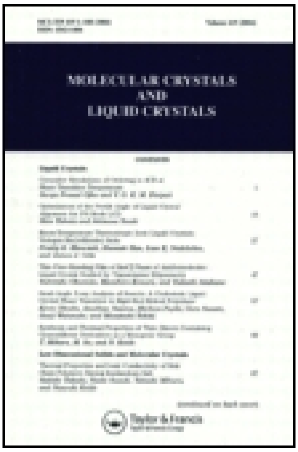

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Novel Photophysical Properties of Fullerene Doped Conducting Polymers

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NOVEL PHOTOPHYSICAL PROPERTIES OF FULLERENE DOPED CONDUCTING POLYMERS.

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Abstract

Photoluminescence in conducting polymer with non-degenerated ground state (NDGS CP) such as polythiophene derivatives, poly(p-phenylene vinylene) derivatives and polyfluorene derivatives is quenched remarkably upon doping by fullerenes such as C_{60} and C_{70}. On the other hand photoconductivity in NDGS CP and also in conducting polymer with degenerated ground state (DGS CP) such as polyacetylene derivatives is enhanced remarkably upon fullerene doping at the excitation of the wavelengths of the inter-band transition in conducting polymer and allowed and forbidden optical transitions in fullerene. Remarkable polarity effect is observed in the spectral response of enhanced photoconductivity. Conducting polymer/C_{60} layer interface exhibits also unique junction characteristics demonstrating sizable photovoltaic effect due to charges photoseparation at the interface.

These unique characteristics are discussed in terms of photo-induced charge transfer between conducting polymers and fullerenes, taking into account the roles of photoexcited self-trapped excitations: exciton-polarons, polarons and charged solitons in polymer main chains and belt-like polaron in C_{60} spherical molecule.

INTRODUCTION

Conducting polymers have attracted much attention from both fundamental scientific and practical view points because they undergo insulator-metal transition and exhibit many other interesting properties upon doping by acceptors such as iodine, BF_4^- and ClO_4^- and

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also donors such as Na or TBA$^+$.[1,2]

On the other hand, C_{60} has exhibited superconductivity[3] and also ferromagnetic behaviour[4] upon alkali metal and TDAE (tetakis(dimethylamino)ethylene) dopings respectively, which stimulated extensive studies of doping effect into fullerides such as C_{60} and C_{70} solids. In these cases fullerides have been considered to be hosts for dopants.

Recently we proposed to use fullerene molecules as dopants to conducting polymers[5] and found the quenching of photoluminescence and enhancement of photoconductivity upon doping.[6-8] Then, similar effects of photoinduced charge transfer has been observed in different types of experiments, such as photoinduced absorption and light induced ESR.[9]

In this paper we discuss the spectral response of the enhanced photoconductivity, its polarity effect as functions of molecular structure of conducting polymer, existence of persistent photoconductivity and also junction characteristics of C_{60}/conducting polymer layered heterojunction.

EXPERIMENTAL

Conducting polymers, poly(3-alkylthiophene) (PAT), poly(9,9-dialkylfluorene) (PDAFl), poly(2,5-dialkoxy-p-phenylene vinylene) (RO-PPV) and poly(o-trimethylsilylphenylacetylene) (PTMSiPA) whose molecular structures are shown in

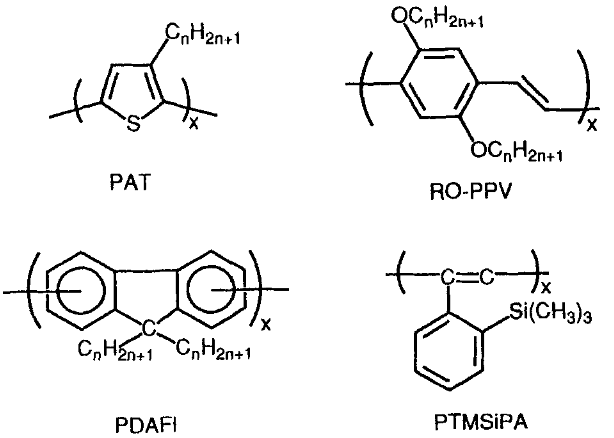


FIGURE 1 Melcular structures of PAT, PDAFl, RO-PPV and PTMSiPA.

Fig.1 were prepared and purified by the method already reported.[10-13] All these polymers are soluble in conventional solvents such as toluene and chloroform.

