SINGLE-STEP SYNTHESIS OF ULTRAFINE PARTICLES OF COMPLEX OXIDES OF TRANSITION METALS FROM NITRATES UNDER THE ACTION OF A GLOW DISCHARGE*

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In the 21st century, one of the main challenges in science and manufacturing is the production of cheap, renewable, and environmentally friendly materials based on nanoparticles [1]. Therefore, complex oxides of transition metals are increasingly gaining popularity. Due to their improved properties, these materials are widely used in various fields, such as electronic devices [2] and catalysis [3]. However, traditional synthesis methods are expensive, require complex equipment, and lead to environmental pollution. Based on this, we propose a new simple method for producing nanoparticles based on the use of low-temperature gas-discharge plasma at atmospheric pressure in air.

The installation was a quartz tube (height 8 cm), the bottom of which was made of stainless steel (cathode) with 0.5 mm holes through which air was supplied at a speed of 5 l/min. The top of the tube was closed with a fluoroplastic cap with a hole for a titanium anode. The interelectrode distance was 5 mm. Discharge current 50 mA. The discharge burning time is 5 minutes. Mixtures of crystal hydrates of various nitrates were poured onto the cathode. The initial mixture of nitrates was $Ni(NO_3)_2$ with $Cu(NO_3)_2$, $Co(NO_3)_2$ with $Ni(NO_3)_2$, $Zn(NO_3)_2$ with $Cu(NO_3)_2$. Compounds were studied in a wide range of initial concentrations, but below we discussed a 1:1 molar ratio, which when mixed gave the corresponding complex oxide. The resulting particles were washed off from all elements of the cell with distilled water into a beaker and then dried at a temperature of 50°C until the water completely evaporated.

The appearance of the particles and their elemental composition were determined using SEM (Tescan Vega 3SBH, Czech Republic) with an EDS system (Aztec EDS, Oxford Instruments Ltd., England). The phase structures of powders were characterized by X-ray diffractometer (XRD: DRON 3 M, Burevestnik, Russia) with Cu Ka radiation of 0.154 nm wavelength. The diffraction patterns were processed using QualX2 software [4] and the open crystallographic COD database [5]. The particle size was obtained using DLS (Photocor Compact-Z, Photocor, Russia).

The resulting particles had a well-developed surface morphology, which makes them promising for future use as catalysts. The average size of the resulting particles according to DLS data was ≈98±20 nm. As a result of plasma treatment of the starting nitrates, according to X-ray diffraction data, the resulting products were crystalline complex oxides $NiCuO_2$, $CoNiO_2$, $ZnCuO_2$. EDS analysis also gave the same composition.

Since there is no information on this type of synthesis, we proposed possible mechanisms for the formation of oxide particles. One of the possible ways for the formation of oxides from nitrates under the action of low-temperature gas-discharge plasma was thermal decomposition. The first stage was decomposition—removal of its own water of crystallization; Next was the decomposition of nitrates into oxides. However, if we look at the literature data on thermal decomposition, we will find that the cathode does not heat up to the temperatures required for decomposition. Most probable, the cathode with nitrates is subjected to ion bombardment, as a result, nitrates can pass into the gas phase, where they interact with active particles from the plasma, followed by the deposition of oxides.

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