Original articles

Electronic structure and composition of tin oxide thin epitaxial and magnetron layers according to synchrotron XANES studies

O. A. Chuvenkova[1], N. I. Boikov[1], S. V. Ryabtsev[1], E. V. Parinova[1], R. G. Chumakov[2], A. M. Lebedev[2], D. Smirnov[3], A. Makarova[4], S. S. Titova[1], K. A. Fateev[1], S. Yu. Turishchev[1]✉

[1]Voronezh State University,
1 Universitetskaya pl., Voronezh 394018, Russian Federation

[2]National Research Center "Kurchatov Institute",
1 Akademika Kurchatova pl., Moscow 123182, Russian Federation

[3]Dresden University of Technology,
18 Zellescher Weg, Dresden 01069, Germany

[4]Free University of Berlin,
22 Arnimallee, Berlin14195, Germany

Abstract

The materials of the tin-oxygen system and thin-film structures based on them are modern and actual for the creation of a wide range of electronic devices, for example, resistive gas sensors of high sensitivity and short response time with low energy consumption and high manufacturability. An important direction in the study of such materials and structures is the control of properties with variations in technological formation regimes. Information on the composition, local atomic and electronic structure of thin layers of the tin-oxygen system with varying approaches to their production is in demand.

The work is devoted to the study of the electronic structure of thin layers of tin oxides obtained by modern methods of molecular beam epitaxy and magnetron sputtering. A study of the local partial density of electronic states in the conduction band by X-ray absorption near edge structure spectroscopy of tin and oxygen has been carried out. The data were obtained using high-intensity synchrotron radiation, which allows varying the monochromatized radiation quantum energy without loss in intensity, that is necessary to obtain high-resolution X-ray spectral data.

It is shown that the composition, local atomic surrounding, electronic spectrum and their features depend on the technology of formation and storage conditions of the studied structures. Synchrotron X-ray spectroscopy data show the presence of intermediate oxides of the tin-oxygen system in the studied materials after prolonged storage in laboratory conditions. The data obtained indicate the possibility of controlled variation in the composition, local atomic surrounding and electronic spectrum of thin-film structures of tin oxides of small thickness. The results of the work can be used in the formation and subsequent modification of thin and ultrathin layers of tin oxides by magnetron sputtering and molecular beam epitaxy, as well as in their further application as active layers of microelectronics devices.

Keywords: Tin and its oxides, Electronic structure, Density of states, Local atomic surrounding, Composition, Epitaxial nanolayers, Magnetron nanolayers, X-ray absorption near edge structure, Synchrotron investigations

✉ Sergey Yu. Turishchev, e-mail: tsu@phys.vsu.ru

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1. Introduction

Thin layers of SnO_2 tin oxide are widely used in various fields of technology, including microelectronics, such as resistive gas sensors, transparent electrodes, catalysts, etc. [1-4]. The methods of producing tin dioxide largely determine its properties. In addition, the properties are influenced by the size and morphology of the obtained materials, in the case of layers, this is primarily the thickness, macro- and microstructure. Thus, due to the significant influence of the surface, the properties of nanometer range structures differ greatly from large ones in size, and can be used to create devices with significantly improved characteristics. These characteristics primarily include response speed, miniaturization, power consumption, and others. The creation of heterostructures based on tin and silicon oxide is also promising for use in such fields of technology as thermoelectrics [5]. One of the effective and high-precision methods of forming heterostructures consisting of ultrathin ordered layers is the method of molecular beam epitaxy [6–8]. This method is characterized by a high degree of perfection of the grown layers, including nanometer thickness, their structural and phase boundaries and the ability to control the composition, structure and functional properties of the structures being formed. This method makes it possible to obtain very thin layers of tin on the surface of prepared substrates, for example, silicon. The magnetron sputtering method makes it possible to obtain high-quality tin layers in a wide range of thicknesses for use in various fields of technology. The advantage of the method is the simplicity and flexibility of controlling the modes of structure formation [9-11]. The most noticeable disadvantage is the difficulty of obtaining structurally highly organized continuous layers of small thickness comparable to epitaxial ones. It is known that the properties of small-dimensional objects are largely determined by the contribution of the surface. Thin layers of the tin-oxygen system are no exception. High-precision experimental methods with increased sensitivity to the composition, the specifics of the local atomic surrounding, the electronic spectrum, and the structure of the surface layers of the object under study are especially in demand. One of

