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DEVELOPMENT OF SiO_2:GeO_2(Ge°) CERAMIC NANOCOMPOSITES VIA SOL-GEL SYNTHESIS FOR THIN-FILM APPLICATIONS IN SOLAR PANEL MANUFACTURING

تطوير مركبات SiO_2:GeO_2 (Ge°) النانوية الخزفية بإستخدام تقنية المحلول الغرواني (سول -جيل) لتوليف الأغشية الرقيقة التي يتم تطبيقها في إنتاج الألواح الشمسية

Marwan F. S. H. AL-Kamali[1*], Andrei A. Boika[2], Yahya T.A. AL-Ademi[3], Yauheni N. Paddenezhny[4], Vladimir A. Levchenko[5]

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ABSTRACT

In this work, we studied the interaction of dopant substances with the surface of the SiO_2 framework of xerogel. For this purpose, samples of SiO_2:GeO_2 and SiO_2:Ge^o compositions synthesized at a temperature of 800 °C were prepared. The studies were carried out using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The results showed a clearly defined globular structure and high homogeneity of the morphology of the samples. The introduction of germanium contributed to the formation of a coating on the surface of SiO_2 globules and an increase in the size of aggregates, which is associated with the agglomeration of germanium ions. Morphology analysis revealed a porous structure, especially pronounced in SiO_2:GeO_2 samples, which can improve the mechanical properties of the materials. Chemical composition and phase analysis confirmed a change in the ratios of elements, indicating the effect of germanium on the physicochemical properties of the xerogel. The obtained results open up new prospects for the application of such nanocomposites in thin-film technologies and composite materials.

Keywords: Nanocomposites, SiO_2-xerogel, germanium, germanium oxide, SEM, X-ray diffraction, surface morphology, dopant substances.

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ملخص البحث:

في هذا العمل، قمنا بدراسة تفاعل المنشطات مع سطح إطار الهلامة الجافة (الأكسروجيل) لأكسيد السليكون .(SiO_2) تم تحضير عينات من التراكيب التالية SiO_2:GeO_2 و SiO_2:Ge^o، حيث تم توليفها وتلدينها عند درجة حرارة 800 درجة مئوية. أُجريت دراسات التركيب السطحي للعينات (المورفولوجيا) باستخدام المجهر الإلكتروني الماسح(SEM) ، وتم قياس التكوين المرحلي باستخدام جهاز حيود الأشعة السينية.(XRD)

أظهرت النتائج بنية كروية محددة بوضوح وتجانسًا عاليًا للعينات، حيث ساهم إدخال الجرمانيوم في تكوين طبقة على سطح كريات أكسيد السليكون .(SiO_2) كما لوحظ زيادة في حجم الركام المرتبط بتكتل أيونات الجرمانيوم. كشف تحليل الشكل عن بنية مسامية واضحة بشكل خاص في عيناتSiO_2:GeO_2 ، مما يمكن أن يحسن الخواص الميكانيكية للمواد. أكد التركيب الكيميائي وتحليل الطور حدوث تغير في نسب العناصر، مما يشير إلى تأثير الجرمانيوم على الخواص الفيزيائية والكيميائية للهلامة الجافة (الأكسروجيل).

تفتح النتائج التي تم الحصول عليها آفاقًا جديدة لاستخدام هذه المركبات النانوية في تقنيات الأغشية الرقيقة والمواد المركبة.

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الكلمات المفتاحية: المركبات النانوية، الهلامة الجافة (الأكسروجيل) لأكسيد السليكون (SiO_2)، الجرمانيوم، أكسيد الجرمانيوم، المجهر الإلكتروني الماسح(SEM) ، جهاز حيود الأشعة السينية(XRD) ، التشكل السطحي، المنشطات.

I. Introduction

In recent years, there has been a significant increase in interest in the development of new materials for thin-film technologies, especially in the context of solar panel production. One promising area is the use of ceramic nanocomposites based on silicon oxide (SiO_2) and germanium oxide (GeO_2), which have unique optical and electrical properties necessary to improve the efficiency of solar cells [1,2]. Sol-gel synthesis is one of the most effective methods for producing such nanocomposites, allowing control over the morphology and structure of materials at the nanoscale. This method ensures a uniform distribution of components in the matrix, which helps improve the mechanical and thermal properties of the resulting materials [3]. SiO_2:GeO_2(Ge°) ceramic nanocomposites can be used as active layers in solar panels, as they are capable of increasing light absorption and, consequently, the efficiency of converting solar energy into electrical energy [4]. In addition, such materials are highly resistant to external influences, making them ideal for use in conditions typical of solar power plants.

Thus, the development of SiO_2:GeO_2(Ge°) ceramic nanocomposites using sol-gel synthesis is an urgent task that can significantly improve solar panel production technologies and, ultimately, increase their commercial attractiveness.

The purpose of the conducted scientific research was to study the technological stages of the synthesis of porous SiO_2 matrices doped with germanium ions, formed by the sol-gel method based on an aqueous dispersion of Aerosil A-380. The goal was to establish the effect of germanium concentration on the structural and functional characteristics of the resulting matrices intended to form targets for vacuum methods of thin film deposition.

II. Materials and Methods

i. Materials

All chemicals listed below were purchased and utilized in the form of silica (technically known as aerosil) under the labels A-380 Nano-powders (SiO_2, 99.9%, with an average size of primary particles ranging from 5–15 nm, a specific surface area of 50–380 m^2/g, an adsorption capacity ranging from 100 to 340 g at 100 g of

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silica, and a bulk density of 35–150 g/l), germanium nitrate (GeN_4O_{12}, 99.9%), and polyvinyl alcohol $(-C_2H_4O)_n$. These chemicals were procured from Vekton Company - Russia. Distilled water was used in preparing the sol of SiO_2. All chemicals were used without additional purification.

