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Conduction band electronic states of ultrathin thiophene-phenylene co-oligomer and substituted biphenyl films on the surface of layer-by-layer grown ZnO

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The results of studying the electronic states of the conduction band and interface potential barrier during the formation of ultrathin films of thiophene-phenylene co-oligomer CH_3-phenylene-thiophene-thiophene-phenylene -CH_3(CH_3-PTTP-CH_3) on the surface of ZnO and films of biphenyl tetracarboxylic dianhydride (BPDA) on the ZnO surface are presented. A 100 nm thick ZnO layer was prepared by atomic layer deposition (ALD). Organic CH_3-PTTP-CH_3 films and BPDA films up to 8 nm thick were formed by thermal vacuum deposition. During film deposition, the electronic characteristics of the surface were studied using total current spectroscopy (TCS) in the energy range from 5 eV to 20 eV above EF. In this energy range, the structure of the maxima of the unoccupied electronic states of CH_3-PTTP-CH_3 and BPDA films was determined. As a result of the CH_3-PTTP-CH_3 film deposition, a decrease in the work function to 4.0 eV was found, compared with the value of the work function of 4.2 eV measured from the ALD ZnO-substrate. This corresponds to the transfer of a negative charge from the CH_3-PTTP-CH_3 film to the substrate. The charge transfer at the interface between the BPDA film and the ALD ZnO-substrate occurs in the opposite direction, since a 4.7 eV increase of the work function was registered during the formation of this interface. The CH_3-PTTP-CH_3 and BPDA films studied and the layer-by-layer grown ZnO film represent a continuous coating on sufficiently large surface areas of the order of $10\,\mu m \times 10\,\mu m$. The roughness of the ZnO surface does not exceed 4 nm, and the surface roughness of CH_3-PTTP-CH_3 and BPDA films was $10-15$ nm.

Keywords: thiophene-phenylene co-oligomers, biphenyl tetracarboxylic dianhydride, ultrathin films, ZnO, atomic layer deposition method, electronic properties, low-energy electron spectroscopy, interface potential barrier.

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1. Introduction

The electronic properties of organic films based on small conjugated organic molecules are of considerable interest in terms of the development of active elements of organic electronics devices [1–5]. Layouts of organic field-effect transistors (OFET) [6–9] were constructed on the basis of thin and ultrathin films of thiophene-phenylene cooligomers (TPCO), and the mobility values of charge carriers of the order of $10^{-1}\,cm^2/(V \cdot s)$ were demonstrated [9]. It has been shown that by attaching electroactive functional groups to TPCO molecules, it is possible to form TPCO *n*- and *p*-films of the [6,7] conductivity type. Using the technique of ultraviolet photoelectron spectroscopy (UPS), the values of the output of some types of TPCO films were determined, the location of characteristic maxima in the valence energy zone [7.10] was established. One of the methods of optimizing the characteristics of organic electronics devices is the use of hybrid (two-component) materials, or the use of several layers of materials based on different molecules [3,11,12]. Films based on molecules of substituted perylene and naphthalenes, such as perylene-tetracarbon dianhydride and naphthalene-tetracarbon dianhydride [13–15], have proven themselves as an electron-acceptor componentas part of the composite. For films of these molecules, it was shown that it is the introduction of functional groups of carboxylic acid anhydride that leads to a decrease in the position of energy levels corresponding to the energy conduction band in these materials [16,17]. Boundary structures based on semiconductor organic films and binary inorganic semiconductors, for example, ZnO, are of interest in terms of use in transistor and photovoltaic

