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Transient Photoinjection of Holes from Amorphous Se into Poly(*N*-Vinyl Carbazole)

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The transient photoinjection of holes from amorphous selenium into layers of poly(*N*-vinyl carbazole) (PVK) has been studied. A variety of techniques are described which lead to the conclusion that no barrier to injection need be invoked. The injection threshold observed in the Se:PVK structure using the xerographic condenser technique can be accounted for in terms of the achievement of space-charge-perturbed currents in the polymer dielectric. The steepness of the observed threshold arises from the field dependence of the drift mobility of holes in PVK.

I. INTRODUCTION

Regensburger[1] has recently described the sensitization of the dielectric poly(*N*-vinyl carbazole) (PVK) by overcoating with a thin layer of amorphous selenium as a sensitizer. The sensitization was studied using a xerographic condenser discharge technique and the results were interpreted as the injection of holes photogenerated in the amorphous Se into PVK. An abrupt drop in the gain of the system was observed on going to lower fields.

Several possibilities exist for the origin of what Regensburger termed a threshold field for carrier injection. These can be conveniently classified into two categories. One would be the presence of some type of "injection barrier" as suggested by Regensburger.[1] 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 it could result from macroscopic mechanical barrier at the interface between the sensitizer and the dielectric. The second category would arise, not because of any injection or interface limitation, but rather from bulk properties. Two outstanding possibilities in this class would be a range limitation in either the sensitizer (or the dielectric) or an absence (or at least reduction) of the field within the sensitizer due to permanent charge accumulation at the sensitizer-dielectric interface. As far as the bulk limitations are concerned, the range limitation can be eliminated in the amorphous Se: PVK system because of the independent knowledge of the ranges of Se[2] and PVK[3] from transient measurements.

The aim of this paper is to indicate from an investigation of transient photoconductivity in Se: PVK structures that a decrease in injection efficiency observed in the xerographic mode can arise from the trap-free space-charge-perturbed currents (TFSCPC) in PVK.

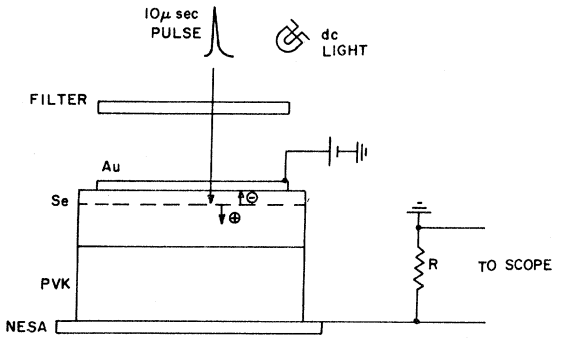

FIG. 1. Schematic representation of the experimental arrangement.

II. EXPERIMENTAL TECHNIQUES AND RESULTS

A. Sample Preparation and Experimental Arrangement

The model composite structure was made in three steps. A layer of PVK ~ 10 μ thick was laid down by dip coating a Nesa slide in a solution of PVK in toluene and cyclohexanone, followed by heating at 100 °C for several hours to remove residual solvents. A thin film of amorphous selenium ~ 3 μ was then vacuum deposited on the PVK. The PVK film was maintained at 55 °C during the selenium evaporation. Finally, a semitransparent top electrode of gold was evaporated to complete the structure.

The experimental arrangement is shown in Fig. 1. A 10-μsec light pulse from a xenon flash tube was appropriately filtered with a narrow-pass band filter, viz., 4330 Å, to produce electron-hole pairs at a distance from the top surface of the amorphous selenium small compared to its total thickness. By applying a positive bias to the top electrode, holes could be driven to the Se : PVK interface, while the electrons were collected at the top electrode. Holes entered the PVK at the interface and their transport through the PVK was measured by time resolving their transit. For some experiments to be described later, a steady dc illumination of the same wavelength was used simultaneously with the light flash. In general, because of the extremely long transits at low fields and their very marked dispersion at all fields, it was found much more useful to measure time-resolved transits in the current mode. The *RC* time constant was, therefore, always maintained much less than the transit time through the PVK.

