

Charge Transport Generated by Electron Beam in Molecularly Doped Polymers

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Charge carrier transport in a typical molecularly doped polymer has been studied by the time-of-flight and radiation induced conductivity methods. In the time range bracketing the conventional transit time a strong disagreement between two sets of data has been found. Unlike time-of-flight signal that rather closely reproduces all salient features reported in literature, the radiation induced conductivity transient lacks the plateau. In fact, it exhibits a strict power law decay $t^{-0.5}$ changing at long times to t^{-1} or $t^{-1.5}$ patterns due to bimolecular recombination or true transit time effects, respectively. At short times, both transients are described by the same $t^{-0.5}$ dependence. These facts suggest the dispersive rather than the Gaussian charge transport.

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Introduction

Molecularly doped polymers (MDPs) are convenient model systems to study charge carrier hopping transport in molecular solids.^{1–3} As a rule, the time-of-flight (TOF) method is used. The plateau on the TOF transient and its scaling with sample thickness are meant to prove specifically the Gaussian mode of charge transport. Accordingly, Bässler's disorder theory is used to describe these results.⁴

There is yet another approach to probe charge carrier transport in dielectrics, notably, the radiation induced conductivity (RIC). It proved exceptionally useful in the study of polymers that usually lack photoconductivity and are unsuitable for the standard TOF technique. A wealth of data now exists about RIC behavior of polymers.^{5–8} The multiple trapping (MT) theory known also as the Rose–Fowler–Vaisberg theory describes these data rather adequately.⁹

It is gratifying that the model photoconducting polymer poly-N-vinylcarbazole (PVK) has been extensively investigated by both of these methods⁹ and the detailed analysis of these comparative studies is now available.¹⁰ In a sense, PVK may be regarded as intramolecularly doped polymer and as such bears a great resemblance to N-isopropylcarbazole-doped polycarbonate.^{11,12}

It is our routine practice to study charge carrier transport in polymers by TOF as well as RIC methods. However, in our case the TOF method employs low energy

electrons (~5 keV) rather than a laser pulse to produce a sheet of charge carriers close to the front surface of the sample.¹⁴

In this article we summarize these results, then proceed to present the results of comparative studies for a typical MPD (RIC versus TOF) and finally give a discussion on the subject.

RIC: Experiment and Theory

The question immediately arises as to whether RIC data obtained with high energy radiation pulses could be of any value for elucidating the transport of electrons or holes photogenerated or contact injected into MPD layers used in photoreceptors and imaging devices. Indeed, despite appreciable differences in charge carrier generation efficiencies the transport properties of free charges emerging after dissociation of initial ion pairs are thought to be representative of a polymer system and not dependent on the generation mode.

The main transport characteristic derived from RIC studies is the effective mobility $\mu_{eff}(t)$. This material parameter is time dependent and by definition is proportional to the current $j(t)$ flowing in a sample after δ -pulse irradiation under condition that the applied electric field is constant and uniform while no charge loss due to bimolecular recombination or exit takes place. Usually RIC is a unipolar phenomenon and $\mu_{eff}(t)$ refers specifically to the majority carriers (in PVK, holes). Moreover, radiation chemistry allows us to evaluate (if only approximately) the concentration of the pulse generated charges and also the mobility.

It has been revealed that the effective mobility exhibits a strict power law decay $t^{-(1+\alpha)}$ over almost five decades in time (from 10 μ s to ~ 1 s at 293 K in PVK), the dispersion parameter α varying between 0.05 and 0.6 in

