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Hole transport in bis(4-N,N-diethylamino-2-methylphenyl)-4-methylphenylmethane

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By time-of-flight photocurrent techniques, hole transport has been investigated in vapor deposited films of the title compound. The measurements were made over a wide range of fields and temperatures which encompassed the glass transition temperature. The results are described within the framework of the disorder formalism due to Bassler and co-workers. The formalism is based on the assumption that transport occurs by hopping in a manifold of states subject to both energetic (diagonal) and positional (off-diagonal) disorder. In this paper, we describe (1) the field and temperature dependencies of the photocurrent transients, (2) the role of polymer dynamics on charge transport at temperatures above the glass transition temperature, and (3) the spectroscopy of interfacial hole states derived from an analysis of photocurrent transients in terms of computer simulations conducted under the premise of energy selective carrier injection into a Gaussian distribution of states.

I. INTRODUCTION

Considerable progress has recently been made concerning the understanding of charge transport in disordered molecular solids such as molecularly doped polymers, pendant group polymers, and main chain polymers. The concept of concurrent energetic (diagonal) and geometric (off-diagonal) disorder has proved to be both necessary and sufficient for rationalizing such recurrent features as (1) the field and temperature dependencies of the mobility,[1,2] (2) the transition from nondispersive to dispersive transport with decreasing temperatures,[3] and (3) the long tails of time-of-flight (TOF) photocurrent transients which, concurrently, show well-developed plateaus suggestive of nondispersive transport. This concept is in accord with the intuition that disorder revealed by optical spectroscopy must also be important for the dynamics of charge carriers. On the other hand, it is a zero-order approach in the sense that it ignores dynamic effects such as the relaxation of a donor or acceptor molecule upon oxidation or reduction.[4,5]

In an attempt to further explore the applicability of the disorder formalism, hole transport of bis(4-N,N-diethylamino-2-methylphenyl)-4-methylphenylmethane (MPMP) has been investigated. One reason for selecting this particular compound was that the glass transition temperature is quite low (283 K). This allows transport measurements in a temperature range above and below T_g in order to study how the onset of dynamical processes in the glass affects the migration of carriers injected into the compound from a photoemitting electrode.

Usually photocurrent transients exhibit an initial drop before a plateau is attached. This reflects the equilibration of carriers within the distribution of hopping states (DOS), provided the population at $t = 0$ is random. The initial current drop should be absent, or at least greatly reduced, if the injection process populated states of the DOS near or below the equilibrium energy. Varying the energy of the injected carriers into the DOS of the transport medium by using injection layers with different ionization potentials should result in a systematic variation of the TOF signal. Not only would the observation of this effect be of interest in providing additional support for the disorder model to explain charge transport, it would also yield valuable information on the relative position of interfacial energies, which is difficult to obtain otherwise via *in situ* measurements. The results of this study demonstrate that the relative position of the energy levels at interfaces between MPMP and either α-Se or an organic pigment is appropriate for studying the phenomenon.

In this paper, we describe (1) characterization of hole transport in MPMP, (2) the role of polymer dynamics on charge transport at temperatures above the glass transition temperature, and (3) the spectroscopy of interfacial hole states derived from an analysis of TOF pulse shapes in terms of computer simulations conducted under the premise of energy selective carrier injection into a Gaussian DOS.

II. EXPERIMENTAL

The molecular structure of MPMP is illustrated in Fig. 1. The preparation of this compound was accomplished by the following procedure. A mixture of 163.3 g (1.0 mole) N,N-diethyl-m-toluidine, 60.1 g (0.5 mole) *p*-tolualdehyde, 150 ml ethanol, and 50 ml concentrated HCl was heated on a steam bath for two days. The mixture was then diluted into water, made alkaline with diluted NaOH, then steam distilled until the distillate was clear. The water layer was decanted from the residue which was further washed with water. The residue solidified on being triturated with ethanol and was then crushed and filtered off. The solid was recrystallized twice from ethanol, mp 378.9–379.8 K, then recrystallized a third time with hot filtration from ethanol containing a small quantity of ethyl acetate, mp 379–379.6 K. The resulting purity by HPLC was 99.7 area %.

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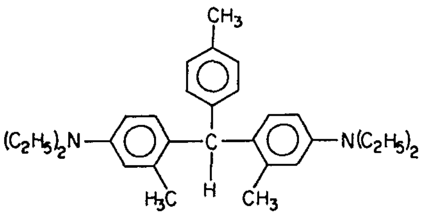

FIG. 1. The molecule structure of bis(4-N,N-diethylamino-2-methylphenyl)-4-methylphenylmethane (MPMP).

Films of MPMP were prepared by thermal sublimation from a resistance heated Ta crucible onto Ni coated polyethylene terephthalate substrates which had previously been coated with 0.30 μm films of either α-Se or N,N′-bis(2-phenethyl)-perylene-3,4,:9,10-bis(dicarboximide) (hereafter perylene). Both the α-Se and perylene layers were prepared by vapor deposition techniques. During the sublimation of the MPMP, the Ta crucible was maintained at 493 K, which resulted in a deposition rate of approximately 40 Å/s. Thicknesses of the MPMP films were between 5 and 9 μm, as determined from cross-section photomicrographs and capacitance measurements. Following the deposition of the MPMP layer, a semitransparent Au electrode was vapor deposited on the free surface. During the deposition of the MPMP and Au layers, the substrate was attached to a liquid-N_2 cooled stage. From differential scanning calorimetry measurements, the glass transition temperature of the MPMP was determined as 283 K.

