Effect of annealing temperature on the morphology, structure, and optical properties of nanostructured SnO(x) films

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Abstract

Nanostructured SnO(x) films were obtained by molecular beam epitaxy (MBE). The morphology, structure, and optical properties of obtained films annealed in the temperature range of 200 °C–1000 °C were studied. The reflection high-energy electron diffraction during the film deposition by the MBE method and the x-ray phase analysis showed that the initial films are in the polycrystalline phase. A single orthorhombic SnO2 phase was obtained for the first time after annealing the SnO(x) film in the air at a temperature of about 500 °C. The sharp change in the optical constants near the temperature of 500 °C was established using ellipsometry. The pronounced absorption edge appears in the short-wave region at temperatures above 500 °C and it disappears at lower temperatures. The film thickness changed non-monotonically during the annealing in the air. At first, it grows from 45 nm to 65 nm (active oxidation to 500 °C), and then (above 600 °C) it begins to decrease. The annealing at temperatures of 500 °C–1000 °C leads to the film compaction, since the film thickness decreases to 50 nm, but the refractive index increases by 10%–15%. Optical constants track the progress of film phase and morphological changes.

1. Introduction

Tin oxides belong to a class of materials combining the high electrical conductivity, the optical transparency in the visible range and the absorption in the ultraviolet region, as well as the infrared light reflection due to the plasma reflection edge. There is a large number of transparent conductive oxides (TCO). Most well-known are oxides based on the metals, such as In, Sn, Zn, Ga, and Cd [1, 2]. The main requirements for transparent conductive films are low resistivity (<0.001 Ω × cm), high optical transparency (>80%) and large bandgap (>3.5 eV). The most widely used until now is tin doped indium oxide (In2O3). The earth crust contains In, as the main oxide element, but its content is 0.00001% and it is a by-product of the Zn and Pb production. Therefore, obtaining tin-alloyed In2O3 films requires substantial financial investments. Among all TCOs, tin oxide films are available, have attractive electron, optical, electrochemical properties [3] and chemical resistance to acids and bases. Furthermore, they are thermally stable and mechanically strong [4]. Compared to tin oxides, the known zinc oxide (ZnO) films are unstable to acids and alkalis. Tin oxide films have their higher charge-carrier concentrations and transmissions in the visible range [5].

Recently, tin oxide films have attracted great attention of scientists and technologists because of their possible applications in solid-state gas sensors, electrodes for electroluminescent displays, in protective coatings, in solar cells and transparent field-effect transistors [6, 7]. Two basic tin oxides, such as SnO and SnO2, are being studied. The existence of these two oxides is associated with the dual Sn valence, with oxidation degrees +2 and +4. Both of these oxides are known to be wide-gap semiconductors with the rutile structure (tetragonal lattice)
for SnO\textsubscript{2} and the PbO structure for SnO [6]. Tin dioxide is an n-type wide-gap semiconductor with the band gap from 3.6 to 4 eV [8, 9] and a transparency more than 85% [10]. SnO\textsubscript{2} films were obtained by such methods as molecular-beam epitaxy (MBE) [11, 12], pulsed laser deposition [13, 14], electron-beam deposition [15], sputtering [16] and sol-gel process [17]. In most contributions, SnO\textsubscript{2} films are polycrystalline. The data on point defects and the effect of the flow rate on the structure and elemental composition of thin tin oxide films are considered in [18]. Oxygen vacancies and Sn interstitials were observed. Tin monoxide is a p-type wide-gap semiconductor, but its band gap is little known; it lies in the range from 2.7 to 3.4 eV [6]. The experimentally obtained SnO band gap value in [19] reaches 3.6 eV. The large absorption coefficient value indicates predominantly direct optical transitions in the ultraviolet (UV) region. The mobility of holes in such oxides can be higher than in oxides based on metal ions, such as Cu\textsuperscript{+}, Ag\textsuperscript{+}, Au\textsuperscript{+}, Zn\textsuperscript{2+}, since the Sn 5S energy level is closer to the O 2p level and the hole localization decreases. Only a few papers have been published on the formation of SnO\textsubscript{2} films. They were obtained by the methods of electron beam evaporation [20], pulsed laser deposition [21], magnetron sputtering [22] and atomic layer deposition [23]. In addition to the structural properties in [19, 24], the optical and electron properties are presented. It is shown that the pressure increase leads to the formation of nanocrystalline orthorhombic SnO and the transmission in the ultraviolet, visible, and near infrared regions. A hydrophobic coating of the higher p-type conductivity occurs at the pressure decrease. The substrate temperature increase results in the formation of the hydrophilic coating with the orthorhombic SnO and tetragonal SnO\textsubscript{2} phases, as well as with the conductivity-type change to the n-type. The absorption edge shift and the bandgap increase were observed at the increase of pressure or substrate temperature. The polycrystalline SnO film synthesis with the conversion to the SnO\textsubscript{2} phase at the growth temperature increase from 400 to 700 °C was reported in [25]. The change of absorption and bandgap at the temperature increase is considered in the same article. Thus, the structural, optical and electron properties of SnO\textsubscript{2} and SnO film properties should be determined by the conditions of their preparation, such as working pressure, substrate temperature, substrate conductivity type, deposition rate, film thickness, deposition method and the approach used.

