JOURNAL OF MATERIALS SCIENCE 27 (1992) 4316–4322

Application of thermally stimulated discharge techniques to a-Se: Te/Se double-layer photoreceptors

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Interface traps in a-Se:Te/Se double-layer xerographic photoreceptors were studied using thermally stimulated discharge (TSD) techniques. Two peaks are found in the TSD current curve which are believed to be due to two distinctly different types of space charge polarization. The well-defined low-temperature peak is centred at approximately $-8\,^{\circ}C$ and it is due to the difference in the dielectric constants and electrical conductivities of a-Se:Te and a-Se layers. It is argued that on the application of an electric field with positive polarity applied to the a-Se:Te layer, the Maxwell–Wagner effect causes the build up of a positive (holes) space charge layer at the interface of the two layers. The observed TSD current peak seems to be dominated by the neutralization currents in the a-Se:Te layer. The broad high-temperature peak occurs between 30 and 50 °C in the thermograms and it is believed to be due to release of holes from the deep traps at the interface between the two layers. The density of these traps is estimated to be $\sim 10^{10}\ cm^{-3}$. The activation energy relaxation corresponds to the effective energy for the detrapping of holes from the interface states and is calculated to be ~0.54 eV.

1. Introduction

In previous papers we described the principles of thermally stimulated discharge (TSD), relevant theories and the method of interpretation [1, 2]. the purpose of the present paper is to discuss application of TSD to amorphous semiconductors. It is well known that electrophotography is one of the outstanding applications of amorphous semiconductors. Amorphous selenium (a-Se) and its alloys are commonly used as photoreceptors in photocopy machines and laser printers [3, 4]. Electron and hole transport in these photoreceptors have been described previously [5–7]. Thermally stimulated discharge has been applied to a-Se before [8–10], but as far as we know, there is no published work on TSD in multilayer amorphous semiconductors.

For the multi-layer photoreceptors such as the popular a-Se:Te/Se double layers, due to the heterogeneity in the sample structure, several polarization effects could be observed when the sample is subjected to an electret formation cycle. The discontinuities in the intrinsic conductivities and dielectric constants within the photoreceptor structure can lead to the accumulation of space charges at the interfaces of the individual layers whenever the system experiences an electrical stress for a period of time which is of the order of its effective dielectric relaxation time. Charge trapping by states associated with the physical heterogeneities of the structures can also cause electrical polarization in the sample. A persistent internal polarization caused by space charges will have a significant impact on the xerographic performance of the photoreceptor.

It will be demonstrated that the application of TSD techniques to multi-layer photo-receptor structures is particularly useful because it is capable of revealing the density as well as the kinetics of the space charge polarization involved.

2. Experimental procedure

Preparation and characterization of the photoreceptor samples have already been described [5–7]. Samples approximately $2.5\ cm^2$ were evaporated with ~ 100 nm thick aluminium top electrodes with a guard-ring configuration, as shown in Fig. 1. After metalization of the top electrodes, the samples were mounted on to glass slides using silver dag (Fig. 1 inset) and then placed in a cryostat for the determination of the TSD current spectrum. The measurements

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were carried out at pressure of $\sim 1 \times 10^{-5}$ torr (1 torr = 133.322 Pa) and in the dark. Samples were normally dark rested in the cryostat for several hours prior to measurement.

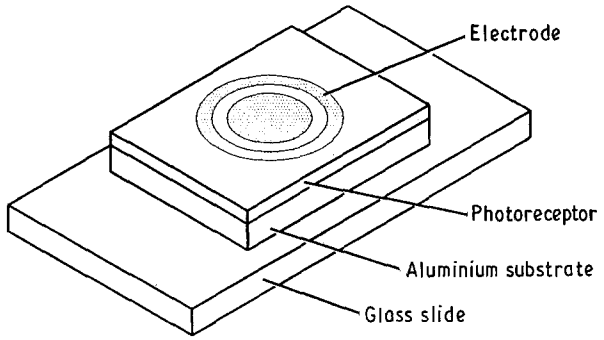
A schematic lay-out of the measurement system is shown in Fig. 2. The basic equipment consisted of a cryostat, where the sample and associated heating and cooling arrangements were housed, a sensitive current detector, a temperature controller and the associated power supplies for polarization of the films.