The drift mobilities were measured by conventional TOF techniques which have been described elsewhere.[2,6] By this method, the displacement of a sheet of carriers injected from a photoemitting electrode is time resolved. Photoexcitation of the α-Se emitter layer was accomplished by 3 ns exposures of 440 nm radiation derived from a dye laser (Laser Sciences, Inc., model 337). For the perylene layers, the wavelength was 600 nm. To ensure that the experiments were performed under the small-signal TOF regime, the exposures were filtered such that the total charge injected into the sample was less than 0.05 CV, where C is the sample capacitance and V the applied voltage. The photocurrent transients were measured with a Tektronix model TD2301 transient digitizer system. All measurements were made in air. Figure 2 includes a schematic diagram of the experimental arrangement.

Mobilities were determined from the conventional expression, $\mu = L^2/\tau_T V$, where L is the sample thickness and τ_T the transit time. The transit times were determined from the intersection of asymptotes to the plateau and trailing edge of the transients. While this technique is the common experimental method for determining transit times, it differs from those normally used in simulation studies where the transit time is defined as the average arrival time of carriers at the collecting electrode. Transit times defined in this manner are typically two to three times greater than those determined by the intersection of asymptotes. This difference, however, is of little significance in determining the field and temperature dependencies of the mobility.

III. RESULTS

Figure 3 shows a series of photocurrent transients, parametric in temperature, measured by injection from a perylene emitter layer. After an initial drop, which becomes RC limited from temperatures above 290 K, a plateau is reached followed by a long tail. As T decreases below 230 K, the pulse shape gradually acquires dispersive features. This behavior is in agreement with literature results on similar materials. A new phenomenon, however, is observed upon replacing the perylene layer with an α-Se emitter. In the latter case, a cusp evolves in the photocurrent transients at temperatures above 263 K. This phenomenon is illustrated in Fig. 4. The effect is field dependent; at low fields the cusp disappears [Fig. 4(b)]. Within the limits of resolution in determining the transit times, mobility values agree with those determined from cusp-free transients measured with perylene emitter layers. In conjunction with the independence of the cusp on exposure intensity, these observations argue against associating the cusp with a space charge effect.

The temperature dependence of the mobility has been measured in the range of 190 to 320 K, encompassing the

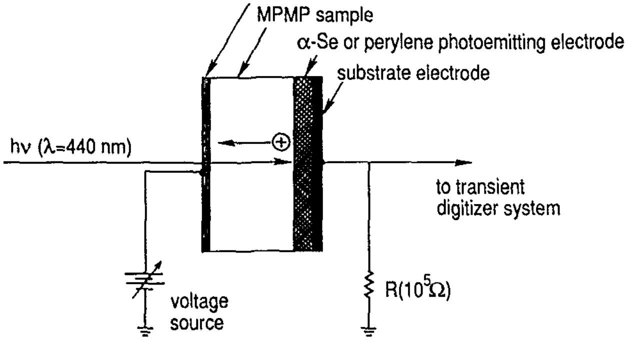

FIG. 2. A schematic diagram of the experimental arrangement.

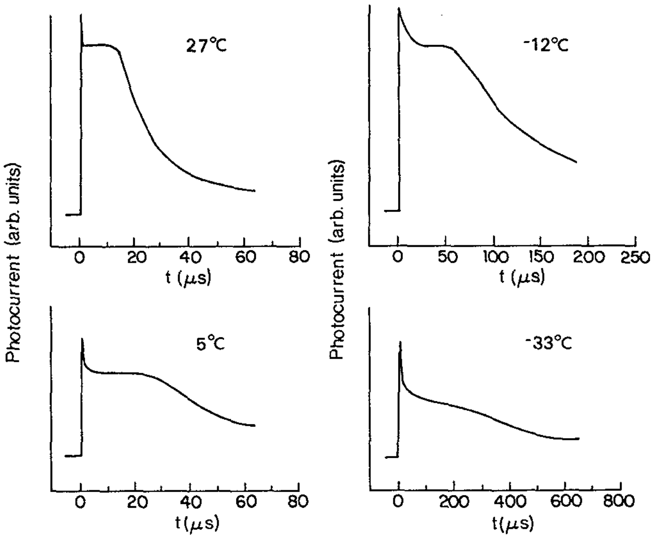

FIG. 3. Photocurrent transients measured with a perylene photoemitting electrode. The sample thickness was 8.7 μm and the voltage 140 V.

