Aging Effects on the Interface State Density Obtained from Current–Voltage and Capacitance–Frequency Characteristics of Polypyrrole/p-Si/Al Structure

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ABSTRACT: A metallic polypyrrole film has been directly formed on a *p*-type Si substrate by means of an anodization method under conditions of constant current density by an electrolyte being held at a constant temperature of 55°C that was composed of 0.40*M* pyrrole and 0.10*M* tetrabutylammonium tetrafluoroborate. An aluminum electrode was used as an ohmic contact. The polypyrrole/*p*-Si/Al structure has clearly demonstrated rectifying behavior by the current–voltage curves studied at room temperature. The capacitance–voltage–frequency curves of the structure have been measured at different frequencies at room temperature in dark. To observe the effect of the aging, the measurements were also repeated 7, 15, 30, 60, and 90 days after fabrication of the polypyrrole/*p*-Si/Al Schottky diode. During the whole measurement process, the probe of holder on sample was fixed to eliminate effects of surface inhomogeneity. The interface state density distribution curves of device have been obtained from forward bias current–voltage (*I-V*) and capacitance–frequency (*C-f*) characteristics as a function of aging time. From these curves, the exponential growth of the interface-state density from midgap toward the top of the valence band is very apparent. The shape of the interface state density distribution curves from the forward bias *I-V* characteristics are very similar to those obtained from *C-f*. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 2313–2319, 2006

Key words: aging; interface state density; Schottky diode; polypyrrole; series resistance

INTRODUCTION

Conducting polymers have potential applications at all levels of microelectronics. During the past two decades, conjugated polymers such as polypyrrole, polyaniline, and polythiophene have been studied extensively because of their improved electronic properties and easy processability.[1–3] Polypyrrole is by far the most extensively studied conducting polymer since monomer pyrrole is easily oxidized, water-soluble, commercially available, and possesses environmental stability, good redox properties, and high electrical conductivity.[4–7] Because of its good intrinsic properties, polypyrrole appears promising for use in batteries, super capacitors, electrochemical (bio) sensors, conductive textiles and fabrics, mechanical actuators, electromagnetic interference shielding, antistatic coating, and drug delivery systems.[8]

As mentioned in references 9–13, Schottky barriers based on semiconducting polymer and metal contact as photovoltaic energy converters and, in particular, the electrical properties of heterojunctions and Schottky barriers fabricated from thin films of polyacetylene, polythiophene, polyparaphenylene, polyaniline, polypyrrole, and poly (*N*-methylpyrrole) have been investigated. The device parameters of these rectifying structures were generally far from ideal diode characteristics. The rectification characteristics of Schottky junctions between polypyrrole and Al, Au, in metals have been reported by different authors.[14,15] Vermeir et al.[16] formed electrodeposited polypyrrole films on chemically modified hydrogen-terminated silicon surfaces that exposed tethered pyrrole units and found that the junctions exhibited diode-like characteristics. The change with aging time of characteristic parameters of polypyrrole and polyaniline/inorganic semiconductor structures have been investigated by Saglam et al.[17,18] and it has been seen that the characteristic parameters obtained from current–voltage and capacitance–voltage measurements such as barrier height, ideality factor, and series resistance of polypyrrole/*p*-Si and polyaniline/*p*-Si structures have changed with increasing aging time. The characterization of current–voltage features of Sn/polypyrrole/*n*-Si structure as a function of temperature has been made by Aydogan et al.[19] and temperature dependences of the ideality factors and barrier heights of diodes have been analyzed in the light of the inhomogeneity model.

The present article focuses on the effects of the time-dependence of the interface state density ob-

tained from current–voltage and capacitance–frequency characteristics of polypyrrole/p-Si/Al structure since stability of the interfaces of metal-semiconductor and polymer-semiconductor structures in the area of electronics devices are important. We report here how interface state density of polypyrrole/p-type Si/Al Schottky diode has changed with increasing aging time. In the present work, we have used polypyrrole for the preparation of the polypyrrole/p-Si/Al diode. The current–voltage and capacitance–frequency measurements of the diode have been carried out under laboratory conditions in dark at room temperature.

