



# Конденсированные среды и межфазные границы

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## Аннотации на английском языке

### Review

Review article

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### Stabilization of food emulsion by polysaccharides and protein-polysaccharide complexes: a short review

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#### Abstract

Emulsions are heterogeneous systems consisting of two immiscible liquids, widely used in the food industry as the basis for some products (mayonnaise, sauces, etc.) and components for the production of functional food products containing systems for targeted delivery of biologically active substances (vitamins, nutraceuticals, flavonoids, etc.). From a thermodynamic point of view, emulsions are unstable systems with excessive surface energy; therefore, they are characterized by rapid destruction through phase separation. For the solution to this problem, emulsifiers are used, amphiphilic molecules of various natures that reduce surface tension, i.e., possess surface activity. However, most of these stabilizers are synthetic and toxic products, which significantly limits their use in the food industry. Natural biopolymers, such as polysaccharides and proteins, as well as their complexes, are amphiphilic macromolecules that combine both polar and hydrophobic fragments, have surface-active properties, low toxicity and excellent biocompatibility, thus they can be considered as promising stabilizers for food emulsions. A special place among polysaccharides is occupied by chitosans and alginates, which, in addition to other advantages mentioned above, are accessible and cheap materials.

The purpose of this work was a brief overview of the prospects for using chitosan, sodium alginate and protein-polysaccharide complexes as stabilizers for emulsions and foams for food application. The article discusses the possibility of using chitosan, sodium alginate, propylene glycol alginate, as well as various protein-polysaccharide complexes as stabilizers for heterogeneous food systems, foams and emulsions, which are the basis of many food products. In addition, special attention is paid to the prospects for the introduction of polysaccharide-based emulsifiers into industrial production and the problems that must be solved for the successful development of emulsions stabilized by biopolymers, which are the basis for the creation of food products, are discussed.

**Keywords:** Chitosan, Sodium Alginate, Protein-Polysaccharide Complexes, Food Emulsion, Stabilization

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## Original articles

Research article

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### Physicochemical study of phase formation in the $Sb_2S_3$ - $Cr_2Te_3$ system

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#### Abstract

Chromium chalcogenides and systems based on them have not been sufficiently studied. Chromium chalcogenide compounds  $Cr_2X_3$  ( $X = S, Se, Te$ ), new phases and solid solutions based on them are widely used in semiconductor technology, since these are materials with thermoelectric and magnetic properties. The purpose of this study was the investigation of chemical interactions in the  $Sb_2S_3$ - $Cr_2Te_3$  system, the construction of a phase diagram, and the search for new phases and solid solutions.

Using the methods of physicochemical analysis (differential thermal, X-ray phase, microstructural analysis, as well as density and microhardness measurements), the chemical interaction in the  $Sb_2S_3$ - $Cr_2Te_3$  system was studied and its phase diagram was constructed. The phase diagram of the system is quasi-binary and is characterized by the formation of a quaternary compound  $Cr_2Sb_2S_3Te_3$ .

Compound  $Cr_2Sb_2S_3Te_3$  incongruently melted at 610 °C. Microstructural analysis showed that at room temperature solid solutions based on  $Sb_2S_3$  were formed in the system, which reached up to 5 mol. %  $Cr_2Te_3$ , and based on  $Cr_2Te_3$  up to – 8 mol. %  $Sb_2S_3$ . The  $Sb_2S_3$ - $Cr_2Te_3$  eutectic formed in the Sb system contains 20 mol. %  $Cr_2Te_3$  and has a melting point of 430 °C. The  $Cr_2Sb_2S_3Te_3$  compound crystallizes in a tetragonal system with the unit cell parameters:  $a = 10.03$ ;  $c = 16.67 \text{ \AA}$ ,  $z = 7$ ,  $\rho_{\text{расч.}} = 5.72 \text{ g/cm}^3$ ,  $\rho_{\text{X-ray}} = 5.765 \text{ g/cm}^3$ .



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**Keywords:** System, Phase, Solid solution, Eutectic, Syngony

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Research article

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**Study of the processes of formation of the structure and strength gain of nanomodified cement systems during long-term hardening**

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**Abstract**

The study investigated the influence of nanoscale admixtures on the structure formation and strength characteristics of cement systems during their hardening with a duration of up to ten years. The study of the processes of structure formation of modified cement systems showed that there is a significant acceleration of the process of cement hydration during the early period of hardening, despite the decrease in water content. It was established that phase transformations and changes in phase composition in all systems were observed throughout the entire studied hardening period. At the same time, in the later stages of hardening, the appearance of stable hydrate new formations (xonotlite, afwillite, ettringite), capable of creating a lower-dimensional, dense and homogeneous structure of nanomodified cement stone was observed. This provides nanomodified cement systems with high compressive strength values ( $R_{com}$ ) both during the early and long stages of hardening. At the same time, the highest strength indicators throughout the entire study period ( $R_{com} = 85$  MPa, with a hardening duration of 28 days and  $R_{com} = 157$  MPa, with a hardening duration of 10 years) was characteristic for the cement system modified with a complex nanoscale admixture based on  $SiO_2$  particles.