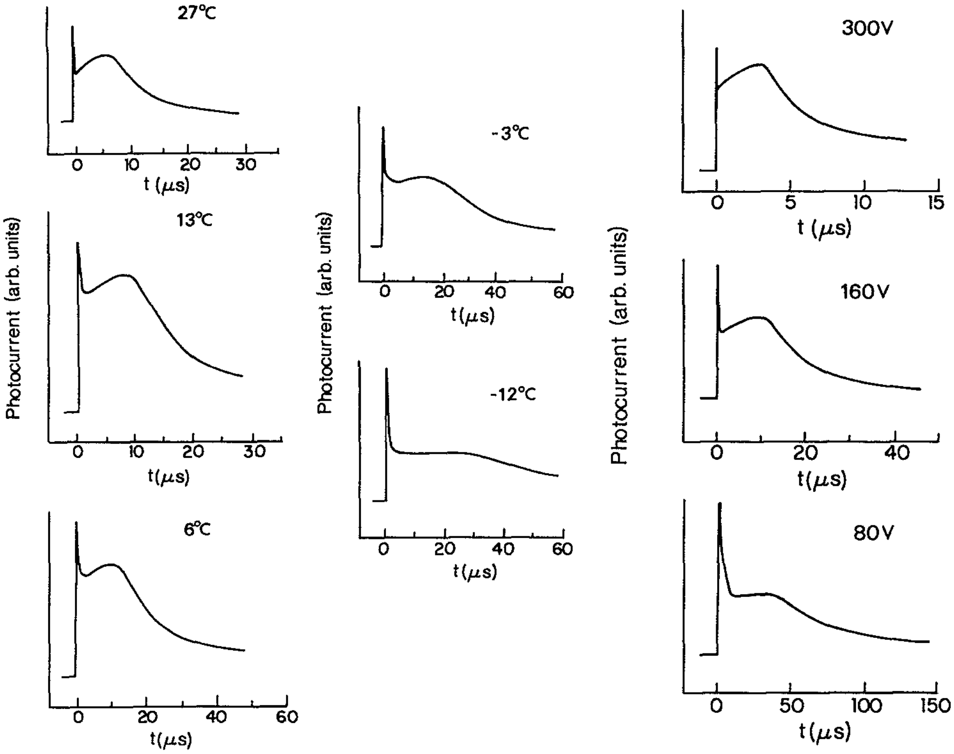

FIG. 4. Photocurrent transients measured with an α-Se photoemitting electrode. In Fig. 4(a), the sample thickness was 8.7 μm and the voltage 140 V. In Fig. 4(b), the thickness was 8.7 μm and the temperature 298 K.

glass transition temperature, 283 K. No significant hysteresis in the mobility was observed in heating and subsequent cooling through the glass transition region. Figure 5 illustrates that for $T < T_g$, the temperature dependence of the low field mobility can be described by an $\mu = \mu_0 \exp[-(T_0/T)^2]$ relationship with $\mu_0 = 0.34$ cm^2/Vs and $T_0 = 758$ K. It is noteworthy that mobilities derived from more-or-less dispersive transients obey the same temperature dependence. Above T_g, slopes of $\ln \mu$ vs T^{-2} plots decrease. This shown in greater detail in Fig. 6 for temperatures in the vicinity of T_g. The field dependence of the mobility follows a $\ln \mu \propto E^{1/2}$ relationship over the entire range of fields investigated. The slopes, $S = \partial \ln(\mu/\mu_0)/\partial E^{1/2}$, vary linearly with T^{-2} with little deviation above T_g.

IV. DISCUSSION

A. Analysis of mobility data

For hopping through a manifold of sites with superimposed diagonal and off-diagonal disorder, computer simulations predict[1,2]

$$\mu(\hat{\sigma},\Sigma,E) = \mu_0 \exp\left[-\left(\frac{2\hat{\sigma}}{3}\right)^2\right]\exp[C(\hat{\sigma}^2 - \Sigma^2)E^{1/2}]. \tag{1}$$

Here, σ is the width of the Gaussian DOS, equivalent to a normalized variance $\hat{\sigma} = \sigma/kT$ of the energy distribution of hopping states, Σ the variance of the wave function overlap term $2\gamma a$ in a Miller–Abrahams[7] type expression for the intersite hopping rate, and C an empirical constant, $C = 2.9 \times 10^{-4}$ (cm/V)$^{1/2}$. Equation (1) is only valid for $\Sigma \gtrsim 1.5$ and moderately large fields. At lower fields ($\lesssim 5 \times 10^5$ V/cm), the field dependence of the mobility should saturate. For $\Sigma \gtrsim 2.5$, the mobilities should decrease with increasing field at low fields. It should be noted, however, that the essential quantity determining the field dependence of the mobility is the drop of the electrostatic potential across a hopping distance. Since the simulations were carried out on a test sample with cubic symmetry and a lattice constant $a = 6$ Å, the electric field applied to a sample of lattice dimensions $a^{\exp}$ must be rescaled by a factor $a^{\exp}/6$ Å for comparing experimental data with those predicted by Eq. 1.

