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A study of molecularly doped polymer-to-metal contacts by steady state injection current measurements

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Abstract. Temperature dependent characteristics of the steady injection current from various metal contacts into molecularly doped polymers were studied. The dopants were DEH, BD, and TPTA. Au contacts produced space-charge-limited currents in all polymers, i.e. were ohmic. Contacts of Cu (in some cases) and of Sn, Al, and In were emission limited, and gave space-charge-free currents containing both Arrhenius and constant/ T^2 temperature terms. The Arrhenius components correlated with the metal workfunctions. The ionization energies deduced for DEH and BD, namely 5.34 and 5.15 eV, respectively, agreed well with the published spectroscopic values. An experimental value for the ionization potential of TPTA, namely 5.42 eV, is found here for the first time. The results suggest steady state measurements as a viable alternative to UV spectroscopy for determining the ionization energies of doped polymers.

1. Introduction

Molecularly doped polymers (MDPs) are widely used in contemporary xerography as transport layers of double-layer organic photoreceptors. The base layer of these photoreceptors consists of a thin film of light-sensitive pigment, in which carrier pairs are photogenerated. This is overcoated by a thicker transport layer. One species of carrier, normally holes, is injected into the transport layer and traverses it without loss by recombination or trapping (see e.g. the review by Borsenberger and Weiss (1993)). The transport molecules are DEH, BD, and TPTA (figure 1). As a preliminary study, we carried out measurements of steady state dark-current injection from various metal electrodes into these transport polymers, which form the subject of the present paper.

We will show that one can infer the ionization energy of the transport molecules from such measurements. The ionization energies of DEH and BD have previously been obtained by spectroscopic means (see e.g. Kitamura and Yokoyama 1990, Kitamura and Chigono 1991). Although Kitamura and Yokoyama (1990) give values for a range of comparable amines, we could not find any report of an experimental value for the ionization energy of TPTA in the literature.

Previously studies of dark-current injection from metal electrodes into polyvinyl carbazole have been reported by Reucroft and Ghosh (1973, 1974) and Akuetey and Hirsch (1991). These show that, after applying the electric field, the current gradually decays to a steady value which is strongly dependent on the metal workfunction. Mori *et al* (1992) made steady dark-current measurements in evaporated BD between an indium–tin oxide anode and cathodes of various metals. They concluded that in these, probably microcrystalline, samples there was evidence of bipolar conduction unlike the case in doped polymer layers

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containing BD. Below 190 K the current was observed to be of Schottky type, above 190 K and at low fields it was space-charge limited; but above 1 MV cm^{-1} it was of Fowler–Nordheim type.

Abkowitz and Pai (1986) studied the hole current injected into polytetraphenyldiamine-(TPD-) doped polycarbonate by Au, Ag or C electrodes, and its temporal evolution. In all cases, the eventual steady current was space-charge limited, a condition which masks the energetics of the contact interface. The authors' main concern, however, was the interpretation of the field and temperature dependence of the current and its relation to the time-of-flight mobility.

In later work, Abkowitz and coworkers carried out similar experiments on the holetransporting, trap-free, condensation polymer, polytetraphenylbenzidine (PTPB), with Al, Au or C electrodes (Abkowitz *et al* 1993a, b, 1995, Abkowitz 1996). The carbon was either glassy or in the form of carbon-loaded polyimide. The latter contact was ohmic, the steady current being limited by space charge in the polymer. It agreed well with that calculated from the theoretical expression for the trap-free, space-charge-limited, current using mobility values determined by time-of-flight experiments. With the other electrodes, the current was emission limited, i.e. the contact was unable to supply a space-charge-limited current in the polymer. The authors pointed out that the ratio of the measured current to the theoretical space-charge limit provides a measure for the injection efficiency. The injection from Al was so weak that the contact could be regarded as blocking for holes. The blocking property of Al, of course, is well known, and is the reason why this metal is the preferred electrode in most of the numerous flight-time mobility measurements on hole-transporting polymers reported in the literature.