these methods is the X-ray absorption near edge structure (XANES) spectroscopy using high-intensity synchrotron radiation. The possibility of changing the quantum energy of synchrotron radiation without changes in its extremely high intensity is a prerequisite for obtaining high-resolution XANES spectra. In the ultra-soft X-ray region of the synchrotron radiation spectrum, XANES spectroscopy is highly sensitive to the local surrounding of a given sort of studied material or structure surface atoms, and therefore is particularly relevant for the analysis of nanostructures of various compositions, including those based on silicon [12–16] and tin [17–21]. This paper presents the results of studies using the XANES synchrotron method of the composition, atomic and electronic structure of thin tin layers formed by molecular beam epitaxy and magnetron sputtering on crystalline silicon substrates.

2. Experimental

The studied samples of "Epitaxy Sn/Si" were obtained by molecular beam epitaxy on a Si (001) substrate with a 50 nm thick Si buffer layer [5]. During the formation of the samples, the purified and dried substrates were transported to an ultrahigh vacuum film growth chamber, where the thermal oxide was desorbed at a temperature of 840 °C. Then, 5 monolayers of tin atoms (~1.6 nm) were grown (deposited) from an effusion cell onto a 50 nm thick silicon buffer layer. Before conducting synchrotron experiments, the samples were stored in the laboratory for several weeks. The method of high-resolution transmission electron microscopy showed the continuity and uniformity of the formed tin layer.

A series of "Magnetron Sn/Si" samples was obtained by magnetron sputtering on a direct current of a tin target with a purity of 99.999% in argon plasma on Si (100) substrates. The argon pressure in the working chamber was 10^{-3} Torr, the discharge current was 60 mA, the voltage was 360 V. The film thickness was determined by the sputtering time at constant operating modes of the formation unit and amounted to 30 nm. Morphology control performed by scanning electron microscopy showed the formation of a continuous uniform granular layer. The size of the single granule did not exceed the thickness

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of the layer. Before conducting synchrotron experiments, the samples were stored in the laboratory for several weeks.

Studies of the samples electronic structure were carried out using the non-destructive XANES method, which allows obtaining information about the local surrounding of absorbing tin and oxygen atoms specificity and the effects of ordering in the structural grid of these atoms of the analyzed surface layer [22]. The XANES method allows obtaining direct experimental information on the distribution of the local partial density of free electronic states in the conduction band of the studied surface layer [14–17, 20]. The high-intensity radiation of the ultra-soft X-ray range of BESSY-II synchrotron, the Russian-German beamline (Helmholtz-Zentrum-Berlin, Berlin, Germany) [23] and KISI-Kurchatov synchrotron, the NANOPES beamline (NRC "Kurchatov Institute", Moscow, Russia) [24] were used. The photon flux was 10^9–10^{11} photons/s, the storage ring current was 50–300 mA. The depth of the analyzed surface layer [25, 26] and the energy resolution for the Sn $M_{4,5}$ and O K edges were ~10 nm and 0.1 eV, respectively. The total electron yield (TEY) detection mode was used when registering the compensation (drain) current of the sample. The vacuum in the experimental chambers was ~ 10^{-10} Torr. The angle of incidence of synchrotron radiation was 90° to the surface plane.

3. Results and discussion

Fig. 1 shows the XANES Sn $M_{4,5}$ spectra of reference materials and studied samples of epitaxial (Epitaxy Sn/Si) and magnetron (Magnetron Sn/Si) tin layers. The reference materials include sintered compressed powder of polycrystalline tetragonal tin dioxide – SnO_2(T), metal foil refreshed in situ in ultrahigh vacuum of the spectrometer without natural oxide – Sn foil refresh, metal foil of tin – Sn foil, for which the XANES Sn $M_{4,5}$ spectra were recorded experimentally, under the same conditions as the studied samples and previously known ones [17, 19]. We also present data obtained from ab-initio calculations [20] of XANES Sn $M_{4,5}$ spectra for compounds unstable in laboratory conditions such as orthorhombic tin dioxide SnO_2(O) and tin single oxide SnO.

