Photoelectronic Properties of Iodine-Incorporated Poly(N-Vinylcarbazole) Films

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SYNOPSIS

Iodine was incorporated into poly(N-vinylcarbazole) (PVK) films by exposing the films to saturated iodine vapor at 100°C for 10 h. The content of iodine in the films was 2.3% by weight. The films were stable at room temperature. The infrared spectra indicate that no chemical reaction has taken place during the incorporation process. The dark conductivity of these films is about two orders of magnitude greater than that of pure PVK films. Carriers can be photogenerated in the films by light illumination from the UV region to the entire visible region. The most significant feature of these films is that the photoconductivity has a peak at the photon energy of 1.8 eV. This is attributed to the charge transfer from the carbazole to I_2. The results of thermally stimulated currents (TSC) indicate that there are deep traps in pure PVK films, but after iodine incorporation, these deep traps disappear or are replaced by a large quantity of shallow traps.

INTRODUCTION

The conductivity of pure poly(N-vinylcarbazde) (PVK) has been found to be due mainly to the hole transport by means of electronic hopping over donorlike monomers.[1,2] Its saturated polymer chain ensures a low dark conductivity, and its large pendent planar carbazole group with an extended π-electron system can be efficiently photoexcited. Its absorption spectrum is confined to the ultraviolet (UV) region. It is well known that the absorption region can be extended from the UV to the visible region by introducing some acceptorlike material into pure PVK to form charge-transfer (CT) complexes,[3-5] such as the PVK-TNF and PVK-I_2 complexes. Although the optical transitions in either pure PVK or pure TNF occur at wavelengths shorter than 450 nm, the CT complex absorption band extends above 630 nm, giving photoresponse in the entire visible region. From the point of view of elec-

tron affinity, iodine would be a good candidate as an acceptorlike material. PVK-I_2 complex films have been fabricated by dissolving PVK and I_2 in benzene followed by evaporation of the benzene,[4,5] but the absorption spectrum of these films is extended only to purple light (500 nm). Since the absorption spectrum of iodine itself is extended to the whole visible region,[5] this CT complex does not behave like the PVK-TNF complex probably because of the noncompatability between the organic and the inorganic materials in the solution.[5] Since there always exists some free volume in any polymer, the incorporation of iodine molecules may be possible. Most polymers are not sensitive to a small amount of impurities because the polymer itself already contains large quantities of defects and impurities. This is why we do not see the effects of iodine in PVK if the content of iodine is small. However, with a large quantity of iodine incorporated into PVK, there will be a significant change in electrical and optical properties. In this paper, we shall present some new results of the iodine-incorporated PVK and show the difference between our iodine-incorporated PVK and the PVK-I_2 complex films.

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EXPERIMENTAL

Optically clear PVK films were prepared by spinning evenly a solution consisting of 15 parts of PVK supplied by Polyscience, 30 parts of tetrahydrofuran, and 55 parts of toluene in weight on a copper plate. After being dried for 10 h in a moisture-free clean chamber at room temperature and pressure of 10^{-5} Torr, the films were taken out from the plate and put into a beaker together with 50 mg of iodine powder. Then, the beaker was sealed by an aluminum foil and heated at 100°C in an oven for 10 h. After that, silver was vacuum deposited on both surfaces of the film as electrodes, one of the electrodes being semitransparent for illumination (300 Å in thickness) as shown in Figure 1. The film thickness measured by the weighing method and the capacitance method was about 10 ± 1 μm, and the electrode size was 2.2 cm in diameter. A gap-type electrode configuration was also used for photoconduction measurements. The gap width was 0.5 mm, and each electrode width was 2 cm. For optical absorption measurements, separate films of about 400 Å in thickness cast evenly on quartz substrates were used.

A 150 W xenon lamp was used as the light source. Broad band filters (bandwidth of 50 nm at 50% of the peak transmission) were employed for producing a narrow band light. The light intensities after passing through various filters were adjusted to keep the number of the illuminating photons to be constant at about 2.2×10^{16} photons/cm^2 s. For the steady and transient photoconduction current measurements, the same technique reported earlier[6] was adopted. For thermally stimulated current (TSC) measurements, a computer-controlled liquid helium system was used and the range of temperatures used was from 150 to 500 K with a heating rate of 12 K/min. The dark current was measured in various temperature steps at a constant applied voltage. At each step, the temperature was kept constant for 30 min so that the steady-state values could be reached. The content of iodine incorporated in PVK was estimated to be 2.3% in weight by the weighing method.
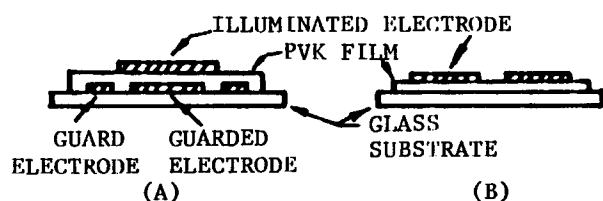

Figure 1 (A) Sandwich electrode configuration; (B) gap electrode configuration.

RESULTS AND DISCUSSION

Infrared Absorption Spectra

The infrared (IR) absorption spectra of pure PVK and iodine-incorporated PVK are shown in Figure 2. It can be seen that there is a new weak IR absorption band at 3420 cm^{-1} for iodine-incorporated PVK and other features are almost identical to those of pure PVK. No substitution reaction has taken place since no new absorption band appears within 1200–1000 cm^{-1}, which corresponds to sensitive vibrational modes in iodinated aromatic hydrocarbons. From the point of view of disassociated energy of a bond, it is hardly possible for a carbazole group to be dissociated from the main chain of a PVK molecule and to combine with a hydrogen atom so that a N—H bond can be formed to create an IR absorption band at 3420 cm^{-1}. We believe that the iodine in iodine-incorporated PVK films exists in the form of molecules rather than of atoms. Thus, this new weak IR absorption band may be attributed to the influence of the strong electronegativity of the iodine molecule that can make the large π-electron clouds of the benzene ring move toward it, although we do not know what the specific vibrational mode is. That the iodine molecules can stay in PVK steadily may be explained as due to the attractive Coulombic force between the carbazole group and the iodine molecule.

