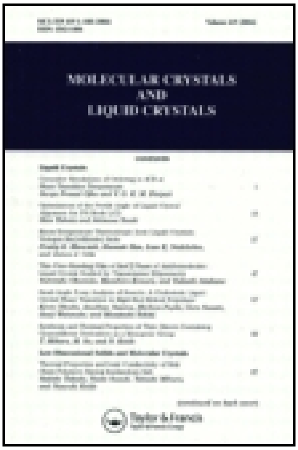

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Photoelectrical Properties of Interfaces Between Organic Films and CdSSe

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Photoelectrical Properties of Interfaces Between Organic Films and CdSSe

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The paper reviews author's own results obtained on heterostructures (HS) of CdSSe with organic photosensitive films, the attention being given to processes of carrier recombination, to the formation of potential barriers near the interface, and to the effect of oxygen. Photochemical processes near top electrodes are discussed. Experimental results characterizing parameters of barriers in such HS are compared with the energy diagram of ionized states and the Anderson model. It is shown that HS can be successfully used for construction of improved organic gas sensors.

Keywords: interface; organic photoconductor; potential barrier; photovoltaic properties; solar cell; molecular gas sensor

1. INTRODUCTION

Early studies of processes in heterostructures (HS) composed of CdS and organic photoconductors were carried out by R. Nelson [1], and by H. Meier and W. Albrecht [2]. It was reported in those papers that open-circuit photovoltage (V_{oc}) increased by 150–200 mV on illuminating HS fabricated of CdS films and merocyanines or phthalocyanines (e.g., CuPc) with non-monochromatic light. It was also shown that the appearance of V_{oc}in such HS is not due to ion motion, by photochemical reactions or by sensibilization effects: electronic processes dominate in these structures and the sign of V_{oc} is defined by the type of conductivity of components as is predicted by the theory of p-n junctions. Moreover, for merocyanine films a possibility of their doping by iodine was shown, resulting in a 50% increase in V_{oc} and short-circuit photocurrent I_{sc} [2]. Since I_{sc} was 3×10^{-8} A/cm^2, the efficiency of such solar cells can be estimated as $\eta < 10^{-8}\%$. The processes of charge carrier recombination were not studied in those papers, nor

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were possible advantages of the above structures is comparison with similar HS consisting of two inorganic semiconductors, nor stability of such cells at normal conditions because of the rapid degradation of their parameters in air[3].

The aims of investigations reviewed in this paper were the following:

1. A study of oxygen effect on photoelectrical properties of the heterostructures and of a possibility of O_2 doping of HS components [4];
2. An investigation of recombination processes near the surfaces of the components and at the HS interfaces [5,6];
3. A search for novel photosensitive organic compounds for HS [5,7];
4. A determination the optimal thickness of solar cell components [6];
5. Minimization of degradation processes (photochemical reactions) in HS [8].

2. OXYGEN EFFECT AND METHODS OF COMPONENTS DOPING

The main reason of small I_{sc} of the above HS is great intrinsic resistance due to low conductivity (σ) and low photosensitivity of both components. It was thus necessary to find simple and effective methods of their doping. O_2molecules in organic layers of p-type create centers of photogeneration of holes, making possible doping of the samples with oxygen by the simplest quenching technique at 350K in air [4]. It was found that such a quenching results after 2–3 hours in an increase of σ of CuPc films by 4 orders, leading to an increase of I_{sc} from 6×10^{-9} to $(6–8)\times10^{-6}$A/cm^2. A similar effect was observed for HS stored in air during 3 days under normal conditions. A further increase of quenching time was ineffective or even led to a decrease in I_{sc}. Since the increase in I_{sc} was smaller than the increase in σ of the CuPc film, I_{sc} of the HS was already limited by thickness of CdS layers and their small σ after such quenching. Moreover, the presence of a dramatic drop of HS photosensitivity in the 2.2–2.5eV range [6] points to a high surface recombination rate of nonequilibrium charge carriers (Sr) in CdS layers. Looking for a way to broaden the spectral region of sensitivity, we substituted the CdS layers by CdS_xSe_{1-x} with x=0.6 (hereafter referred to as CdSSe) in further works. We developed the technology of preparing CdSSe layers exhibiting a high ratio of dark resistance (ρ_d) to the resistance under illumination (ρ_l) [4]. This technology allowed us to obtain CdSSe layers with minimized specific resistance under solar illumination (SI) and Sr.