Figure 5 is an example of a near-perfect $\ln \mu$ vs T^{-2} relationship over a significant range of temperatures. Taking the slope of the curve at the lowest electric field, 7.1×10^4 V/cm, as a measure of the width of the static DOS yields $\sigma = 0.098$ eV for $T < T_g$. This allows translating the data of Fig. 5 into a dependence of the rescaled values of the slope of $\ln \mu$ vs $E^{1/2}$ plots vs $\hat{\sigma}^2$, as suggested by Eq. (1) using $a^{\exp} = 8.5$ Å. The latter value follows from the density and molecular weight of MPMP. The slope of the rescaled S vs vs $\hat{\sigma}^2$ plot is $C = 2.9 \times 10^{-4}$ (cm/V)$^{1/2}$, in excellent agreement with the simulations. Not making the correction for the intersite distance would give $C = 3.5 \times 10^{-4}$ (cm/V)$^{1/2}$. From the abscissa intercept of the extrapolated S vs $\hat{\sigma}^2$ plot, an off-diagonal parameter $\Sigma = 2.0$ follows (Fig. 7). Since in van der Waals coupled organic solids, $2\gamma a$ is typically of order 10, this is equivalent to a 20% variation of intersite coupling due to positional disorder. We consider the fact that chemically different materials such as MPMP, 1,1-bis(di-4-tolylaminophenyl)cyclohexane doped polycarbonate,[2] vapor deposited films of 1,1-bis(di-4-tolylaminophenyl)cyclohexane,[6] and polysilylenes[8] all yield not only the same general pattern for the $\mu(E,\hat{\sigma})$ relationship, but the same numerical constant relating S and $\hat{\sigma}$ as a convincing argument that the disorder formalism is both necessary and

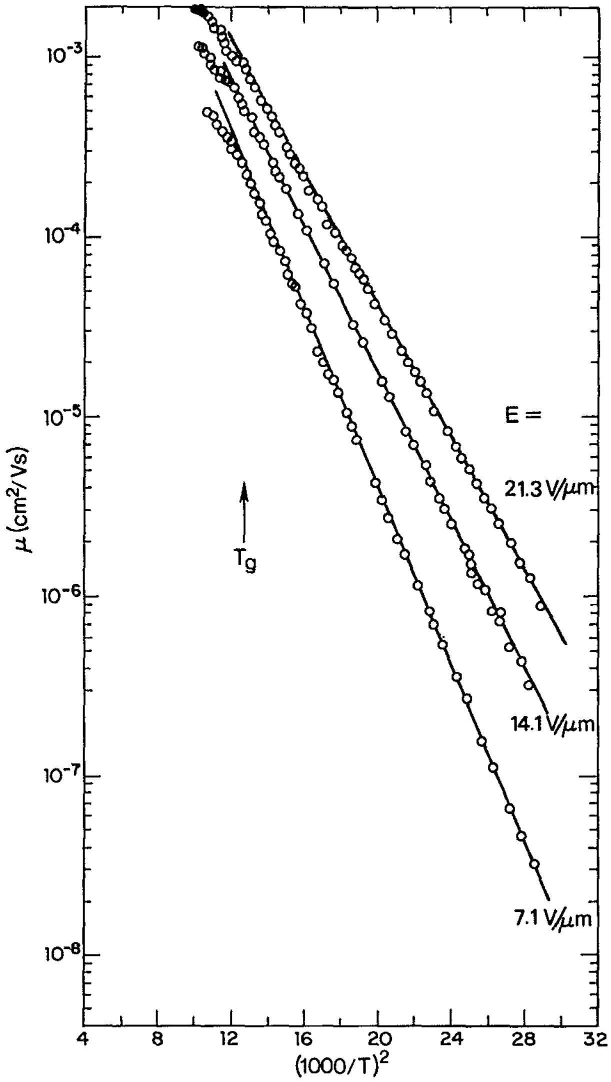

FIG. 5. The logarithm of the mobility vs T^{-2}. The measurements were made with a perylene photoemitting electrode.

sufficient for rationalizing charge transport. This does not rule out the existence of polaron transport. The crucial quantity that determines the magnitude of polaronic effects is the change of the molecular structure upon removing, or adding, an electron. Materials such as MPMP and the arylamine derivatives[6] do not belong to this category. It is suggested that this is due to the fact that the structures of both the neutral and radical cations of these compounds are planar.

The disorder formalism is readily extended to the case where the DOS is not static but dynamic, albeit on a time scale that is slow relative to the average dwell time of carriers on the hopping sites. This situation should be realized in a glass at temperatures not too far above the glass transition temperature where slow local motions of the structural elements occur that are likely to increase the width of the DOS. As a consequence, the mobility should increase with temperature more slowly than expected for a static DOS. Making the plausible assumption that μ_0, the mobility in the absence of energetic disorder, remains unaltered, the effective