Dark Conduction

Figure 3 shows the electric-field dependence of the dark conduction current of pure PVK and iodine-incorporated PVK films. The dark conductivity of iodine-incorporated PVK is about two orders of magnitude greater than that of pure PVK. Most polymers that have saturated main chains are good insulators; pure PVK is a typical example. Generation of free charge carriers in these materials usually involves the breaking of the bonds, which requires energies of the order of a few electronvolts. Therefore, in pure PVK, these are very few free carriers at room temperature. The dark conduction in pure PVK is due mainly to holes injected from the positively biased electrode. An injected hole in PVK is, in fact, a donorlike carbazole deficient of one electron due to its donation of one electron to the electrode. This carbazole then accepts an electron from a neighboring neutral carbazole and, in turn, creates a hole in it, and so on. The hole hops from one group to another, but not necessarily within the same macromolecule. During the transit, most of
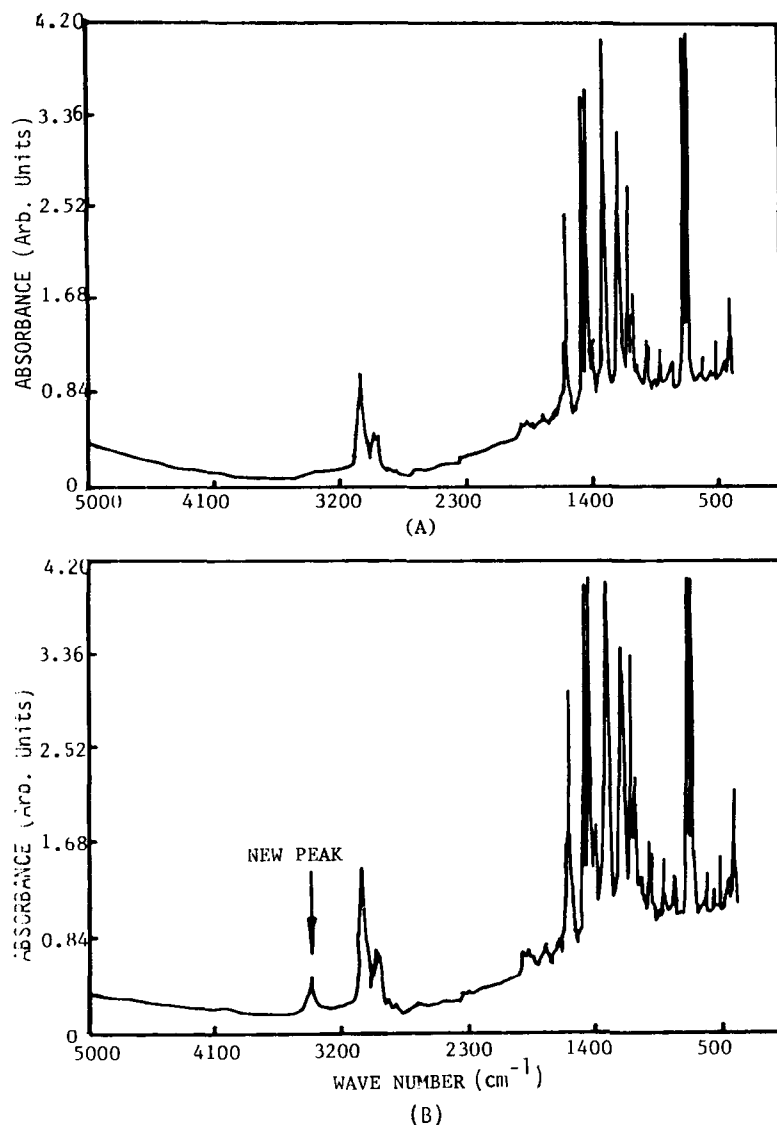
Figure 2 IR absorption spectra: (A) pure PVK; (B) iodine-incorporated PVK.

the time the holes inside are in localized states and only occasionally are thermally released from these states to move in the general direction of the field to other sites. Furthermore, PVK contains a sizable population of charged traps that are generally occupied by electrons and have a large hole capture cross section.[7] These traps will reduce not only the free hole concentration, but also the hole mobility. Therefore, the dark current–voltage (I–V) characteristics of pure PVK shown in Figure 3 may be divided into three regions, namely, (i) the low-field space charge limited region for fields of 10^3–10^4 V/cm, (ii) the medium field contact limited region for fields of 10^4–10^5 V/cm, and (iii) the high-field enhanced thermionic emission region for fields above 10^5 V/cm. Figure 3(B) shows that in the high-field

region the relation between the current I and the applied field F follows closely the Richardson–Schottky (RS) field-enhanced thermionic emission equation

$$J = \frac{4\pi em(kT)^2}{h^3}$$

$$\times \exp(-\Phi_B/kT)\exp(\beta_{RS}F^{1/2}/kT) \quad (1)$$

where J is the current density; ϕ_B is the barrier height; β_{RS} is the Richardson–Schottky coefficient, which is given by

