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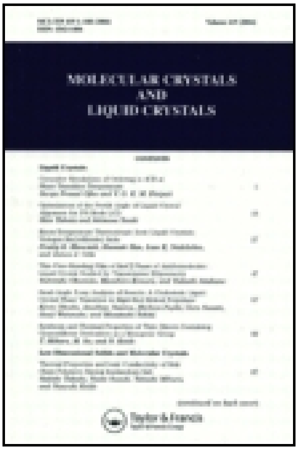

Molecular Crystals and Liquid Crystals

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Application of Semiconducting Electrodes to Measurements of Charge Carrier Mobilities in a Non-photoconducting Organic Crystal; 1,3,5-trinitrobenzene

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Application of Semiconducting Electrodes to Measurements of Charge Carrier Mobilities in a Non-photoconducting Organic Crystal; 1,3,5-trinitrobenzene

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Semiconducting electrodes have been used to photoinject holes and electrons into the non-photoconducting crystal 1,3,5-trinitrobenzene. With these electrodes, the drift mobility tensors for holes and electrons have been measured. Mobilities are $\sim 10^{-2}$ cm^2 V · s and are weakly anisotropic. All mobilities are weakly thermally activated. The overall current transient shape used to calculate these mobilities depended upon the photoconductor used as the electrode, and the major differences are consistent with the presence of interface potential barriers. The carrier injection mechanism is discussed and the field dependence of the injection efficiency evaluated.

1. INTRODUCTION

The mobility of charge carriers in insulators is usually studied by the time-of-flight technique.[1] Despite its conceptual simplicity the widespread use of this technique is limited both by: the necessity to inject a sufficient amount of charge into the insulator and by the

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trapping of charge carriers. In photoconductors, charge carriers are, as a rule, generated using short pulses of strongly absorbed light. For materials which do not exhibit sufficient photoconductivity the time-of-flight technique may be employed using excitation with an electron gun or photoinjection of carriers from an electrode; a metal or semiconductor. The latter method has been successfully applied to many inorganic materials, but very few organics[2-7] in most cases, due to the complexities of generation and transport of charge carriers in such inhomogeneous systems, the analysis of the experimental results proves difficult. Moreover, the most important properties characterizing organic semiconductors such as the energetic positions of the particle and quasiparticle excitation, their transport properties and interactions, are generally poorly understood.

This paper presents measurements of the charge carrier drift mobility tensor in crystals of 1,3,5-trinitrobenzene (TNB), a material well known for its ability to form charge-transfer complexes. Since this crystal is non-photoconducting we used photoinjection from semiconducting selenium (Se) and cadmium telluride (CdTe) layers. Characteristic features of the photoinjection process are discussed.

2. EXPERIMENTAL

The starting material, commercial 1,3,5-trinitrobenzene ($C_6H_3(NO_2)_3$ from BDH Chemicals Ltd., Poole England) was recrystallized from carbon tetrachloride, vacuum sublimed and further purified by zone-refining using several hundred of zone passes under a nitrogen atmosphere. The single crystals were grown from the melt by the Bridgman technique. The crystal boules obtained were quite large (about 2.8 cm long and 1.2 cm in diameter), and allowed a series of plane-parallel samples to be cut with planes perpendicular to the three main crystallographic directions. Crystallographic alignment of the samples was done by standard x-ray techniques. The samples were approximately 1 cm^2 in area and 0.2–0.8 mm thick.

All plates were carefully polished with a tissue wetted with benzene before depositing the evaporated semiconductors contacts. Thin layers of photoconducting Se or CdTe were evaporated in vacuo (about 10^2 Pa), the crystal being kept at room temperature. The thicknesses of the deposited films were estimated to be below 1 μm, but this thickness varied slightly from sample to sample.

The drift mobility measurements were performed by a time-of-flight technique in which a slab of insulator is sandwiched between a

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transparent conducting glass as a front contact and a brass plate mounted on soft spring as a back contact. Sample temperature could be varied above and below room temperature and was monitored by a copper-constantan thermocouple. Carrier motion[8] was monitored on an oscilloscope screen via the current in an external circuit. The transit time of the charges was marked by a sharp decrease in the monitored current. Duration of the exciting light pulse (with photon energies within the range 2–4 eV) was about 1 μs, and the intensity of the light pulse was kept sufficiently low to avoid space charge effects. A sample test with anthracene crystals gave mobilities equal to literature values.

3. RESULTS AND DISCUSSION

3.1. Charge carrier mobilities

Crystalline 1,3,5-trinitrobenzene with blocking contacts showed no noticeable photoconductivity. Attempts were made to photoinject carriers into TNB from such metals as Au, Ag, Al but in vain (signals were close to the noise level). Effective photoinjection of electrons has been found when Se electrodes were used[9] and films of CdTe were found to photoinject case both electrons and holes. Injection efficiency from CdTe electrodes was much lower than for Se, by one to two orders of magnitude.