The detector used for current measurements was a Keithley 610C electrometer capable of detecting currents which may range from 10^{-14}–0.3 A. The simplified electrical circuit diagram is shown in Fig. 3. A protection resistor, R_p, of value 22 kΩ was connected in series in the charging branch of the circuit. A polarizing d.c. field was applied by using a regulated voltage supply (Fluke model 412B).

Usually currents in the range 10^{-12}–10^{-8} A may be produced after thermal stimulation of the polarized, short-circuited samples. No pertinent noise problems were encountered by using short, low-noise coaxial signal cables. The thermally stimulated currents were then recorded using a Bryans chart recorder (model 2700).

Virgin samples were used for each TSD run because the samples were inevitably destroyed after being heated to temperatures far beyond their glass transition temperatures during each run. After being poled at a temperature $T_p = 30.5\,^{\circ}C$ for a period $t_p = 30$ min, the sample was subsequently cooled to $-50\,^{\circ}C$ at a rate of about $-7.5\,^{\circ}C\,min^{-1}$ (with the applied voltage still on). The value of the poling voltage, V_a, was varied from -50 to $+50$ V, with respect to the polarity of the aluminium substrate, i.e. the electrode bordering the SeTe layer.

The heating rate used was $2\,^{\circ}C\,min^{-1}$, because this was found to yield reasonably large currents during the TSD. Higher heating rates were found to cause a high occurrence of sample failure during a run. The thin and brittle (~ 50 μm) photoreceptor layers had a tendency to flake away from the aluminium substrate (~ 0.5 cm thick) due to the different rates of thermal expansions of the photoreceptor layers and of the substrate.



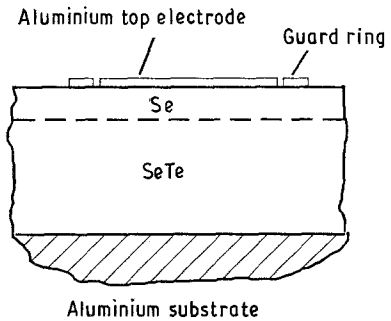


Figure 1 Sample configuration for a-Se:Te/Se double-layer photoreceptor.

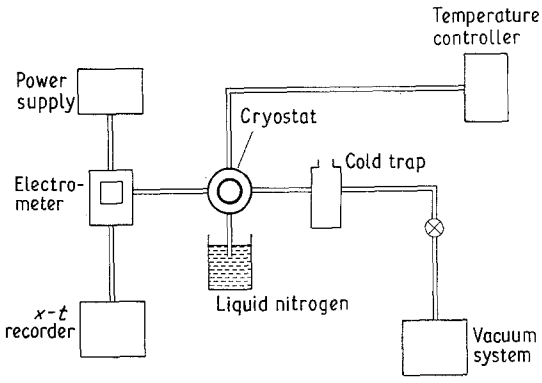


Figure 2 Schematic lay-out of the TSD apparatus.

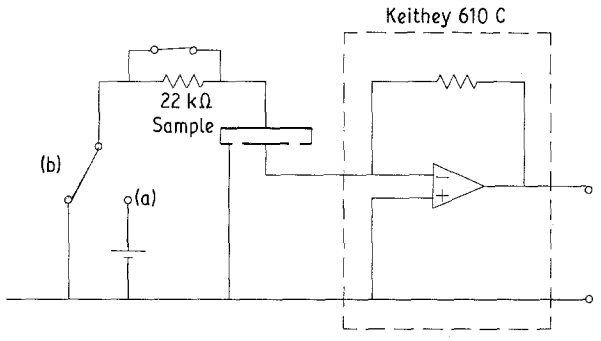


Figure 3 A simplified circuit diagram for current measurement; (a) charging, (b) discharging.

3. Results

The short-circuit TSD current spectra of the photoreceptor layers exhibited very different characteristics when the polarity of the forming electrode was reversed. To aid our analysis, this section will be divided into two sub-sections based on the polarity of V_a, the applied voltage.

