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### **The study of photovoltaic and electrotransport properties of organic solar cells based on a mixture of PTB7-TH thiophene and PC70BM fullerene**

New samples of organic solar cells (OSCs) based on the recently synthesized polymer, Poly[4,8-bis(5-(2-ethylhexyl)thiophen-2-yl)benzo[1,2-b;4,5-b']dithiophene-2,6-diyl-alt-(4-(2-ethylhexyl)-3-fluorothieno[3,4-b]thiophene-)-2-carboxylate-2-6-diyl)], abbreviated as PTB7-TH have been studied. 6 % energy conversion efficiency was achieved for the standard non-inverted electrode configuration, which corresponds to the international performance for such devices. Local transport of holes in the pure PTB7 film and in the mixture with fullerenes was studied by the probe scanning microscopy in the current spreading mode. Current fluctuations on a nanoscale were revealed in the pure PTB7 film and its mixtures. The current fluctuations can be explained by the influence of the structure on the mobility of holes in the framework of the space-charge limited current (SCLC) model. For the first time, estimates of the variation of the hole mobility in PTB7 were obtained in the framework of a semiempirical model. The method of secondary ion mass spectrometry (SIMS) was used to determine the distribution of the donor and acceptor in the PTB7:PC71BM:DIO film. It was found the enrichment of the surface of the film with a polymer, which explains the better efficiency when using an inverted structure. The method for the investigation of the internal structure of photoactive layers has been developed and was used for the first time, which based on obtaining a cut of the film by a focused ion beam and then subsequent measuring the slice by atomic force microscopy. The achieved quality of the cut exceeds the quality of the cut made by of the ultra microtome used in the previous stages of work. However, the phase contrast of the AFM image shows that the fine structure of the film is damaged by a focused ion beam.

*Keywords:* organic solar cells (OSCs), confocal optical microscopy, Raman microscopy, organic semiconductors, energy efficiency, bulk heterojunction, electron microscopy, scanning probe microscopy, atomic force microscopy.

#### *1. Introduction*

Organic solar cells (OSCs) are an interesting and promising field of research in the renewable energetics, which can solve energy problems, because OSCs can be produced by simple methods, borrowing technologies from the printing industry, such as roll technology or spraying [1]. It will allow creating large-area solar panel using a small amount of material. The result will be the low cost solar cells, which will lead to their widespread use and can compete with traditional energy sources. The advantage of OSCs is not only the simplicity and cost-effectiveness of their production, but also the possibility of applying them to flexible substrates, which expands the field of application of solar panels, in particular, their integration into buildings and vehicles. At the moment, the market of organic solar cells is in the initial stage of development. It is well known that many important processes occurring in the OSCs (diffusion and dissociation of excitons, transport of charge carriers, etc.), critically depend on the structure of the consisting films [1-4]. Control and optimization of morphology is an important task in the production of OCSs, which requires the development and application of the newest methods of microscopy, because the donor and acceptor materials in a photoactive layer with a bulk heterojunction form domains with a characteristic size comparable to the exciton diffusion length (~ 10 nanometers). Thus, the use of various complementary analytical methods such as scanning probe microscopy, confocal optical microscopy, electron microscopy, etc. is a necessary step in optimizing morphology and, consequently, improving the performances of the OSCs [5-7]. The use of complex high-resolution optical methods such as Tip-enhanced Raman microscopy (TERS) and scanning near-field optical microscopy (SNOM) [8], for the analysis of organic materials is a laborious task, primarily because of the instability of many organic materials exposed to laser irradiation. As a result, at the present moment there are no described results of the OSCs research using TERS and SNOM methods for investigation of promising samples of highly efficient organic solar cells, which can find wide application in various fields. Purpose of this work is to improve photovoltaic properties of polymer solar cell by optimizing morphology of bulk heterojunction and to work out procedures for studying structure of photoactive materials by advanced microscopic techniques. The novelty of the work lies in the development of the OSCs based on newly synthesized materials and the application of advanced research methods.