Figures 1 and 2 show typical current transients which appeared when the light pulse illuminated Se–TNB and CdTe–TNB junctions, respectively. The charge carriers, which give rise to the transients shown in Figure 1 are electrons. From experiments on different crystal thicknesses and illuminating the Se layer directly or indirectly through the crystal and observing transients for different polarisations of biasing voltage, it was shown unambiguously that transients of electrons generated in Se through TNB samples were being observed.

One can readily see that the shapes of electron transients for Se–TNB and CdTe–TNB are markedly different. This is due to the difference in photoinjection properties between Se and CdTe deposited onto TNB. The efficiency and rate of charge injection from such electrodes will depend on; the nature of the photogeneration process within the electrode; its transport properties; the height and shape of potential barrier formed for carriers being attracted by field from the place of generation to the bulk of insulator, as well as on the space charge (within the insulator and in the interface region). However, for the electrodes used here, the characteristic shapes of the

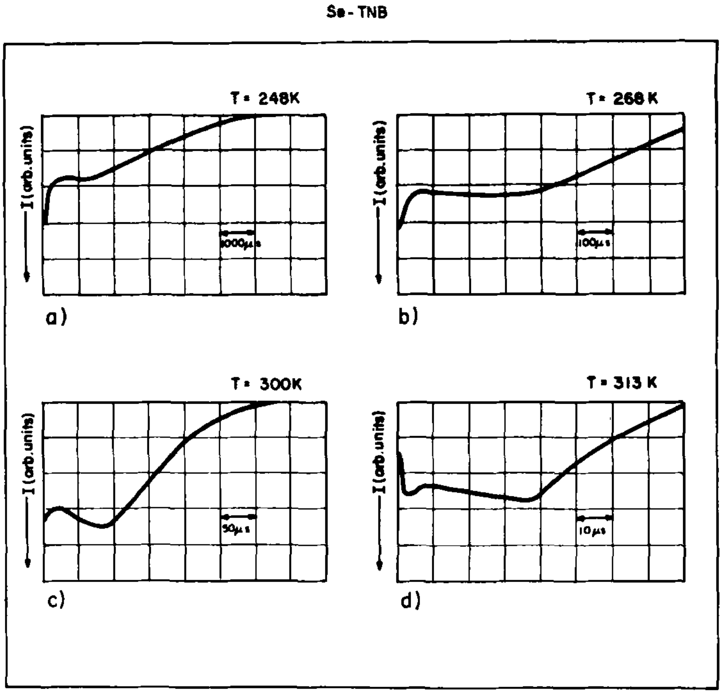

FIGURE 1 Electron current transients in Se–TNB system. Horizontal scale: (a) 1000 μs, (b) 100 μs, (c) 50 μs, (d) 10 μs. $V = 465$ V, thickness of the TNB plate (*c*) amounts to 0.23 mm.

transients are similar to those expected for charge-carrier generation in a narrow region close to the sample surface, where the generation time is short compared to the transit time. The sharp kink in the current transient marks the transit time t_t of charge carriers through the sample. The effective mobility μ was evaluated by measuring t_t and using the well-known formula[10] $\mu = L^2/V \cdot t_t$ where L is the sample's thickness and V stands for the voltage applied.

With Se or CdTe electrodes the mobility in TNB was found to be field-independent for fields up to 5×10^6 V m^{-1} (Figure 3). As expected[10] the experimental points lie on straight lines intersecting the origin and also the transit times showed a square dependence on sample thicknesses.

The temperature dependence of the mobilities of both holes and electrons were studied in detail for the three main crystallographic directions in the range 200–360 K. These results are shown in Figure 4 and Figure 5. Since TNB is orthorhombic (Pbca),[11] the full mobility tensor is known when mobilities along *a*, *b* and *c* are determined.

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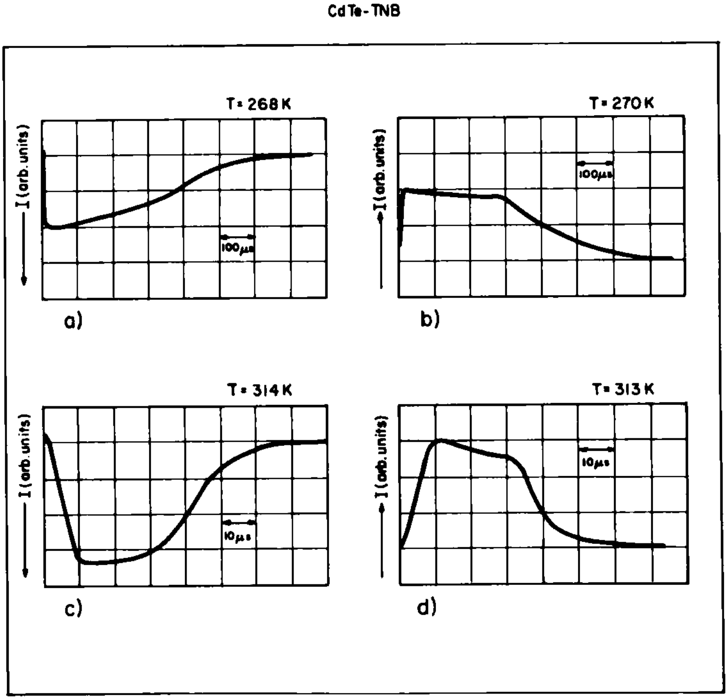

FIGURE 2 Current transients obtained in CdTe–TNB system for electrons (a) and (c) and holes (b) and (d). Horizontal scale: (a) and (b) 100 μs; (c) and (d) 10 μs. $V = +465$ V, thickness of TNB sample amounts to 0.23 mm.

