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Plasma Enhanced Chemical Vapor Deposited Materials and Organic Semiconductors in Photovoltaic Devices

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Abstract

Introduction. PECVD enables fabrication of wide range of advanced materials with various structure such as amorphous, polymorphous, nano-crystalline, nanostructured, microcrystalline etc. and with various electronic properties. The latter can be also changed by different dopingl. PECVD silicon materials are commercially employed in multi-layered PV structures (including ones on flexible substrates). Combining these materials with crystalline silicon active substrate resulted in significant improvement of PCE in hetero junction technology PV structures. Existence of new organic semiconductors (OS) together with understanding of physical properties resulted in fast development of OC PV devices

Aim. To consider both PECVD and OS materials and to present description of fabrication, structure and electronic properties for device application.

Materials and methods. Devices based on non-crystalline materials, devices based on OS, hybrid devices. PECVD and Spin coating technique was used to deposit materials with tunable properties enabling device engineering possibilities.

Results. PECVD and OS materials were analyzed. These materials have different levels of characterization (data volume, interpretation of the results etc.) and of understanding of physics determining device performance. Some examples of these materials in PV including structures with crystalline silicon were considered.

Conclusion. Important advantage of both PECVD and OS materials is that fabrication methods are compatible and allow fabrication of great variety of hybrid device structures on crystalline semiconductors. Advantages of such devices are difficult to predict because of lack of data in scientific literature. However a new area in material science and related devices for further exploring and exploiting has appeared.

Keywords: PECVD materials, plasma deposition, organic semiconductors, photovoltaic devices, hybrid photovoltaic devices

Introduction. Crystalline semiconductors are principal materials for devices in modern solid-state electronics. Their fabrication technology, structure and electronic properties have been extensively studied for years and well reported in literature. Recently new classes materials have appeared, however, and resulted in new types of devices, which are not possible to realize with crystalline semiconductors. One such class of materials is materials (thin films) prepared by means of plasma enhanced chemical vapour deposition (PECVD) and another is class of organic semiconductors (OS). Logically this classification is not accurate because PECVD materials are defined by fabrication method, while OS are defined by chemical structure. Nevertheless, we use further this classification and notification for materials because it is convenient, and these terms are known for specialists and widely used in literature.

Both classes present artificial (human created) materials in contrast to crystalline semiconductors. They have very significant advantages versus crystalline semiconductors:

a) structure of them and consequently electronic properties can be varied in very wide range not limited by bond length (angles) and stoichiometry constrains providing impressive possibilities for material engineering;

b) they are fabricated by low temperature technology compatible with crystalline semiconductors enabling also substrates made of glass, plastics, metal foils etc. which could be also flexible;

c) these materials can be used for large area devices. PECVD is rather mature technology and PECVD materials are commercially used in such important devices as displays and solar cells, while OS technology is only at initial stage of commercialization.
Nevertheless, it is important that they both can be realized by industrial methods, which allow scaling up device production.

In this paper we consider both PECVD and OS materials and present a brief description of fabrication, structure and electronic properties principal for device applications.

Variation of technological parameters during fabrication of these materials results in significant and well controlled changes in electronic properties providing a great promise for material and device engineering. Additionally technological compatibility of them allows design and development of hybrid device structures comprising both PECVD and OS materials. We shall use mostly our results for illustration and will avoid extended discussion due to space constraints.

Experimental.

Fabrication of PECVD Materials. Chemical vapour deposition (CVD) is well developed technique based on thermal decomposition of gases resulting in formation of radicals (partly decomposed molecules with unsaturated chemical bonds) and consequent film growth on a substrate. In order to have reasonable growth rate substrate temperature should be sufficiently high in the range of 600–1000 ºC making impossible to use such substrate as glass. Radicals are principal components for the film growth and are created by only collisions of molecules with sufficiently high kinetic energy, which number depends on temperature and even at high temperature is still not large. Alternatively, plasma of glow discharge can be used for creating radicals. This technique is known as plasma enhanced chemical vapor deposition (PECVD). In this case a reactor can be designed either for inductive or capacitive type of discharge. The latter is conventionally used in both laboratory and industrial equipment.