We also studied the influence of the quenching in presence of different ambients on ρ_l and Sr of CdSSe layers [9]. The films were prepared by vacuum deposition of CdSSe onto SnO_2- coated glass substrates at temperature T_s= 570K. The films of stable tetragonal orientation of crystallites were formed in such a way, the angle of the *c*-axis of crystallites deviating from the normal to the substrate by less than 2^0.

The structures obtained were typically 1μm thick, with In or Al used as top electrodes, and had the following characteristics: $\rho_l = 16$ Ohm cm; $(\rho_d/\rho_l) = 4$; Sr $\geq 10^4$ cm/sec. The films were quenched in air or in $CdCl_2$ and $CdCl_2$+$CuSO_4$vapors to minimize the ρ_l in Sr[9]. The lowest values of ρ_l (and the maximum of the ρ_d/ρ_l ratio) were obtained upon quenching the films during 1 hour at 773K. Results obtained for various methods of quenching are given in Table I, from which it may be seen that only quenching in $CdCl_2$+$CuSO_4$vapors leads to the desired decrease of ρ_l in Sr. The quenching of CdSSe layers in air leads to an increase of ρ_l and Sr, resulting in adecrease in I_{sc} and in a strong drop of the photosensitivity in the short wavelength region [4]. Therefore we used mainly CdSSe layers, quenched in $CdCl_2$ + $CuSO_4$ vapors.

TABLE I The influence of thermoactivation atmosphere on surface resistance and surface recombination rate of CdSSe layers

Ambient	ρ_d *Ohm cm*	ρ_l *Ohm cm*		*Sr cm/sec*	
		Illumination through SnO_2	*Free surface illumination*	*Illumination through SnO_2*	*Free surface illumination*
Before quenching	64	16	14	2×10^4	1.2×10^4
In air	64×10^6	58×10^4	38×10^4	4×10^4	8×10^4
In $CdCl_2$ vapors	54×10^5	85	81	6×10^3	3×10^3
In $CdCl_2$ $CuSO_4$ vapors	12×10^6	11.2	10.6	6×10^3	2×10^3

3. PROCESSES OF THE RECOMBINATION OF NON-EQUILIBRIUM CHARGE CARRIERS

The centers of trapping and recombination of charge carriers arise at interfaces of inorganic HS mainly due to the existence of dangling covalent bonds and diffusion of component atoms in the process of preparation [10,11]. The dangling covalent bonds are absent at the interface between the organic film (OF) and CdSSe, and the diffusion of component atoms is insignificant due to low temperatures of sublimation and the large size of organic molecules. All these factors result in a relatively low concentration of trapping and recombination centers at the CdSSe/organic film interfaces. The formation of barriers at the interface will therefore be controlled by parameters of the energy structure of ionized states of the components.