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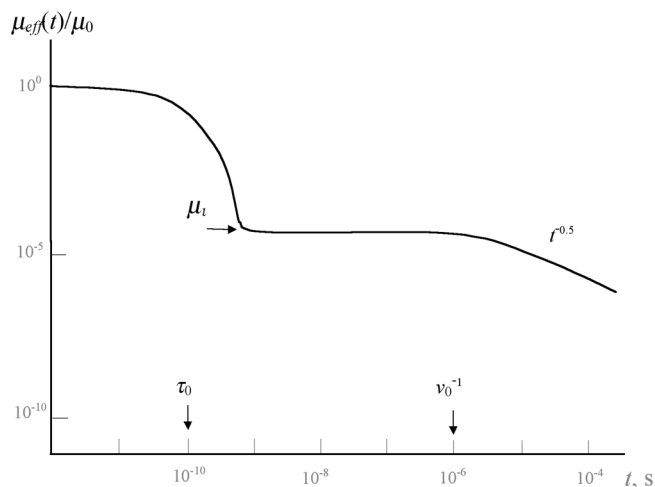


Figure 1. General representation of the effective mobility. Parameters of the model polymer are as follows: $\alpha = 0.5$, $\nu_0 = 10^6 \text{ s}^{-1}$, $\mu_0 = 10^{-5} \text{ m}^2/\text{V} \times \text{s}$ and $\tau_0 = 10^{-10} \text{ s}$.

different polymers.^{6,10} Also α seems to be proportional to the absolute temperature as required by the multiple trapping theory for an exponential trap distribution.¹⁵

In addition to α there are two other parameters characterizing charge carrier transport and the effective mobility in particular. One is the product of the microscopic mobility of majority carriers in extended states (μ_0) and lifetime before trapping (τ_0), also called the *Schubweg*, $\mu_0\tau_0$. The other is the release rate (or frequency factor) of the trapped charges ν_0 . The latter has a specific physical meaning and can be directly determined using short pulses of radiation⁹ (see below).

All these parameters combine to define the so-called initial mobility μ_i which denotes the effective mobility of majority carriers immediately after their trapping. Over time range $\tau_0 \leq t \leq \nu_0^{-1}$ $\mu_{eff} \approx \mu_i$, and is almost constant (Fig. 1). It is this property of the effective mobility that allows ν_0 to be directly measured.⁹

Theoretical treatment of the charge carrier transport in the framework of the MT model is greatly facilitated by the fact that there exists a simple relationship between $\mu_{eff}(t)$ and the so-called τ function first introduced by Arkhipov and co-workers.^{16,17}

$$\mu_{eff}(t) = \mu_0 \frac{d\tau(t)}{dt} \quad (1)$$

For the case of the exponential trap distribution

$$\tau(t) = \frac{\tau_0(\nu_0 t)^\alpha}{\alpha\gamma(\alpha, \nu_0 t)} \quad (2)$$

where $\gamma(a, x)$ is the incomplete gamma function.

For low dose rate irradiation the effective mobility may be regarded as a Green function of the corresponding differential equations of the MT model and will be used later to calculate the current transients.

It has been conclusively shown that charge carrier transport in polymers (including PVK) is dispersive^{9,18} This conclusion agrees with the fact that polymers are disordered molecular solids. MDPs are evidently even more disordered as they lack the short-range order of the

homopolymers. At small doping levels the positional disorder of the dopant molecules approaches gas disorder.

Experimental Procedure

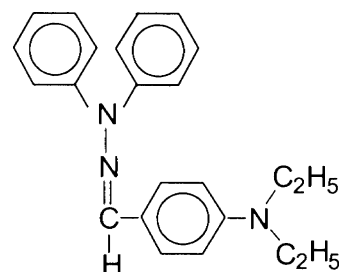
RIC investigations have become a routine laboratory test since the advent of electron guns capable of delivering pulses of 3 to 100 keV electrons as demonstrated by Hirsch as early as 1972^{19,20} using both (RIC and TOF) techniques. These radiation sources are particularly appropriate for studying charge carrier transport in thin polymer films. Our own studies using them date back to 1982.⁶

In the present work we used the electron facility ELA-50 to produce pulses of 3 to 50 keV electrons (beam current up to 1 mA, pulse length from 10 to $10^3 \mu\text{s}$ with rectangular pulse shape). The facility could be operated in a single pulse mode as well as in a truly continuous irradiation regime. Irradiation of polymer samples took place in vacuum ($\sim 3 \times 10^{-2} \text{ Pa}$) at room or elevated (up to 100°C) temperatures.