Interestingly, Abkowitz *et al* (1995) introduced a model to explain how an injection barrier of, say, 0.3 eV could be overcome by tunnelling into a polymer transport state as far as, say, 6 nm from the contact interface. At this distance, a sufficiently high applied field would shift the energetic position of the transport state to coincide with the metal Fermi level. In our opinion, however, the resulting tunnelling rate would become unrealistically low.

2. Experimental details

The doped polymers were prepared by dispersing the transport molecules in polycarbonate (PC) dissolved in dichloromethane, using donor-type dopants which were DEH, TPTA, and BD (see figure 1). All dopant concentrations were 40% by weight. The layers were coated by the wire-bar technique onto aluminized or tinned polyethylene terephthalate films. Sandwich type cells were formed by vapour depositing Au, Cu, Sn, Al, or In contacts on the free surface. The transparency of the top electrodes was controlled by optical transmittance measurement during vacuum deposition and was between 15 and 20%. The thickness of the layers was measured by an Elcometer 300 thickness meter, which offered a 0.1 μ m accuracy. The thickness was 14–20 μ m and the electrode area 0.3–4 cm², the latter controlled by masks. The maximum electrode size was dictated by the geometry of the sample holder in the cryostat.

DC current measurements were carried out in the temperature range of 188–348 K, limited by the minimum detectable signal at the lower end and by the glass transition temperature at the higher end of the range. The equilibrium steady state current was recorded by a Keithley electrometer. Readings were taken systematically 2 h after each voltage step (the decay upon stepping the voltage initially was of 1/t type but became imperceptible after 2 h). The final currents were in the range of 10^{-11} – 10^{-3} A. The measurements were



Figure 1. Chemical structures of the dopant donor molecules studied.

checked on three samples for each structure studied. The results presented below are from representative samples.

3. Results and analysis

Figure 2 shows the steady current–voltage characteristics of the metal–polymer combinations examined at room temperature (298 K), with the following exceptions: (i) the In–DEH data are for 338 K and (ii) the In–TPTA data for 328 K as, in both cases, the currents at room temperature were too low for reliable recording; (iii) the Au–BD and Cu–BD points (which practically coincide, Au giving the slightly higher currents) refer to 288 K, as here the current at room temperature was too high, even for the smallest contact area. It is clear that the highest currents were produced by Au electrodes for each composition, and the lowest by In electrodes. The magnitudes of the In and Al currents suggest blocking contacts, while the Cu and Au currents provide strongly injecting contacts. The above observations for Al and Au agree for TPD with the results of Abkowitz and Pai (1986) and for PTPB with those of Abkowitz *et al* (1993a, b, 1995, Abkowitz 1996).

To study the current-field characteristics, we plotted $\log(J/\mu)$ against log *F*, where *J* is the current density, μ the hole mobility in the polymer, and *F*(= voltage/thickness) the applied field (figure 3). The J/μ representation is used because, in general, μ itself is field dependent. The mobility data were time-of-flight results taken from the work of Schein *et al* (1986), Veres (1995), and Schein and Borsenberger (1993) for DEH, Borsenberger (1990) and Veres (1995) for TPTA, and Nemeth-Buhin and Juhasz (1995) and Veres (1995) for BD. The published mobility data are best described by an exp(-constant/ T^2) temperature



Figure 2. Current–voltage characteristics for the metal–polymer contacts, all at 298 K except In–DEH–338 K, In–TPTA–328 K, and Au–BD and Cu–BD–288 K (layer thickness: DEH, 14 μ m; TPTA, 20 μ m; BD, 14 μ m).

dependence although, in the case of DEH, Schein and his co-workers could not make a clear distinction between an Arrhenius or a constant/ T^2 type behaviour. We carried out time-of-flight measurements to check the mobilities in our DEH, TPTA, and BD layers, and found them to agree well with the published data. In this paper we will follow the constant/ T^2 type description of the mobility as this seems to offer the best fit for all the available data.