3.1. V_a positive

The TSD current spectra for a series of positive poling voltages are shown in Fig. 4. From the plots, it could be observed that well-defined current peaks are present in the temperature regions of about $-8\,^{\circ}C$, followed by the appearance of several irregular TSD current peaks over a broad temperature region

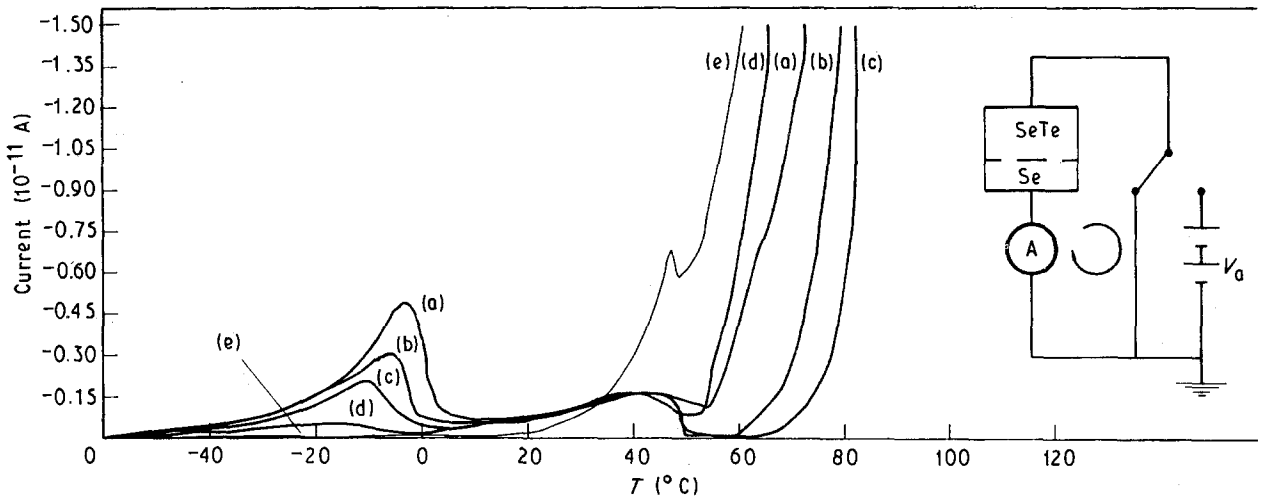


Figure 4 TSD current thermograms of an a-Se:Te/Se double-layer photoreceptor (T_p = 30.5 °C, r = 2 °C min^{-1}, t_p = 30 min). V(V): (a) 50, (b) 30, (c) 20, (d) 10, (e) 0.

between + 30 and + 50 °C. This latter temperature range incidentally also spanned the glass transition temperature of the a-Se and a-SeTe layers, as obtained from our DTA [11]. The low-temperature peaks will first be discussed followed by the high-temperature peaks.

Similar TSD runs were also carried out on samples with a homogeneous structure. The tellurium concentration of the single-layer a-Se-Te photoreceptor is shown in Fig. 5. A representative TSD current thermogram of the sample is shown Fig. 6. As can be seen, no current peaks are observed even though the samples were polarized under identical conditions. This shows that the observed current peaks in the thermograms of Fig. 4 are indeed due to the interfacial nature of the two-layer photoreceptors.

The low-temperature current peaks displayed an asymmetric shape which is reminiscent of dipole disorientation peaks undergoing a Debye relaxation. The plot of the maximum TSD current, I_m, versus the applied voltage is shown in Fig. 7. A linear relationship is found. The plot of the total charge released, Q, as a function of the applied voltage was also found to be linear. The TSD current peaks also exhibit a slight shift towards higher temperatures as the polarizing voltage is increased.

It was reported in the previous paper that a space charge polarization would occur in an electrically heterogeneous sample when an electric field is applied across its electrodes [1]. This Maxwell–Wagner polarization is caused by the accumulation of charge carriers at the boundaries or interfaces of the regions of the different media. Upon cooling and removal of the applied voltage, this layer of space charges would decay at a very slow rate due to the relatively high resistivities of the media at the low temperature. When the temperature is raised, the frozen-in charges will be gradually neutralized by the thermally generated carriers from the bulk of the media bordering the interface. It was shown previously that the decaying density of the frozen-in charge would respond to a linear temperature rise in a manner similar to that of a dipole depolarization [1]. This model for the decay of