The objective of the paper is to show how the MBE method and subsequent annealing in the atmosphere affect the phase transformations in SnO(x) films and changes in their optical and structural properties. The phase transitions are observed from the initial Sn film with a tetragonal lattice (β-Sn) to the SnO\textsubscript{2} and SnO\textsubscript{2} phases at the annealing in the temperature range 200 °C–1000 °C, as will be shown in the article. The annealing process leads to the oxidation and the formation of either individual phases or a phase mixture. Based on the MBE method and the annealing process, nanostructured films with a single orthorhombic SnO\textsubscript{2} phase were first obtained. Besides the crystalline structure, the annealing influences the morphology of films and their optical constants.

2. Experimental section

The nanostructured SnO(x) film samples were obtained using a molecular-beam epitaxy (MBE) machine. The MBE chamber is equipped with an electron-beam evaporator (EBE) for Si and a Knudsen effusion cell for Sn. Sn films were deposited on the Si(1 1 1) substrate with a 100 nm thick SiO\textsubscript{2} layer at the Sn deposition rate of 0.28 Å c\textsuperscript{-1} in the presence of oxygen. Molecular oxygen was supplied into the chamber through a leak. The purity of used oxygen amounts 99.999%. The process pressure during the growth was 10\textsuperscript{-6} Torr. The substrate temperature was chosen below the Sn melting temperature (approximately 180 °C). After the deposition, all samples were pulled out in the air and placed in a quartz tube. Then, they were annealed at the atmospheric pressure in the temperature range of 200 °C–1000 °C. The morphology and surface structure in the growth process in the MBE chamber was observed by reflection high-energy electron diffraction (RHEED).

Furthermore, the film morphology was analyzed by a scanning electron microscope (SEM) (Hitachi SU8220) and an atomic force microscope (AFM). The NT-MDT Solver P47 AFM was used to study the surface morphology. To obtain topography and phase images, a semi-contact mode was used. The crystalline structure and the phase analysis was performed by x-ray Powder Diffraction (XRD), which presents a powder x-ray diffraction system (Shimadzu XRD-7000, CuK\textalpha\, Radiation, λ = 1.54178 Å, linear detector OneSight) in the 2θ range from 10° to 65°. The indexing of diffraction patterns was carried out on the data of the PDF (Powder Diffraction File, released in 2010, International Center for Diffraction Data, Pennsylvania, USA) database. The SEM system allows carrying out the elemental analysis of the grown samples, since it additionally includes the EDX (Energy-dispersive x-ray spectroscopy) Bruker detectors. The XFlash 5060F QUAD detector included in the SEM equipment was used for the analysis of the x-ray spectrum emission energy of the structures obtained by the MBE technique and annealed in the furnace. The analysis of the material chemical state, both in its initial state and after annealing, was carried out using a SPECS x-ray photoelectron spectrometer (XPS). In this work, the characteristic monochromatic radiation AlK\textsubscript{0.1,2} (h\nu = 1486.74 eV, 200 W) was used to excite the spectra. The studies of optical constants in the wavelength range of 250–1000 nm were carried out by the ellipsometry.
method. The measurements were made on the ellipsometers of our own design [26]. The film optical constants restoration included several steps. Firstly, the determination of the SiO₂ layer thickness and optical constants on the masked sample areas, where the SiO₂ sublayer was not covered by Sn was made. Secondly, the determination of the SnO(x) layer thickness and optical constants was carried out. An independent solution of the inverse ellipsometry task was used at each stage for each wavelength with multi-angle spectral measurements (VASE). The film was considered uniform in depth. Therefore, the reconstructed dielectric functions and thickness are effective in case of a not uniform layer structure. The processing on effective medium approximation (EMA) models and the recovery of dielectric SnO(x) cluster functions, after accumulating the sufficient amount of experimental data and comparing these dielectric functions with the microscopy data on the film morphology, are possible.

