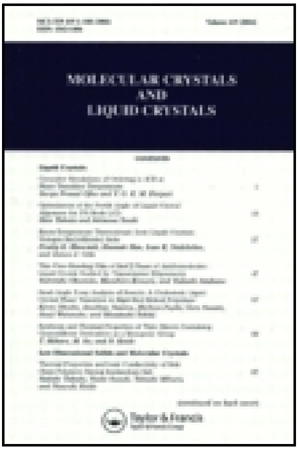

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl16

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To cite this article: Z. Burshtein (1981): Photoinjection of Holes from 9,10-Dichloroanthracene and 9,10-Dibromoanthracene into Para-Terphenyl Single Crystals, Molecular Crystals and Liquid Crystals, 71:3-4, 157-172

To link to this article: http://dx.doi.org/10.1080/00268948108072318

Mol. Cryst. Liq. Cryst., 1981, Vol. 71, pp. 157–172
0026-8941/81/7104-0157/$06.50/0

Photoinjection of Holes from 9,10-Dichloroanthracene and 9,10-Dibromoanthracene into Para-Terphenyl Single Crystals

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(*Received September 24, 1980*)

A study of photoconductivity sensitization of p-terphenyl by application of thin layers of 9,10-dichloroanthracene or 9,10-dibromoanthracene on the crystal surface is presented. The sensitized photocurrents arise from holes, photogenerated in the layers, and then injected into the p-terphenyl crystal. The spectra and voltage and temperature dependences of the sensitized photocurrents are measured and discussed.

I INTRODUCTION

Photoconductivity sensitization of organic materials has attracted considerable interest in the past,[1] much of it as a result of its potential applications to electrophotography. The growing interest in studies related to solar energy conversion devices emphasizes the importance of such studies. Of the numerous past photoconductivity sensitization studies in organics, only few were devoted to single crystals,[2] as usual anthracene being the subject material. In the last two decades considerable knowledge has accumulated on charge carrier generation and transport in several other organic compounds, making possible further insight of the detailed processes determining the sensitization of conductivity. Recently, the electrical transport and photocarrier generation processes of p-terphenyl have been measured in detail,[3,4] making it a proper candidate for photoconductivity sensitization studies.

The application of a sensitizing material on an organic crystal surface might show itself either by increased photocarrier generation yield and/or by

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extension of the optical response to longer wavelengths. The first process involves the photogeneration of excitons in the crystal as an initial step. These excitons diffuse to the surface, where they interact with the sensitizer. If the sensitizer acts as a trap for one type of carrier, the opposite-sign carrier may be injected into the crystal bulk under a proper voltage polarity. In the second process the initial step involves photogeneration of carriers in the sensitizer, which are then injected into the crystal. This process may also be reflected at the same time by photoconductivity sensitization in the fundamental crystal absorption region, if the crystal fluorescence and the sensitizer absorption spectra overlap.

The first two materials chosen to be studied as potential sensitizers to p-terphenyl were 9,10-dichloroanthracene and 9,10-dibromoanthracene (hereafter abbreviated as DCA and DBA, respectively). Some electronic transport and carrier generation properties in these materials have already been studied.[5–7] The absorption spectra of both DCA and DBA are shifted towards longer wavelengths[8] compared to that of p-terphenyl.[9] For DCA the photocurrents were established as due only to photogenerated holes.[6] It was then hoped that it could also act as an efficient electron trap, showing sensitization effects in the absorption spectral ranges of both itself and that of p-terphenyl.

II EXPERIMENTAL

The p-terphenyl crystals used were vapour-grown thin (10–100 μm thick) platelets, with their large surfaces parallel to the crystallographic (a,b) plane.[10] The DCA or DBA layers were applied to one of the crystal surfaces by either of two different ways. In one way, a drop of a dilute solution of DCA or DBA in benzene was let to dry on one of the crystal surfaces in free air. The benzene evaporated within 2–3 seconds, leaving a thin (0.1–1 μm thick) layer of the sensitizer. The other way was by vacuum deposition, also of a layer about 0.1–1 μm thick. The sample was sandwiched between a transparent conducting quartz plate (SnO_2 coated), which served as the front electrode, and mercury, which served as the back electrode. Illumination took place through the front electrode. The crystal holder was mounted in a cryogenic system, where the temperature could be varied between 150–350°K, A copper constantan thermocouple attached to the crystal holder monitored the temperature. The sample was kept under a vacuum of 10^{-5} torr. The optical system consisted of a 2,500 Watt Oriel Xe arc lamp followed by a Jarrell Ash double monochromator. The voltage across the crystal was provided by a Keithley 246 high-voltage supply. The photocurrents were measured by a Keithley 153 microvolt-ameter and recorded by a Yokogawa recorder. The light intensity was calibrated by measuring the photoconductivity of a germanium cell replacing the p-terphenyl cell.