It is known that neutrals (molecules, radicals, atoms) and charged (ions and electrons) particles existing plasma. The charged particles are sensitive to electric field applied to the capacitive electrodes to create the discharge. Both ions and electrons are accelerated in electric field before collisions, but electrons because of lighter mass gain more velocity and kinetic energy from electric field. Concentrations of charged particles in glow discharge plasma are significantly (by factor of 10^3–10^4) less than those of neutrals therefore probability of collisions for charged particles are determined by (electron, ion)-(neutrals) interactions. Between collisions both ion and electron move with acceleration determined by electric field. For the same time of travelling ion velocity increase (consequently increase of kinetic energy) is significantly less (due to mass difference) than that of electron. Dominating collision ion with neutral atom results in effective transfer of kinetic energy gained by ion to neutral thus gas temperature slightly increases. Behavior of electron is significantly different: after 1st acceleration it cannot transfer the gained energy to neutral (because of mass difference) and continues its travel gaining kinetic energy until inelastic collision when it transfers its kinetic energy into internal one (e.g. in ionization process or breaking chemical bond, or exciting core electrons in atom etc.).

Electrical field in discharge results in little increase of gas temperature. While behavior of elec-

trons have principal difference related to significantly less (by factor of 1800) electron mass. Electron practically doesn′t loss its kinetic energy in elastic collisions with heavy particles (neutrals and ions), and after a series collisions, an electron continues to increase its kinetic energy, reaching its mean value ("electron" temperature) in the range of 2…10 eV. Thus, electrons in glow discharge have mean energy enough to break any molecular bond creating radicals. That is why substrate temperature is not of principal importance in PECVD technique. Substrate temperature in PECVD fabrication is in the range of $RT - 300$ ºC, making possible deposition of materials on glass, metal or plastic foils. The latter paves way for flexible large area electronics.

Electric field in PECVD system is formed by application of DC, AC, RF or VHF) voltage from power source. RF discharge is mostly used. At present PECVD technique allows fabrication of devices of square meters area (record seems to belong to "Applied Materials" – 5.2 m^2). For the first time continuous multilayered (about 20 layers) device fabrication has been realized by PECVD roll-to-roll deposition on stainless steel foil by "UNI Solar" [3]. PECVD technique is of principal importance (dominating in the multibillion markets of displays) for fabrication large area displays and occupies significant segment in PV devices. The most important advantages of PECVD are related to facilities for material engineering (creation of artificial materials with controlled structure and electronic properties) and to continuous (or large scale) fabrication of large area devices. Fig. 1 shows PECVD installations:

a) for laboratory research with sample area up to 150×150 mm (from "MVS Inc." USA, located at INAOE, Puebla, Mexico);

b) for industrial scale experiments with sample area to 1000×1200 mm (KAI-1200 from "Oerlikon", Switzerland, located at RDC TFTE, St Petersburg, Russian Federation).

Laboratory installations conventionally comprise several chambers ("multi-chamber cluster tool") including load–lock and transport chamber that allows fabrication of multi-layered structures avoiding cross and ambient contaminations. Large area systems are usually used in modelling processes and prototyping device structures for consequent implementation of the results obtained in some large-scale production facilities.

Fabrication of organic materials. The biggest advantage of organic materials based on polymer is their solution type fabrication processes. In comparison to inorganic material deposition methods, which usually require high substrate temperature and complex high vacuum process, organic materials deposition requires only a neutral atmosphere to reduce ambient contamination that is usually obtained by nitrogen ambient in glovebox systems. Deposition techniques for semiconductor and conductor polymers can be divided in:

1) coating process (spin coating, blade coating, spray coating, etc.);

2) printing process (screen, offset and inkjet printing) [1].

Spin coating system seems to be dominating technique to deposit organic materials in research laboratories because of its "simplicity". However, other methods enabling printing are being developed to reach industrial scale such as screen or inkjet printing for deposition on flexible substrates and large areas.

Electronic properties of organic material thin films depend on the deposition process conditions determined by such factors as viscosity, diffusivity, volatility and dilution method used to prepare the initial

a   *b*

Fig. 1. Photo images of PECVD systems: *a* − laboratory level multi-chambered cluster tool ("MVSyst. Inc.", USA, located at INAOE, Puebla, Mex.; *b* − system for industrial scale experimenting ("Oerlikon, Switzerland", located at TF TE RC)

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chemical solution. Despite the flexibility of fabrication process of organic materials, it has not been found the "best" technique that dominates industry preferences.