With Au contacts to all polymers, and also with a Cu contact to BD, the current was observed to be fully space-charge limited at all temperatures and fields. Examples are the uppermost lines in figure 3. These not only show slopes of two, but are in good quantitative agreement with the theoretical expression $J/\mu = \frac{9}{8}\varepsilon\varepsilon_0 F^2/L$ (Mott and Gurney 1948). The observation of a space-charge-limited current implies on ohmic contact, in which the injection is so strong that a virtual anode forms inside the polymer, screening the interface. Such a contact can give no information about the interface energetics (other than that the barrier to injection must be very low) and, therefore, is of no further interest in this investigation.

For Cu–DEH samples at high temperatures (not illustrated), the plots of $\log(J/\mu)$ against log *F* had slopes between unity and two, suggesting space-charge perturbation. However, the slopes became unity at around room temperature (figure 3). For all other combinations, the slopes were unity throughout the practical temperature range. In these cases we interpret the contacts as weak, emission-limited, Mott contacts (Mott and Gurney 1948), in which the thermal equilibrium concentration p_0 just inside the polymer satisfies the condition $ep_0 \ll CF$ where *C* is the sample capacitance per unit area. Then p_0 is fully determined by the interface energetics; the carrier concentration throughout the sample is constant and equal to p_0 ; and the current is given by $J = ep_0\mu F$ as observed.



Figure 3. J/μ –F characteristics of metal–polymer contacts at room temperature except 338 K for In–DEH, 328 K for In–TPTA, and 288 K for Au–BD and Cu–BD). *F* is the average field i.e. voltage/thickness.

The conduction mechanism in MDPs is by hopping through a manifold of localized states. For the linear, i.e. space-charge-free, $J/\mu-F$ characteristics, the corresponding conductance J/F was found to show a quasi-Poole–Frenkel-type field dependence (figure 4(a)). This is in agreement with observations by other authors for metal interfaces with a number of polymers in which conduction is by hopping, and has been interpreted as Richardson–Schottky emission into the polymers (Mori *et al* 1992, Kanemitsu and Imamura 1987, Reucroft and Ghosh 1974). However, Akuetey and Hirsch (1991) and Abkowitz *et al* (1995) have pointed out that the Richardson–Schottky theory is strictly applicable to emission into free, or quasi-free, space only and is not a proper description for emission into the localized states of the hopping manifold. An appropriate description is an emission-limited Mott contact (see above), with the conduction or valence band of the insulator replaced by the manifold of hopping states. The field dependence of the conductance may then be attributed to that of the mobility. The temperature dependence of the conductance was nearly Arrhenius type but showed deviations from it in favour of a constant/ T^2 -type dependence at high fields and low temperatures (figure 4(b)).

To relate the conductance to the interface energetics, we use the Mott contact model as modified for a Gaussian distribution of polymer transport states (figure 5) by Akuetey and Hirsch (1991). The thermal equilibrium hole concentration in the polymer at the interface is given by them as

$$p_{0} = (2\pi\sigma^{2})^{1/2} N_{m} \exp\left(-\frac{\Delta E_{0}}{kT}\right) \int_{-\infty}^{\infty} \exp\left(\frac{\varepsilon}{kT} - \frac{\varepsilon^{2}}{2\sigma^{2}}\right) d\varepsilon$$
$$= N_{m} \exp\left[-\frac{\Delta E_{0}}{kT} + \frac{1}{2}\left(\frac{\sigma}{kT}\right)^{2}\right]$$
(1)





Figure 4. The typical dependence of space-charge free conductance on square root of field (*a*) and temperature (*b*) (sample, Al–BD, 14 μ m thick).

where σ is the diagonal disorder of the energy of the transport states in the polymer, N_m is the total number of the molecular transport states, ΔE_0 is the difference between the mean energy of the transport manifold and the metal Fermi level, and ε is the energy measured from the mean energy of the transport manifold towards the vacuum level. The model implies that any surface traps present are masked by the thermal equilibrium across the interface. Similarly, a thin oxide layer on the metal is not neglected.