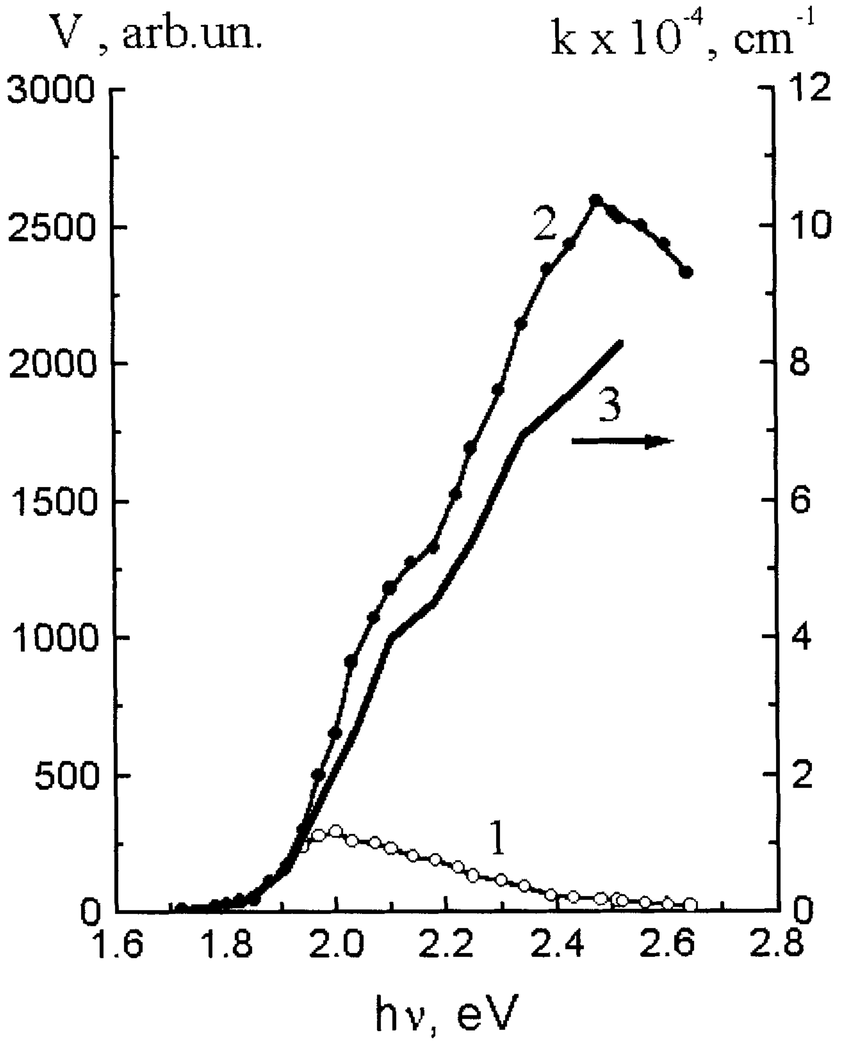

FIGURE 1 Spectral dependences of I_{sc} (excitation through CdSSe layer – curve 1), I_{sc}' (excitation through TTT layer – curve 2) of CdSSe/TTT HS, and the absorbance of CdSSe films (curve 3)

In order to determine Sr, measurements of spectral dependences of I_{sc} and V_{oc} were performed in CdSSe/pentacene (Pn), CdSSe/tetratiotetracene (TTT), and pthalocyanines (Pc): ClAlClPc, PbPc with and without a top CuI electrode, using the Bergman method. The measurements were performed for illumination from the CdSSe (SnO_2) side and from the organic layer side (through the top CuI electrode) [5,6]. The parameters measured under illumination of the organic layer will be hereafter marked with a dash. In all HS studied, the values V_{oc}', I_{sc}' and η' values were found greater than V_{oc}, I_{sc} and η, respectively, whereas Sr was smaller.

Figure 1 shows spectral dependences of V_{oc}' and V_{oc} measured in CdSSe/TTT HS. It becomes clear from the figure that $V_{oc}'(h\nu)$ (curve 2) follows the spectral dependence of the absorbance ($k(h\nu)$) of the CdSSe layer (curve 3). No decrease of V_{oc}' was observed in the strong absorption region of CdSSe. On the contrary, a correlation between $V_{oc}(h\nu)$ and $k(h\nu)$ was observed only at $h\nu<2.1$ eV for CdSSe illumination. For greater k and $h\nu$, V_{oc} decreases owing to greater Sr for CdSSe layer. The latter feature cannot be explained by the decrease of the number of non-intrinsic carriers injected into the space charge region (SCR) near the interface since the same decrease is observed in the spectral dependences of V_{oc}' and V_{oc} for structures SnO_2/CdSSe, for which at $h\nu$=2.4eV $V_{oc}'/V_{oc}< 2$, i.e. is significantly smaller than the respective value for a CdSSe/organic film HS.

These results show that, under illumination of HS from the TTT side, Sr′ is considerably smaller than for CdSSe/CuI structures. A similar trend was observed by us also for other HS (CdSSe with PbPc, ClAlClPc, Pn). This is caused by the fact that main processes of recombination take place in the bulk of CdSSe layers and not near the interface nor in the organic films. Thus the main advantage of an CdSSe/organic film HS is a value of Sr near the interface smaller than of that of HS composed of inorganic semiconductors (CdS/Cu_2S [13]).

4. THE EFFECT OF THICKNESSES OF PHOTOSENSITIVE COMPONENTS

The investigation of the dependence of HS parameters on thicknesses of the components was performed with SnO_2/CdSSe/Pn/CuI samples, fabricated from CdSSe layers quenched in $CdCl_2$+$CuSO_4$ vapors, and Pn films deposited at T_s=360K. The choice of these HS was caused by the absence of polymorphism in Pn and by a better homogeneity compared to phthalocyanines, as well as by a smaller diffusion depth of the top electrode atoms, which allowed us to perform the measurements over a wide range of thicknesses of the organic component. HS characteristics were measured under illumination of both the top and bottom electrodes.

