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# Time of flight results for molecularly doped polymers revisited

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#### Abstract

We examine the published data concerning the shape of the current transients obtained by the time of flight (TOF) technique for molecularly doped polymers and polyvinylcarbazole (PVK) and compare it with the predictions from the existing theories of charge carrier transport in disordered organic solids. We show that TOF current shapes frequently run contrary to theoretical predictions. Plateau appearances on TOF curves may or may not mean the equilibration of the charge carrier transport. Using both TOF and TOF-2 (uniform generation of charge carriers) techniques we demonstrate that hole transport in polycarbonate doped with p-diethylaminobenzaldehyde-diphenylhydrazone (DEH) and in PVK is indeed dispersive despite the fact that samples of these polymers equipped with an a-Se generation layer produce TOF curves with a plateau.

# 1. Introduction

It is now recognized that charge carrier transport in molecularly doped polymers (MDP) involves a highly non-equilibrium early stage when the average (effective) mobility  $\mu_{ef}(t)$ decreases over time. The Gaussian (non-dispersive) transport with constant carrier mobility  $\mu$  and diffusivity *D* is expected to set in some time later, marking a dispersive to Gaussian (DG) transition.

There is no first-principles theory of electronic transport in MDP and all existing models derive from the time of flight (TOF) experiments. In a TOF study one measures the current in a sample (thickness *L*) with two non-injecting electrodes designed to secure a constant and uniform electric field  $F_0$ . The current is due to a drifting sheet of one-sign carriers pulsegenerated at the front electrode. A small signal regime is always employed in order to exclude any field perturbation. Various operational procedures are used to define the time of flight  $t_{tr}$  depending on the theoretical model used to interpret experimental data, so  $\mu_{dr} = L/(t_{tr}F_0)$ .

Dispersive transport with  $\mu_{ef} \propto t^{-1+\alpha}$  (0 <  $\alpha$  < 1.0) leads to an unusual relationship [1–3]:

$$\mu_{\rm dr} \propto (F_0/L)^{(1/\alpha)-1} \tag{1}$$

even if material parameters do not depend on the field. The presence of L in equation (1) is quite unusual. Non-

equilibrium drift mobility is only a convenient means to qualify MDP for a particular application [1]. On several occasions this proviso has been overlooked and the above field dependence explained in terms of steady-state microscopic theories (as, for example, in [4]).

These considerations lend importance to verification procedures intended to ascertain whether one is really dealing with Gaussian transport and whether proper corrections have been made to account for the initial non-stationary stage of the carrier transport.

To achieve this aim we present first the theoretical treatment of the TOF experiment to obtain equilibration criteria, then critically review the published data and finally give recommendations for future work.

# 2. Theory

Two major approaches for describing electronic transport in MDP are as follows: the continuous-time random walk (CTRW) [1] and multiple-trapping (MT) approach [2], where both stress its non-stationary character, and the Gaussian disorder model (GDM) approach [5–7], underlining on the other hand its steady-state features. Since we focus on the TOF current shape it does not matter whether the transport is quasiband (MT) or hopping (CTRW and GDM).



**Figure 1.** Time of flight curves for the true Gaussian transport.  $L = 2 \times 10^{-5} \text{ m}$ ,  $F_0 = 10^8 \text{ V m}^{-1}(1)$ ,  $L = 2 \times 10^{-6} \text{ m}$ ,  $F_0 = 10^7 \text{ V m}^{-1}(2)$  and  $L = 2 \times 10^{-7} \text{ m}$ ,  $F_0 = 10^6 \text{ V m}^{-1}(3)$ .  $\tilde{\mu} = 10^{-11} \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1}$ ,  $\tilde{D} = 2.5 \times 10^{-12} \text{ m}^2 \text{ s}^{-1}$  and  $t_{\text{tr}}$  equals 0.02 s in all cases.