**Keywords:** Cement systems,  $SiO_2$  nanoparticles, Carbon nanotubes, Structure formation, Long-term hardening, Compressive strength

**Acknowledgements:** The experimental studies were carried out using the facilities of the Professor Borisov Centre for Collective Use, Voronezh State Technical University which is partly supported by the Ministry of Science and Education of the Russian Federation, Project No. 075 – 15 – 2021 – 662.

**For citation:** Artamonova O. V., Shvedova M. A. Study of the processes of formation of the structure and strength gain of nanomodified cement systems during long-term hardening. *Condensed Matter and Interphases*. 2024;26(2): 204–212. <https://doi.org/10.17308/kcmf.2024.26/11935>

Research article

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**Photoelectrochemical activity of oxide films on silver-palladium alloys in an alkaline solution**

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**Abstract**

The continuously increasing energy needs of humanity are causing a number of serious environmental problems. One of the methods for the solution of such problems is the photocatalytic or photoelectrochemical production of a fairly environmentally friendly fuel - hydrogen gas. The studies in this field are mainly associated with the search for semiconductor material that is most suitable for photocatalysis. Oxides of some metals, including silver, can be used as such a material. The photocatalytic or photoelectrochemical activity of the oxide is determined by the features of its electronic structure and can increase significantly when combined with another oxide. Therefore, anodic oxidation of binary alloys is considered as an accessible and, most importantly, controlled method for combining oxides of various metals. The aim of this study was to reveal the role of alloying of silver with palladium in the photoelectrochemical activity of oxide films anodically formed in deaerated 0.1 M KOH.

The anodic formation of oxide films was carried out by the potentiodynamic method in an alkaline medium on silver and its alloys with palladium, the concentration of which ranged from 5 to 30 at. %. Photoelectrochemical activity was assessed by the magnitude of the photocurrent generated in the oxide film directly during its formation and subsequent reduction. The photocurrent was measured in a pulsed lighting mode of the electrode surface with a quasimonochromatic LED with a wavelength of 470 nm.

A positive photocurrent was recorded on all studied samples, which indicates the predominance of donor structural defects in the forming oxide film. With an increase in the concentration of palladium in the alloy, the range of potentials of photoelectrochemical activity of formed anodically oxide films expanded. The maximum photocurrent achieved during the anodic potentiodynamic formation of the oxide film was higher, the lower the palladium concentration was. During the cathodic potentiodynamic reduction of the formed oxide films, it was possible to record even higher values of photocurrents than during their anodic formation. The highest photoelectrochemical activity, characterized by a photocurrent density of  $2.89 \mu A/cm^2$  and incidental proton-to-electron conversion efficiency of 7.6%, was observed in the oxide film anodically formed on silver by the time the potential reached 0.6 V. Comparable values of the photocurrent and quantum efficiency ( $2.12 \mu A/cm^2$  and 5.6%) were recorded in the oxide film on the alloy with a palladium concentration of 10 at. % during its potentiodynamic reduction.

**Keywords:** Silver-palladium alloys, Anodic oxide formation, Photoelectrochemical activity, Photocurrent, Cyclic voltammetry

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Research article

<https://doi.org/10.17308/kcmf.2024.26/11936>**High-temperature gallium sesquisulfides and a fragment of the  $T$ - $x$  diagram of the Ga – S system with these phases**N. Yu. Brezhnev<sup>1</sup>, M. V. Dorokhin<sup>2</sup>, A. Yu. Zavrzhnov<sup>1,✉</sup>, N. A. Kolyshkin<sup>3</sup>, I. N. Nekrylov<sup>1</sup>, V. N. Trushin<sup>2</sup><sup>1</sup>Voronezh State University,

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**Abstract**

It is known that phases with disordered stoichiometric vacancies are promising candidates for new materials with outstanding thermoelectric, radiation-resistant, catalytic, and other properties, which can be explained by a large concentration of the so-called stoichiometric vacancies, caused by the fact that their stoichiometry does not correspond to the structural type. It is interesting to search for such compounds in  $A^{III} - B^{VI}$  semiconductor systems, whose sesquichalcogenides ( $Me_2Ch_3$ ,  $Me = Ga, In$ ;  $Ch = S, Se, Te$ ) are known to have both sphalerite and wurtzite structures and the share of stoichiometric vacancies in the cationic sublattice of about  $1/3$ . The purpose of our study was to determine or confirm the high-temperature structures of gallium sesquisulfides and determine the stability regions corresponding to the phases with these structures on refined  $T$ - $x$  diagrams in the high temperature region ( $T = 878$  °C).