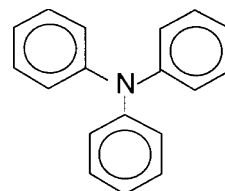
Films of MDP tested were coated on Al disks (40 mm diameter, 100 μm thickness) from a solution, dried and provided with an evaporated Al electrode of 23 mm in diameter on one side only. Film thickness was in 4 to 25 μm range. Dosimetry of the electron beam has been performed by the Faraday cup technique.

Two types of MDP were used. A full program of investigations was realized only with DEH-doped polycarbonate (PC) and not with TFA-doped PC. Dopant loading was 30 wt% in both cases. Chemical structure of these compounds is as follows:

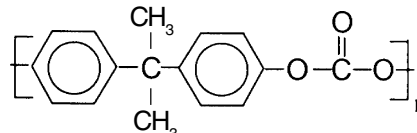
Hydrazone DEH



Triphenylamine



Polycarbonate (PC)



These two MDPs are considered to be representative of the whole class of molecularly doped polymers intensely investigated recently.¹⁻⁴

Special attention has been taken to insure a small signal irradiation thus minimizing possible recombination,

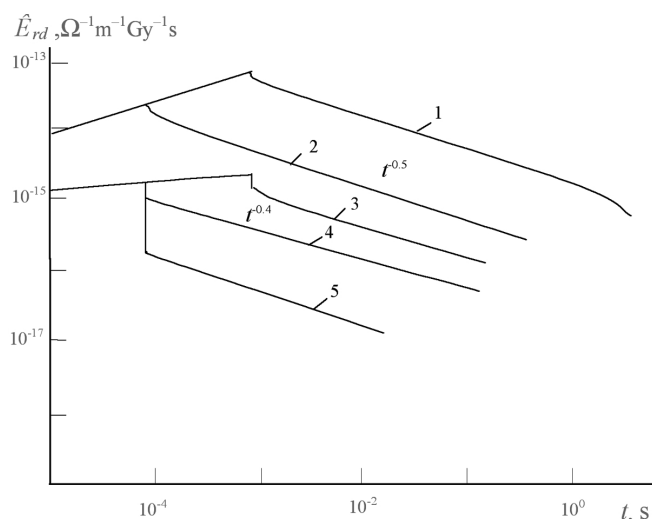


Figure 2. RIC transient in DEH (1,2), and TFA (3,4) doped PC. Curve (5) describes RIC in pure PC. Pulse length (ms) 0.1 (2,4,5) and 1.0 (1,3). Applied electric field 2×10^7 V/m ($T = 293$ K).

dose and field distortion effects. It has been found that high temperature treatment of MDP samples in air at 80°C for 2 to 3 hours completely anneals dose effects.

Experimental Results

As far as the DEH system is concerned TOF measurements largely confirmed the earlier results relating to the plateau region (its length and field dependence).^{21–23} As for TFA-doped PC it was found that TOF signal was too small to yield meaningful results. The reason for this will be discussed later.

It should be stressed that while RIC response of the both tested MDPs was highly reproducible from sample to sample within 20%, the TOF behavior of 10 DEH-doped samples (three different batches) in the plateau region proved exceptionally irregular in a quantitative as well as a qualitative way, in particular, the TOF signal shape depended on the number of electron pulses.

Standard irradiation conditions for RIC studies were as follows: the pulse length $t_0 = 1$ ms, the applied electric field $F_0 = 2 \times 10^7$ V/m, the dose rate $R_0 \approx 4 \times 10^4$ Gy/s and $T = 293$ K. The RIC response due to hole migration through dopant molecules proper can approximately be obtained by subtracting the RIC transient of the binder polymer (PC in our case) from the total detected current signal. These procedures could be easily carried out in all cases except TFA-doped PC during pulse irradiation itself (Fig. 2).