Four series of HS samples were prepared in which only one parameter, viz. the thickness, was varied. The measurements were performed under irradiation of intensity weaker than AMO-2, using interference filters with 2.1 and 2.4 eV transition maxima, to diminish the influence of the intrinsic resistance of HS (1–2 kOhm) on the thickness dependences of V_{oc} and I_{sc}. The dependences of I_{sc}, V_{oc} and η on the thickness of CdSSe layer (d_1) measured on HS having the thickness of the Pn layer (d_2) equal or close to optimum (60 nm) are shown in Figure 2.

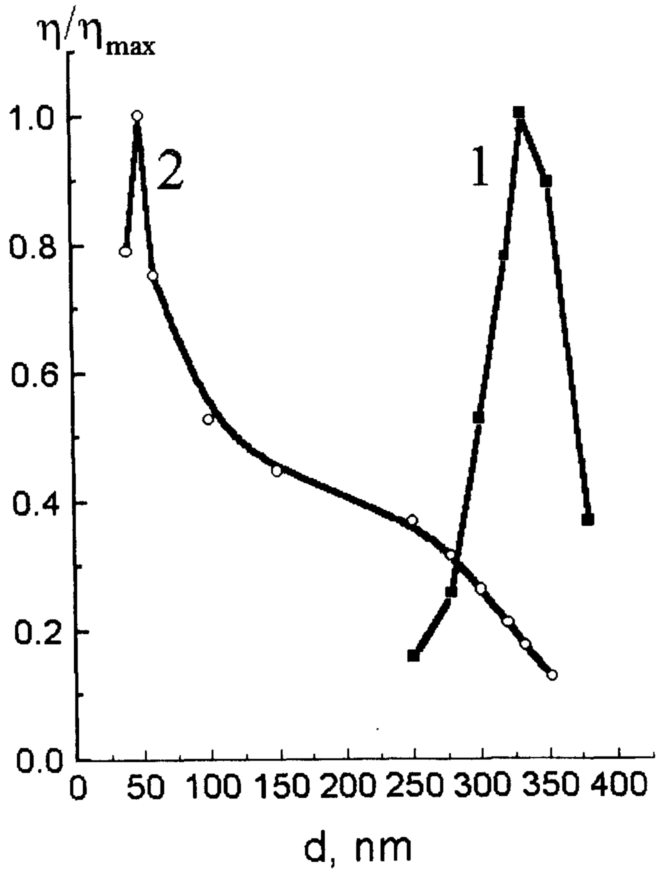

FIGURE 2 Dependences of the efficiency η on layer thickness of components for CdSSe/Pn HS. Curve 1 – CdSSe, curve 2- Pn

Maxima of the dependences of HS characteristics was observed for both regimes at d_1 = 310–350nm. The obtained optimal thickness was $d_1 < L_1 < L_1 + w_1$, where L_1 =200±100nm, and w_1 is the thickness of space charge region (SCR, $w_1 < 120$nm for CdS, CdSe layers [13]). The $\eta(d_2)$ dependence (curve 2) was found qualitatively similar to $\eta(d_1)$ one, but the maximum was in this case observed at d_2=50±10nm, which is smaller than the optimal d_1 value in spite of the fact that $L_2 \cong L_1$. Therefore the maximum on the $V_{oc}(d_2)$ dependence in the

$d_2 < L_2$ region cannot be explained by diffusion processes of excitons and charge carriers in Pn films near the interface, since the optimal d_2 value is close to the thickness of SCR in Pn. We explained the feature by an increase of the efficiency of charge carrier generation under the action of the internal electrical field in SCR. The processes of exciton and carrier diffusion in Pn give rise to a shoulder on the η (d_2) dependence at $d_2 \cong 250$nm. Similar dependences were obtained by us for other CdSSe/organic film HS.

In order to verify the obtained results, a series of CdSSe/Pn HS was fabricated with optimal d_1 and d_2. The efficiency measured for these HS, $\eta = 1{,}2\%$, exceeds the respective value determined for HS with unoptimized thicknesses of the components [7].