C_{60} and C_{70} prepared by an arc discharge utilizing graphite as an electrode and washed with toluene, and provided by Science Laboratory Co.Ltd., were used in this experiment.

Both fullerene (C_{60} and C_{70}) and conducting polymer of appropriate molar ratio were dissolved in toluene, and thin films were prepared by casting the solution on an ITO (In-Sn Oxide) coated and non-coated quartz plates. Gold deposited by evaporation on the film served as a second electrode.

Layer structure of C_{60}/conducting polymer was prepared by the depositions of evaporated C_{60} on conducting polymer coated on the gold evaporated quartz plate. Then the aluminium layer was deposited by evaporation on it as a second electrode.

Absorption and fluorescence spectra were measured by utilizing a spectrophotometer Hitachi 330 and Hitachi F-2000 fluorescence spectrophotometer. Steady-state photoconductivity was measured by irradiating an Xe arc lamp light passing through a monochromator on the sample.

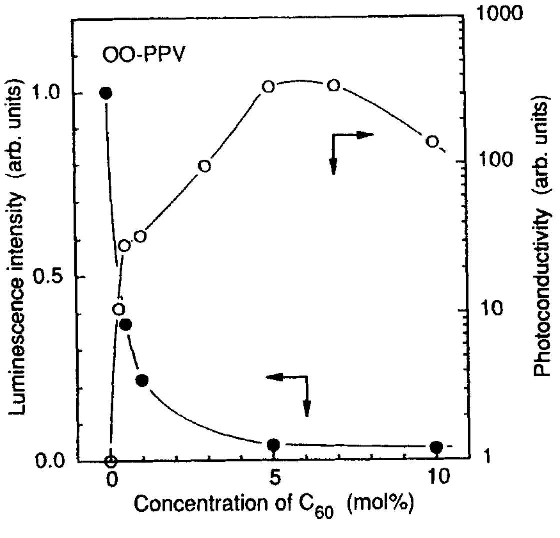


FIGURE 2 Dependence of luminescence intensity and photoconductivity of RO-PPV on the concentration of C_{60}.

RESULTS AND DISCUSSION

In conducting polymers with non-degenerated structure such as PAT, PDAFl and RO-PPV, fluorescence was quenched notably upon C_{60} doping as shown in Fig.2 for example in RO-PPV. On the other hand, photoconductivity was enhanced intensively as also evident in Fig.2 upon amount of C_{60} doping.

It should be noted in Fig.3, that the photoconductivity was enhanced in the wavelength range corresponding to (I) the inter-band transition in conducting polymer, (II) allowed transition in C_{60} $(h_u \rightarrow t_{1g})$ and also the forbidden transition in C_{60} $(h_u \rightarrow t_{1u})$.

These results can be explained by the photo-induced charge transfer between conducting polymers and C_{60}. The scenario of photogeneration is shown in Fig.4,a) for NDGS CP case. In the range (I), photo-excited intrachain exciton-polaron (E_x-P) migrates along polymer main chain and when it encounters with C_{60}, electron may be favourably transferred to C_{60} and relaxes to negative polaron (P_c^-) on C_{60}^-, which results in the quenching of photoluminescence (PL) and enhancement of photoconductivity (PC) due to the migration of positive polarons P^+ on polymer main chain. Note that in pure (undoped) NDGS CP only interchain separation of P^+ from P^- can give photoconductivity, which is rather small due to weak interchain hopping. Presence of C_{60} molecules allows e^- to be captured on it, separated spatially from P^+ on the chains, which suppresses recombination

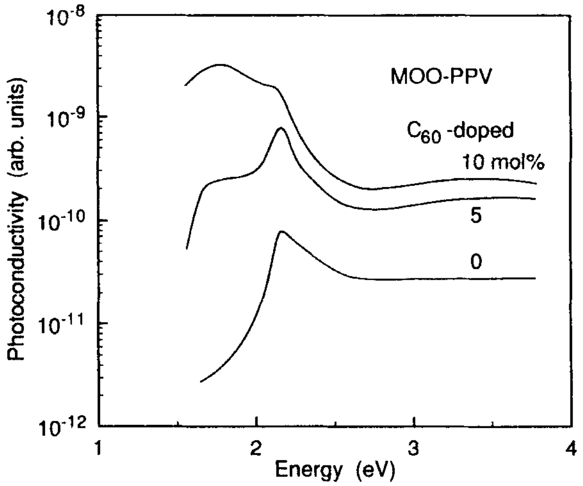


FIGURE 3 Photoconductivity spectra of RO-PPV as a function of C_{60} concentration.