Results for devices

Devices based on PECVD materials. Possibility of doping in PECVD films pioneered by P. Le Comber, W. Spear [2] resulted in development of PV devices firstly with Schottky barrier then with p–i–n junctions. Most developed devices are triple junction from "Uni-Solar" [3] and "micromorph" [4].

Let us consider two representatives of PV devices based on PECVD materials. The first is triple tandem fabricated by means of roll-to roll process on stainless steel substrate. The structure and the fabrication technology have been developed by "Uni Solar" [3]. It comprises 9 PECVD semiconductor layers forming 3 serially connected p–i–n junctions with a-SiH and a-SiGe:H as intrinsic semiconductors, semi- transparent frontal electrode (made of indium tin oxide ITO), conducting grid electrode to improve current collection and some additional layers. Three p–i–n junctions are formed with specially developed intrinsic a-Si:H, a-SiGe:H films with optical gaps and thicknesses of the films designed to optimize optical absorption in a wider wavelength range than that is for one semiconductor thus improving photon absorption and photo-carrier collection. The optimization also includes adjustment of absorption and thicknesses in such way that each p–i–n junction collecting its part of solar spectra should generate the same current otherwise mismatching would create losses and reducing efficiency. The best power conversion efficiency (PCE) achieved (certified) was PCE = 13 % [3] for module area about 1 m^2.

Another PV structure developed for commercial application is called "micromorph" structure [4]. The structure comprises two PECVD p–i–n junctions with amorphous a-Si:H and microcrystalline silicon mk-Si:H. Because of difference in optical band gap (Eg = 1.75 eV for a-Si:H and Eg = 1.1 eV for mk-Si:H the structure provides collection of both visible part of spectra and substantial part of NIR spectrum resulting in increase of photocurrent and efficiency. In micromorph devices, optical optimization of frontal part and rear contact has been applied for effective light trapping and better harvesting penetrated photons. The micromorph structures have been reported with double (one junction with mk-Si:H) and triple (two junctions with mk-Si:H) junctions with stabilized efficiency 11.2 % and 12 %, respectively [4].

Devices based on organic materials. Organic photovoltaic (OPV) solar cell based on solvable compounds, predominantly polymers but most recently also small molecules are increasingly being investigated. This technology promises theoretically low-cost printable PV devices on flexible substrates.

The main difference in function between organic and inorganic active layer is related to creation of rather stable exciton by absorbed photon in organic molecular or polymer absorber in organic photovoltaic (OPV) device. The diffusion length of excitons is typically the order of 10 nm, i. e. around tenth of the thickness of the active layer required to absorb significant proportion of the incident light. As a result, the majority of the photo-generated excitons in a sandwich OPV device decays (recombines) before their collection and does not contribute into current in an external circuit [5, 6]. In order to separate charges of exciton converting them in mobile ones the fundamental bulk. As a heterojunction (BHJ) concept has been developed. It involves organic material composition with the self-assembly of nanoscale heterojunctions (micro heterojunctions) created by spontaneous phase separation of the donor-like (polymer) and acceptor-like (e. g. fullerene) components. Because of this spontaneous phase separation, charge – separating nano-scaled hetero-junctions are formed throughout the bulk of the active layer. In otherwords the charges of the excitons with small diffusion lengths are separated by the local electric fields of the micro heterojunctions. This mechanism provides separation of charges (destruction) of exciton and appearance of mobile charge carriers. However, external electric field for transportation of the separated charges is required and provided by two electrodes with different work function.

After the introduction of the BHJ concept, pioneering researchers started to recognize the importance of precise control of morphology because the device performance is extremely sensitive to the nano morphology of the BHJ film induced by spontaneous phase separation of the D:A blends [7–9] A variety of processing techniques, such as thermal/solvent annealing and processing additives, [10–12] have been devised, and those attempts have enabled us rather fine-tuning 3D nanostructured BHJ morphologies. For further optimization of organic semiconductors, research interests have moved to interface engineering, i. e., inserting interfacial layers (IFLs) between the BHJ film and the electrodes [13–17]. By developing new organic/inorganic interfacial materials or