ii. Synthesis Methodology and Preparation Technique

In the course of the study, highly dispersed silica of the A-380 marka, synthesized by hydrolysis of silicon tetrachloride in an oxygen-hydrogen flame at a temperature above 1000°C, was used to obtain porous SiO_2 matrices. Aerosil powder contains impurities of water, hydrogen chloride, various oxides, and solid particles of silicon dioxide (grit) [5]. Silanol groups are present on the surface of silicon dioxide particles, and ≡Si–O–Si≡ bonds are localized inside the particles. Germanium ions were introduced into the composition in the form of a water-soluble salt—germanium nitrate. The technological process began with the formation of a sol, then a gel, and ultimately, a xerogel. Gelling of the sol (both pure and containing germanium) occurred in open plastic molds in air, after which the gels were dried in a ventilated oven at a temperature of 80°C. To achieve a uniform distribution of dopants throughout the volume of the final products, the xerogel blanks were ground to micron and submicron powders. This made it possible to obtain composite blanks of various geometric profiles using the uniaxial pressing method. In the process of synthesizing germanium compounds of a given stoichiometric composition, the xerogels were subjected to step-by-step heat treatment in air to form a SiO_2:GeO_2 composite material. Then, the synthesis was carried out in a dry hydrogen environment, which made it possible to obtain materials of the SiO_2:Ge^o composition. The final phase transformations were carried out either in air or in a hydrogen environment at a temperature of 800°C for 1 hour. The resulting materials were ground to a state of highly dispersed micro-powders, from which targets were formed in the form of tablets of the desired size using the uniaxial pressing method. An aqueous solution of polyvinyl alcohol with a concentration of 3–6 wt.% was used as a temporary binder, depending on the type of the final product and the pressing mode. The pressure in the hydraulic system of the press during the production of target samples was about 120–125 kg/cm². Thus, binary metal oxide systems of the composition SiO_2:GeO_2 and SiO_2:Ge^o with the atomic ratio of germanium Si:Ge^o = 1:0.05; 1:0.1; 1:0.15; 1:0.5, intended for use in magnetron sputtering in a vacuum [6–8], were successfully formed. The basic scheme for producing finely dispersed micro-powders of the composition SiO_2:GeO_2 and SiO_2:Ge^o is shown in Fig. 1.

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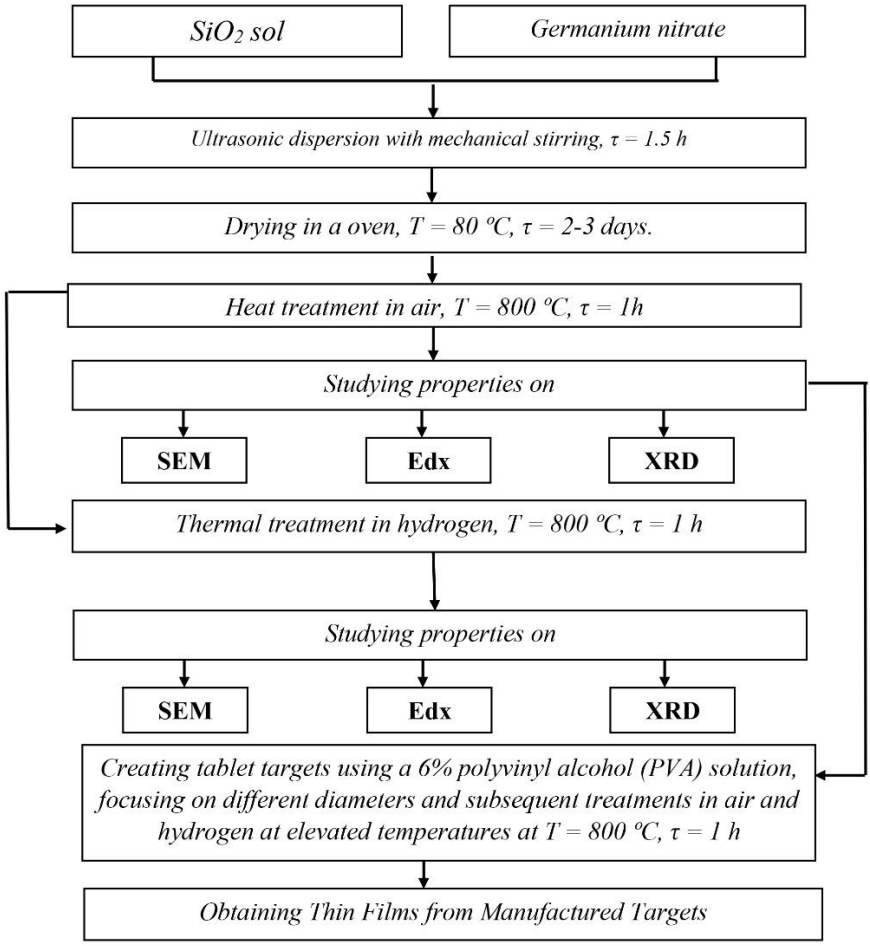

Figure 1 – Scheme of technological stages for obtaining Ge-doped xerogel SiO_2 matrices

iii. Characterization

The surface morphology of the synthesized samples was studied on the central part of the broken SiO_2 xerogel using a scanning electron microscope model S-4800 (Hitachi, Japan) with a resolution of 1 nm. The concentrations of various elements were also determined by the EDX (EDS) method using a Quantex 200 energy-dispersive microanalyzer (without a nitrogen microanalyzer and with an XFlash Detector 5030) with a resolution of 125 eV (Bruker, Germany). The studies were carried out by employees of the STC "Belmicroanalysis" of the branch of "Belmicrosystems" of JSC "INTEGRAL," the management company of the holding "INTEGRAL." To analyze the phase composition of the surface of the synthesized

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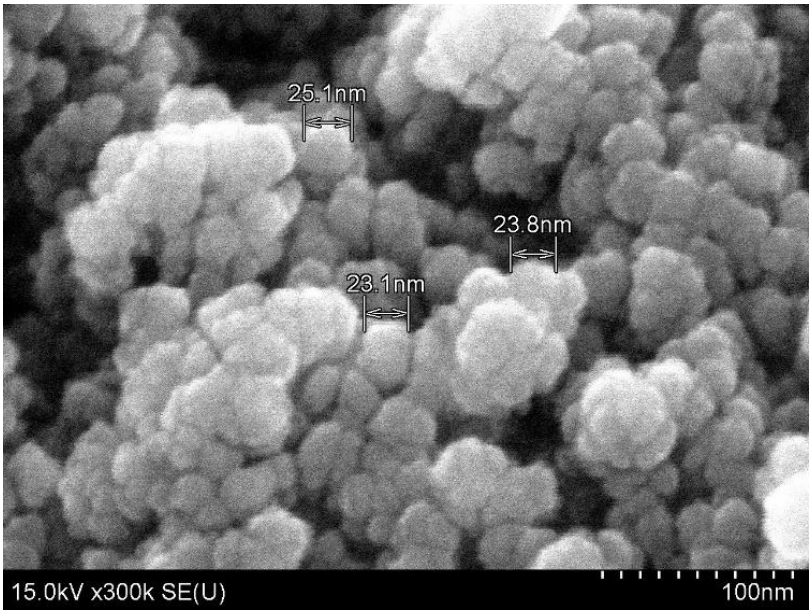
samples, the central part of the SiO_2 xerogels was also studied. Nanostructured micro-powders were analyzed using the X-ray diffraction method on a multifunctional diffractometer GNR APD 2000 PRO; the studies were carried out by employees of the BelCZM Institute of Mechanics of Metal-Polymer Systems named after V. A. Bely.