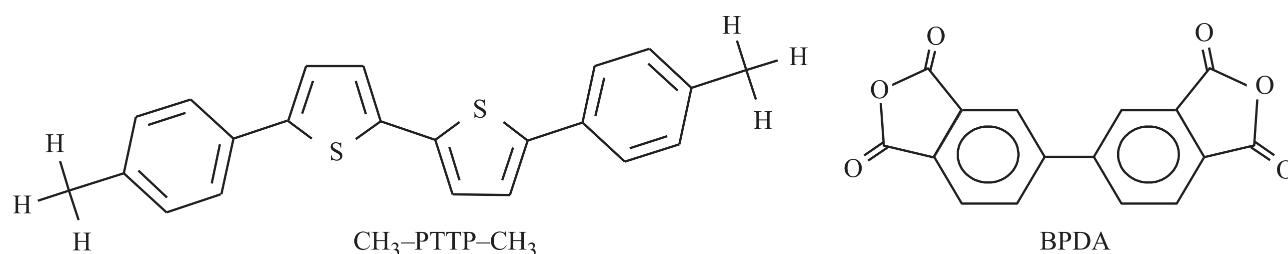

Figure 1. Structural formula of thiophene-phenylene cooligomer molecules 5.5′-bis[4-methylphenyl]-2.2′-bitiophene (CH_3-PTTP-CH_3) and tetracarboxyl of biphenyl dianhydide (Biphenyltetracarboxylic dianhydride, BPDA).

devices [18–21]. One of the well-controlled methods for the formation of (2D) inorganic semiconductors is the method of molecular layering (atomic layer deposition, ALD) [21–23]. With the help of ALD, solid ZnO coatings of the initial silicon substrate with a thickness from several nm to several hundred nm can be formed.

Earlier, we investigated the electronic states of the conduction band of films based on bismethyl-substituted and bis-trifluoromethyl-substituted TFSO molecules on the surface of oxidized silicon and polycrystalline gold [24–26]. Studies of films of substituted pyridines and naphthalenes with functional groups of carboxylic acid anhydride [27,28] have been carried out. The measurements were carried out by total current spectroscopy (TCS) method, and it was shown that the introduction of fluorine-containing functional groups and carboxylic acid dianhydride groups into the composition of molecules leads to a shift in the density maxima of unfilled electronic states (DOUS) towards lower electron energies and to an increase in the electronic output (work function) of organic material. The results obtained by the TCS method correspond well to the results obtained using other techniques, such as X-ray absorption edge spectroscopy (NEXAFS) and electron capture spectroscopy [29,30]. This article presents the results of studies of the electronic properties of structures based on films of dimethyl-substituted thiophene-phenylene co-oligomers CH_3-phenylene-thiophene-thiophene-phenylene -CH_3(CH_3-PTTP-CH_3) on the surface of ZnO and films of tetracarboxylic biphenyl dianhydide (Biphenyltetracarboxylic dianhydride, BPDA) on the surface of ZnO. The ZnO substrates were prepared in the course of work by the ALD method. The results are given of the study by the TCS-structure method of the unfilled electronic states of the conduction band and the characteristics of the boundary potential barrier between organic films and the ZnO substrate. The results of the study of the surface topography of the studied films by atomic force microscopy (AFM) are presented.