introducing alreadydeveloped materials used in other research fields, substantial studies have demonstrated that those materials function as charge-transporting/blocking layers, surface modifiers, and optical spacers, which increase the conversion efficiency of devices with organic semiconductors. More significant advances have been achieved by developing new device architectures [18–22]. For example, multi-junction structures, in which two or more sub-cells with different absorption regions are vertically stacked and interconnected in series or parallel, have allowed a broad solar spectrum to be harvested. Furthermore, the development of various donor materials with different bandgaps and fullerene/non-fullerene acceptors has also been devoted to substantially improving the efficiency of devices with organic semiconductors [23–28]. The combination of new material designs/syntheses and the previously mentioned methods have led to remarkable efficiency enhancements, reaching values over PCE > 11 % [22, 29]. Considering that BHJ organic semiconductors have impressive advantages, such as low-cost printability and extreme mechanical flexibility, when compared to those of amorphous silicon solar cells, the efficiency PCE > 11 % represents an acceptable efficiency level for flexible solar cells and further commercialization of devices with organic semiconductors [30].

Hybrid devices based on PECVD-Polymer materials. Organic-inorganic hybrid solar cells are an alternative to pure organic or inorganic PV devices. Fig. 2 shows an example of a new concept of hybrid photovoltaic structure based on a-Si:H and Polymer organic conductor ITO/ PEDOT:PSS/ (i) a-Si:H/ (n) a-Si:H. Structure was fabricated on Indium Tin Oxide (ITO)
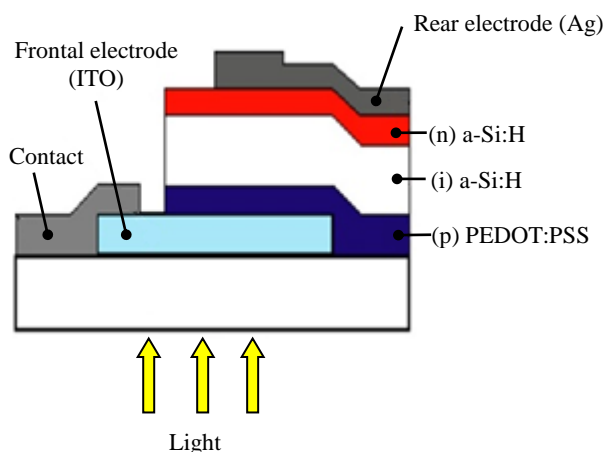
coated glass substrates. PEDOT:PSS precursor was prepared with 1:6 weight ratio. Mixed solution was filtered with a PVDF filter with pore sizes of 0.45 μm. The PEDOT:PSS layer was deposited in N_2 ambient by spin coating. The PEDOT:PSS films with thickness of 45 nm was obtained from 45 μL of solution deposited at rotation speed of 2500 rpm. Inorganic layers were deposited using a cluster multi chamber PECVD system with RF discharge at frequency $f = 13.56$ MHz. The intrinsic a-Si:H layer was deposited from an 10 % SiH_4 + 90 % H_2 gas mixture at pressure $P = 550$ mTorr. The 20 nm thick n-layers were deposited using 0.01 % PH_3 + 9.9 % SiH_4 + 90.09 % H_2 gas mixtures at pressure $P = 550$ mTorr. Finally, the 6 nm thick p-layer was deposited using the 0.26 % B_2H_6 + 21 % CH_4 + 53 % SiH_4 + 25.74 % H_2 mixture at pressure $P = 690$ mTorr. The deposition temperature was fixed at Td = 160 $^{\circ}$C and power at $W = 3$ Watt. The deposition of the top contacts was performed by sputtering of Ag through a metal shadow mask with an area of 0.09 cm^2.