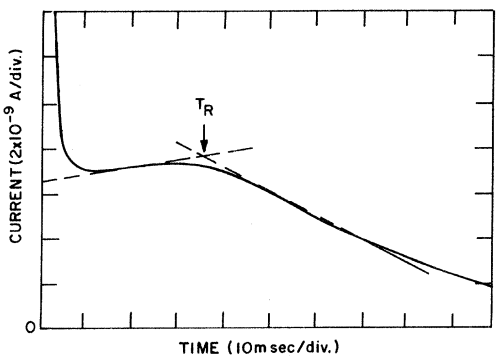

FIG. 2. Typical current pulse showing holes injected from Se transiting PVK.

B. Transient Trap-Free Space-Charge-Perturbed Currents

Figure 2 shows a typical time-resolved transit in the current mode of holes injected from amorphous selenium. The very fast large initial transient is due to the transit of holes across the amorphous Se itself. Since the light pulse duration of 10 μ sec exceeds the hole transit time through the selenium at the fields of interest, this transit is unresolved. The general form of the transit through the PVK is identical to that which can be obtained either by photoemission of hot holes from a metal or by internal generation in the PVK itself.[3] A characteristic feature of the current pulses is the occurrence of a plateau followed by a very long

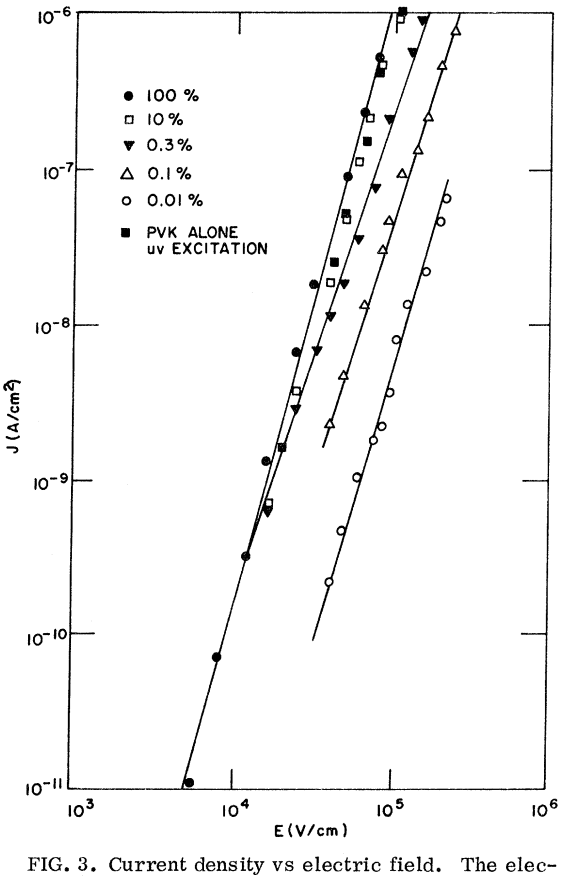

FIG. 3. Current density vs electric field. The electric field is determined from the total applied voltage divided by the PVK thickness.

tail which indicates that a large fraction of the total transported charge exhibits a dispersion in effective transit times. The relatively well-defined shoulder indicates the arrival of the fastest carriers. Figure 3 shows a plot of the average transient current before the arrival of the fastest carrier as a function of applied voltage at different light levels. It can be seen that at the highest light level the transient current is proportional to $\sim V^4$ and is essentially independent of light intensity. As the light intensity is reduced by the introduction of neutral density filters, there is a small but definite decrease in slope to $\sim V^{3.5}$, which occurs at lower voltages as the intensity decreases. No significant change in the shape of the current pulse was observed in going from the small signal to the space-charge-perturbed case. Similar observations have been made in As_2Se_3 films by Scharfe.[4] The absence of a reduced transit time and cusp observed in unique mobility materials for large signals was explained in terms of the dispersion of transit times in As_2Se_3. Because of this dispersion most of the charge moves only a small distance in the transit time of the fastest carriers. The internal field remains therefore essentially constant during the transit, since only a small number of charges are displaced and hence no cusp or reduced transit is observed. In view of the similar dispersive effects in As_2Se_3 and PVK, Scharfe's[4] explanation would seem applicable to PVK also. Figure 4 shows a plot of the reciprocal of the fastest transit time versus applied voltage on a log-log plot. This gives $1/T_R \tilde{\propto} E^{2.5}$ which is a somewhat lower field dependence than reported by Regensburger.[1] The fastest transit time marks the onset of the long tail in the current pulse (see Fig. 2). At the lowest field it is seen that the fastest transit is of the order of several seconds. However, the current does not return to its dark value; i.e., the transit of carriers continues, for several minutes. The mobility in this sample at 10^5 V/cm is $\sim 10^{-6}$ cm^2/V sec.