Various methods of structure and thermal analysis allowed us to prove that at temperatures above 878 °C, close to the stoichiometry of  $Ga_2S_3$ , gallium sesquisulfide has four modifications similar in terms of structure, which are connected with each other and other phases of the Ga – S system by enantiotropic transitions. The study confirmed that  $\gamma$ - $Ga_{2.8}S_3$  with a sphalerite-like cubic structure is formed over a narrow temperature range (878 – 922 °C). The composition of the phase was specified (59.3 mol %). The study demonstrated that at temperatures above 912 °C and a slight excess of gallium (up to ~1 mol %) as compared to the stoichiometry of  $Ga_2S_3$  two modifications are formed: a defected wurtzite-like structure ( $\beta$ - $Ga_2S_3$ ,  $P6_3mc$ ) and its derivative phase, whose structure has a lower symmetry ( $\alpha$ - $Ga_2S_3$ ,  $P6_1$ ) and reaches the stage of congruent melting ( $1109 \pm 2$  °C). The study also accounts for the existence of a distectoid transformation  $\alpha$ - $Ga_2S_3 \leftrightarrow \beta$ - $Ga_2S_3$  (~1040 °C). The fourth modification with a monoclinic structure ( $\alpha'$ - $Ga_2S_3$ ,  $Cc$ ) is stable over a temperature range from room temperature to ~1006 °C. Its composition satisfies the formula of  $Ga_2S_3$ . The article presents a corresponding  $T$ - $x$  diagram of the Ga – S system with the areas of existence of the said phases.

**Keywords:** Ga – S system, Phase diagram, Structure, Stoichiometric vacancies, Vacancy ordering, Synchrotron radiation for the structure analysis

**Acknowledgements:** *In situ* X-ray powder diffraction studies were performed using the equipment of the National Research Centre “Kurchatov Institute”. The authors are also grateful to A.V. Naumov, Associate Professor at the Department of General and Inorganic Chemistry of VSU, for his participation in discussions.

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Research article

<https://doi.org/10.17308/kcmf.2024.26/12070>**Deposition of lead sulfide films from “ $Pb(CH_3COO)_2 - N_2H_4CS$ ” aqueous solutions and their properties**E. A. Gannova, M. V. Grechkina, V. N. Semenov, A. N. Lukin, S. A. Ivkov, T. V. Samofalova<sup>✉</sup>

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**Abstract**

The article presents the results of the study of lead sulfide films obtained by the aerosol pyrolysis of solutions of complex compounds of lead acetate and thiourea at temperatures of 300 and 400 °C. The concentration areas of existence of lead (II) hydroxo complexes were determined. We determined the domination regions of  $[Pb(N_2H_4CS)_4]^{2+}$  complexes, which are precursors during the deposition of lead sulfide films.

The crystal structure, phase composition, and surface morphology of the synthesized films were studied by X-ray phase analysis and atomic force microscopy. It was found that under these deposition conditions, the crystallized PbS films have a cubic structure and are textured in the (200) crystallographic direction. When the concentration of thiourea in the initial solution increases, there is an increase in the values of the average and root-mean-square roughness, as well as the relief height difference of the synthesized samples.

PbS films obtained at a temperature of 400 °C are characterized by a denser packing of grains and a perfect surface microstructure. By optical spectrophotometry, we determined the band gap of synthesized PbS, which is from 0.41 to 0.45 eV for direct allowed transitions.

**Keywords:** PbS films, Aerosol pyrolysis method, Atomic force microscopy, Thiourea complex compounds, X-ray phase analysis, Transmission spectra

**Acknowledgements:** The crystal structure and surface morphology of the samples were studied by X-ray diffraction and atomic force microscopy using the equipment of the Centre for Collective Use of Scientific Equipment of Voronezh State University. URL: <https://ckp.vsu.ru>

**For citation:** Gannova E. A., Grechkina M. V., Semenov V. N., Lukin A. N., Ivkov S. A., Samofalova T. V. Deposition of lead sulfide films from “ $Pb(CH_3COO)_2 - N_2H_4CS$ ” aqueous solutions and their properties. *Condensed Matter and Interphases*. 2024;26(2): 238–246. <https://doi.org/10.17308/kcmf.2024.26/12070>

## Research article

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**X-ray luminescence of  $\text{Sr}_{0.925-x}\text{Ba}_x\text{Eu}_{0.075}\text{F}_{2.075}$  nanopowders**

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**Abstract**

We synthesized powders of single-phase solid solutions  $\text{Sr}_{0.925-x}\text{Ba}_x\text{Eu}_{0.075}\text{F}_{2.075}$  ( $x = 0.00, 0.20, 0.25, 0.30, 0.35$  and  $0.40$ ) by a precipitation technique from nitrate aqueous solutions. The lattice parameters increase linearly as the barium content increases. We recorded a significant increase in the X-ray luminescence intensity of europium at increasing barium content. Upon increasing barium content, the intensity of the luminescence of strong  $^5\text{D}_0 \rightarrow ^7\text{F}_1$  band increases exponentially, and we observed blue and red shifts in the position of the europium luminescence bands for  $^5\text{D}_0 \rightarrow ^7\text{F}_1$  and  $^5\text{D}_0 \rightarrow ^7\text{F}_4$ , respectively.