The quasi-Poole–Frenkel-type field dependence of the conductance (figure 4(a)) agrees with the concept of carrier drift through a Gaussian transport manifold in the polymer. In this situation the drift mobility is modelled by Bässler (1993) as

$$\mu = \mu_0 \exp\left[-\left(\frac{2}{3}\frac{\sigma}{kT}\right)^2\right] \exp\left\{C\left[\left(\frac{2}{3}\frac{\sigma}{kT}\right)^2 - \Sigma^2\right]F^{\frac{1}{2}}\right\}$$
(2)

where μ_0 is a prefactor, *C* is an experimental coefficient, and Σ describes the off-diagonal disorder of the transport manifold. As a check, taking σ from the time-of-flight (TOF) results of Schein *et al* (1986) for DEH, Borsenberger (1990) and Veres (1995) for TPTA, and Nemeth-Buhin and Juhasz (1995) for BD, the parameters Σ and *C* were derived from the field dependence of the conductance and agreed well with the corresponding values obtained by the time-of-flight measurements (table 1).

The zero-field conductance G_0 may therefore be written

$$G_0 = \left(\frac{J}{F}\right) = ep_0\mu(F=0) = eN_m\mu_0 \exp\left\{-\frac{\Delta E_0}{kT} + \frac{1}{2}\left(\frac{\sigma}{kT}\right)^2 - \left(\frac{2}{3}\frac{\sigma}{kT}\right)^2\right\}.$$
 (3)

The temperature dependence of the extrapolated zero-field conductance is shown in figure 6. The slopes of the zero-field conductance G_0 divided by the non-Arrhenius type terms, i.e. G_0/g where $g = \exp[(\frac{1}{2} - \frac{4}{9})(\sigma/kT)^2]$, should reveal the interface barrier ΔE_0 arising from the energy mismatch between the mean transport energy and the metal Fermi level. We



Figure 5. Interface energetics of a metal–polymer contact (EF, metal Fermi level; E_{gd} , metal ground state; E_0 , mean of the Gaussian transport manifold in the doped polymer). (After Akuetey and Hirsch 1991.)

Table 1. Positional disorder Σ and field-effect *C* parameters of drift mobilities inferred from time-of-flight (TOF) measurements, and from injection currents through metal–polymer interfaces. TOF results are taken from ^a Schein *et al* (1986), ^b Borsenberger (1990) or (identically) Veres (1995), ^c Nemeth-Buhin and Juhasz (1995) or (identically) Veres (1995).

	$\begin{array}{c} \text{DEH} \\ \sigma = 0.127 \text{ eV}^{\text{a}} \end{array}$		$\sigma = 0.12 \text{ eV}^{\text{b}}$		$\begin{array}{c} \text{BD} \\ \sigma = 0.11 \text{ eV}^{\text{c}} \end{array}$	
	$C \ ({\rm cm} \ {\rm V}^{-1})^{1/2}$	Σ	$\overline{C \ (\text{cm V}^{-1})^{1/2}}$	Σ	$\overline{C \ (\text{cm V}^{-1})^{1/2}}$	Σ
TOF	$2.9 imes 10^{-4}$	2.79	$2.4 imes 10^{-4}$	3.90	$2.5 imes 10^{-4}$	1.91
Cu	_		$2.3 imes 10^{-4}$	3.74	_	
Sn	$2.8 imes 10^{-4}$	2.84	2.4×10^{-4}	3.48	$2.5 imes 10^{-4}$	1.68
Al	$3.0 imes 10^{-4}$	3.02	$2.5 imes 10^{-4}$	3.96	$2.6 imes 10^{-4}$	2.08
In	—	—	2.4×10^{-4}	4.02		—

note, however, that g is close to unity due to the numerical factors involved so the terms containing σ in (3) are small, and the TOF results are not crucial. As a result of this we have a new way to determine the energy mismatch between the metal Fermi level and the mean of the manifold of the transport states in the MDP.

