THE ROLE OF COULOMB INTERACTION IN PHOTOINDUCED THERMALLY STIMULATED CURRENT AND LUMINESCENCE IN ORGANIC SEMICONDUCTING POLYMERS

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Photoinduced thermally stimulated current (TSC) in conjugated polymers can be controlled by either the thermally activated release of Coulombic unbound (free) trapped charge carriers or by the dissociation of coulombically bound pairs of charges (CPs). In the present work, both TSC regimes have been observed experimentally by combining TSC and thermally stimulated luminescence (TSL) studies in several conjugated polymers. We found that (i) the above-mentioned TSC regimes are determined by the dominant mechanism of charge generation during the optical excitation and, in some conjugated polymers, the regimes can be switched by changing the polarity of the load bias, and (ii) TSC and TSL temperature spectra are similar, when TSC are controlled by the release of unbound charge carriers. However, they are substantially different, when TSC is controlled by the dissociation of CPs, since the Coulomb binding energy strongly affects the dissociation of CPs into free charge carriers. Therefore, it changes the apparent activation energy of TSC. The field effect is explained by a unified model of hopping TSL and TSC in disordered organic semiconductors.

1. Introduction

The trapping of charge carriers in organic electronic materials reduces the carrier mobility, affects the driving voltage, disturbs the internal field distribution, decreases the luminescence efficiency by quenching the emissive excited state, reduces the operation stability of organic devices, and can lead to material degradation via solid-state electrochemical reactions [1]. Reliable techniques for probing the localized states and their energy distributions in organic disordered materials are therefore essential for both the material characterization and the fabrication of advanced organic optoelectronic devices. Methods, based on the thermally assisted release of trapped charges, such as measurements of thermally stimulated current [2–3] and thermally stimulated luminescence [4], are often applied to study traps in disordered materials. Since the mechanisms of charge transport and photogeneration are notoriously different in organic and inorganic semiconductors, the interpretation of TSC and TSL data has to be based on entirely different models in these two groups of materials.

The kinetics of both TSL and TSC in an inorganic semiconductor is controlled by the carrier release from localized states, populated at low temperature by the photoexcitation or strong-field injection from electrodes [5], to a band of extended states, the activation energy of this process being independent of temperature. In contrast, all electronic states are localized in disordered organic semiconductors, and the (variable range) hopping is the only possible charge transport mode in these materials. The analysis of thermally stimulated hopping is much more difficult because the increasing temperature changes both the energy distribution of localized carriers and the energy of the effective transport level [6]. In both organic and inorganic materials, the temperature spectra of TSL

and TSC are expected to be similar because the carrier release from deep states is the rate-limiting step in both processes. However, the experimental TSL peak is often observed at a much lower temperature than the TSC peak in the same material. In addition, TSC peaks, observed in organic materials, increase and shift to lower temperatures with increasing read-out voltage, V_{RO}, applied to the samples during the heating run [2].

This effect is well documented in the literature, and several explanations have been suggested. In general, an increase of the TSC peak intensity and a concomitant increase of the total charge, collected during the heating run, with increasing V_{RO}, indicate an importance of the charge recombination that may occur after the carrier release from traps and that is somehow reduced at larger fields. A more specific model is based on the Poole–Frenkel effect and tacitly assumes the presence of Coulomb centers, although the traps in organic materials are believed to be neutral when empty. The latter notion is supported by numerous charge transport studies demonstrating that the Poole–Frenkel model is not applicable to organic materials for the reasons summarized in [7].

Recently, it has been suggested [8] that the photoinduced TSC in disordered organic materials can be controlled by the dissociation of metastable geminate pairs (GPs) into free charge carriers. That metastable GPs rather than free charges are direct products of exciton dissociation in a photoexcited disordered organic material has been proven by numerous experiments including the field-induced quenching of delayed fluorescence, delayed collection of photogenerated charges, photoinduced quenching of excitons [9], etc. Trapping stabilizes GPs, and, therefore, their lifetime can be especially large at low temperatures. If the load voltage, applied during photoexcitation, is not too high, the dissociation of coulombically bound GPs into free charge carriers is also hardly possible. On the one hand, the Coulomb interaction between sibling carriers does not prevent them from the recombination back into excitons, the carrier release from traps being the rate-limiting step in the TSL kinetics. On the other hand, the Coulomb binding energy strongly affects the rate of GP dissociation into free carriers, and, thereby, it determines the activation energy of TSC.

