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Charge storage mechanism in vacuum deposited PVF films

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Thermally stimulated discharge current (TSD) behavior of vacuum deposited polyvinyl fluoride (PVF) films has been studied as a function of various polarization parameters such as field (1.0×10^3–14.0×10^3 V/cm), temperature (313–453 K), time (3.6×10^3–6.0×10^3 sec), electrode work function (copper, silver, aluminum and indium) and thickness (2000–10000 Å). In a field dependent case the TSD spectra of PVF films show a single relaxation peak centered around 430 ± 1 K. The peak current, charge, peak position and activation energy associated with the peak depend strongly on the polarization parameters. The mechanism of origin of the relaxation peak has been attributed to the space charge polarization where the charge carriers injected at the electrode-polymer interface barrier are displaced at macroscopic distances and get subsequently trapped at trapping levels that are distributed in their activation energies and relaxation times.

1. Introduction

Amorphous selenium (a-Se), in its pure, alloyed and doped forms, finds applications [1–4] as photoreceptor imaging material in electrophotography in general and xeroradiography (XR) in particular. As regards XR, these materials show inadequate X-ray sensitivity, thus limiting its use only for soft tissue applications such as mammography (breast cancer detection at early stage) rather than general bone extremities such as fracture cases. Therefore current thrust of research in the area of XR is to develop dose efficient selenium based imaging materials. For this many fundamental and applied investigations have already been made [5–14] by studying various important parameters in a-Se films and related materials. It includes the study of defect states, charge storage mechanism, transport phenomena, X-ray sensitivity etc. Keeping in view the above aspect of sensitivity enhancement in a-Se films we are currently working on a new concept of incorporating interface barrier layer of suitable polymeric materials in combination with a-Se films in the XR mode. Some of our recent [15] studies have shown promising results in this respect. However, it may be mentioned here that to find the suitability of a polymer as an interfacial barrier layer in XR photoreceptor mode, it is essential to study its various properties such as the mechanism of charge storage, charge transport, trapping etc. Keeping this in view we have made, for the first time, detailed investigations of the charge storage behavior in vacuum deposited polyvinyl fluoride (PVF) films using thermally stimulated discharge current (TSD) technique. In these investigations the TSD behavior of vacuum deposited PVF films has been studied as a function of various polarization parameters such as field (E_p), temperature (T_p), time (t_p), electrode work function (φ) and thickness (d). As regards PVF some investigations have already been made [16, 17] in its vacuum deposited film form and the present detailed investigations throw further light and reveal clearly the charge storage behavior of such films. The interesting results found in these investigations are reported in this paper.

2. Experimental procedure

PVF films were vacuum deposited on to thoroughly cleaned glass slides of size $7.5 \times 2.5 \times 0.13$ cm. These glass slides were cleaned using liquid detergent like genteel and chemical solvent like acetone. After cleaning, the substrates were dried out in the vacuum oven by keeping them at a temperature of about 373 K for about an hour. Films of PVF were vacuum deposited onto these substrates at a pressure $\sim 10^{-4}$ torr. PVF in white shiny powder form was obtained from Poly Sciences Inc., USA. Its molecular weight, glass transition temperature and density were of the order of 110000, 253 K and 1.41 gm/cm^3, respectively. The chemical structure of the PVF being $CH_2{=}CHF$ which has distinct C—F with dipole moment [18] ~ 1.468. The material for vacuum depositing PVF films was kept in tungsten boat inside the vacuum chamber. Glass slides of size mentioned above were kept at the distances of about 18 cm above the tungsten boat. The films were deposited at a rate of 0.01 μm/minute. Subsequently

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these films were annealed in air at about 383 K for about 2 hours to ensure complete adherence of the deposited film to the glass substrate. The films were characterized by studying the infrared (IR) spectra in the range 4000–400 cm^{-1}. In fact the IR spectra of vacuum deposited films were compared (results not shown) with the corresponding spectra of PVF films prepared by solution evaporation technique. The spectra of two types of films showed identical behaviour except for a slight reduction in the intensity of the absorption peaks of the vacuum deposited PVF films. This may be attributed for the reduction in the molecular weight of the polymer on vacuum evaporation as has also been observed by other authors [16] in case of evaporated PVF films. The thicknesses of PVF film was in the range ~2000 Å to 10000 Å. For making TSD measurements, electrodes of various materials were vacuum deposited onto these films in a conventional surface configuration having the polymer film width ~0.08 cm. This surface configuration was obtained by putting of copper wire of width ~0.08 cm on top of the PVF films. The wire masked the polymer film below it. Subsequently electrode materials for contact formation were vacuum deposited onto these masked films. This resulted into a sample having a surface geometry of metal–PVF–metal where the polymer film width being 0.08 cm. Effective dimensions of the PVF samples used for charge storage measurement being 2.5 × 0.08 cm with different polymer film thicknesses. Electrical contacts to these electrodes were made by means of an air drying type of conducting silver paint. TSD measurements were made by polarizing the sample under different polarization conditions and heating it at a constant linear rate under short circuit conditions. The current so released was measured in the form of a TSD spectra using a Keithley 610 C Electrometer coupled to Riken Denshi X-Y recorder.

3. Experimental results

Measurements were started with the virgin sample. A virgin sample of vacuum deposited PVF film of $d \sim 10000$ Å was kept at $E_p = 0$, $T_p = 393$ K and $t_p = 4.5 \times 10^3$ sec, and then heated at a constant heating rate (H) = 0.083 K/sec. No current was found to be released in the external circuit in the temperature range of 313 to 453 K. It shows that no transport of charge takes place in virgin vacuum deposited PVF films when treated thermally in the absence of E_p. However, in the presence of E_p, PVF films get polarized which, on heating at a constant linear heating rate (0.083 K/sec) result in specific TSD spectra. The effect of various polarization parameters on TSD spectra are discussed below.