**Keywords:** Strontium fluoride, Barium fluoride, Europium, X-ray luminescence

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## Research article

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**Coupling of anode reactions in the process of electrooxidation of glycine anion on gold**

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**Abstract**

Electrochemical processes involving organic substances are complex multi-stage reactions. In our opinion, it is incorrect to describe their kinetics using the principle of independent partial processes (or their individual stages) since electrode reactions can be coupled due to competition for active surface sites, due to common intermediate stages, or through an electron. In this case, the theory of coupled reactions or the graph-kinetic method should be used to provide the kinetic description of the process. In general, graph theory makes it possible to identify the relationship between the “structure” and the kinetic behavior of complex systems by means of graphical analysis. In the case of electrochemical reactions, structural elements are substances adsorbed on the metal surface and (or) a set of substances interacting in the reactions. The relationship between their concentrations can be characterized quantitatively by a transformation law, for example, the law of effective masses. Thus, a graph is a set of reacting substances and a sequence of reactions represented graphically. Graphs allow setting a system of kinetic equations and analyzing them by associating a certain behavior of the system with the structure of the corresponding graph. Under the assumption that one intermediate particle is involved in each elementary stage, the kinetic expressions will be linear, which corresponds to the first-order reaction model.

Graph-kinetic analysis of the processes within the  $\text{Au}(\text{Gly}^-, \text{OH}^-, \text{H}_2\text{O})$  system confirmed that the partial multi-stage reactions of anode oxidation of glycine and hydroxyl anions are kinetically coupled. We obtained expressions for partial currents of electrooxidation of hydroxide ions and glycine anions during the anodic process occurring on gold in an alkaline glycine-containing solution. It was shown that with an increase in the anode potential, the nature of the limiting stage of the anodic process changes.

Formal constants of rates and equilibria of electrochemical reactions involving particles of background electrolyte and glycinate ion were calculated. It was found that the rates of partial oxidation reactions of adsorbed OH particles and  $\text{OH}^-$  are significantly higher than those of organic anions ( $\text{Gly}^-$  and  $\text{HCOO}^-$ ). This indicates that the kinetics of the electrooxidation processes of  $\text{Gly}^-$  in the  $\text{Au}(\text{Gly}^-, \text{OH}^-, \text{H}_2\text{O})$  system are determined by the kinetic features of the electrooxidation reactions of hydroxide ions.

**Keywords:** Graph-kinetic analysis, Coupled processes, Electrooxidation, Glycine, Voltammetry

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## Research article

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**Structural, optical, and photocatalytic properties of dispersions of CuS doped with  $\text{Mn}^{2+}$  and  $\text{Ni}^{2+}$** 

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**Abstract**

By calculating the ionic equilibria in the system  $\text{CuCl}_2 (\text{Mn}^{2+}, \text{Ni}^{2+}) - \text{NaCH}_3\text{COO} - \text{N}_2\text{H}_4\text{CS}$ , we determined the concentration regions of the formation of copper sulfide (CuS), both undoped and doped with transition metals (Mn, Ni). Using chemical deposition on frosted glass substrates, we obtained powders and thin films of  $\text{CuS}(\text{Mn})$  and  $\text{CuS}(\text{Ni})$  doped with manganese or nickel with a thickness of 170–200 nm. The X-ray diffraction demonstrated that CuS based dispersions have the hexagonal covelline structure (space group  $P6_3/mmc$ ). The band gap  $E_g$  of CuS films (2.08 eV) grows to 2.37 and 2.49 eV after doping with nickel and manganese, respectively. The study demonstrated that  $\text{CuS}(\text{Ni})$  powders have optimal photocatalytic properties in the visible spectral region. The degree of photodegradation of a methylene blue organic dye increases in alkaline environments.

**Keywords:** Chemical bath deposition, Copper sulfide, Thin films, Powders, Doping, Manganese, Nickel, Methylene blue, Photocatalytic degradation

**Funding:** The research was carried out with the financial support of the Ministry of Science and Higher Education of the Russian Federation (agreement No. 075-15-2022-1118 of June 29, 2022). The optical properties were studied at the Institute of Solid State Chemistry of the Ural Branch of the Russian Academy of Sciences (Grant No. 124020600024-5), and the X-ray crystallography was conducted at M. N. Mikheev Institute of Metal Physics of the Ural Branch of the Russian Academy of Sciences.