EXPERIMENTAL

The samples were prepared using mirror cleaned and polished (as received from the manufacturer) p-type Si wafers (100) orientation and 15–20 Ω cm resistivity. The wafer was chemically cleaned using the RCA cleaning procedure.[20] The native oxide on the front surface of p-Si was removed in a HF:H$_2$O (1:15) solution for 30 s and finally the wafer was rinsed in deionized water for 30 s. Then, low resistivity ohmic back contact to p-type Si (100) wafers was made by evaporating Al, followed by a temperature treatment at 600°C for 3 min in a N$_2$ atmosphere. The ohmic contact made and the edges of the p-Si substrate used as an anode were carefully covered by wax so that the polished and cleaned front side of the sample was exposed to the electrolyte by mounting it in an experimental set-up employed for polymerization. A Pt plate was used as the cathode. Polymerization process was carried out under constant current density conditions of J = 31.85 mA/cm^2 at about 1 min. The polypyrrole/p-Si diodes were fabricated by an electrolyte being held at a constant temperature of 55°C that was composed of 0.40M pyrrole and 0.1M tetrabutylammoniume tetrafluoroborate. The electrolyte solution was prepared in a propylene carbonate solvent (Merck). The diode parameters extracted from the current–voltage (i.e., the saturation current density, the rectification ratio, ideality factor, and barrier height) and capacitance–voltage characteristics (i.e., the built-in voltage and the charge carrier concentration) are strongly influenced by the nature of the dopants, by the thickness, and by the preparation temperature of the polypyrrole layer. The increase of preparation temperature of the polypyrrole layer, from 278 to 323 K, improves significantly the diode behavior. Results from the current–voltage measurements at different temperature indicated that the junction of the diodes primarily controls the current–voltage characteristics. The rectification ratio increases rapidly with the increase of the preparation temperature, and simultaneously, the saturation current density decreases largely. This may be due to the enhancement of the defect concentration in polypyrrole with increasing preparation temperature, which decreases the so-called active acceptor concentration, and hence, enhances the depletion width of junctions. This assumption is supported by results of the complex impedance measurements.[21] Therefore, when prepare the polypyrrole/p-Si Schottky diodes, the electrolyte temperature being held at a 55°C. The thickness of polypyrrole layer was calculated as 200 nm approximately from high frequency C-V measurements.

The current–voltage and capacitance–frequency characteristics of device were measured using a HP 4140B picoampermeter and a HP model 4192A LF impedance analyzer, respectively, at room temperature in the dark. Moreover, to observe the effect of the aging, the measurements were also repeated 7, 15, 30, 60, and 90 days after fabrication of the polypyrrole/p-Si contact. During the whole measurement process, the probe of holder on sample was fixed to eliminate effects of surface inhomogeneity. Similarly, Polypyrrole/p-Si Schottky diode was stored in atmosphere at clean room air for 90 days at room temperature. An average humidity of % 50 measured by a Lambrecht model hygrometer and an average temperature of 25°C characterize the clean room air.

RESULTS AND DISCUSSION

When the Schottky barrier diodes with a thin interfacial layer are considered, it is assumed that the forward bias current of the device is due to thermionic emission current.[22] The insulating interfacial layer disconnects the interface states from the metal, making them communicate with the semiconductor more readily than with the metal. The insulating interfacial layer lowers the background current and facilitates the observation of the signal signatures arising from these states.[23] The energy band diagram of the polymer/p-Si Schottky diode with a thin interfacial layer and interface states has been shown in Figure 1. The current (I)–voltage (V) equation in respect to the thermoionic emission theory in the presence of interfacial layer is given by[24]

$$I = AA^*T^2 \exp\left(\frac{-q\Phi_b}{kT}\right)\left[\exp\left(\frac{q(V-IR_s)}{nkT}\right)-1\right] \quad (1)$$

where A is the effective area of diode; A^* is the effective Richardson constant; Φ_b is the barrier height; T is the temperature; q is the elementary charge; k is Boltzmann constant; R_s is the series resistance, and n is the ideality factor. The saturation current, I_o, may be denoted by