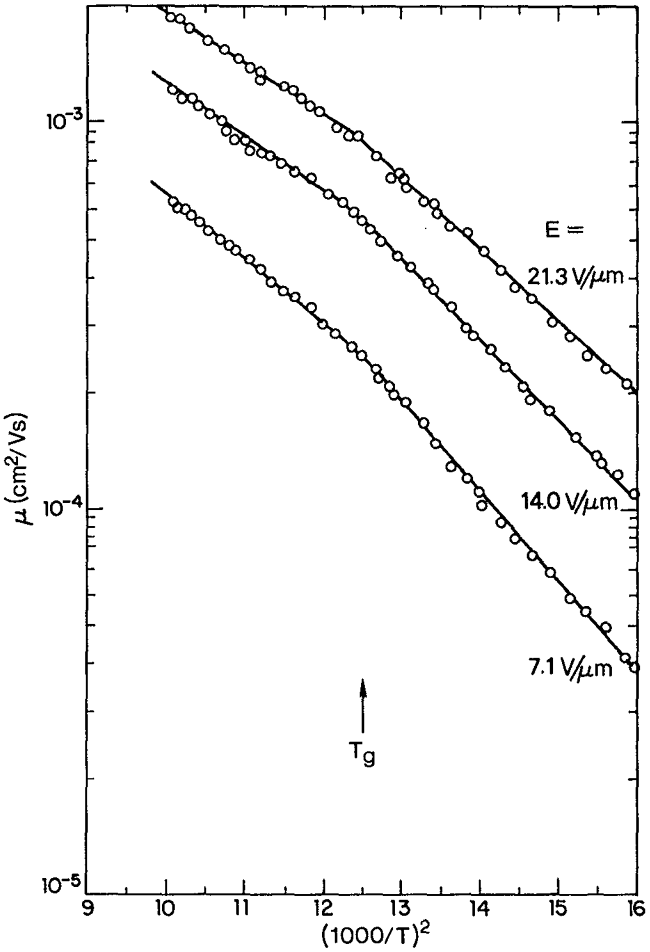

FIG. 6. The data of Fig. 6 in the region of the glass transition temperature.

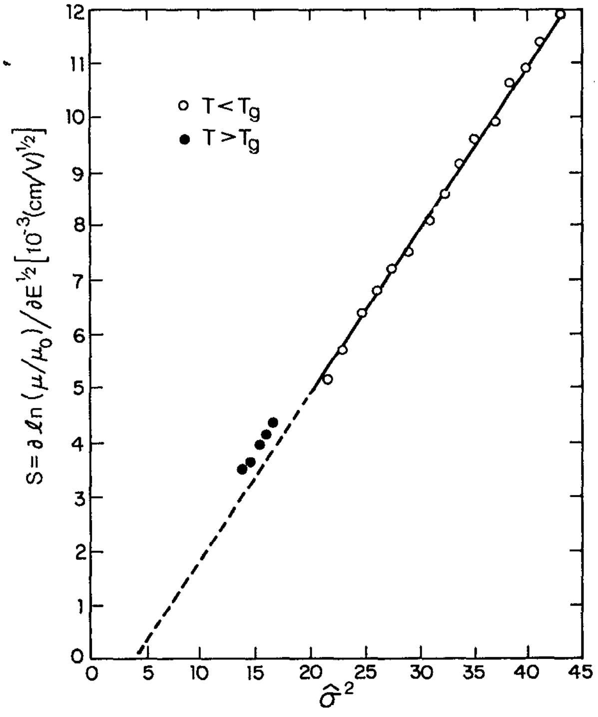

FIG. 7. S vs $\hat{\sigma}^2$.

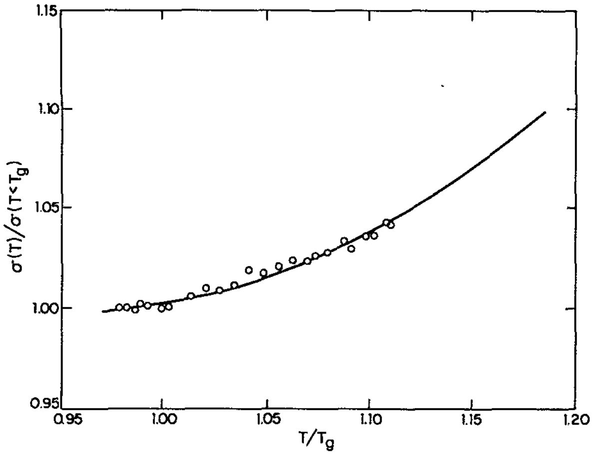

FIG. 8. $\sigma(T)/\sigma(T<T_g)$ as a function of reduced temperature, T/T_g. The solid curve is the average measured width of four different polysilylenes: (1) poly(dihexylsilylene), (2) poly (methylphenylsilylene), (3) poly(dibutylsilylene), and (4) polydibutlylgemylene. From M. A. Abkowitz, H. Bässler, and M. Stolka (Ref. 8).

disorder parameter $\sigma(T)$ can be calculated from the temperature dependence of the mobility according to Eq. (1). In the low field limit,

$$\sigma(T) = \tfrac{3}{2}[\ln(\mu_0/\mu)]^{1/2}. \tag{2}$$

Equation (2) allows determining the temperature dependence of σ above T_g. Data obtained from the $E = 7.1\times 10^4$ V/cm curve of Fig. 6 are shown in Fig. 8. It is noteworthy that a plot of $\sigma(T)$ values normalized to the width of the DOS at $T < T_g$ vs temperature, normalized to the glass transition temperature, is coincident with the analogous curve obtained from the analysis of hole mobilities in polysilylenes with various substituents.[8] This supports the argument that the deviation of the $\mu(T > T_g)$ from the extrapolation of $\mu(T < T_g)$ is a signature of the glass dynamics above T_g. It is well known that the dynamic properties of glasses are more-or-less materials invariant, if plotted as a function of the reduced temperature. T/T_g.[9] A recent analysis also suggests that for temperatures $T_g < T < T_c \approx 1.3\ T_g$, they are controlled by slow noncollective motions of the glass forming elements in a random potential.[10] T_c is the divergence temperature above which collective effects, tractable within the framework of mode coupling theory,[11] become important. Observing the manifestation of the onset of glass dynamics above T_g on charge transport in a variety of chemically quite different systems such as a molecular glass, molecularly doped polymers,[12,13] and main chain polymers like the polysilylenes[8] is another convincing test for the applicability of the disorder formalism for treating the motion of charge carriers in disordered molecular solids.

