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SYNTHESIS OF ZnO NANOPARTICLES BY THERMAL DECOMPOSITION OF BASIC ZINC CARBONATE

SYNTEZA NANOCZĄSTEK ZnO NA DRODZE TERMICZNEGO ROZKŁADU ZASADOWEGO WĘGLANU CYNKU

The paper presents a method for obtaining nanoparticles of ZnO by thermal decomposition of the Zn-containing compounds. The experiment was based on the thermal decomposition of basic zinc carbonate to zinc oxide (with a content of 58-61 wt.%). Basic zinc carbonate was analysed by derivatography and then annealed at a selected temperature (about 600°C) for about 1 h. Products of thermal decomposition of the compound were studied by XRD analysis and SEM scanning microscopy.

Keywords: ZnO nanoparticles, thermal decomposition, X-ray methods, SEM

W pracy zaprezentowano metodę otrzymywania nanocząstek ZnO na drodze termicznego rozkładu związków zawierających Zn. W eksperymencie wykorzystano termiczny rozkład zasadowego węglanu tlenku cynku (o zawartości od 58-61% mas.). Zasadowy węglan cynku poddano analizie derywatograficznej a następnie wygrzewaniu w określonej temperaturze (ok. 600°C przez ok. 1 godz.). Produkty termicznego rozkładu związku badano analizą XRD oraz mikroskopią skaningową SEM.

1. Introduction

For the production of nanoparticles, two basic techniques are used, i.e. top-down and bottom-up [1,2]. They differ from each other in the process direction. Top-down technique used for a long time consists in grinding the material to a very fine form, while bottom-up method allows making large structures from molecules or single atoms. [3]. Nanoparticles can also be synthesised by electrochemical anodic dissolution of the metal [4,5]. In the reference literature, numerous techniques differing in process parameters are described to obtain nanoparticles of zinc oxide by thermal decomposition of the Zn-containing compounds [6-12].

Zinc oxide as a chemical compound has a broad spectrum of applications. Zinc oxide powders are used in the foundry industry [13]. They are also used in the chemical industry for the production of paints, rubber and plastics, as well as in the pharmaceutical and cosmetic industry. Because of so wide range of the application potentials of the ZnO powder [14], the size and morphology of this material play an important role [15,16].

This paper presents a method for obtaining ZnO nanoparticles by thermal decomposition of a Zn-containing compound.

2. Experimental part

2.1. Test materials, methods and equipment

To obtain ZnO nanopowder, analytically pure basic zinc carbonate ($[ZnCO_3]_2 \cdot [Zn(OH)_2]_3$) supplied by SIGMA-ALDRICH, containing from 58-61 wt.% Zn, was used.

To determine the temperature and time of the decomposition of this compound, a derivatographic analysis was conducted (Fig. 1 a, b, c), and then the sample was annealed at 600°C for one hour in a silite furnace. The test sample was heated and cooled together with the furnace. After thermal treatment, the sample was fractionated and for further analysis the fractions below 0.04 mm (so-called "bottom" fractions) were used.

To identify the chemical composition, the resulting powder was analysed by X-ray diffraction technique (XRD) (Fig. 2). The analysis used the characteristic X-rays of copper cathode CuK α = 0.145 nm. The study was conducted at a voltage of 45 kV and a current of 40 mA, using a Kristalloflex 4H X-ray diffractometer made by Siemens. Qualitative analysis of the material was carried out in Bragg-Brentano geometry on a polycrystalline material.

The size of grains in the resulting powder (Fig. 3 a-d) was determined by SEM, using a SEM Philips XL30 scanning electron microscope with LINK ISIS EDX system and ESEM Philips.
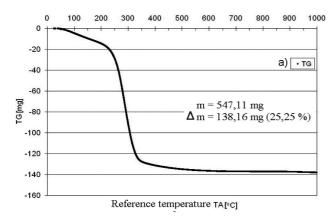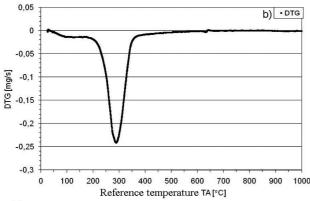
3. Results and discussion

Figure 1 shows the derivatographic analysis (TG, DTG, DTA curves) of basic zinc carbonate heated at a rate of V = 10°C/min. As can be seen from the recorded curves, heating of the sample to 1000°C results in a complete thermal

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decomposition of the compound at 600°C (TG and DTG curves), which is accompanied by an endothermic effect recorded in the form of a large peak at 300°C (DTA curve). The total weight loss of the sample after one hour heating at 600°C amounts to about 25 wt.% (TG curve).
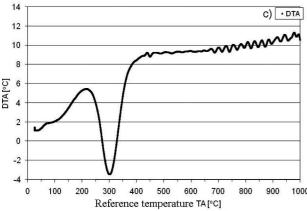

Fig. 1. Derivatographic analysis of basic zinc carbonate: a) TG curve; b) DTG curve; c) DTA curve

The result of the thermal decomposition of basic zinc carbonate is the precipitation of a solid phase in the form of zinc oxide and of a gaseous phase in the form of water and carbon dioxide. The gaseous products of reaction are removed from the system, which correlates with the thermal analysis (Fig. 1: TG and DTG curves). On the basis of these arguments and literature review [11,17], the course of a chemical reaction can be written down according to equation (1):

$$[ZnCO_3]_2 \cdot [Zn(OH)_2]_3 = 5ZnO + 3H_2O + 2CO_2 \quad (1)$$

Figure 2 shows the results of X-ray diffraction of the obtained powder with reflections characteristic for this phase.
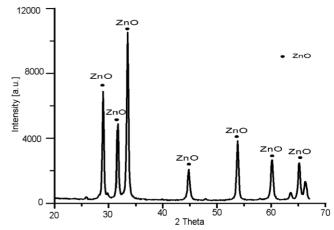

Fig. 2. Phase analysis of ZnO powder

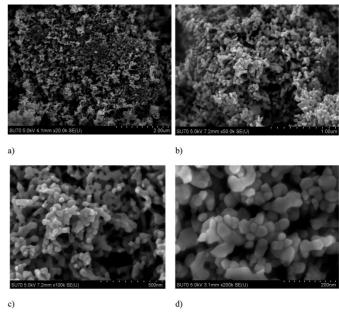The XRD analysis indicated the presence of zinc oxide in the resulting material.

Fig. 3. SEM microscopy of zinc oxide ZnO nanoparticles at different magnifications: a) x 20 k, b) x 50 k, c) x 100 k, d) x 200 k

Figure 3 presents a microscopic image of the obtained ZnO nanoparticles shown at different magnifications. The studies of the nanomaterial show different sizes of the particles comprised in a range of 40 <d <60 nm. The microstructure of nanocrystalline ZnO has a skeletal form resulting from the process of coagulation (Fig. 3 a and b). The nanoparticles formed as a result of the decomposition have the shape of faceted crystals (Fig. 3d). This material is characterised by high porosity.

4. Summary

The study shows that it is possible to obtain zinc oxide ZnO nanoparticles (top-down method) by thermal decomposition of the zinc compound with a high zinc mass content. The zinc oxide nanoparticles is a polydisperse system with the grain size comprised in a range of 40 <d <60 nm. It is characterised by skeletal microstructure and high porosity.

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