III. Results & Discussion

In order to study the interaction of dopant substances with the surface of the globules of the SiO_2 framework of the xerogel, two types of samples were prepared for studying their morphology using scanning electron microscopy (SEM): SiO_2:GeO_2 and SiO_2:Ge^o (see Figs. 2 and 3). A clearly defined globular structure of the xerogels and high homogeneity of their initial morphology were observed. Large globular aggregates that form the framework of the xerogel consist of primary aerosil particles, the sizes of which are about 5–15 nm, according to the specifications for the A-380 marka.



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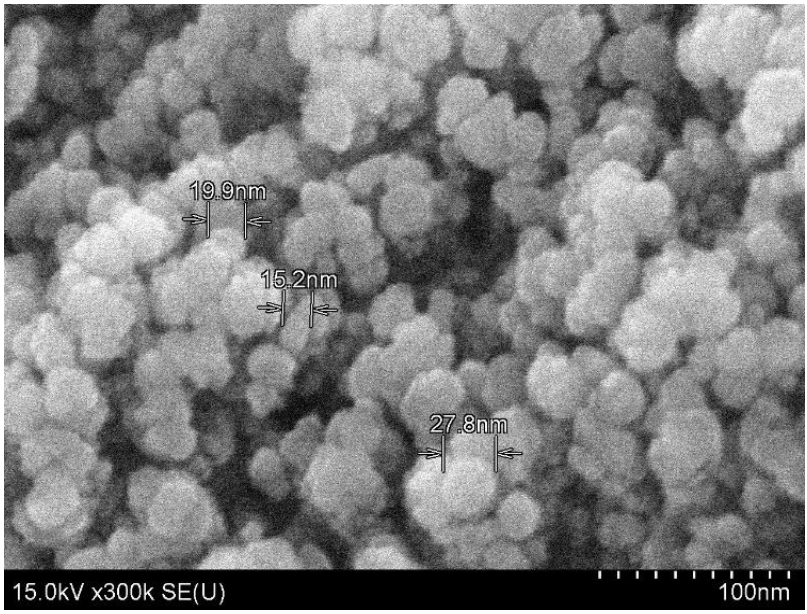

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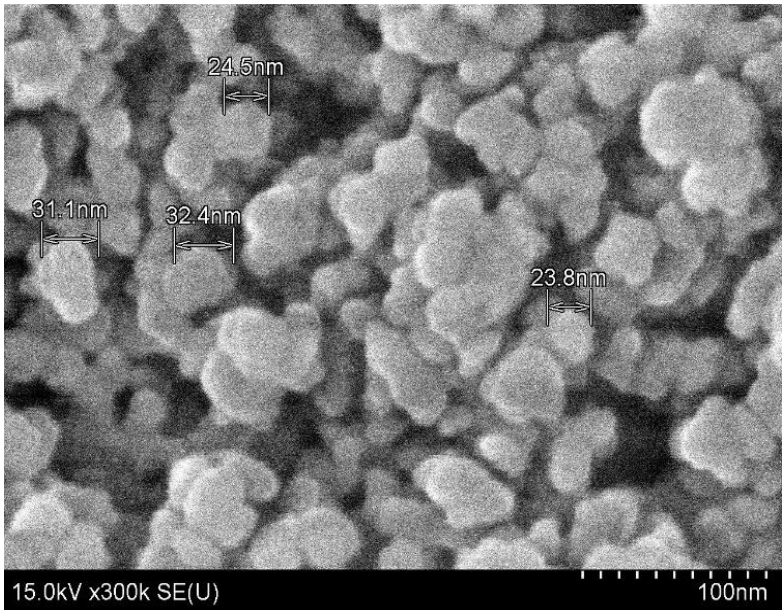

c)

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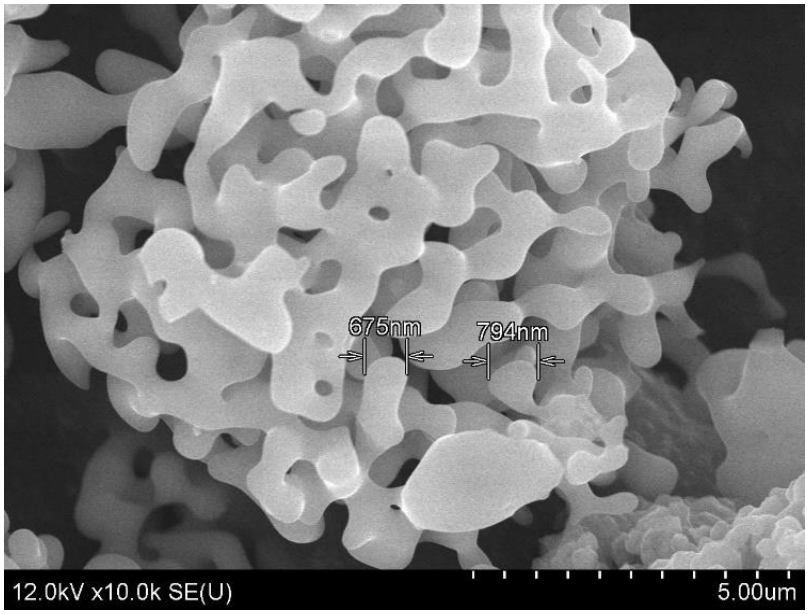
Figure 2 – SEM image of a xerogel micro-powder formed on the basis of a sol of an aqueous dispersion of SiO_2 of the A-380 marka. The xerogel was treated in air at a temperature of 800 °C for 1 hour. (a - Contains germanium nitrate with a concentration of 0.20 mol per 1 mol of sol; b - Contains germanium nitrate with a concentration of 0.30 mol per 1 mol of sol; c - Contains germanium nitrate with a concentration of 0.40 mol per 1 mol of sol.)