It is found that RIC in DEH-doped PC measured at the end of the pulse and reduced to a unit dose rate, K_{rd} (1 ms) = $8 \times 10^{-14} \Omega^{-1} \times m^{-1} \times Gy^{-1}s$ and is almost 50 times larger than in TFA system while α is larger on the contrary in the latter (0.6 versus 0.5). This finding explains probably our failure to measure TOF transient in TFA-doped PC. Judging by K_{rd} value, DEH-doped PC may be placed between polystyrene ($\sim 6.5 \times 10^{-14} \Omega^{-1} \times m^{-1} \times Gy^{-1}s$) and PVK ($\sim 3.5 \times 10^{-13} \Omega^{-1} \times m^{-1} \times Gy^{-1}s$). Again, like these polymers it features a constant dispersion parameter over a time range of 5 decades (from $10 \mu s$ to ~ 1 s). At longer times this decay pattern is obscured by bimolecular recombination or transit time effects.

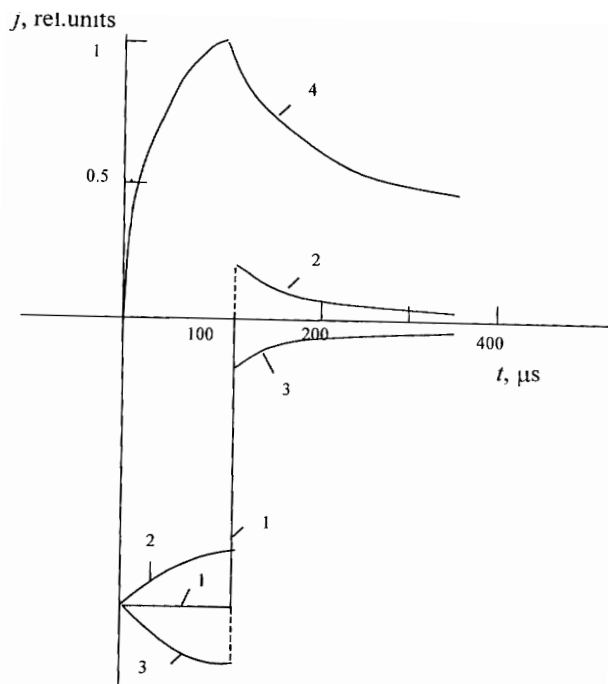


Figure 3. Early part of TOF (2,3) and RIC (4) transients in DEH-doped PC. Curve (1) in a TOF experiment corresponds to a radiation pick-up signal at zero applied voltage. Curve (4) is given not to scale. Transients (2) and (3) are registered for different polarities of the applied voltage.

The fact that we deal with free holes escaping geminate recombination in ion pairs is confirmed by the strong dependence of K_{rd} on F_0 : $K_{rd} \propto F_0^{1.35}$ in the field range from 1 to 2×10^7 V/m (the radiation induced current $j_{rd} \propto F_0^{2.35}$, stronger than quadratic dependence).

At 293 K the temperature coefficient $d \ln K_{rd}/d(1/T)$ of K_{rd} is only ~ 0.1 eV, then increases and reaches a value of ~ 0.4 eV at 355 K which is well above the glass transition temperature (~ 337 K according to Ref. 22). All temperature changes are completely reversible. Note that at 355 K, α is approximately 0.6.

Once it has been established that charge carrier transport in DEH-doped PC is indeed dispersive in the time domain ($10 \mu s - 1$ s) bracketing the usual values of TOF transit times reported in literature^{21–23} it became imperative to investigate TOF transients in this system over the widest time range.