B. Analysis of time-of-flight transients

Upon analyzing the TOF profiles, the following questions are raised: (1) what is the origin of the cusp at high temperatures and electric fields upon photoinjection from α-Se, (2) why does the initial current drop occur on a shorter time scale than in earlier studies on materials such as 1,1-bis(di-4-tolylaminophenyl)cyclohexane doped polycarbonate,[2] and (3) why can conventional TOF signals be observed at temperatures at which one would expect the transients to be dispersive? According to an earlier simulation study,[3] the transition from nondispersive to dispersive transport should occur at

$$\hat{\sigma} > 1.22[\ln(6kTL/ea^2E)]^{1/2}. \tag{3}$$

Physically, the transition was defined by the condition that the transit times are less than 0.1 times the time carriers need to relax towards energetic equilibrium. In TOF profiles, it is manifested by the gradual erosion of the current plateau. For $E = 10^5$ V/cm and $L = 10\ \mu$m, Eq. (3) yields $\hat{\sigma} = 4.4$, equivalent to $T = 257$ K. Experimentally, however, transit times can be distinguished from double linear current vs time plots at temperatures as low as 230 K. One explanation would be that the temperature dependence of the mobility overestimates the width of the DOS. A possible reason for this could be that part of the activation energy is due, for instance, to polaron formation. Apart from the consistency of the analysis of the field and temperature dependencies of the mobility described in the preceding section, the magnitude of the tails of TOF profiles argue against this possibility. The dispersion of the tails of the photocurrent transients is defined by $d = (t_{1/2} - t_0)/t_{1/2}$.[14] Here, t_0 is the time at which the asymptotes to the TOF transients intersect and $t_{1/2}$ is the time at which the current has decayed to 1/2 of the plateau value. The parameter d is related to the diffusivity D via

$$d = (\pi D/\mu V)^{1/2}. \tag{4}$$

Previously, it has been shown by experiment,[14] simulations,[15,16] and by analytic theory of transport controlled by multiple trapping,[17] that the presence of disorder gives rise to an anomalous field-dependent spreading of the carrier packet. It can be quantified in terms of an apparent diffusivity, $D(E,\sigma,\Sigma)$, that depends on both the degree of disorder and the electric field. In Fig. 9, the quantity

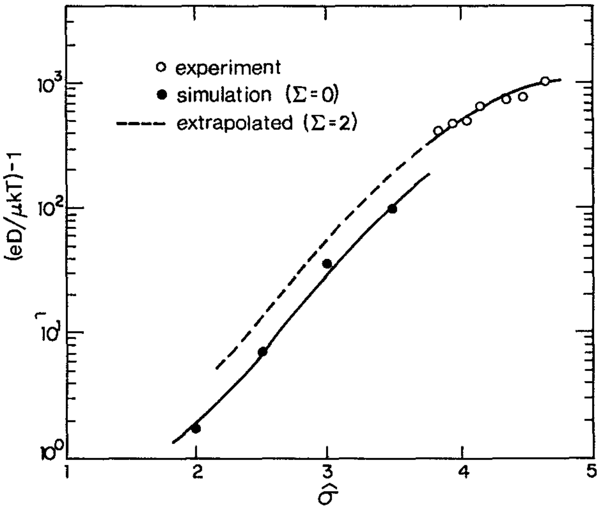

FIG. 9. $(eD/\mu kT) - 1$ determined from the tail of the photocurrent transients (open cirlces) and simulations for $\Sigma = 0$ (solid circles) and $\Sigma = 2$ (extrapolated), respectively. The field was 2×10^5 V/cm. From L. Pautmeier, R. Richert, and H. Bässler (Ref. 16).

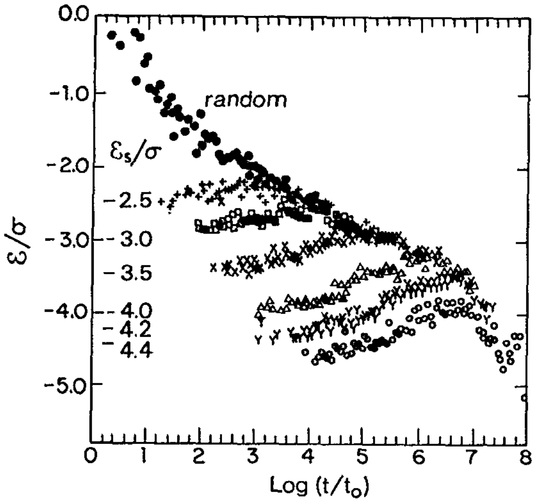

FIG. 10. Energetic relaxation of charge carriers generated at random and at specified energies within a Gaussian DOS of width σ as a function of time. The time is normalized to the dwell time of a carrier on a hopping site in the absence of disorder. The simulation consisted of 8000 lattice planes ($a = 6$ Å, wave function overlap parameter $2\gamma a = 10$, $E = 6 \times 10^5$ V/cm, and $\hat{\sigma} = 3.5$.