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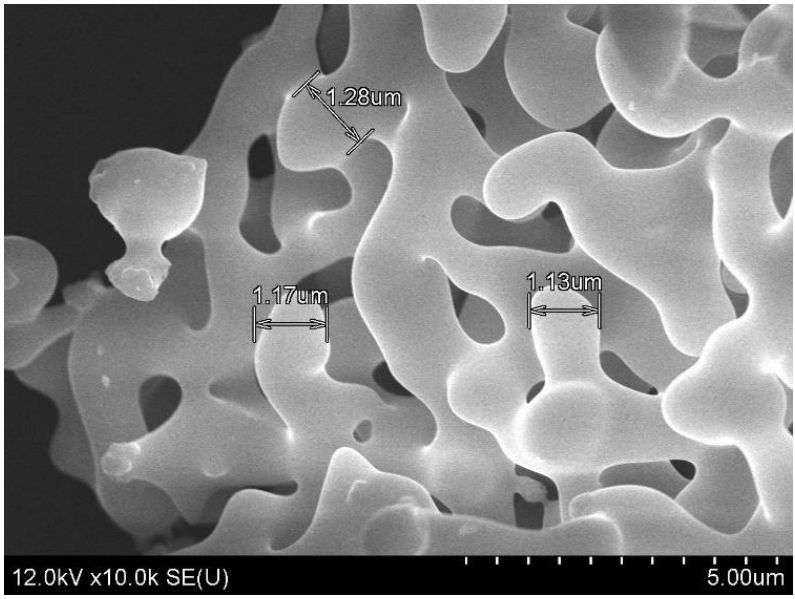

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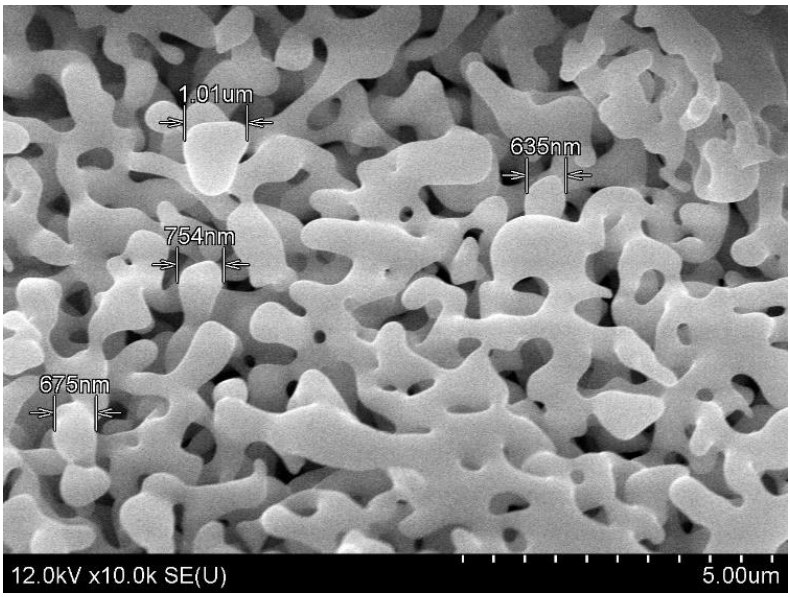

c)

Figure 3 – SEM image of the xerogel micro-powder formed on the basis of a sol of an aqueous dispersion of SiO_2 of the A-380 marka. The xerogel was treated in hydrogen at a temperature of 800 ºC for 1 hour. (a - Contains germanium nitrate with a concentration of 0.20 mol per 1 mol of sol; b - Contains germanium nitrate

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with a concentration of 0.30 mol per 1 mol of sol; c - Contains germanium nitrate with a concentration of 0.40 mol per 1 mol of sol.)

Due to the high degree of hydration, germanium nitrate fills the interglobular and interpore spaces of the xerogel. The formation of matrices based on such xerogels with the composition SiO_2:GeO_2 restores the "relief" of the xerogel structure, indicating the mutual chemical inertness of the SiO_2 and GeO_2 phases (see Fig. 2). The transformation of germanium oxide (Ge) into a reduced metallic state leads to the alignment of the overall structure of the xerogel. Larger aggregates are observed in the samples, with sizes varying from 635 to 1200 nm, likely due to the agglomeration of germanium ions during heat treatment (see Fig. 3).

It was found that germanium ions modify the internal structure of the xerogel, creating a unique coating on the surface of the SiO_2 globules. The observed effect is likely due to the high concentration of germanium nitrate added to the initial SiO_2 sol (with ratios of Si:Ge atoms = 1:0.05, 1:0.10, …, 1:1, etc.), as well as the significant sorption capacity of the xerogel itself [9–10].

Analysis of the obtained xerogels of the SiO_2:GeO_2 and SiO_2:Ge° compositions, synthesized at a temperature of 800 °C, using SEM, showed that no separate formation of micro- and nanoparticles was detected when studying the surface morphology by the SEM method. Most likely, germanium oxide and reduced germanium form a two-dimensional (2D) coating covering the entire inner surface of the xerogel (see Fig. 4). Such a mechanism is proposed in [9–10].