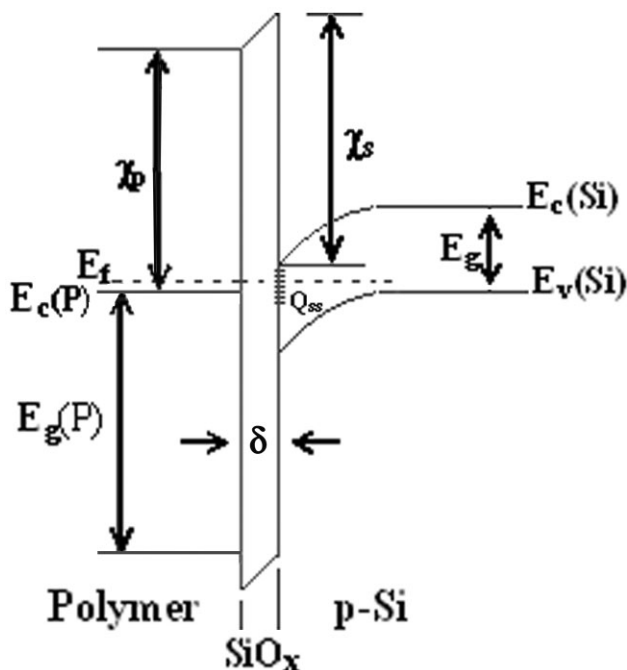$$I_0 = AA^*T^2\exp\left(\frac{-q\Phi_b}{kT}\right) \quad (2)$$

Figure 1 The energy band diagram of the polymer/p-Si Schottky diode with a thin interfacial layer.

Figure 2 The forward and reverse bias current–voltage characteristics for immediately and 90 days measured data of polypyrrole/p-Si Schottky diode.

and is obtained by extrapolation of the forward or reverse bias I-V curve to zero applied voltage. The slope of linear portion of the I-V curve gives the ideality factor, which means that the deviation from the ideal I-V characteristics can be due to the presence of an indefinable interfacial layer that introduces the interface states located at polypyrrole/p-Si interface. Such a layer may be formed during the surface preparation.

Figure 2 shows the forward and reverse bias current–voltage (I-V) curves measured immediately and 90 days after fabrication of the polypyrrole/p-Si/Al structure. The values of the parameters obtained from these characteristics are given in Table I. The values of the barrier height of the device were calculated from the y-axis intercept of the semilog-forward bias I-V characteristics according to eq. (2). The values of the ideality factor n were calculated from the slope of the linear region of the forward bias I-V characteristics according to eq. (1). As can be seen in Figure 2, the forward bias I-V curve of the polypyrrole/p-Si Schottky diode deviates from linearity above approximately 0.35 V. The curvature downward in the forward bias I-V plot at sufficiently large applied voltage is due to the substrate series resistance and to the continuum of the interface states.[25] As can be seen Table I, the values of the ideality factor n obtained from Figure 2 have slowly decreased from 2.00 to 1.93 with increasing aging time. From the value of n, we deduce that the device represent polypyrrole-interfacial layer-semiconductor configuration rather than an ideal diode.
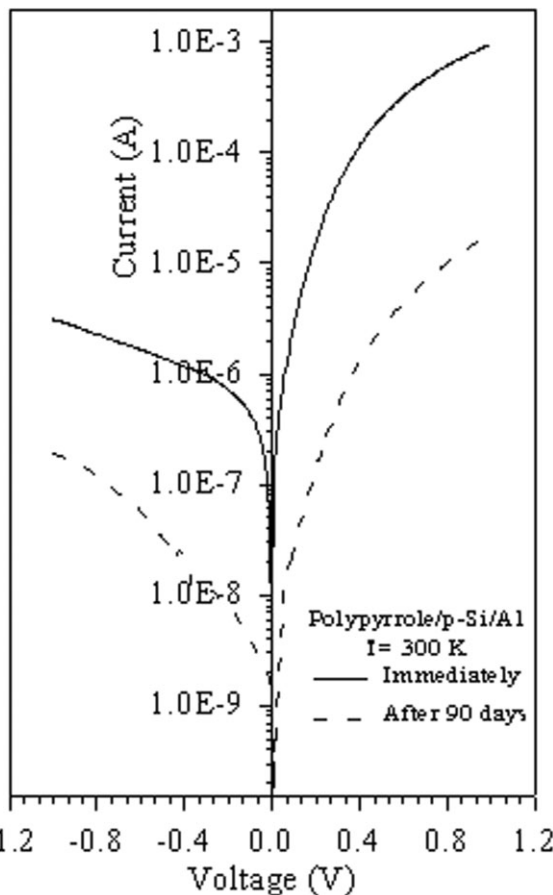
Having n values significantly greater than unity is a result of the interface states and interfacial layer in the polypyrrole/p-Si interface.[26,27] In this kind of device, since the front surface of p-Si is exposed to air before forming the polypyrrole, there is probably an insulating oxide layer between the polymer and p-Si substrate. In the case of Schottky barrier devices, smaller n values imply a lower density of the interface states.[27] Again, the values of the barrier height of the device obtained from the forward bias I-V characteristics in Figure 2 have increased from 0.68 eV to 0.80 eV with increasing aging time. This increase in the values of