The proposed approach is new and original, as it involves the use of advanced methods of production and characterization of OSCs samples, in particular using the latest research methods based on a combination of scanning probe microscopy and confocal optical microscopy. On the one hand, this combination significantly expands the functionality of the apparatuses for a comprehensive study of the physical-chemical properties of materials. In particular, such an analytical complex allows automating a physical experiment and simultaneously conducting mechanical, electrical, magnetic, optical and spectroscopic measurements. On the other hand, the integrated solution provides a qualitatively new tool to probe materials - the near field, which is created with the help of a «hybrid» probe - an optical antenna and laser radiation. With the help of optical-amplified probes, plasmon microscopy of high resolution and, in particular, TERS microscopy used by us, began to develop actively.

## 2. Experimental results

The fabrication of organic solar cells was carried out completely on the equipment of the solar energy laboratory at the NLA Nazarbayev University, which includes a double glove box with spin-coater located inside and a device for thermal deposition of the upper electrode. By using this equipment, samples of organic solar cells based on a mixture of thiophene (PTB7-TH) and fullerene (PC70BM) were obtained. The complete solar cells configuration is glass substrate coated with thin layer of tin-indium oxide (ITO), a spin-coated PEDOT:PSS layer (a hole transport layer), a spin-coated photoactive layer consisting of a mixture of a donor and an acceptor and a thermally evaporated upper aluminum electrode. The upper electrode is not applied to samples intended for investigation by various types of microscopy. All the main processes were carried out in a glove box filled with nitrogen, which avoids contact with the air and moisture.

The first two stages of device assembly were performed out of a glove box, and the rest in a glove box filled with nitrogen. The substrates were made of glass substrates (2x2 cm) half-coated with ITO with a surface resistance of 10-15 ohms/cm<sup>2</sup>, which serves as the anode.

Deposition of PEDOT: PSS:

PEDOT:PSS layer performs three functions:

1. Smooths the surface of ITO.
2. Coordinates the energy of the ITO surface with the active layer.
3. Serves as a material with hole conductivity (Blocks electrons from the anode).

Obtaining high-quality PEDOT:PSS layer is critical to the efficient operation of solar cells and is often the most difficult part of the device assembling. For the adequate deposition of PEDOT: PSS, a clean and hydrophilic surface is required, which must be achieved with the thorough cleaning of the ITO surface. It is very important that the active layer does not come into contact with ITO, which is ensured by the uniform deposition of PEDOT: PSS without pinholes. The purity and composition of the PEDOT:PSS solution is also very important. We used PEDOT: PSS made of Ossila, which is filtered before using through 0.45 µm PVDF filter.

PEDOT: PSS layers were deposited by spin-coating techniques at a rotation speed of 5000-6000 rpm for 30 seconds to obtain a film thickness of 30-40 nm. For better performance of devices PEDOT:PSS should be annealed. Devices with different annealing times of PEDOT: PSS were produced from 1 min to 30 min at temperatures from 75 to 180 °C, the results showed that the annealing time temperature range does not affect the initial efficiency if during this time the devices were kept in a glove box with an inert atmosphere. Thus, after coating PEDOT:PSS to remove residual water, the samples were annealed for 5 minutes at 150°C in glove box. However, after forming the PEDOT: PSS film, it is important to minimize the exposure of the films to air, as the performance of the devices deteriorates rapidly.