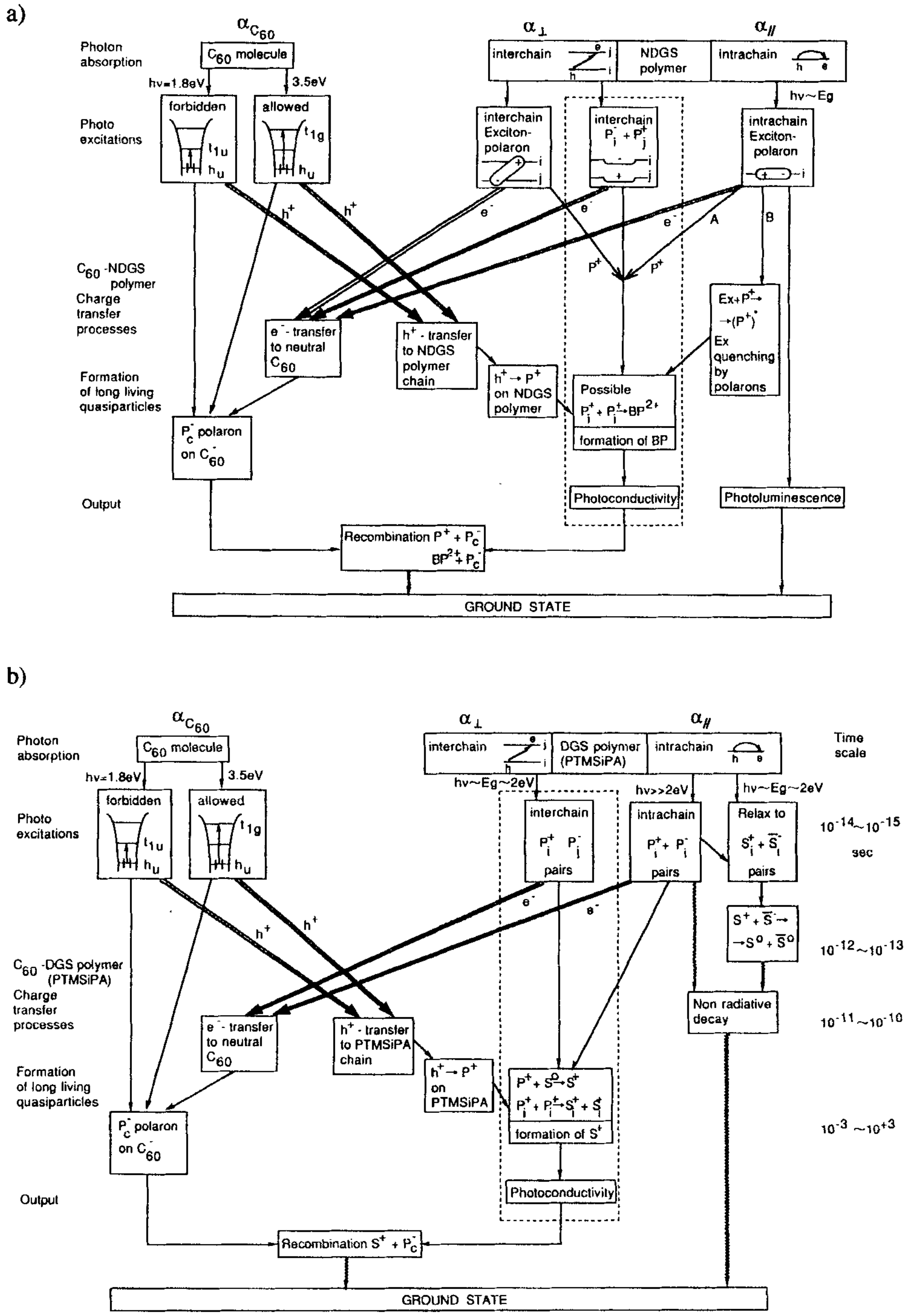


FIGURE 4 The scenario of photo-induced charge transfer: a) in NDGS CP, b) in DGS CP.

and thus increases concentration of charge carriers. In the case of photo-excitation in the wavelength region (II) and (III), electron is excited from h_u to t_{1g} and t_{1u}, respectively and the hole in C_{60} should be transferred to the valence band of conducting polymer (where it relaxes to P^+) which again contributes to enhancement of the photoconductivity.