III RESULTS AND DISCUSSION

The carrier transport and photoconduction properties of p-terphenyl has been recently studied in detail.[3,4] Photocurrents appear at wavelengths shorter than $\lambda = 0.36\ \mu m$. They are related to both hole and electron photogeneration, and involve the generation of singlet excitons as an initial step. The crystal is fully transparent at wavelengths longer than $\lambda \simeq 0.36\ \mu m$, and practically no photocurrents may be recorded in that spectral range. This situation changed when a thin layer of either DCA or DBA was applied to the crystal back surface. The crystal response to illumination with the sensitized side positive was extended to longer wavelengths (i.e., lower photon energies), up to $\lambda \simeq 0.46\ \mu m$. No change of the photocurrent spectrum was seen compared to the situation with a clean crystal when the sensitized side was negative. These features are seen in Figures 1 and 2, where the photocurrents quantum yield $\eta = J/qF$, where J is the photocurrent density, q is the electronic charge and F is the photon flux density, are plotted vs photon energy for DCA and DBA sensitizations, respectively. The DBA sensitized photocurrents (Figure 2) were much higher than those with DCA (Figure 1). They are plotted in Figure 2 on a logarithmic scale for easier comparison between the photocurrents with the DBA side positive or negative. The photon flux varied between 10^{14} to 10^{15} photons cm^{-2} sec^{-1} in the studied spectral range. All photocurrents in the spectral range shown were proportional to the incident light intensity. The plotted yield η is thus independent of the light intensity.

In fact, when the sensitized sides were positive, some slow carrier trapping occurred during illumination, and the photocurrents decayed within 20–30 sec after the onset of illumination to about 85–90% of their initial value (then they remained approximately constant). This decay results from an electric polarization of the crystal, opposing the external voltage, which is always associated with excess carrier trapping. When DCA was used as a sensitizer, the photocurrents obtained with the sensitized side negative (Figure 1) showed similar decay characteristics. For DBA, on the other hand, these photocurrents (Figure 2) *decayed in time to zero!* The charge Q transferred by the current always satisfied $Q = cV$, where c was the sample geometric capacitance and V the applied voltage. This meant that the photocurrents obtained with the DBA side negative were blocked, causing complete electrical polarization. This point will be later discussed in detail. The polarization could be removed by illuminating the crystal with light of wavelength $\lambda \simeq 0.35\ \mu m$, while both the electrodes were short-circuited to ground. The data shown in Figures 1 and 2, as well as in all other figures, correspond to the initial photocurrent signals as measured during 2–3 sec immediately after the onset of illumination. Crystal depolarization was practiced between successive measurements.[2-3] The data in Figure 1 correspond to

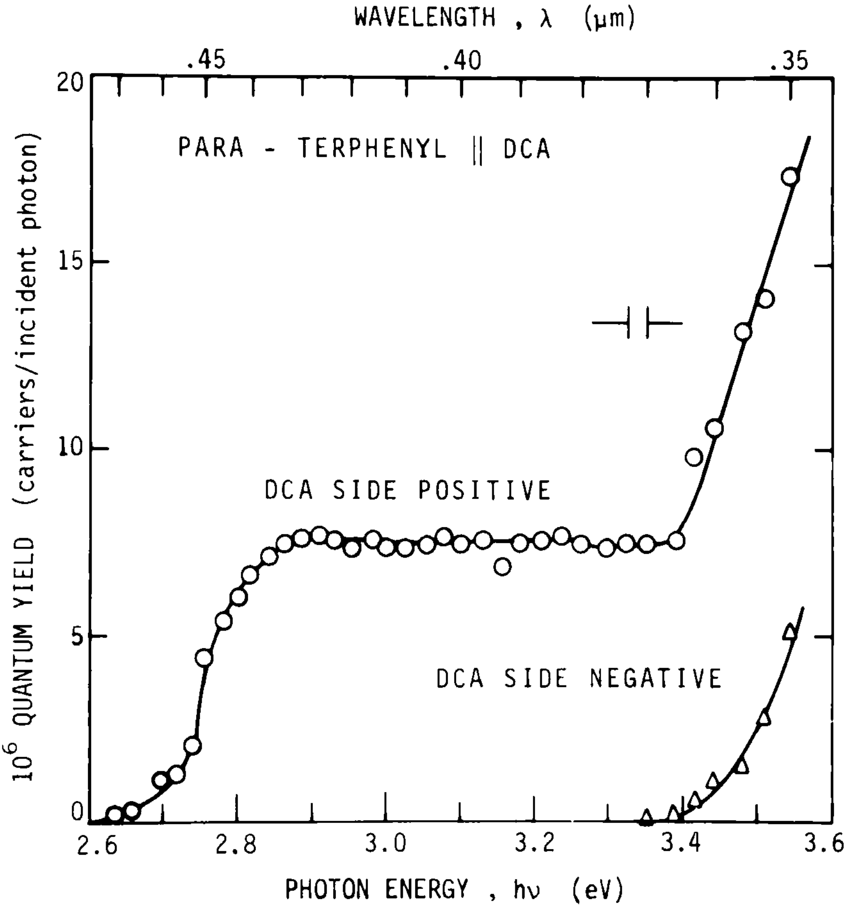

FIGURE 1 Room temperature photocurrent quantum yield vs photon energy with both a positive and a negative polarity to the 9,10-dichloroanthracene layer side. Voltages of ±500V across 85 μm thick p-terphenyl sample.

a case where the DCA layer was applied by letting a drop of a DCA solution in benzene to evaporate in free air. The data in Figure 2, on the other hand, were obtained by application of the DBA layer by vacuum deposition. However, the method by which the DCA or DBA layers were applied had no effect on any of the photoconductivity characteristics.