Initially, the deposition of pure polymers from the solution was performed by spin-coating, then mixtures of a donor and an acceptor were coated. A relatively new polymer, PTB7-TH, was used as polymer donors, which had showed good energy conversion efficiency (9.2 %) and was well studied. As an acceptor, a compound based on PC70BM fullerenes was used. The method of dynamic spin-coating was used, in which the solution was dropped to the substrate already rotating to a constant speed. This method allows the use of a small amount of material and achieves good repeatability in the production of samples. A standard spin-coating regime was used to coat photoactive organic layers, during which the substrate was rotated for a period of 15 seconds to a constant predetermined speed, then the drop of solution was applied to a spinning substrate, followed for another 15 seconds rotating with constant speed. These parameters did not change, because their effectiveness was confirmed by numerous experiments. The amount of solution dropped to the rotating substrate was also constant-30 microliters.

The quality of films deposited on the substrate was determined primarily by visual inspection. Samples suitable for use in organic solar cells should have uniform color without visible irregularities in the region of

samples covered with indium tin oxide. Estimation of the surface quality of films on a nanometer scale was carried out using atomic force microscopy.

After coating of the active layer, an 100 nm thick aluminum electrode was deposited by thermal evaporation at a high vacuum ( $10^{-7}$  atm.) at a deposition rate of 1.5 Å/sec. Then, after deposition of the Al electrode, the samples were annealed to remove residual solvent and improve the crystallinity of the consisting films. Annealing at a temperature of about 150 °C for 15 minutes gives optimum performance. At the same time, no apparent changes in the morphology measured by the AFM occur, perhaps because of more small-scale changes not visible by atomic force microscopy.

In the Figure 1 is shown chemical structures donor material, PTB7-TH and acceptor material based on fullerene derivatives, PC70BM.

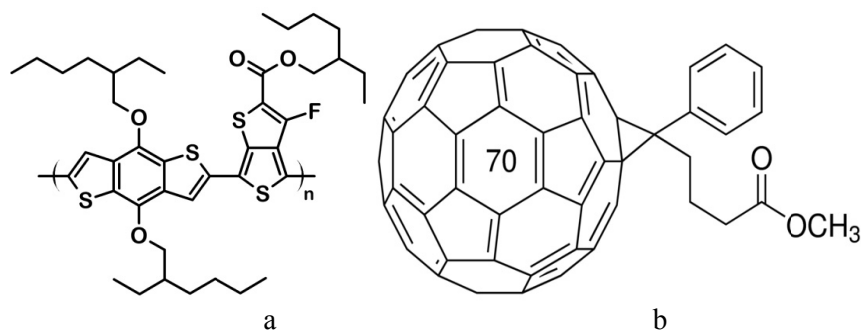


Figure 1. Chemical structure of PTB7-TH (a) and PC70BM (b)

The influence of the diiodooctane (DIO) additive on the morphology and local electrical properties of the PTB7-TH: PC71BM film and on the photovoltaic characteristics of the devices was studied.

Figure 2 and Table 1 show the I-V curve and photovoltaic performances of OSCs based on PTB7-TH: PC70BM with and without the addition of 3 % DIO. With the addition of DIO, the sizes of fullerene domains reduce and simultaneously better mixing of heterophases occurs. These all lead to enhancing of interface area of polymer/ fullerene and alleviate exciton dissociation to free charges. Transient absorption spectroscopy already revealed effective photogeneration of charge carriers for the smallest amount of DIO 0.6 and 1 %, with the greatest efficiency being detected for 3 % DIO [9]. This is explained by the better morphology of the mixture at this concentration, as a result the recombination of charge carriers reduces, which in turn increases the photovoltage of the devices. An increase in the DIO content to 10 % negatively affects the efficiency of cells, which is possibly due to the ineffective formation of free carriers and trapping in trapped locations in the interconnected donor-acceptor mixture. However, the mechanism of DIO's influence on the photovoltaic properties of polymer solar cells is not yet completely clear and requires further study.

As can be seen from Figure 2 and Table 1, DIO significantly improves photovoltaic performance and device efficiency. The efficiency of the device with DIO is twice more than no DIO device. In our work, 6 % efficiency was achieved.