In the present work, we report on the comparative studies of TSC and TSL in two different types of conjugated polymers with respect to the magnitude of the energetic disorder, namely a weakly disordered novel derivative of the well characterized ladder-type poly(para-phenylene) (LPPP), a diaryl (diphenyl)-substituted LPPP derivative (PhLPPP), and on a strongly energetically disordered polyphenylenevinylene derivative (PhPPV).

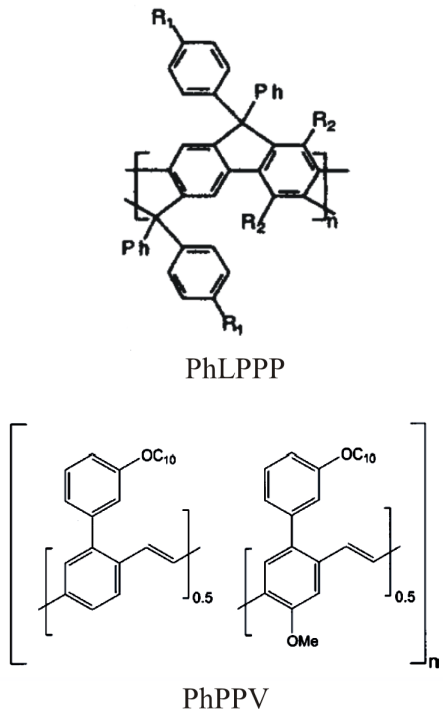

Fig. 1. Molecular structures of studied conjugated polymers

2. Experimental

The structural formulas of the conjugated polymers used in this study are shown in Fig. 1. Samples for TSC measurements were fabricated as single-layer OLEDs (active area ~0.1 cm^2) with indium-tin-oxide (ITO) and Al electrodes. Typically, 200-nm-thick polymer films were spin-coated from 1÷2 wt.% toluene solutions onto cleaned ITO-patterned substrate (1000 rpm, 30 s) in a nitrogen-flooded glovebox. Subsequently, films were dried at room temperature and transported to a high-vacuum metal deposition chamber, where a 100-nm-thick Al cathode was evaporated on the top of the films at a base pressure of 10^{-6} mbar with a deposition rate of 5 Å/s. Samples for TSL measurements were prepared by the spin coating of toluene solutions on quartz or stainless steel substrates.

TSC measurements were carried out in a continuous flow cryostat. The samples were photoexcited by illumination through the ITO electrode with 445-nm light, i.e. within the lowest excited state of the studied

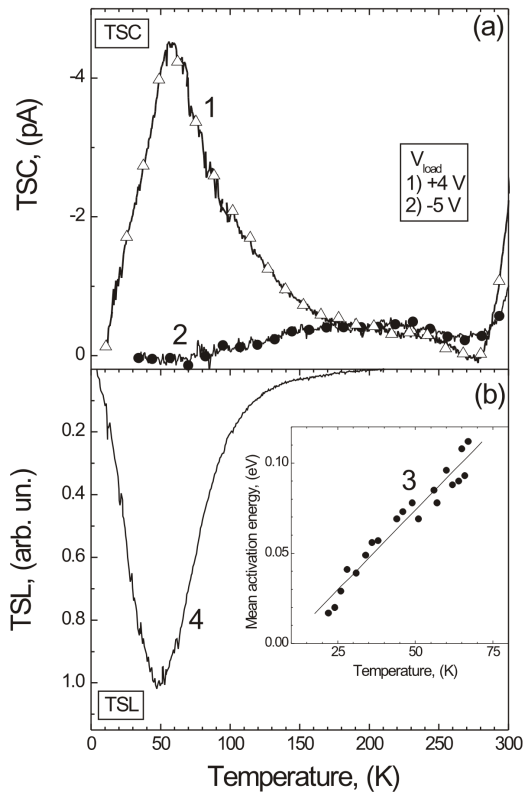

Fig. 2. (*a*) TSC from a PhLPPP sample recorded after the optical trap filling at the positive $V_{\rm load} = +4$ V (curve *1*) and negative $V_{\rm load} = -5$ V (curve *2*) load voltages at the excitation at 445 nm. TSC was recordered at zero read-out voltage. (*b*) TSL glow curve of a PhLPPP film after the excitation at 4.2 K at 436 nm (curve *4*). The inset shows the temperature dependence of the mean activation energies in PhLPPP determined by fractional TSL

