SYNTHESIS, MORPHOLOGY AND STRUCTURAL PROPERTIES OF MICROCRYSTALLINE $RbSm(MoO_4)_2$

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This study is a part of wide current observation aimed at exploring new useful materials among complex molybdate compounds. Double molybdates $A^+Ln^{3+}(MoO_4)_2$, where A^+ is an alkali element or Tl^+ and Ln^{3+} is an rare earth element, show different crystal structures depending on element content. These molybdates are promising as new laser host materials. Present study is aimed to evaluate the formation of $RbSm^{3+}(MoO_4)_2$, in quasi-binary systems Rb_2MoO_4-$Sm_2(MoO_4)_3$ and observe the morphological and structural properties of the molybdate.

The commercial molybdenum trioxide, rubidium carbonate (special purity grade) and samarium oxide with content of the major substance more than 99.9% were taken as starting materials. Crystalline molybdate samples were prepared in ceramic crucibles using the standard solid state reactions. Initially, rubidium and samarium molybdates were obtained. To avoid a loss of molybdenum oxide due to its high volatility, heat treatment of stoichiometric mixtures was started at T = 450°C and followed by step-wise temperature increasing up to T = 600°C for Rb_2MoO_4 and 800°C for $Sm_2(MoO_4)_3$, respectively. A powder mixture of these compounds was ground in an agate mortar, preheated at T = 450°C for about 50 h and then was fired at T = 600°C by 150 h to yield $RbSm(MoO_4)_2$. Finally, the samples were cooled to room temperature. All synthesis procedure were carried out in the air.

Micromorphology of the final product was observed by SEM with LEO 1430 (СКР Nanostructures) device. Microcrystals are formed by slightly agglomerated plate-like crystals with typical dimensions 12×6×1 μm with smoothed edges. Crystal habit is appeared to be governed by a layered structure typical for $MLn(MoO_4)_2$ -type molybdates. The diffraction data for Rietveld analysis were collected at room temperature (298 K) with a Bruker D8 ADVANCE powder diffractometer in the Bragg-Brentano geometry and linear Vantec detector (*CuKα* radiation, step size 0.016°, counting time 2s per step). The refinement of structure with *Pbcn* space group was stable and led to minimal *R*-factor. Formation of $RbSm(MoO_4)_2$ with structural parameters a = 514.31(2), b = 1881.95(7), c = 816.41(3) pm, V = 790.21(5)×10^6 pm^3 was found.

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