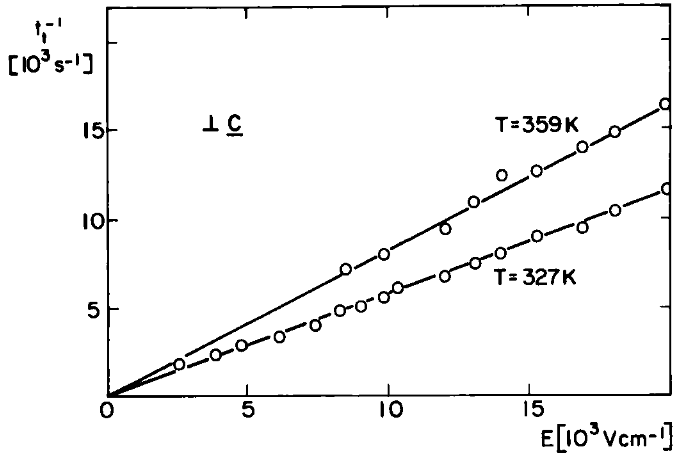

FIGURE 3 The plot t_t^{-1} vs E for the plate of Se–TNB cut perpendicularly to the crystallographic axis a, b and c.

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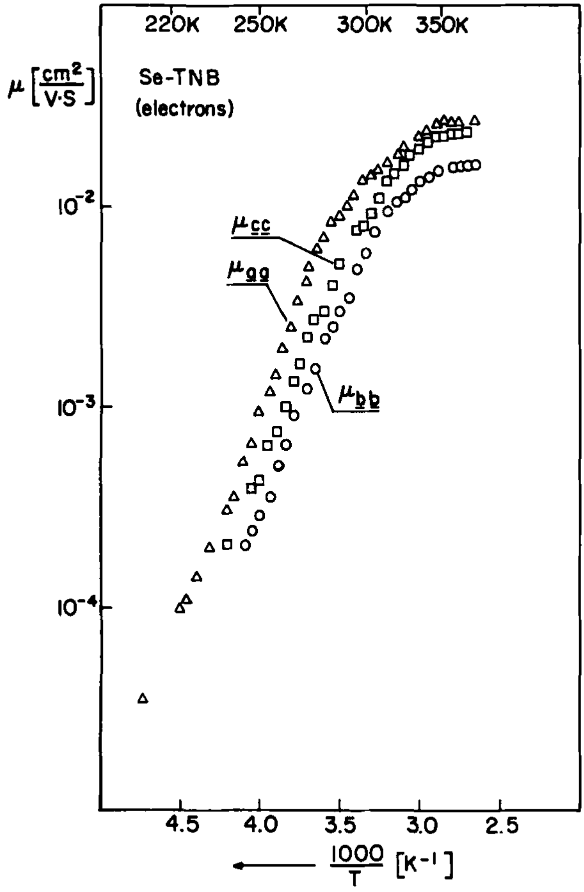

(a)

FIGURE 4 Temperature dependence of electron drift mobilities for (a) Se–TNB and (b) CdTe–TNB systems, with plates of TNB cut perpendicularly to the crystallographic axis *a*, *b* and *c*.

Close inspection of the experimental results shown in Figure 4 reveals a systematic difference between the magnitudes of electron drift mobilities for Se–TNB and CdTe–TNB systems (especially for high temperature region) whereas the anisotropy remains the same. Nevertheless, the temperature dependences of electron drift mobility in TNB measured with Se or CdTe photoinjecting electrodes are very similar, which suggests that their shape does not depend on electrode properties but only on trapping phenomena and the anisotropy factors of single crystal TNB itself. The difference in the values measured for Se and CdTe electrodes will be further discussed in the subsequent section.