3. Results and discussion

3.1. Structure of initial and annealed SnO(x) films (RHEED, XRD)

The formation of nanostructured SnO(x) films was investigated in the temperature range of 200 °C–1000 °C. At the first stage, the Sn film was deposited on the Si(111) substrate with a 100 nm thick SiO₂ layer at 180 °C. The molecular oxygen flux was supplied in the MBE chamber during the Sn growth from the Knudsen effusion cell. Based on the RHEED data, the morphology and structure of the growing films were controlled in the growth process. In figure 1 is the RHEED pattern from the Sn film grown in the presence of oxygen. The obtained film is polycrystalline, as shown in the figure. This conclusion can be made by the presence of rings on the RHEED pattern. Further, the samples in the air were unloaded and annealed at the atmospheric pressure. In order to determine the crystalline structure and phase composition, it was necessary to use XRD. The x-ray phase analysis pattern. Further, the samples in the air were unloaded and annealed at the atmospheric pressure. In order to determine the crystalline structure and phase composition, it was necessary to use XRD. The x-ray phase analysis pattern. Further, the samples in the air were unloaded and annealed at the atmospheric pressure. In order to determine the crystalline structure and phase composition, it was necessary to use XRD. The x-ray phase analysis pattern. Further, the samples in the air were unloaded and annealed at the atmospheric pressure. In order to determine the crystalline structure and phase composition, it was necessary to use XRD.

The range of angles 2θ = 28°–29° from the measurements was excluded, since it contains an intense (111) Si substrate peak. The XRD patterns for the as-grown sample, as well as for the samples annealed at 200 °C and 600 °C, are demonstrated in figure 2(a). The annealing temperature at the atmospheric pressure was varied from 200 to 800 °C. Several samples from this series are shown in the figure. The annealing of all films lasted one hour. The calculated diffraction patterns of determined Sn, SnO and SnO₂ material phases are shown in figure 2(b), too. The as-grown sample diffraction peaks at 2θ = 30.6°, 32.0°, 43.9° and 44.9° correspond to the (200), (101), (220) and (211) planes of the tetragonal Sn crystal (β-Sn, PDF #010-75-9188). Based on the RHEED patterns it can be concluded that the Sn film is polycrystalline. The annealing temperature increase results in the appearance of the SnO phase. The sample annealed at 200 °C mainly contains SnO (Romarchite Tin Oxide, PDF #000-55-0837), as demonstrated in figure 2(a). Stannous tin oxide (SnO) is crystallized in the tetragonal structure. However, the (200) and (101) peaks related to β-Sn are still observed at this temperature. The transformation of the crystalline film structure in stannic tin oxide (SnO₂) takes place at 500 °C. The diffraction peaks at 2θ = 29.9°, 31.3°, 34.2°, 35.8° and 38.2°, corresponding to the (111), (020), (002), (021) and (200) planes of orthorhombic SnO₂ (α-SnO₂, PDF #010-75-9495), are pointed out in figure 2(a) for the sample annealed at 600 °C. A similar result with orthorhombic SnO₂ was obtained for tin oxide nanowires [27]. The nanowire array included both normal rutile structured SnO₂ and orthorhombic structured SnO₂. Bulk orthorhombic SnO₂ was synthesized at the high pressure of 158 kbar [28]. Furthermore, the appearance of the
mixture of o-SnO$_2$ and $\beta$-Sn is reported in [29]. The formation of orthorhombic SnO$_2$ was explained by the oxygen deficiency during the annealing of disordered (amorphous or nanoparticle) tin oxide. Although the XRD analysis in [29] showed the existence of o-SnO$_2$, this phase is in the mixture with $\beta$-Sn. The data of the x-ray phase analysis reported in this article suggest the transition from tetragonal SnO to o-SnO$_2$, which is a single phase. Taking into account the XRD results, the orthorhombic SnO$_2$ phase was observed starting from 500 °C. The further temperature increase up to 800 °C did not change the SnO$_2$ phase. Nevertheless, the small tetragonal SnO$_2$ part was revealed in addition to orthorhombic SnO$_2$.