We shall first concentrate on discussing the DCA sensitization results, as given in Figure 1. The fast rise of the photocurrents at both polarities for photon energies above 3.4 eV coincides with the onset of the strong p-terphenyl crystal light absorption.[9] The light is fully absorbed in a region about 0.1 μm thick at the surface near the illuminated front electrode, and this

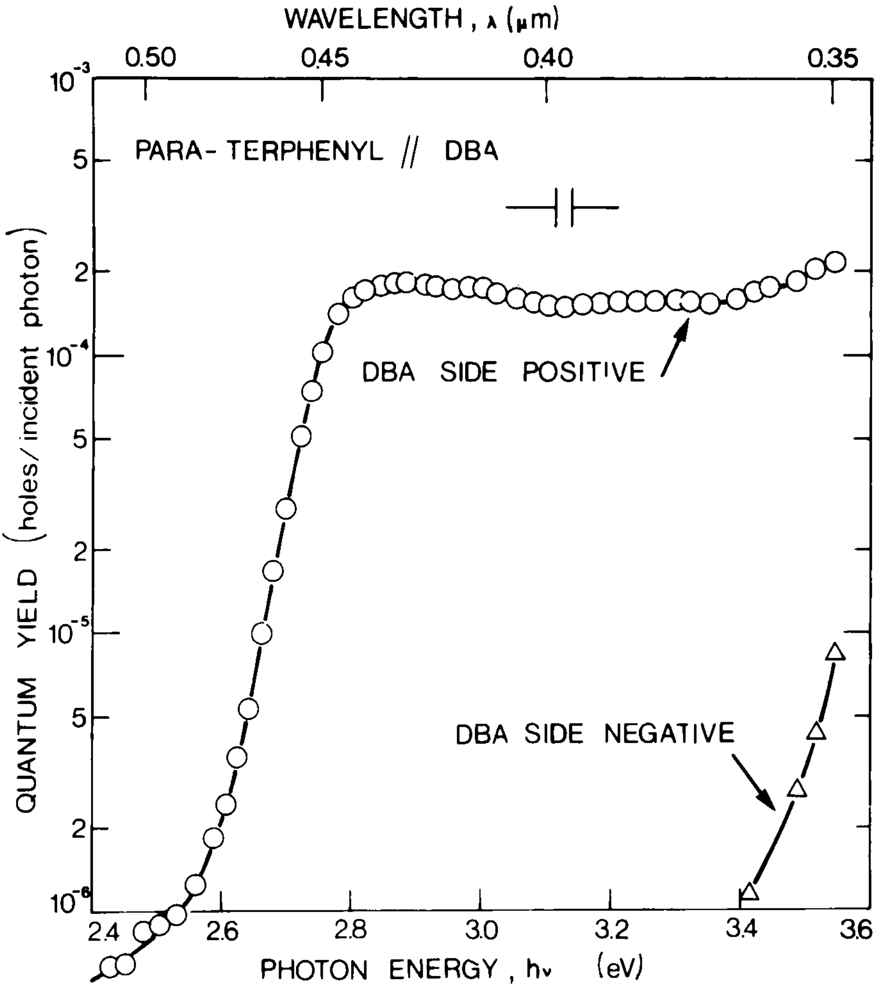

FIGURE 2 Semi-log plot of room temperature photocurrent quantum yield vs photon energy with both a positive and a negative polarity to the 9,10-dibromoanthracene layer side. Voltages of ± 150V across 40 μm thick p-terphenyl sample.

region acts as a source for free carriers. When the DCA side was positive, the illuminated side was negative, and electrons were swept into the crystal bulk, giving rise to electron conduction. The opposite polarity is similarly related to hole conduction. These photocurrents were proportional to the light intensity only up to a photon energy of about 3.55 eV. At higher photon energies they were proportional to the 3/2 power of the light intensity, consistent with the measurements of Gonzalez *et al.*[4]

The photon energy threshold of the sensitized photocurrents, shown in Figure 1 for the DCA side positive, coincides with the onset of the strong singlet-singlet light absorption of DCA.[5,8] Naturally, these photocurrents are interpreted as hole photocurrents, initially generated in the DCA layer from dissociation of photogenerated singlet excitons, and then injected into the p-terphenyl crystal. Indeed, it was seen that the photogeneration of holes in DCA is very efficient.[6] Electron photocurrents, on the other hand, could not be seen[11,6] in neither α or β crystalline forms of this material,[12,6] which meant that either photogeneration of free electrons is very inefficient, or that electrons have a very low mobility.

The nature of photoconduction in DBA[7] is less known than in DCA. Particularly, it is not yet known whether holes and electrons have comparable contributions to photoconductivity. The present results given in Figure 2, indicate, in a similar way to the case of DCA, the occurrence of very efficient hole injection from DBA into p-terphenyl. This proves that at least hole photogeneration is very efficient (also, like in DCA, as a result of the dissociation of photogenerated singlet excitons). As in Figure 1, the photocurrents above photon energies $h\nu = 3.4$ eV, with negative polarity to the DBA side, are hole photocurrents which are generated at the front electrode. The electron photocurrents, generated at the front electrode when the DBA side was positive, are reflected in Figure 2 by a moderate increase of the quantum yield in that spectral range ($h\nu \gtrsim 3.4$ eV). The DBA side still continues to inject holes by the effect of emitted fluorescence,[13] which overlaps the absorption of DBA.[8] With DCA as a sensitizer, this effect was much less important due to the much smaller hole injection efficiency (Figure 1).