A comparison of the absolute magnitude of the observed currents with those which should be calculated theoretically is made difficult by the large dispersion in transit times exhibited by PVK. The problem is that it is not clear what precise mobility value or field dependence should be used in a modified Child's law to calculate the maximum current which PVK can sustain. Certainly one would conclude from their order of magnitude that the observed currents are trap-free space-charge-perturbed (TFSCP). In addition, it has been possible to achieve the same absolute light-independent current levels by charge generation in the PVK itself, using intense ultraviolet light pulses. The conclusion is that the uppermost curve in Fig. 3 is very close to the maximum bulk current that this thickness of PVK can sustain, and using Child's law $J = 10^{-13}\kappa\mu E^2/L$, a mobility of $\sim 4\times10^{-7}$ cm^2/V sec is estimated at 10^5V/cm from this curve. This is quite close to the value calculated from Fig. 4.

It is relatively easy to see how this limitation is reached in this system. In order for the current flow in PVK to be TFSCP, essentially one CV of charge must be moved across the Se and through the PVK film. In view of the relatively high mobility of holes in amorphous Se, it is difficult with normal light intensities to achieve generation rates which will allow a CV to be transported across Se within its transit time. In other words, even though the space-charge limit may be reached in PVK, the charge producing this is in general transported through the Se without space-charge perturbation in the transit. Nevertheless, once the CV of charge has been brought to the interface, the Se feels the effect of its space charge. The field at the illuminated Se surface and within its bulk has been reduced to zero, until the original surface charge is restored after the charging time RC. Since the transit time at low fields in the PVK may be several seconds, both charge generation (which is field dependent) and motion in the Se would, for times longer than

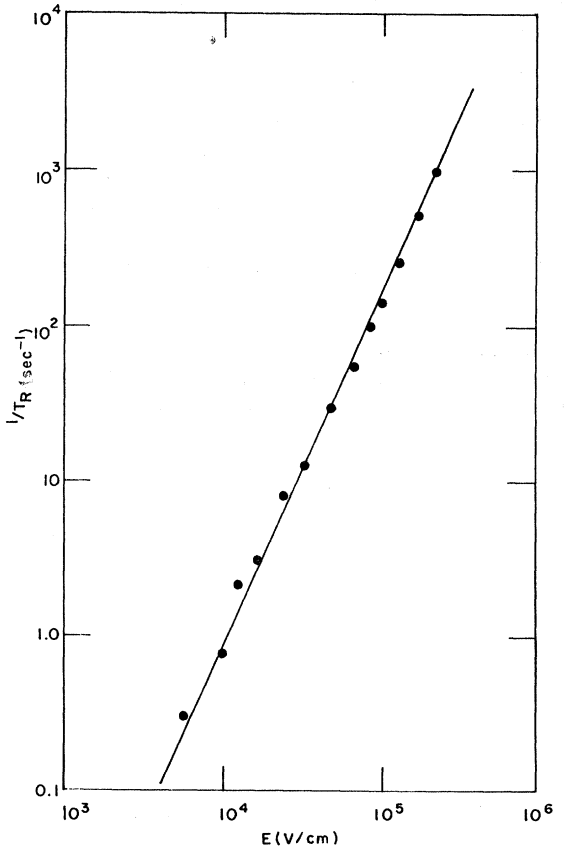

FIG. 4. Reciprocal of fastest transit time vs electric field.

TABLE I. Results of graphical integration of Se and PVK current pulse.

E(V/cm)	Se (C)	PVK (C)	PVK/Se
9.4×10^{3}	2×10^{-10}	5×10^{-10}	2.5
1.2×10^{4}	2.5×10^{-10}	5.5×10^{-10}	2.2
2.4×10^{4}	4.5×10^{-10}	7×10^{-10}	1.5
3.2×10^{4}	7.5×10^{-10}	1×10^{-9}	1.3
4.8×10^{4}	10^{-9}	2×10^{-9}	2.0
6.5×10^{4}	1.5×10^{-9}	3×10^{-9}	2.0
8.1×10^{4}	2.0×10^{-9}	4×10^{-9}	2.0
1×10^{5}	2.2×10^{-9}	5×10^{-9}	2.3
1.2×10^{5}	3.0×10^{-9}	8×10^{-9}	2.6
1.6×10^{5}	3.5×10^{-9}	1.8×10^{-8}	5.1
2.0×10^{5}	4.4×10^{-9}	2.0×10^{-8}	4.6
2.4×10^{5}	4.8×10^{-9}	2.4×10^{-8}	5.0