2. Experiment

The substrate used for the application of organic material was the surface of a ZnO semiconductor prepared by the atomic layer deposition (ALD) method. Diethylcinc reagents and deionized water were used for synthesis. The temperature during synthesis was maintained in the range of 200−250°C and layers of O and Zn atoms were alternately deposited on the surface of a silicon substrate [31]. Taking into account the applied number of layers, the estimate thickness of ZnO films was 100 nm. Earlier, by the X-ray photoelectron spectroscopy method, we established the predominance of the O atoms content at 5−10%, in the composition of the formed ZnO layers, compared to the content of Zn atoms [23]. Using the X-ray diffraction method, it was found that the structure of the formed ZnO coatings corresponds to the structure of the Wurtzite type, and the surface roughness of the substrate does not exceed 4 nm [23]. The ZnO coatings prepared by the ALD method have a bandgap of 3.4 eV, i.e., typical for these semiconductor materials [21]. For the preparation of thiophene-phenolic oligomer films CH_3-HTTP-CH_3 (Fig. 1) was used, which was synthesized according to the previously described method [2]. Tetracarboxylic biphenyl dianhydride (3, 3′, 4, 4′-Biphenyltetracarboxylic dianhydride, BPDA) (Fig. 1) was purchased from Sigma-Aldrich. Decontamination of materials was carried out by holding in vacuum at a base pressure of 10^{-6} Pa and a temperature of 100°C for a period of 2−3 h. Thermal vacuum deposition of CH_3-HTTP-CH_3 coatings and BPDA coatings up to 10 nm thick on a ZnO substrate was carried out at a rate of 0.1 nm/minute. During the deposition of films, the pressure in the vacuum chamber was allowed to increase by an order of magnitude from the base pressure (10^{-6} Pa). The films formed in this way CH_3-PPTP-CH_3 demonstrate reflexes during X-ray diffraction measurements corresponding to periods 3.8 and 1.9 nm [26]. Therefore, the authors believe that the films CH_3-HTTP-CH_3 have a polycrystalline structure.

Measurements of the electronic properties of the films were carried out by total current spectroscopy (TCS) [32,33] for a series of sequentially deposited films of each type with an increase in the total thickness of the organic coating to 8−10 nm. In the TCS experiment, an incident electron beam with a cross-sectional area of 0.2−0.4 mm^2 was directed along the normal to the surface under study and the energy derivative of the total current $S(E)$ passing through the sample was recorded using a lock-in amplifier. The energy of electrons in the testing beam was varied in a range from

0 to 25 eV relative to the vacuum level (E_{vac}) of the surface under study. When the energy of the incident beam and E_{vac} of the surface coincide, the primary maximum of TCS is recorded. Taking into account the calibration of the TCS instrument on a known surface, for example, on the surface of highly Oriented pyrolytic graphite (HOPG — Highly Oriented Pyrolytic Graphite), it is possible to establish the energy location of E_{vac} relative to the Fermi level of the system (E_F). Well-reproducible values of the electronic output operation of about 4.6 eV [34] are characteristic of the HPP surface of the HOPG. When the beam energy exceeds the value of E_{vac} of the surface, a fine structure of the total current spectra (FSTCS) is formed. The FSTCS carries information about the location of the features of the Density of Unoccupied Electronic States (DOUS) of the studied material [25,33,35]. The topography of the ZnO coating surface and the surfaces of the studied films CH_3-PPTP-CH_3 and BPDA were studied using the AFM technique in a semi-contact mode on an Integra Spectra microscope (Spectrum Instruments Ltd.).

3. Results and discussion

The structure of unfilled electronic states of the conduction band during thermal deposition of an organic film on an ALD ZnO substrate was studied experimentally by the TCS method. The energy range for recording the fine structure of the total current spectra (FSTCS) is usually in the range from 5 to 25 eV above E_F. The FSTCS maxima are caused by an increase in the intensity of the entrance of incident electrons into the studied surface, which should be associated with an increase in the density of the allowed electronic states at a given energy. The FSTCS series measured during the deposition of CH_3-HTTP-CH_3 films on the AL ZnO surface is shown in Fig. 2. When the thickness of the organic coating is 0 nm, that is, before the application of the organic film, the FSTCS of the investigated ZnO substrate was measured. TSTCS of ZnO demonstrates several clearly distinguished maxima, including those at energies of 8.0, 10.5, and 16.0 eV. We have observed such a FSTCS before when studying the surface of AL ZnO [23].