The TOF transient at early times ($t \leq 3t_0$, $t_0 = 0.1$ ms) is simply proportional to the RIC signal but is approximately 50 times smaller, all other factors unchanged (for both polarities of the applied field, see Fig. 3). Accordingly, very strong field effects persist. Then, as the observation time increases, there appears a clear asymmetry as regards the field polarity. At last the plateau region is reached and only hole transient current persists. It is here that sample-to-sample scatter of plateau characteristics (length, presence or absence of the cusp) has been observed. In most samples tested (Fig. 4) we saw clear shoulder on the current transient waveform which is usually taken to signify the non-dispersive transport of holes across the film to obtain the drift mobility.^{1–4,21–23}

It should be emphasized that the RIC current transient measured on the same sample under the same con-

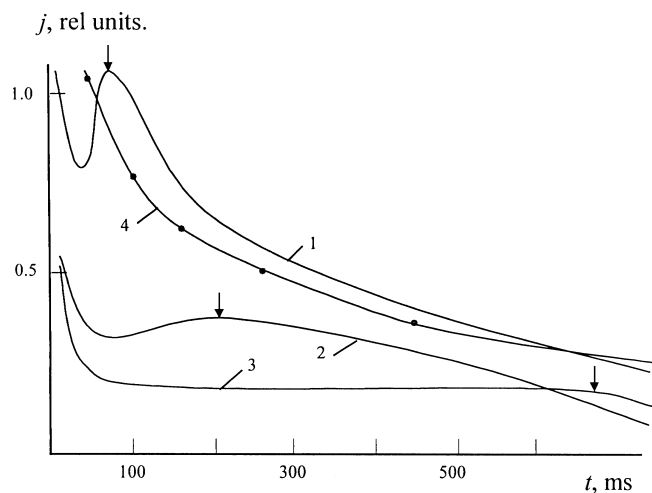


Figure 4. TOF (1,2,3) and RIC (4) transients in DEH-doped PC at the plateau region ($t_0 = 0.1$ ms, 293 K, $L = 25$ μm). Applied electric field (in units of 10^7): 1.7 (1,4), 1.1 (2) and 0.85 (3). The arrows indicate transit times t_{tr}^* .

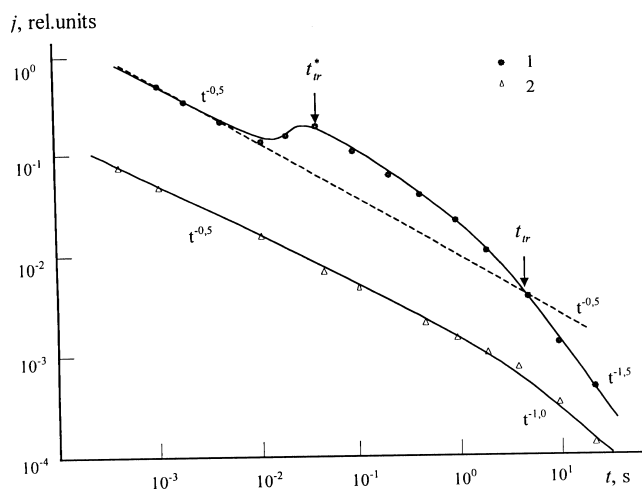


Figure 5. TOF (1) and RIC (2) transients taken in DEH-doped PC to compare their behavior around t_{tr}^* . While TOF curve shows a clear plateau with a cusp the RIC transient follows $t^{-0.5}$ power-law in this region. Pulse length 10 μs .

ditions shows no sign of a plateau (Figs. 4 and 5). The current decays as $t^{-0.5}$ over the whole time region where the plateau appears on a TOF curve. Moreover, once current decay of the TOF transient is observed well beyond the tail section another transit event may be detected when at $t \geq 1$ s the current decay takes on $t^{-1.5}$ power law dependence. At high temperatures (>330 K) the plateau altogether disappears from the TOF current transients (Fig. 6).