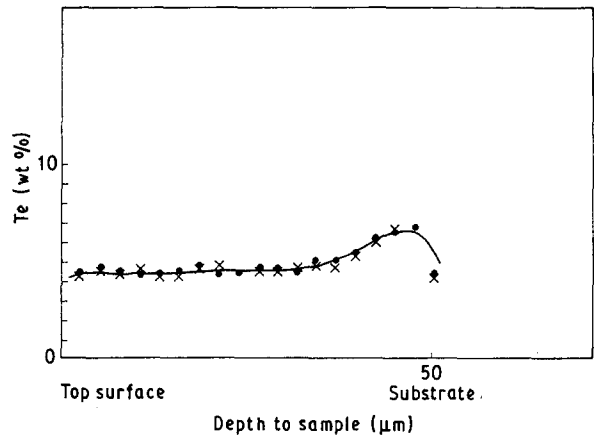


Figure 5 Tellurium concentration profile in the single-layer a-Se:Te/Se photoreceptor. (●) First run, (×) second run.

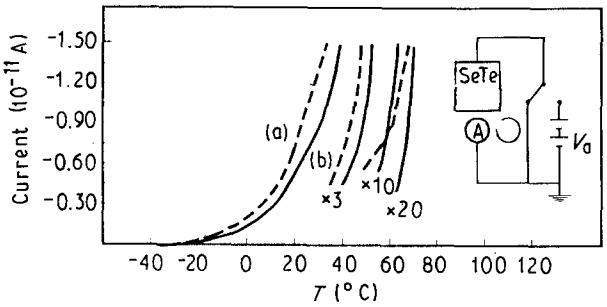


Figure 6 TSD current thermograms of a homogeneous single-layer a-Se:Te photoreceptor (T_p = 30.5 °C, r = 2 °C min^{-1}, t_p = 30 min). V_a(V): (a) 50, (b) 10.

the frozen-in interfacial charges is used for the analysis of the low-temperature peaks.

The sign as well as density of the charges stored at the interface, $Q(\infty)$, could be estimated by the use of Equation 23, in Part I [1]. The ratio $\varepsilon_1\delta(T)/\varepsilon\delta_1(T)$ in this equation plays an important role in determining the magnitude and sign of the charges. Using approximate room-temperature values for the electrical conductivities and static dielectric constants as $\delta(T) = \delta_{Se}(T) \simeq 10^{-11}\,\Omega^{-1}\,\text{cm}^{-1}$,

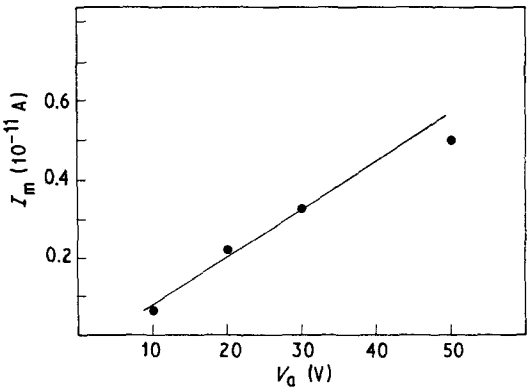


Figure 7 Peak current maxima as a function of the applied polarization voltage for the low-temperature Maxwell–Wagner peaks.

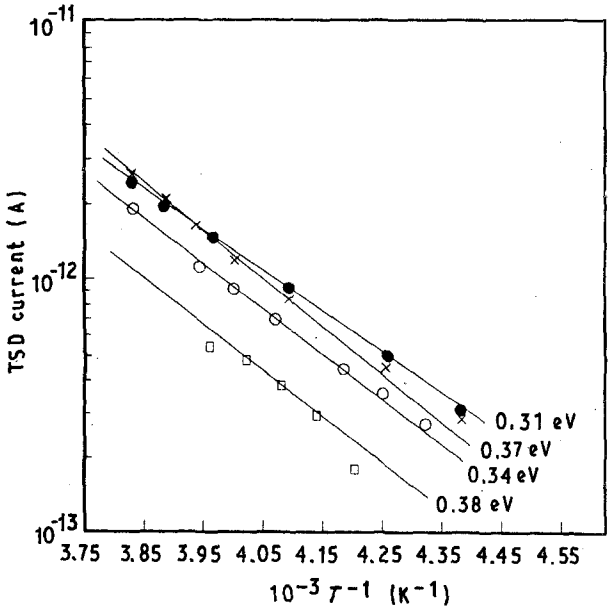


Figure 8 Initial rise plots of the low-temperature Maxwell–Wagner peaks. V_a(V): (×) 50, (●) 30, (○) 20, (□) 10.