#### 2.1. True Gaussian transport

Once carriers move with constant  $\tilde{\mu}$  and  $\tilde{D}$  (presumably, this is the case in crystalline solids) the TOF current is given by the well-known expression [7]

$$j(t) = \frac{\sigma_0 e \tilde{\mu} F_0}{L} \left[ 1 - \frac{1}{2} \operatorname{erfc} \left( \frac{L - \tilde{\mu} F_0 t}{\sqrt{4 \tilde{D} t}} \right) \right]$$
(2)

where  $\sigma_0$  is the planar carrier density, *e* is an electronic charge and erfc(*x*) is the complementary error function. Kinetic coefficients  $\tilde{\mu}$  and  $\tilde{D}$  are expected to be related by Einstein's formula. Time of flight  $t_{tr} = L/(\tilde{\mu}F_0)$  marks the moment  $t_{1/2}$ at which the current drops to half its initial (plateau) value and  $t_{tr} = t_{1/2}$ .

The relative width  $W = (t_{tr} - t_0)/t_{tr}$  is often used to characterize the transit time dispersion. Here,  $t_0$  is the 'usual' transit time obtained from the intersection of tangents to the TOF transient on linear axes (figure 1). The current curve is normalized by its initial (plateau) value while the time is plotted in units of  $t_{1/2}$ . It is important that the tangent goes through  $j(t_{1/2})$ , as shown on the figure (curve 2).

through  $j(t_{1/2})$ , as shown on the figure (curve 2). According to [6]  $W = \left[\frac{\pi D}{\mu F_0 L}\right]^{1/2}$  which reduces to  $\left[\frac{\pi kT}{eF_0 L}\right]^{1/2}$  once the Einstein's relation holds. Under typical experimental conditions ( $F_0 = 2 \times 10^7$  V m<sup>-1</sup>,  $L = 20 \ \mu$ m and 290 K) the equilibrium  $\tilde{W} \approx 0.014$ .

For curve 1 in figure 1 the computed W (0.0198) compares rather favorably with the graphically found one (0.021). Curves 2 and 3, which clearly differ, refer to  $F_0$  and L, each reduced, by factors of 10 and 100 respectively, their ratio being kept constant. The tail is rather symmetrical with respect to the time of flight. Note that W for curve 2 was found to be 0.20 both theoretically and graphically while for curve 3 the whole procedure seems inadequate (formally defined Wexceeds unity).



**Figure 2.** Transient curves in the dispersive regime calculated using the  $\tau$ -approximation of Arkhipov and Rudenko [8]. Surface (1, 3) and uniform generation (2, 4).  $F_0 = 10^8$  (1, 2) and  $10^7$  V m<sup>-1</sup> (3, 4). Pulse length = 2.5  $\mu$ s,  $\mu_0 = 10^{-5}$  m<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>,  $\tau_0 = 4 \times 10^{-11}$  s,  $\nu_0 = 2 \times 10^6$  s<sup>-1</sup>,  $L = 10^{-5}$  m,  $\alpha = 0.70$ .

### 2.2. Dispersive transport

Dispersive (or anomalous) transport was discovered in the early 1970s and was originally described by the semiphenomenological CTRW model [1]. The real understanding of the TOF current shape has been provided by MT theory [2, 3].

A lg j-lg t plot has two asymptotes: the preflight one  $j \propto t^{-1+\alpha}$  ( $t \ll t_{tr}$ ) and the post-flight one  $j \propto t^{-1-\alpha}$  ( $t \gg t_{tr}$ ) whose intersection operationally defines  $t_{tr}$  as figure 2 shows (curve 3). Typical values of the dispersion parameter  $\alpha$  for MDP range from 0.6 to 0.75 (see below). For calculations for figure 2 we used the  $\tau$ -approximation, though for  $\alpha = 0.70$  its application is rather questionable.

It is important that the change of excitation mode as realized in TOF (surface) and TOF-2 (uniform generation) techniques does not affect the main transient features. Indeed, the gross effect consists in the slight acceleration of the preflight current fall (compare curves 1 and 3 with curves 2 and 4) while the time of flight shortens by a factor of  $(\sqrt{3})^{1/\alpha}$ . The post-flight asymptote does not change at all [9].