On the other hand in PTMSiPA of degenerated structure, fluorescence was not observed even in the non-doped state. Therefore quenching of photoluminescence by C_{60} doping can not be studied. However the photoconductivity was found to be enhanced upon C_{60} doping just as in the case of NDGS CP in the wavelength also corresponding to (I) the inter-band transition, (II) excitation due to allowed transition in C_{60} and (III) forbidden transition in C_{60}.

In the wavelength ranges (II) and (III) enhanced photoconductivity can be explained by the electron excitation from h_u to t_{1g} and t_{1u} levels of C_{60} respectively, and the transfer of hole to the valence band of conducting polymer. The detailed scenario of photoconductivity enhancement in C_{60} doped DGS CP is shown in Fig.4,b).

Upon the photoexcitation at the wavelength corresponding to the inter-band transition of conducting polymer, the enhancement of photoconductivity also can be explained by taking into account the role of neutral solitons (S^o), charged solitons (S^+) and polarons (P^+) interacting with C_{60} molecules. In this case one should note that fast relaxation of intrachain photoexcited electron-hole pair into soliton-antisoliton pair: S^+ - S^- (which decays nonradiatively through transformation into neutral S^o - S^o pair) may inhibit the charge transfer to C_{60}, as sketched at Fig.4,b).

In this case the enhancement may however exist as indeed we have observed and it comes mainly from P^+ - P^- pairs photoexcited either in one chain (which becomes possible at excitation with much larger photon energy than the band edge), or on adjacent chains with formation of interchain exciton, which dissociates when encountered to C_{60}, with electron transferred to C_{60} t_{1u} level.[14] On the other hand photopumping of holes from photoexcited C_{60} is still effective channel of photoconductivity enhancement even in DGS CP as evidenced by peaks of PC at 1.8 eV and 3.5 eV, corresponding to optical transitions in C_{60}. So C_{60} plays a role of sensitizer in both NDGS and DGS CPs in its own absorption bands, while the channel of polymer photoexcitation differs due to decreased role of intrachain excitations for charge transfer from DGS CP to C_{60}, due to S - S pairs nonradiative relaxation.[14]

As evident in Fig.5, in this case remarkable polarity effect was observed in the enhanced photoconductivity spectrum. That is, only for the light irradiation from positive electrode side, large signal was observed in the wavelength regions (I) and (II). However in the range of (III), for both negative and positive polarities, that is, for the excitations both from the cathode side and anode side similar magnitude of photoresponse was observed.

This is explained clearly by the migration of photo-generated positive polarons in conducting polymer, because in the ranges (I) and (II) the carrier can be only

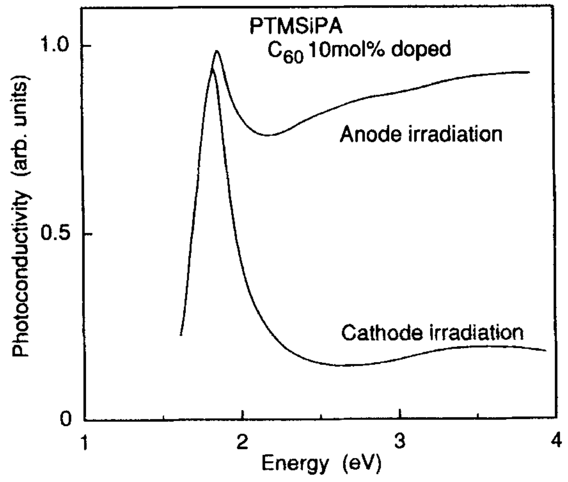


FIGURE 5 Photoconductivity spectra of C_{60}-doped PTMSiPA under: a) cathode and b) anode irradiation.