XANES Sn $M_{4,5}$ (3d) absorption spectra represent the distribution of p states in the conduction band, which, according to dipole selection rules, reflect transitions from core 3d states to free p- and f-states in the conduction band. The distribution of the main spectral features and their relative intensities, given in a single range for the convenience of comparing the spectra in Fig.1, show that the spectra of the used references are sufficiently different from each other and from the formed nanolayers in terms of their fine structure. A detailed discussion of the reference spectra fine structure is presented in [19–20]. In the spectrum of the studied magnetron layer of the Magnetron Sn/Si sample, a sufficiently pronounced structure of the absorption edges M5 and M4 is observed, the most intense features of which are located at energies ~487.1 eV and ~495.5 eV, B-C and G in
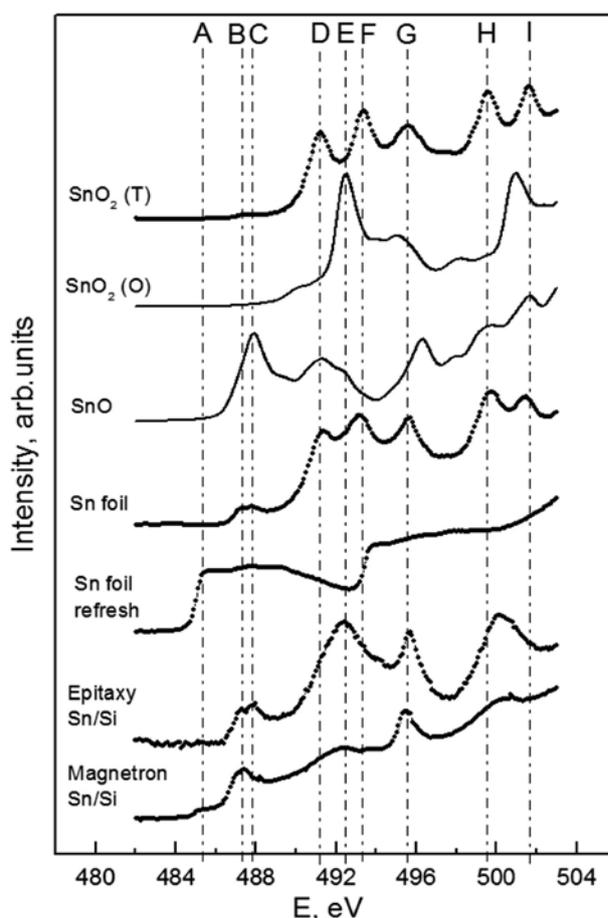

Fig. 1. XANES Sn $M_{4,5}$ of references (SnO_2(T), SnO_2(O), SnO, Sn foil, Sn foil refresh) and studied samples of epitaxial (Epitaxy Sn/Si) and magnetron (Magnetron Sn/Si) tin layers