The mean values derived for ΔE_0 from three samples of each kind are listed in table 2. The mean depth of the transport states E_0 below the vacuum level is calculated from the metal workfunctions E_F as $E_0 = E_F + E_0$. The metal workfunctions were taken from Michaelson (1978). These values apply to ultra-clean metal surfaces, whereas our electrodes were evaporated in an ordinary, untrapped, silicone-vapour-pumped laboratory system, so that the interfaces would certainly have been partly contaminated. We argue that, nevertheless, the clean-metal values are applicable. This is because, as long as the electrodes possess bulk metal properties, and the contamination barriers are insulating or only weakly semiconducting and sufficiently thin, the band bending across the barriers is negligible. Thermal equilibrium between the metal Fermi level and the polymer transport



Figure 6. The temperature dependence of extrapolated zero-field conductance $G_0 = (J/F)_0$ and G_0 divided by the non-Arrhenius factors g (see text).

Table 2. Activation energies ΔE_0 and mean depth of transport states $E_0 = E_F + E_0$.	The last
column lists the published spectroscopic values of ionization energies.	

	$\begin{array}{l} \text{Cu} \\ E_F = 4.65 \text{ eV}^{\text{a}} \end{array}$	$ Sn $ $ E_F = 4.42 \text{ eV}^a $	Al $E_F = 4.28 \text{ eV}^a$	In $E_F = 4.12 \text{ eV}^{a}$	Ionization energy (eV)
DEH	_				
ΔE_0 (eV)		0.92 ± 0.05	1.07 ± 0.07		
E_0 (eV)		5.34 ± 0.05	5.35 ± 0.07		5.32 ^a
TPTA				_	
ΔE_0 (eV)	0.79 ± 0.04	0.97 ± 0.05	1.14 ± 0.08		
E_0 (eV)	5.44 ± 0.04	5.39 ± 0.05	5.42 ± 0.08		5.4 ^a
BD	—				
ΔE_0 (eV)		0.72 ± 0.04	0.88 ± 0.05	1.02 ± 0.08	
E_0 (eV)		5.14 ± 0.04	5.16 ± 0.05	5.14 ± 0.08	5.11 ^a

^a For references see the text.

level is established by tunnelling through the barrier, whose precise nature and thickness are immaterial (cf Akuetey and Hirsch 1991). Of course, the presence of a barrier reduces the tunnelling rates. This could lead to an eventual field saturation of the contact, but such a transition was never observed.

The ionization energy cited for DEH is the average value of the results of Kitamura and Yokoyama (1990), Kitamura and Chigono (1991), Yajima and Kawamura (1993), and Kanemitsu *et al* (1991); the ionization energy for TPTA is an *estimate* based on molecular size from a range of comparable amines reported by Kitamura and Yokoyama (1990)—the value of 5.7 eV cited by Veres and Juhasz (1997) is actually that reported for TPA; and the ionization energy for BD was taken as an average between the results of Kitamura and

Chigono (1991), Enokida *et al* (1990), and Hayashi *et al* (1992). From the good agreement with the published ionization potential data we conclude that ΔE_0 , as derived from the Arrhenius term of the conductance, represents the energy barrier at the interface.

The 1/T = 0 intercepts derived according to figure 6 should correspond to $eN_m\mu_0$ (see (3)), thus knowing N_m from the concentration and molar weight of dopants and the density of the polymers ($N_m = 1.4 \times 10^{21}$ cm⁻³ for TPTA, 8.3×10^{20} cm⁻³ for DEH, and 8×10^{20} cm⁻³ for BD) should reveal the mobility prefactor μ_0 . This may then be compared with the values independently obtained from TOF experiments (table 3). The agreement with the drift mobility is again remarkable. This shows that the transport manifold is not fully occupied but the steady state current carriers traverse the sample in the same way as the thermalized mean of a charge packet, in contrast to the conclusions of Akuetey and Hirsch (1991).

Table 3. Drift mobility prefactors μ_0 (cm² V⁻¹ s⁻¹) inferred from TOF measurements (for references see table 1) and injection currents through metal–polymer interfaces.

