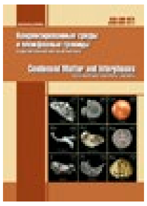

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Structure and composition of a composite of porous silicon with deposited copper

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Abstract

Porous silicon is a promising nanomaterial for optoelectronics and sensorics, as it has a large specific surface area and is photoluminescent under visible light. The deposition of copper particles on the surface of porous silicon will greatly expand the range of applications of the resulting nanocomposites. Copper was chosen due to its low electrical resistivity and high resistance to electromigration compared to other metals. The purpose of this research was to study changes in the structure and composition of porous silicon after the chemical deposition of copper.

Porous silicon was obtained by the anodisation of monocrystalline silicon wafers KEF (100) (electronic-grade phosphorus-doped silicon) with an electrical resistivity of 0.2 Ohm·cm. An HF solution in isopropyl alcohol with the addition of H_2O_2 solution was used to etch the silicon wafers. The porosity of the samples was about 70 %. The porous silicon samples were immersed in copper sulphate solution ($CuSO_4 \cdot 5H_2O$) for 7 days. We used scanning electron microscopy, IR spectroscopy, and ultrasoft X-ray emission spectroscopy to obtain data on the morphology and composition of the initial sample and the sample with deposited copper. The chemical deposition of copper on porous silicon showed a significant distortion of the pore shape as well as the formation of large cavities inside the porous layer. However, in the lower part the pore morphology remained the same as in the original sample. It was found that the chemical deposition of copper on porous silicon leads to copper penetrating into the porous layer, the formation of a composite structure, and it prevents the oxidation of the porous layer during storage. Thus, it was demonstrated that the chemical deposition of copper on a porous silicon surface leads to visible changes in the surface morphology and composition. Therefore, it should have a significant impact on the catalytic, electrical, and optical properties of the material.

Keywords: Porous silicon, Composites, Copper, Ultrasoft X-ray emission Spectroscopy, Electronic structure

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1. Introduction

Nowadays, the demand for nanostructured and nanoscale systems is increasing significantly [1–2], as they are widely used in microelectronics and optoelectronics [3–8], as well as in medicine and chemistry [9–11]. One of the most common nanomaterials used in modern industry and science is porous silicon (por-Si) [12–15]. Porous silicon is a material obtained by anodising monocrystalline silicon. Depending on the properties of the original silicon wafer and the anodisation process parameters, por-Si can have different morphology and different optical and electrophysical characteristics [16–18]. Various electrolytic cell configurations are used to produce por-Si [19–21]. In contrast to bulk silicon, porous silicon exhibits a number of properties such as a high specific surface area, photo- and electroluminescence, and biocompatibility. Due to these unique properties, por-Si can be successfully applied in optoelectronics, micromechanical systems, and biomedicine. Recently, the deposition of metals on porous silicon has been extensively studied, which will greatly expand the application of the resulting composite material [22–25].

It is known that copper has a lower electrical resistivity and has a higher electromigration immunity compared to aluminium [26], which may be of use in electroluminescence and gas sensors.

In study [27], porous silicon with electrochemically deposited copper particles was successfully applied as a photodetector. The authors of [28] obtained samples of porous silicon with Cu particles which exhibited infrared luminescence bands with peaks at 660.6 and 802.2 nm. Also, por-Si with deposited Cu particles is used as a sensor showing high catalytic activity, reproducibility, wide range of study, as well as stability of operation [29, 30]. When porous silicon doped with copper particles is used as a gas sensor for phosphine detection, the sensitivity is 5 times better than that of its counterparts [31].

There are various methods of copper deposition on porous silicon: vacuum evaporation, electrodeposition, chemical deposition, and immersion plating. The advantage of the chemical deposition of copper is the deep penetration of metal atoms into the pores [32]. In addition, this method is simple and inexpensive.

The choice of the method of film deposition on porous silicon is largely determined by the requirements for the resulting material. In turn, the mechanical, chemical, and physical properties of the films depend on the microstructural characteristics of por-Si: pore size and distribution, texture, etc.

Thus, the aim of this research was to study the change in the structure and composition of porous silicon after chemical deposition of copper.