the transit time in Se and shorter than the transit time in PVK, be small. Precisely this mechanism can be observed by use of an accelerated-transit-time technique. The apparatus can be adjusted to display the current pulse due to charge motion in the selenium. At low fields there is sufficient time to manually give the sample a second light pulse before the CV of charge due to the preceding pulse has hardly moved away from the interface. Under such circumstances, essentially no selenium response is seen; i.e., as far as the Se is concerned it is polarized. After waiting for several transit times, the Se signal is restored to its unperturbed value. The waiting period can be substantially reduced by increasing the driving field on the PVK during the transit of carriers which initially started their drift in a lower field.

The observation of transits, albeit with considerable dispersion, down to very low fields certainly rules out the cause of the injection threshold field as solely arising from any loss of carriers during the transport across the PVK. This important conclusion was first reached by Regensburger on the basis of his transit measurements. [1] However, a further possibility was that substantial carrier loss occurred in the interface region, perhaps for a variety of reasons. Although it is possible to argue that any carrier loss, which was the controlling factor in determining the current level in the PVK, would of necessity mean that the current was light-intensity dependent, attempts were made to quantitatively assess the loss of carriers. The most direct way to do this would be to measure the height of the integrated charge displacement through the PVK. However, as pointed out earlier, the transit time and therefore the required integration times at low fields becomes restrictively long. A more reliable way, though still with limited accuracy, was employed. The area under the current curve for the Se part of the current pulse and for the PVK were independently measured graphically. The results are given in Table I. Although considerable scatter exists, clearly no substantial loss of carriers exists anywhere in the system, of the order required to account for the sharp drop in apparent injection efficiency. Indeed, it is clear from the quite small currents ($\sim10^{-11}$ A/cm^2) at which PVK becomes TFSCP at low field that, if any significant loss occurred, no transit current pulse would even be observed. This follows from the fact that at low fields, where transit times are of the order of seconds, RC considerations limit the maximum series resistance to $\sim10^{8}\ \Omega$. The noise level is ~1 mV, so the minimum observable current level is $\sim10^{-11}$ A. The TFSCPC is therefore itself only just observable at low fields.

A further crucial and confirmatory test was employed to decide whether the observed currents are solely determined by bulk transport in the PVK, and not by an injection rate over any potential barrier, or release from traps, at the interface. This was to measure the temperature dependence of the light-independent transit currents. The results of these measurements are shown in Fig. 5. Here, the average transient current during the transit and the reciprocal of the fastest transit time are plotted as a function of reciprocal temperature. The temperature range extended from -30 to $+60\,^{\circ}$C. The activation energy of ~0.5 eV obtained from these curves represents the thermal activation energy for hole transport in PVK. Measurements at different fields indicate that this activation energy is independent of field. The very close agreement in slope between

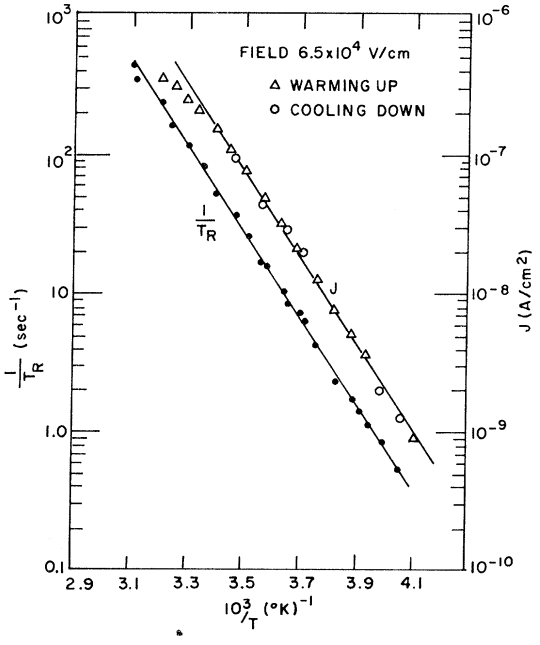

FIG. 5. Reciprocal of fastest transit time and current density vs reciprocal temperature.