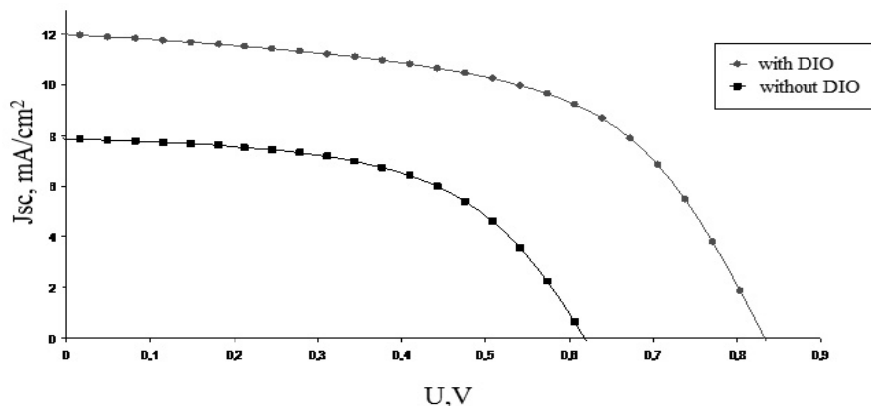


Figure 2. I-V curve of PTB7-TH:PC70BM with and without DIO

**Photovoltaic performances of standard devices based on PTB7-TH: PC70BM**

| Voc, V | Jsc, mA/cm <sup>2</sup> | Fill Factor | Efficiency, % |
|--------|-------------------------|-------------|---------------|
| 0.83   | 12.1                    | 56          | ~6            |
| 0.62   | 7.9                     | 55          | ~2.7          |

Therefore, OSCs based on the recently synthesized polymer donor PTB7-TH has been developed and obtained power conversion efficiency comparable to those published in the references for a standard (non-inverted) architecture. The next step is to optimize the resulting devices using an inverted architecture, which requires developing new processes.

*The study of charge carrier transport by the space-charge limited current method*

Currently, one of the most intensively studied polymers is polythieno[2,4,10]-thiophene-cobenzodithiophene (PTB7), which has proved to be a promising donor material for OSCs [8, 11-13]. In this our work films of pure PTB7 deposited on glass/indium-tin oxide/PEDOT:PSS substrates were studied.

Various studies show that the morphology of pure PTB7 is quite nonuniform and is characterized by the presence of a crystalline and amorphous phase [14], which certainly must affect the processes of charge transfer. The dimensions of the crystallites in PTB7 have typical dimensions comparable with the diffusion path of excitons (10 nm) [11, 12] and it is to be expected that structural nonuniformity will have a significant impact on both the hole transport processes in the PTB7 phase and on the OSCs efficiency overall.

In order to study the processes of transfer of positive charge carriers on the nanometer scale, the spreading currents and local I-V characteristics in pure PTB7 and mixtures were studied by means of the ACM in the spreading current measurement mode (c-AFM) (Fig. 3).

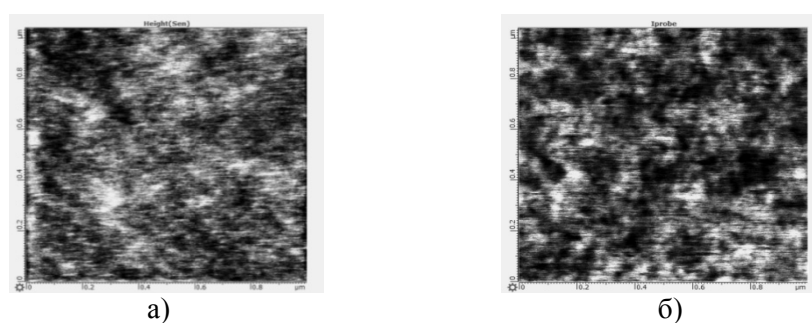


Figure 3. AFM measurements: a) topography, б) current distribution at electric potential of +0.5 V

Measurements of the spreading currents were made in contact mode with a gold-coated needle, at a voltage of + 0.5V on the needle. Under such conditions, holes are injected into the polymer from the gold probe. Figure 3 shows the current distribution on the surface of the PTB7 film. With a positive electric potential on the needle, the current distribution does not correlate with the topography of the surface.