5. THE ENERGY STRUCTURE OF HS

Properties of HS are defined mainly by the energies of bands allowed for charge carriers (ionized states [12]) of contacted semiconductors, i.e. by the energy structure (ES) of HS [10]. A model of ES for an ideal HS is due to Anderson [10,11]. An ideal HS is a structure which is created by two contacted semiconductors, with the following conditions fulfilled: 1) the band model can be applied to them; 2) concentrations of interface states are insignificant; 3) tunnel effects do not play a significant role in the processes of charge carrier transport. The HS ES in this case is defined by electron affinity (A_c), ionization energy (I_c) and band gap (E_g), which have been determined for most inorganic semiconductors and for acenes [12]. The reliability of the Anderson model was confirmed for $A^{II}B^{VI}$- based HS, for example for ZnTe/CdSe [10,15].

The band model is widely used to explain photoelectrical properties of organic compounds which are sensitive in the visible and near infrared regions (Pn, TTT, Pc). The concentrations of surface states appear rather low; moreover, processes of diffusion of organic molecules in $A^{II}B^{VI}$ are slow owing to low sublimation temperature and large size of organic molecules. Therefore this model can be valid for HS from $A^{II}B^{VI}$ and photosensitive organic films. According to the model, a band bending occurs due to electron flow through the interface until an equilibrium is attained, i.e. until the energies of the Fermi levels (E_f) in both semiconductors become equal. This results in a band bending and creation of potential differences in the HS components (V_{d1} and V_{d2}; hereafter, parameters of the inorganic semiconductor will be labeled with the index "1"). The total contact difference of potentials is $V_D = V_{d1} + V_{d2}$ and is equal to the difference between the thermoelectric work functions of electrons or energies of Fermi levels of isolated semiconductors.

Except for particular cases, one should expect discontinuities of the energies of the valence band and the conduction band to appear at the interface: $\Delta E_v = I_{c1} - I_{c2}$, $\Delta E_c = A_{c1} - A_{c2}$. The structure of ES has been described in detail in [10], and the types of ES for studied HS have been summarized in this book.

TABLE II HS parameters

Parameter	*Illumination through*	*Organic material*			
		TTT	*Pn*	*PbPc*	*ClAlCPc*
V_b, V	--	0.42	0.6	0.7	0.8
J_{sc}, A/m^2	SnO_2	10	4	6.2	9.2
	CuI		4.9	10	11
V_{oc}, V	SnO_2	0.37	0.58	0.55	0.72
	CuI		0.59	0.60	0.73
η, %	SnO_2	0.15	0.11	0.18	0.35
	CuI		0.12	0.32	0.41
V_d (ΔE_f)		0.4	0.6		
I_c, eV		4.75	5.07	(4.97)	(5.14)
A_c, eV		2.7	2.87	3.58	3.65
E_g, eV		2.0	2.20	(1.39)	(1.6)
ΔE_c, eV		2.07	2.05	1.24	1.17
ΔE_v, eV		2.03	1.75	1.81	1.62

Table II lists the parameters of HS prepared on the basis of CdSSe films quenched in the $CdCl_2$+$CuSO_4$ vapors. The parameters of investigated CdSSe/organic film HS are in a satisfactory agreement with energy parameters obtained from the Anderson model. For example, the barrier height (V_b) is equal to V_d obtained from E_f difference. It should be noted that η decreases with increasing ΔE_c (or ΔE_v). ES of ideal CdSSe/Pn and CdS/Cu_2S structures are shown as an example in Figure 3.

6. REASONS FOR HS DEGRADATION

In spite of a considerable improvement of η of solar cells and HS based on CdSSe, the possibility of their practical application remains vague since processes of degradation in air have not been sufficiently well understood. It was found in[3]that organic solar cells are stable in vacuum but undergo an irreversible degradation in air. It was found in [16] that in solar cells based GaClPc,