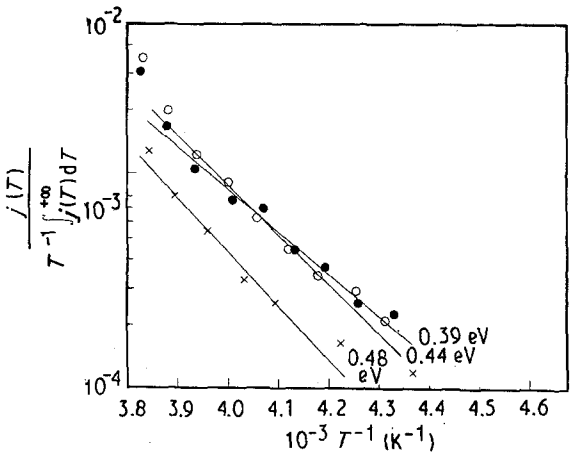


Figure 9 BFG plots of the Maxwell–Wagner peaks of the a-Se:Te/Se double-layer photoreceptors. V(V): (×) 50, (●) 30, (○) 20.

$\delta_1(T) = \delta_{SeTe}(T) \simeq 10^{-9}\,\Omega^{-1}\,cm^{-1}$, $\varepsilon = \varepsilon_{Se} = 6.8$, $\varepsilon_1 = \varepsilon_{SeTe} = 7.5$ [12, 13], the sign of $Q(\infty)$ is found to be positive (i.e. holes), for positive values of V_a. A comparison between the theoretical density of charges that could ultimately be stored for the different applied voltages and the actual magnitude of the charges released as found by a graphical integration of the TSD current peaks reveals that this quantity is consistently less than the former. Equation 31 in Part I [1] shows that the released TSD current is an algebraic sum of the two opposite currents in the respective a-Se and a-SeTe layers. In fact, using the values of the material constants that were noted previously, the external TSD current as described by this equation would be dominated by the component in the a-SeTe layer. Hence, the density of charges as determined by the graphical integration of the TSD current peaks could not have fully accounted for the total density of frozen-in charges.

Using the method of initial rise described in Part II [2], the apparent activation energy derived from the peaks centred at about − 8 °C is found to range from 0.31–0.38 eV. These plots are shown in Fig. 8. The BFG plots (named after Bucci, Fieschi and Guidi [13]), an example of which is shown in Fig. 9, also yield values ranging from 0.39–0.48 eV. The apparent energies of activation that were calculated from the TSD current peaks would actually be a hybrid value of the energies of activation for ohmic conduction in the a-SeTe and of the a-Se layers. However, because the electrical conductivity of the a-SeTe layer is much larger than that of the pure a-Se layer throughout the temperature range of the current peak, it can be argued that the calculated apparent activation energies would correspond to that of conduction in the a-SeTe layer. The values of the activation energy for low-field d.c. conduction in amorphous alloys of SeTe (10–30 wt % Te) lie in the range 0.40–0.80 [12, 14]. These values were determined via conventional current–voltage–temperature measurements.

As the samples were heated further, broad TSD current peaks were observed in the temperature range within which the peaks coincided with the glass transition regions of the a-Se and a-SeTe layers. This observation strongly suggests that these current peaks are closely related to the onset of major structural relaxations in the samples.

It is expected that due to the mismatch in the amorphous lattices of the a-Se and a-SeTe layers at the junction, defect states would be present. These states at the interface could act as traps for any mobile carriers traversing across the junction. During the polarization state of the TSD run these states are filled and remain so before being discharged.

As the temperature of the sample is raised, the individual rates of structural relaxations of the a-Se and a-SeTe layers would cause a dramatic change in the physical characteristics of the interface which leads to the release of the trapped carriers. The trapped carriers are "shaken out" of the interfacial states as the system re-adjusts to the changing physical stresses. The rate of release of the trapped carriers increases as the glass transition is approached.