The observed current nonuniformity (Fig. 4) reflect the influence of various processes on the transport of positive charge carriers in the PTB7, including the effect of the film structure. In general, the observed nonuniformity current distribution in the pure PTB7 film is a new result, which has not yet been described in the literature.

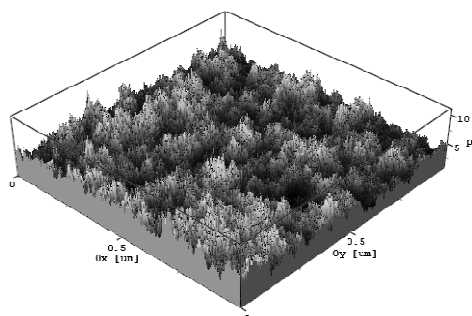


Figure 4. 3D image of current distribution at electric potential of +0.5 B

Figure 5 I-V shows curves of PTB7 of different thicknesses, as can be seen from Figure 6 the curves are straightened in double logarithmic coordinates with exponent of  $2.61 \pm 0.05$ , which indicates that the current limited by space charge under the influence of the Frenkel effect [13].

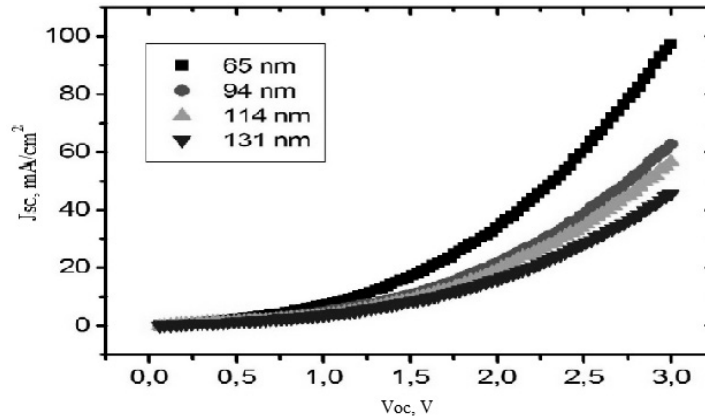


Figure 5. Averaged I-V curves of c-ACM in pure PTB7, obtained on an area of  $1 \times 1 \mu\text{m}$  ( $10 \times 10$  points)

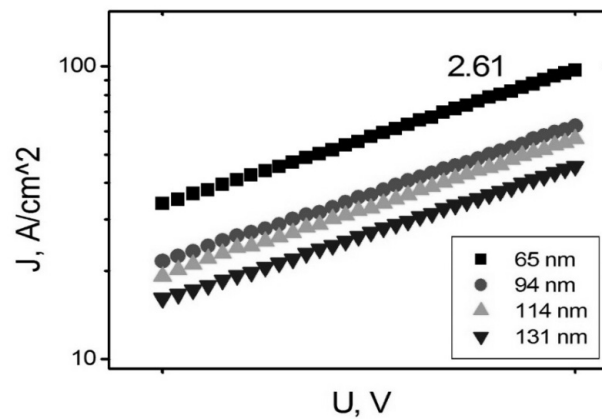


Figure 6. Averaged I-V curves of c-ACM in PTB7 in double logarithmic coordinates, obtained on an area of  $1 \times 1 \mu\text{m}$  ( $10 \times 10$  points) in the SCLC region