TABLE I
The Experimental Values of Parameters Obtained from Current–Voltage and Capacitance–Voltage Characteristics of the Polypyrrole/p-Si/Al Schottky Diode with Increasing Aging Time

		I-V		C-V
Aging time ↓	n	Φ_b (eV)	Rs (Ω)	Φ_b (eV)
Immediately	2.00	0.68	500	0.67
90 Days	1.93	0.80	16500	0.78

the barrier height with increasing aging time may be attribute increase in the series resistance values of polypyrrole/p-Si/Al Schottky diode. Both barrier height and series resistance have been increased with increasing aging time. While Rs increases with time, current transport across polypyrrole/p-Si interface as a result of the increasing voltage drop across the polypyrrole layer decreases (see Fig. 2) with respect to same voltage value. Therefore, it seems that the barrier height increases because of the decreasing of the current. The series resistance values of polypyrrole/p-Si/Al structure have been calculated using Cheung's functions with increasing aging time. The values of the series resistance of the device obtained from the forward bias I-V characteristics in Figure 2 have increased from 500 ohm to 16,500 ohm with increasing aging time. The reason of this increase is that the doping agent in polypyrrole is gradually replaced by oxygen, which leads to a slow decrease of its conductivity. The presence of the interfacial layer in the diodes causes the effective barrier height to decrease or the reverse current to increase with increasing reverse bias as a result of the increasing voltage drop across the interfacial layer. Furthermore, the Fermi level for the majority carriers rises on the semiconductor side when the diode is biased in the forward direction. Most of these carriers will be injected directly into the metal, forming a thermionic emission current, while some of them are trapped by the interface states. This charge-capture process results in an increase in the effective barrier height, thereby reducing the diode current.[22] Again, as can be seen in Table I, the values of barrier height of the polypyrrole/p-Si/Al Schottky diode calculated using reverse bias capacitance–voltage characteristics have increased from 0.67 eV to 0.78 eV with increasing aging time. It is seen that there is a good agreement between the values of the barrier height obtained from I-V and C-V characteristics. The reverse bias capacitance–voltage characteristics of the polypyrrole/p-Si/Al Schottky diode have been not shown here.

The properties of the interface states can be studied using a variety of techniques involving the capture or the emission of charge carriers, such as current–voltage and capacitance–voltage measurements, deep-level transient spectroscopy, Schottky capacitance spectroscopy, and admittance spectroscopy.[28–30] The interface states can be examined in two subgroups. At first, they are in equilibrium with the metal and, in the second situation, with the semiconductor. In general, the interface states in equilibrium with the semiconductor do not contribute to the capacitance at sufficiently high frequencies because the charge at the interface states cannot follow the ac signal. In this case, the Schottky barrier diode is the space-charge capacitance only. At low frequencies, the contribution of the interface states to diode capacitance decreases with
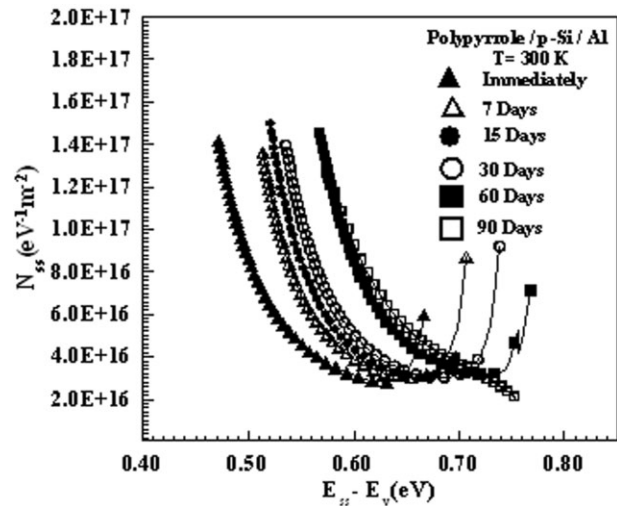
Figure 3 The energy distribution curves of the interface states obtained from the forward bias I-V characteristics with increasing aging time.