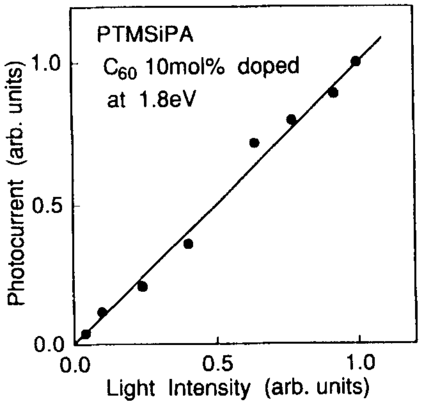


FIGURE 6 Dependence of photoconductivity of C_{60} doped PTMSiPA on the light intensity.

photogenerated at the area near the irradiated side electrode. The fact that the photoconductivity was observed for the case of the positive polarity means that predominant migrating carriers are positive carriers, most probably charged solitons, migrating through the bulk of the sample to negative electrode. However in range (III) carrier is uniformly generated in the bulk of sample. Therefore polarity of the applied voltage has no effect in the later case.

In the present experiment of relatively weak light intensity, the photoconductivity was linearly dependent on the light intensity as shown in Fig.6.

It can not be excluded that at larger light intensities, when accumulation of long living polarons on C_{60}^- is possible due to their suppressed recombination with P^+, the saturation of PC can be observed. We have observed even at low pumping density the effect of persistent photoconductivity (i.e. the memory effect of dark conductivity enhancement due to residual charges after switching off the light source). This effect is under detailed investigation currently. It is found that after switching off the light source the dark conductivity is always enhanced several times, depending on the spectral range used for photoexcitation. Only annealing to rather high temperatures can restore the initially low dark conductivity, confirming that charges are accumulated in some deep traps with life times of hours even at room temperatures, meaning that recombination of charges is strongly suppressed.

Let now summarize this part of our study of C_{60}-conjugated polymer composites. We have demonstrated above that C_{60} is an example of so called weak dopant[15,16], or in other words "photo-dopant", which can transfer the charge to the polymeric chain not in the ground state (i.e. in dark conditions) but only upon photoexcitation. Contrary to well known conventional or strong dopants, like K, Na or I_2, ClO_4^- etc.[1,2] which increase dramatically the dark conductivity and change optical properties of conjugated polymers in their ground state, the fullerene photodopant can increase significantly the photoconductivity by 10-100 times and change the optical properties mainly in nonequilibrium photoexcited state upon light irradiation, when a certain number of charge carriers, exist in the sample. Note that this number is enhanced compared to case of pure polymer (without C_{60} molecules), because in pure polymer positive and negative charges are quickly recombining in quasi-one-dimensional geometry of chained polymer, while in case of C_{60} photodopants the spatial separation of electrons on C_{60}^- from holes in P^+ on polymeric chains suppresses their recombination. This suppression of recombination is apparently strongly enhanced by the self-trapping effects both in polymer (in which charges are relaxed into deep states of polaron/bipolarons or solitons) and in fullerene (where a belt-type atomic deformation along the perimeter of C_{60} sphere is known to exist, due to Jahn-Teller effect on triply degenerate t_{1u} level.[14] This later effect of relaxation needs more theoretical understanding, but the effect of persistent photoconductivity observed here implies that this self trapping is important phenomena.

Actually in this general problem of weak dopant which exhibits strong self-trapping there are many questions that need more deeper understanding and thus more experimental study to clarify carefully each step of the process of charge separation and relaxation. Let here point out some of this important steps:

1. Photoluminescence quenching, We have mentioned above only one possible source of PL quenching due to dissociation of exciton-polaron (E_x-P) with e^- transfer to neutral C_{60}: E_x - P + C_{60} → P^+ + C_{60}^-. As noted in our earlier paper,[5] one can not exclude at present stage that the transfer of excitation energy due to dipole-dipole Förster type interaction also contributes to PL quenching:
E_x-P + C_{60} → $(C_{60})^*$, where $(C_{60})^*$ defines excited state of neutral C_{60} molecule (which exhibited $h_u \rightarrow t_{1u}$ transition, that becomes allowed in polymeric matrix.[7]) As we found in our preliminary results on the existence of persistent photoconductivity, C_{60}^- cation and P^+ (or BP^{2+}) are longly living states and after their accumulation new channels for nonlinear PL quenching may open. Firstly E_x-P may interact with P^+ and transfer energy to P^+ as already observed in nonlinear PL quenching in pure PPV upon high density of photopumping[17]:
E_x-P + P^+ → $(P^+)^*$, The theory for this E_x-P decay reaction has been proposed already[18] although it is not understood completely. Note that if initially P^+ is bound to C_{60}^- by Coulomb attraction, then in $(P^+)^*$ excited state it may become free giving rise to additional nonlinear enhancement of photoconductivity, which may be observed at high light intensity.