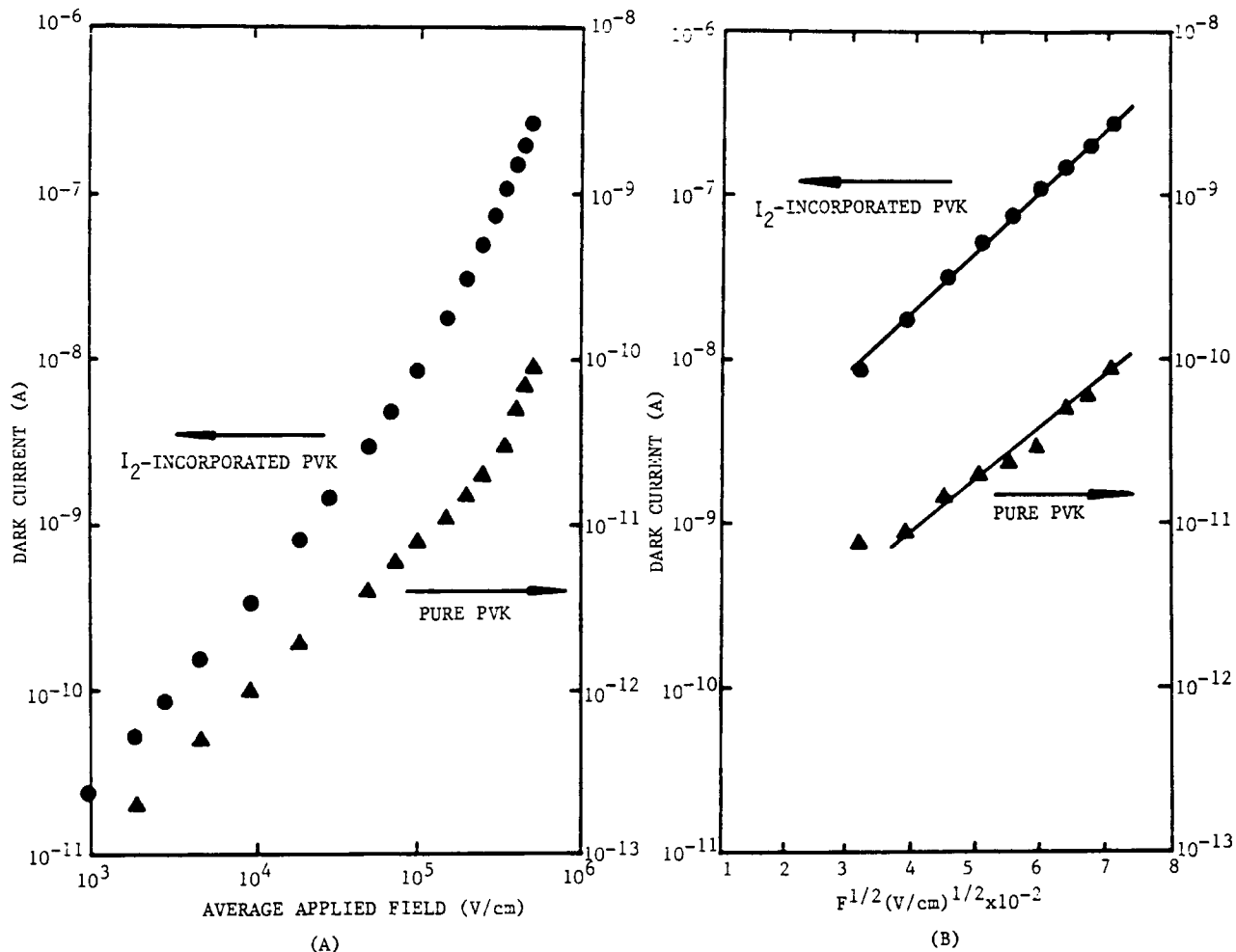$$\beta_{RS} = (1/2)(e^3/\pi\epsilon_0\epsilon_r)^{1/2} \quad (2)$$

Figure 3   (A) Dark I-V characteristics of pure PVK and iodine-incorporated PVK; (B) $I - F^{1/2}$ characteristics in the high-field region.

and other constants have the usual meanings. The slope of the straight lines shown in Figure 3(B) yields a value of 7.2×10^{-3} $(V/cm)^{-1/2}$ for β_{RS} for pure PVK, which is close to the theoretical value of 7.5×10^{-3} $(V/cm)^{-1/2}$ calculated from eq. (2) using a value of 3.75 for ϵ_r.[8,9]

For iodine-incorporated PVK, the conduction current is about two orders of magnitude greater than that for pure PVK. This increase in dark conductivity may be attributed to the charge transfer from the carbazole groups to I_2 molecules and also to the replacement of the original deep traps with shallow traps due to the iodine incorporation. This, in turn, increases the thermal generation of holes in the bulk at room temperature. This may be why the medium-field contact-limited region disappears in the I-F characteristics for iodine-incorporated PVK. In the high-field region, the relationship of ln

$I \propto F^{1/2}$ holds as shown in Figure 3(B). But in this case, the dark conduction may be controlled by the Poole–Frenkel (PF) effect rather than by the Richardson–Schottky (RS) field-enhanced thermionic emission. Since the expression for the PF effect is so similar to that for the RS thermionic emission, it is not possible to distinguish between these two mechanisms simply by the I-V characteristics.

The temperature dependence of the dark current for both pure PVK and iodine-incorporated PVK follows closely the simple Arrhenius relation

$$I = I_0 \exp(-E_a/KT) \qquad (3)$$

as shown in Figure 4, where E_a is the activation energy and I_0 is a constant. From these results we have calculated the activation energies. For pure PVK, E_a is about 1.03 eV, which may be associated with
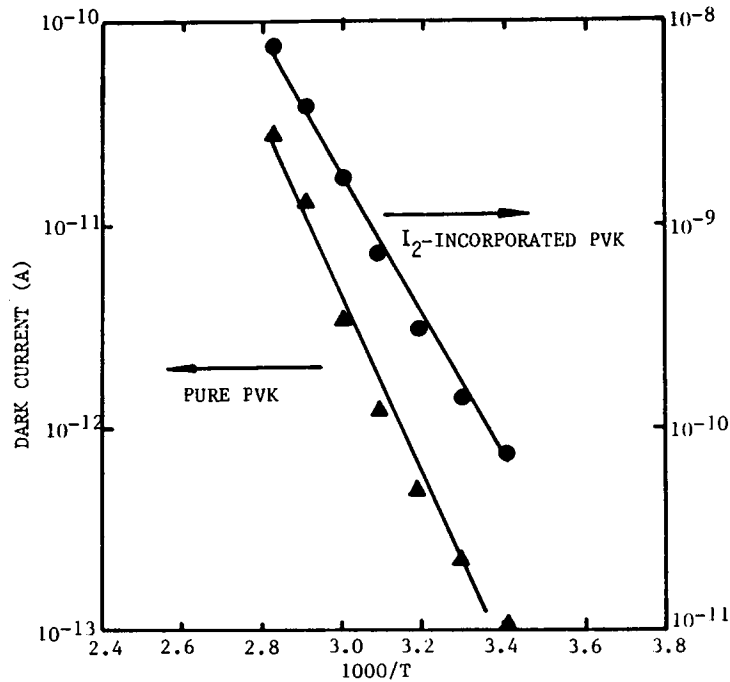
Figure 4   Dark current as a function of temperature for pure PVK and iodine-incorporated PVK at an average applied field of 1×10^5 V/cm.

the energy required for thermionic emission, while for iodine-incorporated PVK, E_a is about 0.71 eV, which may be associated with the energy required for detrapping the trapped holes.