In MT formalism the carrier packet spreads in the field direction due stochastic processes of carrier trapping and thermal release, carriers drifting with constant quasi-free mobility  $\mu_0 (\approx 10^{-5} \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1})$  during very short intervals of time  $\tau_0 (\approx 10^{-10} \text{ s})$  between successive events of release and capture [9]. The frequency factor  $\nu_0 \sim 10^6 \text{ s}^{-1}$ . The spreading is extremely large, so the root mean square dispersion  $\omega = [\langle \Delta x^2 \rangle]^{1/2}$  is equal to the mean displacement  $\langle x \rangle$  [10] and as a result is proportional to the electric field ( $\omega \propto F_0$ ). In fact, the packet drifts as much as it spreads.

Obviously, the form of the packet differs markedly from the Gaussian [8]. At transit time, the mean carrier displacement approximately equals the sample thickness. As a result, the packet spreading at  $t_{\rm tr}$  is also close to *L*.

The case of  $0.5 < \alpha \le 1.0$  relates to a mildly anomalous transport while for  $\alpha > 1.0$  a DG transition is bound to occur. Since  $\tau$ -approximation no longer holds, one resorts to Laplace transformation of differential equations [11–13]



**Figure 3.** TOF curves computed for the box-type MT model using exact solutions presented in [15]. L = 1 (1), 3 (2), 10 (3), 100  $\mu$ m (4).  $\mu_0 = 10^{-5} \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1}$ ,  $\tau_0 = 10^{-10} \text{ s}$ ,  $\nu_0 = 10^6 \text{ s}^{-1}$ . The maximum trap energy is 5kT,  $D_{\text{F}} = 0.84 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ .  $F_0 = 10^7 \text{ V} \text{ m}^{-1}$  for all curves.

or even to the fractional Fokker–Planck approach [14]. It has been ascertained that for  $\alpha = 0.8-0.9$  a well-defined shoulder rather than a featureless slope changeover (as given by the  $\tau$ -approximation [8, 10]) appears on TOF curves when represented on a linear j-t plot.

MT theory for  $\alpha < 1.0$  predicts that a lg j-lg t plot is universal as regards field or thickness variation. Moreover, TOF curves depend only on the ratio  $F_0/L$  unlike the true Gaussian transport (see section 2.1).

## 2.3. General case of transport with a DG transition

Now let us discuss the MDP transport in the most general way. For specific results we rely on MT schemes allowing the DG transition (monoenergetic [15] and box-like [11, 15] for example) or the GDM.

In all MT models predicting a DG transition (except the degenerate case of monoenergetic traps) there is an initial current fall  $j \propto t^{-1+\alpha(t)}$  with  $\alpha(t) \rightarrow 1.0$  as  $t \rightarrow \infty$  (a semi-infinite sample is implied). If the sample thickness *L* and electric field  $F_0$  are such that  $\alpha(t_{\rm tr}) \ge 0.95$  the plateau is bound to appear. It is remarkable that normalized full time TOF transients do not change if the ratio  $F_0/L$  stays constant. We verified this result for curve 2 in figure 3 by changing these quantities simultaneously by up to 100 times. Such a result is familiar for truly dispersive transport [8–10] but here it extends to transit times much larger than the equilibration time  $\tilde{t}$ , i.e. well into the non-dispersive regime.

This theoretical result defines the so-called field diffusion whose diffusivity is proportional to the electric field squared [8]:

$$D_{\rm F} \propto F_0^2$$
. (3)

Indeed, the substitution of relation (3) into equation (2) proves that the resulting TOF current depends only on the ratio  $F_0/L$ . Note that for the field independent *D*, as is the case for truly equilibrium transport, this is not so (figure 1).



**Figure 4.** As figure 3, except that  $F_0 = 10^6$  (1),  $10^7$  (2),  $3 \times 10^7$  (3) and  $10^8$  V m<sup>-1</sup> (4).  $L = 3 \mu m$  for all curves.

If  $F_0$  and L change separately the effect is conspicuous. Thus L variation (curves 1 to 4 in figure 3) reduces W from 0.285 to 0.030 (theory predicts a 10-fold decrease),  $\tilde{t}$  being unchanged. Increasing  $F_0$  (figure 4) results in the plateau shortening (curves 1–3) and eventually in its disappearance (curve 4) as Gaussian transport gradually gives way to a dispersive one.