2. Experimental

Porous silicon samples were produced from monocrystalline silicon wafers (electronic-grade phosphorus-doped silicon, orientation 100, and resistivity of 0.2 Ohm·cm) by electrochemical anodisation in an electrolyte based on hydrofluoric acid [33]. Then, the samples were immersed in a colloidal $CuSO_4 \cdot 5H_2O$ solution (C = 0.1 mol/dm^3) for 7 days (at room temperature, in low light) [34]. The pH of the solution, at which copper was deposited, was 3–3.5. Next, the samples were washed in isopropanol and stored under laboratory conditions in sealed polyethylene bags.

A comparative analysis of the pore size and the thickness of the porous layer of the original sample and the resulting sample was carried out by scanning electron microscopy (JSM-6380LV microscope with microanalysis unit).

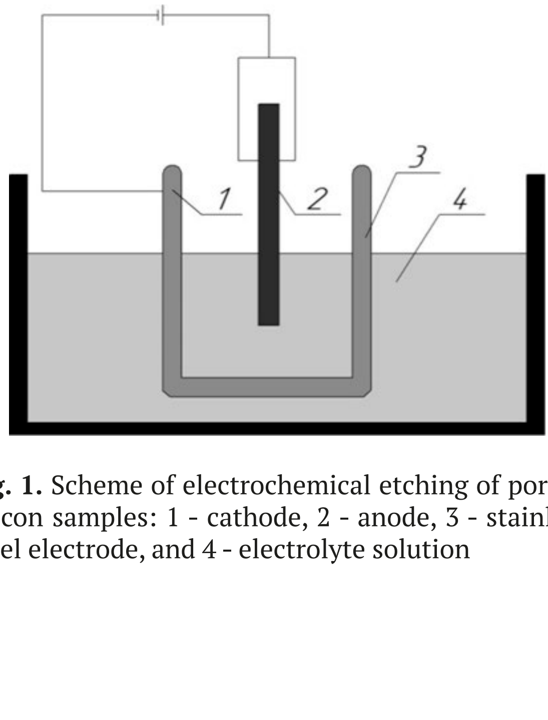


Fig. 1. Scheme of electrochemical etching of porous silicon samples: 1 - cathode, 2 - anode, 3 - stainless steel electrode, and 4 - electrolyte solution

The electronic structural features and phase composition of the initial sample of porous silicon and the resulting sample containing metal were determined by ultrasoft X-ray emission spectroscopy (USXES). The Si $L_{2,3}$ spectra of porous silicon samples were obtained using an RSM-500 X-ray spectrometer-monochromator, which allows the study of spectra in the wavelength range from 0.5 to 50 nm. The analysis depth was 20 nm, at an electron energy of 1.5 keV exciting the X-rays. The USXES spectra were modelled by weighting coefficients using custom-made software. When modelling the Si $L_{2,3}$ spectra of por-Si samples, we used reference spectra of monocrystalline silicon c-Si, amorphous hydrogenated silicon a-Si:H, low-coordinated silicon Si_{lc}, silicon suboxide SiO_x (x ~ 1.3), and silicon dioxide SiO_2 [35–36]. The modelling error was defined as the difference between the areas under the experimental and modelled Si $L_{2,3}$ spectrum that did not exceed 10%. The tests were carried out two weeks after the samples were prepared.