In view of these results we propose a simple procedure to define another transit time t_{tr} by intersecting the early part of a TOF decay curve ($t^{-0.5}$) with the one at long times ($t^{-1.5}$) using the $\log j - \log t$ plot. As expected, $t_{tr} \gg t_{tr}^*$, the latter quantity being conventionally defined (Fig. 5). The dependence of t_{tr}^* and t_{tr} on changing film thickness, the applied electric field and temperature is presented in Table I, t_{tr} varying in accordance with the multiple trapping theory.^{25,26}

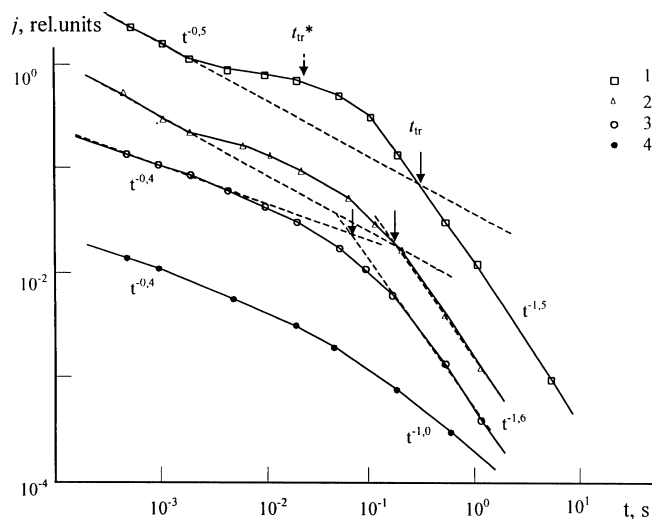


Figure 6. TOF (1,2,3) and RIC (4) transient in DEH-doped PC. Temperature of the sample (K): 308 (1), 333 (2) and 353 (3,4). Sample thickness 4.3 μm , applied voltage 100 V. Arrows indicate appropriate transit times (see text). The curves are displaced arbitrarily on the Y-axis to underline effects relating to transit time.

TABLE I. Summary of Experimental Results for DEH-Doped PC as Obtained by TOF Technique

Experimental conditions	Transit time, ms		Drift mobility, $\text{m}^2/\text{V}\cdot\text{s}$	
	t_{tr}^*	t_{tr}	μ_{dr}^*	μ_{dr}
1. Temperature 300 K $L = 4.3$ μm $F_0 = 2.3 \times 10^7$ V/m	40	300	4.7×10^{-12}	6.2×10^{-13}
$L = 18$ μm $F_0 = 2.8 \cdot 10^7$ V/m	40	4×10^3	1.8×10^{-11}	1.8×10^{-13}
$L = 7$ μm $F_0 = 1.45 \times 10^7$ V/m	—	2×10^3	—	2.5×10^{-13}
2. Temperature 333 K $L = 4.3$ μm $F_0 = 2.3 \times 10^7$ V/m	—	160	—	1.1×10^{-12}
3. Temperature 353 K $L = 4.3$ μm $F_0 = 2.3 \times 10^7$ V/m	—	80	—	2.2×10^{-12}

It should be emphasized that as expected μ_{dr}^* values found in the present work agree favorably with those cited earlier in literature. Our doping level of DEH corresponds to ~ 1.05 mole/dm³. For this very system, μ_{dr}^* according to Ref. 22 equals $4 \times 10^{-11} \text{ m}^2/\text{V} \times \text{s}$ for 298 K and 2×10^7 V/m for unspecified film thickness (10 to 50 μm). This figure is slightly less ($\sim 3 \times 10^{-11} \text{ m}^2/\text{V} \times \text{s}$) for 1.14 mole/dm³ loading in the same conditions (film thickness ~ 20 μm).²³

For a 50 wt% DEH-doped PC (1.7 mole/dm³) film (18 μm thick) this value is even smaller ($\sim 2 \times 10^{-11} \text{ m}^2/\text{V} \times \text{s}$) at 294 K and 2.2×10^7 V/m.²¹ Two of these works used the material itself as the photogenerator^{21,22} while the third²³ employed a special 1 μm thick charge generation layer.