Thermally Stimulated Current (TSC)

Figure 5 shows the TSC curves for pure PVK and iodine-incorporated PVK. It is obvious that the
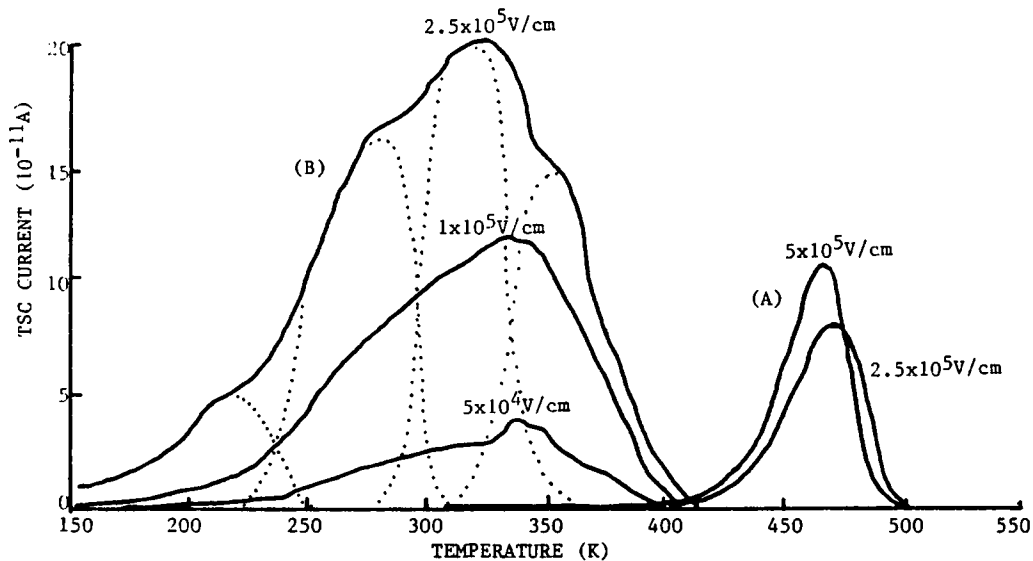

Figure 5   Thermally stimulated currents (TSC) with a heating rate of 12 K/min at various applied fields: (A) pure PVK; (B) iodine-incorporated PVK. The dotted lines represent the component TSC curves split from the TSC curve for iodine-incorporated PVK for $F = 2.5 \times 10^5$ V/cm.

temperature for the peak TSC in pure PVK (470 K) is much higher than that in iodine-incorporated PVK (340 K). This means that the traps in pure PVK are in levels deeper than those in iodine-incorporated PVK. Another significant difference is that the half-peak width of the TSC current for pure PVK (50 K) is much narrower than that for iodine-incorporated PVK (150 K). This implies that there exists mainly one trap energy level in pure PVK, whereas in iodine-incorporated PVK, the traps may be distributed in multiple energy levels. The area under the TSC curve represents the quantities of the traps. It can be seen that there are many fewer traps in pure PVK than in iodine-incorporated PVK.

A TSC curve for a single-trap energy level has one maximum whose position depends on the trap depth, the capture cross section of the trap, and the heating rate. The peak positions of TSC curves also depend on the applied electric field as shown in Figure 5. If several discrete distribution levels of traps are present, the TSC curve may be considered to consist of several peaks, each originating from a distinct trap energy level. For the calculation of single-trap depth from one TSC curve, it is necessary to know the temperature for the peak occurrence T_m and the left and the right half-peak temperature T' and T''. The single-trap depth then can be determined from the following relation.[10]

$$\frac{kT_m}{\Delta E} = \frac{T'' - T'}{T_m} (1.2\gamma - 0.54)$$

$$+ 5.5 \times 10^{-3} - \left(\frac{\gamma - 0.75}{2}\right)^2 \quad (4)$$

where $\gamma = (T'' - T_m)/(T_m - T')$. Equation (4) is a good approximation when $10 < \Delta E/kT_m < 35$ and $0.75 < \gamma < 0.9$. But when the half-peak width is broader than 50 K, the single-trap model will not hold any more. If we look at the shape of the broad peak of TSC for iodine-incorporated PVK more closely, it can be seen that this broad peak could be considered to be superposed by several single narrow peaks, as shown by the dotted lines in Figure 5 for the case with $F = 2.5 \times 10^5$ V/cm. As a rough estimation, we consider a broadest single peak at which the single-trap model is still valid. Then, we have $\Delta E = 35 \, kT_m$. Based on this assumption, we have $\Delta E = 1.42$ eV for pure PVK and $\Delta E_1 = 0.63$ eV, $\Delta E_2 = 0.84$ eV, $\Delta E_3 = 0.98$ eV, and $\Delta E_4 = 1.07$ eV for iodine-incorporated PVK.

It should be emphasized that this high-temperature TSC measurement in pure PVK has not been reported before possibly because of the restriction of the instrument. The reason for us to make this experiment is that from the TSC curves measured by Pai[11] it is obvious that the peaks which would appear above 400 K could be much higher, broader, and, therefore, much more important than those appearing at about 220 K. It can be concluded that the majority of the traps in pure PVK can only be released at temperatures above 400 K. The appearance of the low-temperature TSC peak (about 220 K) may be attributed to the increase of average mobility of the carriers.[12,13]

Photoelectronic Properties

As the IR spectra indicate that the iodine in iodine-incorporated PVK remain in the form of molecules, it is expected that some of the I_2 molecules act as acceptors to promote the charge transfer between the carbazole groups and I_2 molecules and some modify the local potential to create shallow traps and to suppress the original deep traps. We believe that our iodine-incorporated films are quite different from the PVK–I_2 complexes reported by Hermann and Rembaum[4,5] because their technique used for forming CT complexes in PVK is different from ours. In the following discussion, we refer to our samples as iodine-incorporated PVK and to theirs as PVK–I_2 complexes.