The dependence of the current,  $I$ , on the applied voltage,  $V$ , in this case, is described by a semi-empirical equation [8]:

$$I = A_{eff} \alpha_0 \mu \epsilon^{0.89\gamma} \left(\frac{V}{L}\right)^{\frac{1}{2}} \frac{V^2}{L^3} \delta \left(\frac{L}{D}\right)^{1.6 \pm 0.1}, \quad (1)$$

where  $A_{eff}$  — area of a contact of AFM needle with sample surface площадь;  $\alpha$  — prefactor;  $\delta$  — experimental coefficient ( $\delta=7,8$ );  $\gamma$  — coefficient determining a field or concentration dependence of hole mobility;  $D$  — diameter of probe-sample contact area;  $\epsilon$  — relative dielectric constant;  $\epsilon$  — dielectric constant (in the case of PTB7= 3);  $\mu$  — mobility of charge carriers;  $L$  — thickness of the sample. Contact area  $A_{eff}$  can be calculated using the Hertz model, where the contact radius of the needle and the surface is defined as:

$$a = \left( \frac{3FR}{4 \left( \frac{1-V_1^2}{Y_1} + \frac{1-V_2^2}{Y_2} \right)} \right)^{\frac{1}{3}}, \quad (2)$$

where  $F$  is the force of pressing the needle against the surface,  $R$  is the radius of the needle rounding,  $\nu$  is the Young's modulus, and  $Y$  is the Poisson's ratio. At  $F = 20 \text{ nN}$ ,  $R = 35 \text{ nm}$ ,  $\nu = 79 \text{ GPa}$ ,  $\nu = 0.44$  for Au and  $\nu = 3 \text{ GPa}$ ,  $\nu = 0.35$  for the polymer, the contact radius of the needle and surface is  $12 \text{ nm}$  and the contact area is  $452 \text{ nm}^2$ .

After taking logarithm (1) we get the following equation:

$$\ln\left(\frac{IL^{\Delta}d^{1.6}}{A_{eff}V^2}\right) = 0.89\gamma\left(\frac{V}{L}\right)^{\frac{1}{2}} + \ln(\alpha_0\mu\delta). \quad (3)$$

In order to determine the dependence of the current on the thickness of the sample, a dependence of  $I \approx L^{\Delta}$  is made (Fig. 7). The exponent = 1.04 was used in Equation 3.

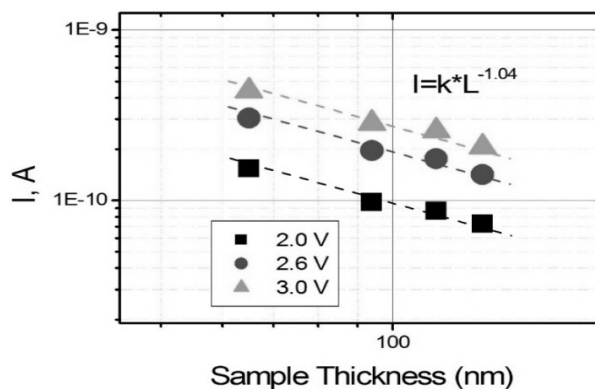


Figure 7. Dependence of the current,  $I$ , on the thickness of the PTB7 sample for different values of the voltage  $V$

The I-V curves are linearized in coordinates  $\left(\frac{V}{L}\right)^{\frac{1}{2}} = f\left(\ln\left(\frac{IL^{1.04}d^{1.6}}{A_{eff}V^2}\right)\right)$  and along a straight line segment, the mobility of the charge carriers is determined (Fig. 8).