3.1. Field dependence

The field dependent TSD spectra obtained for vacuum deposited PVF films of $d \sim 10000$ Å and polarized under identical conditions of $T_p = 393$ K and $t_p = 4.5 \times 10^3$ sec but for different E_p are shown in Fig. 1. Curves A, B, C, D and E correspond to E_p of 1.0×10^3, 2.0×10^3, 4.0×10^3, 8.0×10^3 and 14.0×10^3 V/cm, respectively. It is seen from Fig. 1 that a single relaxation peak centered around 430 ± 1 K is obtained in TSD spectra at all E_p. It is also seen that peak current (I_M) and the charge (Q) associated with the relaxation peak depend strongly on E_p. In fact charge Q associated with the peak is the covered area under the peak which has been calculated by the product of $I \times t$ (where I is the value of current on Y-axis and t is the time inherent in the temperature on X-axis). To see the dependence of both I_M and Q on E_p, a plot of I_M vs E_p and Q vs E_p have been made as shown in Figs 2a and b, respectively. It is seen from these figures that both I_M and Q vary non-linearly with the increase in E_p showing a tendency of saturation towards high fields.

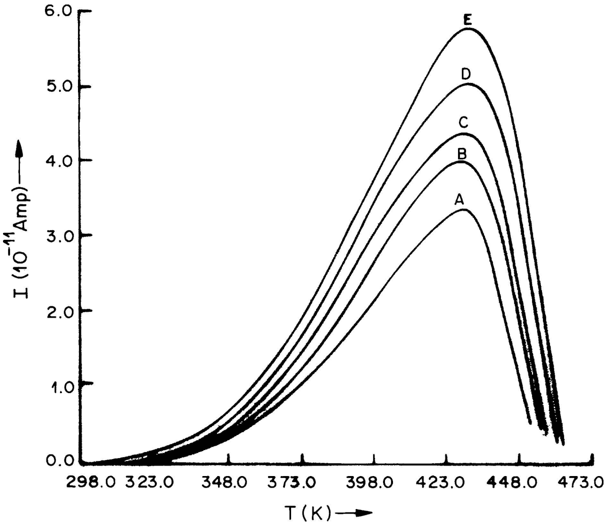


Figure 1 TSD spectra of vacuum deposited PVF films ($d \sim 10000$ Å), polarized at $T_p = 393$ K for $t_p = 4.5 \times 10^3$ sec but for different E_p. Curves A, B, C, D and E correspond to E_p of 1.0×10^3, 2.0×10^3, 4.0×10^3, 8.0×10^3 and 14.0×10^3 V/cm, respectively.

The activation energy (U) associated with the relaxation peak has been calculated using the initial rise method [19]. Plots of log I vs $1/T$ has been made for all the curves under the TSD spectra of Fig. 1. These plots are shown in Fig. 3. The values of U obtained from the slope of these plots have been found to be ~0.65 eV at all fields. Using this value of U, other electrical parameters such as relaxation time at infinite temperature (τ_0), relaxation time at peak temperature (τ_M) and relaxation time at room temperature (τ_{300}) associated with the peak have also been calculated [20] and are tabulated in Table I.

3.2. Temperature dependence

The temperature dependent TSD spectra for vacuum deposited PVF films of $d \sim 10000$ Å and polarized under identical conditions of $E_p = 8.0 \times 10^3$ V/cm and $t_p = 4.5 \times 10^3$ sec but for different T_p are shown in Fig. 4. Curves A, B, C, D and E correspond to T_p of 313, 353, 393, 433 and 453, respectively. It is seen from Fig. 4 that in this case also a single relaxation peak is obtained in the TSD spectra. The peak position in this case is found to be dependent on T_p and lies in the temperature range of 410 to 445 K. To see the actual dependence of T_M on T_p, a plot of T_M vs T_p has been

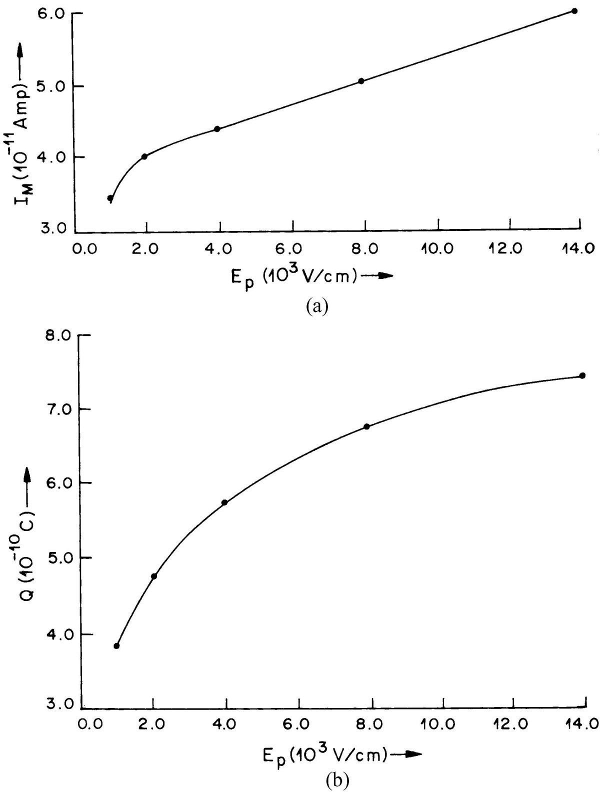


Figure 2 (a) Plots of I_M vs E_p for the curves A, B, C, D and E for Fig. 1. (b) Plots of Q vs E_p for the curves A, B, C, D and E for Fig. 1.

made and it is shown in Fig. 5. It is seen from this figure that T_M varies linearly with the increase in T_p. Further it is seen from Fig. 4 that both I_M and Q associated with the relaxation peak depend on the value of T_p. Plots of I_M vs T_p and Q vs T_p have also been made and shown in Figs 6a and b, respectively. It is seen from these plots that I_M and Q increase almost in a linear way with the increase in T_p.