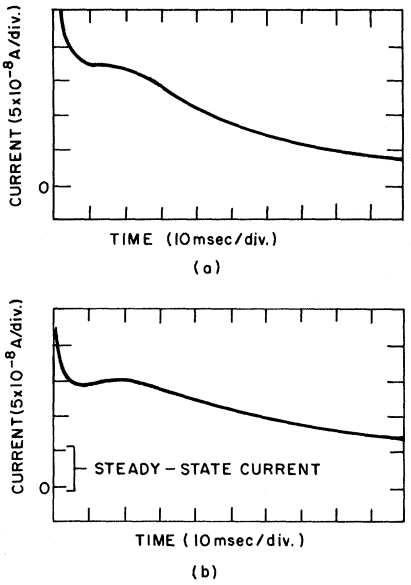

FIG. 6. (a) Trace of TFSCPC transit pulse in PVK. (b) Trace of TFSCPC produced by simultaneous dc and pulse excitation. The dc current was adjusted to be one division.

these two curves suggests that the rate of charge injection from the selenium into the PVK is bulk limited by the PVK. This rules out the possibility that any potential barrier or release from interface traps determines the injection rate. Experimentally it was not feasible to make these temperature measurements at fields below 6.5×10^4 V/cm, so the conclusions from these experiments can only be drawn for fields in excess of this value. The data were taken during cooling down and warming up. The lack of hysteresis in the data would not be expected if trapping at the interface occurred to any significant degree, since any trapping at room temperature would be vastly amplified at lower temperatures.

C. Transient and Steady-State TFSCPC

This is a remarkably simple way of showing that the light-independent currents achieved in PVK were TFSCPC. The basis for this experiment is that, under one most important condition, if the currents were TFSCPC then simultaneous steady-state and transient currents must always add up to the TFSCPC. This is shown in Fig. 6, where in (a) the transient current represents the full TFSCPC, whereas in (b) the steady-state current produced by steady light almost reduces the transient current by an amount equal to the steady-state current. The condition under which these currents are additive to the TFSCPC value is that there has to be no permanent trapping in the steady state; i.e., the steady-state current has also to be trap free. This is in fact verified by the direct observation of the transition from transient to steady-state currents. The way in which this was achieved is indicated in Fig. 7. Instead of using a light flash, step-function illumination was used. Because of the long transits in PVK at low fields, a camera shutter is sufficiently fast to establish the steady-state light level in a time much shorter than the transit time through the PVK. It is therefore possible to see directly the transit through the PVK and then the transition to steady-state injection in PVK. It can be seen that there is only about a 50% reduction from the transient current level to the steady-state current level indicating a remarkable absence of permanent trapping. The condition to observe the addition of transient and steady-state currents is therefore satisfied. The reason for the reduction in the current level between the transient and steady-state time scale is a change in the average effective drift mobility. Direct evidence for this conclusion is described in Sec. II D.

D. Method of Interrupted Transits

This technique was first used by Gibbons and Spear [5] to measure the deep-trapping lifetime and therefore the range of electrons in orthorhombic sulfur crystals. The very long transit times, and hence deep-trapping lifetime in PVK, therefore make it an admirable candidate for this type of experiment. Figure 8 indicates the sequence of events in this experiment. Initially a field is applied to commence the drift of carriers through the sample. After some fraction of the transit is complete the field is removed or interrupted for a

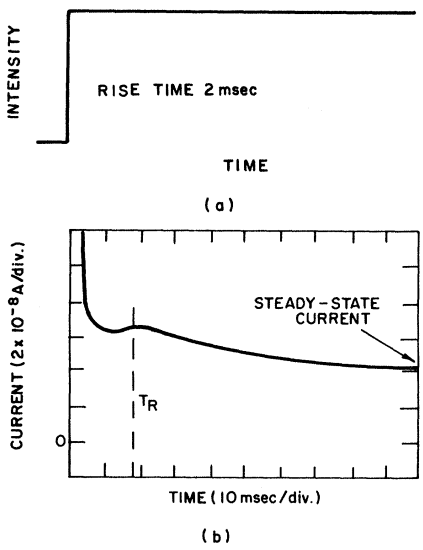

FIG. 7. (a) Application and duration of the step-function illumination. (b) Current response to such excitation. The steady-state current was observed for several hours without decay.