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Research article

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### Theoretical exploration of halogenated anthracene derivatives: unraveling electronic and molecular insights

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#### Abstract

This research article delves into the profound ramifications of halogenation on anthracene within the captivating domain of polycyclic aromatic hydrocarbons (PAHs). By employing Density Functional Theory (DFT) calculations, the study comprehensively explores the intricate interplay between halogen atoms and the molecular framework of anthracene. The entwining of halogens such as fluorine, chlorine, and bromine with aromatic rings orchestrates a symphony of changes, reshaping electronic structures, reactivity, and optical behaviors. This investigation traverses diverse analytical landscapes, encompassing molecular orbitals and Density of States analysis, UV-visibility spectra, infrared spectroscopy, nuclear magnetic resonance (NMR), and natural bond orbital (NBO) analysis, unveiling the intricate tapestry of molecular modifications. The electronic transitions, vibrational signatures, and NMR shifts of halogenated derivatives illuminate the dynamic effects of halogenation. Moreover, the study contemplates their potential across medicinal, environmental, and optoelectronic landscapes. Ultimately, this exploration presents a comprehensive narrative that harmonizes theoretical insights with practical applications.

**Keywords:** PAHs, Halogenation, Electronic Properties, DFT, Energy States, Frequency Analysis

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Research article

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### The influence of benzoic acid moisture on the proton exchange process in lithium niobate crystals

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#### Abstract

The purpose of this study was the investigation of the influence of water impurities in benzoic acid, used as a source of protons during proton exchange on lithium niobate crystals, on the process of formation of proton exchange waveguides, their structure and phase composition.

To carry out the research, prism coupling method, X-ray diffraction analysis, IR absorption spectroscopy, and optical microscopy in polarized light were used. It was established that an increase in the moisture content in benzoic acid affected the optical characteristics of the waveguides and slightly increased the stress (strain) of the proton exchange layers. Subsequent annealing significantly equalized the characteristics of the waveguides.

When performing proton exchange, the moisture content of benzoic acid must be taken into account to obtain reproducible and stable performance of integrated optical devices with proton exchange waveguides.

**Keywords:** Lithium niobate, Proton exchange, Waveguide, Structure, Phase composition, Benzoic acid, Melt, Moisture

**Funding:** The study was funded by the Russian Foundation for Basic Research and the Perm Krai within the framework of scientific project No. 20-42-596001.

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Research article

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### Anodic dissolution and passivation of manganese monosilicide in fluoride-containing sulfuric acid solutions

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#### Abstract

The purpose of this study was to investigate the anode resistance of manganese monosilicide MnSi in fluoride-containing sulfuric acid solutions and the concentration effect of sodium fluoride on the anodic dissolution and passivation of the silicide.

The study was carried out on a single-crystal MnSi sample in 0.5 M H<sub>2</sub>SO<sub>4</sub> + (0.0025–0.05) M NaF solutions. The study presents micrographs

and elemental composition of the electrode surface after anodic polarization from  $E_{\text{corrosion}}$  to  $E = 3.2$  V in 0.5 M  $\text{H}_2\text{SO}_4$  and 0.5 M  $\text{H}_2\text{SO}_4 + 0.05$  M NaF solutions. A stronger etching of the electrode surface was observed in the presence of fluoride ions; elemental analysis showed an increase in the oxygen content in certain areas of the silicide surface associated with the formation of manganese and silicon oxides and their partial removal at high polarization values.

The kinetic regularities of the MnSi-electrode anodic dissolution were studied by the methods of polarization, capacitance, and impedance measurements. It was established that the addition of fluoride ions leads to weaker barrier properties of the silicon dioxide surface film, which determines the high silicide resistance in a fluoride-free medium. The order of the reaction was calculated for the MnSi anodic dissolution for NaF depending on the potential. In the region of low anodic potentials (from  $E_{\text{cor}}$  to  $E \approx -0.2$  V), the reaction order ranged from 1.8 to 1.1, which was due to the high influence of silicon in the composition of the silicide and its oxidation products. With an increase in the polarization value (up to  $E = 0.9$  V), the reaction order decreased to 0.5. An increase in the contribution of manganese ionization and oxidation reactions to the kinetics of the anodic dissolution of the silicide was observed. The silicide passivation in a fluoride-containing electrolyte was characterized by higher values of the dissolution current density ( $10^{-4}$ – $10^{-3}$  A/cm<sup>2</sup>) as compared to a fluoride-free electrolyte ( $10^{-6}$  A/cm<sup>2</sup>), the reaction order in region of the passive state was  $\sim 1.0$ . Passivation was due to the formation of  $\text{MnO}_2$  and  $\text{SiO}_2$  oxides on the surface. In the transpassivation region ( $E \geq 2.0$  V), there was a weak dependence of the current density on the concentration of fluoride ions. Oxygen release was observed on the surface of the electrode, and the formation of  $\text{MnO}_4^-$  ions was recorded in the near-electrode layer. The article discusses mechanisms and kinetic regularities of anodic processes on an MnSi-electrode in sulfuric acid solution in the presence of fluoride ions.

**Keywords:** Manganese monosilicide, Sulfuric acid electrolyte, Sodium fluoride, Anodic dissolution, Impedance

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Research article

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### Stabilization of the $\text{Ba}_4\text{Y}_3\text{F}_{17}$ phase in the NaF-BaF<sub>2</sub>-YF<sub>3</sub> system

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#### Abstract

The paper describes the study of the phase formation in the NaF-BaF<sub>2</sub>-YF<sub>3</sub> system. It involved solid-phase sintering of the components in a fluorinating atmosphere at 750 °C for two weeks and quenching them in liquid nitrogen.

