EFFECT OF TIN OXIDES ON THE PHASE COMPOSITION AND STRUCTURE OF COO-AL,O₃-SNO₂ AND NIO-AL,O₃-SNO₂ SPINELS OBTAINED BY SHS METHOD

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Spinels are a traditional material for the production of ceramic pigments with a wide range of colors. Sky-blue pigment (ceruleum) is known to contain tin dioxide, cobalt aluminate (blue cobalt), as well as magnesium and zinc oxides. Therefore, in the work, SnO₂ tin oxide is added as an additive that influences on the color of pigment.

Pure color pigments were obtained by the SHS method under the air atmosphere in the MgO-CoO-SnO₂-Al₂O₃ system from MgO, Al₂O₃, Co₂O₃, SnO₂ oxides and aluminum powder (ASD-4). The thermogravimetric analysis conducted using a SDT Q600 thermal analyzer showed that the oxidation of aluminum proceeded through stages:

 $SnO_2+4Al+2O_2=Sn+2Al_2O_3(1)$, $CoO+2Al+O_2=Co+Al_2O_3(2)$, $4Al+3O_2=2Al_2O_3(3)$

The green mixture is heated during these reactions before starting the synthesis of spinels.

CoO+Al₂O₃=CoAl₂O₄ (4), MgO+ Al₂O₃=MgAl₂O₄ (5), 2CoO+SnO₂=Co₂SnO₄ (6)

The increase in mass after ~ 1100°C is connected with oxidation of tin.

 $2Sn+O_2=2SnO(7)$, $2SnO+O_2=2SnO_2(8)$

Tin and its oxide SnO are strong reducing agents, which leads to the formation of a large number of cobalt inclusions in the product during synthesis.

X-ray diffraction analysis (DRON-UM1 diffractometer with filtered Co $k\alpha$ radiation) of MgO-CoO-SnO₂-Al₂O₃ pigment showed that the products contained corundum and the large amount of cobalt along with the spinel phases.

The study of the pigment section showed that the main phases were a mixture of cobalt and magnesium aluminospinels, and there were also the blue inclusions of Co₂SnO₄. In addition, particles of Co₃Sn₂ intermetallide were detected, which was confirmed by micro-X-ray spectral analysis (Camebax) (Fig. 1).

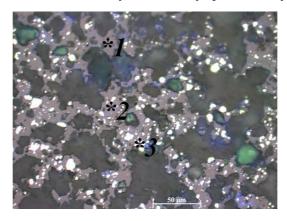


Fig. 1. Micrograph of MgO-CoO-SnO₂-Al₂O₃ pigment, Axiovert 200M, where 1 is (Co, Mg)Al₂O₄, 2 is Co₂SnO₄, 3 is Co.

IR spectroscopic analysis (Nicolet 5700 FT-IR spectrometer) showed that MgO-CoO-SnO₂-Al₂O₃ pigment had absorption bands typical for the spinel structure.

Spinel-type pigment synthesized by the SHS method in the MgO-CoO-SnO₂-Al₂O₃ system has a bright blue color; however, a large amount of the formed metal phase significantly degrades its quality.