The values of U associated with the relaxation peak have again been evaluated by initial rise method. Log I vs $1/T$ plots are shown in Fig. 7 and the values of U have been found to lie in the range 0.55 eV to 0.69 eV. It is observed from these values that U depends strongly on T_p. To see its actual dependence on T_p the plots of U vs T_p have been made and are shown in Fig. 8. It is seen from Fig. 8 that U increases almost in a linear way with the increase in T_p. The values of other relaxation parameters such as τ_0, τ_M and τ_{300} etc. have also been evaluated as in the previous case of E_p variation and are given in Table II.

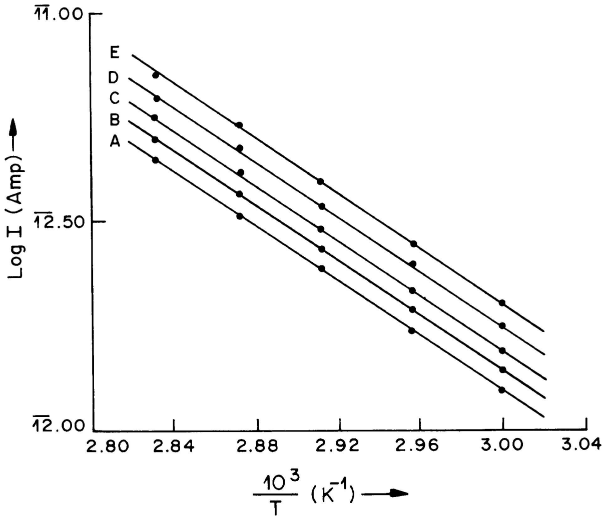


Figure 3 Plots of Log I vs $1/T$. Curves A, B, C, D and E correspond to E_p of 1.0×10^3, 2.0×10^3, 4.0×10^3, 8.0×10^3 and 14.0×10^3 V/cm, respectively.

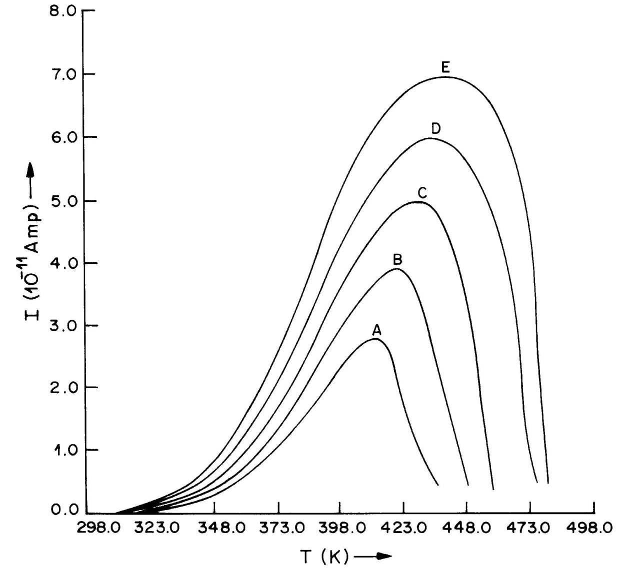


Figure 4 TSD curves of vacuum deposited PVF films ($d \sim 10000$ Å), polarized at $E_p = 8.0 \times 10^3$ V/cm for $t_p = 4.5 \times 10^3$ sec but for different T_p. Curves A, B, C, D and E correspond to T_p of 313, 353, 393, 433 and 453 K, respectively.

3.3. Time dependence

The time dependent TSD spectra obtained for vacuum deposited PVF films of $d \sim 10000$ Å and polarized under identical conditions of $E_p = 8.0 \times 10^3$ V/cm and $T_p = 393$ K but for different t_p are shown in Fig. 9. Curves A, B, C, D and E in the figure correspond to t_P of 3.6×10^3, 3.96×10^3, 4.5×10^3, 5.4×10^3

TABLE I Depolarization kinetics data calculated by the initial rise method for peak of the vacuum deposited PVF films of thickness 10000 Å polarized at $T_p = 393$ K for $t_p = 4.5 \times 10^3$ sec but for different E_p. The films were heated during the depolarization at $H = 0.083$ K/sec

Curve (Fig. 1)	$E_p \times 10^3$ (V/cm)	T_M (K)	$I_M \times 10^{-11}$ (Amp)	$Q \times 10^{-10}$ (C)	U (eV)	τ_0 (sec)	$\tau_M \times 10^2$ (sec)	τ_{300} (sec)
A	1	430	3.4	3.88	0.65	7.07×10^{-6}	2.94	5.89×10^5
B	2	429	4.0	4.85	0.65	6.75×10^{-6}	2.93	5.63×10^5
C	4	431	4.35	5.81	0.65	7.39×10^{-6}	2.96	6.17×10^5
D	8	430	5.0	6.82	0.65	7.07×10^{-6}	2.94	5.89×10^5
E	14	431	5.8	7.40	0.65	7.39×10^{-6}	2.96	6.17×10^5