In Figure 3 we see the variation with voltage of the photocurrents quantum yield at the wavelengths $\lambda = 0.35\ \mu$m and $\lambda = 0.43\ \mu$m for the p-terphenyl $\|$ DCA system. With $\lambda = 0.35\ \mu$m, which is strongly absorbed in p-terphenyl, the hole injection from the DCA is negligible. Both hole and electron photocurrents (full and open circles in Figure 2) rise fast with the voltage, but then, above 20–30V, the rise becomes much slower. The inflection voltage at 20–30V is interpreted[4] as the voltage necessary for the carriers to escape surface recombination as well as bulk trapping. The further linear increase of the photocurrents demonstrates the effect of the field on the carriers' generation process proper. Onsagers' theory[14,15] predicts for a bulk generation process a linear η vs voltage curve, with a positive intercept at zero voltage. The expected analytical form is given by[15]

$$\eta = \eta_0(1 + AE)\exp(-E_a/k_BT), \tag{1}$$

$$A = (1/4\pi\epsilon_0)(q^3/2\epsilon_r(k_BT)^2),$$

Where E_a is the energy required for the thermalized carrier to escape the Coulomb attraction of the opposite-sign carrier, k_B is Boltzmann's constant, T

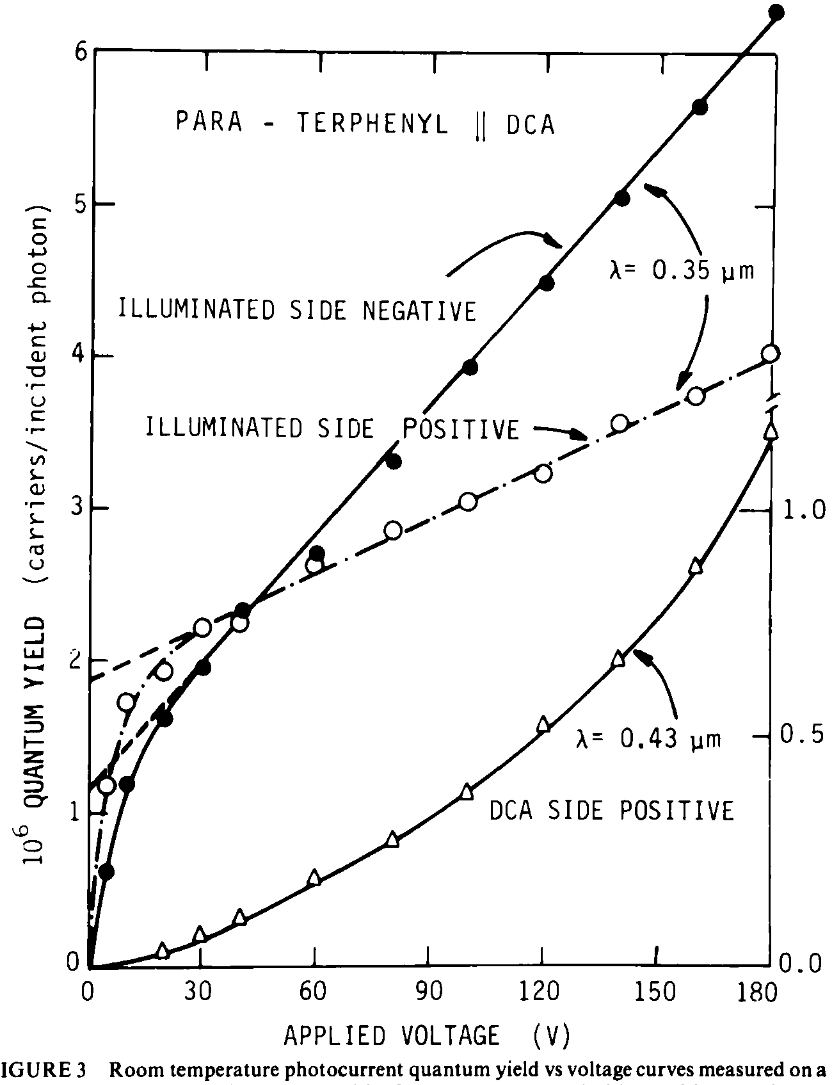

FIGURE 3 Room temperature photocurrent quantum yield vs voltage curves measured on a p-terphenyl || DCA system with both polarities for $\lambda = 0.35\ \mu m$, and with a positive polarity to the DCA side for $\lambda = 0.43\ \mu m$. Ordinate scale for the latter is marked on the right. Same sample as in Figure 1.

is the absolute temperature, E is the external electric field, ϵ_0 is the permittivity of free space and ϵ_r is the relative dielectric constant of the medium. The predictions of Onsager's theory (Eq. 1) are basically fulfilled for both hole and electron photocurrents. However, the zero voltage intercept is higher for holes, but the slope is smaller. By separate fitting of the experimental curves to Eq. (1), one finds two separate values for ϵ_r, namely $\epsilon_r = 2.3$ and $\epsilon_r = 0.6$ for the hole and electron curves, respectively (Figure 3). These results are consistent with the previous measurements of p-terphenyl photoconductivity, both under pulsed[3] and continuous[4] illuminations, in the sense that the fit of the hole curve to Eq. (1) always yielded a higher value for ϵ_r. Eq. (1) should be considered only as an approximation. For example, it does not predict different curves for holes and electrons, which is inconsistent with the experimental results.[3,4] The fact that one obtains for ϵ_r a value which is smaller than unity should be considered in that context. As seen in Figure 3, at high voltages the electron generation quantum yields are higher than the hole generation yields and this shows itself also in Figure 1, where the spectrum was taken with a voltage of 500V.