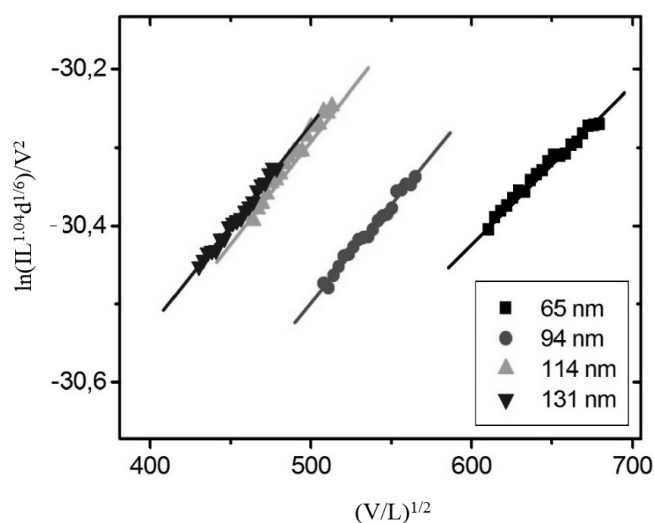


Figure 8. Computed I-V curves extracted from the SCLC region from curves shown in Figure 2

The values of hole mobility calculated in terms of equations (2) and (3) in pure PTB7 at different thicknesses are presented in Table 2.

Table 2

**The calculated value of the hole mobility in pure PTB7 for different values of the sample thicknesses**

| Sample thickness, nm                              | 65                   | 94                  | 114                 | 131                  |
|---|----------------------|---------------------|---------------------|----------------------|
| $\mu$ , $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ | $1,19 \cdot 10^{-3}$ | $1,0 \cdot 10^{-3}$ | $1,0 \cdot 10^{-3}$ | $0,91 \cdot 10^{-3}$ |

It is known that one of the most accurate methods for determining the mobility of charge carriers in organic polymers is the time-of-flight (TOF) technique [5-7, 14]. The obtained value of the mobility of holes in pure PTB7 completely coincides with the values determined by TOF technique [3].

### 3. Conclusion

New samples of organic solar cells (OSCs) based on the recently synthesized polymer, Poly[4,8-bis(5-(2-ethylhexyl)thiophen-2-yl)benzo[1,2-b;4,5-b']dithiophene-2,6-diyl-alt-(4-(2-ethylhexyl)-3-fluorothieno[3,4-b]thiophene)-2-carboxylate-2,6-diyl)], abbreviated as PTB7-TH have been studied. 6 % energy conversion efficiency was achieved for the standard non-inverted electrode configuration, which corresponds to the international performance for such devices. Local transport of holes in the pure PTB7 film and in the mixture with fullerenes was studied by the probe scanning microscopy in the current spreading mode. Current fluctuations on a nanoscale were revealed in the pure PTB7 film and its mixtures. The current fluctuations can be explained by the influence of the structure on the mobility of holes in the framework of the space-charge limited current (SCLC) model. For the first time, estimates of the variation of the hole mobility in PTB7 were obtained in the framework of a semiempirical model. The method of secondary ion mass spectrometry (SIMS) was used to determine the distribution of the donor and acceptor in the PTB7:PC71BM:DIO film. It was found the enrichment of the surface of the film with a polymer, which explains the better efficiency when using an inverted structure. The method for the investigation of the internal structure of photoactive layers has been developed and was used for the first time, which based on obtaining a cut of the film by a focused ion beam and then subsequent measuring the slice by atomic force microscopy. The achieved quality of the cut exceeds the quality of the cut made by of the ultra microtome used in the previous stages of work. However, the phase contrast of the AFM image shows that the fine structure of the film is damaged by a focused ion beam. The obtained data of distribution of the donor and acceptor in the PTB7:PC71BM:DIO film can be used in the future works for better optimizing of photoactive morphology and developing new selective contacts suitable for bulk heterojunction film surface enriched with a polymer in order to further boost device performance.