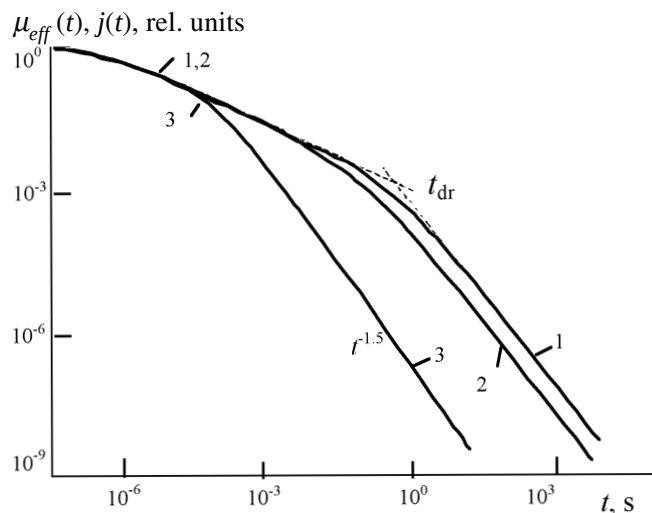


Figure 7. TOF (1,3) and RIC (2) transient accounting for hole transit across the 20 μm polymer film (1,2) and across the irradiated layer 0.6 μm thick (3). This last case corresponds to the majority carriers being drawn to the front (irradiated) electrode as proposed earlier in Ref. 20. Curve (1) illustrates the fact that to define t_{tr} accurately one needs to register the current transient over almost 5 orders of magnitude around the expected transit time.

Discussion

As a matter of fact, both RIC and TOF methods are widely used to measure the drift mobility of charge carriers in dielectric media, be it a gas, liquid or solid.²⁷ Most, if not all, of these results refer specifically to the equilibrium drift mobility which is independent of time. The case of dispersive charge transport has been considered by Hughes²⁸ in relation to RIC and TOF experiments. We have extended this analysis by applying accurate numerical calculations using Eqs. 1 and 2.

The fitting procedure not included here and found elsewhere^{29,30} gives the following results for MT model parameters for DEH-doped PC: $\alpha = 0.5$, $v_0 = 10^6 \text{ s}^{-1}$, and $\mu_0\tau_0 = 10^{-15} \text{ m}^2/\text{V}$. For $\mu_0 = 10^{-5} \text{ m}^2/\text{V} \times \text{s}$ we have $\tau_0 = 10^{-10} \text{ s}$. These data were used in Fig. 1.

First of all, we would like to stress that unlike $\mu_{eff}(t)$ the drift mobility μ_{dr}^* (or μ_{dr}) is an ill-defined quantity (see Fig. 7). It can be seen that to find t_{tr} accurately enough one has to register $j(t)$ over a time domain covering 5 decades with t_{tr} at its center (the analysis refers to an ideal TOF geometry implying generation of holes as a delta-function of both coordinate and time).

Relaxing the coordinate restraint we introduce the generation layer with a finite thickness (for 6 keV electrons about 0.6 μm) and a specific depth dose profile. Now the TOF current transients due to majority carriers are expected to be seen for both polarities of the field, one being much shorter than the other (Fig. 7). This explains the observed asymmetry mentioned earlier.

The focus of our numerical analysis consists in comparison between RIC and TOF electron beam induced current transients in the plateau region. From Fig. 7 it is clear that the transit event is equally well detected by both these methods though the kink on the $\log j - \log t$ plot of the RIC transient is less conspicuous. The absence of any mark on the RIC transients as to the exit of charge carriers at times $t \approx t_{dr}^*$ serves only to suggest that there is no real transit by the bulk generated holes

which dominate the RIC signal. In our view the plateau is specifically a TOF phenomenon.