From the above it is established that voltages of 20–30V in the particular sample studied are sufficient to make the carriers overcome all surface recombinations and bulk trapping. Therefore, the hole injection photocurrents from the DCA side reflect the hole injection rate proper for voltages above 20–30V. The voltage dependence of these photocurrents under illumination with $\lambda = 0.43$ μm is shown by triangles in Figure 3. The behaviour is different than with strongly absorbed light. The quantum yield varies superlinearly with the voltage V; it is approximately proportional to $V^{1.6}$. The same was found for all sensitized photocurrents.

In Figure 4 we see, on a log-log scale, the variation with voltage of the photocurrents quantum yield at wavelengths $\lambda = 0.35$ μm and $\lambda = 0.44$ μm for the p-terphenyl || DBA system of Figure 2. Electron photocurrents obtained with the illuminated side negative for $\lambda = 0.35$ μm are masked by the parallel hole injection from the DBA. Therefore, only hole photocurrents obtained with the illuminated side positive are shown (circles in Figure 4). They are very similar to those obtained in Figure 3, except for the inflection voltage being higher, 80–100V in that p-terphenyl sample. This means that a voltage of about 80–100V is necessary for holes to overcome surface recombination and bulk trapping in that sample. Indeed, the same inflection voltage shows up for the hole injection current-voltage characteristics measured with the DBA side positive at $\lambda = 0.44$ μm (triangles in Figure 4). The initial low voltage rise is very fast, but then, above 80–100V, it attains a smaller slope of 2.0. The hole injection quantum yield is then proportional to V^2 in a p-terphenyl || DBA system. This was verified for the entire spectral range of sensitized photocurrents.

The process of carrier injection between two insulators is in principle different

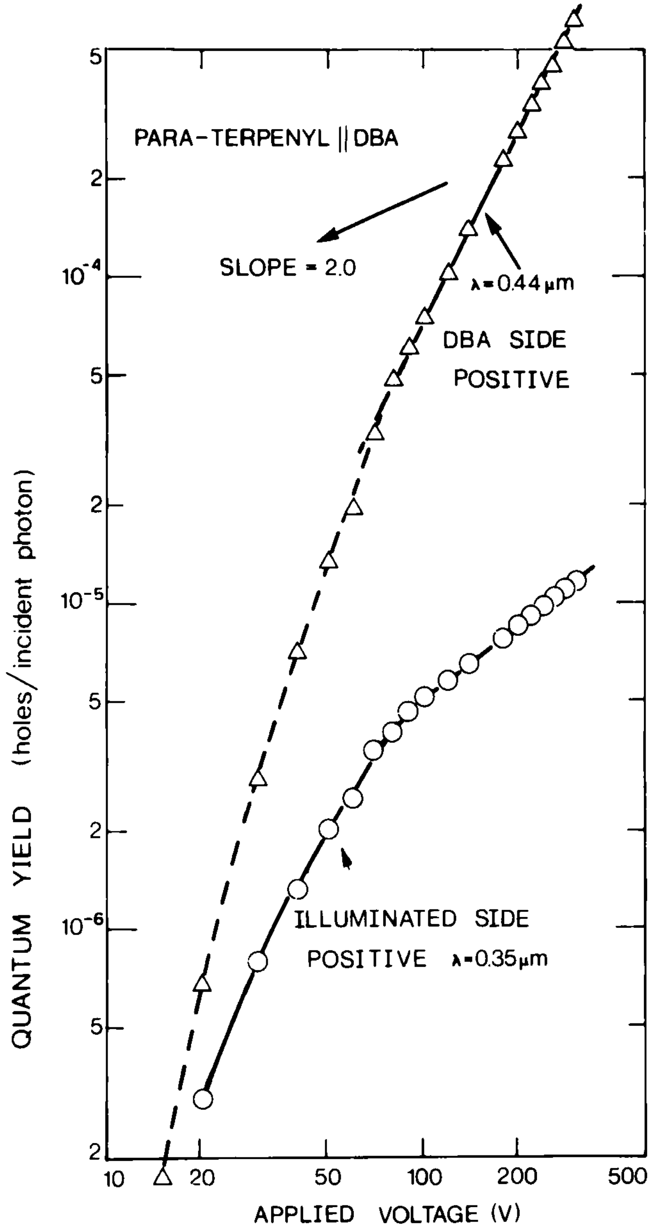

FIGURE 4 Room temperature log-log plots of photocurrent quantum yield vs voltage curves measured on a p-terphenyl || DBA system, with a positive polarity to illuminated front electrode at λ = 0.35 μm, and with a positive polarity to back electrode, which is sensitized with DBA, at λ = 0.44 μm. Same sample as in Figure 2.

than carrier injection from a metal or a semiconductor. When one of the latters is the carrier's source, an injected carrier is subject to an image force. The electric field dependence of the injection rate[16] depends on the energy and thermallization length of the injected carrier. Between insulators, no image force is present. The question arises whether an energy barrier exists for the hole injection process, or the holes are injected with excess energy. If the holes were injected with excess energy from the sensitized side it would mean that the layer constituted an *energy barrier* for holes moving in the *opposite* direction. In that case we would see that the hole photocurrents, obtained by illuminating the front electrode with light strongly absorbed in p-terphenyl, decay to zero as illumination proceeds. The holes injected from the front electrode side will accumulate near the back layer, and the space-charge thus produced will prevent further currents. This indeed happens with a DBA layer, but does not happen with DCA. As mentioned in the beginning of this section, there was a slight decay of the photocurrents also with a DCA layer, to about 85–90% of the initial value. That, however, was typical to some small trapping in p-terphenyl, and appeared to be similar with plain p-terphenyl samples. Thus, we may conclude that the DCA layer does not constitute an appreciable blocking to hole motion from the p-terphenyl to the DCA. The DBA layer, on the other hand, is blocking.