The prepared samples were placed in nickel capillaries, which, together with barium hydrofluoride, BaF<sub>2</sub>·HF, were placed in copper containers. The containers were sealed by argon arc welding. The fluorinating atmosphere was created by pyrolysis of barium hydrofluoride, BaF<sub>2</sub>·HF. X-ray powder diffraction was carried out using a Bruker D8 Advanced diffractometer (CuK $\alpha$ -radiation). TOPAS, DifWin, and Powder 2.0 software were used to process X-ray diffraction patterns.

Sodium fluoride is a good sintering additive, its introduction in the amount of 5 mol % NaF was enough to synthesize sintered mass with clear X-ray diffraction patterns. The experiment revealed the formation of a solid solution based on the  $\text{Ba}_4\text{Y}_3\text{F}_{17}$  compound with a trigonally distorted fluorite structure (space group  $R\bar{3}$ ) with a content of up to  $\sim 20$  mol % of NaF. The parameters of the trigonal cell were related to the parameter  $a_0$  of the fluorite subcell by the ratios  $a \sim \sqrt{7}/2a_0$  and  $c \sim 2\sqrt{3}a_0$ . The general formula for the resulting solid solution is  $\text{Ba}_{1-x}\text{Y}_x\text{Na}_x\text{F}_{2+x-y}$ . The introduction of sodium fluoride reduced the parameters of the trigonal lattice and was accompanied by the formation of anion vacancies. Structure stabilization expressed in the expansion of the homogeneity region of the phase based on  $\text{Ba}_4\text{Y}_3\text{F}_{17}$  seems to be associated with the disappearance of interstitial fluorine ions surrounded by anions in the  $\text{Ba}_4\text{Y}_3\text{F}_{17}$  structure, both in the cuboctahedral cavity of the  $\text{Y}_6\text{F}_{36}$  clusters and in the centre of the  $\text{F}_8$  cubes.

The corresponding solid solution can be used to create new photonics materials. The NaF-BaF<sub>2</sub>-YF<sub>3</sub> system is similar to the previously studied NaF-BaF<sub>2</sub>-GdF<sub>3</sub> system.

**Keywords:** Sodium fluoride, Barium fluoride, Yttrium fluoride, Solid solution

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Research article

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### Synthesis and characterization of lead and cadmium hexaborates doped with Cr<sup>3+</sup>

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#### Abstract

Borates doped with transition metals (Mn, Cu, Cr) exhibit a significant and long-lasting luminescence at room temperature, high power, and other outstanding characteristics. Therefore, the purpose of the study was to establish the possibility of the formation of borate materials containing chromium and the determination of their structure and thermal properties.

New phases of variable composition were synthesized in the  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} \cdot x\text{Cr}^{3+}$  system by heterovalent substitution of  $\text{Cd}^{2+}$  ions with  $\text{Cr}^{3+}$  ions using solid-phase reactions at 640 °C. The phases were isolated in the concentration range  $0 \leq x \leq 7.0$  mol % and characterized by X-ray phase analysis (XRD), differential scanning calorimetry (DSC) and IR spectroscopy. According to XRD and IR spectra, the resulting borates crystallize in a monoclinic cell and are assigned to one structural type (space group  $P2_1/n$ ,  $Z = 4$ ).

The crystallographic characteristics of the new phases have been determined. The crystal lattice parameters and their volumes decrease monotonically, indicating the formation of a continuous series of substitutional solid solutions in the studied concentration range. According to the DSC results, the sample  $\text{PbCd}_{2-x}\text{B}_6\text{O}_{12} \cdot 0.03 \text{Cr}^{3+}$  melts incongruently at 729 °C.

**Keywords:** Polycrystals of lead and cadmium borates, Cr<sup>3+</sup> ions, Solid-state reaction method, Solid solutions, X-ray phase analysis, DSC, IR spectroscopy

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Research article

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### The synergy of transformation of isomorphous phyllosilicate structures

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#### Abstract

The article presents an analysis of the synergy of structural transformations of phyllosilicates subjected to high temperatures and microwave radiation in terms of the destruction and formation of crystal structures. Studying the synergy of transformations of crystal structures is essential for the development of new materials and technologies, because it helps to create materials with unique properties, which cannot be obtained when using the same factors separately.

The material used in the study was a polymineral complex, which contained quartz, montmorillonite, kaolinite, chlorite, paragonite, and iron oxides (listed from the largest to the smallest mass fraction). The methods used in the study allowed us to assess the structural transformations. Averaged structural formulas of the studied phyllosilicates were calculated using the oxygen method combined with a recalculation method based on the results of a microprobe analysis. Differential thermal analysis demonstrated a synergistic effect of high temperature and microwave fields registered as a decrease in the temperature gradient of the ceramics and initiation of the sintering process at lower temperatures and with greater intensity. All the treated samples contained the amorphous phase in significant concentrations: from 15 to 25 vol. %. Half of the three-layer phyllosilicates (chlorite and montmorillonite) were destroyed by the microwave field. Kaolinite and paragonite practically did not react to external factors. The synergistic effect was the most obvious in the structural transformations of silicon and iron oxides.