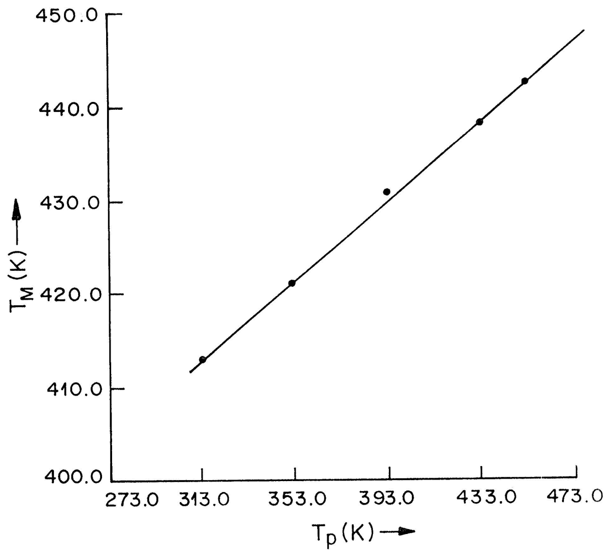
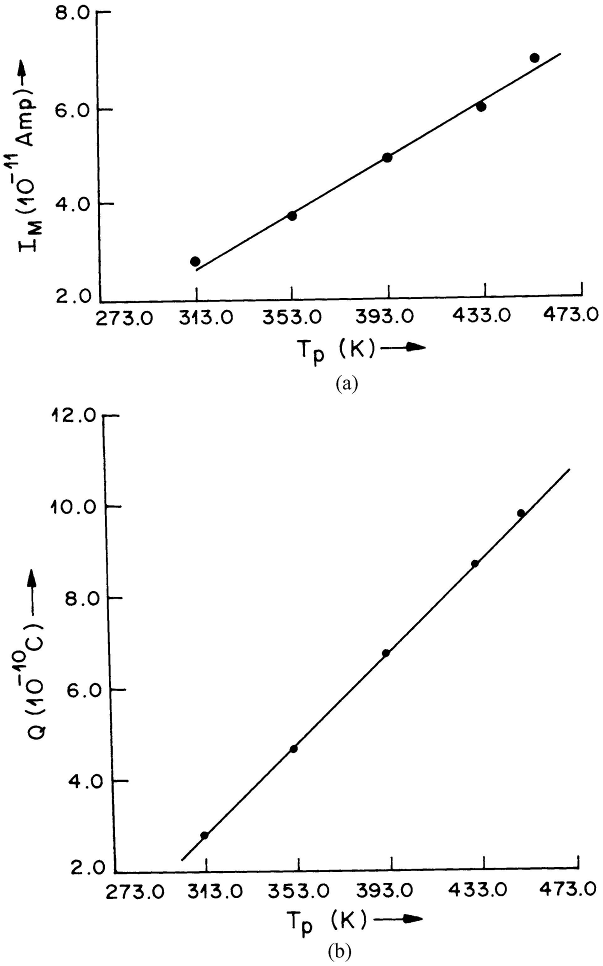


Figure 5 Plots of T_M vs T_p for the curves A, B, C, D and E for Fig. 4.



(a)

(b)

Figure 6 (a) Plots of I_M vs T_p for the curves A, B, C, D and E for Fig. 4. (b) Plots of Q vs T_p for the curves A, B, C, D and E for Fig. 4.

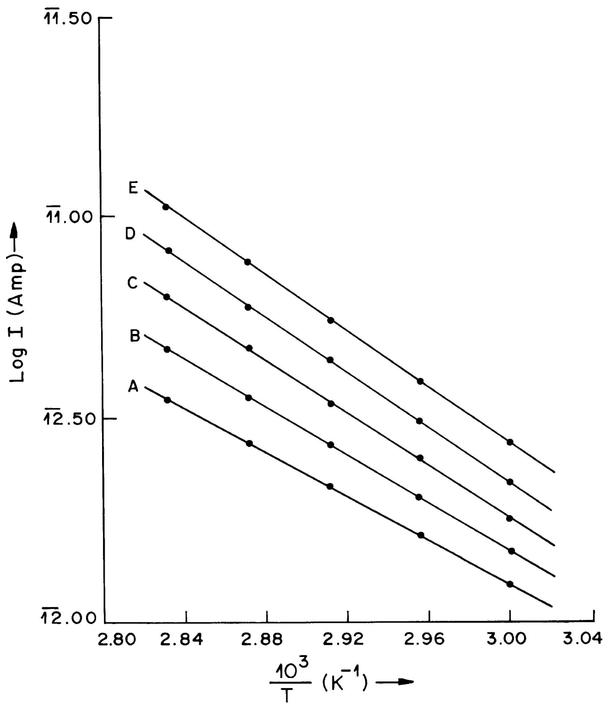


Figure 7 Plots of Log I vs $1/T$. Curves A, B, C, D and E correspond to T_p of 313, 353, 393, 433 and 453 K, respectively.