GDM predictions are quite similar [6, 16, 17]. As Monte Carlo simulations allow automatic accounting for the normal diffusion, relation (3) holds only for medium to strong fields when  $D_{\rm F} \propto F_0^2$ . As  $F_0 \rightarrow 0$ , the contribution of the field diffusion becomes negligible and the Einstein relation is gradually recovered. Typical values of W vary from 0.12 to 0.6 depending on the width  $\sigma$  of the Gaussian hopping site manifold and the temperature.

#### 2.4. Implications for experimental studies

It is useful to characterize the TOF plateau and the subsequent trailing edge in terms of parameters  $\psi$  and  $\beta$ . The first is effectively the plateau slope  $(j \propto t^{-\psi})$  while the second refers to current decay following a transit time  $(j \propto t^{-\beta})$ . Thus, we have three parameters  $(W, \psi \text{ and } \beta)$  which describe experimentally observed TOF transients and the following criteria (requirements) allowing one to verify whether the observed plateau is indeed a proof of the genuine DG transition.

R1. A plateau should not rise to its end (cusps contradict the theory).

R2. The plateau starting point  $t_i$  should not depend on L.

R3. As  $\psi \to 0$ ,  $\beta$  should become larger than unity for  $t \ge 3t_{tr}$ .

R4. W is expected to decrease as L increases according to a relationship  $W \propto L^{-1/2}$  for well-defined plateaus.

R5. *W* must increase with  $F_0$  causing plateau shortening and its eventual disappearance at high electric fields.

The situation gets more complicated for would-be plateaus with  $\psi \approx 0.1$ –0.3. The problem here is whether this should be ascribed to an ongoing DG transition or to the dispersive transport with  $\alpha = (1 - \psi)$  close to unity. Theory gives an unequivocal answer to this question as well. If the post-flight current decay follows the power-like dependence  $j \propto t^{-2+\psi}$ , the transport is indeed dispersive. However, if  $\beta \ge 4.0$  the DG transition occurs.

## 3. Analysis of the TOF data

Contrary to the R1 case, cusp appearance has not been a rare event at any stage of the MDP research in the world's major laboratories [18–24]. Routinely, the cusp was treated like a plateau by averaging its top, without taking any trouble to find out the reason for its appearance. Now we are going to address TOF curves with a plateau proper, trying to ascertain whether plateau presence really indicates a DG transition.

TOF curves with a flat plateau ( $\psi \approx 0$ ) are better analyzed in two separate groups depending on the W value. In the first, W = 0.3-0.6 and there are exceptionally long tails [25-34]. The second group [17, 20, 36-41] includes MDP whose W ranges from 0.1 to 0.2. The reason for such an approach is that the former seemingly defy interpretation in terms of the DG transition whereas the latter seem to be in accord with it.

Stolka *et al* [25] have shown that *L* variation from 4 to 90  $\mu$ m left the drift mobility unchanged. The MPD studied was PC + TPD (this designation implies polymer matrix followed by a dopant in standard notation).

Later, detailed studies of Yuh and Stolka [28] introduced a concept of *W*-characterization. They confirmed that mobility was independent on the sample thickness (PC + TPD) and revealed that *W* does indeed diminish with increasing sample thickness (0.34 for  $L = 18 \ \mu\text{m}$  and 0.20 for  $L = 74 \ \mu\text{m}$  at 10<sup>5</sup> V m<sup>-1</sup>) in apparent accord with R4. In contrast, *W* was found to be field independent ( $W \approx 0.34$  at 10<sup>7</sup> and  $6 \times 10^7$  V m<sup>-1</sup> for  $L = 18 \ \mu\text{m}$ ) in violation of R5. The tail was rather asymmetric.