The cross-section scanning electron microscopy (SEM) image (in secondary electron regime) of the hybrid photovoltaic structure on flexible substrate is shown in Fig. 3. AZO layer has columnar structure and PEDOT:PSS layer, deposited on AZO is rather inhomogeneous at the PEDOT:PSS/a-Si interface. However, PEDOT layer "heals" rather rough AZO surface, preparing smooth and planar surface for the deposition of amorphous silicon film. It is interesting to note, that the substrate defect (crack) is translated through the AZO layer and this is interrupted due to the organic polymer layer.
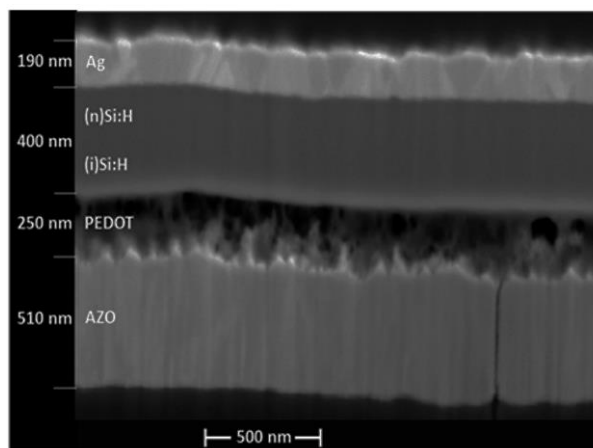

Fig. 2. Hybrid photovoltaic structure based on a-Si:H and Polymer organic conductor ITO/ PEDOT:PSS/ (i) a-Si:H/ (n) a-Si:H. PEDOT:PSS film deposited by spin coating (45 nm) 45 μL. Inorganic layers deposited by multi chamber PECVD system with RF PECVD

Fig. 3. Cross-section scanning electron microscopy (SEM) image (from secondary electron regime) of the hybrid photovoltaic structure on flexible substrate (PEN/AZO/PEDOT:PSS/ (i) a-Si:H/ (n) a-Si:H/Ag stack)

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The electronic characteristics of PEDOT:PSS can be modified by dilution method. Thus, performance characteristics of devices can be control by modification of PEDOT:PSS film. Fig. 4 shows the $J(V)$ characteristics and performance characteristics (V_{oc} and J_{sc}) of hybrid ITO/ PEDOT:PSS/ (i) a-Si:H/ (n) a-Si:H photovoltaic (PV) structures incorporating the post-deposition isopropanol (IPA) dipped PEDOT:PSS films. Structure with PEDOT:PSS film with 45 min of IPA dipping time showed the best performance with J_{sc} = 15.29 mA/cm^2, V_{oc} = 0.61 V, FF = 36.5 % and PCE = 3.40 %. Fig. 4, b displays the values of Jsc and Voc for untreated and IPA dipped samples as function of dipping time. The values of Jsc show an increase from 9.52 mA/cm^2 for the untreated PEDOT:PSS structure to 15 mA/cm^2 for the IPA dipped PEDOT:PSS structure with 45 dipping time, this may be due to the decrease of the resistivity of their IPA dipped PEDOT:PSS films. It is interesting to note some the maximum values such as J_{sc} ~ 15 mA/cm^2 obtained in these structures. The values are very similar to those in the best p–i–n structures based on a-Si:H or even better [17]. However, the shunt and serial resistances in the PEDOT:PSS/ (i) a-Si:H structures are the main issue to be solved in order to increase the FF values above 50 %. The substitution of the p-type a-Si:H:B by a PEDOT:PSS layer results in improvement of frontal interface properties and simplification of fabrication process of p–i–n structures based on amorphous silicon.

Hybrid devices using crystalline semiconductors, non-crystalline PECVD and organic materials (HJTOS structures). Combination of well-developed crystalline silicon (c-Si) solar cell with PECVD layers has provided substantial improvement in efficiency from 17 to 24 % [31]. The structure is notified as hetero-junction transitions (HJT) structure. Therefore, in this section we consider an example of such HJT device structure comprising both crystalline semiconductor and PECVD layers schematically shown in Fig. 5, a.

The base of the structure is n-doped c-Si wafer. On the top of the wafer intrinsic a-SixC1-x:H film (with optimized x), then intrinsic a-Si:H film are deposited. Above that it is p-doped microcrystalline silicon and then transparent conductive oxide (ITO) covered finally with electrode grid. Thus some junctions:

a) between p-mk-Si:H and a-Si:H film;

b) between a-Si:H and c-Si (because of optical gap difference) are created forming electric field on frontal side that improves collection of charge carriers generated by short wavelength photons.

