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EFFECT OF MECHANOCHEMICAL ACTIVATION ON HIGH-TEMPERATURE SYNTHESIS AND PHASE FORMATION IN THE ZnO-MgO-Co_3O_4-$Mr(NO)_2 \cdot 6H_2O$-Al_2O_3-Al SYSTEM

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Increased demand for inorganic pigments is due to their longer service life compared to organic pigments. Stable colors, chemical resistance to acids and alkalis, resistance to high temperatures, and environmental friendliness - these are the properties of spinels that are required for coloring ceramic and construction products, food ware, plastics, rubber, etc. Spinel pigments have also proven themselves for coloring polymers used for 3D-printing. Energy saving in the production of refractory materials is becoming increasingly important. There has been a growing interest in combining self-propagating high-temperature synthesis (SHS) and mechanochemical activation (MA) methods of initial reagents. This is due to the fact that the preliminary MA of powder reaction mixtures extends the combustion range for inorganic materials, reduces their initial ignition temperatures, and helps to achieve homogeneity of the final product [1].

Oxides of ZnO, MgO, Co_3O_4, Al_2O_3, magnesium nitrate $Mg(NO_3)_2 \cdot 6H_2O$, and Al powder (ASD-4) were used to obtain blue pigments in the system ZnO-MgO-Co_3O_4-$Mr(NO)_2 \cdot 6H_2O$-Al_2O_3-Al by SHS. Starting powders were mixed and subjected to MA in a M3 planetary ball mill (45 g acceleration) with 1 dm^3 steel drums using steel balls with a diameter of 7 mm. The mass of the powder mixture was 0.05 kg, and the ball-to-powder mass ratio was 4:1. Synthesis was conducted in a gradient furnace in the air at atmospheric pressure. The upper part of the samples, where the furnace temperature was maximum, was ignited. The heat transferred from the spiral initiated a chemical reaction, resulting in a combustion wave propagated from top to down. Temperature-time profiles during SHS of spinel were measured using a 100 μm diameter tungsten-rhenium thermocouple BP5-BP20 placed in the center of the samples.

The main SHS parameters (Fig. 1) and the composition of combustion products were studied as a function of MA time for the cobalt-containing reaction mixture.

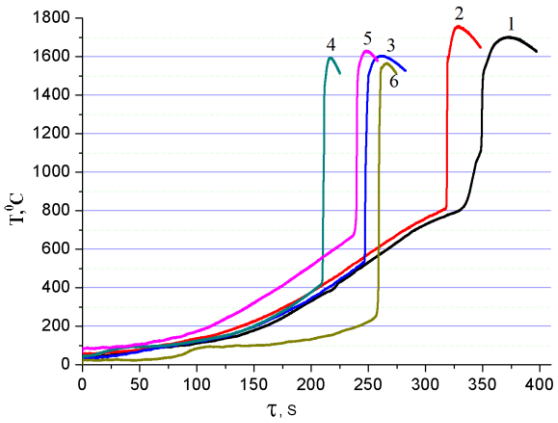

Fig. 1. Thermograms of SHS of blue pigment at different MA times, where (1) no MA, (2) 5 sec MA, (3) 30 sec MA, (4) 60 sec MA, (5) 90 sec MA, (6) 120 sec MA.

It was shown that an increase in the MA time leads to the change in the maximum combustion temperatures and the initial ignition temperatures, which depends on mixing, particle size and structural changes on the surface of starting components. IR spectroscopic analysis performed on a Nicolet 5700 FTIR spectrometer confirmed changes in the molecular structure of reactive compositions depending on the time of MA of a starting reaction mixture. Scanning electron microscopy (Philips SEM 515) showed the nuclei of a new phase (spinel) on the surface of Al_2O_3 particles, which influence the combustion process of the system. The MA of the starting reaction mixture improves the purity of pigment colors.

REFERENCES

[1] A.S. Rogachev, Mechanical activation of heterogeneous exothermic reactions in powder mixtures/RUSSIAN CHEMICAL REVIEWS (2019) V. 88, №9.-P.875-900; DOI 10.1070/RCR4884