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Original article

Application of Sorption Analysis in the Study of Various Nanomaterials Used in Electronics Depending on their Composition and Production Conditions

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Abstract

Introduction. At present, sorption methods of analysis, including the thermal desorption of inert gases, are widely adopted to characterize the porous structure parameters of nanomaterials having a wide range of applications. Nitrogen thermal desorption belongs to the group of non-destructive techniques that provide a rapid analysis of the following parameters exhibited by nanomaterials: specific surface area, average particle size, mesopore size distribution, as well as the presence or absence of micropores in the system. In this work, mesoporous silicon and calcium hydroxyapatite powders are selected as the objects of research. Since modern interference optical filters are cumbersome and expensive to use, meso- and nanoporous silicon nanostructures are of interest in the implementation of filters for fiber-optic communication systems. Hydroxyapatite can potentially provide high corrosion resistance while posing no risk of toxicity to the environment. In addition, anti-corrosion hydroxyapatite coatings are of decisive importance for the practical application of magnesium alloys used to reduce the weight of vehicles, aircraft, and electronics housings.

Aim. To consider the application of the thermal desorption of inert gases, specifically nitrogen thermal desorption, in the study of the porous structure parameters of nanomaterials having various compositions on the example of mesoporous silicon and hydroxyapatite.

Materials and methods. In this work, the thermal desorption of inert gases and capillary condensation were applied to study the porous structure parameters of hydroxyapatite and porous silicon powders. In particular, the nitrogen thermal desorption method was implemented using a Sorbi MS instrument equipped with a Sorbi Prep sample preparation station.

Results. Recommendations are provided on choosing the mass of the adsorbent material required for the study, the sample preparation conditions, as well as the relative partial pressure range of the gas adsorbate. The selected sample types were found to lack a micropore system in the structure. Finally, the dependence of the specific surface area of hydroxyapatite powders and the parameters of its mesoporous structure on heat treatment conditions was analyzed.

Conclusion. The study of nitrogen adsorption and capillary condensation allows the porous structure parameters of hydroxyapatite and porous silicon to be reproduced, which is of great importance for their use in medicine and radio electronics as anticorrosion coatings, as well as for the implementation of optical filters.

Keywords: specific surface area, porous materials, sorption analysis, porous silicon, hydroxyapatite

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**Особенности применения сорбционного анализа
для исследования различных наноматериалов электроники
в зависимости от состава и технологических условий получения**

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Аннотация

Введение. В настоящее время сорбционные методы анализа, включая метод тепловой десорбции инертных газов, широко применяются для характеристики параметров пористой структуры наноматериалов широкого спектра функционального назначения. Тепловая десорбция азота относится к группе неразрушающих методик, обеспечивающих экспресс-анализ таких параметров наноматериалов, как удельная поверхность, средний размер частиц, распределение мезопор по размерам, наличие или отсутствие микропор в системе. В данной статье в качестве объектов исследования выбраны порошки мезопористого кремния и гидроксиапатита кальция. Наноструктуры на основе мезо- и нанопористого кремния представляют интерес при реализации фильтров для систем волоконно-оптической связи, поскольку современные интерференционные оптические фильтры громоздки в использовании и дороги. Гидроксиапатит потенциально обеспечивает высокую коррозионную стойкость и не токсичен для окружающей среды. Антикоррозионные покрытия на его основе имеют решающее значение для практического применения магниевых сплавов, которые используются для уменьшения массы транспортных средств, самолетов, корпусов электроники.

Цель работы. Рассмотрение особенностей применения метода тепловой десорбции инертных газов, в частности азота, для исследования параметров пористой структуры наноматериалов различного состава на примере мезопористого кремния и гидроксиапатита.

Материалы и методы. Применение метода тепловой десорбции инертных газов и капиллярной конденсации для исследования параметров пористой структуры порошков гидроксиапатита и пористого кремния. Метод тепловой десорбции азота реализован с помощью прибора Сорби МС, оснащенного станцией пробоподготовки Сорби Преп.

Результаты. Предложены рекомендации по выбору массы материала-адсорбента, требуемой для исследования, выбору условий пробоподготовки и диапазона изменения относительного парциального давления газа-адсорбата. Установлено, что выбранные типы образцов характеризуются отсутствием системы микропор в структуре. Проанализирована зависимость удельной поверхности порошков гидроксиапатита и параметров его мезопористой структуры от условий термообработки.