Another new possibility is the interaction of E_x-P with persistent P_c^- on C_{60}^-. Two processes may contribute to such interaction:
E_x-P + C_{60}^- → C_{60}^{-2}, the formation of BP on C_{60}, since C_{60} is a good acceptor, which can accept up to 6 electrons. Also the back transfer of e^- to E_x-P can not be excluded: E_x-P + C_{60}^- → P^- + C_{60}, so the careful study of time dependences and high optical pumping ESR and PC are required to clarify which of this interesting reactions between E_x-P and C_{60} takes place.

2. Photoconductivity enhancement, In our simple explanation above the enhancement of photoconductivity comes from the suppression of recombination of P^+ and P_c^-, due to their spatial separation. However we have not considered the special role of self-trapping effect. Here it should be mentioned that due to the belt type one-dimensional nature of P_c^- in C_{60} the overlapping of wave functions of P^+ (in CP chains) and P_c^- can be strongly changed due to geometrical reasons, depending on the relative orientation of the belt (ring) to the π-orbitals on conjugated segment of the chain which contains P^+. This

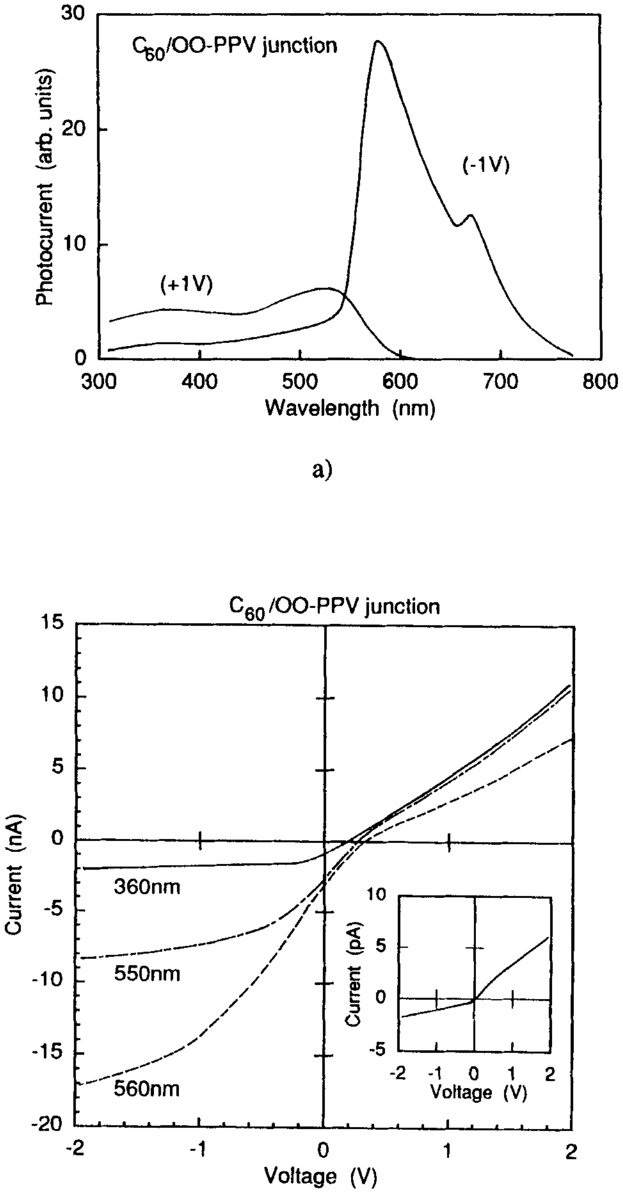
suppression of overlapping may be the reason for the observed persistent photoconductivity, since charges in C_{60} belt P_c^- may live long time, while P^+ should contribute to dark conductivity being not able to recombine with opposite charges due to negligible overlapping of their wave functions.

Another point which needs careful understanding is the Coulomb attraction of P^+ to P_c^-: simple estimates show that at 4 Å separation between C_{60} and CP chain the binding energy may be as large as 0.2 - 0.4 eV, meaning that at small C_{60} concentrations all P^+ should be bound at room temperature to P_c^-. Probably the excess of kinetic energy which P^+ has after the dissociation of E_x-P, or after hole transfer from C_{60} is enough to avoid binding at first stages, while interaction with E_x-P may provide a source of kinetic energy helping to unbound P^+ at later stage.