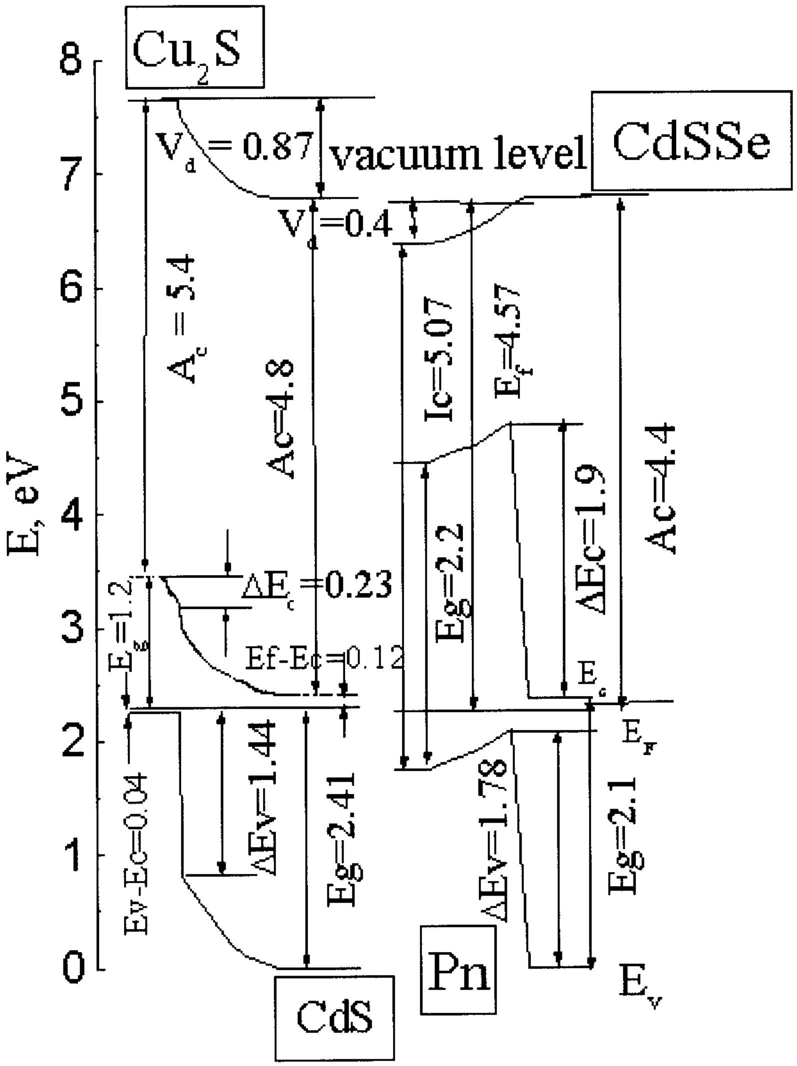

FIGURE 3 Energy diagrams of CdS/Cu_2S and CdSSe/Pn heterostructures

AlClPc and $(AlPc)_2O$, supplied with various top electrodes and periodically illuminated with a 30mW/cm^2 polychromatic light during 5 minutes, $V_{oc}$is does not change during 1 hour exposure of the samples air, whereas I_{sc}monotonically decreases and after 1 hour is equal to 10–20% of the initial value (I_{sco}). The degradation rate strongly depends on the top electrode material: for example, for GaClPc solar cells the rate increases in the order: Sb, Sn, Al+Mo, Al, i.e. the degradation is associated with the tendency to oxidation.

Three series of SnO_2/CdSSe/ClAlClPc/CuI HS were prepared to investigate the processes of degradation. In order to lower the degradation rate, HS of the third series were coated by a layer of hermetic (elastosyl 137–180), which effectively protects against drying and decreases the O_2 diffusion rate, being transparent in the visible and UV regions [8]. The first and third series of samples were stored in the darkness and HS were illuminated only during 5-minute cycles of measurements (photoexposure dose <20J/cm^2). The samples of the second series were stored under natural solar illumination. Since the intensity of solar light changes during the day, the measurements were always performed at the same hour (2p. m., when the intensity is at maximum). To minimize the influence of initial HS parameters on the degradation kinetics, the obtained V_{oc}' and I_{sc}' were normalized to their initial values and averaged. The dependences on the storage time t obtained by such a procedure are shown on Figure 4. It appears from the figure that V_{oc}' of HS stored in the darkness does not change during 1000 hours, whereas I_{sc}' decreases by 25±5%, i.e. it is smaller than the respective change observed during 1 hour in a Schottky solar [16]. A 15±5% increase of V_{oc} during 2 days without any further changes was observed in all coated HS (cf. Figure 4). I_{sc} reached its maximum (4.5 I_{sco}) after 6±2 hours and then decreased. However, in most HS I_{sc} exceeeded I_{sco} even after 1000 hours. Contrary to the above findings, a fast degradation of HS characteristics was observed in samples stored under ambient conditions (second series). In those samples, after 24 hours, under a total photoexposure dose 20kJ/cm^2. V_{oc} and I_{sc} decreased by ca. one order of magnitude, the decrease of I_{sc} being always more pronounced than that of V_{oc}. It should be noted that under these conditions we still could not record any changes in absorbances of the samples.