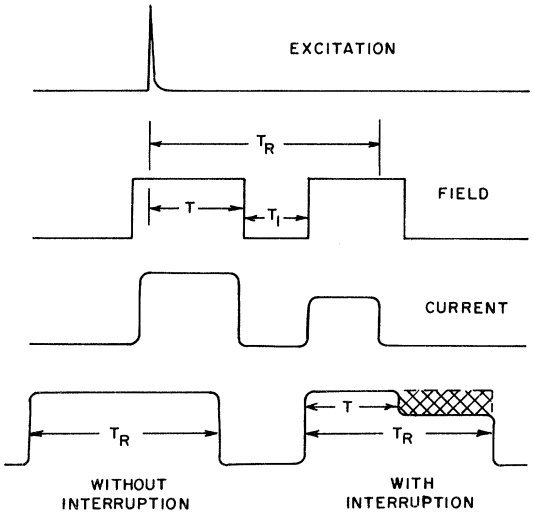

FIG. 8. Schematic representation of the method of interrupted transits to determine deep-trapping lifetimes. Shaded area in last pulse indicates number of carriers permanently lost during the interruption (after Gibbons and Spear, Ref. 5).

vaying length of time; during this interval the sample is shorted. This effectively makes the transit time infinitely long compared with the deep-trapping lifetime and trapping can occur. When the field is restored, the carriers remaining free resume their drift at the same velocity. As a result of the loss of carriers, the transient current resumes at a lower value. By measuring this reduction of current as a function of the interruption time, the deep-trapping lifetime can be determined if, as was the case for electrons in orthorhombic sulfur, [5] the current decayed exponentially with interruption time. Figure 9 shows the result of this type of experiment on PVK. The transit times in PVK are so long that the interruptions can be performed manually. It is seen that after reestablishment of the drift field the current level is lower, suggesting

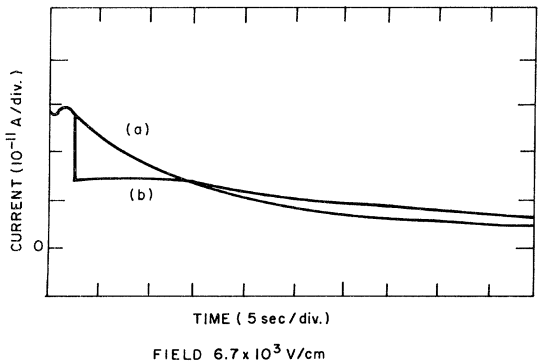

FIG 9. Results of interrupted transit experiment on PVK. (a) Current pulse without interruption and (b) current pulse after transit was interrupted for 4.5 sec. Trace (b) has been drawn leaving out the interruption period.

a loss of carriers. However, unlike the case of sulfur, it is seen that the interruption has in fact changed the duration of the current pulse. The transit now takes a longer time, after allowing for the interruption, than for the same drift field without interruption. It is concluded that during the interruption some adjustment occurs which results in a lower average drift mobility. In any event, on a time scale of seconds no permanent deep trapping is evident, but a transition to a lower effective drift velocity is directly observed. This may be due to interaction with shallow traps on this time scale.

III. DISCUSSION

A number of experiments have been described which indicate that TFSCPC can be achieved and that, because of the field-dependent mobility of holes in this material, these currents increase proportionally to $\sim V^4$. More importantly, however, it is seen that, because of the extremely low drift mobility of holes in PVK, the absolute value of these currents at relatively high fields, i.e., $\lesssim 4 \times 10^4$ V/cm are only $\lesssim 10^{-8}$ A/cm^2. It is easy to show that the generation-limited currents in amorphous Se, with a light flux of 2×10^{12} photons/sec