The TSD current peaks also exhibited a saturation effect, i.e. the amount of released charge as found from the area underneath the peaks, was independent of the

magnitude of the charging voltage, V_a. This observation is believed to be due to the finite density of the interfacial traps that were filled during polarization. The graphical integration of the TSD peaks yields an average value of about 1.53×10^{-9} C cm^{-2} which corresponds to a density of 9.56×10^{9} cm^{-2} of released electronic charges.

At this point it is also interesting to consider a simple estimate for the density of interfacial states that may arise from the mismatch in the packing order of the atoms at the junction of the layers. Taking the bulk density of a-Se to be 4.3 g cm^{-3} [15] and that for a-SeTe with 10 at % Te as 4.4 g cm^{-3} [16], it can be easily shown that the number of atoms of Se or SeTe units in the solid would be about 3.28×10^{22} and 3.07×10^{22} cm^{-3}, respectively. Using $n_s = (n_b)^{2/3}$ for a uniform distribution of atoms in a cubic array, where n_b and n_s are the number of units in the bulk and in the surface, it can be shown that the values of n_s for Se and SeTe are 1.02×10^{15} and 0.98×10^{15} cm^{-2}. If the difference between these two quantities is taken as the surface density of available sites for the lattice defects, it may be argued that an upper limit of about 4×10^{13} cm^{-2} states would be present at the interface of the two layers. A comparison with the density of released charges obtained from the TSD current peaks shows that about 1 in 4.1×10^{3} interfacial states were actually filled when the sample was polarized under the experimental conditions used.

The apparent activation energy for the TSD peaks calculated from the initial rise plot in Fig. 10 is found to be 0.54 eV. This corresponds to the effective depth of the interfacial states of the junction. It should be noted that activation energy of structural relaxations associated with the glass transition of homogeneous single-layer a-Se is of the order of 2.50 eV [11, 17] and of a-SeTe:Cl (5.5 wt% Te, p.p.m. Cl) is about 2.26 eV [11]. By relating the values of the activation energies for the structural relaxations with that of the activation energy of the TSD relaxation current, it can be said that a "full-blown" structural re-adjustment (involving the breaking of bonds and also micro-Brownian motions) would not be required in releasing the trapped carriers.

The broadness of the peaks also suggests that the relaxation is a distributed one. Unfortunately, our attempts to resolve the peaks following the "peak cleaning" principle [18] did not result in clear, well-defined sub-peaks from which further inference could be made. This experimental difficulty is believed to be directly attributable to the heterogeneous nature of the sample. Repeated sequences of heating and fast cooling as required by the technique can also cause irreversible changes to the state of the still frozen-in polarization, making the results of subsequent TSD runs uncharacteristic of the total polarization state that would have been revealed. However, it is clear that from the very broad peaks observed and their close proximity to the glass transition regions of the individual layers, the actual relaxation involved would be governed by some form of cooperative behaviour.

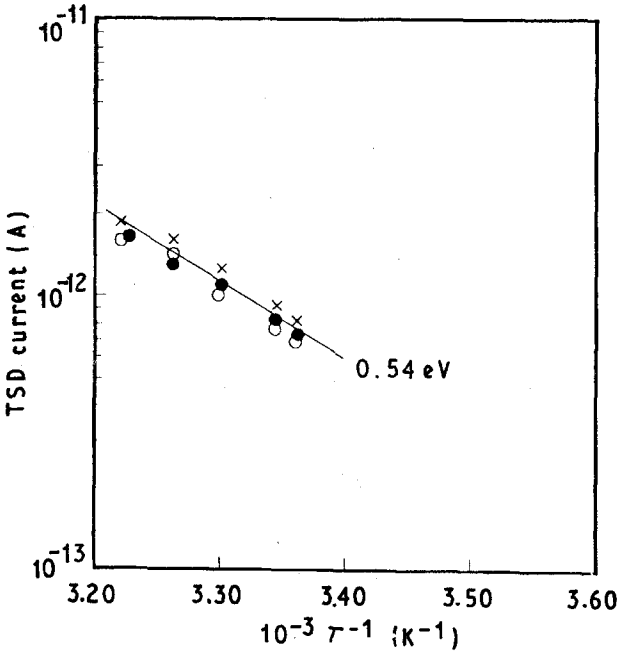


Figure 10 Initial rise plot of the broad detrapping peak of the a-Se:Te/Se double-layer photoreceptors. V_a(V): (×) 50, (●) 30, (○) 20.