Anyway the careful study of persistent photoconductivity and effects of high photopumping are in progress now to understand the origin of PC enhancement and its time evolution. One should note that the mobility of P^+ also may be influenced by C_{60} dopants, however this effect may be not so large as in the case of small size strong dopants[19] due to a large size of C_{60} molecule and thus smaller effect of interchain compression upon charging, which has been observed in CP doped by small size strong dopants.[20]

Let now turn to C_{60}/conjugated polymer heterojunctions, which actually resembles the model of molecular type Donor-Acceptor diode of Aviram and Ratner.[21] In the Al/C_{60}/conducting polymer/Au layer structure, photo response was observed upon light irradiation as shown in Fig.7,a). In this case, it should be noted (in this figure OO-PPV with octyl side chain was used as a conducting polymer) large polarity effect was also observed. The sample was irradiated by monochromated light from the side of semitransparent Au electrode deposited on glass.

In the case of forward bias (that is, Au electrode is positively biased) larger dark current has been observed than in the backward bias (Au is negative) in consistency with D-A rectification,[21] since OO-PPV is a donor and C_{60} is an acceptor in this D-A rectifier, as sketched at Fig.8. The original idea of Aviram and Ratner[21] was to inject electrons to LUMO of A - molecule at forward bias from low work function electrode (Al in our case), and to inject holes to HOMO of D from hole injecting contact (Au in our case), so that recombination of e^- and h^+ at interface will complete the forward dark current as sketched at Fig.8. Clearly at backward bias such process needs much higher voltage for the transmission of current, providing rectification as observed in our case, with the only difference that charges are not free e^- and h^+ on molecular levels, but rather P^+ and P_c^- on the corresponding local levels of polarons. Same D-A layer upon light irradiation



a)

b)

FIGURE 7 a) Photoresponse of C_{60}/OO-PPV layered heterojunction and b) the current-voltage dependence of C_{60}/OO-PPV photocell in various spectral ranges of photoexcitation. Inset: the current-voltage under dark.

provides photoinduced charge separation at the interface due to favourable e^- transfer from LUMO of D (or Ex-P level of OO-PPV in our case) to LUMO of A (to t_{1u} level of C_{60}) or hole transfer from h_u level of C_{60} to valence band of polymer, followed by self trapping to corresponding polaronic states, providing photovoltage at open circuit or photocurrent (if short circuited or upon backward bias).

Upon photoexcitation indeed at the backward bias large signal was observed in the range of photon energy near the band gap. In the spectrum, photoresponse was also found at around 680 nm which corresponds to the transition from $h_u \rightarrow t_{1u}$ in C_{60}. However, much smaller signal was observed at the wavelength larger than the band gap of OO-PPV and the response at higher photon energy was negligible.

On the other hand in the case of positive polarity (Au is positively biased) photoresponse was only observed at photon energy larger than the band gap energy as shown at Fig.7. The corresponding open circuit voltage Uoc= 0.2 ev at 560-580 nm excitation, while the short circuit current Isc = 3 nA is comparatively small due to large resistance of the thick enough layer of insulating C_{60}.

It should also be noted in Fig.7,b) that the current-voltage characteristics of this layered structure element was strongly dependent on the wavelength of the excitation. That is, for the photon energy much larger than the band gap energy of OO-PPV, the photoresponse for the backward bias saturates with increasing voltage. On the other hand, that at photon energies around the band gap photocurrent increased more with voltage.

