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Deposition of tin and gold on porous silicon by vacuum thermal spraying

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Abstract. In this work, a tin-gold layer was deposited on porous silicon (KEF 100) substrates by vacuum-thermal process (VUP) to improve the performance of sensors. By the method of X-ray photoelectron spectroscopy (XPS) we analyzed the surface of the materials of the original porous silicon, as well as porous silicon with tin and gold, according to the method [1]). XPS overview spectra allow identifying elements present on the sample surface, as well as determining their oxidation state and concentration. Using this information, valuable data on the surface composition can be obtained and the chemical structure of the sample can be analyzed. The results obtained showed that using the vacuum-thermal method can be successfully applied to obtain nanocomposites of porous silicon with tin and gold. The obtained nanocomposites contain phases of tin dioxide, tin suboxide/monoxide and metallic tin. Compared to tin, the gold film is formed in a smoother, more uniform manner. XPS spectra show that the gold is metallic, free of impurities and oxides.

Keywords: porous silicon, composites, tin, gold, thin films

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Осаждение олова и золота на пористом кремнии методом вакуумно-термического напыления

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Аннотация. Методами атомно-силовой микроскопии и рентгеновской фотоэлектронной спектроскопии получены данные о морфологии и составе образцов пористого кремния с вакуумно-термическим испарением оловом и золотом. Установлено, что химическое осаждение тонких металлических слоев на пористый кремний приводит к образованию композитной структуры, при этом наблюдается частичное окисление оловянной пленки, а пленка золота остается металлической.

Ключевые слова: пористый кремний, композиты, олово, золото, тонкие пленки

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Introduction

Porous silicon (por-Si) is a very important material for the fabrication of various types of sensors. Its nanostructure with many micropores and a large internal surface area has a number of unique properties that make it attractive for a variety of applications. One application of porous silicon is the creation of sensing elements for gas and chemical sensors. Due to its large surface area, porous silicon can effectively sorb target substances and provide their detection at very low concentrations [1]. At the same time, the porous structure allows for a fast and sensitive sensor response. Functionalization of porous silicon surface with tin and gold is an important step in the process of creating sensors and other devices based on porous silicon. It allows to expand the capabilities and improve the properties of this material, making it even more attractive for a wide range of applications [2].

Materials and Methods

To obtain samples of porous silicon with deposited metals, the method of thermal vacuum sputtering through a mask on the VUP-4 unit was used. Porous silicon (KEF) with resistivity of 0.2 Ohm·cm^2, with orientation ⟨100⟩, obtained by electrochemical anodization in an electrolyte based on hydrofluoric acid and isopropyl alcohol [3] was used as a substrate.

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The Sn (por-Si-Sn) and Au (por-Si-Au) layer with a thickness of about 200 nm was sputtered using VHF-000 grade tin and OSF gold wire on a “cold” porous silicon substrate. The sputtering rate was 3–5 nm/s, at a residual gas pressure in the chamber of about $5 \cdot 10^{-5}$ to 10^{-4} mmHg. The morphology of the obtained samples was examined on an atomic force microscope (SOLVER P47 PRO). Image processing using NOVA software allowed to analyze the morphological features of the surface. In addition to the morphology analysis, the surface of por-Si, por-Si-Sn and por-Si-Au samples was analyzed by XPS X-ray photoelectron spectroscopy at the SPECS facility, according to the method [4].

Results and Discussion

Fig. 1 shows AFM images of the surface. When tin and gold film is deposited on the porous silicon surface, the surface becomes smoother (Fig. 1, *b*, *c*). For the original porous silicon sample, the roughness is 70 nm, and when tin is deposited, the roughness decreases to 45 nm and gold decreases to 20 nm. The average particle size of tin on porous silicon is 50 nm and gold is about 60 nm, which is due to partial coagulation of the particles.

Survey spectra of XPS are required to investigate the chemical structure and composition of surface materials. The obtained results allow to understand the structure and composition of the material, as well as to optimize the manufacturing processes. The NIST database [5] was used to decipher the obtained XPS spectra.