The main reason of HS degradation are photochemical reactions which lead to the creation of trapping centers in the organic layer and CuI films leading to an increase of ρ. Consequently, the intrinsic resistance should also increase, resulting in a decrease of I_{sc}, more pronounced than the corresponding change of V_{oc}. Apparently, the photochemical reactions take place only at the interface between the top CuI electrode and the organic film, or in the CuI layer. To check this supposition, additional measurements of the degradation of surface resistance of CuI layer were performed (Figure 4). It may be seen that the surface resistance increases after 1000 hours by one order of magnitude for samples stored in darkness and by 3 orders for samples stored under abbient conditions. It is therefore supposed that such an increase is the main reason of the decrease of I_{sc} of HS.

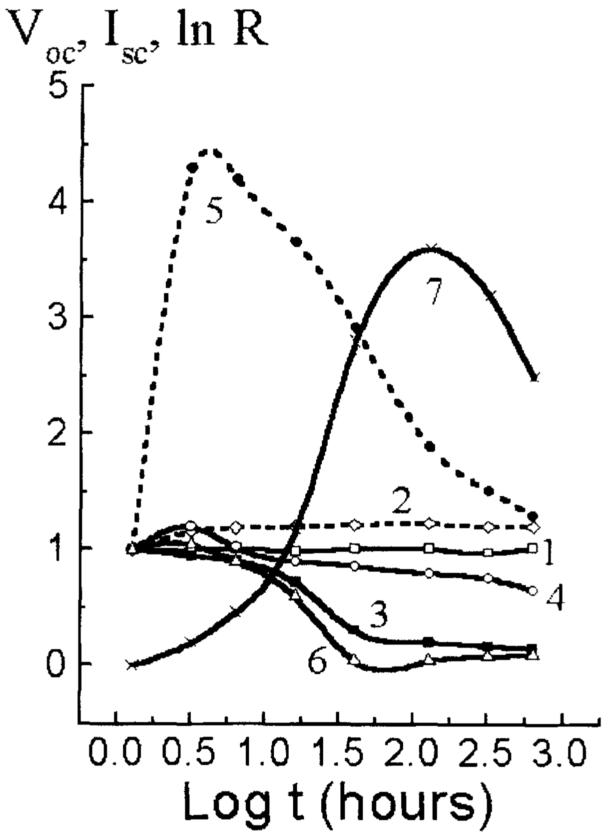

FIGURE 4 Dependnces of V_{oc} (curves 1–3), I_{sc} (curves 4–6) and the resistance of top CuI electrode (7) of unprotected (1,3,4,6) and protected (2,5) CdSSe/ClAlClPc HS on the storage time in the darkness (1,2,4,5) and for illuminated samples (3,6,7). All dependences are normalized to the initial values

The rate of the photochemical reactions is small in darkness (no decrease of V_{oc} was observed in the first and third series of HS). The interaction with oxygen leads to a doping of organic films but does not cause any degradation of solar cells, contrary to what was proposed earlier [3]. The O_2 doping leads to an improvement of parameters of solar cells which is confirmed by an increase of V_{oc} in samples exposed to the air, and by the presence of a maximum on the dependence of I_{sc} on the storage time in HS coated with a hermetic. This process is faster in unprotected HS. The decrease in I_{sc} for both protected and unprotected HS is caused by the increase of the resistance of CuI, since V_{oc} do not change. The increase of the resistance of CuI increase can be explained by a change of its stoichiometry. Similar results were observed for SnO_2/CdSSe/PbPc/CuI HS.

The efficiency of chemical and photochemical reactions at the CdSSe/organic film interface is smaller than at a metal/organic film interface. The processes of degradation are caused by photochemical reactions (oxidation) in the top electrode layer and near its interface with the organic film [3,8,16].

7. THE POSSIBILITY OF USE OF HS AS GAS SENSORS

Preparation of gas sensors with fast response is one of the most prospective emerging applications of organic HS [17]. Advantages of such gas sensors are their better selectivity in comparison with metal oxide – based sensors, and their relatively long service in comparison with biosensors [17–19].

