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The porous structure of copper-cadmium oxide system prepared by AC electrochemical synthesis

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Abstract

The porous structure of nanoparticles of copper-cadmium oxide system prepared by AC electrochemical synthesis in sodium chloride with a concentration of 3, 15, 25 wt. % has been studied. The obtained data indicate that at a higher current density and sodium chloride concentration of 3 % wt. in the electrochemical oxidation of cadmium and copper products are formed with the structure of mesopores and channels. In this case, the specific surface area has the greatest value (19.4 m^2/g) and a maximum pore volume (0.0778 cm^3/g). Thus, the obtained data allow predicting the operating parameters of the electrolysis to obtain a copper - cadmium oxide system of a predetermined porous structure.

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Keywords: electrolysis, alternating current, dispersion, mesopore, specific surface area, copper-cadmium oxide.

1. Introduction

Technologies of electronic products, which are used as transparent materials, are based on the use of active oxide semiconductors and multicomponent dielectrics[1, 2]. A special role belongs to the semiconductor of n- and p-type[3]. The actual problem is the mutual dosage in a wide range of the ratios of nanostructured materials having different types of conductivity to create composites of the specified composition and products on their base with unique properties. Synthesized materials are used for production of sorbents, catalysts, sensors, electrochemical and photochemical devices[1, 4-8].

Currently, there is a natural interest in the methods of metal oxides synthesis and their dual systems based on the processes taking place in non-equilibrium conditions[9,10]. Unique opportunity to obtain double-oxide systems is the alternating current use under electrochemical synthesis. In particular, the use of alternating current of industrial

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frequency allows obtaining dual-oxide systems with particles in the nanometre range and uniform distribution of the components of the matrix[11].

Despite the fact that electrochemical synthesis is characterized by high energy consumption, it results in the formation of the power-saturated nanomaterials[12].

This work covers the pore structure characterization of products of simultaneous AC electrochemical oxidation of a copper-cadmium system. The main characteristic of powders of oxides is dispersion. Average rate of dispersion is the specific surface area (SSA). The porosity of the product also affects the value of a specific surface area. During the electrochemical oxidation of cadmium and copper products are formed with macro- and micropores and channels. The reasons for this are the non-equilibrium conditions of the process, the presence of current plots on the surface of non-conductive metals, the internal stress of generated particles, etc[13].

2. Materials and Method

The synthetic procedure was as follows: electrochemical metal oxidation was carried out in aqueous sodium chloride solution with concentrations of 3, 15, 25 wt % at the temperature of 100°C and at variation of the current density from 1 to 3 A/cm^2. Sodium chloride was used as an electrolyte (mark "analytical grade") and the plates of copper (mark M1) and cadmium (mark A0) were used as soluble electrodes[14]. The selected electrolyte concentration is based on earlier studies on the speed of individual oxidation of cadmium and copper[15,16].

The surface area of the particle was determined with the device ASAP 2400 Micromeritics. The cross-section of the nitrogen molecule was assumed to be 0.162 nm^2. Total pore volume was calculated at relative N_2 pressure (P/P_0) of p/p0=0.05-0.33. Adsorption measurements with the help of the device ASAP 2400 Micromeritics conducted with the accuracy of the method is ±2.5%. The results are shown in Table 1.

3. Results and Discussion

The characteristics of the porous structure of metal oxides depend on the conditions of electrolysis that have been studied previously. In particular this applies to density changes of the alternating current and the concentration of the electrolyte parameters that have the greatest impact on the speed of processes. Table 1 shows the values of specific surface area, total volume and pore diameter of dry products from electrolysis of cadmium and copper, obtained for different values of the modes of synthesis. The table shows that the obtained products of the electrolysis of copper and cadmium have a developed surface. The highest values of specific surface of samples are obtained by electrolysis in solutions of sodium chloride with a concentration of 3% and 15 wt. % at a current density of 3A/cm^2 (19.4 and 15.6 m^2/g). Thus, we can conclude that the specific surface area increases with decreasing electrolyte concentration and increasing current density. This occurs because a higher current density produces more non-equilibrium process conditions and a product structure is characterized by a large number of defects and has a high surface energy. Also, the findings show that the products have low total pore volume from 0.0065 to 0.0486 cm^3/g. The average value of the pore diameter varies in the range of 11-24 nm.

Figures 1-3 present the integral dependence of the distribution of pore size for different concentrations of sodium chloride in solution.

Table 1. The pore structure characterization of the products of AC electrochemical oxidation of copper-cadmium system in sodium chloride solutions

Samples	Concentration of sodium hydroxide, wt %	Current density, A/cm^2	Surface area, m^2/g	Pore volume, cm^3/g	Pore diameter, nm
1	3	1	8.2	0.0289	14.0
2	3	2	2.2	0.0064	11.6
3	3	3	19.4	0.0778	16.0

4	15	1	2.7	0.0078	11.5
5	15	2	11.5	0.0716	24.9
6	15	3	15.6	0.0485	12.4
7	25	1	3.9	0.0129	13.2
8	25	2	4.2	0.0141	13.2
9	25	3	2.4	0.0142	18.5