Similar investigation has been reported by Schein [33] for L changing in PS + DEH from 3.8 to 42  $\mu$ m ( $\psi = 0.07$ ) to show that W remained constant (0.40 to within 5%). 10fold field increase produced no noticeable variation in the normalized linear TOF plot (violation of both R4 and R5). The same result has been reported in [30] (violation of R5), this time for W = 0.3. The authors of [30] point out that there is a disparity of over two orders of magnitude in the model tetraphenylbenzidine main-chain polymer between the measured  $W \approx 0.3$  and  $\tilde{W} \approx 0.002$  found from the solid-state hole diffusion data. This finding proves our earlier assertion that field diffusion may be responsible for this discrepancy.

It should be remembered that Pfister [35] reported TOF data demonstrating that in a PC + TPA system at the highest temperature (383 K) or loading (50 wt%) and  $\psi \leq 0.1$ , the post-transit decay proceeded with  $\beta \approx 1.7$  for 1.5 decades. These findings obviously contradict traditional interpretation in terms of a true DG transition and favor the persistence of the dispersive transport in spite of the plateau appearance.

Now we address TOF data relating to the second group of papers [17, 20, 36–41]. It has been shown that MDP with high drift mobility (PS and PC used as a polymer matrix plus dopants at high loadings) as well as evaporated films of dopants themselves [20, 36] featuring exceptionally low W = 0.1– 0.2 have produced evidence in support of the GDM theory. Thus, experiment has borne out the theoretical  $W \propto L^{-1/2}$ dependence [6] and in a rather sketchy lg *j*–lg *t* representation the existence of  $\beta$  in excess of 4.5 [19, 20, 39, 41]. The strongest indication of this effect has been presented in [37] for a photoconductive polymer poly(methylphenylsilylene). In this case  $\beta \ge 10$  at  $t \approx 1.7 t_{tr}$  ( $\psi \approx 0$ ), thus conforming to R3. But still it is worth mentioning that samples of PS + 30% TTA from the same batch had *W* varying uncontrollably from 0.12 to 0.3 [41].

No attempt has been made to test thickness (or field) independence of  $t_i$  in MDP. Judging by data presented in [28] this parameter scales like  $t_{tr}$  instead of being constant, in clear contradiction to the theory.

It should be recalled that early results with some MDP and PVK clearly favored dispersive rather than Gaussian transport (the first theory of dispersive transport (CTRW) was devised to explain those results). Of special importance is the proof of the dispersive transport in PVK provided in [4], as all possible measures have been taken to guarantee the highest purity standard of the samples to ward off any claims on that account by GDM proponents. A year earlier, there appeared an investigation on carrier transport in PVK claiming to prove a DG transition based on plateau appearance [18]. Again, TOF curves featured a cusp and the post-transit current decay followed a mild  $j \propto t^{-1.2}$  pattern over two decades in time, thus grossly violating the GDM application criteria.

#### 4. Electron-gun based studies

It has become clear to us that in order to resolve the standing controversy, the experimental basis of investigations should be enlarged by including the TOF-2 variant. This approach allows circumventing possible surface release effects by generating the vast majority of carriers uniformly in the sample. Besides this, a detailed study of the early time current waveform has also been undertaken.

This approach used the electron facility ELA-65, capable of providing short pulses  $(2-20 \ \mu s)$  of 3-7 (TOF) and 50-65 keV (TOF-2) electrons, respectively. As a result, the two techniques could be applied to the same sample in identical conditions (all experiments were conducted in high vacuum). As a prototype MDP we chose PC doped with (30 or 50)% DEH (by weight) and PVK. Data reduction agreed with recommendations given in section 2.2.

Our main results are as follows [9, 42–46]. TOF mobility data are very close to those reported in the literature and qualitatively agree with Pfister's observations [35]: a shoulder  $j \propto t^{-0.16}$  rather than a plateau is observed in PVK while in PC–30% DEH a shoulder, a cusp and even a plateau do appear. Judging by the current post-transit decay and again in full accord with Pfister's recommendations [35]  $\alpha$  is 0.6 (PVK) and 0.7–0.75 (MDP).

The most revealing information comes from TOF-2 measurements. TOF-2 curves for both polymers are in perfect agreement with MT predictions and with the above TOF figures for the dispersion parameter  $\alpha$ .