increasing frequency.[31] Experimentally measured capacitance corresponding to low frequency in the data obtained from the capacitance–frequency measurements is approximately equal to the sum of the space-charge capacitance (C_{sc}) and the interface capacitance (C_{ss}).[30]

The downward concave curvature of the forward bias I-V plots at sufficiently large voltages has been attributed to the presence of the interface states which are in equilibrium with the semiconductor, apart from the effect of R_s.[25] In this region, the ideality factor n is rather controlled by the interface states and the series resistance. The curves of the interface state energy distribution or density distribution can thus be determined from data of this region for the forward bias I-V in Figure 2.[32] The curves obtained by taking into account the bias dependence of the ideality factor and barrier height are given in Figure 3. As can be seen in Figure 3, the exponential growth of the interface-state density from midgap toward the top of the valence band is very apparent. The curves of the interface state energy distribution or density distribution have shifted toward midgap with increasing aging time. We think that this is due to the increase in series resistance from approximately 500 to 16,500 ohms. The interface state density we have determined from the forward bias I-V is of the order of magnitude of 10^{16}/m^{2}eV.

The capacitance of the devices depending on frequency is given as follows[33,34]:

$$C = C_{sc} + C_{ss} \text{ (at low frequency)} \qquad (3)$$

$$C \cong C_{sc} \text{ (at high frequency)} \qquad (4)$$
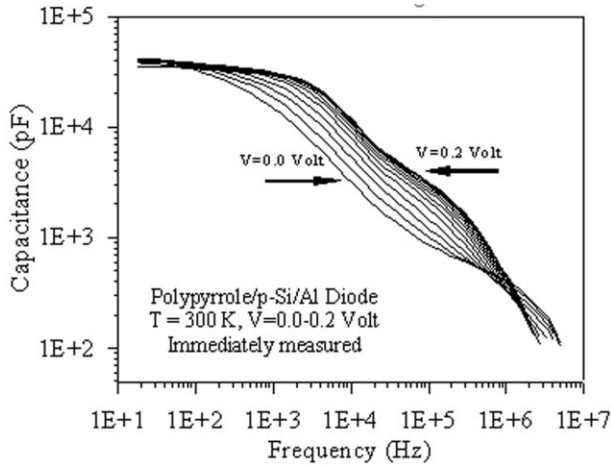
Figure 4 Experimental *C-f* curves for immediately measured data of polypyrrole/*p*-Si/Al Schottky diode as a function of applied forward bias.

According to Nicollian and Goetzberger,[35] the interface state capacitance can be described as:

$$C_{ss} = AqN_{ss}\frac{\arctan(\omega\tau)}{\omega\tau} \tag{5}$$

where τ is time constant and can be written as

$$\tau = \frac{1}{v_{th}\sigma N_d}\exp\left(\frac{qV_d}{kT}\right) \tag{6}$$

where σ is the cross section of interface states, v_{th} the thermal velocity of carrier, and N_d the doping concentration. The interface density for small values of $\omega\tau$ equals to[33–35]

$$N_{ss} = \frac{C_{ss}}{qA} \tag{7}$$

where A is the diode area. The interface state capacitance C_{ss} is determined from the vertical axis intercept of $C_{ss} - f$ plots.

In *p*-type semiconductors, the energy of the interface states E_{ss} with respect to the top of the valence band at the surface of the semiconductor is given by[34]