polymers, from a 75-W Xenon lamp. The samples were cooled down to about 15 K in dark under zero applied voltage and illuminated for 5 min with applied load voltage $V_{\rm load}$. After the photoexcitation, the samples were kept for 5 min or longer at the initial low temperature with a biased readout voltage $V_{\rm RO}$ for the complete decay of the isothermal current. The TSC current was measured at a constant heating rate of 10 K/min by means of an electrometer that also operated as a low-noise voltage source. TSL measurements were carried out using a home-built setup operable from 4.2 to 350 K using a temperature controlled helium cryostat. After the cooling down to 4.2 K, the samples were photoexcited (typically for 30 s) using the emission from a high-pressure 500-W mercury lamp. After the photoexcitation, the TSL signal was detected in the photon-counting mode with a cooled photomultiplier positioned next to the cryostat window. The TSL measurements were performed at the constant heating rate $\beta = 9$ K/min. The details of our TSC and TSL setups have been described elsewhere [2, 3].

3. Results

It has been found that, in PhLPPP samples, the TSC temperature spectra essentially depend on the polarity of the load voltage, $V_{\rm load}$. Hereafter, the polarity of $V_{\rm load}$ refers to the potential of the ITO electrode relative to the Al contact. Figure 2,*a* shows the typical TSC spectra of PhLPPP recorded after the trap filling by the photoexcitation of a sample at the positive $V_{\rm load}=+4$ V (curve *1*) and negative $V_{\rm load} = -5$ V (curve *2*) at $T = 10$ K. All these spectra were recorded at zero read-out voltage, so the TSC was driven just by the build-in potential of the sample, $V_{\rm built-in} = -1.3$ V that was measured by applying a compensation voltage, at which the photocurrent diminishes during the photoexcitation. As one can see from Fig. 2,*a*, a pronounced low-temperature peak with maximum at ∼55 K dominates the TSC spectrum, when the photoexcitation is performed under a positive load voltage (curve *1*). This peak is absent under the negative load voltage, and TSC appears only at relatively high temperatures as a very broad peak with weakly distinguished maxima around 160 and 220 K (curve *2*). In general, the magnitude of a TSC signal increased with the exposure and saturated at a photoexcitation time of ∼5 min used in the present study. The dark TSC current observed at T>280 K is probably due to the dark injection of charge carriers from the electrodes driven by small residual voltages from the high-impedance preamplifier of the electrometer.

A TSL glow curve of a PhLPPP film after the excitation with 436-nm light is presented in Fig. 2,*b* (curve *4*). The PhLPPP films show TSL only in the low-temperature region as a broad peak with a maximum at ∼50 K. The fractional TSL measurements showed that the mean activation energy $< E_a >$ increases linearly with temperature (see the inset in Fig. 2,*b*) implying a quasi-continuous distribution of localized states in PhLPPP. Remarkably, the TSL glow curve almost completely coincides with the low-temperature TSC peak measured in PhLPPP with a positive $V_{\rm load}$ biased during the illumination (Fig. 2, curves *1* and *4*). However, the temperature dependence of TSL is clearly different from the TSC spectrum recorded for a negative $V_{\rm load}$ (Fig. 2,*a*, curve *2*).

Figure 3,*a* illustrates the typical TSC spectra of a PhPPV sample (curves *1*, *2* and *3* were monitored with

$V_{\rm load} = +7$ V, $V_{\rm load} = 0$, and $V_{\rm load} = -7$ V, respectively). A TSC curve of this polymer is a single peak with maximum at a relatively high temperature, although the onset of a TSC signal practically coincides with the starting temperature of the heating run. It is not surprising that the TSC spectra of PhPPV are broad and shifted to higher temperatures, because this polymer is a rather strongly disordered material. In contrast with PhLPPP polymer, the temperature range and the shape of the TSC spectrum in PhPPV samples are not so sensitive to the load voltage and its polarity (Fig. 3,*a*). As depicted in the inset to Fig. 3,*a*, the TSC intensity only weakly changes with load voltage when $V_{\rm load}$ is negative and zero, or when a low positive voltage (about ~1.5 V) was applied to the ITO electrode to compensate the $V_{\rm built-in}$ of the device. The TSC intensity increases with the positive load voltage when $V_{\rm load} > V_{\rm built-in}$.