Carrier Generation

To avoid the light penetrating through the illuminated electrode to the opposite electrode, a gap electrode configuration as shown in Figure 1(B) was used. For pure PVK, the photocurrent I_{ph} is much larger when the illuminated electrode is at the negative polarity than when it is at the positive polarity under UV light illumination, as shown in Figure 6(A). Since only the positively biased electrode could inject holes, leading to the formation of a trapped homo-space charge near the injecting electrode, the field is suppressed near the positive electrode and enhanced near the negative electrode. The UV light generates singlet excitons in pure and iodine-incorporated PVK with the concentration higher near the illuminated electrode. These excitons produced near the negatively biased electrode will diffuse toward the positively biased electrode. The collision between these excitons and traps (traps with trapped holes act as electron traps) generates free holes provided that the applied field is large enough to facilitate carrier separation based
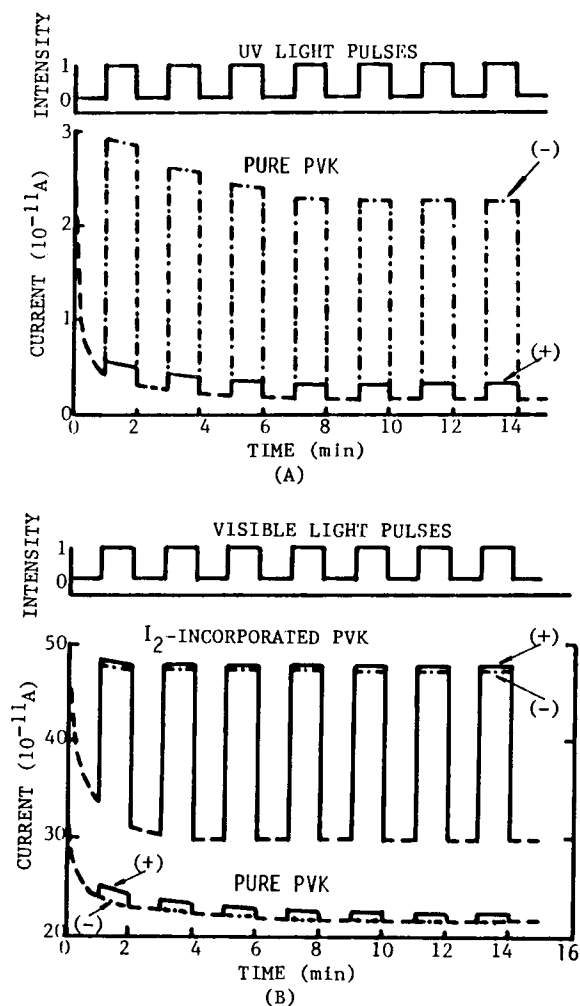
UV LIGHT PULSES

PURE PVK

(−)

(+)

TIME (min)

(A)

VISIBLE LIGHT PULSES

I_2-INCORPORATED PVK

(+)

(−)

(+)

PURE PVK

(−)

TIME (min)

(B)

Figure 6 Photocurrent superposed on to the dark charging current for the gap electrode configuration with the illuminated electrode biased at negative 200 V (−) and at positive 200 V (+). Light pulse duration is 1 min. (A) UV illumination for pure PVK; (B) visible illumination for pure PVK and iodine-incorporated PVK.

on the Onsager dissociation processes. This is why the photocurrent is higher when the illuminated electrode is negative than that when it is positive, as shown in Figure 6(A). This trend is just reversed for the illumination light in the visible region that is not absorbed by pure PVK. In this case, the photocurrent must be due to photoinjection of holes from the electrode. As expected, photoinjection occurs only when the illuminated electrode is positively biased for pure PVK, as shown in Figure 6(B).

For iodine-incorporated PVK, the visible light illumination causes the excitation of the electrons in the carbazole groups to make charge transfer to I_2 molecules, which requires only an energy of 1.8 eV, as shown in Figure 7. It is likely that since the con-

centration of I_2 is less than that of PVK in our films, the transport of holes generated by the excitation is associated with uncomplexed PVK, whereas the transport of electrons depends on the presence of I_2 in a manner similar to that in the PVK–TNF system.[14] The iodine-incorporated film may be regarded as a system consisting of uncomplexed carbazole units, uncomplexed I_2 molecules, and CT complexes. The transport mechanism for both holes and electrons is due to an intermolecular hopping process. For electrons, the transport could be due to hopping between uncomplexed I_2 molecules, between CT complexes, or both. This may be why the buildup of the photocurrent under light illumination and the current decay after the removal of the light illumination involve a bimolecular process, which will be discussed in under Current Transient below.