Our experiments demonstrated for the first time the mechanism of formation of magnetite and hematite crystals from X-ray amorphous iron-containing films covering the particles of clay minerals. The use of the MW field and the resulting dehydration led to the formation of crystals of nuclei of iron oxides. The following high-temperature processes activated an increase in aggregated iron (magnetite and hematite). The X-ray diffraction analysis determined the presence of a synergistic effect in the evolution of structures, which could not be identified by means of IR-spectroscopy. The EPR spectroscopy allowed us to register the states of rare irregular cells with foreign paramagnetic atoms. The shift of the foreign Fe<sup>3+</sup> from the geometric centers of octahedral crystal cells towards the minima of potential energy caused by the Jahn-Teller effect decreased the potential energy of the crystal lattice. At the same time, some chemical bonds forming the crystal cell became stronger, while others weakened.

**Keywords:** Crystal cell, Transformation, Montmorillonite, Kaolinite, Chlorite, Paragonite, Microwave field, Heat treatment

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Research article

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### Structure and electrical transport properties of cation-deficient derivatives of layered neodymium–barium ferrocuprocobaltite

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#### Abstract

Layered double perovskites (LDP) based on rare earth elements, barium, and 3d-metals with high electrical conductivity and electrocatalytic activity in the oxygen reduction reaction are promising cathode materials for medium-temperature solid oxide fuel cells based on proton- or oxygen-conducting solid electrolytes. For the improvement of the functional characteristics of LDP, various strategies are used: a) creating composites based on LDP, b) the partial substitution of cations, and c) the creation of a deficiency of cations in various positions in the LDP structure. The advantage of the latter strategy is that it does not require complicating the chemical and, as a rule, phase composition of the LDP. The purpose of this study was the investigation of the effect of neodymium and barium deficiency on the structural and electrical transport characteristics of NdBaFeCo<sub>0.5</sub>Cu<sub>0.5</sub>O<sub>6-δ</sub> LDP.

The samples were obtained by the ceramic method and characterized using X-ray phase analysis, IR absorption spectroscopy, iodometry, electron microscopy, thermal analysis, as well as electrical conductivity and thermo-EMF measurements methods.

Creation of up to 10 mol. % of vacancies in neodymium or barium sublattices had little effect on the values of the oxygen nonstoichiometry index (δ) and unit cell parameters of NdBaFeCo<sub>0.5</sub>Cu<sub>0.5</sub>O<sub>6-δ</sub> derivatives. However, it led to an increase in the crystallite size (determined by the Scherrer, Williamson–Hall and size–strain methods) and the thermal stability of these phases. The values of electrical conductivity and the Seebeck coefficient of ceramics, in general, increased, and the activation energies of the electrical transfer process decreased when a deficiency of neodymium or barium was created in its structure. In the temperature range 300–700 K, the weighted mobility of charge

carriers (“holes”) varied within 0.04–0.8 cm<sup>2</sup>/(V·s) and increased with increasing temperature, which is typical for the polaron conduction mechanism, and their concentration varied in the range (0.1–3)·10<sup>20</sup> cm<sup>-3</sup>, increased exponentially with increasing temperature and, in general, when a deficiency of neodymium or barium in the NdBaFeCo<sub>0.5</sub>Cu<sub>0.5</sub>O<sub>6-δ</sub> structure was created.

**Keywords:** Layered double perovskites, Cation deficiency, Structure, Thermal stability, Electrical conductivity, Thermo-EMF, Weighted mobility, Concentration of charge carriers

**Acknowledgments:** studies using powder X-ray diffraction, IR absorption spectroscopy, electron microscopy and thermal analysis were carried out using the equipment of the Centre for Physico-Chemical Research Methods of the Belarusian State Technological University.

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Research article

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### Synthesis and sensory properties of tungsten (VI) oxide-based nanomaterials

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#### Abstract

The purpose of this work was to develop a methodology for the synthesis of WO<sub>3</sub>-based nano-scale materials, to provide their characterization, and to study their sensory properties.

