Fabrication of Layered Niobium Oxysulfide and Investigation of Structural and Elemental Composition

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Layered oxide materials are composed of repeating two-dimensional host oxide layers with sandwiched cations in-between. The host oxide layers were delaminated by intercalating with the large size of amine molecules. The delaminated host layers have planar surfaces with an area of a few micrometers square with the thickness of approximately one nanometer, therefore they are called nanosheets. When the colloidal solution of nanosheets with negative charge and an aqueous solution of cationic species were mixed at appropriate pH, the re-stacked layered oxides can be fabricated immediately. The greatest advantage of this method is to intercalate any cation easily at the room temperature. But now, there are just a few types of nanosheets reported for this issue, and it is very important to synthesis new type of nanosheets to develop various novel functional layered materials. Therefore, the purpose of this research is synthesizing the layered niobium oxysulfide, and investigating the structure and elemental composition. The starting material of $K_4Nb_6O_{17}$ was prepared by conventional solid state reaction. The powder was heat-treated in the mixture of H_2S and N_2 gases. The final product was treated with H_2SO_4 to protonate the interlayer. The proton exchanged powder was stirred in the Tetrabutylammonium (TBA) solution to intercalate with amine molecules. The material was analyzed by XRD, XPS, SEM, and ICP measurements. The powder heat-treated in the presence of H_2S at 923K was revealed to have the layered structure differently from the XRD pattern of $K_4Nb_6O_{17}$. The SEM microphotograph of the powder also showed that it had the platelike microcrystals. The elemental composition of the powder was revealed that it contained the K, Nb, O, and S according to the XPS 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 H_2S heat-treated powders have the layered structure and have the capability of intercalating large size amine molecules

(2006 MRS FALL MEETING ABSTRACT, Symposium QQ.3.15, 2006.11)