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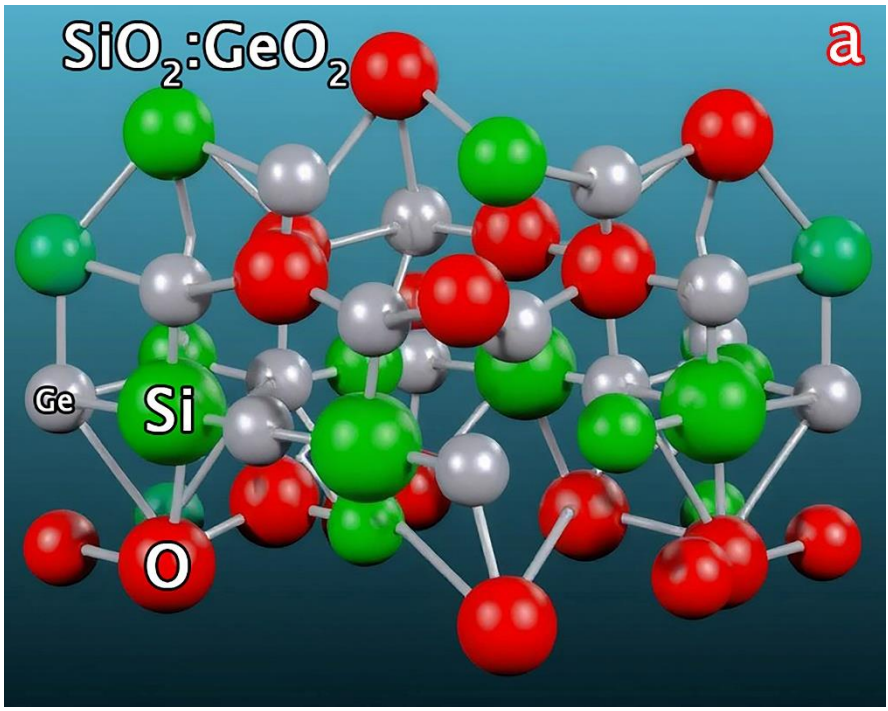

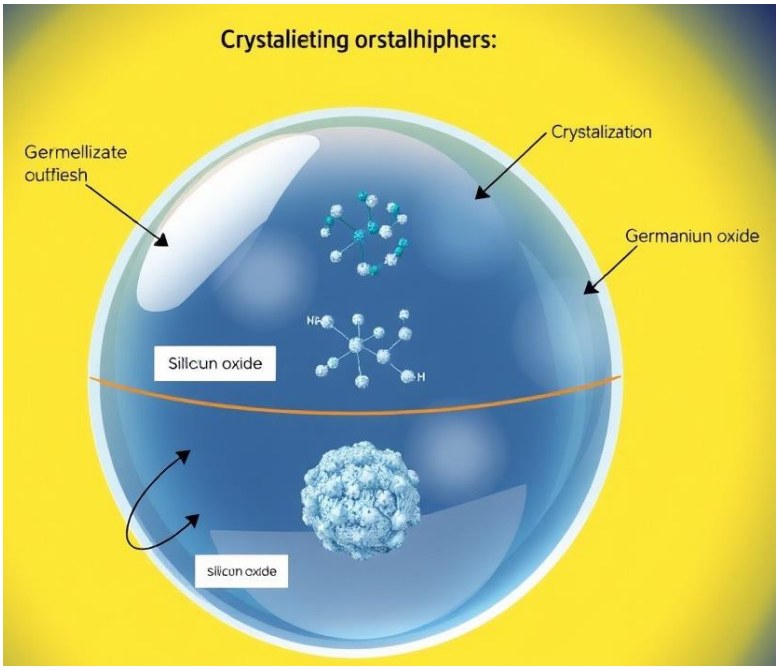

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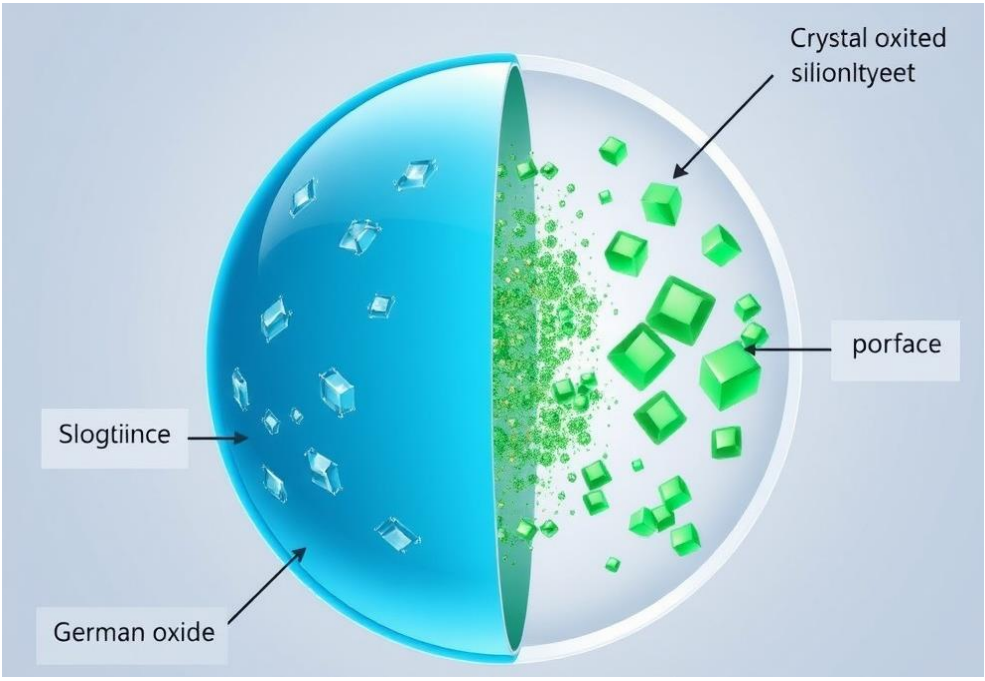

Figure 4. Colored schematic illustration of a possible mechanism promoting the formation of Ge particles in the Si–Ge–O system.

SiO_2:GeO_2 nanocomposites exhibit a uniform distribution of particles ranging in size from 15 to 32 nm, indicating a high level of dispersion and a spherical particle shape (see Fig. 2). In SiO_2:Ge^o samples, larger aggregates are observed, with sizes varying from 635 to 1200 nm, likely due to the agglomeration of germanium ions during heat treatment in a hydrogen atmosphere and the reduction of germanium oxide to Ge^o (see Fig. 3).

SEM images also show the presence of a porous structure in both types of nanocomposites, which is an important aspect for their application in thin-film technologies. The pores range in size from 10 to 30 nm, contributing to improved adhesion and mechanical properties of the materials [11]. The SiO_2:GeO_2 samples exhibited more pronounced porosity compared to SiO_2:Ge^o, which may be attributed to the optimal concentration of germanium facilitating the more efficient formation of pores in the matrix. SEM observations also confirmed that phase transformations occurred during heat treatment at 800 °C, which manifested as changes in particle morphology and the formation of new structures characteristic of composite materials [12].

