Optical Properties of ITO—Epoxy Resin Composite Films

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The optical characteristics of transparent and electrically conductive composite coatings consisting of nanostructured antireflective films of indium tin oxide ITO obtained by magnetron sputtering onto a hot substrate and epoxy resin were studied. The process of filling the voids in the ITO film structure with epoxy resin is examined, along with the influence of the filling degree on the optical properties of the coating. It is shown that at the initial filling stage, the antireflection effect of the coating is enhanced compared to the original ITO film, whereas complete filling leads to reflection and transmission spectra that exhibit an interference pattern due to light interaction with the coating. The experimental results show good agreement with data from computer simulations of coatings, where the model parameters were derived from scanning electron microscopy images. The developed model can be further applied to the fabrication of multicomponent nanostructured coatings with tailored properties.

Keywords: indium tin oxide, ITO, nanostructured film, transparent conductive oxides, nanorods, numerical simulation.

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1. Introduction

In recent years, the nanostructured films of transparent conductive oxides have been considered as functional coatings demanded in fabrication of various optoelectronic devices. Such films can form antireflective, diffusing, and reflective surfaces depending on the method and conditions of application, as well as the combination of surrounding layers [1–3]. Nanostructured layers of indium tin oxide (ITO) applied to the surfaces of LED crystals, displays, and protective glasses provide anti-glare properties and increase the efficiency of light output [4–6]. The nanometer-sized inhomogeneities in the structure of these films are distributed in such a way that a medium with an effective refractive index that monotonically declines in the direction perpendicular to the substrate is formed in the film. Such films can be obtained by vacuum deposition methods (electron beam evaporation or magnetron sputtering) on substrates heated to high temperatures [7–9]. It is believed that the formation of ITO nanocrystals in the structure of such films occurs as a result of an autocatalytic process according to the vapor-liquid-crystal (VLC) mechanism [10]. The characteristic dimensions of the threads are usually tens of nanometers in diameter and can reach micron values in length. With such parameters, the resulting film acquires a larger specific surface area, tens of times greater than the specific surface area of a dense film [11]. A greatly enlarged specific surface area of the film facilitates its use as a working element of gas sensors [12,13], but may affect the optical properties of the films when exposed to environmental factors. To increase the resistance of nanostructured films to external impact, a method for forming a thin protective coating of Al_2O_3 using atomic layer deposition (ALD) was previously proposed [14]. Film protection provides mechanical reinforcement of individual film nanocrystals and also isolates the ITO surface from aggressive chemical compounds. Along with that, the profile of the effective refractive index in the film practically does not change due to the small thickness of the dielectric protective layer. When thicker layers of Al_2O_3 are applied using the same method it results in formation of the dome-shaped structures on the film surface, which contribute to increased light extraction from high-refractive index materials. In paper [6], a more than one and a half-fold increase in brightness of an electroluminescent display was observed when using such a coating instead of the traditionally used planar layers. However, the formation of coatings using atomic layer deposition methods requires introduction of an additional expensive technological operation into the fabrication process. An alternative method of forming a protective coating is to apply liquid epoxy resin and then allow it to cure. This method is used, for example, to apply protective films and form lenses during the mass production of LEDs and photodiodes. It should be taken into account that morphology and optical properties of the resulting coating will depend on the degree of filling and wettability of the ITO film with epoxy resin. The wettability of the materials surface is known to have a huge dependence on the degree of development of the material's surface [15,16]. In study [17] it was shown that films obtained by magnetron sputtering of ITO targets onto a hot substrate had pronounced hydrophilic properties. The wettability of the surface of such films with other liquids, interaction of the film material with resin, as well as the effect of the degree of filling on the optical characteristics of the resulting composite coating was not covered in the