As the film of the cooligomer CH_3-HTTP-CH_3 with a thickness of up to 8 nm FSTCS of the substrate faded, and at the same time the intensity of new FSTCS maxima increased (Fig. 2). The FSTCS of the surface with characteristic maxima H_1–H_6 was formed at a film thickness of CH_3-PPTP-CH_3 5–8 nm. The maxima of H_1–H_6 are characteristic of FSTCS films CH_3-HTTP-CH_3 and on the surface of other substrates, oxidized silicon, polycrystalline gold [24,26]. During the deposition of the BPDA film, the FSTCS ALD of the ZnO substrate faded and the FSTCS increased with maxima B_1–B_4 (Fig. 3). To analyze the rate of increase of the FSTCS of the deposited coating with an increase in the coating thickness in the case of a film CH_3-PTTP-CH_3, it is convenient to choose a maximum of H_2 at an energy of 6.0 eV. The maxima of H_2 and H_4 are

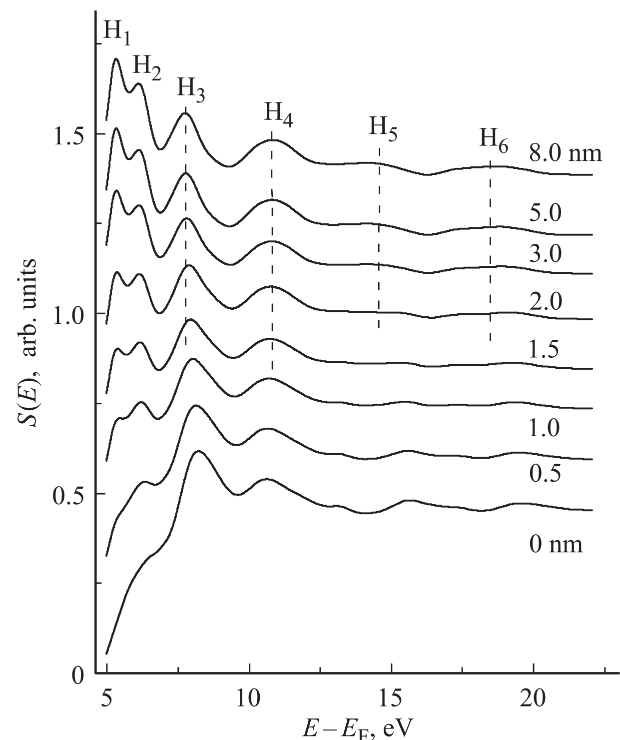

Figure 2. FSTCS in the process of deposition of films CH_3-PTTP-CH_3 onto the ZnO surface formed by the ALD method. Captions near curves specify the corresponding thickness of organic coating from 0 to 8 nm. The most distinct maxima of H_1–H_6 in the FSTCS of films CH_3-HTTP-CH_3 are marked. Vertical dotted lines are shown for convenience of maxima comparison.

also quite clearly expressed (Fig. 2), but they are located at energies close in value to the energies of the location of sufficiently intense maxima of the ZnO substrate. In the case of a BPDA film, it is convenient to choose a maximum of B_1 for the analysis of the growth of FSTCS (Fig. 3). For electrons with energy 6 eV above the level E_F free path length (λ) is approximately 4 nm [36]. To analyze the attenuation of the FSTCS substrate, it is convenient to choose the difference in the intensities of the FSTCS maximum at 10.5 eV and minimum at 14 eV. With an electron energy of 11–14 eV above the level of E_F — is approximately 3 nm [36]. As discussed in detail in the works [33,36], we should expect an exponential increase in the FSTCS intensities of the deposited coating and the attenuation of the features of the FSTCS substrate with a change in the thickness of the coating (d) in the case of the formation of a solid organic coating. The exponent of this exponential dependence is given by the expression $2d/\lambda$ [36]. As a result of the analysis, the exponential nature of the increase in the intensities of the FSTCS maxima CH_3-PTTP-CH_3 and BPDA was established. The appearance of the FSTCS maxima characteristic of these films was detected at an early stage of deposition, when the thickness of the deposited layer was 0.5nm.