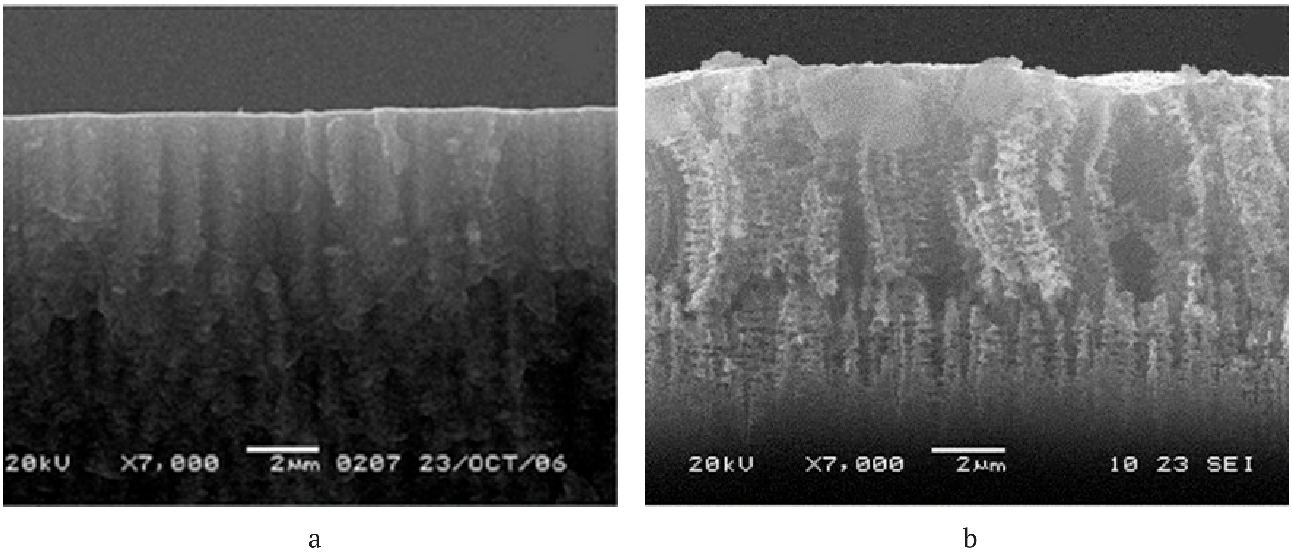
In order to obtain data on the chemical bonds and their possible deformations on the surface of the por-Si samples, IR spectroscopy was carried out. IR transmission spectra of porous silicon samples were obtained using a Vertex 70 FTIR spectrometer (Bruker) with an ATR unit. All tests were performed one month after the samples were prepared.

3. Results and discussion

Fig. 2 shows the SEM images of the cleavages of the original porous silicon and the samples with chemically deposited copper. The average pore diameter in porous silicon is ~100–150 nm, which is typical for por-Si obtained by the previously described method. Image analysis shows that the pore shape was distorted by the chemical deposition of copper on porous silicon. Inside porous silicon, cavities up to 4–5 μm high were formed, which is approximately half the height of the original sample (~10 μm). There is no apparent change in the lower part, and the predominant orientation (100) perpendicular to the surface is retained.

To confirm the presence of copper in the porous layer, an energy dispersive microanalysis of the sample cleavages was carried out (Fig. 2). It showed the presence of ~10 at. % of copper (light inclusions). Similar results were obtained earlier during tin deposition [37]. The nucleation and growth of copper inclusions can be explained mainly by physical adsorption onto a substrate with a sufficiently high specific surface area.

The USXES Si $L_{2,3}$ surface spectra of the samples are shown in Fig. 3. Modelling results using the reference sample spectra showed that the porous silicon samples with deposited copper differ significantly in their phase composition from the original porous silicon. The surface layer of the original porous silicon contains crystalline and



a b

Fig. 2. SEM images of the porous silicon cleavage with nanoparticle inclusions: a: original porous silicon, b: porous silicon with deposited Cu (light inclusions)

