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MORPHOLOGY AND ELECTRICAL PROPERTIES OF ITO FILMS OBTAINED ON SILICON SUBSTRATES BY CVD METHOD

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ITO films were obtained on silicon substrates using an improved chemical vapor deposition (CVD) method in a quasi-enclosed volume at normal atmospheric pressure, without using a carrier gas. The resulting films had a thickness of 2.8–3.0 microns and a fairly low sheet resistance. Using an SPM 9700HT type scanning probe microscope, the surfaces of 500×500 nm ITO film samples were examined, and the results are presented in the form of two-dimensional (2D) and three-dimensional (3D) images. The electrophysical properties of the grown films were studied by the Hall method and it was shown that the films have an n-type conductivity, a mobility of $\mu \approx 2.5$ cm^2/(V×s) and a concentration of charge carriers $n \approx 1.35\times10^{20}$ cm^{-3} and a sheet resistance of $\rho \approx 1.85\times10^{-5}$ Ω·sq^{-1} (ohms per square). It is shown that our modified method of chemical vapor deposition makes it possible to obtain ITO films with good characteristics acceptable for use in optoelectronics and photovoltaics devices as a transparent contact layer.
Keywords: *Indium tin oxide; CVD method; Concentration and mobility of carriers; Sheet resistance*
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1. INTRODUCTION

ITO (Indy Tin Oxide) thin films attract a lot of attention due to their optical transparency and low resistivity [1, 2]. They are widely used in various optoelectronic devices as transparent electrical contacts or electrodes [3, 4]. For example, they are used in LEDs [5-7], flat-panel displays [8], thin-film solar cells (SC) [9-14], and various sensors [15]. Optoelectronics typically uses transparent conductive thin-film ITO electrodes with a ratio of $In_2O_3:SnO_2 = 9:1$ (90 wt.% In and 10 wt.% Sn) [16, 17]. They are also used as an anti-reflective wide-band "window" in SC due to their high refractive index (n = 1.97-2.06) and relatively low sheet resistance (≤ 10^{-2}–10^{-3} Ω·sq^{-1}). At the same time, ITO contact layers with high charge-carrier mobility and low sheet resistance <10 Ω·sq^{-1} with optical transparency (≥80%) in the visible and near-infrared (IR) ranges make it possible to ensure high efficiency of SC [9-14]. However, due to the growing technological demand for transparent conductive contacts for flat-panel displays with a larger area, as well as for SC and LED structures, new materials with higher electrical and optical properties are required [8, 16], as well as relatively affordable and easy-to-manage technologies for their production [15]. Although ITO films are transparent in the visible range (380-780 nm), their transmission coefficients are low in the near-infrared (IR) range (950-1800 nm). This leads to a decrease in the efficiency of SC in the long-wavelength range of the solar radiation spectrum [9-14]. Therefore, all this requires improving the transparency of ITO films in the near-infrared range to increase the efficiency of SC, as well as the development of relatively inexpensive technological methods for producing ITO films.

To improve transparency while maintaining good electrical conductivity of ITO films, researchers use various methods. Methods for obtaining oxide films with high mobility of charge carriers are presented in [14], and the mechanisms underlying high mobility in oxide films are discussed. Another interesting way to improve the electrical conductivity of oxide films while maintaining their transparency is to create multilayer structures in the form of dielectric/metal/dielectric (ITO/metal/ITO) [6, 17, 18]. Because multilayer films reduce the reflection of light from metal and, due to a sufficiently thin (not strongly affecting transparency) metal layer, provide high electrical conductivity of films [17-19]. In the message [20], polycrystalline ITO films with good optoelectronic characteristics and a homogeneous surface were obtained by chemical deposition from the gas phase (CVD) followed by annealing at a temperature of 550°C in a nitrogen (N_2) atmosphere. The use of the CVD process for the synthesis of ITO, as described in this paper, may become an alternative to the method of magnetron deposition of ITO films, which are quite suitable for use in various optoelectronic devices.