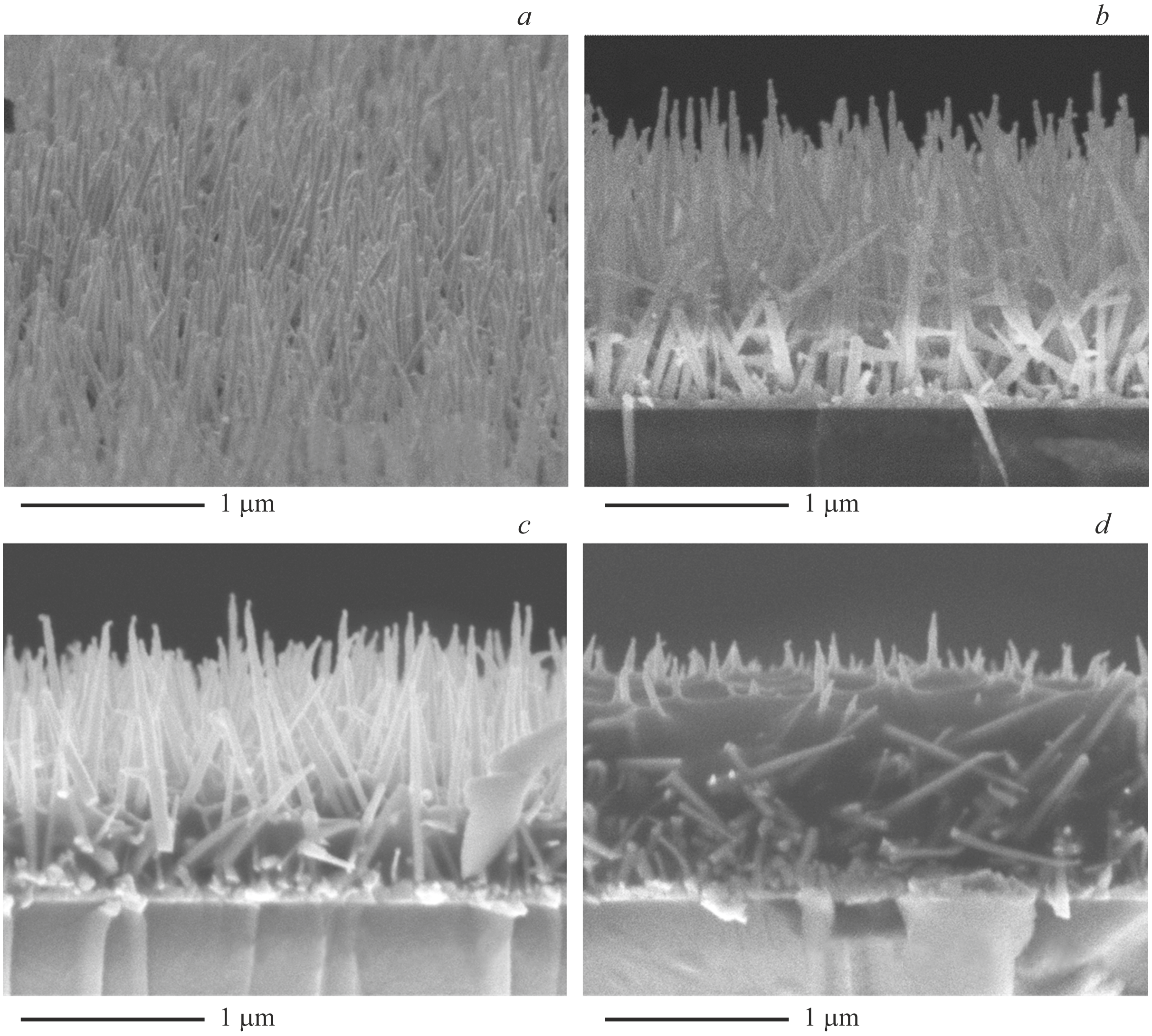

Figure 1. SEM images of ITO films. Top view of initial ITO film at an angle of 10° (*a*), cleavages with different degree of the film filling with epoxy resin: without resin (*b*), intermediate filling (*c*), fully filled (*d*).

above-mentioned work. A computer model based on the finite difference time domain (FDTD) method, widely used in nanophotonics research, was also built [18,19]. The model allows describing the optical properties of such a coating and optimizing the layers thicknesses and the nanocones sizes.

2. Experiment procedure

Nanostructured ITO films were deposited by magnetron sputtering from an In_2O_3-SnO_2 (90/10 wt.%) target onto a substrate heated to 550 °C. A detailed description of the film formation procedure is given in [6]. The films thickness is $\sim$ 1300 nm, while the mass of the material in the film is equivalent to a dense 150 nm thick film, which is caused by a significant decline in density of the nanostructured material when applying ITO onto a heated substrate. Liquid jewelry epoxy resin EpoximaxX Epoxy Crystal PLUS was applied from a pipette to the prepared film samples, forming a coating of variable thickness when spread. Thus, samples of composite films ITO−epoxy resin with different thicknesses of the filling material were obtained, which made it possible to study the optical properties of composite films at various stages of filling the nanostructured ITO material with epoxy resin. After the epoxy resin had cured, the transmission and reflection spectra were measured, and images of the cleavages of the manufactured films were obtained using JEOL JSM-7001F scanning electron microscope. The transmission and reflection spectra of the obtained samples were measured using Optronic Laboratories OL 770 spectroradiometer.