Заключение. Исследование процессов адсорбции и капиллярной конденсации азота позволяет воспроизвести параметры пористой структуры гидроксиапатита и пористого кремния, что является важным показателем для их применения в медицине и электронике в качестве антикоррозионных покрытий и для реализации оптических фильтров.

Ключевые слова: удельная поверхность, пористые материалы, сорбционный анализ, пористый кремний, гидроксиапатит

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Introduction. Porous silicon nanopowders are characterized by a high specific surface area contributing to the increased reactivity of this material. As a result, ultrafine silicon powders find widespread use in the chemical industry as catalytic additives and adsorbents, in the production of ceramics and cement, as well as in various areas of medicine and in the implementation of filters for fiber-optic communication systems [1, 3–7]. For example, the prototypes of near UV and visible band-stop filters were developed in [1], including a band-stop filter on the basis of plasmon resonance in composite nanostructured layers (porous silicon-silver).

Biocompatible hydroxyapatite powders are employed in the manufacture of bioceramics. In particular, calcium hydroxyapatite is widely used in such areas of medicine as dentistry and bone tissue engineering as a substitute material for damaged segments [8–13]. Another significant area involves the production of anticorrosion hydroxyapatite coatings. For example, a successful formation of crystalline hydroxyapatite coatings on pure magnesium and its alloys is described in [2]. In addition, the study of nitrogen adsorption and capillary condensation processes allows the porous structure parameters of hydroxyapatite to be reproduced.

Presently, nitrogen sorption at 77 K is commonly used to analyze materials having pores within the size range of 0.5...50 nm. The mechanism underlying this method can be described as follows. At low relative pressure (0.02...0.1), the adsorbate starts to fill micropores. Once adsorption in the micropores is complete, monolayer adsorption takes place. Initially, capillary condensation can be observed in relatively small mesopores when the relative pressure and pore width correspond to the Kelvin equation. A desorption isotherm is obtained by reversing the adsorption process, releasing the liquid adsorbate, and reducing the equilibrium relative pressure [14, 15]. The evaporation process takes place at the condensed liquid meniscus.

The present article aims to consider the application of the thermal desorption of inert gases, specifically nitrogen, when studying the porous structure parameters of nanomaterials characterized by various compositions on the example of mesoporous silicon and calcium hydroxyapatite.

Materials and Methods. Powdered porous silicon was produced from *p*- and *n*-type wafers at the Voronezh State University. These powders were obtained under ultrasonic, electrochemical, and mechanical impact.

Особенности применения сорбционного анализа для исследования различных наноматериалов электроники в зависимости от состава и технологических условий получения
Features of the Sorption Analysis Application for the Study on Various Nanomaterials of Electronics, Depending on the Composition and Technological Conditions of Obtaining

Hydroxyapatite powders were produced via chemical deposition, with the hydrochemical deposition unit comprising the following elements: an ES-61201 magnetic stirrer with heating, a LOIP LT-208 circulating bath, and an inert holder of the reaction bath (inhouse assembly). Calcium nitrate and diammonium phosphate were used as initial precursors. In some cases, the resulting structures were subjected to microwave radiation [9, 11].

The sorption properties of nanomaterials were studied using a Sorbi MS instrument equipped with a Sorbi Prep sample preparation station (META CJSC, Novosibirsk, Russia).

When studying the porous structure parameters of nanomaterials via the sorption method, it is essential to correctly choose the mass value of adsorbent material required for the study, select a sample preparation mode, and establish the relative partial pressure range of the gas adsorbate at which the measurements are to be performed.

1. Choice of the mass value for the test material. Sample collection.

When studying compositions via nitrogen thermal desorption, the choice of the mass value of the material under study is determined by two factors: possibility of obtaining a stable desorption signal used to calculate the desorbed gas volume; total surface area to be measured.

2. Mode selection and sample preparation for the material under study.

Sample preparation of the material under study typically involves the controlled heating of the sample in a flow of inert gas (helium). Preparation varying in terms of heating temperature and duration is primarily aimed at removing moisture and surface contamination.

3. Measurements within the given range of the relative partial pressures of gas adsorbate P/P_0 .

The range of P/P_0 is selected depending on the considered porous structure parameter. The measurement of specific surface area using the Brunauer-Emmett-Teller (BET) method and of the outer surface area, as well as the plotting of mesopore size distribution, imply the choice of different research modes.