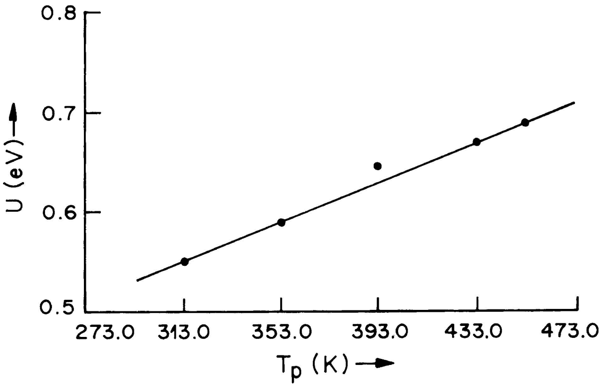


Figure 8 Plots of U vs T_p for the curves A, B, C, D and E for Fig. 4.

and 6.0 × 10^3 sec, respectively. It is seen from Fig. 9 that here again a single relaxation peak is observed. The peak position in this case lies in the range 420 to 440 K. Thus it is observed that in this case also the peak temperature T_M of the relaxation peak depends on the value of t_p. To see the actual dependence of T_M on t_p, a plot of T_M vs t_p have been made and it has shown in Fig. 10. It is seen from the figure that T_M varies almost in a linear way with t_p. It is further seen from Fig. 9 that both I_M and Q associated with the relaxation peak increase with the increase in t_p. The actual variation of I_M vs t_p and Q vs t_p is shown in Figs 11a and b, respectively. Here again it is seen that both I_M and Q vary almost linearly with the variation in t_p. However, it may be mentioned here that the respective change in the values of T_M, I_M and Q are less in t_p case as compared to the corresponding changes in the case of T_p variation.

The initial rise plots of log I vs $1/T$ for the calculation of U in this case is shown in Fig. 12. The values of U obtained from these plots have been found in the range 0.62 to 0.68 eV. The actual dependence of U on t_p is shown in Fig. 13. It is seen from this figure that U varies linearly with the increase in t_p. The values of other relaxation parameters such as τ_0, τ_M and τ_{300} etc. have been calculated using the above values of U and are tabulated in Table III.

TABLE II Depolarization kinetics data calculated by the initial rise method for peak of the vacuum deposited PVF films of thickness 10000 Å polarized with $E_p = 8.0 \times 10^3$ V/cm for $t_p = 4.5 \times 10^3$ sec but for different T_p. The films were heated during the depolarization at $H = 0.083$ K/sec

Curve Fig. 4	T_p (K)	T_M (K)	$I_M \times 10^{-11}$ (Amp)	$Q \times 10^{-10}$ (C)	U (eV)	τ_0 (sec)	$\tau_M \times 10^2$ (sec)	τ_{300} (sec)
A	313	413	2.8	2.78	0.55	6.22×10^{-5}	3.21	1.08×10^5
B	353	421	3.9	4.13	0.59	2.68×10^{-5}	3.11	2.20×10^5
C	393	430	5.0	6.82	0.65	7.07×10^{-6}	2.94	5.89×10^5
D	433	438	6.0	9.59	0.67	5.77×10^{-6}	2.96	1.04×10^6
E	453	443	6.98	10.58	0.69	4.15×10^{-6}	2.94	1.63×10^6

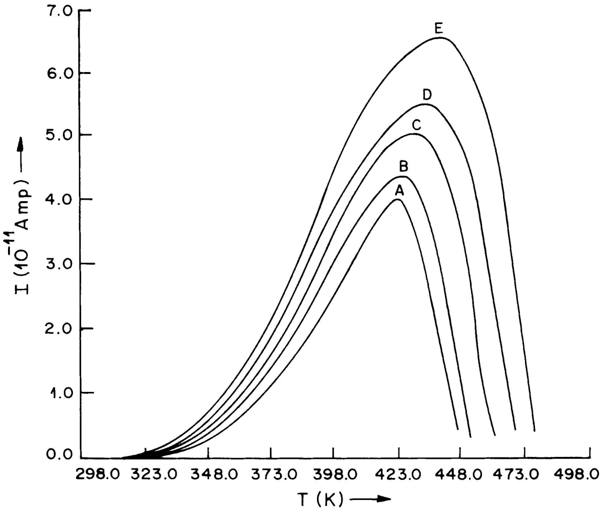


Figure 9 TSD spectra of vacuum deposited PVF films ($d \sim 10000$ Å), polarized at $E_p = 8.0 \times 10^3$ V/cm, $T_p = 393$ K but for different t_p. Curves A, B, C, D and E correspond to t_p of 3.6×10^3, 3.96×10^3, 4.5×10^3, 5.4×10^3 and 6.0×10^3 sec, respectively.

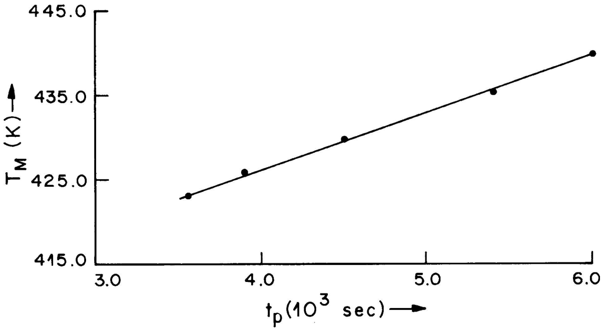
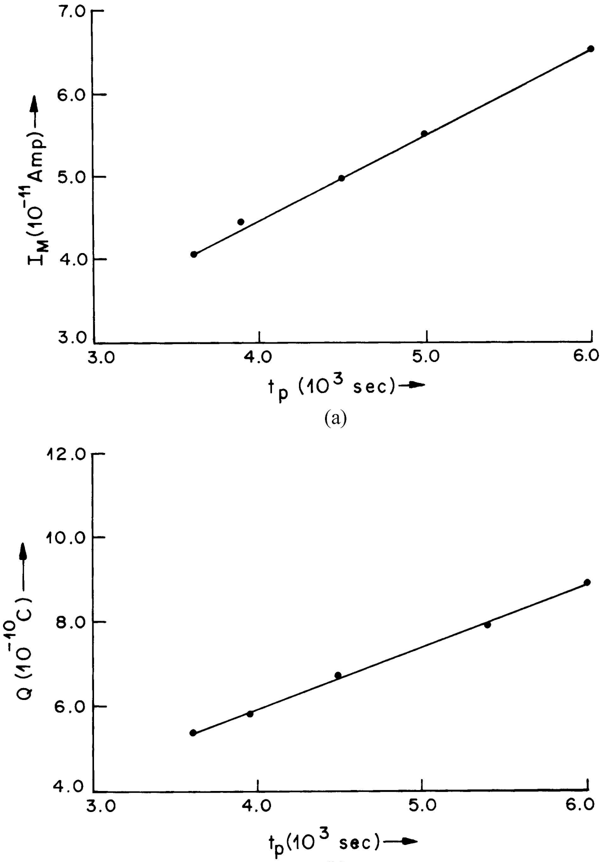