3.2. Morphology and chemical composition of nanostructured SnO(x) films (SEM, AFM, EDX and XPS)

To determine the morphology and surface structure of the grown films, AFM and SEM investigations were carried out. The SnO(x) films are found to be the nanostructured films. The SEM images of the SnO(x) film surface at the annealing temperatures of 400 °C and 800 °C are demonstrated in figures 3(a) and (c). The visual differences are observed at the temperature change. A more detailed analysis can be carried out considering the distribution histograms for the number of islands on the size of the base, which are shown in figures 3(b) and (d). The results obtained from the island distributions suggest the formation of the bimodal island distribution at the low annealing temperature (figure 3(b)). The island density and average island size reach $7.2 \times 10^9$ cm$^{-2}$ and 87.5 nm, respectively. These data are obtained at the consideration of the entire island selection. However, the bimodal island distribution is observed. Therefore, it is important to note that two medium sizes are formed in the regions of about 70 nm and 100 nm. The annealing temperature increase of SnO(x) film up to 800 °C results in the formation of a more uniform island distribution, since the ratios of maxima for the number of islands in the distributions differ more than by a factor of 2. The ratio of the maxima corresponding to 70 nm and 110 nm, shown in figure 3(d), rises related to the value, which is obtained at 400 °C (figure 3(b)). In the case of annealing temperature 800 °C, the average island size equals 76.5 nm. It may be concluded that the SnO(x) film morphology begins to transform. A further rise of the annealing temperature noticeably modifies the film morphology. To observe the changes in the morphology, the AFM measurements were additionally performed. The AFM height (figure 4(a)) and phase (figure 4(b)) images for the sample annealed at 1000 °C are presented in figure 4. The AFM phase image allows completing the relief features, which are observed in figure 4(a). The islands contained in the nanostructured SnO(x) film begin to disintegrate into the smaller nanoclusters during the annealing process at 1000 °C. Nevertheless, the initial islands still preserve their boundaries. The changes in the morphology observed at the film annealing can influence the optical constants, as well as the optical properties of obtained SnO(x) films. It will be considered in our article later. The elemental analysis of the grown
samples was studied by EDX spectroscopy. The elemental distribution map for the SnO(x) film is demonstrated in figure 5. It includes the data on the morphology, since the mapping for Sn, O, Si and C overlays the information obtained from secondary electrons. The Sn, O and Si elements correspond to yellow, green and blue colors, respectively. Furthermore, carbon traces can be observed. It is due to the sample exposition to the atmosphere. Based on the map of elements, it can be concluded that the islands are enriched by Sn and O.

The chemical state of SnO(x) films was investigated by x-ray photoelectron spectroscopy. The photoelectron spectra of the Sn3d_{3/2} region for the initial sample and the sample annealed at 500 °C are shown in figures 6(a) and (b), respectively. The decomposition of the Sn3d_{3/2} region, shown in figure 6 (a), reveals the presence of peaks corresponding to different Sn oxidation states of 0 (metal), 1 +2 (SnO) and +4 (SnO_2). Since β-Sn, in the polycrystalline phase, takes place for the initial sample based on the results of the x-ray phase analysis, the SnO and SnO_2 phases obtained from the photoelectron spectra are in the amorphous state, which was induced by the as-grown sample oxidation during the unloading of this initial sample in the atmosphere. The analysis of
Sn3d3/2 photoelectron spectrum for the sample annealed at 500 °C demonstrates the existence of only single SnO2 phase. It is also confirmed by the XRD technique.

Further, the knowledge about the morphology, structure and compounds allows clarifying the measurement results of optical constants.

3.3. Optical properties of SnO(x) films (Ellipsometry)
The optical constants of nanostructured SnO(x) films studied by the ellipsometry method were obtained at different annealing regimes. The refractive index and absorption coefficient for the SnO(x) film at the temperature increase of the atmospheric annealing are shown in figures 7 and 8. The behavior of the refractive index and high absorption coefficient values correspond to the high metallic Sn content in the as-grown layer. The films begin to exhibit dielectric properties at the annealing temperature increase. A smooth change of the refractive index and the pronounced absorption edge in the UV range are observed. The absorption edge shifts from 3.76 eV for 550 °C and to 3.9 eV at temperatures of 700 °C and above. Furthermore, the absorption takes place in the visible part of the spectrum. It is inherited from the initial film and may indicate an insignificant concentration of unoxidized metal Sn clusters. This concentration equals approximately 1% on our estimations from the EMA modeling. The film thickness changes non-monotonically during the annealing process. At first, it grows from 45 nm to 65 nm, since the active oxidation continues to 500 °C. Then the film thickness begins to decrease above 600 °C. Annealing the film at the temperatures of 900 °C–1000 °C leads to the film compaction.