Analysis of XPS-spectra of Si 2*p* porous silicon (Fig. 2) showed the presence of silicon dioxide (Si 2*p*, 103.5 eV), silicon suboxide (Si 2p, 100.5-103.0 eV) and unoxidized crystalline or amorphous silicon (Si 2*p*, 99.5 eV) in the sample [5–7].

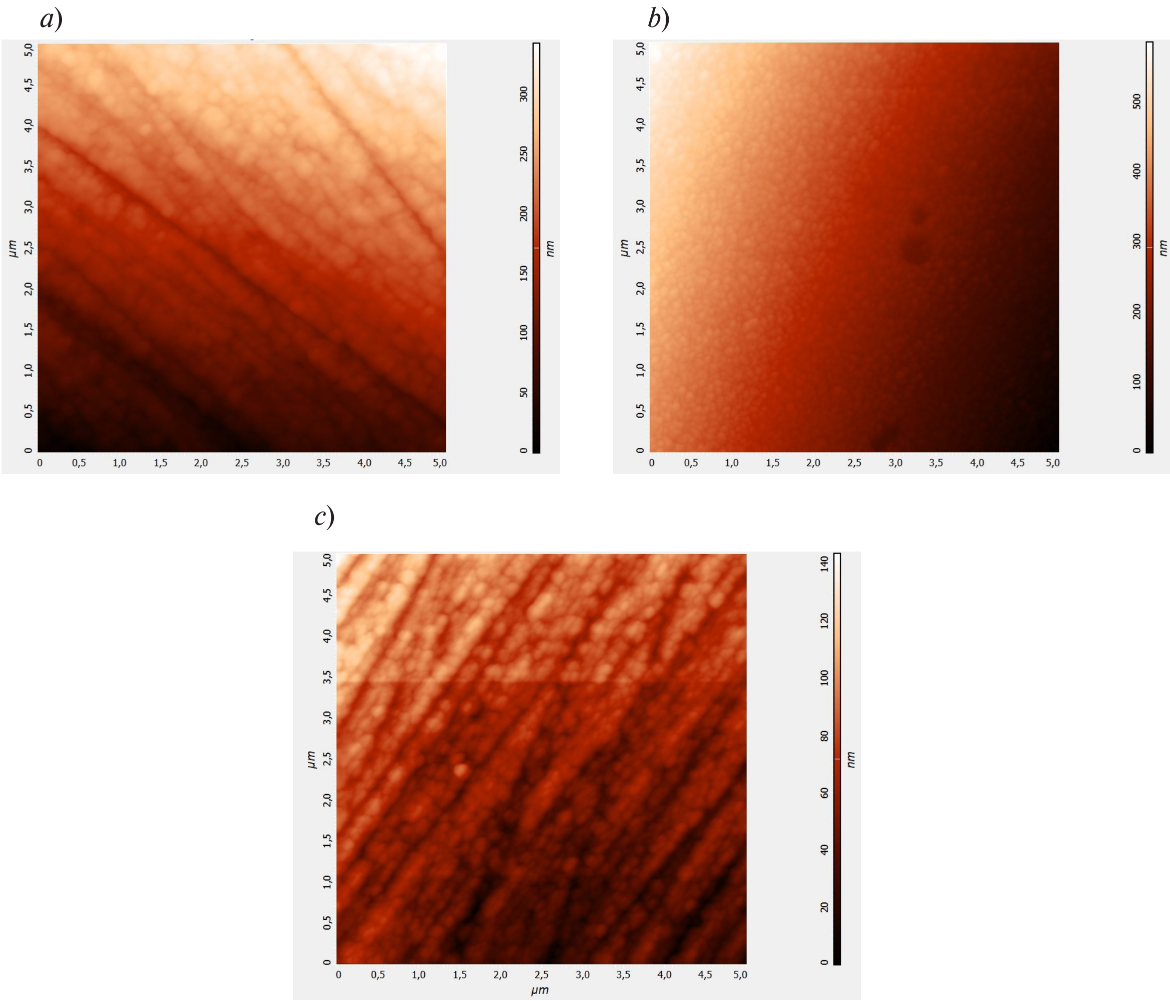

Fig. 1. Surface morphologies of samples obtained by AFM: por-Si (*a*), por-Si-Sn (*b*), por-Si-Au (*c*)

