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Hierarchical self-assembly of SiO_2-SnO_2 nano- and microstructures in combined sol-gel systems

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Abstract. Using the IR spectroscopy method, studies were carried out on the processes of hierarchical self-assembly of SiO_2-SnO_2 nano- and microstructures in combined sol-gel systems obtained by mixing film-forming sols with different maturation times, which meets the goals and objectives of nanostructural engineering. Characteristic absorption peaks were identified that correspond to the process of hydrolytic polycondensation and carry information about the process of self-assembly in the analyzed systems.

Keywords: nanostructure engineering, hierarchical self-assembly, sol-gel technology, spectroscopic investigation

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Иерархическая самосборка нано- и микроструктур SiO_2-SnO_2 в комбинированных золь-гель системах

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Аннотация. С использованием метода ИК-спектроскопии проведены исследования процессов иерархической самосборки нано- и микроструктур SiO_2-SnO_2 в комбинированных золь-гель системах, полученных путем смешения пленкообразующих золей с различным временем созревания, что отвечает целям и задачам наноструктурной инженерии. Установлены характеристические пики поглощения, соответствующие процессу гидролитической поликонденсации и несущие информацию о процессе самосборки в анализируемых системах.

Ключевые слова: наноструктурная инженерия, иерархическая самосборка, золь-гель технология, спектроскопические исследования

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Introduction

In recent years, wide-bandgap semiconductor metal oxides such as ZnO, SnO_2, TiO_2, In_2O_3, etc. have attracted increasing interest. First of all, this is due to the wide scope of their practical application, including, among other things, solving gas sensory problems [1]. At the same time, it is becoming increasingly important to develop new methods and approaches to nanostructure engineering that make it possible to control the properties of these materials both through macro-level influences and by controlling the process of their synthesis at the earliest stages. For example, it is known that varying the maturation time of a film-forming sol leads to a new type of micro- and nanostructure of the metal oxide material [2].

Materials and Methods

Combined sol-gel SiO_2-SnO_2 systems were prepared within the framework of nanostructure engineering methods and approaches, which were first proposed to control the process of hierarchical self-assembly of zinc oxide nano- and microstructures [3]. The main idea is to mix film-forming sols with different maturation times in a given volume ratio (1:1, 2:1, 1:2). As part of this study, an analysis of a two-component SiO_2-SnO_2 system with a mass fraction of tin dioxide of 80 wt% was carried out. Thin films based on combined sol-gel systems were synthesized by applying film-forming sols to substrates made of oxidized monocrystalline silicon KEF (100) measuring 10×10 mm^2 by centrifugation (at a speed of 4000 rpm), followed by annealing in air atmosphere (for 30 min at temperature 550 °C).

The qualitative composition of film-forming sols, as well as the processes of hierarchical self-assembly, were studied by IR spectroscopy on an IR-Fourier spectrometer FSM 1201 (Infraspek LLC, Russia) within the method of multiple attenuation of total internal reflection using a MNPVO36 ZnSe cell. The surface morphology of the samples obtained on the basis of combined sol-gel systems was studied using the MIRA3 (Tescan) scanning electron microscope.

Results and Discussion

Fig. 1 shows the IR spectra of combined sol-gel systems SiO_2-SnO_2 with a maturation time of 1 hour (sample no. *1*) and 24 hours (sample no. *5*), respectively, as well as IR spectra of sols obtained by mixing samples no. *1* and no. *5* in a volume ratio of 2:1 (sample no. *2*), 1:1 (sample no. *3*) and 1:2 (sample no. *4*), respectively. Analysis of the presented IR spectra shows that mixing film-forming sols with different maturation times does not lead to the emergence of new characteristic vibrational modes. This allows concluding that new chemical bonds are not formed, and there is no noticeable change in the qualitative composition of combined sol-gel systems when mixing sols with different maturation times.

To analyze self-assembly processes occurring in the considered combined sol-gel systems, the most informative is the absorption peak with a maximum of 1010 cm^{-1} (inset in Fig. 1), which corresponds to the results of previous studies [4]. This vibrational mode corresponds to symmetrical stretching vibrations of Si-O-Si and a decrease in transmission with increasing maturation time of film-forming sols corresponds to the process of hydrolytic polycondensation, the consequence of which is the hierarchical self-assembly of nano- and microstructures of SiO_2-SnO_2, which have a fractal nature [5].