Figure 10 Plots of T_M vs t_p for the curves A, B, C, D and E for Fig. 9.



(a)

(b)

Figure 11 (a) Plots of I_M vs t_p for the curves A, B, C, D and E for Fig. 9. (b) Plots of Q vs t_p for the curves A, B, C, D and E for Fig. 9.

3.4. Electrode work function dependence

The TSD spectra obtained for vacuum deposited PVF films of $d \sim 10000$ Å and polarized under identical conditions of $E_p = 8.0 \times 10^3$ V/cm, $T_p = 393$ K and $t_p = 4.5 \times 10^3$ sec but for different electrode metals are shown in Fig. 14. Curves A, B, C and D in Fig. 14 correspond to electrode metal of copper (Cu), silver (Ag), aluminum (Al) and indium (In), respectively in their decreasing order of electrode work function (φ) [21], i.e., $\varphi_{Cu} > \varphi_{Ag} > \varphi_{Al} > \varphi_{In}$. The values of φ of these electrode materials along with their ionization potential (IP) and electron affinity are also compared [18] in Table IV with the IP [18] and electron affinity [22] of the PVF polymer used in the investigation. It is seen from the Fig. 14 that in this case also a single relaxation peak centered at 430 ± 1 K is obtained. It is further seen from the figure that both I_M and Q associated with the relaxation peak depend on electrode material used and increase with the increase in φ of the electrode material. However, this case is similar to that of E_P variation where the peak position remains fixed and U as evaluated by initial rise method (results not shown) also remains the same.

3.5. Thickness dependence

The TSD spectra obtained for vacuum deposited PVF films polarized under identical conditions of $E_p = 8.0 \times 10^3$ V/cm, $T_p = 393$ K and $t_p = 4.5 \times 10^3$ sec but for different d are shown in Fig. 15. Curves A, B, C and D in figure correspond to d of 2000, 5000, 8000 and 10000 Å, respectively. It is seen from Fig. 15 that a single relaxation peak centered at 430 ± 1 K is obtained. It is further seen that both I_M and Q depend on the d of the film. With the increase in d these quantities increase initially and than show saturation to-

TABLE III Depolarization kinetics data calculated by the initial rise method for peak of the vacuum deposited PVF films of thickness 10000 Å polarized with $E_p = 8.0 \times 10^3$ V/cm at $T_p = 393$ K but for different t_p. The films were heated during the depolarization at $H = 0.083$ K/sec

Curve Fig. 9	$t_p \times 10^3$ (sec)	T_M (K)	$I_M \times 10^{-11}$ (Amp)	$Q \times 10^{-10}$ (C)	U (eV)	τ_0 (sec)	$\tau_M \times 10^2$ (sec)	τ_{300} (sec)
A	3.6	423	4.05	5.39	0.62	1.22×10^{-5}	2.99	3.19×10^5
B	3.96	426	4.40	5.81	0.63	1.05×10^{-5}	2.98	4.03×10^5
C	4.5	430	5.00	6.82	0.65	7.07×10^{-6}	2.94	5.89×10^5
D	5.4	435	5.45	7.82	0.67	5.03×10^{-6}	2.92	9.10×10^5
E	6.0	440	6.50	8.97	0.68	4.78×10^{-6}	2.94	1.27×10^6

TABLE IV Work function, ionization potential and electron affinity of the electrode materials and polymer PVF

S. No	Electrode material	Work function (eV)	Ionization potential (eV)	Electron affinity (eV)	Ionization potential of PVF (eV)	Electron affinity of PVF (eV)
1	Copper	4.47	7.72638	1.235	10.363	1.91
2	Silver	4.28	7.57624	1.302		
3	Aluminum	4.20	5.98577	0.441		
4	Indium	4.0	5.78636	0.3		