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Fig. 1, respectively. Thus, spin-doublet splitting of these edges is observed ~8.4 eV. These features correspond in their energy position to tin single oxide SnO with a fine structure of the XANES spectrum at energies of ~487.9 eV, ~491.3 eV. At the same time, a less intense structure is also observed at energies 485.1 eV (A) and 493.5 eV (F), with the same spin-doublet splitting, corresponding to metallic tin without natural oxide (reference spectrum of the Sn foil refresh sample). Another "set" of high-energy features at energies of ~492.3 eV and ~500.7 eV corresponds to orthorhombic tin dioxide. Thus, in the sample obtained by magnetron sputtering, single oxide and orthorhombic tin dioxide were formed along with metallic tin in a surface layer of ~ 10 nm, accessible to XANES sensing when registering Sn $M_{4,5}$ absorption edges. Thus, the 30 nm tin layer obtained by magnetron sputtering of a tin target does not completely oxidize when exposed to air, even for a long time. However, the oxides formed in this layer are unstable under normal conditions. In addition, there is no phase of stable tetragonal tin dioxide with a characteristic fine structure of the M4 edge of DFG (Fig. 1) at energies 491.3 eV, 493.2 eV and 495.6 eV and the M5 edge of HI at energies ~499.5 eV and ~501.6 eV, respectively. Note that when considering the tin metal foil (Fig. 1), stored without any special conditions, the formation of stable tetragonal tin dioxide along with unstable phases of tetragonal tin single oxide and orthorhombic tin dioxide is observed on its surface, as evidenced by the "blurring" of the dip E between the features D and F at energies 491.3 eV and 493.2 eV. On the surface of the studied 30 nm tin layer obtained by magnetron sputtering, only the initial stage of tin oxidation is observed, passing through the formation of intermediate unstable phases of single oxide and orthorhombic tin dioxide. The absence of a stable phase of tetragonal tin dioxide, despite being in laboratory conditions for several weeks, indicates, perhaps, an insufficiently elapsed period of oxidation of the tin layer or the need for additional conditions for its production.

When considering the XANES Sn $M_{4,5}$ absorption edge of the epitaxial tin film (Epitaxy Sn/Si), it can be seen that it also has a very pronounced fine structure. The most intense features were noted at energies of 492.3 eV (E) and 500.3 eV, which, in general, are located closest to the characteristic features of the fine structure of the orthorhombic tin dioxide SnO_2 (O) spectrum. We note the observed electronic states in the region of low-energy spectral features ~487.2 eV and ~495.6 eV, which also correspond to tin single oxide. It is worth noting that the distribution of electronic states in the energy range of ~ 487–488 eV, unlike tin layers obtained by magnetron sputtering, has a double, split structure of the sun at energies of ~ 487.2 eV and 487.8 eV. We observe a similar peak splitting for the surface of the metal tin foil (Sn foil), which is probably due to the simultaneous presence of vacancies in the sublattice of oxygen and the phase of tin single oxide on the surface of this sample. The high intensity of the fine structure features of XANES Sn $M_{4,5}$, corresponding to orthorhombic tin dioxide, indicates the predominance of this phase compared with tin single oxide. Thus, in the sample of tin layers formed epitaxially, the same phases of the "intermediate" tin oxides SnO and SnO_2(O) are observed as in the layers obtained by magnetron sputtering, except for metallic tin. The latter fact is related to the critically small thickness of the epitaxial tin nanolayer, all atoms of which are exposed to interaction with oxygen atoms. In particular, due to the silicon buffer layer presence under the epitaxial tin layer. This layer of crystalline Si, as a result of its noticeable electronegativity, attracts oxygen atoms diffusing through the thin tin layer at least to the tin-silicon interface. At the same time, the XANES Sn $M_{4,5}$ spectrum also lacks fine structure features corresponding to the phase of stable tetragonal tin dioxide. Thus, it is possible to epitaxially obtain thin layers of intermediate phases of tin oxides. To obtain a stable phase of tin dioxide SnO_2(T), the formation regimes should be changed or additional modifying conditions should be applied, for example, oxidative annealing.

Fig. 2 shows the O K XANES spectra of references (SnO2(T) and Sn foil) and studied samples of magnetron (Magnetron Sn/Si) and epitaxial (Epitaxy Sn/Si) tin layers. XANES O K (1s) absorption spectra represent transitions from the core 1s oxygen level to free p states in the conduction band. It can be seen that the

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spectra of the presented reference samples, as in the case of the absorption spectra of XANES Sn M4.5, differ in their fine structure. Thus, tetragonal tin dioxide has a pronounced peak A at an energy of 533.9 eV, as well as features B (536.5 eV), C (538 eV), D (540 eV), E (541.2 eV), F (544.2 eV) and G (549.2 eV). The main peaks A at an energy of 533.85 eV and D at an energy of 540 eV are most pronounced in tin metal foil. The remaining features of the fine structure are smoothed out. The lack of data on the oxygen edge in orthorhombic tin dioxide and tin monoxide makes the interpretation of the oxygen edge of the studied samples insufficiently complete and is the subject of further research.