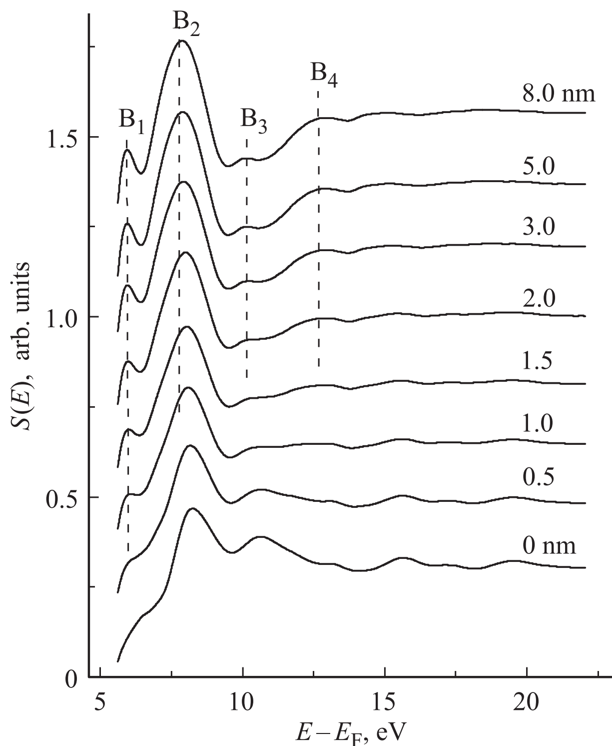

Figure 3. FSTCS in the process of deposition of BPDA films on the ZnO surface formed by the ALD method. Captions near curves specify the corresponding thickness of organic coating from 0 to 8 nm. The most distinct maxima of B_1-B_4 in FSTCS of BPDA films are marked. Vertical dotted lines are shown for convenience of maxima comparison.

By measuring the energy position of the primary TCS maximum during the deposition of the organic layer, the changes in the value of E_{vac} of the studied surface relative to E_F were analyzed. The values of $E_{vac}-E_F$, i.e. the output of the ZnO substrate surface was 4.2±0.1 eV. This corresponds well to the results of studies of the ALD ZnO surface, but is significantly lower than the value of the output of the surface of the single crystal ZnO (000-1) [23,37]. As a result of the deposition of the film CH_3-PTTP-CH_3, a decrease in the output operation to 4.0 eV was detected (Fig. 4, *a*). The same value $E_{vac}-E_F$ was established by us earlier when studying films of CH_3-PTTP-CH_3 on the surface $(SiO_2)n-$Si [24]. Taking into account the literature data, it should be assumed that in the absence of interaction at the substrate/organic film interface, the docking of energy levels and the E_{vac} level occurs in a fairly narrow layer, with a thickness of 1−3 nm [38–40], and upon reaching of such a thickness of the organic coating, the values of the output work do not depend on the substrate material. At the same time, the role of the substrate can become decisive in the formation of a boundary barrier in the case of the formation of boundary electronic states or the diffusion of substrate atoms into an organic film [40–42]. Thus, when studying films of CH_3-PTTP-CH_3 on the surface of polycrystalline gold, the values of the output of these films exceeded 4.6 eV [26]. A decrease in the value of $E_{vac}-E_F$ during the deposition of CH_3-PPTP-CH_3 on an ALD ZnO substrate corresponds to the transfer of a negative charge from an organic film into the substrate. The charge transfer at the boundary of the BPDA and ALD ZnO film of the substrate occurs in the opposite direction, since during the formation of this boundary barrier, an increase in the output operation to 4.7 eV was registered (Fig. 4, *b*). It should be assumed that, similarly to well-known organic materials based on perylene-tetracarbon dianhydride and naphthalene-tetracarbon dianhydride [13–15], BPDA films have sufficiently high values of electron affinity. Therefore, in the case of BPDA films, the values of $E_{val}-E_F$ exceed similar characteristics of films of bis-methyl substituted TPCO and the studied surfaces of ALD ZnO and oxidized silicon.