Another important observation is that, at variance with the PhLPPP samples, the TSL and TSC temperature spectra of PhPPV are very different at any load voltage. A typical TSL glow curve of PhPPV films is presented in Fig. 3,*b*. The TSL peak of PhPPV films is located at much lower temperatures than TSC and shows two features, namely a stronger peak at 50 K and a weaker feature at about 100 K, as it can be clearly seen from the deconvolution of the TSC glow curve into two Gaussian peaks (Fig. 3,*b*, dotted lines). The inset to Fig. 3,*b* shows the temperature dependence of the mean activation energy as determined from fractional TSL measurements, and it proves that the TSL kinetics is controlled by a quasi-continuous distribution of intrinsic localized states and, probably, of extrinsic traps in this polymer.

Figure 4,*a* illustrates the evolution of the TSC peak in PhPPV samples with increase in the read-out voltage $V_{\rm RO}$. The magnitude of the TSC peak increases and shifts to lower temperatures with increasing$V_{\rm RO}$ (Fig. 4,*a*, curves *1*–*7*). At $V_{\rm RO} = -7$ V (Fig. 4,*a*, curve *5*) or higher negative load voltage, the measured TSC peak is located at 100 K and apparently coincides with the high-temperature TSL peak (curve *8*). No further shift to lower temperatures of the TSC peak with increasing $V_{\rm RO}$ was observed (the maximum read-out voltage $V_{\rm RO} = -18$ V was applied (Fig. 4,*a*, curve *7*)). We found, however, that this peak can be observed even without optical trap filling at higher $V_{\rm RO}$ voltages, and its magnitude is proportional to the applied read-out voltage implying the polarization origin of this peak, which masks the true behavior of TSC at negative read-out voltages below −7 V. We should note that the PhLPPP polymer used in this study did not show any

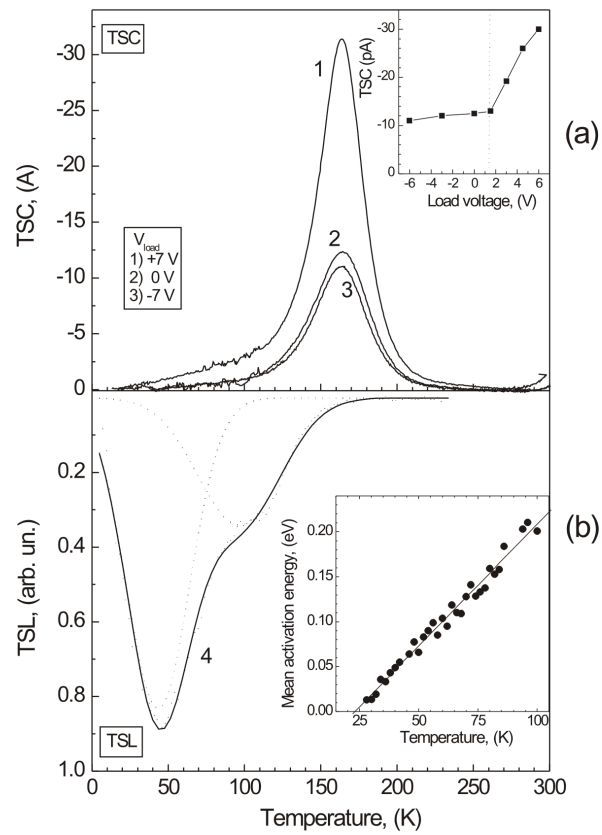

Fig. 3. *a*) TSC from a PhPPV sample recorded after the optical trap filling at the positive $V_{\rm load} = +7$ V (curve *1*), zero (curve *2*), and negative $V_{\rm load} = -7$ V (curve *3*) load voltages at the excitation at 445 nm. TSC was recorded at zero read-out voltage. The inset shows the TSC magnitude at different load voltages. *b*) TSL glow curve of a PhPPV film after the excitation at 4.2 K (curve *4*, symbols). Deconvolution of the TSC curve with two Gaussian functions is shown by dotted lines. The inset shows the mean activation energies (symbols) of the polymer measured by the fractional TSL technique; the solid line depicts an extrapolation of experimental data by a linear function

polarization peaks. Despite the concealing polarization effects, the data shown in Fig. 4,*a* clearly indicate that the difference in the TSC and TSL spectra of PhPPV is getting reduced with increasing $V_{\rm RO}$ (c.f. Fig 4,*a* and 4,*b*).