Photoconduction and Photoabsorption

The photocurrent as a function of photon energy for pure PVK at various applied fields is shown in Figure 8. Within the range of the photon energies used, there is negligible absorption in pure PVK and, therefore, carrier generation in the bulk is unlikely to take place. The photocurrent is due mainly to photoemission of holes from the positively biased electrode. According to the Fowler equation for photoemission from a metallic electrode to an insulator,[15] the quantum yield is proportional to $(h\nu - \phi_B)^2$, where $h\nu$ is the photon energy and ϕ_B is the threshold barrier height. By assuming that the quantum yield is proportional to I_{ph}/F, where I_{ph} is the photocurrent and F is the average applied field, the extrapolation of the plot $(I_{ph}/F)^{1/2}$ as a function of $h\nu$ to the zero current abscissa as shown in Figure 8(b) gives ϕ_B. For the Ag-illuminated electrode, ϕ_B is about 1.7 eV. That the data for the photon energy of 3.1 eV are far above the straight line as shown in Figure 8(B) may be caused by the filter bandwidth (~ 50 nm), which would make the point include some photons with energy extending to 3.3 eV that becomes absorbed by pure PVK. The small current in the near-infrared region may be attributed to the contribution by the field-enhanced thermionic emission. Our estimated 1.7 eV for ϕ_B for the Ag electrode is in close agreement with the photoemission threshold of 1.8 eV for the Ag electrode reported by Reucroft and Ghosh.[9] However, our results show that ϕ_B is not sensitive to the applied field, at least for the range of the fields from 1×10^5 to 5×10^5 V/cm in this investigation. This may be attributed to the homo-space charge formed by free

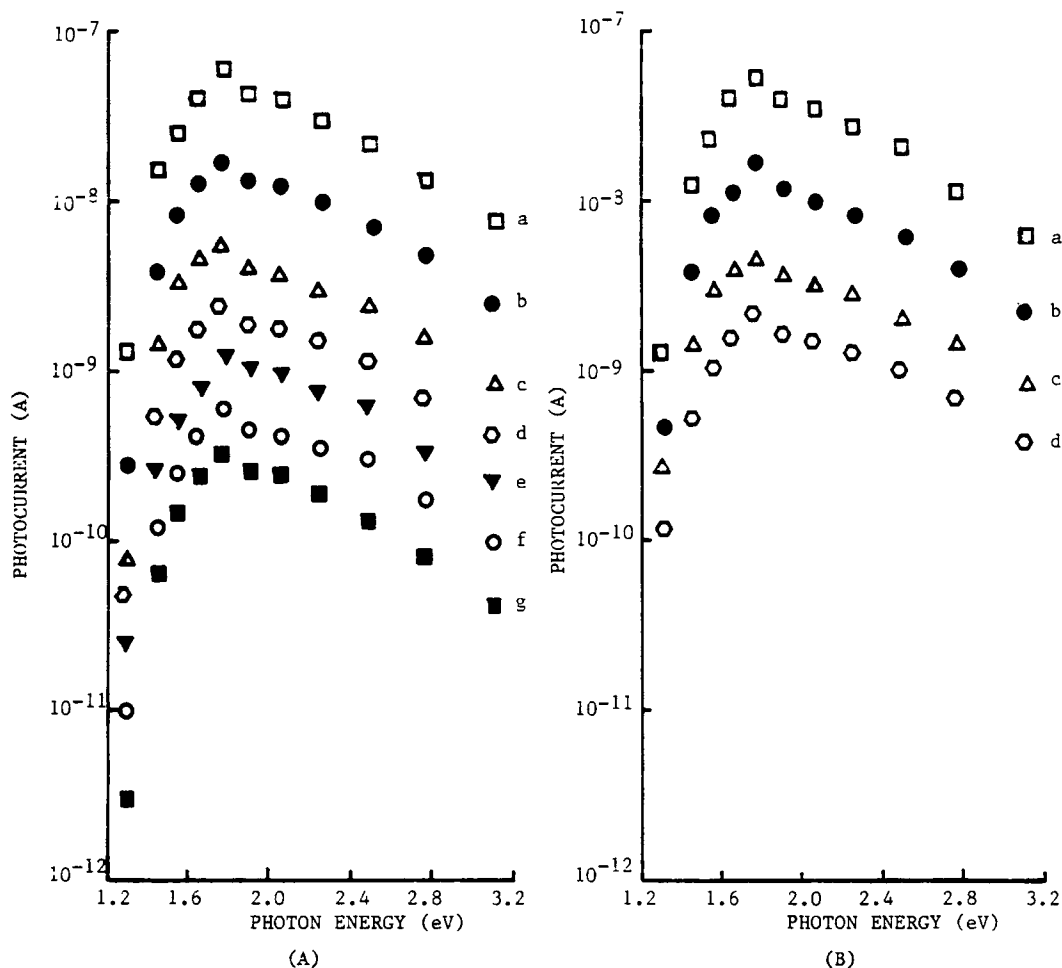**Figure 7**   Photocurrent as a function of photon energy for iodine-incorporated PVK at various electric fields: (a) 1×10^5 V/cm; (b) 5×10^4 V/cm; (c) 2×10^4 V/cm; (d) 1×10^4 V/cm; (e) 5×10^3 V/cm; (f) 2×10^3 V/cm; (g) 1×10^3 V/cm. (A) Illuminated electrode at positive polarity; (B) illuminated electrode at negative polarity.

and trapped holes near the hole-injecting contact, which shields from the effect of field-induced Schottky barrier lowering.

Figure 7 shows the photocurrent as a function of photon energy for iodine-incorporated PVK at various electric fields. There are three features in this figure: (1) There is almost no difference in photocurrent between the case with the illuminated electrode positively biased and that with it negatively biased; (2) photocurrent is observable even when the applied field is as low as 10^3 V/cm; (3) there exists a photocurrent peak at about 1.8 eV. The relative optical absorption for the PVK–I_2 complexes, I_2, and iodine-incorporated PVK are shown in Figure 9. It is well known that the absorption bands of PVK and TNF are in the UV region,[3] but the formation of PVK–TNF complexes results in a broad-band absorption—extending to the visible region. However, the absorption of PVK–I_2 complexes does not