	DEH	TPTA	BD
TOF	$9.2 imes 10^{-4}$	$1.1 imes 10^{-1}$	1.1×10^{-3}
Cu	_	$5.8 imes 10^{-2}$	_
Sn	$5.4 imes 10^{-4}$	$7.8 imes 10^{-2}$	$8.8 imes 10^{-4}$
Al	$7.2 imes 10^{-4}$	$9.0 imes 10^{-2}$	$7.9 imes 10^{-4}$
In	—	$8.2 imes 10^{-2}$	—

4. Discussion

To maintain a constant, thermal-equilibrium, carrier concentration p_0 in the polymer independent of current, the forward current injected across the contact interface must exceed the current drawn at the highest field. In the classical Mott contact, p_0 is stabilized by backward thermal diffusion. In the metal–polymer contact, the only way a charge can reach a localized state in the doped polymer is by tunnelling. The tunnelling probability decreases exponentially with the distance of the state from the interface and with the square root of its potential energy. The number of carriers available for forward tunnelling is determined by the Fermi–Dirac distribution in the electrode metal. The saturated tunnelling current into a Gaussian distribution of transport states is field independent and has the form (Akuetey and Hirsch 1991)

$$J \cong 3 \times 10^{10} \sigma (E_0 - E_{gd}) \exp\left(-\frac{\Delta E_0 - \sigma}{kT}\right) \exp(-\alpha \langle R \rangle) \tag{4}$$

with energies in electron volts, where E_{gd} is the ground state of the electrode metal (figure 5), α is the decay constant of a carrier localized on a site and $\langle R \rangle$ is the average separation of the sites at the hopping level. With a total site concentration of $N_m \approx 10^{21}$ cm⁻³ in all three polymers, $\langle R \rangle \approx 1$ nm for all. Taking for α a reasonable value of 7 nm⁻¹ (see e.g. Borsenberger 1990) in each case, we estimate currents several orders of magnitude higher than the maximum measured steady currents at all temperatures and fields. This confirms that the supply rate is always sufficient to maintain a thermal equilibrium condition, even in the In–TPTA system where the interface barrier is the highest (0.12 eV). A non-thermalequilibrium, trap-release-controlled, current was observed by Akuetey and Hirsch (1991) at an Al–PVK interface where the barrier is ~ 2 eV. However, unlike the case here, such a current gives a strongly non-linear J/μ –F characteristic.

The Arrhenius-type component of the activation energies measured under space-chargefree conditions showed a clear correlation with the metal Fermi levels published by Michaelson (1978). The correlation allows calculation of the mean energy of the transport manifold, which should correspond to the ionization potential of the transport molecules in the polymer, i.e. $E_0 = E_F + E_0$ (figure 5). Ionization potentials have been measured by a number of authors using ultraviolet (UV) photoemission spectroscopy. The agreement between our results and the ionization energies obtained from the UV emission spectra, summarized in table 2, suggests that our approach is self-consistent and that the use of the clean-metal workfunctions is justified. We therefore conclude that the ionization energies of MDPs can be deduced from injection current measurements.

5. Summary and conclusion

Interface phenomena were studied by way of equilibrium dark-current measurements on polycarbonate layers doped with DEH, TPTA, and BD and furnished with various metal electrodes. The currents were space-charge limited for Au contacts to all polymers and for the Cu–BD combination and also for Cu–DEH at high temperatures. However the currents were space-charge free for In, Al, and Sn contacts to all polymers, as well as for the Cu–TPTA structure. In these cases, the contacts were interpreted to be of emission-limited, thermal equilibrium, Mott type, masking any interface traps and providing a temperature dependence for the current with both Arrhenius and constant/ T^2 terms. The Arrhenius activation energies correlated well with the metal Fermi energies. This suggested that these energies represent the difference between the metal Fermi level and the transport level in the polymer, and thus led to the determination of the ionization potentials. The results agree well with the published spectroscopic data for DEH and BD. For TPTA, no such data are available, but our result, 5.42 eV, is comparable with the literature values for similar amines.

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