Computational modeling was performed using the finite difference method with Comsol Multiphysics® software package and Electromagnetic Waves (Frequency Domain) calculation interface, with the choice of a quadratic shape

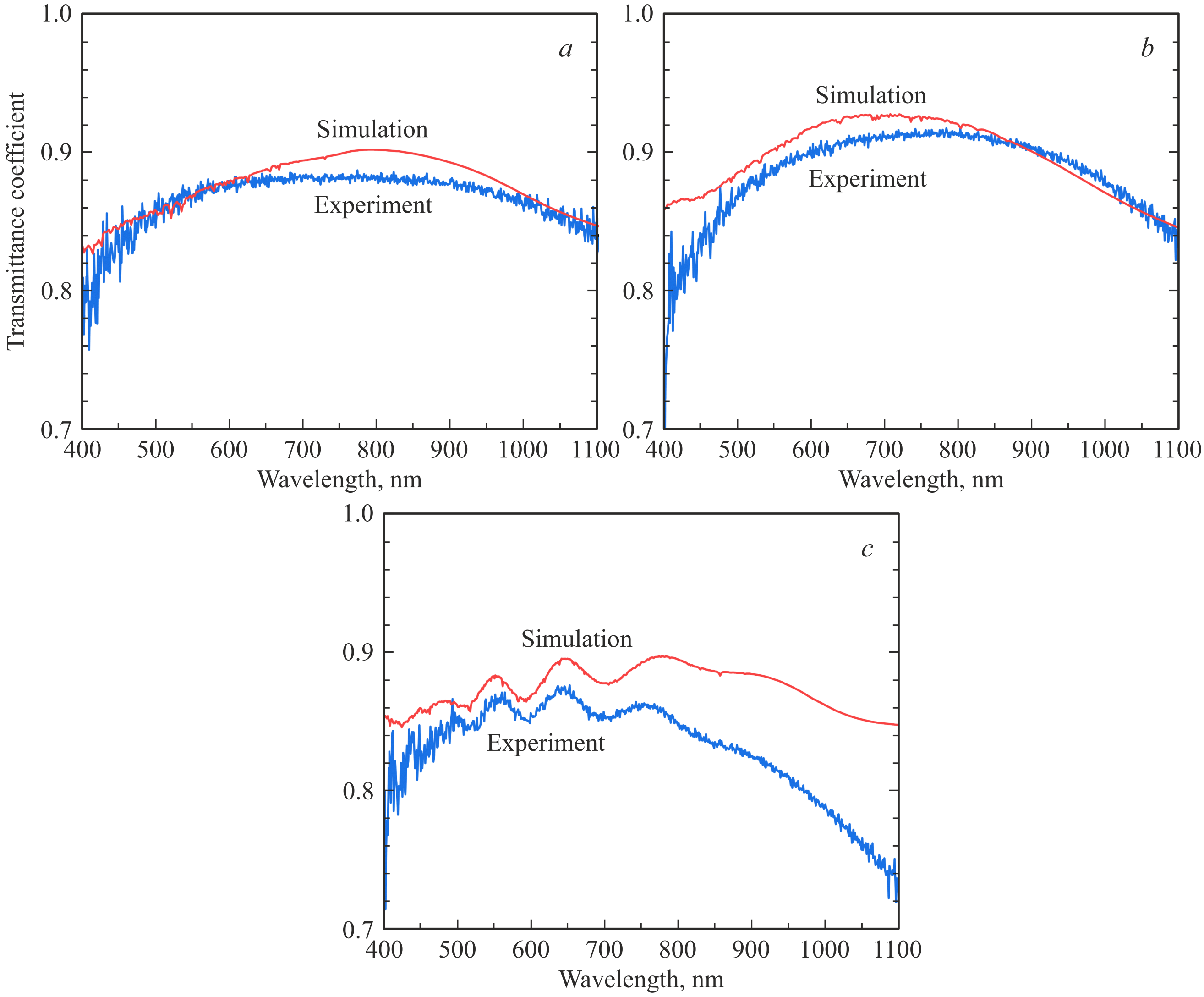

Figure 2. Experimentally obtained and calculated transmission spectra of ITO films with different degrees of filling with epoxy resin: without resin (*a*), intermediate filling (*b*), completely filled (*c*).

function using the nonlinear Newton method and the direct solver PARDISO.