extend the absorption band beyond the I_2 absorption band, whereas the absorption of iodine-incorporated PVK does extend to a much longer wavelength region. That the shape of the photocurrent spectrum of iodine-incorporated PVK is similar to that of optical absorption of I_2 implies that the CT between inorganic and organic groups is similar to interband transition in inorganic crystals. The difference is that there are a large quantity of trap states in iodine-incorporated PVK so that the absorption peak becomes very broad. This is why the photons of higher energies in the UV region do not induce a larger current in iodine-incorporated PVK. It should be noted that the formation of PVK–I_2 complexes involves structure changes of PVK upon complexing.[16] We believe that in our iodine-incorporated PVK the PVK structure remains unchanged. Although the CT take place between PVK and I_2, they may not be complexed.
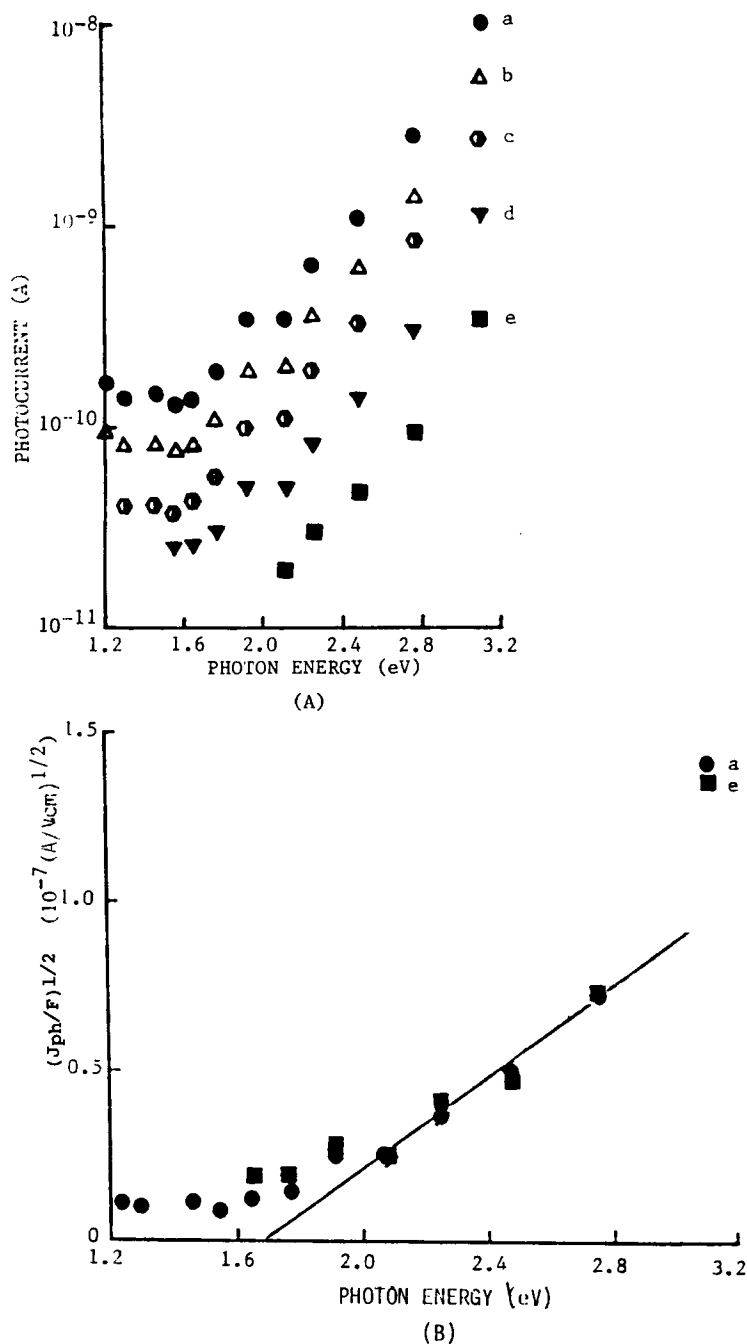
Figure 8 Photocurrent as a function of photon energy for pure PVK at various electric fields: (a) 5×10^5 V/cm; (b) 4×10^5 V/cm; (c) 3×10^5 V/cm; (d) 2×10^5 V/cm; (e) 1×10^5 V/cm. (A) I_{ph} vs. $h\nu$; (B) $(I_{ph}/F)^{1/2}$ vs. $h\nu$.

Current Transient

The buildup of the photoinduced current after the application of light illumination and the decay of this current after the removal of the light illumination as functions of time have been measured. A dc bias field was first applied to the film until the current reached a steady-state value. After that, the film was subjected either to a step function illumi-nation or to a sudden switch-off of the light. The time evolution of the current was recorded with a chart drive recorder. Typical results are shown in Figure 10. For pure PVK, the photocurrent buildup and the current decay follow closely the monomo-lecular process that can be expressed as
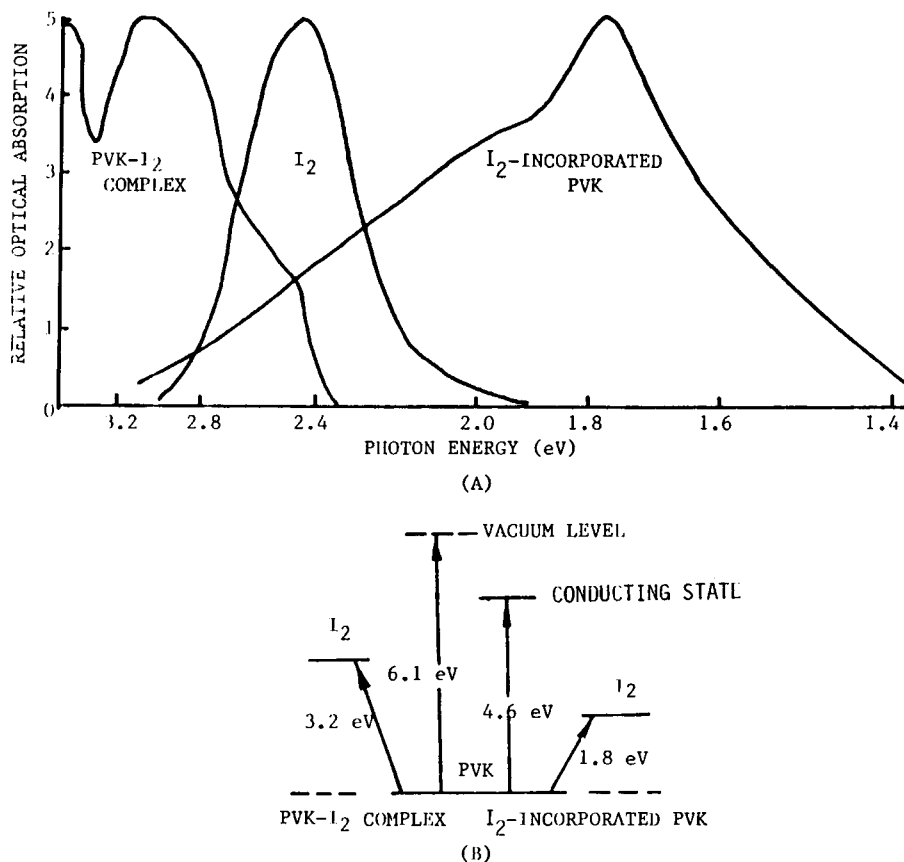
$$I(t) = I_0(1 - e^{-t/\tau}) \tag{5}$$