On the rare (back) side a-Si:H and n-doped mk-Si:H forms junction with built in electric field improving transport of photo-generated charges and also contribute to photocurrent because of carrier photo-generation due to absorption of long wave length photons. Thus, better harvesting of both short wave length (on the frontal side) and long wave length on rear side, together with improving charge collection, results in significant improvement inefficiency of up to PCE = 26 % [32]. Here it is worth to noticed that p-mk-Si:H, a-Si:H, a-SixC1-x:H, n-mk-Si:H layers are deposited by PE CVD technique. An example of current-voltage characteristics $J(U)$ under sun illumination is presented in Fig. 6, a. As seen the HJT structure shows short circuit current density Jsc = 36 mA/cm^2 and excellent current collection up to voltage $U \approx 0.6\,V$. How-
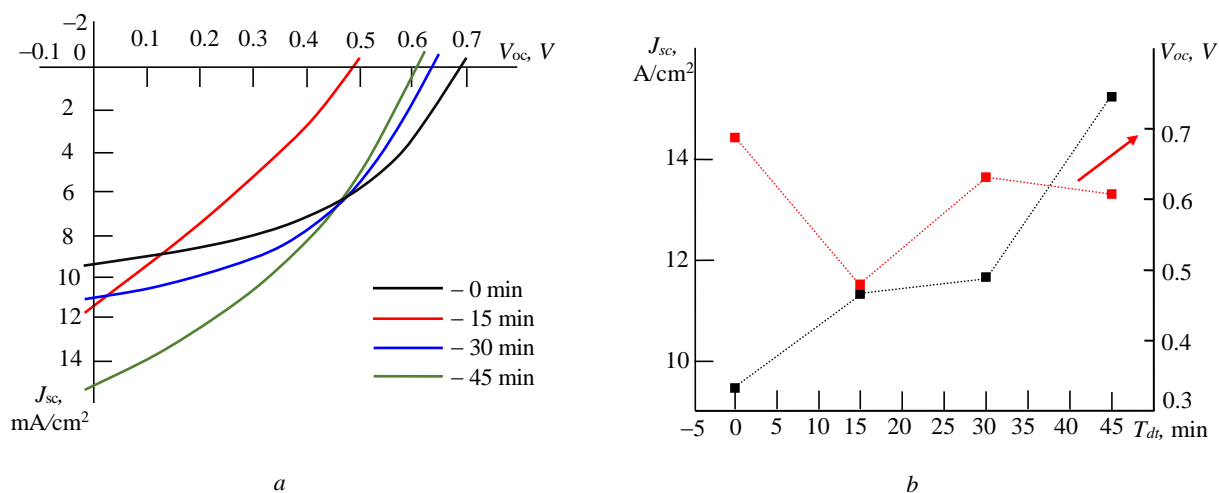
Fig. 4. Current density – voltage $J(V)$ characteristics and J_{sc} and V_{oc} parameters extracted from $J − V$ curves of hybrid solar cells structures for different dipping time in isopropyl alcohol: $a − J(V)$ characteristics under AM 1.5 solar illumination; $b − J_{sc}$ and V_{oc} parameters extracted from $J(V)$ curves as a function of dipping time in isopropyl alcohol

ever, it would be of interest to study tandem structure consisting of both HJT (bottom junction) and hybrid h–n–i–p structure (top junction) based on PE CVD and organic films. $J(V)$ characteristics measured for these two junctions (in tandem) and also for integral tandem structure are shown in Fig. 6, *b*. The lower J_{SC} and V_{OC} values observed in the HJT structure incorporated in the tandem are related to filtering incident light by top side junction.

Fig. 7, *a* represents spectral dependence of external quantum efficiency (EQE, measured in a. u.) for the same sample of HJT solar cell (see structure in Fig. 5, *a*). This graph demonstrates effective harvesting of photons by the structure in the range of wavelength from $\lambda \approx 450$ nm to 1100 nm (practically entire visible and partly NIR part of sun spectrum).