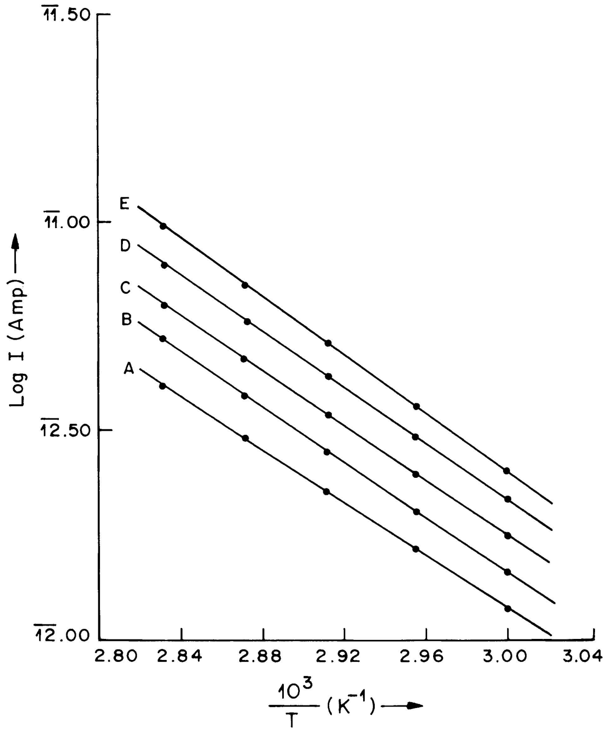


Figure 12 Plots of Log I vs $1/T$. Curves A, B, C, D and E correspond to t_p of 3.6×10^3, 3.96×10^3, 4.5×10^3, 5.4×10^3 and 6.0×10^3 sec, respectively.

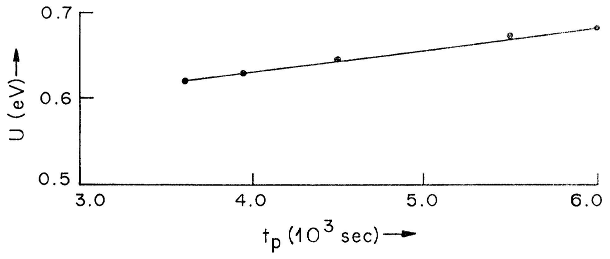


Figure 13 Plots of U vs t_p for the curves A, B, C, D and E for Fig. 9.

wards higher d as shown by their respective plots in Figs 16a and b, respectively. Also the U and relaxation parameters remain the same as in the case of E_P and φ variation.

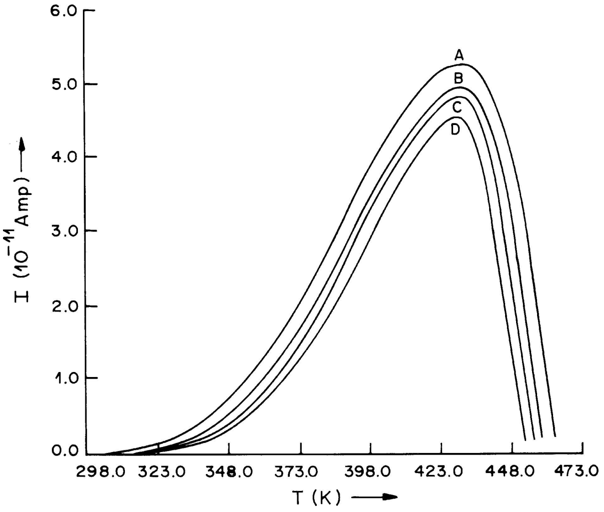


Figure 14 TSD spectra of vacuum deposited PVF films ($d \sim 10000$ Å), polarized under identical conditions at $E_p = 8.0 \times 10^3$ V/cm, $T_p = 393$ K and $t_p = 4.5 \times 10^3$ but for different electrode metals. Curves A, B, C and D correspond to electrode metals of Cu, Ag, Al and In, respectively.

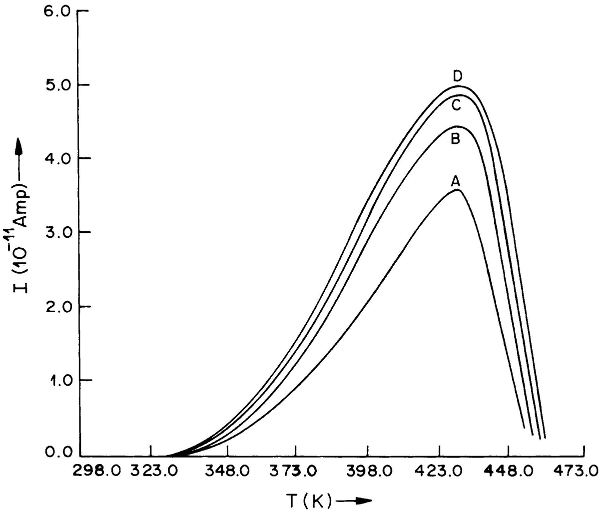
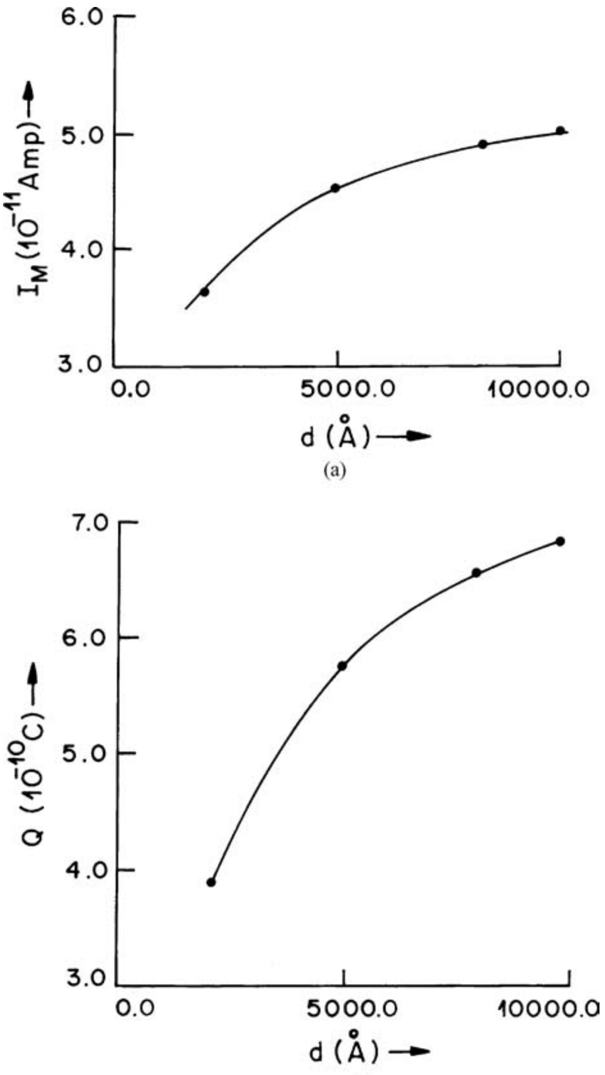