3. Experimental results and discussion

Figure 1 shows SEM images of a film with different degrees of filling with epoxy resin. As can be seen from the images the resin fills the film uniformly without any voids. At the same time, it does not encapsulate all of its nanocrystals in a thin layer, as happens when forming a protective layer by atomic layer deposition, but gradually fills it from the bottom up until it covers the entire film. The morphology of a film completely filled with resin also differs from the morphology of a film coated with a thick layer of aluminum oxide obtained by ALD method. In the latter case, as discussed above, dome-shaped structures of a near micron size are observed.

The transmission spectra obtained in the experiment for samples with different degrees of film filling with resin are shown in Figure 2. Comparing the data in Figure 2, *a* (for the original film) with the data in Figure 2, *b* (for the film with intermediate filling with epoxy resin), it can be concluded that initially, when a small amount of epoxy resin is applied, an antireflection effect is observed, most likely associated with the smoothing of the profile of the effective refractive index of the initial film. The fact is that the examined structured ITO films consist of a dense sublayer, providing lateral conductivity, from which whisker-like crystals are drawn (Figure 1). At the transition from the sublayer to the layer consisting of nanocrystals, a sharp drop in the effective refractive index should be observed. Therefore, filling this transition with an epoxy resin layer with a refractive index lower than that of the sublayer smooths out the jump in refractive index in the film, which reduces the reflection of transmitted light.

Subsequently, as the voids in the film are filled with resin, the positive effect of smoothing the refractive index profile is neutralized, and when the film is completely filled with resin (Figure 2, *c*), interference extremes are observed in the transmission spectrum of the sample, which is because a fairly significant reflection occurs at the outer boundary of the coating.

The modeling of the studied samples was carried out as follows. The geometry of the structure used in modeling is shown in Figure 3. Within this model, the composite coating is represented as two-dimensional layers corresponding to the different layers of the coating: substrate, dense ITO sublayer, and epoxy resin. The nanostructured coating is shown as a set of triangular peaks, the sizes of which and the distance between them are determined by a random distribution. The optical parameters of ITO are taken from [20], parameters of glass Schott N-BK7 — were taken from Comsol Multiphysics on the basis of the manufacturer's specification (SCHOTT Zemax catalog).

In constructing the model, we were guided by the approach described in detail in [21]. The calculations used a plane wave propagating along the Z axis, simulating normal incidence of light. To record the spectral data of transmission and reflection, two frequency-dependent monitors were placed in the substrate and above the plane wave source, respectively. At the boundaries in the direction of the axis Z, perfectly matched layers (PMLs) were used to absorb both propagating and evanescent waves with minimal reflections from the boundaries of the computational domain. In direction of X axis, periodic Floquet boundary conditions (which establish a phase shift of the electromagnetic wave at the opposite boundaries of the computational domain) were used, which made it possible to reduce memory requirements and limit the size of the computational domain to 2000–2500 nm. In order to avoid the appearance of features in transmission/reflection spectra associated with a specific implementation of the location and heights of the peaks, the results were averaged over a set of 64 different random implementations. The maximum grid element size was limited to 1/8 of the smallest wavelength of the selected wavelength range, divided by the refractive index in the corresponding region.

According to the scanning electron microscopy data, the thickness of the dense sublayer of ITO film in the model was 50 nm, the height of the nanowires varied according to a random uniform distribution in the range of 200–1300 nm, and the base diameter was in the range of 10–50 nm.

The calculated transmission spectrum of the sample without epoxy resin is shown in Figure 2, *a*. Subsequently, a layer of epoxy resin was added to the obtained and verified model. A graphical representation of the model used is shown in Figure 3, *a*. The distribution of the electric field strength in a film completely filled with epoxy resin obtained as a result of the simulation is shown in Figure 3, *b*.

Since a gradient in the epoxy resin layer thickness was observed in experimental conditions, samples with epoxy resin were analyzed in order to determine the model epoxy resin layer thickness that would achieve the best agreement between the experimental and calculated data. Thus, the best fit for the sample with a small amount of epoxy resin (Figure 2, *b*) was achieved during optimization by parameters with averaging over the thickness of the epoxy layer in the range of 75–175 nm, and for the sample with complete filling with epoxy resin (Figure 2, *c*) — in the range of 900–1200 nm.