The results of diagnostics of the topography of the studied surfaces: the surfaces of the ALD ZnO substrate, the surfaces of the films CH_3-PTTP-CH_3 and BPDA are shown in Fig. 5. All the materials studied are a continuous coating on sufficiently large areas of the surface of the order of 10, μm×10 μm. The studied surfaces have a granular structure with varying grain sizes and varying degrees of roughness. Thus, the characteristic grain size of the ALD ZnO coating in the surface plane lies within 50−200 nm, and the characteristic height difference does not exceed 4 nm (Fig. 5, *a*). The established roughness of the ALD ZnO layer corresponds to the results of our studies of this material by X-ray diffraction [23]. The film CH_3-PTTP-CH_3 is formed by grains with a size of 200 nm or more in the surface plane, and the height difference reaches 15 nm (Fig. 5, *b*). The BPDA film is formed by grains, 50−100 nm in size in the surface plane, and the height difference reaches 10 nm (Fig. 5, *c*). The formation of continuous films of

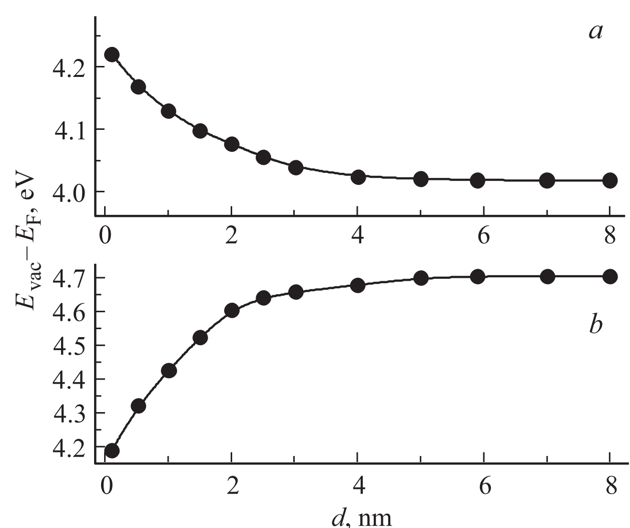

Figure 4. Analysis of the energy position of the primary TCS maximum as the thickness of the organic coating increases. Changing the position of the vacuum level E_{vac} relative to E_F during thermal deposition of the film (*a*) CH_3-PTTP-CH_3 and (*b*) BPDA.