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2. MATERIALS AND METHODS

There are various methods to obtaining ITO films; for example, in [16, 21, 22] ITO films were obtained by high-frequency reactive sputtering with simultaneous ion treatment. Some researchers prefer to use the sol-gel method to obtain In_2O_3 and SnO_2 films [23, 24]. However, in some cases, the CVD method (chemical vapor deposition) is used to obtain high-purity and high–quality films [25]. In the CVD method, reagents are fed directly to the surface of the substrate, where the vaporized molecules of the substances that make up In_2O_3 and SnO_2 thermally decompose and react to synthesize ITO layers consisting of a mixture of In_2O_3 and SnO_2 on the surface of the substrate. In this case, the decomposition of the reagents occurs at temperatures above 120-140 °C and the ITO deposition process is carried out in air, since the deposition product is an oxide, and the oxygen environment does not interfere with the synthesis of ITO oxide material.

In this paper, to facilitate the control of the ratio of the components of the film composition, we modified the method of chemical vapor deposition (CVD) in such a way that the deposition of ITO layers is carried out under quasi-closed volume conditions, i.e., under atmospheric air and pressure conditions [26]. In short, in this method, the deposition process does not occur in a carrier gas stream but in a quasi-enclosed volume at normal atmospheric pressure, which makes it possible to create a large partial pressure of the components close to the saturation pressure of their vapors for a given temperature [20]. This, on the one hand, makes it possible to increase the growth rate, and on the other hand, to reduce the loss of reagents and increase the percentage of raw materials used. The film growth rate in this method is almost 10 times higher than with conventional technology. Figure 1 shows a block diagram of an upgraded ITO film production facility on various substrates at different temperatures of the evaporator and substrate.

ITO films were deposited on silicon substrates by decomposing vapors of alcohol solutions of indium and tin chlorides in the temperature range of 120-140 °C (evaporator temperature) and at substrate temperatures of ~240-250°C. The technological modes were set by controlling the temperature of the substrate, the evaporators to which solutions of indium and tin chlorides are supplied, as well as the ratio of the solution components and the rate of solution supply. The precursors were alcoholic solutions of indium chloride ($InCl_3$) and tin chloride ($SnCl_2$), which we prepared by separately heating metallic indium (In-99.99) and tin (Sn-99.99) in dilute (1:1) hydrochloric acid (HCl-extra pure). During the deposition of ITO films in our upgraded CVD method, the precursor solution is fed to an evaporator heated to 120-140°C, where indium and tin chlorides evaporate from the evaporator, decompose, and to form indium and tin oxides (In_2O_3 and SnO_2) under the influence of temperature. Which then, continuing to move in the direction of evaporation, are directed towards the substrate, and deposited on it. The properties of ITO films obtained by this method depend mainly on the temperatures of the substrate, and the evaporator.

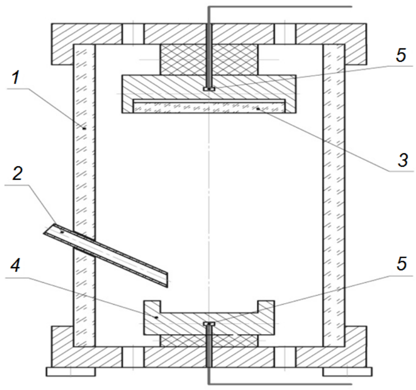

Fig. 1. Block diagram of a CVD reactor for deposition of ITO films on various substrates at different substrate and evaporator temperatures, as well as in different ratios of the In_2O_3:SnO_2 film composition: 1-quartz reactor; 2-tube for supplying reagent solution to the evaporator; 3-substrate; 4-evaporator; 5-thermocouples for controlling the temperature of the substrate and evaporator

3. RESULTS AND DISCUSSIONS

ITO thin films used in many optoelectronic devices are typically less than one micrometer thick. The thicknesses of the ITO films obtained by us, measured on a microinterferometer MII-4 microscope, were 2.8-3.0 µm. We previously demonstrated in [27, 28] that ITO films obtained by the above-described method exhibited a polycrystalline structure with a specific texture. Therefore, we do not present the results of the X-ray diffraction analysis of the obtained ITO layers here.

Studying of the sample surface $0.9In_2O_3$:$0.1SnO_2$ was performed on a scanning probe microscope of the SPM 9700HT type. The measurement was carried out in contact mode, and, for this purpose, a sample area measuring 500×500 nm was selected. Results of the study of the sample surface $0.9In_2O_3$:$0.1SnO_2$ in the form of two-dimensional (2D) and three-dimensional (3D) images are shown in Figs. 2 and 3.

Fig. 2 shows that the film is almost uniform with a small texture (Fig. 3). The sharp peaks of the crystals indicate the nanocrystalline structure of the film. As can be seen from Fig. 3, the heights of the texture peaks are approximately 20-40 nm, but there are also a small number of peaks exceeding 50-60 nm.