3.2. V_a negative

The TSD current thermograms for negative applied voltages are shown in Fig. 11. No clear or well-defined peaks that could be attributed to a Maxwell–Wagner mechanism could be observed. Instead, several small and broad peaks are present followed by current reversals as the T_g region was approached. Only a qualitative discussion of these peaks is possible, as attempts to increase the magnitudes of the peaks by using larger poling voltages or higher heating rates resulted in frequent sample failure. The use of longer polarization time also did not significantly affect the final results. It is clear that the use of Equation 31 in Part I, is not adequate for describing the observed TSD behaviour [1]. It appears that a space charge polarization due to the Maxwell–Wagner effect was not successfully achieved when the polarity of the forming electrode was reversed. It must be stated that the successful formation of a space charge layer at the interface of two media depends on whether the incoming conduction current is greater or smaller than the outgoing one. It has been established that the d.c.

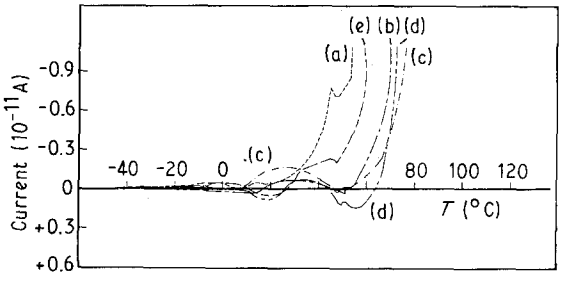


Figure 11 TSD current thermograms of a-Se:Te/Se double layers for negative applied polarization voltages ($T_p = 30.5$ °C, $r = 2$ °C min^{-1}, $t_p = 30$ min). V_a(V): (a) 0, (b) − 10, (c) − 20, (d) − 30, (e) − 50.

conductivity of certain alloys of a-SeTe can be about 10^2 times higher than that of a-Se [12, 19]. It has also been found from thermo-electric measurements that a predominantly p-type conduction is exhibited by a-Se [20, 21]. The isoelectronic addition of tellurium would not affect this property.

For positive values of V_a, the requirement that is outlined above was easily satisfied. This has resulted in holes being immobilized at the interface of the layers. For negative values of V_a, however, the outgoing hole current was greater than the incoming current. This has resulted in an insignificant density of charges being immobilized with the final consequence of small current peaks being released during the TSD run. It is also believed that the inter-play of the small neutralization currents and the ubiquitous parasitic background current was responsible for the current reversals detected.

4. Discussion

We have shown that the short-circuit TSD current behaviour of a d.c. polarized a-SeTe/a-Se double-layer photoreceptor is dependent on the polarity of the forming electrodes. For negative polarities of the aluminium substrate, no distinct TSD current peaks which could be attributed to the neutralization of a layer of previously frozen-in interfacial space charges were observed. However, distinct TSD current peaks were observed when positive charging voltages were employed.

Two current peaks, directly attributable to the presence of a heterogeneity in the samples, were observed. The low-temperature peak, centred at about $-8\,^{\circ}C$, is essentially a dielectric relaxation peak. This is due to the response of the intrinsic electrical conductivities of the component layers of the laminate to the linear temperature rise. The high-temperature peak, which spanned a broad temperature range between $+30$ and $+50\,^{\circ}C$, is believed to be due to the release of trapped carriers from states associated with the mismatch in the amorphous lattices of the layers.

From the direction of the released TSD currents and by the use of the Maxwell–Wagner model to describe the isothermal charging of the a-SeTe/a-Se junction, it is believed that a layer of positive space charges (holes) were accumulated when the forming electrode was made positive. During the subsequent TSD, thermally generated carriers (electrons) from both sides of the interface were driven to the interface where they neutralized the immobilized holes. From a consideration of the relative magnitudes of the d.c. conductivities and the dielectric constants of the individual layers, it is also concluded that the TSD neutralization current that was externally detected would be dominated by the component in the a-SeTe layer.