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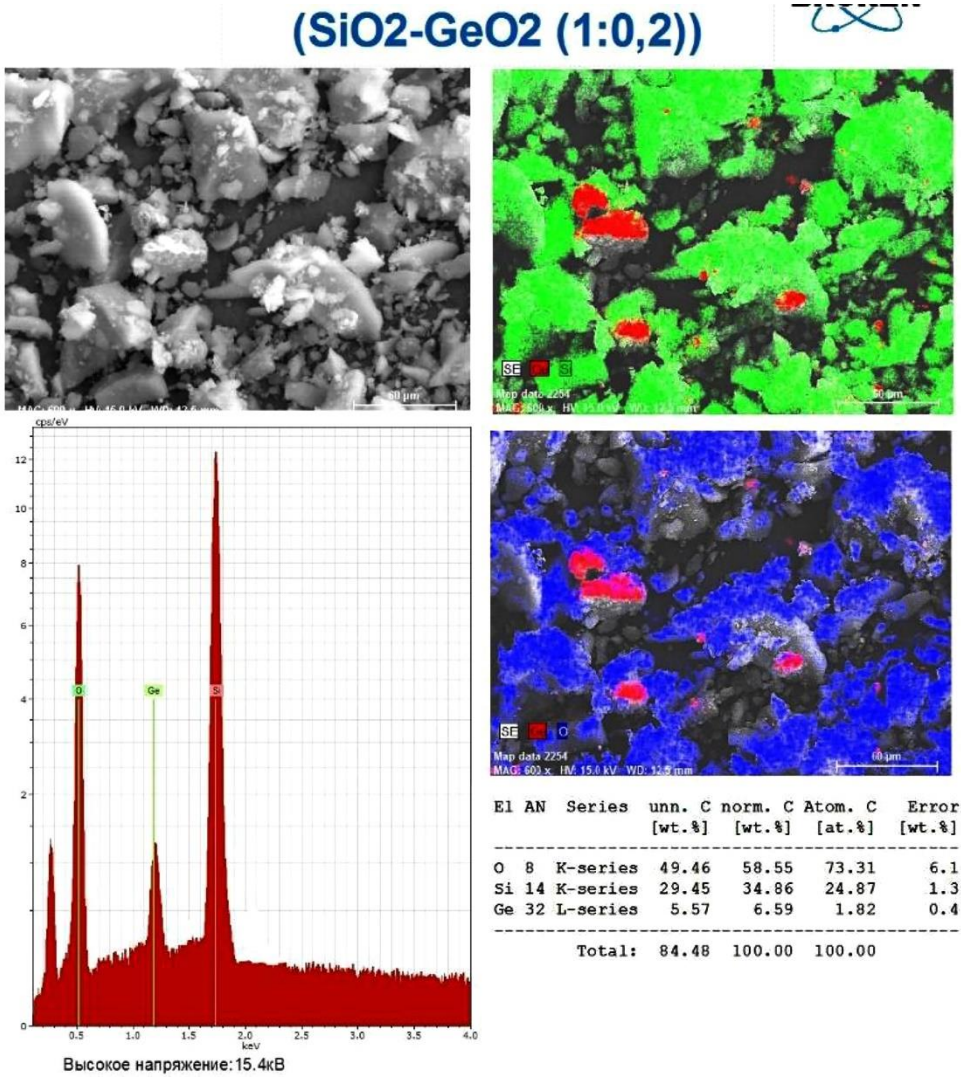
During the study of SiO_2:GeO_2 nanocomposites synthesized at 800 °C, the elemental composition was analyzed using energy-dispersive X-ray spectroscopy (EDX). The results show the following elemental ratios (see Fig. 5):

Si:Ge ratio ≈ 1:0.2: (Silicon content (Si): 24.87 at.%, Germanium content (Ge): 1.82 at.%, Oxygen content (O): 73.31 at.%).

Si:Ge ratio ≈ 1:0.3: (Silicon content (Si): 31.03 at.%, Germanium content (Ge): 2.41 at.%, Oxygen content (O): 66.56 at.%).

Si:Ge ratio ≈ 1:0.4: (Silicon content (Si): 32.09 at.%, Germanium content (Ge): 2.98 at.%, Oxygen content (O): 64.93 at.%).



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
O	8	K-series	49.46	58.55	73.31	6.1
Si	14	K-series	29.45	34.86	24.87	1.3
Ge	32	L-series	5.57	6.59	1.82	0.4
		Total:	84.48	100.00	100.00	

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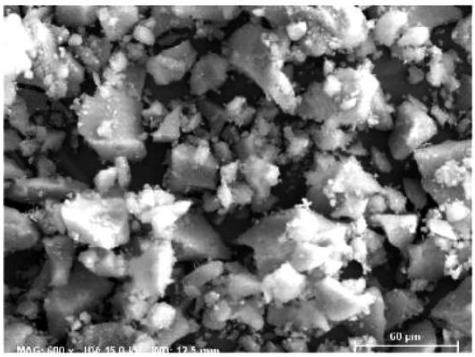
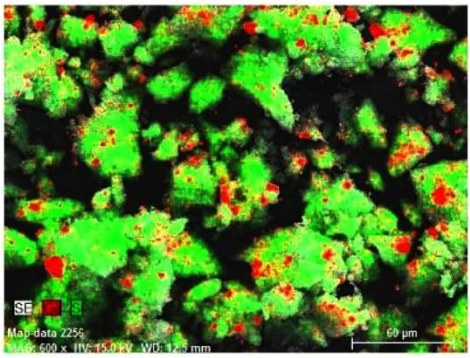
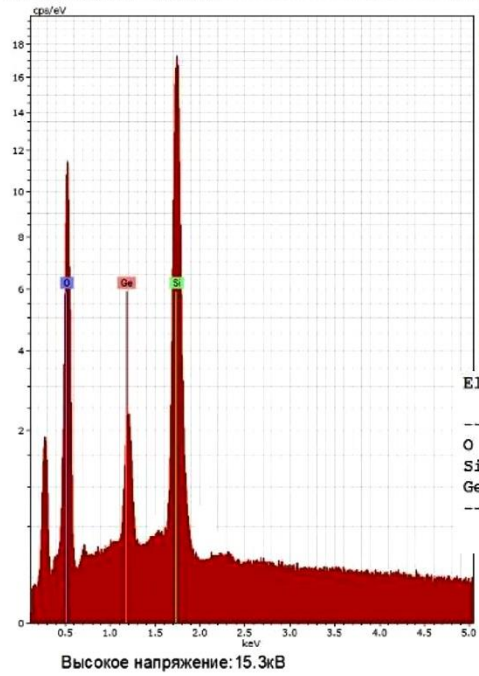
(SiO2-GeO2 (1:0,3))