Figure 9 (A) Comparison of optical absorption spectra among PVK-I_2 complex,[5,16]I_2, and iodine-incorporated PVK; (B) energy-level diagram for PVK, PVK-I_2, and iodine-incorporated PVK.

for the current buildup and

$$I(t) = I_0 e^{-t/\tau} \tag{6}$$

for the current decay, where I_0 is the initial value of the current and τ is the characteristic time constant. For iodine-incorporated PVK, the current transient follows the bimolecular process that can be expressed as

$$I(t) = I_0 \tanh(\beta t) \tag{7}$$

for the current buildup and

$$I(t) = I_0(1 + \alpha t)^{-1} \tag{8}$$

for the current decay, where α and β are constants. For pure PVK, the photocurrent buildup and the current decay are in good agreement with the mono-molecular process because in pure PVK, where the carriers are mainly holes, recombination results from the encounter of holes with hole traps (trapped

electrons in traps act as hole traps). For iodine-incorporated PVK, however, the photocurrent buildup is not quite consistent with the bimolecular process, but the current decay approximately follows this process because the current buildup is due mainly to the charge transfer between PVK and I_2 and the current decay is due mainly to the recombination of electrons and holes.

The current decay in iodine-incorporated PVK depends on the history of illumination: The longer the illumination, the slower the current decay. The current decay depends also on the light wavelength. After the illumination with all different wavelengths in the visible light region, the current decay after the removal of the light illumination could last as long as 24 h. Even after short-circuiting the electrodes in the dark or under illumination, the charge carriers could not be eliminated. A similar persistent conduction phenomena has also been observed in other PVK-based photoconductors.[17,18] It is likely that the lifetime of the charge remaining in I_2 after CT in iodine-incorporated PVK is several orders of magnitude greater than those of the excited singlet

EXPERIMENTAL CURVE

THEORETICAL CURVE

$I(t)/I_0 = 1 - e^{-t/\tau}$ $\tau = 0.3$min

$I(t)/I_0 = e^{-t/\tau}$ $\tau = 0.3$min

TIME (min)

(A)

EXPERIMENTAL CURVE

THEORETICAL CURVE

$I(t)/I_0 = \tanh(\beta t)$ $\beta = 0.8$min^{-1}

$I(t)/I_0 = 1/(\alpha t + 1)$ $\alpha = 1.46$min^{-1}
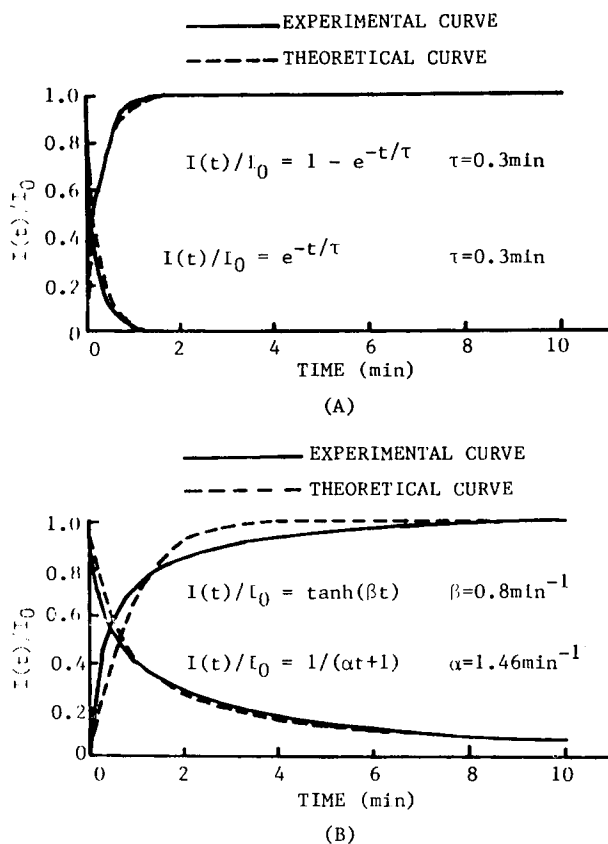
TIME (min)

(B)

Figure 10 The buildup of the photoinduced current after the application of a step-function illumination and the decay of the photoinduced current after the switch-off of the light as functions of time for (A) pure PVK, $\lambda = 500$ nm, $F = 10^5$ V/cm and (B) iodine-incorporated PVK, $\lambda = 700$ nm, $F = 10^5$ V/cm.

(nanoseconds) or triplet (microseconds) states in pure PVK.

CONCLUSIONS

On the basis of the experimental results described above, the following conclusions are drawn:

1. The iodine in iodine-incorporated PVK remains in the form of molecules. There may be a weak attractive force binding the carbazole groups and the iodine molecules.

2. The dark conductivity of iodine-incorporated PVK films with 2.3% of iodine in weight is about two orders of magnitude greater than that of pure PVK films. Photoabsorption is extended from the UV region to the entire visible region. There is a peak at the photon energy of 1.8 eV.

3. For iodine-incorporated PVK, the buildup of

the photocurrent after illumination does not closely follow the bimolecular process, but the current decay after the removal of the light follows approximately the bimolecular recombination process.

4. There are deep traps in pure PVK, but after the incorporation of iodine, the presence of deep traps disappears and a large number of shallow traps is introduced.

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