$$E_{ss} - E_v = E_f + q\Psi_s \tag{8}$$

where E_f is Fermi level, q is the electron charge, and Ψ_s is the surface potential.
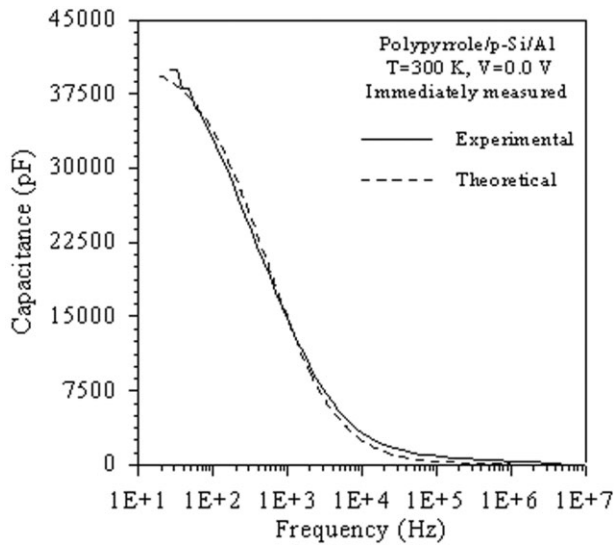
Figure 4 shows the immediately measured capacitance with steps of 0.02 V as a function of the frequency with bias voltage as a parameter. From this figure, we can observe that in the frequency range 20–3 × 10³ Hz, the capacitance is weakly dependent on the frequency. When the frequency applied is in-

creased to $>10^3$ Hz, the capacitance shows strong frequency dependence and tends to decrease more rapidly. Generally, the capacitance measured for a Schottky diode is dependent on the reverse bias voltage and frequency. The voltage and frequency dependence is due to the particular features of a Schottky barrier, impurity level, high series resistance, etc. At low frequency, the capacitance measured is dominated by the depletion capacitance of the Schottky diode, which is bias-dependent and frequency-independent. As the frequency is increased, the total diode capacitance is affected not only by the depletion capacitance, but also by the bulk resistance and the dispersion capacitance, which is frequency-dependent and associated with hole emission from slowly responding deep impurity levels.[36] Because of these effects, the capacitance dependence on bias becomes less pronounced or disappears.

Figure 5 shows the *C-f* plots for the polypyrrole/*p*-Si/Al structure at zero bias voltage as a function of aging time. As seen in Figure 5, there are three regions in this graph. This case can be explained by whether the interface state charges contribute to the diode capacitance or the charge at the interface states can follow an alternating-current signal. Usually, at the interfacial layer and semiconductor interface, there are various kinds of states with different lifetimes. At low frequencies, all the interface states affected by the applied signal are able to give up and accept charges in response to this signal. The interface state capacitance appears directly in parallel with the depletion capacitance, and this result in a higher total value of the capacitance for Schottky diodes than if no interface states were present. At intermediate frequencies, some, but not all, of the interface state charge will participate in small signal measurements, and values of the capacitance observed will be between the low- and high-frequency values. If the capacitance measurements are made at sufficiently high frequencies,
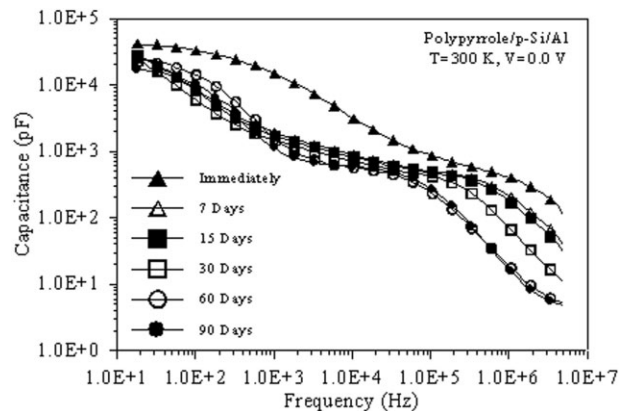
Figure 5 Experimental *C-f* curves of polypyrrole/*p*-Si/Al Schottky diode with increasing aging time at V = 0.0 Volt.

Figure 6 The experimental and theoretical forward bias interface state capacitance plot as a function of the frequency at bias voltage of 0.0 V for immediately measured date of polypyrrole/p-Si/Al Schottky diode.

the interface state charges do not contribute to the diode capacitance. This will occur when the time constant is too long to permit the charge to move in and out of the interface states in response to an applied signal.[37]

At a given forward bias, the value of the space-charge capacitance in high frequency region of the experimentally obtained forward bias C-f plots are subtracted from the experimental junction capacitance C, thus the interface states capacitance C_{ss} is obtained, then $C_{ss} - f$ plots for the diodes. Figure 6 shows the theoretic and experimentally immediately measured C-f plots for the polypyrrole/p-Si/Al Schottky diode at zero bias voltage. In the plateau region, eq. (7) is applicable and the value of the interface state density N_{ss} for each applied bias voltage can be deduced directly from the ordinate of the plateau. Then, we fitted eq. (5) to the experimental values $C_{ss} - f$ to obtain relaxation time of the interface state density τ (the dashed lines in Fig. 6). Based on our results, there is a good agreement between experimental and theory. To obtain the dependence of N_{ss} and τ on the bias, the fitting procedure was repeated for various values of the bias voltage as in Figure 6. The dependence of N_{ss} on the bias was converted to a function of E_{ss} using eq. (8) and this plot is shown in Figure 7.