Figure 15 TSD spectra of vacuum deposited PVF films, polarized under identical conditions at $E_p = 8.0 \times 10^3$ V/cm, $T_p = 393$ K and $t_p = 4.5 \times 10^3$ sec but for different d. Curves A, B, C and D correspond to d of 2500, 5000, 7500 and 10000 Å, respectively.



(a)

(b)

Figure 16 (a) Plots of I_M vs d for the curves A, B, C and D for Fig. 15. (b) Plots of Q vs d for the curves A, B, C and D for Fig. 15.

Based on the analysis of these TSD results we have suggested dominant mechanism of charge storage in vacuum deposited PVF films as discussed below.

4. Discussion

It is known [20] that polarization in dielectric/polymeric materials takes place as a result of one or more than one of the following mechanisms. It includes the dipolar orientation, Maxwell-Wagner-Sillars (MWS) effect (displacement of charge carrier at microscopic distances and their subsequent trapping), space charge effects (displacement of charge carrier at macroscopic distances and their subsequent trapping), ionic effects etc.

The mechanism of dipolar orientation and MWS effect are the cases of uniform polarization wherein the charge associated with the relaxation peak varies linearly with the increase in E_p. Only difference between the two being that in case of dipolar polarization one has permanent dipoles whereas in MWS polarization the dipole formation takes place inside the polymer by displacement of charge carriers at microscopic distances and their subsequent trapping thus giving uniform polarization effect. On the other hand the mechanism of space charge polarization and ionic polarization is a non-linear case wherein the charge carriers injected in the polymer or present inherently are displaced at macroscopic distances and subsequently trapped in a non-uniform way. Thus it is clear from above discussion that one can arrive in establishing the dominant mechanism of polarization in polymers by analyzing the TSD spectra as a function of various polarization parameters.

It is seen from Figs 2a and b that I_M and Q associated with the relaxation peak depend strongly on E_p. In fact these parameters show a tendency of saturation towards higher E_p as seen from these figures. It clearly indicates that the present case of PVF films is a case of non-linear/non-uniform polarization thus ruling out the first two mechanisms of uniform polarization, i.e., dipolar orientation and MWS effect. Therefore it may be suggested that polarization in vacuum deposited PVF films is governed dominantly by a non-linear process such as space charge polarization or ionic polarization. This suggested mechanism finds further support from the observation that both I_M and Q associated with the relaxation peak depend non-linearly on the d of the PVF film as well, as seen from Figs 16a and b, respectively.

The dependence of TSD spectra on φ of the electrode materials, however, indicates that the origin of space charge in vacuum deposited PVF films may be dominantly due to injected charge carriers rather than ionic ones present inherently. Also the order of the U (0.55 to 0.69 eV) associated with the relaxations peak supports further the origin of the peak due to the injected charge carriers rather than the inherently ionic ones as in the later case the value of activation energy is usually much higher than these values. This injection mechanism finds further support from Fig. 14 where I_M and Q associated with the peak depend strongly on the electrode-polymer interface barrier. In case of electrode having maximum φ/IP/electron affinity (interface barrier being minimum as the polymer IP/electron affinity being higher than that of the electrodes used) I_M and Q are maximum. Similarly in case of electrode with least φ (interface barrier being highest) these quantities are minimum. These observations therefore suggest that it a clear case of injection mechanism.

In view of above therefore, it may be suggested that polarization in vacuum deposited PVF films is dominantly governed by space charge effects where the charge carriers injected into the polymer by surmounting the electrode-polymer interface barrier get displaced at macroscopic distances and are subsequently trapped. Further it is seen from Figs 8 and 13 that U associated with the charge carriers depend both on the T_p or t_p and it varies linearly with the increase the T_p or t_p thereby suggesting that the present case is not a case of discrete relaxation rather it is a case of distributed polarization where injected charge carriers are trapped at energy levels which are distributed in their activation energies and relaxation times that lie in the range 0.55 to 0.69 eV and 1.08 × 10^5 to 1.63 × 10^6 sec (room temperature values), respectively.

5. Conclusion

In conclusion it is suggested that charge storage in vacuum deposited PVF films is governed dominantly by space charge effects where charge carriers are injected in to the polymer by surmounting the electrode-polymer interface barrier and get displaced at macroscopic distances and subsequently trapped at energy levels which are distributed in their activation energies and relaxation times and lie in the range 0.55 to 0.69 eV and 1.08×10^5 to 1.63×10^6 sec, respectively.

The above injection limited and charge trapping properties of PVF films indicate that these films may find possible applications as interface barrier layer in a-Se based XR photoreceptors for enhancing their X-ray sensitivity.

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