Electrophysical parameters of ITO films ($0.9In_2O_3$:$0.1SnO_2$) were measured by the Hall method (HMS-7000). The results showed that the grown ITO layers have an n-type conductivity with a sheet resistance of $\rho \approx 1.85 \times 10^{-5}$ $\Omega \cdot sq^{-1}$ and a mobility of $\mu \approx 2.5$ $cm^2/(V \times s)$ at a charge carrier concentration of $n \approx 1.35 \times 10^{20}$ cm^{-3}.

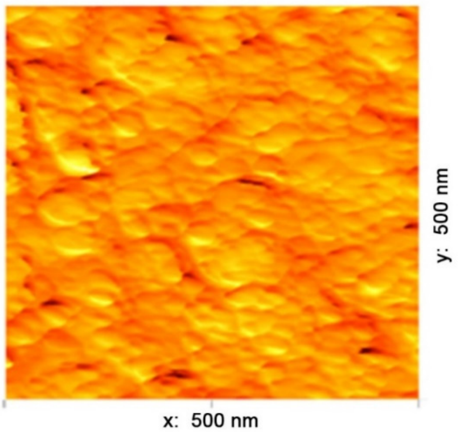

Figure 2. Two-dimensional (2D) image (500×500 nm) of the $0.9In_2O_3$:$0.1SnO_2$ sample surface, captured using SEM

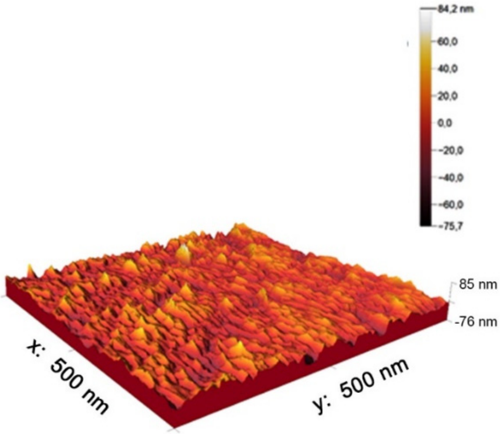

Figure 3. Three-dimensional (3D) image (500×500 nm) of the $0.9In_2O_3$:$0.1SnO_2$ sample surface, captured using SEM

The temperature dependencies of the concentration (Fig. 4) and mobility (Fig. 5) of charge carriers, as well as the sheet resistance (Fig. 6) of the ITO film obtained at a substrate temperature of 240°C (optimal, determined experimentally), were studied. It can be seen from Fig. 4 that the concentration of charge carriers depends nonmonotonically on temperature and varies almost twice in the temperature range from 300 to 420 K, which in turn leads to a decrease in resistivity by nearly a factor of two (Fig. 6).

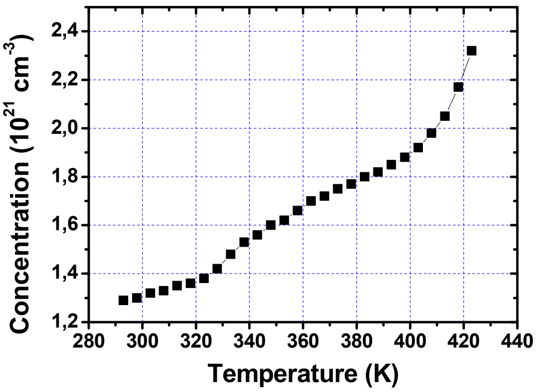

Figure 4. Temperature dependence of the charge carrier concentration in an *n*-ITO ($0.9In_2O_3$):$0.1SnO_2$) film grown on a polycrystalline *p*-Si substrate. (The sample was grown at a substrate temperature of 240°C)

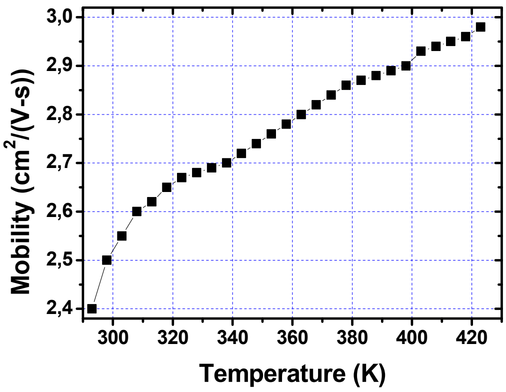

Figure 5. Temperature dependence of the mobility of charge carriers of the *n*-ITO film ($0.9In_2O_3$:$0.1SnO_2$) grown on a polycrystalline *p*-Si substrate. (The sample was grown at a substrate temperature of 240 °C)

