nanosheets which have about 1nm thickness and submicrometer length. Nanosheets can be restacked to layered oxides or deposited as thin films by using the electro-static principles. According to our research, new functional materials can be easily fabricated by mixing the solutions of nanosheets and guest species. However, most of the reported nanosheets were oxide type. Therefore, the aim of research is synthesis of S-doped layered material by our proposed method which is the heat-treatment of layered oxide in H₂S/N₂ gases and researching the delamination and intercalation properties.

Layered potassium niobate (K₄Nb₆O₁₇) was synthesized by the conventional solid state reaction. After washing the powder, layered oxysulfide was prepared by heat-treatment with the mixture of H₂S and N₂ gases. The powder was treated with 2M H₂SO₄ for 48h to protonate the interlayers. The proton exchanged powder was stirred in tetrabutylammonium (TBA) to intercalate the amine molecules.

The new layered material was produced by the heat-treatment in the presence of H₂S at 923K with K₄Nb₆O₁₇ 3H₂O, of which the XRD pattern resembled K_x(H₂O)_yNbS₂. According to the XRF and XPS measurement, however, the elemental composition was estimated as K_xNbS_{2-y}O_y (x and y would be in the range 0.2-0.4 and 0.5-1.0). The suspension of partially delaminated layered oxysulfide sheets was obtained by washing proton exchanged powder with water several times. It was confirmed that the suspension have some slabs which have about 1.5nm thickness and 200nm length by AFM measurement. The XRD pattern of TBA intercalated powder confirmed the intercalation with the appearance of a peak in lower angle side. These results showed that the H₂S heat-treatment niobium layered oxysulfide have the capability of the ion exchanging, intercalation properties and delamination of host layer.