In most cases, changes of various bulk properties are used for detection of different gases, although response times of bulk sensors to the gas concentration changes are of the order of several minutes. Therefore, to decrease the response time one should either reduce film thickness or monitor changes of observables related to parameters of barriers formed near top electrodes in sensors based on sandwich structures [17]. The latter method seems to be preferable because modulated light can be used for the excitation of I_{sc}'. As frequency dependencies of the response of such structures in the air and in vacuum differ greatly[17,19], this feature allows one to increase the selectivity and the signal-to-noise ratio by the choice of a proper frequency. It was found that an IR diode coupled with a barrier structure based on PbPc film is optimal for O_2 monitoring [19]. To improve the parameters of PbPc-based gas sensors, it is necessary to enhance the barrier height. Substitution of the electrode material will hardly help since metals forming higher potential barriers with PbPc tend to oxidize rapidly [19]. Therefore to decrease response time requires to either reduce film thickness or to use for the registration change of properties, caused by barrier existence near top electrode in sensors based on sandwich-structures (SS) [17]. HS consisting of ClAlClPc and CdSSe was chosen as a model structure. The height of the potential barrier for this structure reaches 0.72eV (Table II), i.e. is greater than that of Ni/PbPc. In this case the spectral range of the excitation is wide (the whole visible and near IR region); moreover, the contributions of organic and inorganic semiconductors can be separated since ClAlClPc absorbs in the region of transparency of CdSSe, whereas practically the entire absorption of CdSSe falls into the transparency window of ClAlClPc. We used the CdSSe/ClAlClPc HS and the reference SS supplied with two ohmic electrodes. The measurements of the oxygen effect on I_{sc} were carried out upon illumination of the samples with light modulated at various frequencies. Green and IR diodes were used, shining from opposite sides, and exciting only the CdSSe layer and ClAlCLPc layer, respectively.

It was found that both absolute and relative increase of I_{sc} with increasing oxygen partial pressure are greater for HS than for SS (two orders of magnitude vs. 11 times, respectively) [20]. The absolute I_{sc} change reaches its maximum at 400 – 500 Hz and this frequency is optimal for O_2 detection. The adsorption of oxygen gives rise to the formation of local centers (charge transfer complexes) near HS interfaces, their relaxation times being of the order of 0.01–0.1s. The formation of these centers in the space charge region of organic component is proved by the fact that relative changes of I_{sc} are not only smaller but have also opposite directions. Different directions of I_{sc} changes for organic and inorganic layers can be due to opposite directions of the shift the Fermi levels upon adsorption of O_2. This is logical taking into account the fact that semiconductors with different types of conductivity were used in the fabrication of HS.

8. CONCLUSIONS

1. The rate of surface recombination at the CdSSe/organic film interface is much smaller than at interface with metals, SnO_2, or Cu_2S. Therefore, the efficiency of CdSSe/organic film HS solar cells is greater for the illumination through the organic layer.
2. The potential barrier height near the CdSSe/organic film interface increases in the series: TTT, Pn, PBPc, ClAlClPc from 0.4 to 0.8eV
3. Use of $CdS_{0.6}Se_{0.4}$ instead of CdS allows one to increase the efficieny of HS with ClAlClPc by the factor of 2.
4. To minimize the recombination rate on free surface of CdSSe films and to increase their conductivity under solar illumination, it is necessary to quench the films in $CdCl_2$+$CuSO_4$ vapors at 773K.
5. Optimal layer thicknesses of CdSSe, allowing one to achieve maximum of η, are close to diffusion length of nonequlibrium charge carriers, and optimal thicknesses of Pn are close to the thicness of the space charge region.
6. The degradation of solar cells is mainly due to photochemical reactions in the top electrode or near its interface. The rate of the above reactions can be decreased by hermetization. It was shown that the degradation of CdSSe/ClAlClPc solar cell is slower than that of ClAlClPc/metal solar cell.

7. Doping of organic film (CuPc) with oxygen results in an increase of I_{sc} and η of CdS/CuPc HS by over two orders of magnitude without any appreciable decrease of V_{oc}.
8. The sensitivity of organic film/CdSSe HS to oxygen is by an order of magnitude greater than that of organic film /metal structures. The gas sensitivity in such HS is defined by the organic layer, and CdSSe allows only to increase the height of the potential barrier. Such HS are prospective for the development of HS sensors.

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