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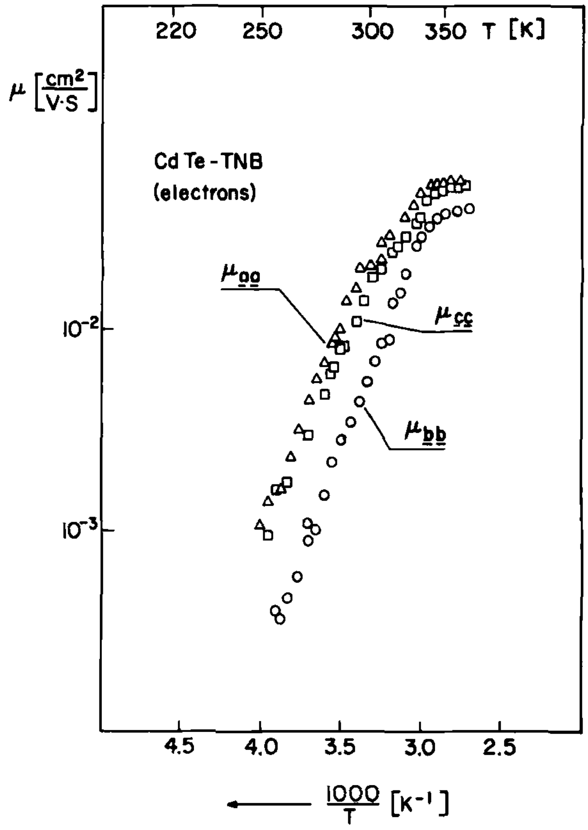

(b)

FIGURE 4 *Continued*

The form of temperature dependence of mobilities in *a*, *b* and *c* crystallographic directions suggests that in the low temperature (200–300 K) region mobilities are thermally activated, the activation energies are ~ 0.37 ± 0.02 eV for electrons in all directions and 0.4 ± 0.1 eV and 0.35 ± 0.1 eV for holes at (*a*, *c*) plane and for *b* direction respectively.

The activated temperature dependence is characteristic of a mobility limited by shallow trapping;[12] though the nature of traps responsible for this effect (impurities or structural defects) is unknown but it should be noted that similar results were obtained on three or more crystal plates for each direction.

It can be seen in Figure 4 and Figure 5, that the mobilities tend to saturate at higher temperatures (above *ca* 330 K). One may therefore suppose that in the high temperature region the effective mobility

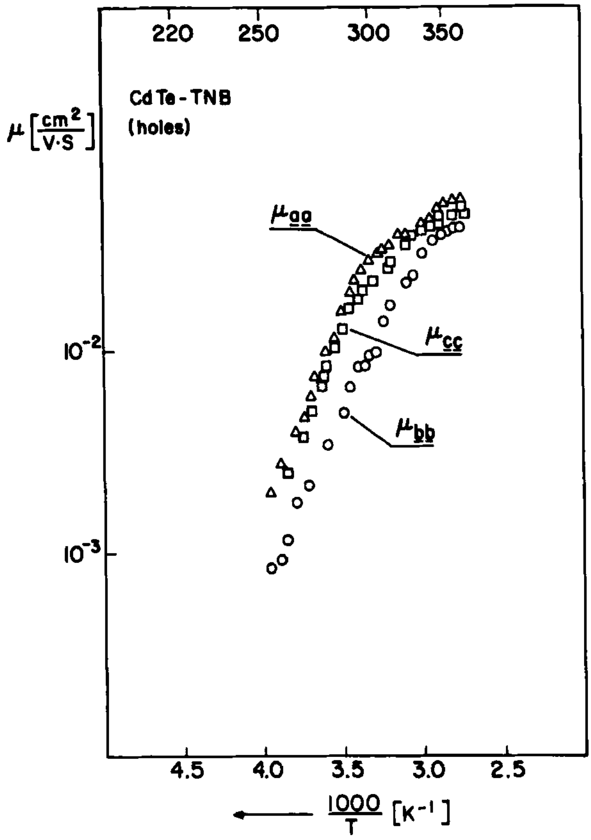

FIGURE 5 Temperature dependences of hole drift mobilities in a TNB single crystal for plates cut perpendicularly to crystallographic axes *a*, *b* and *c*.

approaches its microscopic value μ^0. If the mobility is controlled by a single set of traps their energies being taken as 0.37 eV for electrons, and 0.4 eV and 0.35 eV for holes (*a* and *c* and *b* direction, respectively), one can employ the well-known formula,[12,13] $\mu^0/\mu = 1 + N_t/N_v\exp(E_t/k_BT)$ where N_v is the valence (conduction) band effective density of states, N_t is the trap density, E_t is the trap depth and k_B is Boltzmann's constant, to calculate μ^0. This equation is valid provided that the trapping and release times are both short compared to the effective transit time and only for the limiting case where the lattice mobility can be described by the band model. When mobility is described by the hopping model, equation (2) should be used in its generalized form. The generalized equation has the exponential term