It is worth noting that the TSC peak position did not change upon increasing the film thickness from 200 to 400 nm, and, therefore, the observed field dependence of TSC spectra cannot be explained by the so-called "transport peak" [10], i.e. by the assumption that TSC is controlled by the transport of released carriers across the sample.

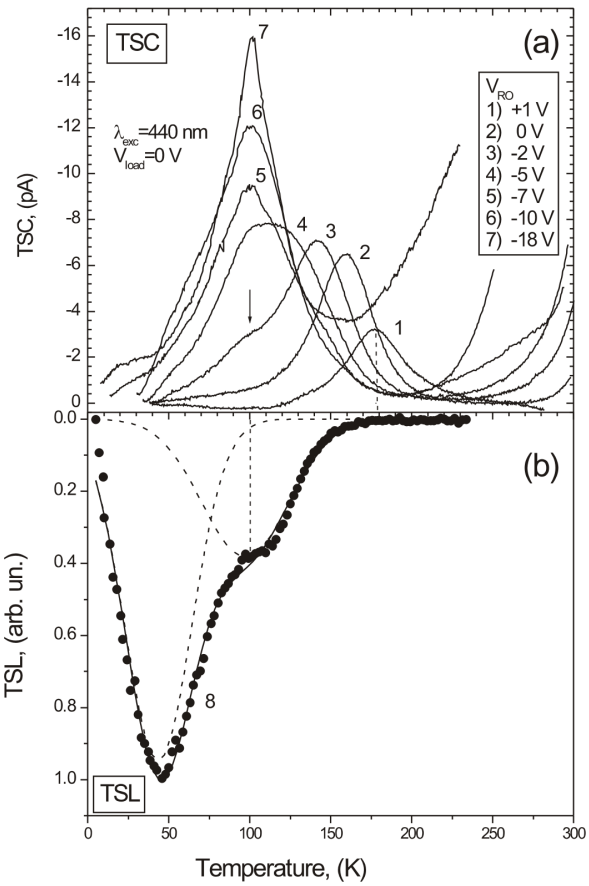

Fig. 4. *a*) TSC spectra from a PhPPV sample after the optical trap filling at zero load voltage are recorded (curve *1–7*) at different read-out voltages V_{RO}. Note that the above values should be corrected by a built-in potential of the device of about −1.5 V. *b*) TSL glow curve of a PhPPV film after the excitation at 4.2 K (curve *4*, symbols) is given for comparison

4. Discussion

The results of our study clearly demonstrate that the TSC spectra of photoconducting polymers essentially depend on the conditions, under which traps have been populated upon the optical excitation, notably upon the load voltage applied during the photoexcitation. Such a behavior could be rationalized in terms of the field-dependent charge carrier photogeneration in conjugated polymers. Free charge carriers (*unbound electrons and holes*) can be generated in pristine organic materials by the photoexitation within the lowest excited state only at a sufficiently strong electric field [6]. However, coulombically bound geminate CPs rather than free carriers are expected to be generated in organic materials at zero or very low applied fields [6]. Since only trapped carriers can survive the long isothermal relaxation following the photoexcitation, and the carrier release rate from traps should not be strongly affected by the Coulomb interaction, the TSL kinetics should be similar for the recombination into excitons of both free carriers and CPs. In contrast, the kinetics of TSC must be strongly different for CPs and coulombically unbound carriers. In order to be transported and contribute to TSC, the carriers bound in CPs have first to be released from traps and then to escape from the Coulomb potential well. The second step is not relevant for independent carriers. The Coulomb binding energy of CPs contributes to the activation energy of charge carriers bound in CPs. A distinguished feature of this contribution is that it is controlled by an external electric field. Since the effective depth of a Coulomb potential well decreases with increase in the field, i.e. read-out voltage, the carriers trapped in CPs can be released at lower temperatures, which leads to a concomitant shift of the TSC temperature spectrum.