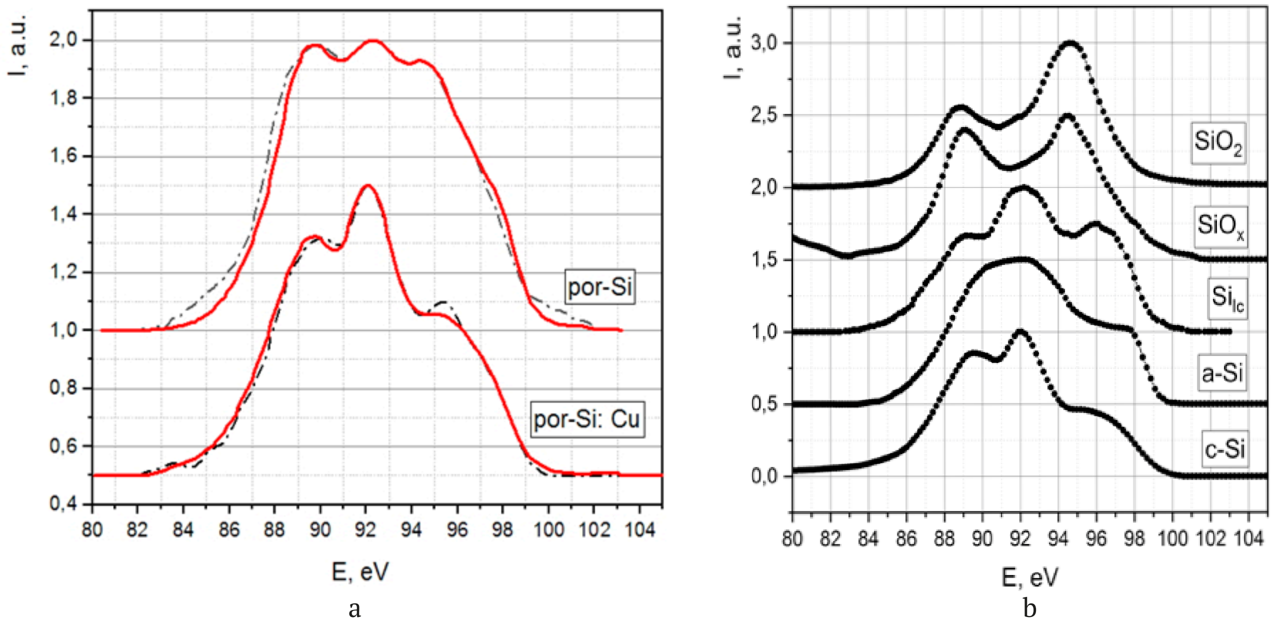


Fig. 3. USXES Si $L_{2,3}$ spectra of the porous layer of the original sample and samples with deposited copper

partially disordered silicon, amorphous silicon, silicon suboxide, and silicon dioxide (Table 1).

The composite sample obtained by chemical deposition of copper has significantly higher percentage of unoxidised crystalline c-Si and partially disordered silicon Si_{lc} phases (~95 % vs. 60 %) with a significantly lower percentage of oxide phases of SiO_x+SiO_2 (Table 1). Most likely, it is due to the fact that the introduction of a sufficiently large (up to 10 at. %) amount of copper particles into the porous layer leads to the formation of a continuous thin layer on the pore surface. In turn, this largely prevents further oxidation of the porous layer during storage.

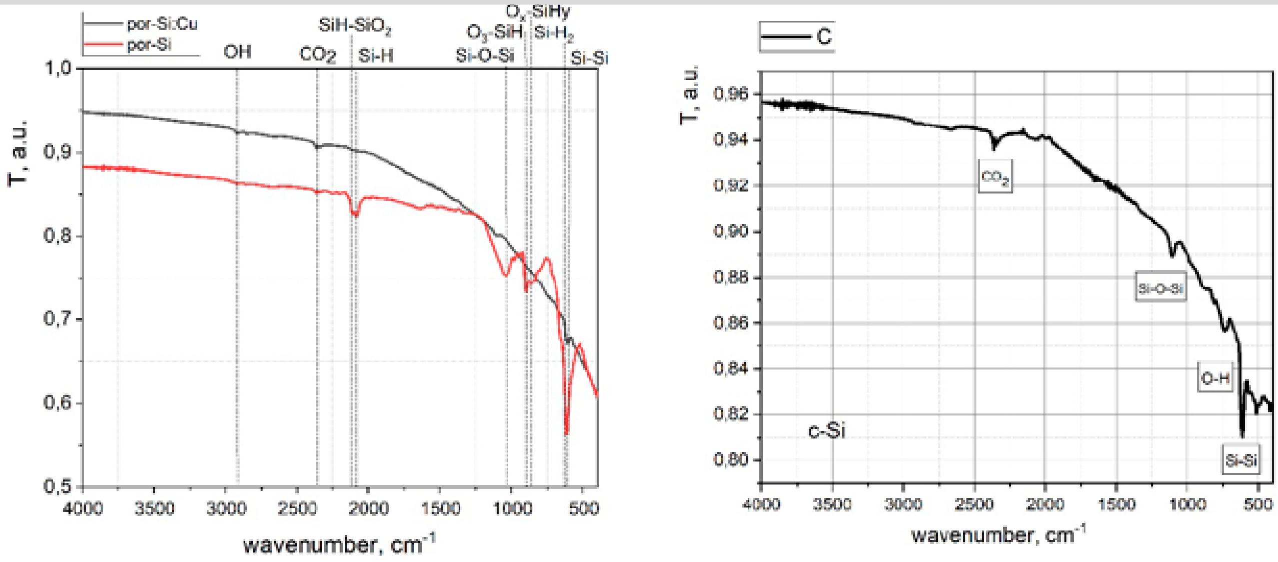
The transmission spectra of the samples of porous silicon and porous silicon with deposited copper, obtained by IR spectroscopy using the ATR unit, are shown in Fig. 4. Despite the fact that the analysis depth of this method is ~1–2 μm, while the depth of the USXES method is 20 nm, the obtained data correlate with each other well enough.