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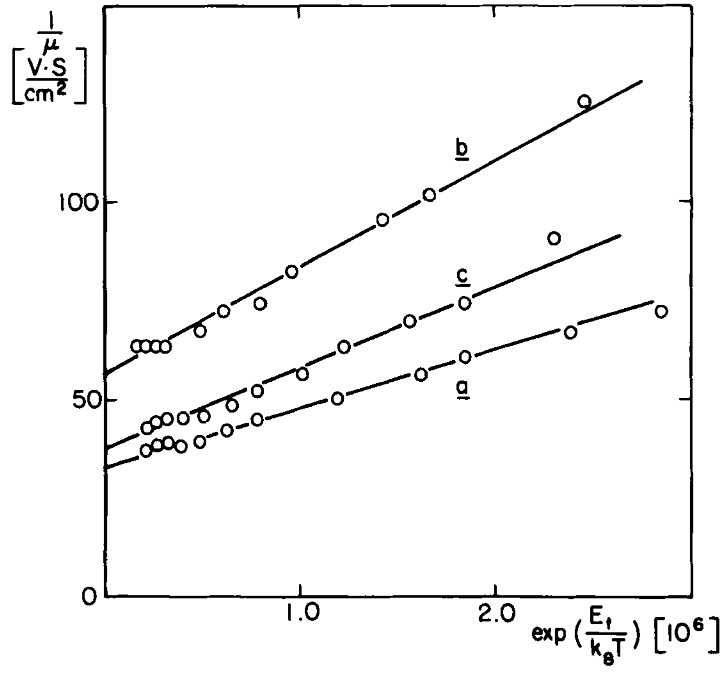

FIGURE 6 Evaluation of μ^0 and $N_t/N_V(\nu_V/\nu_t)$ ratio from plots of $1/\mu$ vs $\exp(E_t/kT)$ for a TNB crystal in *a*, *b* and *c* directions, using Se electrode as an injecting contact.

multiplied by an attempt-to-escape frequencies ratio term equal to ν_V/ν_t[13]

$$\frac{\mu^0}{\mu} = 1 + \frac{N_t}{N_V}\frac{\nu_V}{\nu_t}\exp\left(\frac{E_t}{k_B T}\right) \tag{1}$$

Indeed, at room temperature drift mobilities in TNB are of the order 10^{-2} cm²/V · s suggesting that transport of charge carriers can be described by the small polaron hopping model.[14] Typical plots of μ^{-1} vs. $\exp(E_t/k_B T)$ obtained in the high temperature region for Se–TNB are shown in Figure 6. The linearity of the plots supports the supposition that the lattice mobility μ^0 does not exhibit a strong temperature dependence within the temperature range under consideration.

As shown in Table I, the anisotropy of drift mobility is rather small and similar for both holes and electrons, *viz.* $\mu^0_{aa} : \mu^0_{bb} : \mu^0_{cc} = 1 : 0.7(\pm 0.1) : 0.9$. The experimental spread of the values of μ^0 was not greater than 10%. Almost isotropic transport of carriers in TNB is

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TABLE I

Tensors of electron and hole drift mobility for TNB single crystal

Lattice mobility		Electron		Hole	
$\mu^\circ \left[10^{-2}\frac{\text{cm}^2}{\text{V}\cdot\text{s}}\right]$	Se–TNB	CdTe–TNB	$\frac{N_t}{N_V}\left(\frac{\nu_V}{\nu_t}\right)^a$	CdTe–TNB	$\frac{N_t}{N_V}\left(\frac{\nu_V}{\nu_t}\right)^a$
$\mu^\circ aa$	3.1	6.2	average	5.9	average
$\mu^\circ bb$	1.8	4.4	5×10^{-7}	4.4	3×10^{-7}
$\mu^\circ cc$	2.8	5.6		5.2	

[a] see text

consistent with the structure of a TNB crystal[11] in which 16 molecules per unit cell are arranged in such a way that there is no preferred direction in which the overlap of orbitals would lead to facilitated transport.

The confusing difference in the absolute values of electron mobilities as measured with Se and CdTe electrodes (3.1 and 6.2 cm^2/V s for μ_{aa}) needs, clearly, an explanation; strangely, the anisotropy of the values measured remains almost the same for both experimental arrangements.

3.2. Generation and photoinjection of charge carriers

The current transients observed have been attributed to transport of charge carriers through the plates of TNB, the transit times being at least two orders of magnitude longer than those which could be expected for carriers moving through the layers of Se and CdTe only. Thus, we expect that carriers generated in this layers of the semiconductor are injected into transport states of the molecular crystal. In the case of such structures one should always expect the presence of an "injection barrier". This could have its origin in a potential energy barrier due to the mismatch of electronic energy levels in the sensitizer and the dielectric or in interface effects. Generally, one can also expect that the electric field at the interface may be modified by the presence of accumulated space charge. Thus the injected current may be either contact or bulk limited via space charge, however, it is difficult to distinguish between them.

Typical time-resolved transients of electrons injected from amorphous selenium shown in Figure 1 may be characterized by the following features: (i) an initial spike; (ii) a distinct current increase for relatively short times; (iii) a slow decay for times greater than the

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transit time. Under similar conditions the electron transients for CdTe–TNB (Figure 2a, c) are markedly different, there being no initial spike nor increase, and the tail of the trace being also shorter. Furthermore for the CdTe electrode system there is a 7–8 μs gap between the beginning of the light pulse and time when current reaches a plateau, a feature which was not seen for any sample with an evaporated Se electrode.