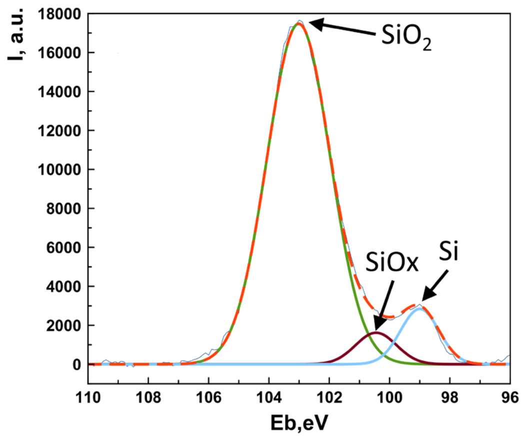

Fig. 2. XPS Si 2*p* spectra with decomposition into por-Si components
(each line corresponds to a spectrum component modeled using a Gaussian)

Fig. 3, *a* shows the XPS spectra of the Sn 3d level backbone. Analysis of the shape and position of XPS Sn 3*d*5/2 spectra and their decomposition into components shows that Sn in the surface layer is in the form of metallic tin (spectrum component with E_b = 485.0 eV) and natural oxide SnOx, where $x \approx 2$ (E_b = 486.5–487.0 eV).

Fig. 3, *b* shows the XPS spectra of the Au 4*f* gold film's Au 4*f* island levels. Analysis of the shape and position of the XPS Au 4f spectra and their decomposition into components shows the absence of visible oxidation of Au (spectrum component with E_b = 84.5 eV).

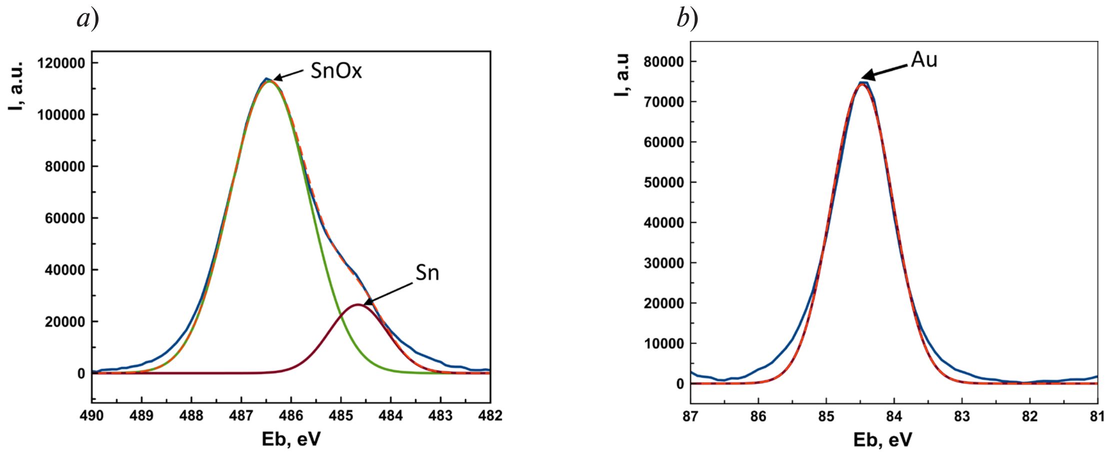

Fig. 3. XPS reference spectra of: Sn 3*d*5/2 tin film
with component decomposition (*a*); Au 4f gold film with component decomposition
(each line corresponds to a spectrum component modeled using a Gaussian) (*b*)

Conclusion

In this work, nanostructured composites of porous silicon with deposited layer of tin and gold were prepared by vacuum-thermal evaporation method. The results showed that the vacuum-thermal method can be successfully applied to obtain nanocomposites of porous silicon with tin and gold. The XPS data agree with the results of atomic force microscopy and confirm the formation of Sn and Au layers on the surface of porous silicon. The obtained nanocomposites with tin contain tin dioxide and metallic tin phases. Compared to tin, the gold film is formed smoother, more uniform, free of impurities and oxides. In general, the conducted studies provide a comprehensive view of the structure and chemical composition of por-Si-Sn and por-Si-Au film samples, which is important for understanding their physicochemical properties and potential applications in electronics and optoelectronics.

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