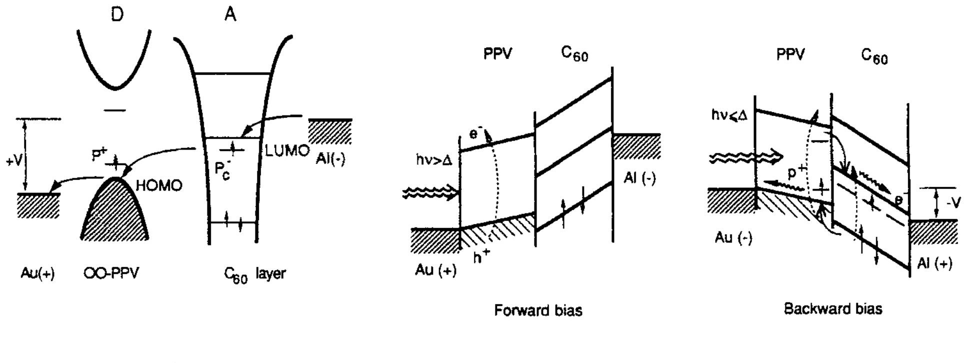


FIGURE 8 a) Rectification in D-A molecular layer diode at forward bias V>0, under dark conditions, b) photogeneration of e^--h^+ pairs at the electrode region; photo-induced charge transfer at interface is not important, c) photo-induced charge transfer at C_{60}/OO-PPV interface (shown by solid arrows) contributes to enhanced photoresponse at hv<Δ.

These results on Al/C_{60}/OO-PPV/Au layer can be explained as follows. In the case of backward bias, the response should be due to the photoinduced charge transfer between OO-PPV and C_{60} crossing the junction as sketched at Fg.8,c). Photoseparated P^+ in OO-PPV and P_c^- in C_{60} layers drift down the potential and are collected at electrodes. Note that electric field drop is mainly in C_{60} layer due to its higher resistivity. This behaviour is quite similar to the photocurrent enhancement in the conventional p/n type photodiode at backward bias. However the light with energy much larger than the band gap of OO-PPV does not reach the junction area due to large absorption in the bulk of OO-PPV. Therefore the response is mainly observed in the region around the band gap energy, where E_x-P excited at the interface can dissociate into P^+ and P_c^- due to e^- transfer. At 680 nm the h_u - t_{1u} transition in C_{60} layer is also possible followed by h^+ transfer to OO-PPV layer (Fig.8,c)), which gives a small peak in photoresponse at backward bias of Fig.7,a).

On the other hand, in the case of positive bias the carriers excited near the Au electrode by light of photon energy larger than the band gap can contribute to the photoresponse, since P^- from OO-PPV will be drifted to Au, while P^+ may drift up the electric field and can be collected at Al electrode if have chance to cross the interface due to thermal excitation or tunneling across the barrier between OO-PPV and C_{60}, shown at Fig.8,b). In this case also the light with the energy around the band gap can reach the junction area and create photoseparated carriers. However, because the polarity of Al is positive, the electron even if have been transferred to C_{60}, can not be collected at Al electrode (and hole in P^+ separated at interface can not be collected at Au electrode), and thus not contributing to photocurrent, resulting thus in the negligible response for positive bias.

The saturation in current voltage characteristics observed for backward bias can be interpreted as follows. The excited carrier density at around junction by light with photon energy much larger than the band gap is very low because such light is attenuated strongly by the absorption of OO-PPV. In such a case, the saturation effect can occur as observed in the experiment due to the limited concentration of excitons (or P^- in OO-PPV) to provide the electron for the transfer from OO-PPV to C_{60}. That is in this case the photoexcitations generation is rate determining factor for the photoresponse. Therefore, with increasing light intensity, the number of carriers separated across the interface also increases and the saturation field in the current-voltage characteristics, needed to collect all this carriers should shift to higher voltage.

So the effect of the photoinduced charge transfer between OO-PPV and C_{60} takes place only at the interface region, due to dissociation of neutral photoexcitations both in C_{60} and OO-PPV, which can not be influenced by electric field. This photoinduced charge transfer contributes to photocurrent only upon backward bias (and give sizable photovoltaic effect with Uoc = 0.2 eV),while the carriers generated at the electrode regions give rise of photocurrent at forward bias.

SUMMARY

Novel photo-induced effects such as quenching of luminescence, enhancement of photoconductivity and its polarity effect were observed in C_{60} doped conducting polymer and also C_{60}/conducting polymer junction. The results are explained by taking into account the electronic energy states of C_{60}, corresponding to so called weak acceptor, whose LUMO level, namely t_{1u} lies within the energy gap of conducting polymer. The dynamics of self trapped excitations: exciton-polaron and polaron in conducting polymers with non-degenerated ground state and solitons in the conducting polymer with degenerated ground state, together with polaron in C_{60} molecule is shown to be important for the processes of charge separation and recombination, enhancing the number of photogenerated carriers, which have rather long life time, as confirmed by observation of persistent photoconductivity.

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