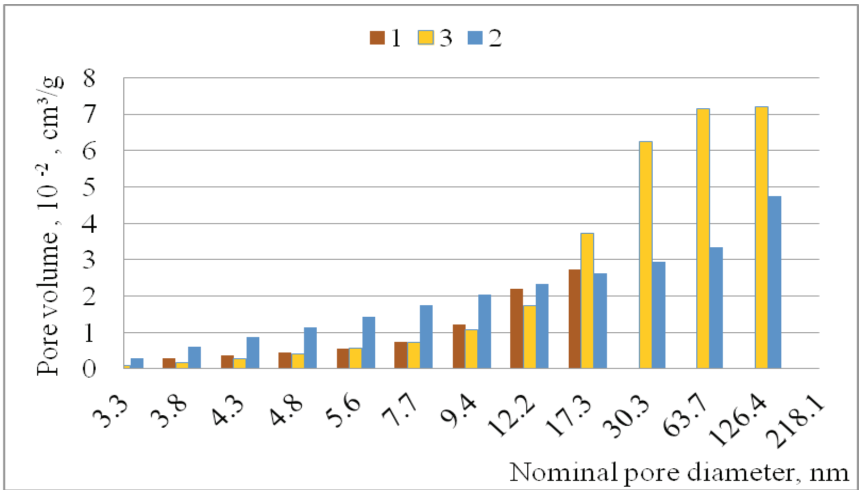

Fig.1. The distribution of the pore sizes for samples 1, 2, 3.

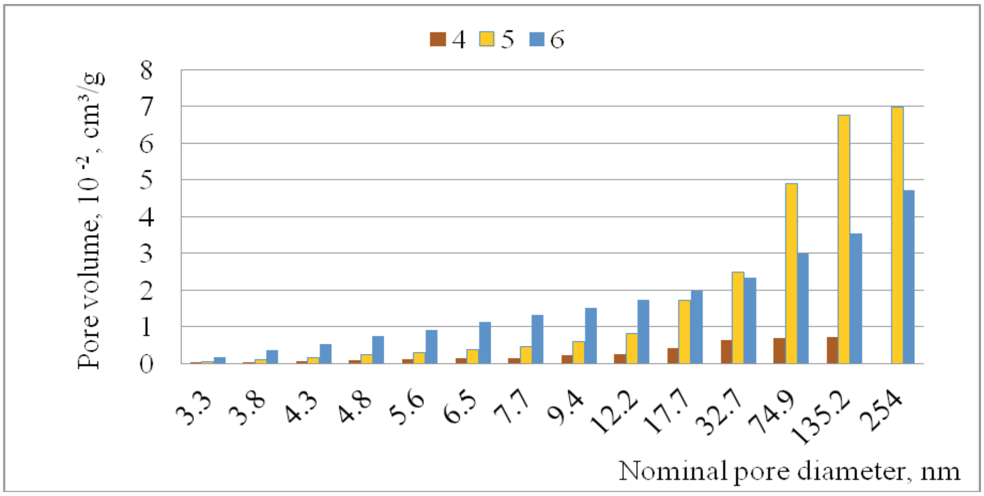

Fig.2. The distribution of the pore sizes for samples 4, 5, 6.

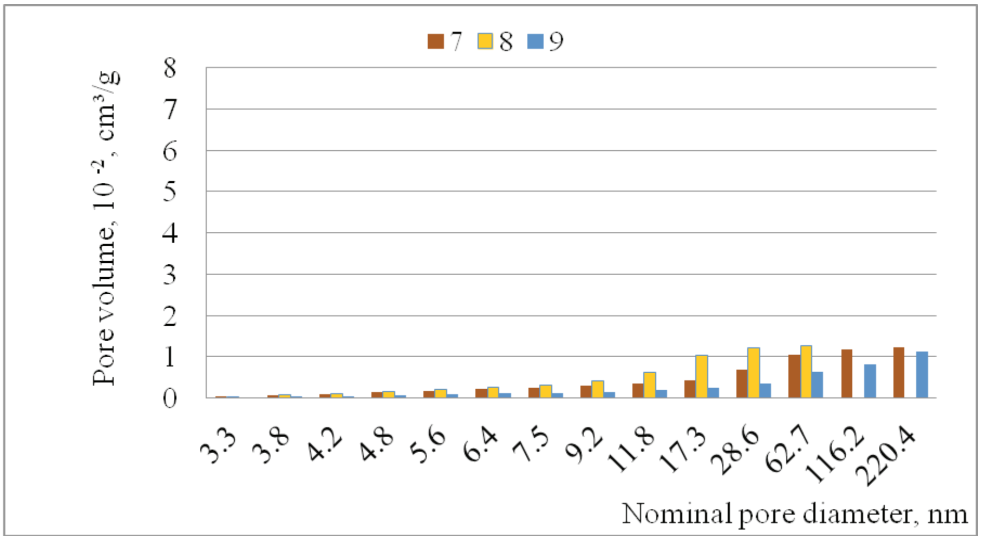

Fig.3. The distribution of the pore sizes for samples 7, 8, 9.

From the histograms, it is evident that the porous structures of the synthesized samples are characterized by a predominance of mesopores. The maximum total pore volume has a sample 3 (Figure 1), the minimum values belong to the products 7, 8, 9 (Figure 3), obtained in solutions of sodium chloride with a concentration of 25 wt. % and at current densities of 1, 2, 3 A/cm^2. However, the product 2, synthesized in solutions of sodium chloride with a concentration of 3 wt.%, contains no more than 17 nm and has a minimum pore volume (0.0064 cm^3/g).

4. Conclusions

1. The material has a maximum diameter of pores in mesoscale range (16-18 nm) at a higher current density.
2. The highest specific surface (19.4 m^2/g) and pore volume (0.0778 cm^3/g) are characterized by the sample obtained at a current density of 3 A/cm^2 and the concentration of sodium chloride of 3 % wt.
3. For the formation of a developed porous structure of the material it is advisable to carry out the electrochemical oxidation of cadmium and copper with an alternating current at low electrolyte concentrations and high current densities.

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