In Figure 5 the hole photoinjection quantum yield from DCA into p-terphenyl, η, is plotted vs $1000/T$ on a semi-log scale for various voltages. The quantum yield is seen to be practically temperature independent. If a considerable barrier existed for hole injection from the DCA layer into the p-terphenyl crystal, we would expect it to be thermally activated; but it is not. The activation energy which may be assigned to the data is only $\sim 5 \times 10^{-3}$ eV. Therefore we conclude that the valence bands of DCA and p-terphenyl are very close to each other, and no considerable barrier is produced for hole motion in either direction. The energy gap of DCA is also smaller than the p-terphenyl energy gap. It is clear, then, why no electron photoinjection takes place, and at the same time, why no barrier is produced for electron motion from p-terphenyl into DCA.

In Figure 6 the hole injection quantum yield from DBA into p-terphenyl is plotted vs $1000/T$ on a semi-log scale for various voltages. Unlike the p-terphenyl || DCA system, it is clear here that holes are injected into p-terphenyl with excess energy. The temperature dependence seen in the figure is thus free of any interface effects. At sufficiently high voltages ($\geqslant$ 80–100V, see Figure 4), such that every injected hole reaches the other end of the p-terphenyl crystal without trapping, the temperature dependence reflects the generation processes in the DBA layer proper. This is most likely the case for 300V (Figure 6) where the activation energy E_a is about 0.025 eV. For lower voltages it also might reflect the temperature effect on the trapping in the p-terphenyl bulk.

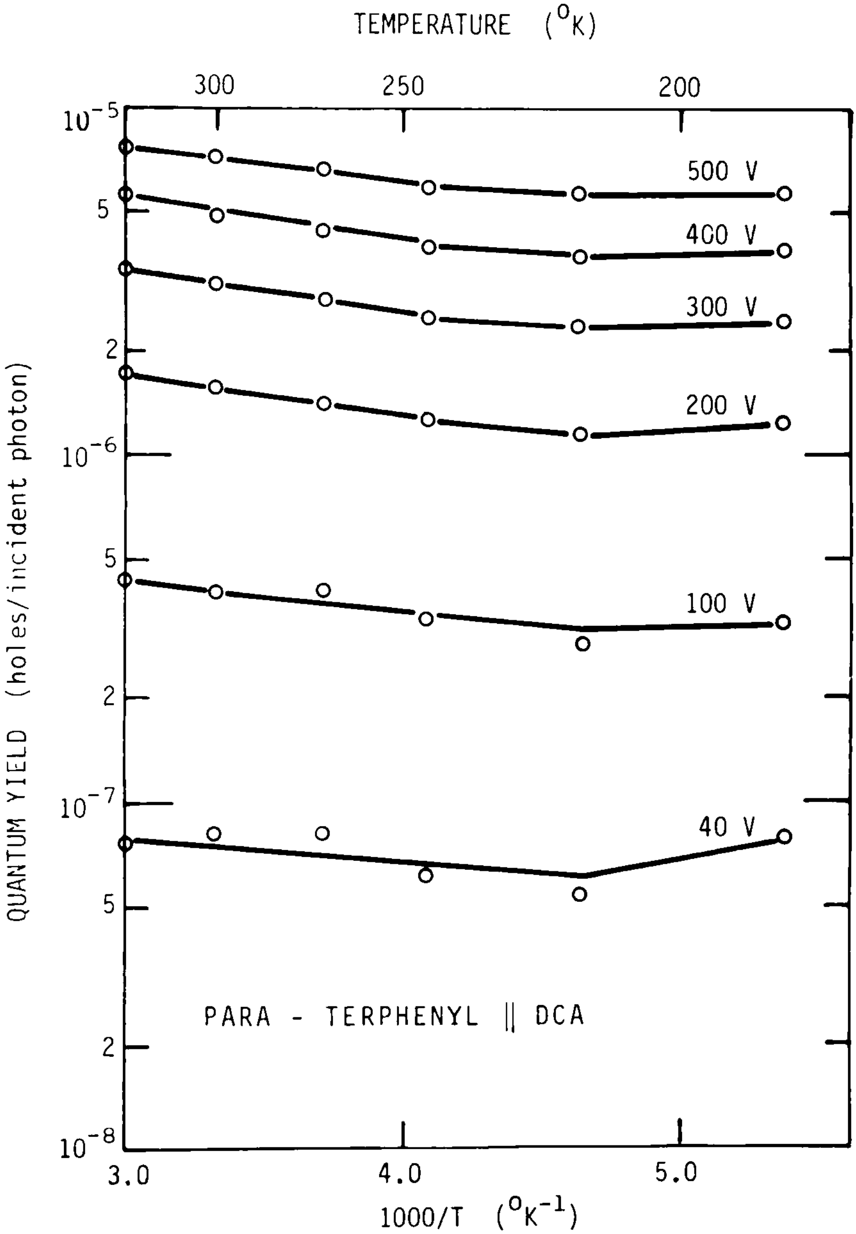

FIGURE 5 Semi-log plot of hole injection quantum yield vs 1000/*T* measured for various voltages with the DCA layer positive at wavelength $\lambda = 0.43\ \mu m$. Same sample as in Figure 1.