Figure 5. The elemental composition map obtained by the EDX spectroscopy method from the sample annealed at 400 °C (This sample corresponds to that shown in figure 3(a)). The Sn and O elements correspond to yellow and green colors, respectively.

Figure 6. Photoelectron spectra of the Sn 3d3/2 regions for the as-grown sample (a) and for the sample annealed at 500 °C (b).
The film thickness decreases up to 50 nm, whereas the refractive index increases by 10%–15%, as shown in figure 7. The optical constants, thereby, track the progress of phase and morphological changes in the film. It would be important to note the sharp change in the behavior of optical constants near the temperature of 500 °C. Apparently, this temperature is characteristic in the oxidation process. The pronounced absorption edge observed in the shortwave region at the higher temperatures disappears at the temperatures below 500 °C. Comparing the obtained data with the literature, it turns out that the energies of 3.7–3.9 eV correspond to the SnO2 band gap [30, 31]. According to the reported results from the review [32], heterogeneous materials SnO + SnO2 have the refractive index range of 1.7–2.1 in the average wavelength range (400–800 nm). The refractive index of the films obtained in our work for this spectral interval is slightly lower. It seems to us that it is related to the developed layer morphology. Thereby, the voids occupy part of the film volume. The film porosity can reach 40%, as we estimated. This indicates the film island character. Also, the conclusion is confirmed by the results of scanning electron microscopy and atomic force microscopy. The literature data for stannous tin oxide (SnO) are heterogeneous. The bandgaps are reported to be 2.5 eV [33], 2.85 eV [34], 3.2–3.3 eV [35] and 3.6 eV [36]. However, such values were not observed in our films. There is a wide absorption band in the range of 1.9–3.4 eV. Similar behavior of the non-monotonic extinction coefficient was observed in [37]. We associate this absorption with an insignificant (∼1% by volume) amount of unoxidized metallic Sn clusters.

4. Conclusion

Nanostructured SnO(x) films were obtained by molecular beam epitaxy by the Sn deposition in the oxygen flux on the SiO2/Si substrate. The morphology, structure and optical properties of the obtained films annealed in the
temperature range of 200 °C—1000 °C were studied. The reflection high-energy electron diffraction during the film deposition by the MBE method at the temperature of 180 °C and the x-ray phase analysis showed that the initial films are in the polycrystalline phase. Annealing the SnO(x) films in the atmosphere at a temperature of about 500 °C resulted in the appearance of the orthorhombic SnO2 phase, which was previously obtained using a high pressure of about 158 kbar. The annealing temperature increase to 800 °C created a small fraction of the tetragonal SnO2 phase. Using scanning electron microscopy and atomic force microscopy it was found that the films are nanostructured. The island density and average island size reach 7.2 × 10^9 cm^-2 and 87.5 nm, respectively, after the annealing at the temperature of 400 °C. The annealing temperature increase of the SnO(x) film up to 800 °C results in the formation of a more uniform island distribution having the island density and average island size of about 9.2 × 10^8 cm^-2 and 76.5 nm, respectively. It may be concluded that the SnO(x) film morphology begins to transform. A further rise of the annealing temperature noticeably modifies the film morphology. The islands in the nanostructured SnO(x) film begin to disintegrate into the smaller nanoclusters during the annealing process at 1000 °C. The optical film constants were studied by the ellipsometry method before and after the annealing in the atmosphere in the temperature range of 200 °C—1000 °C. The high absorption coefficients correspond to the high metallic tin content. The films begin to exhibit their dielectric properties at the annealing temperature increase. A smooth change in the refractive index and the pronounced absorption edge in the UV range were observed. The absorption edge shifts from 3.76 eV for 550 °C and to 3.9 eV at temperatures of 700 °C and above. Also the absorption in the visible part of the spectrum was observed. It is inherited from the initial film and it may indicate an insignificant concentration of unoxidized metal Sn clusters (approximately 1%) of the total.

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