The nanopowder was made by slowly adding nitric acid to an aqueous solution of ammonium paratungstate, (NH<sub>4</sub>)<sub>10</sub>W<sub>21</sub>O<sub>41</sub>·xH<sub>2</sub>O, followed by centrifugation, drying, and calcination. The size of tungsten trioxide grains, which was 10–20 nm, was determined by transmission electron microscopy. According to X-ray phase analysis, the powder, which was calcined at a temperature of 500 °C, mainly consisted of a triclinic phase. Subsequently, diammine palladium (II) nitrate and terpeniol were added to the WO<sub>3</sub> nanopowder to form a paste. The resulting paste was applied to a special dielectric substrate and calcined to a temperature of 750 °C. As a result, a fragile tungsten trioxide-based gel formed. The mass fraction of palladium in the fragile gel was 3%. The sensory properties of the obtained gas-sensitive material were studied under stationary (300 °C) and non-stationary temperature conditions (quick heating to 450 °C and slow cooling to 100 °C). A sharp increase in the sensitivity of a tungsten trioxide-based sensor was observed under non-stationary temperature conditions which depended on the composition of the gas-sensitive layer.

**Keywords:** MOX sensor; Sensitivity; Temperature modulation

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### Short communication

Short communication

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### Influence of the particle size of sulfonated cation exchange resin on the physicochemical properties and surface morphology of MK-40 heterogeneous membranes

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#### Abstract

The article presents the results of the study of the physicochemical properties and surface morphology of MK-40 heterogeneous membranes with a given particle size of sulfonated cation exchange resin within the ranges of < 20 μm, 32–40 μm and 56–71 μm with the resin-polyethylene ratio of 65:35. The experimental membranes were manufactured at LLC Innovative Enterprise *Shchekinoazot* (Russia). A comparative analysis of the characteristics of the membranes showed that their main physicochemical properties change in accordance with the changes in surface parameters. With an increase in the particle size of the ion exchange resin, the moisture content and the thickness of the membranes decreased together with the surface macroporosity. What is more, the exchange capacity of the membranes did not depend on the particle size of the ion exchange resin.

Quantitative analysis of SEM images of swollen membranes revealed a number of peculiarities of their surface morphology. First, it was established that membranes with a size of resin particles within the range of 56–71 μm had small ion-exchangers with a radius of  $R < 10$  μm and no large ( $R > 20$  μm) ion-exchangers. Secondly, it was found that the fraction of the ion exchanger and the weighted average radius of the ion exchange resin particles decreased by 14% and 15% respectively with an increase in the preset dimensions of the ion exchanger. Thirdly, with an increase in the size of the resin particles, the surface macroporosity decreased by 7%, while the size of the macropores increased by more than 20%. To explain the revealed peculiarities of the surface morphology of MK-40 heterogeneous membranes with different particle sizes of sulfonated cation exchange resin, it is necessary to study the structure of their internal phase.

**Keywords:** Heterogeneous cation exchange membrane, Particle size of ion exchange resin, Surface morphology, Physicochemical properties

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Microphotographs of membrane surfaces were obtained using the equipment of the Centre for Collective Use of Scientific Equipment of Voronezh State University. URL: <https://ckp.vsu.ru>

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Short communication

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**$1/f^2$  noise as a precursor of structural reconstructions near the melting point of crystalline materials with different types of chemical bonds**

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**Abstract**

Transition phenomena near the melting point (premelting effect) are fundamental processes occurring in materials with different types of chemical bonding. At  $T \geq 0.8T_m$ , there is a fluctuating heat release. The analysis of fluctuation processes allows obtaining information about dynamic reconstructions in various subsystems and interconnections between them within the system. The purpose of this work was to study the spectral characteristics of heat fluctuations in stationary modes of premelting materials with ionic, covalent, and metallic chemical bonds (KCl, Ge, Cu, and Sb) and to determine the type of the fluctuation process using the Hurst parameter.

The spectral characteristics of heat fluctuations in stationary modes of pre-melting KCl, Ge, Cu, and Sb at  $T \sim 0.9T_m$  were determined by wavelet analysis. This method allows analyzing the behavior of complex systems at critical points in order to identify certain correlations and development trends in them.

The study showed that in the premelting region, the frequency spectrum of heat fluctuations was characteristic of  $1/f^2$  noise or nonlinear Brownian noise, which is a precursor of structural reconstructions during phase transitions. The type of fluctuation processes in the premelting region of KCl, Ge, Cu, and Sb was determined using the Hurst parameter ( $H$ ). It was shown that in stationary modes of premelting  $H > 0.5$ . Consequently, the previous trend of the process dynamics was very likely to develop in the same direction. However, with a decrease in the energy of the chemical bond  $H \rightarrow 0.5$ , which indicated a decrease in the stability of the system and a likely change in the development trend for the structural reconstructions in the premelting transition region.

Thus, near the melting point, there are unstable dynamic states, which are precursors to structural changes in the system, which have certain developmental trends. This should be taken into account when calculating the stability and reliability of materials and systems.

**Keywords:** Premelting, Fluctuations, Wavelet analysis,  $1/f^2$  noise, Hurst parameter, Structural reconstructions

**For citation:** Mashkina E. S.  $1/f^2$  noise as a precursor of structural reconstructions near the melting point of crystalline materials with different types of chemical bonds. *Condensed Matter and Interphases*. 2024;26(2): 362–366. <https://doi.org/https://doi.org/10.17308/kcmf.2024.26/12071>