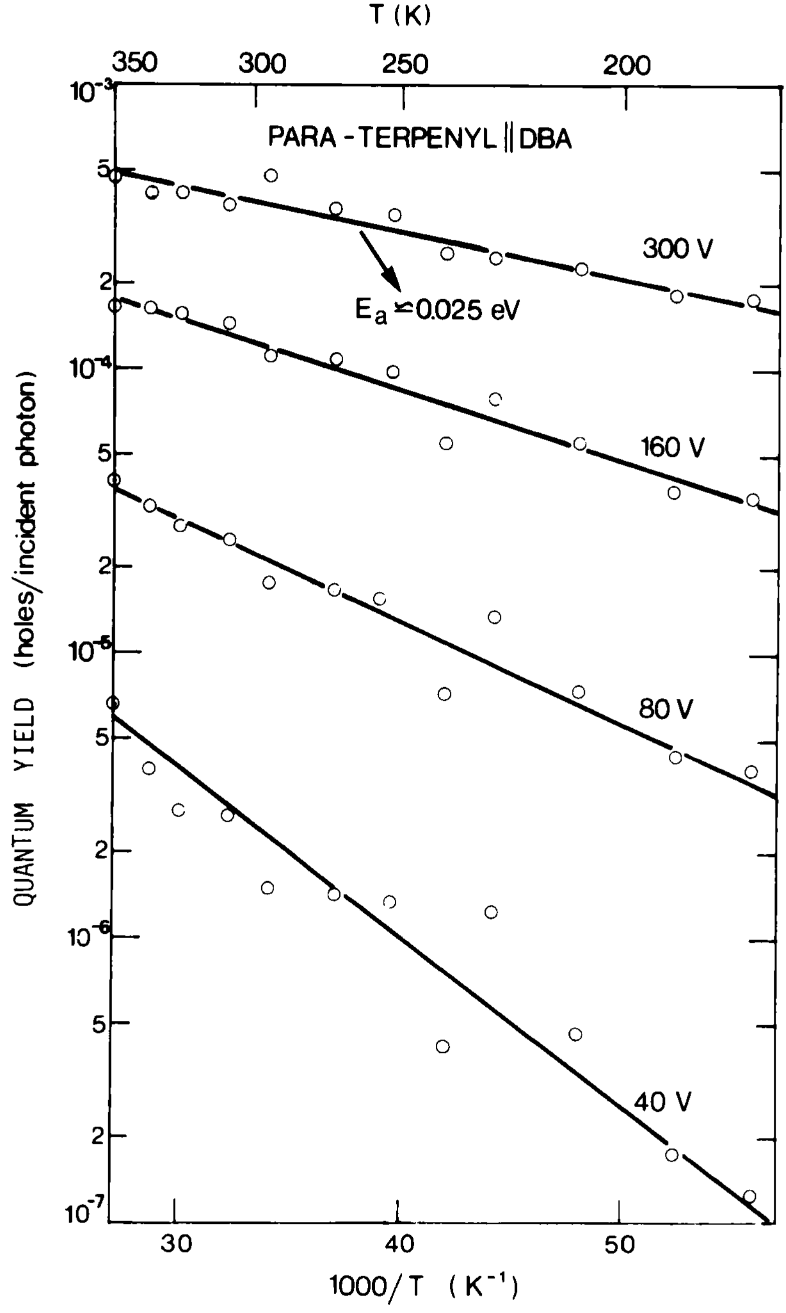

FIGURE 6 Semi-log plot of quantum yield vs 1000/*T* measured for various voltages with the DBA layer positive at wavelength $\lambda = 0.44$ μm. Same sample as in Figure 2.

Indeed, at low voltages (40V, 80V, Figure 6) one obtains stronger thermal activations than for the higher voltages.

As a result of the above we may conclude that the photocurrents observed in the spectral range $h\nu < 3.4$ eV for both DCA and DBA sensitizing materials reflect the hole generation rates at the sensitizing layers proper. For sufficiently high voltages, such that every hole that has been injected into the p-terphenyl crystal from the sensitizing layer, is collected at the counter electrode, the photocurrent quantum yield η is given by

$$\eta = \begin{cases} & \alpha_s(h\nu)l \ll 1: \\ \alpha_s(h\nu)f(E)l & \mu_p E\tau_p \gg l \\ \alpha_s(h\nu)f(E)\mu_p \mathrm{E}\tau_p & \mu_p E\tau_p \ll l \\ & \alpha_s(h\nu)l \gg 1: \\ f(E) & \mu_p E\tau_p \gg \alpha_s(h\nu)^{-1} \\ \alpha_s(h\nu)f(E)\mu_p E\tau_p & \mu_p E\tau_p \ll \alpha_s(h\nu)^{-1} \end{cases} \qquad (2)$$