A remarkable characteristic of the TSC phenomenon in PhLPPP samples is that the samples show distinctly different TSC spectra depending on the polarity of a load voltage. The TSC spectra of this polymer are dominated by strong low-temperature peaks at 55–60 K at positive V_{load} (Fig. 2,*a*), while, at a negative V_{load}, TSC is observed only at high temperatures while virtually no TSC signal can be found at low temperatures. This asymmetry in the TSC spectra of PhLPPP with respect to the polarity of V_{load} can be explained by the charge generation at only one of the electrodes known as surface electrode-sensitised charge photogeneration [5]. A metal electrode very efficiently quenches singlet excitons due to the dipole-dipole coupling to metal electrons [6]. Therefore, the charge generation is expected to occur only at the transparent ITO electrode [5,6] where the charge transfer dominates over the exciton quenching. At an ITO/polymer interface, electrons can be easily transferred from an excited molecule to a vacant electron state above the Fermi level of ITO, while holes are pushed into the bulk by a positive potential of the ITO-electrode. This process is believed to be very efficient due to a large energy difference between the Fermi level of ITO (4.9 eV) and the LUMO levels of most conjugated polymers, e.g. of PhLPPP (2.8 eV). The photocurrent (I_{ph} *vs* V_{load}) dependence was also found to be clearly asymmetric: the photocurrent increases much steeper for positive V_{load} suggesting the efficient photoinjection

of holes from the positively biased ITO electrode. In contrast, for negative load voltages, the photocurrent is considerably lower and most likely caused by the relatively weak photogeneration of charge carriers in the bulk.

Since mostly holes are accumulated in photoexcited polymer films, the low temperature TSC peaks in PhLPPP samples are most probably related to hole traps. The similarity of the low-temperature TSC peaks with TSL temperature spectra in PhLPPP (cf. curves *1* and *4* in Fig. 2) suggests that the released holes in TSC are not coulombically bound with electrons into charge pairs. Otherwise, the thermally assisted dissociation of charge pairs into free carriers would requires an additional activation energy, and the TSC peak should have been shifted to higher temperatures with respect to TSL. Hole mobilities in PhLPPP exceeds the electron mobility by several orders of magnitude, which implies localized states for holes be much shallower than the electron traps. Electron traps cannot be seen in TSL, since holes are liberated at lower temperatures and recombine with deeply trapped electrons constituting geminate pairs.

The ITO-sensitized surface photogeneration of holes can be terminated by applying a negative bias to the ITO electrode. Under such conditions, both electron and holes are produced due to the relatively weak bulk photogeneration. Remarkably, the low-temperature hole-related TSC peak disappears in PhLPPP upon the bulk photogeneration at a negative V_{load} (curve *2* in Fig.2,*a*), although the hole traps should be inevitably populated during the bulk photogeneration as well. This puzzle can be resolved only by the assumption that, during the bulk photogeneration, the trapped holes are not free but bound in electron-hole pairs. The coulombically bound holes can easily recombine, and hence they produce the TSL emission observed experimentally at low temperatures (Fig. 2,*b*, curve *4*). But, due to the Coulomb binding, they are hardly able to dissociate into free charge carriers at lower temperatures and to contribute to TSC. The latter process becomes feasible at elevated temperatures. Therefore, the growth of the TSC signal in the temperature range from 50 to 150 K (curves *2* in Fig.2,*a*) may reflect the dissociation of coulombically bound charge pairs. However, the higher-temperature part of the TSC spectra of these polymers at negative V_{load} can also be affected by the thermal release of deeply trapped electrons as found recently for a polyfluorene polymer [3]. Since TSL is a competitive process to TSC, the geminate pairs contribute mainly to the TSL signal at lower temperatures, while TSC reveals the reminder of charge carriers that have avoided the recombination at lower temperatures.