The features observed in Se–TNB may be explained as follows. The initial spike (*ca* 6–8 μs in width) is probably due to generation and transport of electrons across the α-Se layer (electron mobility in amorphous selenium is about 10^{-2} cm^2/V·s [15]. The spike width was slightly field dependent and its height both temperature and field dependent.

The slow rise of the photocurrent in CdTe-TNB structure is reminiscent of the feature observed in As_2S_3–CdI_2 films [16], which was explained as 'delayed generation' of carriers. This feature is, most probably, responsible for the difference in our mobility values measured with Se and CdTe contacts, respectively. A simple calculation shows that this difference is too large to be due to differences in transit times through Se and CdTe layers, transit times in those layers being smaller than the average spread of measured t_t for the total system. Thus, a mechanism has to be postulated which temporarily slows down electrons before their injection into the insulator. A possibility that the effective electric field in the insulator is lowered in the case of Se–TNB structures, has been rejected since the t_T^{-1} vs. E plot (Figure 3) does not exhibit either significant deviations from linearity or a non-zero intercept.

Obvious mechanisms which could be responsible for the 'delayed generation' should take into account trapping of electrons within the Se layer as well as at the interface, the trap release times being of the order of t_t. Another mechanism which can explain the features observed in the transients and the underestimated measured mobility values is a limited rate of injection due to an existence of a potential barrier. The carriers generated in Se may be thought to form a reservoir at the interface, the injection rate being governed by the properties of the barrier.[17]

In order to provide some insight into the injection process we have determined electric field and temperature dependences of the photoinjected current. Figure 7 shows the current measured in the plateau region of the transient vs. electric field for Se–TNB structures. The current is a quadratic function of the applied field which implies that the total charge collected at the rear electrode is proportional to the

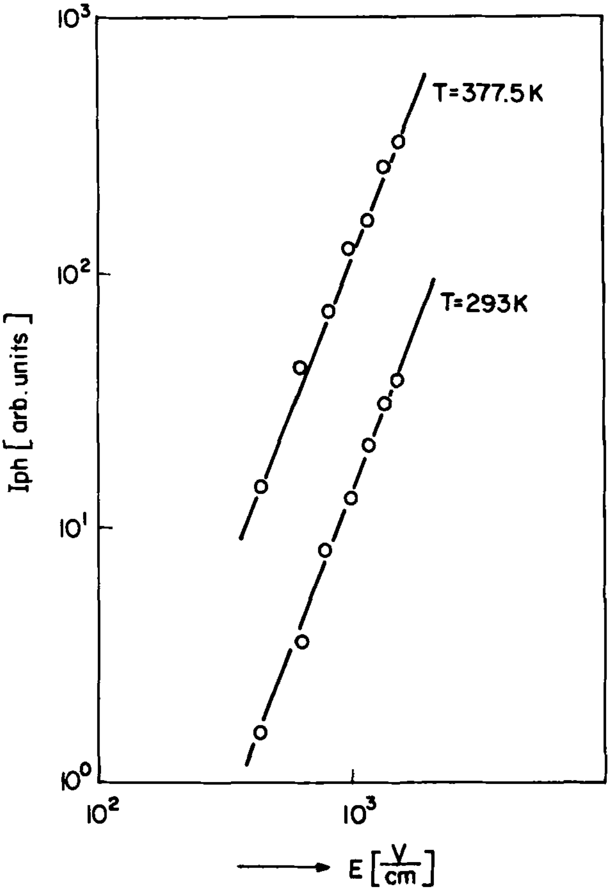

FIGURE 7 Electric field dependence of photocurrent for Se–TNB system at 293 K and 377.5 K.

electric field. Indeed, if we disregard trapping the charge Q is equal to

$$Q = I_{ph} \cdot t_t = I_{ph} \cdot \frac{LE}{\mu} \cdot E \tag{2}$$

This dependence may be thought to arise from the superposition of the field dependence of photogeneration in Se and the field dependence of the photoinjection process. Generation of charge carriers in Se is described by the Onsager theory of geminate recombination.[18,20] The theory predicts only a slight field dependence of Q for fields lower than *ca* 10^4 V cm^{-1}. However as pointed out by Weisz *et al.*[21] the

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photocurrent produced by generation of carriers in a thin surface layer of a photoconductor is modified by the presence of surface recombination.

The model presented in ref. 21, and derived assuming that the recombination-limited lifetime of a carrier at the surface τ_s is much shorter than that in the bulk, leads to the formula

$$Q(V) \cong [2K/(1+K)]CV \qquad (3)$$

where

$$K = \rho(0)\mu\tau_s \exp(-2)/2\epsilon \qquad (4)$$

$\rho(0)$ being the surface density of generated charges and ϵ being the dielectric constant.