where μ_p is the hole mobility in the sensitizing layer, $\alpha_s(h\nu)$ is the (photon energy dependent) singlet absorption coefficient, E is the external electric field, $f(E)$ is the (electric field dependent) fraction of singlet excitons generated in the sensitizing layer, which dissociate leaving a free hole, l is the layer thickness and τ_p is the hole lifetime in the sensitizing layer. The independence of the photoductivity quantum yield on the light intensity indicates that τ_p is governed by trapping, rather than by direct recombination. It is seen in Eq. (2), that in the photon energy range, where $\alpha_s(h\nu)l \ll 1$, the quantum yield should reflect the spectral variation of the absorption coefficient $\alpha_s(h\nu)$. This is indeed seen in Figures 1 and 2 in the DCA and DBA absorption tails, respectively. If the layer is sufficiently thick, then in the spectral region where $\alpha_s(h\nu)l \ll 1$, the photocurrent spectrum may saturate, provided the hole schubweg $\mu_p E\tau_p$ is longer then the light absorption depth $\alpha_s(h\nu)^{-1}$. The existance of structureless plateaus in the high photon energy regions of Figures 1 and 2 indicates that this is the case for both DCA and DBA layers. Furthermore, we may conclude that for both layers the schubweg $\mu_p E\tau_p$ is always longer than the layer thickness l. This is a result of the fact that the photocurrents have identical voltage dependences throughout the entire spectral range. When $\mu_p E\tau_p \ll l$, the voltage dependence in the tail region should show a higher power (Eq. 2).

As seen in Eq. (2), our quantum yields in the plateau regions are equal to the fraction of photogenerated singlet excitons $f(E)$, which dissociate leaving a free hole. This fraction is proportional to $E^{1.6}$ for DCA, and to E^2 for DBA. In crystalline β — DCA[6] the free hole generation efficiency varied linearly with the external field. In crystalline α — DCA, on the other hand, the photocurrents (studied with a surface-type cell) varied only linearly with the voltage,[5,7] which means that carrier generation was independent of the electric field. A similar result was obtained for a DBA crystal.[7] The difference in power dependences of $f(E)$ is probably due to the amorphous structure of the layers. So far no clear cut explanation has been provided for many cases in which the carrier generation rates varied with the electric field (very often linearly). Attempts to explain such behaviours were basically along two lines. One way was to modify Onsager's theory[14] by assuming high local mobility anisotropy.[17] This modification eliminates the zero-field positive intercept of Onsager's theory.[13] The other proposal, by Weisz *et al.*,[18] suggests that very fast carrier trapping takes place in the region where the photocarriers are produced. With increasing external electric field, the carriers' transit times across the generation layer become shorter, and more carriers escape the fast trapping region into the crystal bulk. The result is a linear electric field dependence. Neither of the above seems appropriate for our cases. Firstly, the layers are very likely amorphous; secondly, Weisz model is not applicable. The layer thickness is of the same order as the generation region width (the light penetration depth) for the wavelengths of measurement (beginning of the plateaus). The present results conform to a general behaviour, measured for many materials, including for DCA,[6] but no more insight is gained concerning the field effect on carrier generation processes in DCA and DBA.

Temperature variations may affect the quantum yield η in the plateau region by the influence on the fraction of dissociating photogenerated singlet excitons $f(E)$. In both the DCA α and β crystalline forms, the photoconductivity was reported to increase with temperature. In β- DCA it has increased by an order of magnitude[6] between 240° K to 320° K. In α-DCA and in DBA crystals, activation energies of 0.1–0.2 eV were reported.[7] When measurements were taken with the single crystal forms in surface-type cells,[5,7] the separation between the electrodes was of the order of a millimeter. It is then likely that the schubweg $\mu_p E \tau_p$ was smaller than the distance between the electrodes, L. In that case the photocurrents are proportional to the ratio $\mu_p E \tau_p / L$. Temperature variations of μ_p and τ_p could then be of major importance. The rather small temperature dependences of the photocurrents in our sensitizing layers may be explained by their small thickness ($\sim 1 \mu$m), as well as by their different structure (amorphous or policrystalline).

When the crystals were turned such that the sensitized sides were directly illuminated, basically all the characteristics, namely, spectral shape, voltage and temperature effects were the same. Of course, photocurrents in the photon energy range 2.6 eV–3.4 eV were seen only when the front electrode was positive, and were absent in the opposite polarity. Also, the photocurrents due to light absorption in the p-terphenyl were almost absent, indicating that only a small proportion of the light reached the p-terphenyl sensitizer interface for $h\nu > 3.4$. Finally, there was no indication of sensitization in the fundamental light absorption range of p-terphenyl which could be interpreted as p-terphenyl singlet dissociation at surface states. Attempts to see such effects were made by applying extremely thin layers of the sensitizers, about 100–200Å in thickness, and illuminating the samples through the layers. For such layers, sensitized photocurrents in the long wavelength range were detectable, but much smaller, and in the fundamental singlet-singlet absorption of p-terphenyl ($h\nu > 3.4$ eV) they were the same as for the plain p-terphenyl crystal.

IV SUMMARY

This work presents a study of photoconductivity sensitization of p-terphenyl by 9,10-dichloroanthracene or 9,10-dibromoanthracene layers applied to the crystal surface. The photoconductivity response was extended to wavelengths as long as 0.46 μm only when the sensitizing layer was positively biased. The sensitized photocurrents varied as the 1.6 power of the external voltage for DCA and as the 2.0 power for DBA. With DCA, the photocurrents were practically insensitive of temperature variations in the range 180–330°K. With DBA, on the other hand, the photocurrents showed slight thermal activation, with an activation energy of about 0.025 eV. The data suggest that the photocurrents arise from holes, initially generated in the sensitizing layer via dissociation of its photogenerated singlet excitons, and then injected into the p-terphenyl crystal. The energetic locations of the valence bands of DCA and p-terphenyl are very close, and no energy barrier is established for hole motion at their interface. The DBA valence band, on the other hand, is lower in energy. Holes generated in p-terphenyl are blocked by a DBA layer, and holes generated in DBA enter the p-terphenyl with excess energy.

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