Better shortwave response has been reported in hybrid structures with organic semiconductors in frontal part of the device structure [16]. Therefore, it would be of interest fabricate tandem structure with

top junction with organic semiconductors. An example of cross-section diagram for such structure is presented in Fig. 5, *b*. Bottom junction is reproduced HJT structure (Fig. 5, *a*), on the top of which hybrid junction is placed. The latter comprises glass substrate, transparent conductive layer (aluminium doped zinc oxide, AZO), *n*-type a-Si:H, intrinsic a-Si:H, p-type organic semiconductor PEDOT:PSS, and second transparent conductive oxide (AZO). We could expect better current collection for short wavelengths, because frontal built-in electric field is determined by the interface (PEDOT:PSS)-(i-a-Si:). Current-voltage
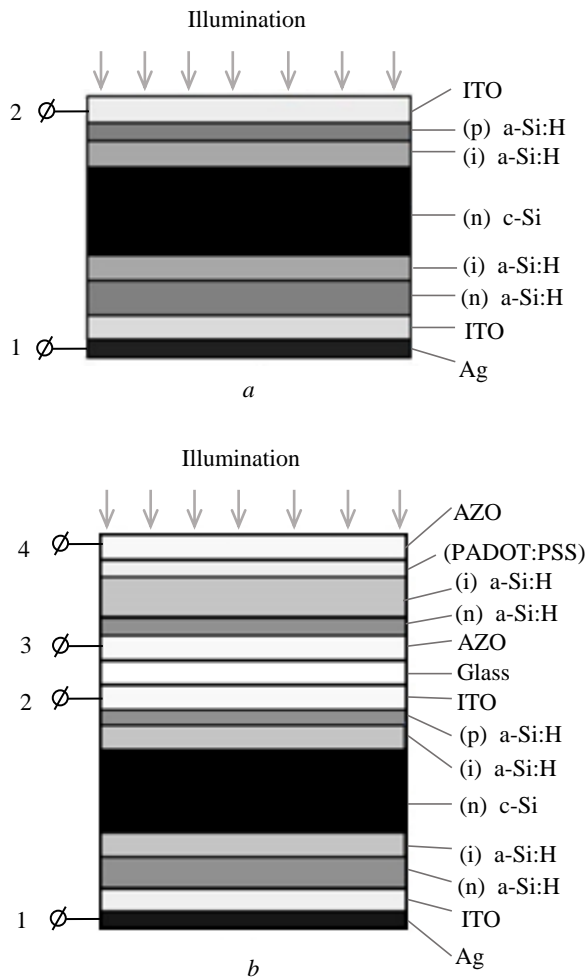

a

b

Fig. 5. Cross sectional view of HJT device with crystalline silicon: *a* − PECVD materials; *b* − HJT with OS incorporated
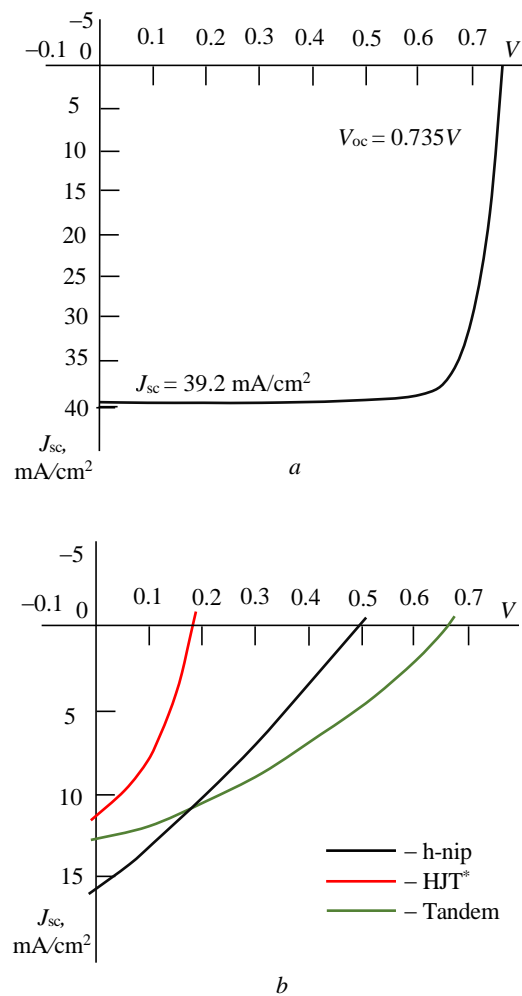