In the experiments described here the photon energies (between 2 and 4 eV) are sufficiently high to assume that the photogeneration in

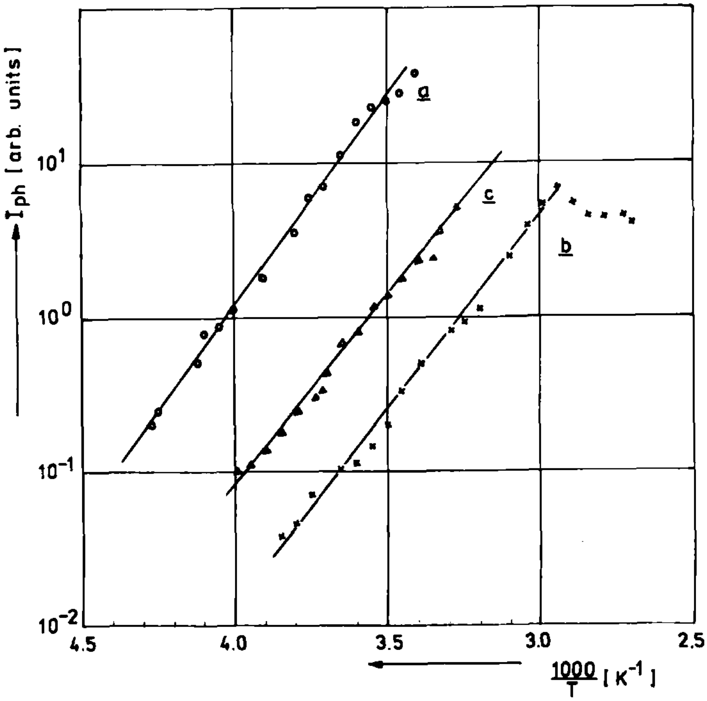

FIGURE 8 Photocurrent I_{ph} versus $(1000/T)$ for Se–TNB system. (Apparent departure from straight line behavior for *b* direction we attribute to the phase transition in selenium from the red to the grey form).

Se takes place in a thin surface layer (absorption coefficient $\alpha \geq 10^4$ cm^{-1},[22]) thus a linear dependence of Q on the electric field may be thought to arise from surface recombination. Nevertheless, one cannot exclude the possibility that the total electric field dependence observed does contain effects due to the dependence of the *injection* of carriers on the field. This effect should not, however be very strong, since the Q vs. E dependence to be observed is not very large.

In the course of this study we have also determined temperature dependence of the photocurrent at a given electric field for the Se–TNB system. The results of these measurements are shown in Figure 8. Such measurements together with measurements of temperature dependence of mobility can serve as a test to decide whether the observed currents are solely determined by the bulk transport in the

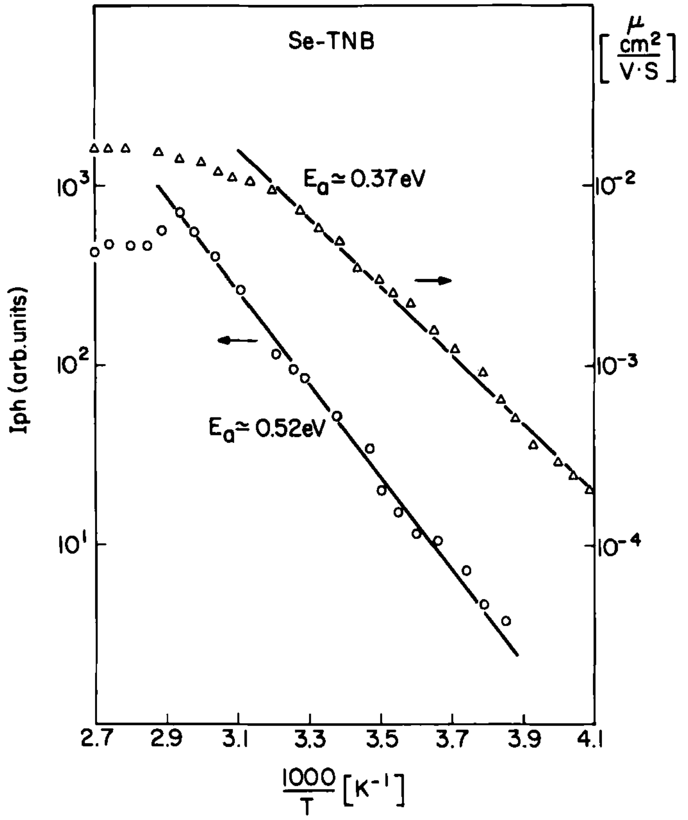

FIGURE 9 Mobility of electrons in the *b* direction of TNB and the photocurrent density vs reciprocal temperature.

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insulator and not by either an injection rate limited by a potential barrier, or by release from traps at the interface.[23]

Activation energies of the photocurrent are about 0.52 eV for *a*, *b* and *c* crystallographic directions whereas activation energies of drift mobilities are close to 0.37 eV. In Figure 9 we have compared this for the *b* direction. Assuming Eq. (4) to hold then the activation energies should fulfill the relation

$$E_Q^a = E_{I_{ph}}^a - E_\mu^a \qquad (5)$$

where E_Q^a, $E_{I_{ph}}^a$ and E_μ^a stand for activation energies of the collected charge Q, photocurrent I_{ph} and mobility μ respectively.