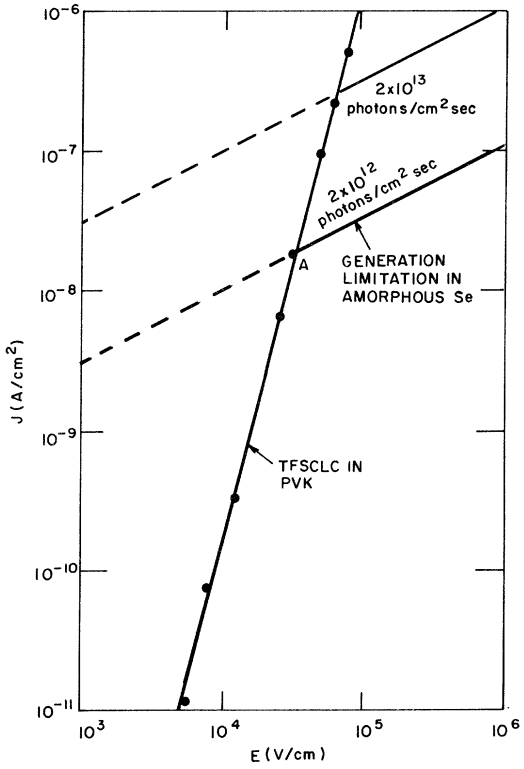

FIG. 10. Composite TFSCPC and amorphous Se generation-limited current curves. The TFSCPC curve for PVK was experimentally determined for a 10-μ sample. The generation-limited current curves for amorphous Se were calculated assuming a generation efficiency $\alpha E^{0.5}$, with a value of unity at 10^6 V/cm.

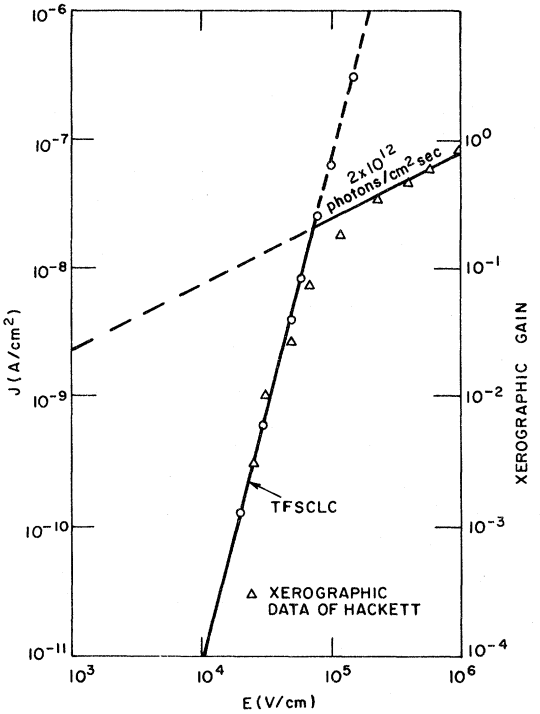

FIG. 11. Comparison of measured xerographic gain-vs-field curve with that constructed using a composite of TFSCPC curve, measured on the same sample, and the amorphous Se generation-limited current curve. The latter was computed for the light level used in the xerographic measurements with the experimentally observed gain of 0.8 at 10^6 V/cm (Ref. 8).

cm^2, will be larger than these maximum currents that a 10-μ film of PVK can sustain at these fields. This is shown in Fig. 10. The amorphous Se generation-limited currents shown were computed assuming a quantum efficiency of 30% for a photon energy of 3.0 eV, at a field of 10 V/μ. They are assumed to be steady-state currents, i.e., measured on a time scale long compared with the transit time[6]; trapping effects are neglected. It is seen that for fields to the right of point A the currents through the combined Se-PVK structure will be emission or generation limited and therefore thickness independent. To the left of A the currents through the combined structure will be bulk limited by the PVK and therefore thickness dependent. It is apparent that the combination of the two curves indicated by the full lines has the characteristic shape of the xerographic gain curve observed in this sample which is shown in Fig. 11. If this combination is to explain the xerographic gain curve, however, one further condition must be met. The TFSCPC curve represents current measured in the constant voltage mode. Since the TFSCPC is a bulk limitation, the current must reflect the voltage dependence of the amount of charge flowing and the voltage dependence of the transit time of that charge. For this limitation, the voltage dependence of the current is the same whether the currents are measured within a transit or in the steady state. This is not true for generation-limited currents. On the other hand, measurements made in the open circuit mode, i.e., xerographically, result in equivalent xerographic currents proportional to the initial dv/dt. If measured on a time scale long compared with a transit, they do not reflect the field dependence of the transit time. Therefore, in order for the equivalent xerographic current to have the same voltage dependence as the TFSCPC, the initial dv/dt must be measured in a time $\lesssim$ a transit time. In the case of a trap-free space-charge-limited discharge in the xerographic or V mode the transit time for the CV of charge is strictly infinite[7] so that this condition will be satisfied.