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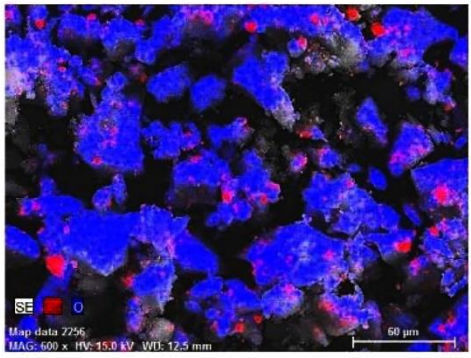

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [wt.%]
O	8	K-series	31.21	50.43	66.56	3.6
Si	14	K-series	25.54	41.27	31.03	1.1
Ge	32	L-series	5.13	8.29	2.41	0.3
		Total:	61.88	100.00	100.00	

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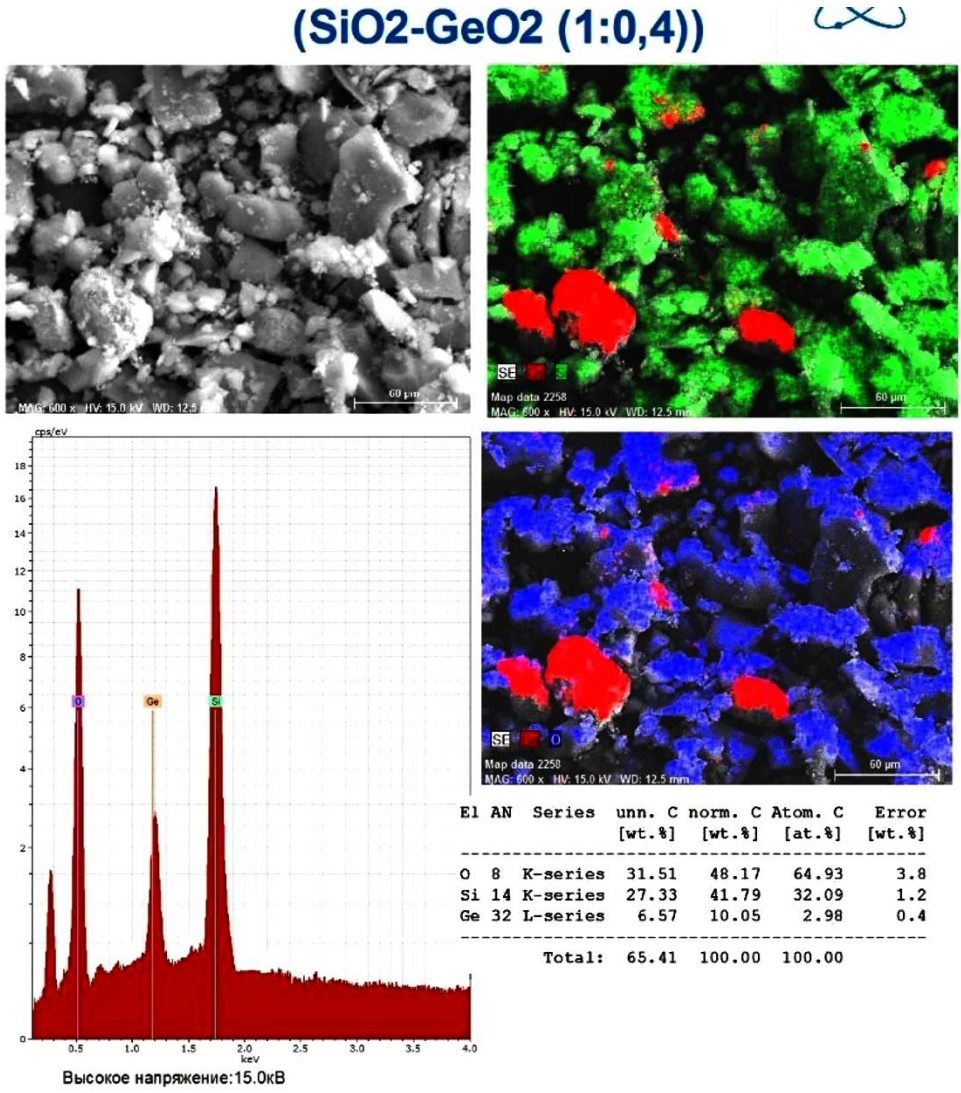

Figure 5. EDX analysis of SiO_2:GeO_2 nanocomposites after heat treatment in air at 800 °C.

Thus, the influence of germanium ions on the structural changes of the composite, including its physicochemical properties, is confirmed. These changes can be used for further research in the field of thin-film technologies and composite materials [11–12].