When considering the oxygen absorption edge in a sample of tin layers obtained by magnetron sputtering, A peak is observed at an energy of 533.9 eV, as well as D at an energy of 540 eV. In general, the edge structure is similar in its features and their energy position to the fine structure of the absorption spectrum of the Sn foil reference, where there are also no pronounced features of the B, C, E and G of the spectra of the tin dioxide reference. The difference observed only in the distribution of intensities between the two main peaks of the structure A and D and in the observation of an insignificant dip in intensity at an energy of 541.6 eV.

In the epitaxially obtained sample (Epitaxy Sn/Si), A peak is also observed at an energy of 533.9 eV and D at an energy of 540 eV. However, the intensity of the low-energy peak A is lower than that of peak D. Here, a wide feature in the D peak region probably indicates the oxidation of the crystalline silicon epitaxial buffer surface, which is located under the nanolayer of epitaxial tin. We noted above that the tin-silicon layer boundary is available for interaction with atmospheric oxygen. At the same time, this interface is in the range of the XANES O K spectrum probing depth. Moreover, the shape and position of feature D correlates well with the data on the fine structure of the XANES O K absorption edges of naturally oxidized silicon [for example see 14]. Thus, the signal from the oxidized silicon atoms of the Si-Sn interface is superimposed on the signal from the tin oxide of the epitaxial nanolayer, forming the spectrum shown in Fig. 2.
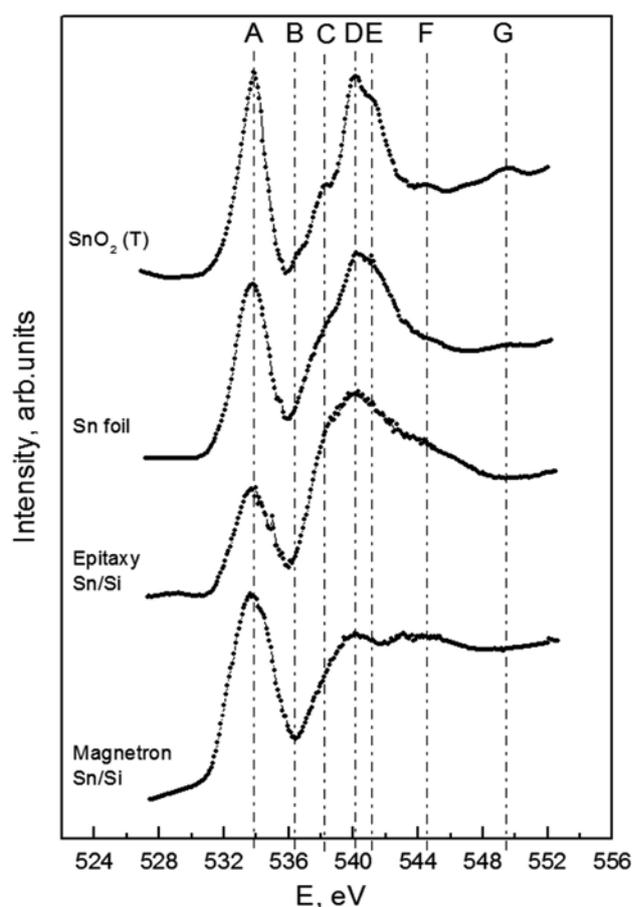

Fig. 2. XANES O K of references (SnO$_2$(T), Sn foil) and studied samples of epitaxial (Epitaxy Sn/Si) and magnetron (Magnetron Sn/Si) tin layers