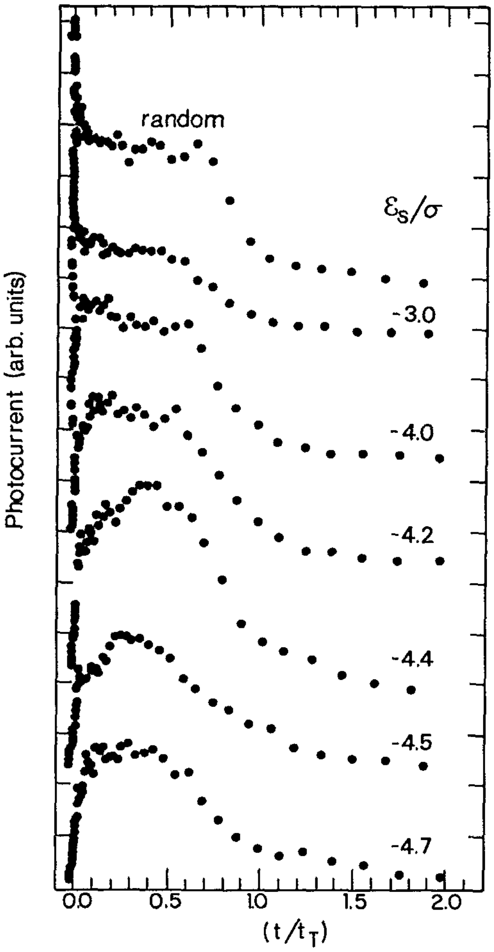

FIG. 11. Simulated photocurrent transients parametric in the start energy ϵ_s/σ (relative to the center of the DOS) in a semilogarithmic representation. Note that the arrival time is virtually independent of ϵ_s/σ. The field was 6×10^5 V/cm.

$f(E) - 1 = (eD/\mu kT) - 1$ is plotted vs $\hat{\sigma}^2$ and compared with simulation data for the case of vanishing off-diagonal disorder ($\Sigma = 0$). The comparison illustrated in Fig. 9 measures the deviation from classic Einstein behavior. For $\Sigma = 2$ and $f(E) - 1$, the data are approximately a factor of 2 larger. Unfortunately, the very substantial increase in computational time prevents simulating the case for $\hat{\sigma} > 3.5$. It is, nevertheless, obvious that the experimental $f(E) - 1$ curve is a continuation of the simulation data. This demonstrates that the tails of the photocurrent transients are consistent with the disorder parameters inferred from the field and temperature dependencies of the mobility, yet inconsistent with the concept that only part of the temperature dependence of the mobility is due to disorder.

Anticipating that the above inconsistencies have a common origin which is related to energy-selective, i.e., nonstatistical, carrier injection into the DOS at the interface between the emitter and transport layer, we conducted a Monte Carlo simulation of this phenomenon. The procedure was the same as in earlier studies.[18,19] We selected a cubic sample consisting of 8000 lattice planes ($a = 6$ Å) with energetic disorder only ($\hat{\sigma} = 3.5$, $\Sigma = 0$) and an electric field of 5×10^5 V/cm. Contrary to earlier studies, the carriers were started at specific energies. It should be noted that the inclusion of off-diagonal disorder does not affect the energetic relaxation and, concomittantly, the photocurrent relaxation to any significant degree. Figure 10 shows a family of plots of the carrier mean energy as a function of time and initial energy, ϵ_s. As ϵ_s decreases $\langle \epsilon(t) \rangle$ remains constant for an increasing span of time and passes through a maximum of increasing magnitude before merging into the $\langle \epsilon(t) \rangle$ curve obtained under energetically random starting conditions. Appearance of the maximum accounts for the fact that carriers, initially started at a site surrounded by higher energy neighbors only, require activation to higher energy sites before being able to begin relaxing to deeper states within the DOS. This relaxation pattern, in principle known from earlier studies,[19] is reflected in the associated photocurrent transients. The sequence of TOF plots illustrated in Fig. 11 confirms the intuitive notion that the initial drop of the photocurrent due to carrier equilibration gradually vanishes as ϵ_s approaches the equilibrium occupational density of states, known to be centered at $\langle \epsilon_\infty \rangle = \sigma\hat{\sigma}$ below the center of the DOS having a width σ. As ϵ_s drops below ϵ_∞, the photocurrent displays a fast initial rise, because carriers need to become activated before contributing to the photocurrent. It is followed by a gradual increase, reflecting the slow approach of the mean energy of the moving carrier packet towards equilibrium. If the fastest carriers reach the collecting electrode before equilibrium has been attained, a cusp evolves in the photocurrent transient replacing the plateu. This occurs when ϵ_s is swept across a relatively narrow energy slice $-4.2\sigma > \epsilon_s > -4.6\sigma$ below the center of the DOS. This effect is more clearly observed if TOF transients are plotted on a double logarithmic scale, as illustrated in Fig. 12.