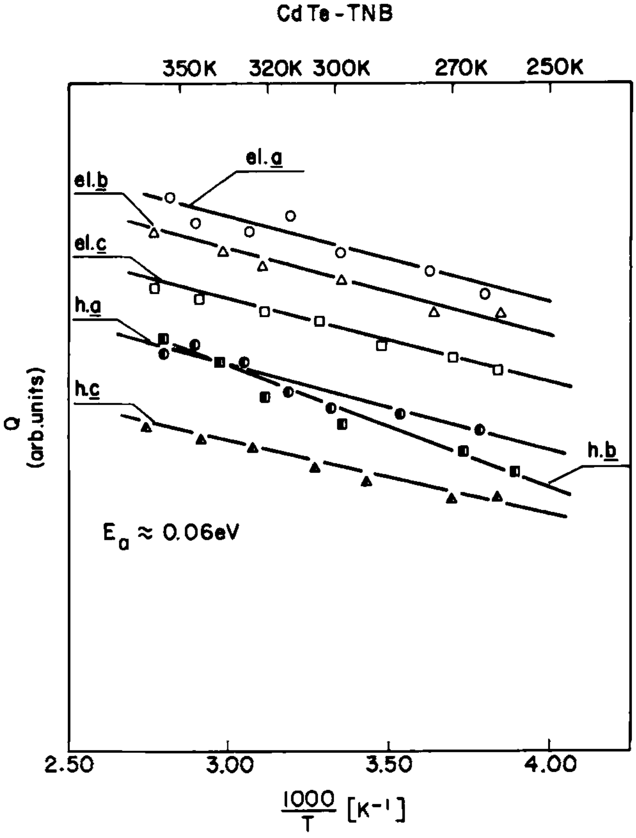

FIGURE 10 Total charge injected into TNB from CdTe under short light pulse illumination versus reciprocal temperature.

The photoinjection activation energy is thus $E_Q^a \cong 0.15$ eV which suggests some form of a potential barrier exists at the Se–TNB interface. Alternatively, one can assume that shallow interface traps determine the rate of injection and its temperature dependence.

The CdTe–TNB system exhibits a different behaviour. The collected charges were almost two orders of magnitude smaller than in Se–TNB. Holes and electrons were injected with approximately the same efficiency which is to some extent astonishing. In order to evaluate the activation energy for injection of electrons and hole we have plotted the total charge collected Q versus reciprocal temperature —see Figure 10. The charge collected was obtained by graphical integration of the photocurrent signal which was easier in the case of CdTe–TNB due to the absence of long current tails. Although some scatter exists, it is clear that for both electrons and holes the activation energy is the same and is very small ~ 0.06 eV.

Since the energy gap in CdTe is only 1.53 eV,[24] the presence of the injection of both types of carriers can only be rationalized if one assumes a "hot carrier" injection. On the other hand, the absence of the injection of holes in Se–TNB is likely to be due simply to a mismatch of appropriate valence bands.

4. CONCLUSIONS

To measure carrier transport properties in organic insulators which are not photoconducting requires either excitation from an electron gun or photoinjection from a suitable electrode. In comparison to inorganic studies, semiconductor electrodes have seldom been used coupled with organic materials. We have applied selenium and cadmium telluride electrodes to single crystal trinitrobenzene and succeeded in measuring the mobility tensor for holes and electrons. The drift mobilities measured were all prone to shallow trapping, in general being ~ 2×10^{-2} cm^2/V sec for both holes and electrons. Little anisotropy was observed as expected from the crystal structure. The highest mobility measured was 6×10^{-2} cm^2/V s for electrons in the *aa* crystal direction.

The shapes of the current transient for the Se and CdTe electrodes were different, particularly for short times. This difference is due to properties of the semiconductor-TNB interface. Measurements of the current transients and injection efficiencies with different crystal thicknesses and excitation geometries showed that these differences relate to the interface between the semiconductor-bulk material. The

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photoinjection activation energy E_a^a for electron injection from selenium shows either a potential barrier exists at the interface, or interface states out as carrier traps, either of which will determine the rate of carrier injection and its temperature dependence. CdTe electrodes show a different behaviour. Injection efficiency is two orders of magnitude lower, but the injection activation energy for both holes and electrons is very small, ~ 0.06 eV.

It was also found that Se electrodes only gave electron injection, no hole injection could be observed. This can be understood in terms of the mismatch in energy between the appropriate valence bands. However CdTe electrodes gave injection of both holes and electrons. Since the energy gap in CdTe is 1.5 eV, smaller than that of TNB, this can only be rationalized assuming "hot carrier" injection, a phenomenon which has been observed in inorganic systems. It does appear however that the application of semiconducting electrodes to organic non-photoconductors can give information relating to the nature of the injection process and the existence of interface barriers and/or states.

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