It is well known that fundamental results in photo-physics (Onsager theory of geminate recombination, Langevin mechanism of bimolecular recombination, dispersive charge carrier transport in PVK and As_2Se_3) have been obtained by employing bulk photo-conductivity measurements instead of the near surface generation of the photocarriers using UV laser pulses.^{31–35} Pulse photoconductivity results are greatly missed in MDPs.

On the weight of all our previous studies of RIC in polymers including PVK and specifically molecularly doped polymethylmetacrylate (PMMA) as well as polyvinylchloride (PVC)³⁶ and taking into account the striking similarity between RIC response of DEH-doped PC and that of PVK and polystyrene we perceive the dispersive rather than Gaussian hole transport in this MDP under conditions used in this study.

In our previous work³⁶ with molecularly doped PMMA and PVC it has been shown that only electron donors were active in PVC and electron acceptors in PMMA. No great differences were detected as to the specific nature of the compounds used in each group (donors or acceptors). On the contrary, large variations of hole drift mobilities (TOF technique only) were found in Ref. 23 concerning electron donors used to dope PC or polystyrene. It just may be that this effect explains the low activity of TFA molecules in PC compared to DEH as evidenced by our RIC measurements.

Model Considerations

Now we would like to make some general remarks about the microscopic picture of the hole transport in DEH-doped PC. Our approach to the problem of charge carrier transport in homogeneous polymers assumes carrier hopping on a densely packed manifold of transport (hopping) centers retaining the short range order of the respective single crystal.^{10,37} The central idea which distinguishes it from the Bässler's disorder theory is that the energy scatter concerns only a small fraction ($\leq 1\%$) of hopping centers which begin to act as traps. The majority of isoenergetic centers build up a transfer band (analogue of the conduction band or extended states) with microscopic mobility μ_0 equal to that in the respective single crystal. The origin of the energy scatter (and traps themselves) is ascribed to the elementary voids of the fluctuation free volume³⁸ forming an association with the normal hopping centers. Trap release rate (or frequency factor v_0) have been found to be modulated by molecular relaxations (specifically, hindered rotations) at elevated temperatures. To insure $\alpha \approx \text{const}$ over a wide range of time, trap energy distribution is taken to be exponential.

These considerations allow one to reconcile hopping transport of charge carriers with the multiple trapping formalism (and RFV model based on it) that describes RIC of polymers so well.⁹ Close analogy of RIC response of DEH-doped PC and that of PVK and polystyrene suggests that the above considerations are possibly applicable to it.

Indeed, in this case short-range order in the packing of dopant molecules may be lacking. Nevertheless, if the real reason for their energy scatter is the fluctuation free volume then we again come to the concept of the transfer band, this time in the form of the diffusion cluster in the bond disordered system.³⁹ The low value of the frequency factor ($\sim 10^6 \text{ s}^{-1}$) is rather surprising but in line with most common polymers including PVK. To

understand this one needs information about relaxation dynamics (translational as well as rotational) of small guest molecules (DEH, TFA etc.) in rigid polymer matrices. The case of lightly doped MDPs is even more complex and merits special consideration. Effects of spatial correlation due to charge–dipole interactions are to be properly accounted for as well.^{40,41}

We eagerly await future pulse photoconductivity studies in a number of MDPs to supplement our electron beam measurements.

Conclusions

Molecularly doped polymers are disordered polymer systems if only because of positional disorder of the dopant molecules. By analogy with common polymers (photoconductive PVK included) charge carrier transport in MDPs is expected to be dispersive rather than Gaussian. To prove this assertion we performed extensive studies on DEH-doped PC using both RIC and TOF techniques. Except plateau region of the TOF transients excited by electron beam all observations could be consistently explained in terms of the MT model incorporating the multiple trapping formalism.

So, TOF excited by electron beam and RIC lead to contradictory results regarding hole transport in MDP. We believe that only parallel measurements of TOF and bulk conductivity generated by light and electron beam will help to understand the nature of this effect.

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