Thus, the following parameters are selected for the Sorbi MS instrument used in the present work:

– specific surface area: BET method; relative partial pressures of the gas adsorbate P/P_0 within the range of 6–20 %;

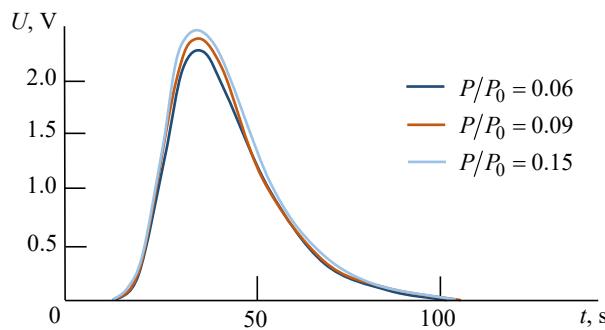


Fig. 1. Nitrogen thermal desorption lines observed at the initial stage of studying mesoporous silicon powder having a specific surface area of $400 \text{ m}^2/\text{g}$
 Рис. 1. Линии тепловой десорбции азота, наблюдаемые на начальном этапе исследования порошка мезопористого кремния с удельной поверхностью $400 \text{ м}^2/\text{г}$

– micropore presence indication (pores smaller than 2 nm): t -method proposed by de Boer; relative partial pressures of the gas adsorbate P/P_0 within the range of 15–40 %;

– mesopore size distribution: capillary condensation of inert gas; relative partial pressures of the gas adsorbate P/P_0 within the range of 6–97 %.

Results. In this work, we studied the porous structure parameters exhibited by nanomaterials of various compositions (silicon; hydroxyapatite) that are characterized by different specific surface areas.

The study consisted in analyzing a series of adsorption isotherms within the relative partial pressure range of the gas adsorbate (nitrogen), determining the specific surface area of each sample via a standard method (BET), as well as establishing the presence/absence of micropores in the sample. Fig. 1 shows nitrogen desorption lines constructed using mesoporous silicon samples, with the area of each formed peak being proportional to the volume of adsorbed/desorbed gas. The lines obtained at the

relative partial pressure of the gas adsorbate within the applicability limits of the BET model are shown as an example.

The study of mesoporous silicon powders revealed that insufficient sample mass can significantly limit the analysis. In order to obtain a stable desorption signal, this method requires a sample mass of at least 5 mg, while the recommended sample preparation conditions include 473 K and 40 min, with the recommended relative partial pressure range of the sample varying from 5 to 98 %. If the pressure of the gas adsorbate exceeds 98 %, the gas flow regulator might not operate properly, resulting in poor data analysis. Since powders lack a system of pores smaller than 2 nm, it seems unnecessary to examine a narrower range (from 5 to 40 %), which is traditionally used in sorption analysis to study micropores. The studies indicate that the specific surface area of powdered mesoporous silicon corresponds to the range of $60 \text{ mg}^2/\text{g} \dots 500 \text{ mg}^2/\text{g}$.

The dependence of the specific surface of powders (Table) and mesoporous structure parameters (Fig. 2 a, b) on the heat treatment conditions was analyzed in the study of hydroxyapatite samples.

The recommended mass of hydroxyapatite samples when using the nitrogen thermal desorption

Specific surface area values for hydroxyapatite samples

Значения удельной площади поверхности для образцов гидроксиапатита

$T, \text{ K}$	$t, \text{ min}$	$S_{\text{BET}}, \text{ m}^2/\text{g}$
No annealing	–	54
423	60	81
573	60	90
873	60	49
1173	60	7

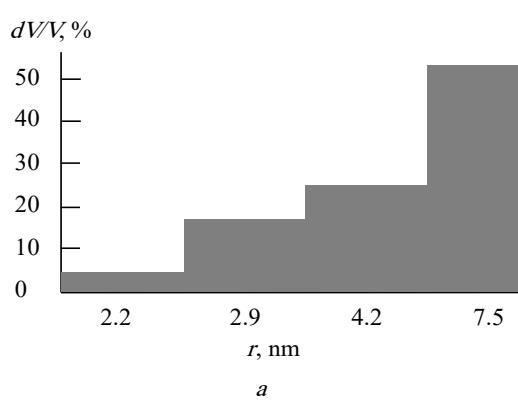
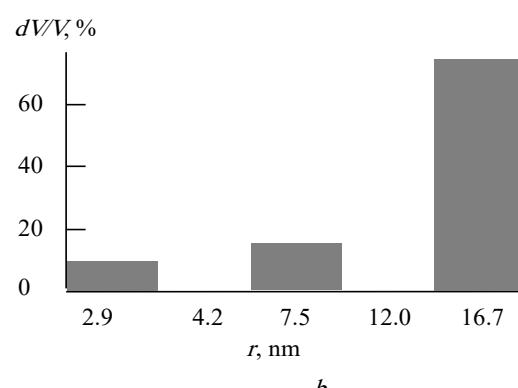