The temperature-dependent appearance of a cusp in experimental TOF plots is in accord with the prediction of the simulations. As T increases, the equilibrium energy $\langle \epsilon_\infty \rangle = \sigma^2/kT$ relative to the center of the DOS. This is illustrated

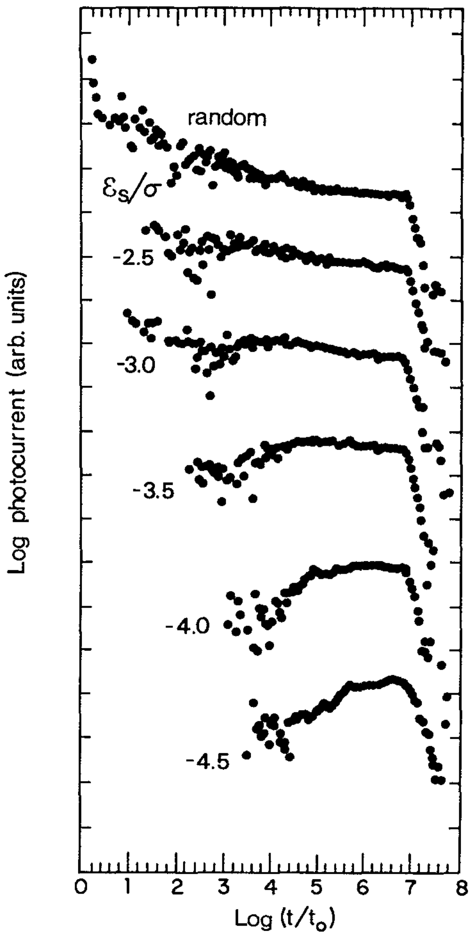

FIG. 12. Simulated photocurrent transients in a double logarithmic representation, parametric in the start energy ϵ_s/σ.

in Fig. 13 for a DOS having a Gaussian width of 0.1 eV. Within a temperature range, $270 < T < 300$ K, $\langle \epsilon_\infty \rangle$ drops by 0.04 eV. If holes are injected from a photoemitting electrode with a fixed energy of the valence states, the energy mismatch increases by the same amount. This is in agreement with the simulation results.

Additional support for this interpretation comes from the field dependence of the cusp. Previous simulations indicated that $\langle \epsilon_\infty \rangle$ increases with the field and causes the mobility to increase accordingly.[16] For $\sigma = 0.1$ eV, the equilibrium energy of excess holes is expected to drop by 0.030 eV within a field range $0 < E < 6 \times 10^5$ V/cm. Increasing E, therefore, contributes to the energy mismatch between the injection level and the equilibrium transport energy that has to be compensated by thermal activation while the carriers traverse the sample. From the temperature dependence of the onset of the cusp, one can estimate the relative positions of the injection level and the center of the DOS of the transport medium, respectively. For an α-Se:MPMP interface, we estimate this difference, which is identical with the difference of ionization potentials, to be 0.5 to 0.6 eV. Since the cusp is not observed with the perylene:MPMP configuration, the difference is estimated to be 0.1 to 0.2 eV less. The ionization potential of α-Se has been reported as -5.9 eV.[20] Assuming the mean energy of hole transport states is 0.2 to 0.3 eV above this, would then place the injection level of α-Se at -5.6 to -5.7 eV below vacuum. This, in turn, suggests that the ionization potential of MPMP should be in the range of -6.1 to -6.3 eV, in agreement with solution electrochemical measurements.[21]

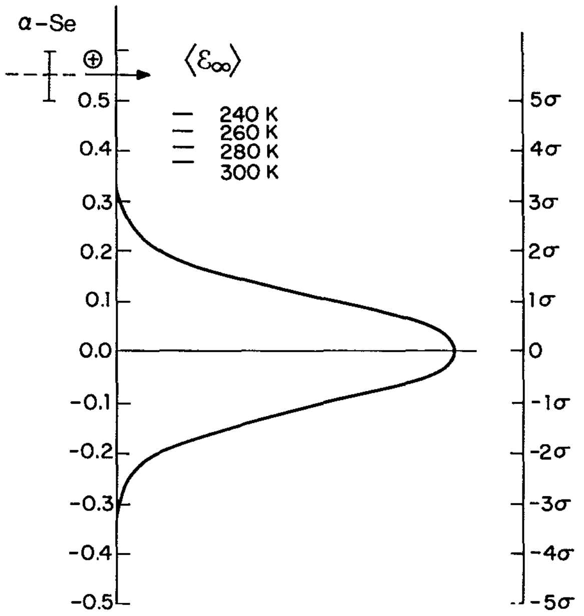

FIG. 13. Schematic illustration of the energetics of charge transfer at the interface between an α-Se photoemitting electrode and MPMP.

A necessary consequence of energy selective injection into states near the quasi-equilibrium energy is that the initial relaxation of carriers is diminished. This provides a straightforward explanation for the extension of the nondispersive transport regime to temperatures at which one would expect dispersive behavior otherwise.

V. CONCLUDING REMARKS

Apart from supporting the disorder formalism for rationalizing hole transport in MPMP, the present study establishes a method of describing the energetics of the charge transfer process at the interface between a photoemitting electrode and a transport medium by analyzing the shape of the photocurrent transients. The method is analogous to site selective optical spectroscopy of random media where spectral diffusion is diminished as a spectrally narrow excitation source is scanned across an inhomogeneously broadened absorption profile towards the tail states of the DOS.[22]

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