**Figure 5.** Comparison of TOF-2 (1) and TOF (2, 3) curves for a sample of PC + 50% DEH 16.4  $\mu$ m thick equipped with an a-Se layer on a linear (a) and on a logarithmic scale (b).  $F_0 = 2.4 \times 10^7$  (1, 2) and  $1.2 \times 10^7$  V m<sup>-1</sup> (3); room temperature [42]. For curves 2 and 3, which were made to coincide on the plateau, *W* is 0.61. The time constant *RC* and the pulse length are much less than the observation times.

An important observation is that the application of an a-Se generation layer to samples of DEH-doped PC or PVK leads to the formation of a slightly cusping plateau. For a range of fields it turns into a flat plateau (figure 5) similar to that reported in [26, 29] and in full accord with our results obtained by the optical TOF technique using an a-Se layer [46].

Finally,  $t_i$  for TOF curves for PC–50% DEH with a wouldbe plateau scales with the field in exactly the same way as the time of flight (figure 6). These preliminary results show that  $t_i$ should not be regarded as an equilibration time (as a rule, the plateau duration comprises about 1.3–1.5 decades irrespective of the sample length or applied electric field).

All these facts combine to show definitively that carrier transport in the tested DEH-doped PC and PVK is dispersive with rather high dispersion parameter (0.6–0.75 at room temperature). TOF results are severely interfered with by some extraneous factor, presumably surface traps [47]. The exact mechanism of this interference still needs to be understood. This conclusion is probably applicable to those MDP whose TOF signature contains a plateau with a long dispersive tail.



**Figure 6.** TOF curves for a sample of PC + 50% DEH 14  $\mu$ m thick in a logarithmic representation. The electric field equals  $3.6 \times 10^7$ (1) and  $3.6 \times 10^6$  V m<sup>-1</sup> (2). The arrows indicate the beginning of the would-be plateaus (unpublished results). Pulse length 20  $\mu$ s; *RC* is near 50 (1) and 500  $\mu$ s (2).

It is not completely clear whether this inference could be extended to MDPs with rather short tails.

Therefore, our next aim is to prepare MDP samples (with and without a-Se layers) having W = 0.1-0.15 and  $\beta \ge 6.0$ in order to put them under TOF-2 scrutiny as earlier suggested in [9, 43]. Our preliminary results on PS + 30% TTA featuring high drift mobility (3 × 10<sup>-8</sup> m<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> as in [41]) show that hole transport is indeed dispersive [48]. However, in contrast to the W = 0.1-0.3 found in [41], we have registered a much higher value (0.6) in full accord with  $W \approx 0.65$  reported for PC + 40% TTA [49].

It is interesting that Schein [49] discussed similar discrepancies between experimental TOF data and predictions from both the polaron and the GDM theories at some length as early as 1992.

Recent Monte Carlo simulations in the framework of the correlated disorder model (CDM) show that the equilibration time may be very short;  $\psi$  may approach zero and  $\beta$  ranges from 3 to 5 [50, 51].

Original oscilloscope traces presented in two of the most recent works by Borsenberger [52, 53] graphically illustrate the seriousness of the situation with MDP samples supplied with a generating layer (a-Se, dye, etc). In the first, one can see a poorly defined plateau ( $\psi \ge 0.2$ ) and an extremely long trailing edge ( $W \approx 0.5$ ). The second paper reports that the normal TOF signal may acquire a severely cusping form in one of the two equally acceptable configurations with respect to the a-Se layer and the light beam. In addition, Bässler *et al* [24] have recently demonstrated that the generation layer might exert a profound influence on the form of the TOF current.

It is instructive to remember that only the combination of both TOF and photoconductivity (the analog of our TOF-2) techniques proved definitively the dispersive rather than the Gaussian hole transport in yet another model disordered solid, namely photoconductive chalcogenide glass  $As_2Se_3$  [54, 55]. Of course, this is an inorganic substance, but according to Baranovskii (chapters 2 and 6 in a recently published book [56]), photo-physicists working with organics would gain much both theoretically and experimentally by borrowing ideas from the inorganic front. We fully subscribe to this assertion.

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