In Fig. 11 the experimentally determined TFSCPC curve has been plotted for a Se:PVK sample. Superimposed on the TFSCPC is xerographic gain data determined from initial dv/dt measurements.[8] In these measurements a photon flux of 2×10^{12} photons/cm^2 sec was used and a gain of 0.8 achieved at 10^6 V/cm. These data determine the Se generation current curve and the high-field experimental points must lie on this. The position of the other points is then determined. Figure 11, therefore, demonstrates unequivocally that it is possible to account almost exactly for the xerographic gain-vs-field curve in both shape and absolute magnitude. No adjustment of any parameter is required and no xerographic data are involved. Apparently this was not the phenomenon observed by Regensburger,[1] since his data were obtained under space-charge-free conditions.[9]

So far we have dealt exclusively with the fact that it is relatively easy to become bulk limited in the Se:PVK system, because of TFSCPC in PVK, and that this can produce a falloff in the apparent injection efficiency. An important point to stress is, however, that *any* limitation which arises from a field dependence of the amount of injected charge which varies as $E^{0.5}$ to $E^{1.0}$ may produce a very similar falloff in injection efficiency. This stems from the fact that on xerographic time scales one may measure currents, or equivalently initial dv/dt, on a time scale at low fields which is short compared with the transit time. A good example of this is shown in Fig. 3. Here, in the light-intensity-dependent region the currents are generation limited, but because these currents are observed on a transit time scale in the PVK, the currents involve the field-dependent charge generation in Se proportional to $E^{0.5}$ and the transit time in PVK so that the slope of J vs E changes by $E^{0.5}$. In other words any measurement on a time scale short compared with the transit time in PVK will result in the observation of the strong field dependence of the transit time

in this material. Therefore, the strong field dependence of the apparent injection efficiency is not a unique characteristic of the space-charge-limited current, but is characteristic of measurements made while a transit is still proceeding. A typical xerographic apparatus response time is ~0.1 sec and, referring to Fig. 4, it is seen that the transit time of this PVK sample already begins to exceed this value at fields $\sim 3\times10^4$ V/cm. The transition referred to above is, therefore, expected to occur in the range of field values necessary to account for the observed xerographic gain-vs-field curves. This bulk limitation does not arise from any permanent loss of carriers, but from the fact that, because of their low velocity, the carriers have not sufficient time to transit the PVK before the initial dv/dt is measured. In a xerographic sense, the usual meaning of a bulk limitation is a range limitation, i.e., $\mu\tau E <$ sample thickness, where τ is the bulk deep-trapping lifetime. It is now clea that another bulk limitation can arise if the time required to transport the charge is inconveniently long. By an arbitrary cutoff in the time of observation, this will be an operational range limitation, and the xerographic gain at a given field will be inversely proportional to the PVK thickness. Colloquially stated, it matters how fast the carriers move as well as how far.

ACKNOWLEDGMENTS

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Transient Accumulation of Interfacial Charge in Photoconductor-Dielectric Systems

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This paper presents experimental results which reveal the occurrence of free-carrier accumulation (i.e., no trapping and no potential-energy barrier to injection) at a photoconductor-dielectric interface. The space-charge-limited current of the dielectric emerges as a key parameter in the process. Several predictions concerning the rate of accumulation of free charge at the interface and the dependence of that rate on applied voltage and current levels in the photoconductor are made using a qualitative model. The experiments were carried out on a model system, amorphous-Se—PVK [poly(*N*-vinyl carbazole)], where by means of light the Se is made a conductor of variable "dark" conductance. Direct observation of the development of interfacial charge has been possible by use of a double-relay technique. The variation with applied voltage and light intensity of the time for accumulation of a CV of charge conforms qualitatively with the predictions of the theoretical model.

I. INTRODUCTION

A recent paper[1] has described a limitation which can account for observed xerographic gain-vs-field curves in structures consisting of films of the polymer poly(*N*-vinyl carbazole) (PVK) overcoated with amorphous selenium. The limitation arises because even at relatively high fields the emission-limited current of the Se can be larger than the trap-free space-charge-limited currents (TFSCLC) in the PVK. In the experiments on the amorphous-Se–PVK system,[1] the assumption of capacitive field division throughout the measurement time for the emission-limited region was justified implicitly in terms of the high resistivities of the amorphous Se and PVK. By this criterion, structures incorporating a dielectric of relatively low resistivity on a high-resistivity dielectric, such as PVK or amorphous Se, would quickly develop essentially zero field in the more conducting dielectric. The experiments on the amorphous-Se–PVK system[1] indicated that the relative resistivities of the dielectrics in a composite structure are not, per se, the determining factor.

The purpose of this paper is to present experimental evidence which monitors the transition from