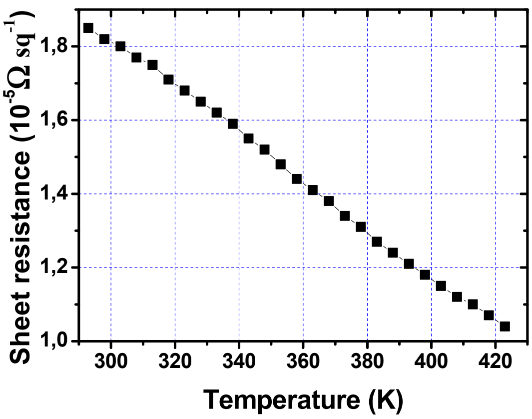

Figure 6. Temperature dependence of the sheet resistance of the *n*-ITO film ($0.9In_2O_3$:$0.1SnO_2$) grown on a polycrystalline *p*-Si substrate (The sample was grown at a substrate temperature of 240°C)

This indicates that the film exhibits semiconductor properties, as confirmed by studies of the temperature dependence of charge-carrier mobility (Fig. 5). As shown in Fig. 5, the mobility of charge carriers increases non-monotonically with temperature.

In some papers, to improve optical characteristics, specifically to enhance transparency, film oxidation was employed, which the authors considered a diffusion process. In the pre-oxidation experiments conducted in these studies, the film did not have time to oxidize in 30 seconds at a temperature of 550°C and below. The authors of [29] claim that it is possible to achieve film transparency at lower temperatures (200-220°C), but this requires longer annealing times (>60 min). This was also confirmed by the results we obtained, as well as the findings of other studies that varied widely in their annealing regimes. For example, during low-temperature annealing for up to 1000 min at 125-165°C [30] and 30-60 min at 200-300°C [31].

4. CONCLUSIONS

Using the modified CVD method, ITO films with a thickness of ~3.0 μm were obtained on *p*-type silicon substrates, and their structural and electrical properties were investigated. The resulting layers exhibit n-type conductivity and a nanocrystalline structure, with a specific electrical resistance of 1.85×10^{-3} sq^{-1}, making them suitable for use as transparent conductive contact layers in solar cells and LED structures.

The studies conducted in this work have demonstrated that our modified CVD method can produce ITO films at relatively low substrate temperatures (240-250°C), with thicknesses of several micrometers and good properties under normal atmospheric pressure and quasi-enclosed volume conditions. We hope these preliminary results will provide valuable insights and support future research on the synthesis of high-quality ITO films. The composition, structure, and properties of such coatings can be adjusted by varying the evaporator and substrate temperatures, as well as through additional post-application treatments.

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МОРФОЛОГІЯ ТА ЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ПЛІВОК ІТО, ОТРИМАНИХ НА КРЕМНІЄВИХ ПІДЛОЖКАХ МЕТОДОМ CVD

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Плівки ІТО були отримані на кремнієвих підкладках за допомогою вдосконаленого методу хімічного осадження з парової фази (CVD) у квазізамкненому об'ємі за нормального атмосферного тиску без використання газу-носія. Отримані плівки мали товщину 2,8–3,0 мікрона та досить низький шаровий опір. За допомогою скануючого зондового мікроскопа типу SPM 9700HT були досліджені поверхні зразків плівок ІТО розміром 500×500 нм, а результати представлені у вигляді двовимірних (2D) та тривимірних (3D) зображень. Електрофізичні властивості вирощених плівок досліджували методом Холла, і було показано, що плівки мають n-тип провідності, рухливість $\mu \approx 2{,}5$ см^2/(В×с) та концентрацію носіїв заряду $n \approx 1{,}35\times10^{20}$ см$^{-3}$, а також шаровий опір $\mu \approx 1{,}85\times10^{-5}$ Ом на квадрат. Показано, що наш модифікований метод хімічного осадження з парової фази дозволяє отримувати плівки ІТО з хорошими характеристиками, прийнятними для використання в оптоелектроніці та фотоелектричних пристроях як прозорий контактний шар.
Ключові слова: *оксид індію-олова; метод CVD; концентрація та рухливість носіїв; шаровий опір*