The decay of the space charge layer at the interface is caused by intrinsic conduction rather than by the redistribution, i.e. self-drift of the charges. Several experimental observations consolidate this view. The positions of the TSD current peaks did not shift towards lower temperatures as the polarizing field was increased. For a decay via self-drift of the charges, it is expected that a lowering of T_m, the peak temperature, would be observed due to the speeding up of the decay caused by the higher driving field. The "charge-ratio" plots of the TSD current peaks were also found to be non-linear. A linear plot would be characteristic of the decay of the space charges via a self-drift. The average relaxation time of the decay determined from the TSD current peaks were of the order of 10^3–10^4 s. If the decay of the interfacial charges was to occur via a drift towards the electrodes, and the times quoted above were to represent the average time taken for the majority of the carriers to reach the electrodes, then unrealistically low values for the effective drift mobilities of the carriers would result. Using the simple relation for the drift mobility $L^2/T_t V$, where L is the distance to be traversed, T_t is the transit time and V is the driving voltage (taken to be ~ 50 V), then the effective drift mobility, μ, would be of the order of 10^{-12}–10^{-11} $cm^2\,V^{-1}\,s^{-1}$. These values would be too low compared to known values of effective drift mobilities for holes or electronic a-Se or a-SeTe alloys [5–7].

The apparent activation energy for the relaxation was about 0.38–0.48 eV. This would correspond to the activation energy for the conduction of intrinsic, thermally generated electronics in the a-SeTe layer. The values calculated from the TSD current thermograms are of the same order as those that were determined via more conventional methods.

The magnitude of the charge ultimately recovered is found to be less than the theoretical value of charges that were frozen-in at the interface. This experimental result is in agreement with the general TSD behaviour of heterogeneous physical systems involving space charges whereby the so-called Gross charge invariance principle is violated [22, 23]. Only in the unlikely case where the a-Se layer is fully insulating would the principle be obeyed and the magnitude of the theoretically stored charges be fully recovered [1].

Broad current peaks observed in the T_g regions of the layers are believed to be due to the emptying of filled traps. It is assumed that defect states at the interface could act as traps for the mobile carriers. These defect states probably arise from the misfits and stresses of the a-SeTe and a-Se lattices at their contact interface and as such, the occupancies and the actual densities of these states can dramatically change when the glass transition is approached. The density of the defect states, found from the TSD current peaks, is about 9.56×10^9 cm^{-2}.

It is found that the emptying of the defect states is a distributed process as evinced by the broad TSD current peaks. This is due to the close dependence of the kinetics of the trapping states to the structural relaxations of the layers in the sample at the glass transition. Assuming a simple thermally activated behaviour for the relaxation, an apparent activation energy of 0.54 eV was found. This value would represent the effective energy required for the release of trapped holes from the interface states.

5. Conclusions

The impact of a Maxwell–Wagner polarization on the

overall transport of the excess, photogenerated charges through a heterogeneous xerographic photoreceptor is dependent on the time delay between sensitization of the photoreceptor (corona charging) and the photo-discharge. This delay is usually of the order of seconds or less [3]. The time needed for the build-up of the equilibrium charges at the interface is essentially determined by the effective ohmic relaxation time of the sample which, for a typical two-layer structure, would be of the order of several minutes [2].

Thus it can be said that a Maxwell-Wagner polarization will not be a significant problem to the xerographic performance of the photoreceptor as long as the sensitization/photo-discharge delay is short compared to the effective relaxation time of the heterogeneous structure. However, it should be noted that the relaxation time is a temperature dependent quantity, being considerably shorter if the temperature is increased. This effect may become significant if high-temperature working environments are encountered.

Time-of-flight experiments on multi-layer photoreceptors do not show any effects that may be attributed to the presence of a space charge layer at the interfacial regions of the samples [24]. The time delay between the application of the applied driving field and the photoexcitation was of the order of 10^{-6} s. Therefore, the times involved in these experiments are too short to cause any significant build-up of a Maxwell–Wagner effect.

It is believed that the deep interface states that were revealed by the TSD results will have a significant effect on the long-term cyclic property of the photoreceptor. A build-up of trapped charges at the interface of the layers of the heterogeneous structure will lead to undesirably high values of the so-called residual potential which, in turn, will cause a significant degradation to its xerographic performance [24].

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Received 9 May
and accepted 20 September 1991