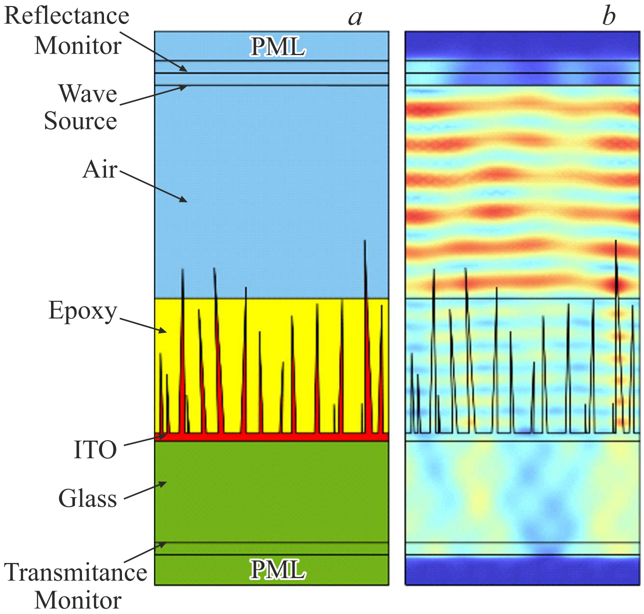

Figure 3. A diagram of the geometry of the computational domain indicating the layers corresponding to the substrate, sublayer and ITO nanowires, epoxy resin and air (*a*), and an example of the distribution of the electric field strength arising in this geometric configuration at an incident wavelength of 470 nm (*b*).

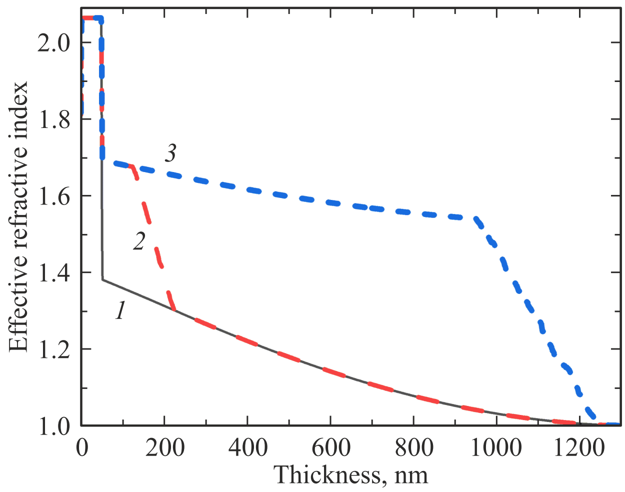

Figure 4. The dependence of the effective refractive index on the height obtained in the model for a wavelength of 470 nm for ITO films with different degrees of filling with epoxy resin: without resin (*1*), intermediate filling (*2*), completely filled (*3*).

Figure 4 shows the calculated profiles of the effective refractive index of the coatings obtained from the results of modeling ITO films with the spectra shown in Figure 2. Although gradual filling with epoxy resin leads to a decrease in the antireflective properties of nanostructured ITO films, the nanowires of this material, having a higher refractive index than epoxy resin, provide a refractive index gradient even when the coating is completely filled with resin. At the same time, the coating is provided with additional mechanical strength and chemical protection.

4. Conclusion

Thus, in this work, the nature of filling of nanostructured ITO films with epoxy resin was investigated, and the transmission and reflection spectra of the obtained samples with different degrees of filling were measured and modeled. It is worth noting that the morphology of the resulting coating differs from the morphology of ITO−Al_2O_3 coating at both the initial and final stages of filling the films with protective material. The epoxy resin initially provides additional light transmission to the film, but with further filling, light transmission is reduced due to a change in the effective refractive index profile of the coating. However, the obtained calculated effective refractive index profiles reveal a refractive index gradient even in a fully filled coating, which should provide such coatings with an advantage in light extraction compared to flat ITO−epoxy resin films. At the same time, such coatings are given additional mechanical strength and chemical protection.

The basic principles of modeling developed and verified within this study enable model prediction of the properties of complex, nanostructured multicomponent media to fabricate the coatings with specified optical properties.

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Conflict of interest

The authors declare that they have no conflict of interest.

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