### Acknowledgements

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### **РТВ7-ТН тиофен және РС70ВМ фуллерен қоспалары негізіндегі күн ұяшықтарының фотовольтаикалық және электротранспорттық қасиеттерін зерттеу**

РТВ7-ТН аббревиатуралы жуырда синтезделген полимер негізінде ОСБ жаңа үлгілері зерттелді. Энергияны қайта қалыптастыру тиімділігі стандартты инверттелмеген электродтар конфигурациясы үшін 6%, осындай аспап үшін халықаралық көрсеткіштерге сәйкес келеді. РТВ7 донорында кемтіктердің жергілікті тасымалы және оның фуллеренді фотобелсенді қоспалары сканерлеуші зондтық микроскоппен тоқтың жайылуын өлшеу режимнің көмегімен зерттелді. РТВ7 таза қабықшасының және оның қоспаларының нанометрлік шкаласында ток флуктуациясы анықталды. Ток флуктуациясы құрылымының ТООЗ моделі шегінде кемтіктердің қозғалғыштығы әсерімен түсіндірілді. Жартылай эмпирикалық модель шегінде РТВ7 кемтіктердің қозғалғыштық вариациясын бағалау алғаш рет алынды. Қосымша иондық масс-спектрометрия әдісі РТВ7:РС71ВМ:ДИО қабықшасындағы донор және акцептор бөлінулерін анықтау үшін қолданылды. Инверттелген құрылымды қолданғанда жоғарылатылған тиімділікті түсіндіретін қабықша бетінің полимермен сапаландырылғаны анықталды. Атомдық-күштік микроскоп әдістерімен кезекті қиықты өлшеу фокусталған иондық шоғырлы қабық қиығын алуға негізделген фотобелсенді қабаттардың ішкі құрылымын зерттеу әдісі бірінші рет жасалды және қолданылды. Алынған қиықтың сапасы жұмыстың алдыңғы кезеңдерінде пайданылған ультрамикротом қиығының сапасынан асып түседі. АСМ фазалық контрасты қабықтың кішкене құрылымы фокусталған иондық шоғырмен зақымдалғанын көрсетті.

*Кілт сөздер:* органикалық күн ұяшықтары, конфокальді оптикалық микроскопия, комбинациялық шашырау микроскопиясы, органикалық жартылайөткізгіштер, энергиятиімділік, көлемді гетерооту, электрондық микроскопия, сканерлеуші зондтық микроскопия, атомдық-күштік микроскопия.

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### **Изучение фотовольтаических и электротранспортных свойств солнечных ячеек на основе смеси тиофена РТВ7-ТН и фуллерена РС70ВМ**

Исследованы новые образцы ОСБ на основе недавно синтезированного полимера с аббревиатурой РТВ7-ТН. Достигнута эффективность преобразования энергии 6% для стандартной неинвертированной конфигурации электродов, что соответствует международным показателям для такого прибора. Исследован локальный транспорт дырок в доноре РТВ7 и его фотоактивных смесях с фуллеренами с помощью режима измерения тока растекания сканирующей зондовой микроскопии. Обнаружены флуктуации тока на нанометровой шкале в пленке чистого РТВ7 и его смесях. Флуктуации тока объяснены влиянием структуры на подвижность дырок в рамках модели ТООЗ. Впервые получены оценки вариации подвижности дырок в РТВ7 в рамках полуэмпирической модели. Метод вторичной ионной масс-спектрометрии применен для определения распределения донора и акцептора в пленке РТВ7:РС71ВМ:ДИО. Обнаружено обогащение поверхности пленки полимером, что объясняет повышенную эффективность при использовании инвертированной структуры. Разработан и впервые применен метод исследования внутренней структуры фотоактивных слоев, основанный на получении среза пленки фокусированным ионным пучком с последующим измерением среза методами атомно-силовой микроскопии. Полученное качество среза превосходит качество среза ультрамикротомом, использованного на предыдущих этапах работы. Однако фазовый контраст АСМ показывает, что мелкая структура пленки повреждена фокусированным ионным пучком.

*Ключевые слова:* органические солнечные батареи (ОСБ), конфокальная оптическая микроскопия, микроскопия комбинационного рассеяния, органические полупроводники, энергоэффективность, объемный гетеропереход, электронная микроскопия, сканирующая зондовая микроскопия, атомно-силовая микроскопия.