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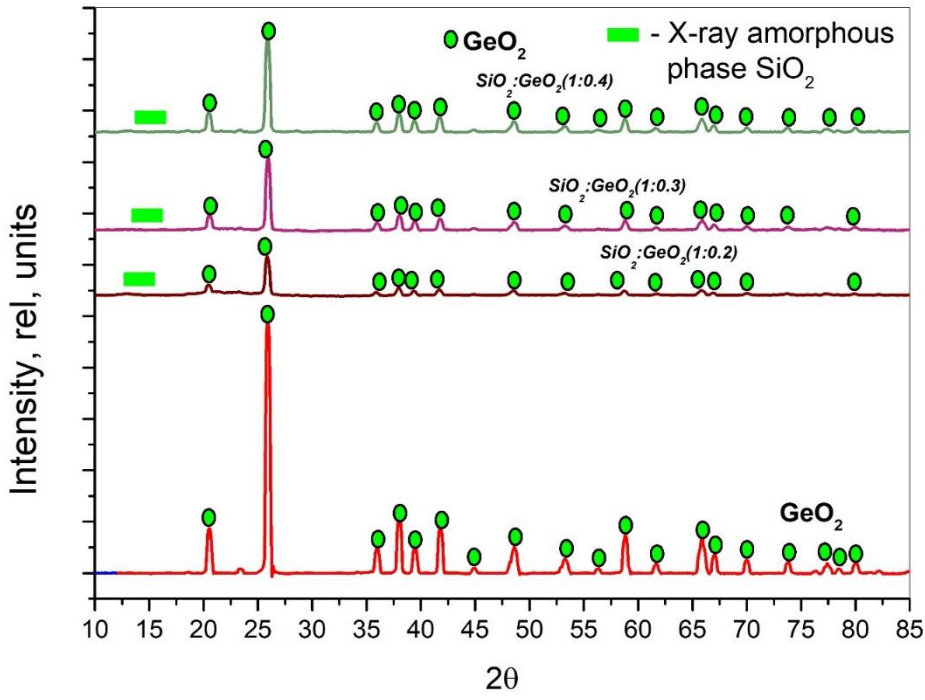
As a result of the phase composition analysis of the SiO_2:GeO_2 and SiO_2:Ge° nanocomposites synthesized at 800 °C using X-ray diffraction (XRD), the following data were obtained:

For the SiO_2:GeO_2 composition (Fig. 6a): A peak is observed at $2\theta \approx 22.0°$, corresponding to silica (SiO_2), which is in an amorphous form. Peaks are also recorded at $2\theta \approx 20.5°$, 25.8°, 35.95°, 37.99°, 39.4°, 41.7°, 48.6°, 53.2°, 58.8°, 65.9°, 67.0°, and 70.1°, associated with germanium oxide (GeO_2). It is important to note that silicon oxide (SiO_2) did not crystallize at 800 °C, indicating its amorphous nature.

For the SiO_2:Ge° composition (Fig. 6b): When germanium oxide is reduced in the SiO_2 matrix with a concentration of 0.4 mol%, partial crystallization of silicon dioxide occurs, as evidenced by the appearance of a peak. Peaks were also recorded at $2\theta \approx 27.4°$, 45.5°, 53.8°, 66.2°, 72.9°, 83.8°, and 90.2°, corresponding to reduced germanium (Ge), indicating the transformation of germanium oxide into a metallic state.



a)

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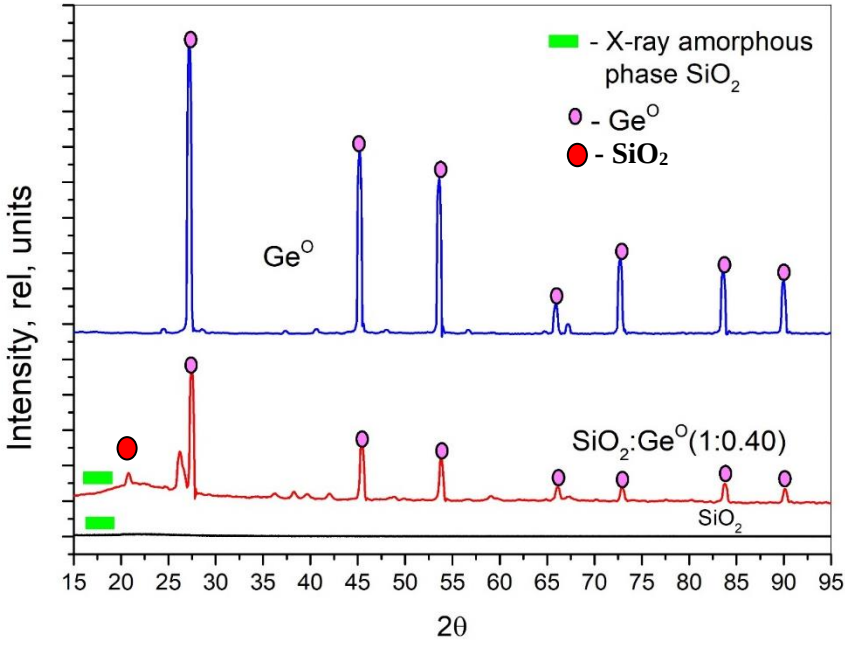

b)

Figure 6. XRD spectra of xerogel micro-powders formed from an aqueous dispersion of Aerosil A-380 and germanium nitrate: (a) annealed in air at T = 800 °C for 1 hour; (b) annealed in hydrogen at T = 800 °C for 1 hour.

The X-ray diffraction analysis confirms that the introduction of germanium into the xerogel affects the phase composition of the materials. Despite the high processing temperature, SiO_2 retains its amorphous structure, which can influence the properties of the obtained composites and their potential applications in various fields, including nanotechnology and thin-film technology [11–12]. These peaks confirm the presence of germanium oxide and reduced germanium phases in the samples, indicating changes in the phase composition at high processing temperatures.

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IV. Conclusions

As a result of the research conducted, the following scientific and technical results were obtained:

- Influence of germanium on the properties of xerogel: The introduction of germanium into SiO_2 xerogel significantly alters its morphological and physicochemical characteristics. SiO_2:GeO_2 and SiO_2:Ge^o samples demonstrate a clear globular structure and high homogeneity, making them promising for use in various fields.
- Formation of a porous structure: It has been established that germanium forms a two-dimensional coating on the surface of SiO_2 globules, contributing to the formation of a porous structure, especially in SiO_2:GeO_2 samples. This can improve adhesion and mechanical properties, which are important aspects for thin-film technologies.
- Particle size: SiO_2:GeO_2 nanocomposites show a uniform distribution of particles ranging in size from 15 to 32 nm, indicating a high level of dispersity and a spherical particle shape. In SiO_2:Ge^o samples, larger aggregates ranging in size from 635 to 1200 nm are observed, likely due to the agglomeration of germanium ions during heat treatment.
- Changes in chemical composition and structure: Chemical and phase analysis confirmed that germanium not only modifies the chemical composition of the xerogel but also contributes to changes in its structure. This opens up new perspectives for further research and development in the field of composite materials.
- Analysis of the chemical and phase composition indicates that germanium affects the changes in both chemical and physicochemical properties of the xerogel, opening up new opportunities for application in thin-film technologies and composite materials.

Thus, the results of the study highlight the importance of investigating the interaction of additives with the main components of xerogels to create materials with desired properties.

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