As can be seen in Figure 7, the exponential growth of the interface-state density from midgap toward the top of the valence band is very apparent. The curves of the interface state energy distribution or density distribution have shifted toward midgap with increasing aging time. The interface state density we have determined from the C-f is of the order of magnitude of 10^{14}/m^2eV. The shape of the interface state density

distribution curves from the forward bias I-V characteristics in Figure 3 are very similar to those obtained from C-f. When the interface state density distribution curves from the C-f characteristics in Figure 7 are compared with those from the forward bias I-V characteristics in Figure 3, it is seen that the interface states density from the C-f characteristics is far smaller than that determined from the forward bias I-V characteristics; it is almost two orders of magnitude smaller. As explained by Victorovitch et al.,[38] the large difference between the interface state density values from the C-f and I-V characteristics is caused by the local trap-density effect at the oxide-semiconductor interface. The traps are essentially concentrated across the current parts; I-V curves provide an estimate of the local density of traps in the areas crossed by the current. Therefore, the average density of traps derived from admittance measurements can be significantly lower than the local density[38] (by about two orders of magnitude for our own experimental values).

As can be seen in Figures 3 and 7, the interface state density distribution curves obtained from I-V and C-f characteristics have been showed exponential rise toward the top of the valence band in the band gap. Again, the interface state density distribution curves in Figures 3 and 7 have been shifted toward midgap with increasing aging time. The reason of this shift is that the doping agent in polypyrrole is gradually replaced by oxygen, which leads to a slow decrease of its conductivity.[17] This would explain the dramatic increase of the series resistance (from 500 Ω to 16,500 Ω) of the diode. The interface state density distribution curves will be shifted toward midgap because of increasing series resistance.
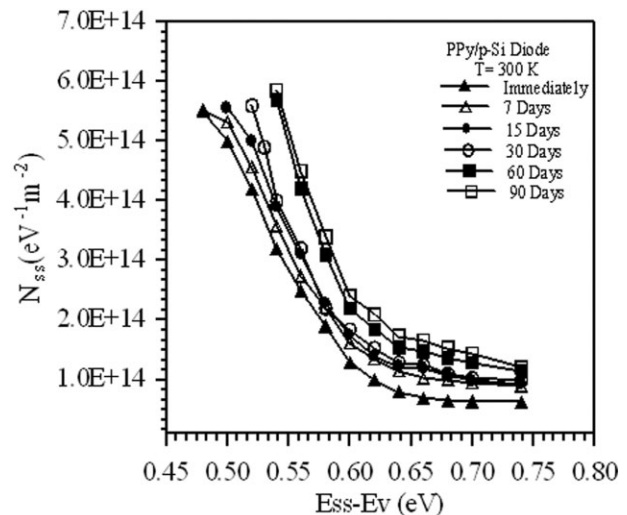
Figure 7 The energy distribution curves of the interface states obtained from the C-f characteristics with increasing aging time.

CONCLUSIONS

The energy distribution of the interface states of the polypyrrole/p-Si/Al Schottky diode have been determined from the I-V and C-f characteristics as a function of increasing aging time. It has been seen that the shape of the interface state density distribution curves from the forward bias I-V characteristics are very similar to those obtained from C-f. At the same time, it is seen that the interface states density from the C-f characteristics is far smaller than that determined from the forward bias I-V characteristics; it is almost two orders of magnitude smaller. This difference between the interface state density values from the C-f and I-V characteristics is caused by the local trap-density, which affects the areas crossed by the current at the oxide-semiconductor interface. Again, the density of interface states distribution profile as a function of $E_{ss} - E_v$ (from I-V and C-f) has been shifted toward midgap with increasing aging time. This cause has been attributed to the increasing of series resistance of polypyrrole/p-Si/Al structure with increasing aging time. Most of the aging effect has been attributed to replacement of the doping agent in PPy gradually by oxygen, to partly an ionic activity within the oxide film on p-type Si.

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