Fig. 2. Pore distribution histogram for hydroxyapatite: a – without heat treatment;
 b – heat-treated at 1173 K

Рис. 2. Гистограмма распределения пор для образца ГАП: a – без термообработки;
 b – с термообработкой при температуре 1173 К



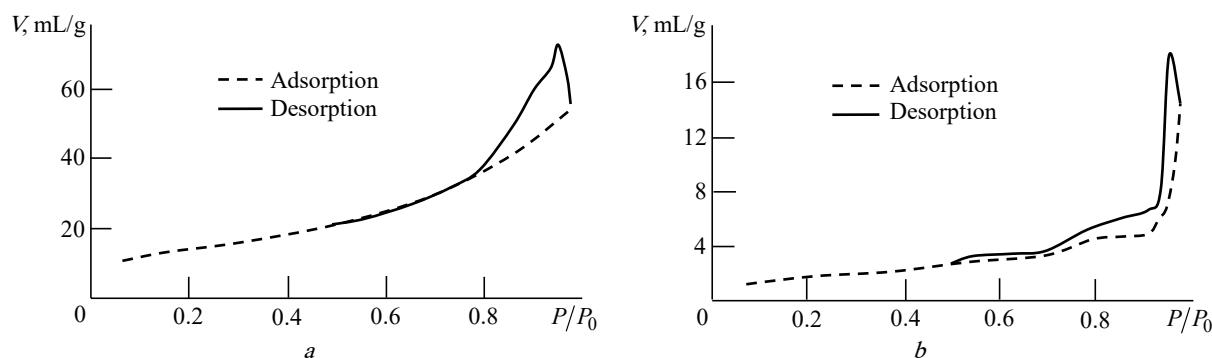


Fig. 3. Complete adsorption isotherm for hydroxyapatite: *a* – without heat treatment; *b* – heat-treated at 1173 K

Rис. 3. Полная изотерма адсорбции для ГАП: *a* – без термообработки; *b* – с термообработкой при температуре 1173 К

method ranges from 150 to 1000 mg, depending on the heat treatment conditions. The sample preparation conditions are as follows: 423 K and 60 min.

Histograms showing mesopore size distribution were constructed drawing on the analysis of complete isotherms of nitrogen adsorption on hydroxyapatite powders. Fig. 3, *a*, *b* presents an example of nitrogen adsorption isotherms for untreated hydroxyapatite and hydroxyapatite heat-treated at 1173 K.

It can be concluded from comparing the data given in the table with those presented in Fig. 2 and 3 that the loss of specific surface area at 1173 K is attributable to pore expansion, which is consistent with the histogram of pore size distribution (Fig. 2) and the disappearance of a pore system with an average radius of 4.2 and 12 nm during sintering (or particle enlargement). The relatively small specific surface area of the untreated sample can be attributed to the presence of moisture, which was removed in all other samples.

Conclusion. In this work, the application of the thermal desorption of inert gases is examined when studying nanomaterials of various compositions on the example of mesoporous silicon and hydroxyapatite.

The recommendations on choosing the mass value for the receipt of a stable desorption signal, as well as sample preparation conditions for the study of mesoporous silicon and hydroxyapatite, are outlined. As a rule, when studying materials via the thermal desorption of inert gases, insufficient sample mass can significantly limit the analysis. Conversely, in the case of nanomaterials having a high specific surface area, an excessive mass of the adsorbent can lead to the receipt of an invalid signal from the thermal conductivity sensor, as well as peak truncation.

The study of sorption properties enables a rapid and inexpensive analysis of the structural characteristics exhibited by hydroxyapatite, porous silicon, and other powder nanomaterials used in radio electronics.

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