4. Conclusions

There is a general agreement between the data on the analysis of synchrotron XANES spectra of tin (Sn M$_{4,5}$) and oxygen (O K). On the surface of the studied Epitaxy Sn/Si and Magnetron Sn/Si samples there are oxides similar to natural ones found on the surface of tin foil, but different from tin dioxide tetragonal modification. The layers obtained by molecular beam epitaxy are completely oxidized with the predominance of intermediate phases of tin oxides. Tin layers obtained by magnetron sputtering contain the same phases of intermediate tin oxides SnO and SnO$_2$(O) on the surface, however, the presence of non-oxidized metallic tin is noted. That is, tin layers undergo the same stages of oxide formation during oxidation from the surface, regardless of the method of obtaining these layers. At the same time, the result of interaction with atmospheric oxygen significantly depends on the thickness

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of the formed nanolayer as an indicator of the amount of tin metal available for oxidation. Absence of obvious traces (fine structure of spectra) in the recorded synchrotron data of stable tetragonal tin dioxide indicates insufficient formation conditions for its formation. Thus, the considered approaches require additional effects or conditions to obtain nanolayers of stable tetragonal tin dioxide. The obtained data show the possibility of fine, through the modes of formation and composition, control of the local atomic structure and electronic spectrum of thin layers of tin oxides formed by molecular beam epitaxy or magnetron sputtering, which is important for use in modern structures, including microelectronic ones.

Contribution of the authors

All authors made an equivalent contribution to the preparation of the publication.

Conflict or interests

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

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Information about authors

Olga A. Chuvenkova, Cand. Sci. (Phys.-Math.), Senior Researcher, Joint Scientific and Educational Laboratory "Atomic and Electronic Structure of Functional Materials" of Voronezh State University and the National Research Center "Kurchatov Institute", Voronezh State University (Voronezh, Russian Federation).

https://orcid.org/0000-0001-5701-6909
chuvenkova@phys.vsu.ru

Nikolai I. Boikov, Engineer-Physicist, Joint Scientific and Educational Laboratory "Atomic and Electronic Structure of Functional Materials" of Voronezh State University and the National Research Center "Kurchatov Institute", Voronezh State University (Voronezh, Russian Federation).

https://orcid.org/0000-0002-0512-8666
boykov-hfmm@bk.ru

Stanislav V. Ryabtsev, Dr. Sci. (Phys.-Math.), Head of the Institute of Physics, Voronezh State University (Voronezh, Russian Federation).

https://orcid.org/ 0000-0001-7635-8162
ryabtsev@phys.vsu.ru

Elena V. Parinova, Cand. Sci. (Phys.-Math.), Assistant Professor, General Physics Department, Voronezh State University (Voronezh, Russian Federation).

https://orcid.org/0000-0003-2817-3547
parinova@phys.vsu.ru

Ratibor G. Chumakov, Cand. Sci. (Phys.-Math.), Senior Researcher of the National Research Center "Kurchatov Institute" (Moscow, Russian Federation).

https://orcid.org/0000-0002-3737-5012
ratibor.chumakov@gmail.com

Alexei M. Lebedev, Cand. Sci. (Phys.-Math.), Senior Researcher of the National Research Center "Kurchatov Institute" (Moscow, Russian Federation).

https://orcid.org/0000-0002-4436-6077
lebedev.alex.m@gmail.com

Dmitry Smirnov, Cand. Sci. (Phys.-Math.), Researcher, Institut für Festkörper- und Materialphysik, Technische Universität Dresden (Dresden, Germany).

anna.makarova@fu-berlin.de

Anna Makarova, Cand. Sci. (Phys.-Math.), Researcher, Institut für Chemie und Biochemie, Freie Universität Berlin (Berlin, Germany).

anna.makarova@fu-berlin.de

Sofiia S. Titova, Teacher of General Physics Department, Voronezh State University, (Voronezh, Russian Federation).

https://orcid.org/0000-0001-6860-401X
titova@phys.vsu.ru

Kirill A. Fateev, Laboratory Assistant in Physics of General Physics Department, Voronezh State University, (Voronezh, Russian Federation).

fateev@phys.vsu.ru

Sergey Yu. Turishchev, Dr. Sci. (Phys.-Math.), Associate Professor, Head of the General Physics Department, Voronezh State University (Voronezh, Russian Federation).

https://orcid.org/0000-0003-3320-1979

tsu@phys.vsu.ru