b

Fig. 6. Current density − voltage characteristics $J(V)$ measured under sun illumination: *a* − for only HJT* structure; *b* − in the integral tandem structure, which consists of bottom HJT junction and top h-nip junction; in the tandem $J(V)$ characteristics were measured separately for h-nip top junction providing $V_{OC} = 0.495$ V, $J_{SC} = 15.2$ mA/cm², for HJT* bottom junction providing $V_{OC} = 0.19$ V, $J_{SC} = 11.2$ mA/cm² and for the integral tandem providing $V_{OC} = 0.665$ V, $J_{SC} = 13$ mA/cm²
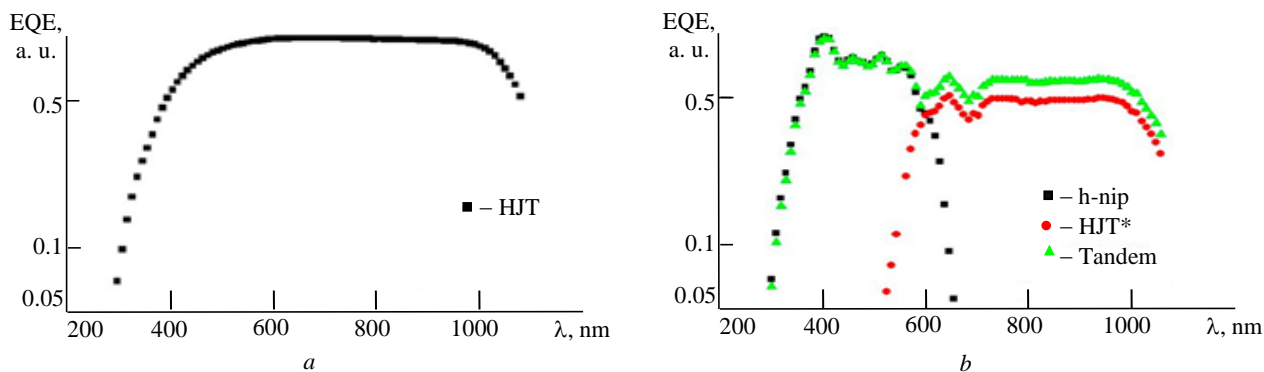
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Fig. 7. Spectral dependence of external quantum efficiency EQE = $f(\lambda)$: *a* − for HJT* structure; *b* − in the integral tandem structure, which consists of bottom HJT junction and top h-nip junction; in the tandem EQE = $f(\lambda)$ characteristics were measured separately for h-nip top junction, for HJT* bottom junction and for the integral tandem

characteristics $I(V)$ of this structures represented in Fig. 6, *b* are measured for bottom HJT junction, top hybrid junction and tandem. The structure has not been optimized to achieve high current therefore J_{sc} is significantly less, mostly because the top junction works as optical filter, however both the junctions demonstrate their functions and open circuit voltage is equal to sum both junctions. Spectral characteristics of the tandem are shown in Fig. 7, *b*, one can see that HJT OS tandem demonstrate better response in the range of $\lambda = 300…450$ nm. It should be noted also that fabrication of the tandem demonstrates compatibility of fabrication processes for crystalline silicon, PECVD materials and organic semiconductors.

Outlook. In this paper we have briefly described and analyzed two classes of materials: PECVD and organic semiconductors. These both are artificial materials with impressive possibilities for material engi-neering. However, they have different level of both characterization (data volume, interpretation of the results etc.) and understanding of physical processes determining device performance. We have also considered some examples of these materials in photovoltaic devices in different combinations including structures with crystalline silicon.

A very important advantage of both PECVD and organic materials is that technologies of their fabrication are compatible and allow a fabrication of hybrid device structures on crystalline semiconductors (e.g. on crystalline silicon). This paves the way for a great variety of hybrid device structures. At present advantages of such devices are difficult to predict because of shortage of data reported in scientific literature, but new territory in material science and related devices has definitely appeared for further exploring and exploiting.

Author's contributions

Andrey Kosarev, supervision of the study, writing a working version.
Ismael Cosme, research, processing of results, preparation of organic materials.
Svetlana Mansurova, research, optical end electrical characterization of sampels.
Dmitriy Andronikov, research, processing of results, PECVD fabrication of silicon (Si) based materials.
Alexey Abramov, research, methodology development, optical end electrical characterization of sampels.
Igor Shakhray, research, processing of results, fabrication of PECVD materials, optimization properties for device application.
Eugeny Terukov, concept development, methodology development, general management, assessment of results.

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