OBTAINING OF COBALT FERRITES BY ATMOSPHERIC PRESSURE DISCHARGE IN AIR*

D.A. SHUTOV[1], K.V. SMIRNOVA[2], A.N. IVANOV[1], S.I. KARTASHOV[1], V.V. RYBKIN[1]

[1] *Ivanovo State University of Chemistry and Technology, Ivanovo, Russian Federation*
[2] *A.V. Topchiev Institute of Petrochemical Synthesis RAS, Moscow, Russian Federation*

Currently, several methods are used to synthesize cobalt ferrite such as the hydrothermal synthesis, mechanochemical synthesis, sol-gel method, micro-emulsion method, co-precipitation, CVD and methods using low-temperature gas-discharge plasma. Of course, each of these methods has its own advantages and disadvantages. We propose a new method for obtaining cobalt ferrite nanoparticles from a mixture of cobalt(II) and iron(III) nitrates located on the cathode of an atmospheric pressure DC discharge in air. The main advantage of the method is the short (several minutes) process time and any additional calcination not required.

The setup scheme is shown in Fig. 1. A direct current glow discharge was ignited between a titanium anode (1) and an aluminum plate (2) that served as a cathode. The cathode-anode distance, *d*, was 1 cm. The open surface of the cathode was limited by a ceramic tube (5) 2 cm in diameter. The discharge current varied in the range (60-100) mA. The discharge burning time was 5 min. The mixture of $Co(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ (analytical grade) various molar ratios were used as objects of study. A weighed portion of powder mixtures of salts (4) was placed on the cathode surface. Under the action of the discharge, particles of the substance were formed on the surface of the tube and cathode, which were collected by washing the tube and cathode with distilled water. The insoluble fraction formed a colloidal solution, which was centrifuged for 15 minutes at a speed of 15,000 rpm. The obtained particles were dried on glass substrates at a temperature of 50°C during the day. XRD (DRON 3M X-ray diffractometer, Burevestnik, Russia), SEM (TESCAN VEGA3 SBH, Czech Republic), energy dispersive spectroscopy (Aztec EDS, Oxford Instrumental, England), dynamic light scattering (Photocor Compact–Z, Russia), magnetometry (VSM, Cryogenic Limited, England) were used for powders analysis.

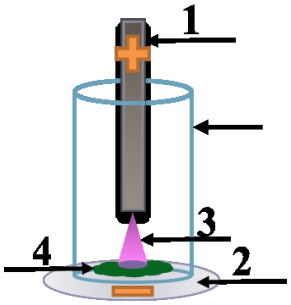

Fig. 1. Scheme of the experimental setup. 1 – titanium anode. 2 – aluminum cathode. 3 – plasma region. 4 – initial salt of metal nitrates (crystal hydrates). 5 – quartz (ceramic) tube.

The data of energy dispersive X-ray spectroscopy and X-ray phase analysis showed that the synthesized powders have a complex phase and chemical composition, which depends on the Fe:Co molar ratio in the initial salts. The best result in terms of yield of cobalt ferrite is obtained with Fe:Co=2:1. The resulting material contains 86 wt. % Fe_2CoO_4, also 13.5 wt. % Fe_2O_3 and 0.5 wt. % Fe_3O_4. At other ratios, Co_3O_4 is also formed. According to dynamic light scattering data, the obtained powders consist of two characteristic fractions. The main fraction (94%) is represented by particles 105 ± 4 nm in size. And the other fraction (6%) consists of particles 18 ± 4 nm in size. The obtained substances have a well-developed surface, which makes them attractive for use in catalysis. The resulting materials have magnetic properties. So, for powders obtained from salts with Fe:Co=2:1 the coercive force was ~490 Oe. The saturation magnetization was ~52 emu/g, and the remnant magnetization was ~22 emu/g. The disadvantages of the method include the fact that the resulting powders, in addition to cobalt ferrite, contain up to 10 weight percent impurities. But, apparently, the process can be optimized by changing the ratio of the initial components, the conduction time, and the discharge current. The available data suggest that in this way it is possible to obtain not only cobalt ferrite, but also ferrites of other metals.

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