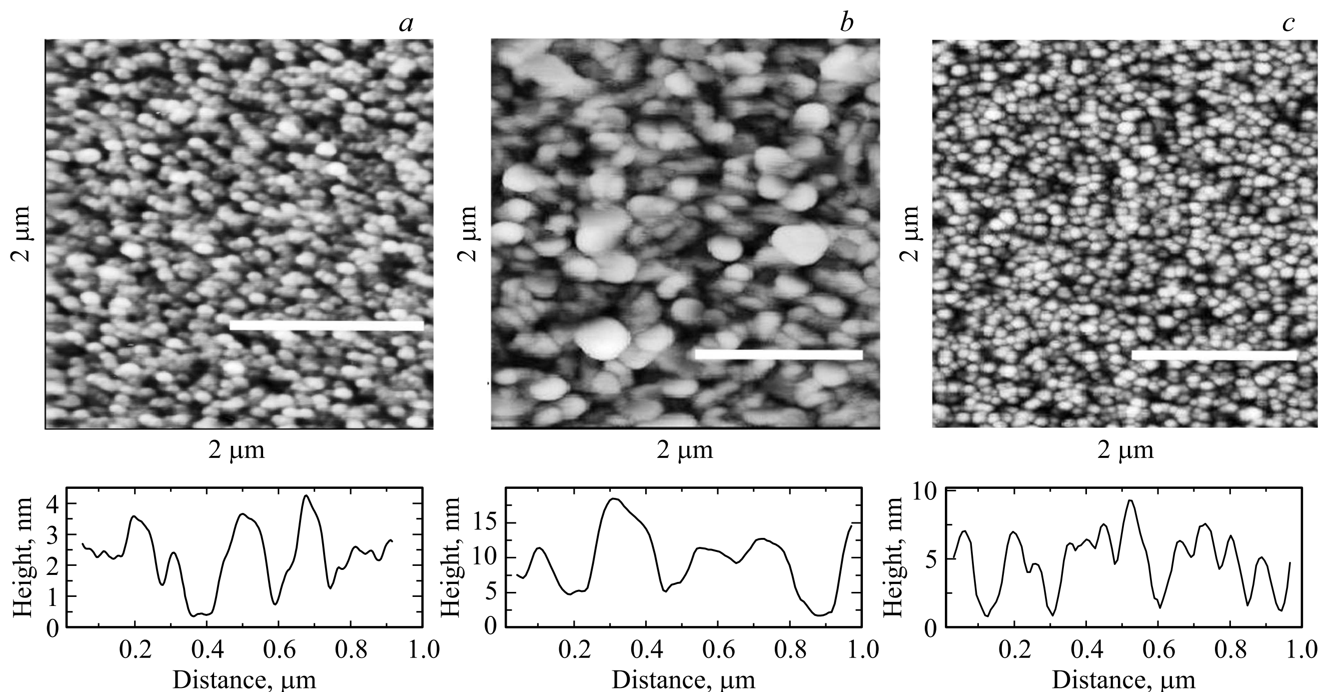

Figure 5. (*a*) $2\,\mu$m$\times 2\,\mu$m AFM-image of the surface area of the ZnO layer, prepared by the ALD method. Gradations of gray color from black to white correspond to the height difference from 0 nm to 20 nm. The profile of the surface area on the segment marked with a white horizontal bar is shown below. (*b*) $2\,\mu$m$\times 2\,\mu$m AFM-image of the surface area of the film CH_3-PTTP-CH_3 on the surface of ALD ZnO. Gradations of gray color from black to white correspond to the height difference from 0 to 40 nm. The profile of the surface area on the segment marked with a white horizontal bar is shown below. (*c*) $2\,\mu$m$\times 2\,\mu$m AFM is an image of a section of the BPDA film surface on the ALD ZnO surface. Gradations of gray color from black to white correspond to the height difference from 0 to 30 nm. The profile of the surface area on the segment marked with a white horizontal bar is shown below.

CH_3-PTTP-CH_3 and BPDA makes it possible to analyze changes in the intensity of the FSTCS maxima, assuming an exponential increase in the intensity of the maxima of the deposited layer and exponential attenuation of the substrate signal [33,36].

4. Conclusion

By the method of thermal vacuum deposition, CH_3-PTTP-CH_3 films and BPDA films with a thickness of 8−10 nm were formed on the surface of layer-by-layer grown ZnO. By the TCS method, as the thickness of the organic film increases, the structure of the maxima of the unfilled electronic states of the films CH_3-PTTP-CH_3 and BPDA in the conduction band in the energy range from 5 to 20 eV above E_F. As a result of the deposition of the film CH_3-PTTP-CH_3, a decrease in the output operation to 4.0 eV was found, compared with the value of the output operation 4.2 eV, measured for ALD ZnO substrate. This corresponds to the transfer of a negative charge from the film CH_3-PTTP-CH_3 to the substrate. Charge transfer at the boundary of the BPDA and ALD ZnO film of the substrate occurs in the opposite direction, since during the formation of this boundary barrier, an increase in the output operation to 4.7 eV was registered. The studied films CH_3-PTTP-CH_3 and BPDA and layer-by-layer grown ZnO represent a continuous coating on sufficiently large surface areas of the order of $10\,\mu$m$\times 10\,\mu$m. The roughness of the ZnO surface does not exceed 4 nm, and the roughness of the surfaces of the films CH_3-PTTP-CH_3 and BPDA is 10−15 nm.

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Conflict of interest

The authors declare that they have no conflict of interest.

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