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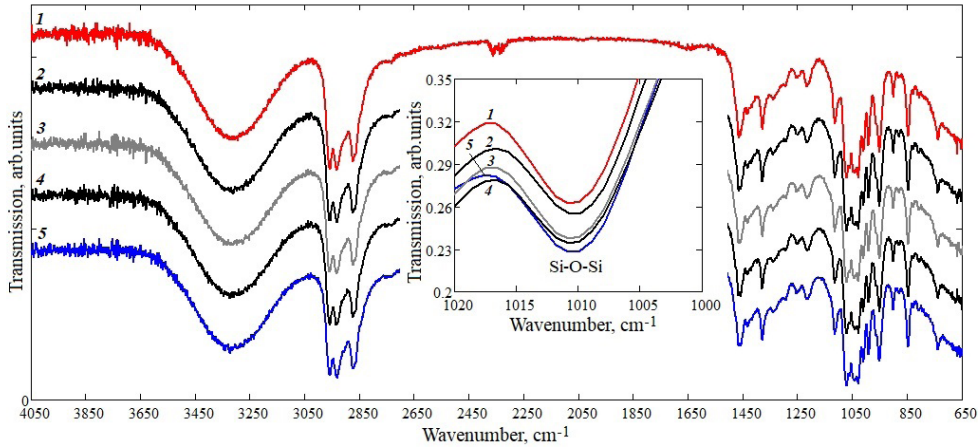

Fig. 1. IR transmission spectra of combined sol-gel systems with different maturation times: *1* corresponds to 1 hour, *2* to 1 hour + 24 hours in a volume ratio of 2:1, *3* to 1 hour + 24 hours in a volume ratio of 1:1, *4* to 1 hour + 24 hours in a volumetric ratio of 1:2, *5* to 24 hours

Fig. 2, *a* shows a diagram illustrating the relationship between the IR transmission rate of combined sol-gel systems and the maturation time, as well as the volume ratio of the mixed sols.

The analysis of the diagram indicates that for combined sol-gel systems under study, an increase in the maturation time leads to an apparent decrease in the transmission rate from 0.263 to 0.229 for 1 hour and 24 hours, respectively. Taking into account that the studied vibrational mode corresponds to the process of hydrolytic polycondensation, this can be attributed to the enlargement of fractal clusters during self-assembly [6]. Mixing film-forming sols in a given volume ratio results in the fact that in the illustrative diagram the minimum transmission rate of the analyzed vibrational mode is located closer to the film-forming sol, the ratio of which is larger. This leads to an assumption that mixing sols with different maturation times creates a new type of size distribution of fractal clusters and, ultimately, to a new type of structure of thin films synthesized from combined sol-gel systems. In particular, mixing film-forming sols with maturation times of 1 hour and 24 hours in the volumetric ratio of 2:1 results in forming SiO_2-SnO_2 nano- and microstructures (Fig. 2, *b*) with hierarchical spatial organization [7].

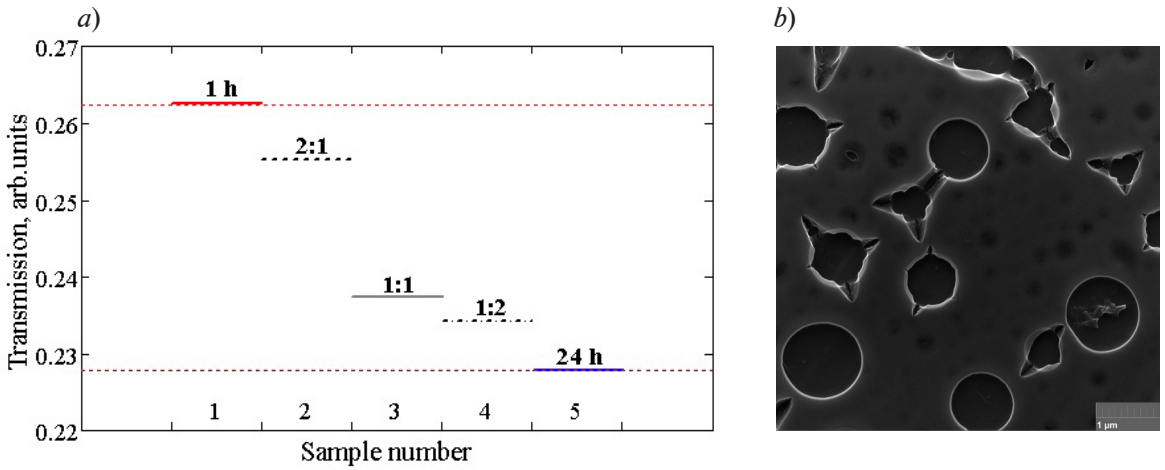

Fig. 2. Illustrative diagram (*a*) of IR transmission rate through combined sol-gel systems and SEM image (*b*) of thin film synthesized from combined sol-gel systems obtained by mixing sols with maturation time of 1 hour and 24 hours in volume ratio 2:1

Conclusion

Thus, this study shows that infrared spectroscopy is a promising method for studying the hierarchical self-assembly of SiO_2-SnO_2 nano- and microstructures in combined sol-gel systems. It has been established that the mixing of sols with different maturation times affects the intensity of the characteristic peaks and absorption bands corresponding to the process of hydrolytic polycondensation. The obtained spectroscopic patterns were verified by experimental data from scanning electron microscopy of thin films synthesized from combined sol-gel systems.

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