In contrast, PhPPV is known as a much stronger energetically disordered polymer as evidenced by its hole mobility that is by several orders of magnitude smaller than that in the ladder-type PPP or polyfluorene polymers. The TSL glow curve shown in Fig. 4,*b* suggests a double-peak DOS distribution in this polymer with the deeper peak possibly related to some sort of aggregate-type traps. The behavior of TSC spectra in PhPPV samples upon V_{load} appears to be very different from that observed for PhLPPP samples, namely: (i) TSC spectrum profile in PhPPV is not changed with a load voltage, as it follows from Fig. 3,*a*; (ii) TSC spectrum is substantially different from the TSL temperature spectra irrespective to the V_{load} (c.f. Fig. 3,*a* and 3,*b*); (iii) TSC magnitude at zero V_{load} (or at V_{load} compensating $V_{\mathrm{buit-in}}$ potential of the device) is almost the same as that obtained at negative V_{load} (c.f. curve *2* and *3* in Fig. 4,*a*), although the photocurrent during the photoexcitation was very different. This implies that the TSC magnitude in PhPPV samples at such V_{load} is virtually not sensitive to the concentration of free charge carriers generated during the photoexcitation and contributing to the photocurrent at low temperatures.

All the above observations can be explained by a premise of the *dominant role* of coulombically bound charge pairs on TSC in PhPPV samples. It seems that bound charge pairs are efficiently created in PhPPV not only at zero V_{load} but at any applied electric field, thus implying a strong tendency to the Coulomb bounding of generated charge carriers in this polymer via a bimolecular process. Hence, depending on the type of polymers, TSC phenomena can be controlled by the detrapping of unbound charge carriers or the dissociation of coulombically bound charge pairs.

An additional evidence for the role of the Coulomb interaction on the TSC spectra of PhPPV comes from the dependence of the TSC spectra on the read-out voltage (V_{RO}) applied during the heating run (Fig. 4,*a*). Since increasing the external electric field makes a Coulomb potential well effectively shallower, one could expect that the temperature spectra of TSC controlled by the dissociation of coulombically bound charge pairs should be shifted toward lower temperatures. This shift has indeed been observed in PhPPV, while no effect of the read-out voltage on the TSL peak position was found. A straightforward evidence of the recombination of charge carriers during the heating run comes from the fact that TSC increases with increase in V_{RO} that cannot affect the amount of the total stored charge.

At low fields, the released holes mostly recombine with still trapped electrons, and the probability of this recombination decreases with increase in the fields, thus resulting in an increase of the total charge collected in a TSC experiment. At $V_{\mathrm{RO}} = -7$ V, TSC spectra (Fig. 4,*a*, curve *5*) coincide well with the TSL peak at about 100 K, by indicating that the Coulomb attraction in charge pairs at such electric fields is completely compensated by the applied field, and TSC is fully controlled by the detrapping of coulombically unbound charge carriers.

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РОЛЬ КУЛОНІВСЬКОЇ ВЗАЄМОДІЇ В ФОТОІНДУКОВАНИХ ТЕРМІЧНО СТИМУЛЬОВАНИХ СТРУМІ ТА ЛЮМІНЕСЦЕНЦІЇ В ОРГАНІЧНИХ НАПІВПРОВІДНИКОВИХ ПОЛІМЕРАХ

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Р е з ю м е

Фотоіндукований термостимульований струм (ТСС) в спряжених полімерах може визначатись або термічно активованим вивільненням кулонівськи незв'язаних носіїв заряду із пасток, або дисоціацією кулонівські зв'язаних пар носіїв заряду (ЗП), що генеруються під дією фотозбудження. В даній роботі обидва режими ТСС спостерігались експериментально в ряді спряжених полімерах при комбінованому дослідженні ТСС та термостимульованої люмінесценції (ТСЛ). Ми виявили, що: (1) вказані режими ТСС визначаються домінуючим механізмом фотогенерації носіїв зарядів і в деяких спряжених полімерах можливо спостерігати перехід від одного до іншого режиму при зміні полярності прикладеної при фотозбудженні напруги, (2) криві температурної залежності ТСС та ТСЛ є аналогічними у випадку коли ТСС визначається звільненням кулонівськи незв'язаних носіїв заряду, проте вони суттєво відрізняються, коли ТСС визначається дисоціацією ЗП, оскільки кулонівська взаємодія сильно впливає на дисоціацію ЗП на вільні носії заряду, і відповідно на ефективну енергію активації ТСС. Вплив поля пояснюється в рамках узагальненої моделі ТСС і ТСЛ для невпорядкованих органічних напівпровідників із стрибковим типом транспорту носіїв заряду.