The IR transmission spectra of porous silicon samples after 30-day storage in air under laboratory conditions show the typical features of this material (Table 2) [38, 39]. Analysis of the por-Si spectrum suggests the presence of the main transmission band corresponding to Si-Si vibrations (616 cm^{-1}) and different configurations of Si-H_x bonds (625, 2084, and 2200 cm^{-1}), as well as bonds of the O_x-SiH_y (865 cm^{-1}) and O_3-SiH (900 cm^{-1}) types. In the range of wavelength numbers between 2500 and 4500 cm^{-1}, almost no peculiarities were observed in the spectra of the samples. The absorption bands in the region of 2360 cm^{-1} correspond to adsorbed CO_2.

The IR spectrum of the sample with deposited copper is generally similar to the spectrum of the original crystalline silicon substrate (Fig. 4). It shows much less pronounced features in the same areas as in the original porous silicon. The absorption band corresponding to Si-O-Si bonds and the bands characteristic of Si-H_x and O_y-Si-H_y bonds are practically absent. It should

Table 1. Phase composition of samples of original porous silicon and samples with chemically deposited copper

	"Phases", %					
20 nm	nc-Si	Si_{lc}	a-Si:H	SiO_x	SiO_2	Error, %
por-Si, 20 nm	19	5	35	28	13	5
por-Si: Cu, 20 nm	80	13	–	–	7	5



a b

Fig. 4. IR spectra of porous silicon and por-Si with deposited copper (a), as well as reference spectra (b)

be noted that Si-H_x and Oy-Si-H_y bonds in porous silicon actively participate in oxidation processes during storage of samples. They are replaced by Si-O-Si bonds and cause changes and degradation of various functional characteristics of the structures [40].

When studying the kinetics of sorption of porous silicon in air, its oxidation was observed after 30 days [41]. In the case of porous silicon with precipitated copper no such changes occur. Therefore, the absence of oxidation after 30 days of exposure of the samples to air may indicate significant stabilisation of the composition and surface properties of the composite, as we assumed further changes in the functional characteristics of the original porous silicon in the process of natural ageing. The assumed mechanism of slowing down the oxidation of porous silicon is that copper prevents oxygen from penetrating into the porous layer when interacting with the atmosphere. It is oxidised in the first place.

Table 2. IR absorption bands in porous silicon and the composite with chemically deposited copper

Wave number, cm^{-1}	Por-Si
615	Si-Si val. sym.
625	Si-H_2 pend.
865	O_x-SiH_y deform., SiF
900	O_3-SiH deform.
1057	Si-O-Si val. TO
2084	Si-H val. long.
2200	SiH-SiO_2 struc.
2360	CO_2

4. Conclusions

In this study, a method for the chemical deposition of copper into porous silicon from an aqueous solution of copper sulphate was developed. The results showed that copper penetrates the pores reasonably well when using chemical deposition and slows the oxidation of the porous layer during long-term storage in air. Thus, the methodology developed in the study can be successfully applied to create composite materials with improved properties.

Contribution of the authors

The authors contributed equally to this article.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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