

Apparent dependence of conductivity of a conducting polymer on an electric field in a field effect transistor configuration

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A curious effect is reported whereby an electric field apparently greatly affects the conductivity of an organic polymer, poly-3,4-ethylenedioxythiophene (PEDOT), doped to the “metallic” conducting regime when it is used in an all-organic polymer field effect transistor configuration. The response time for change in current in the source/drain PEDOT polymer with a change of gate voltage is slow ($\leq \sim 2$ s), suggesting that ionic diffusion is involved in the phenomenon. It is suggested that the electric field changes only the conductivity of the lowly conducting polymer matrix, which contains highly conducting islands of PEDOT, thus changing the extent of percolation of electric charge between metallic islands, and thereby affecting the bulk conductivity of the PEDOT source/drain material. © 2002 American Institute of Physics. [DOI: 10.1063/1.1511291]

I. INTRODUCTION

Classical (inorganic) field effect transistors (FETs) operate by the principle that the conductivity of an (inorganic) semiconductor, which is connected to an electrical circuit by a “source” terminal and a “drain” terminal attached to opposite ends of the active semiconductor, can be increased or decreased by the presence of an electric field which is supplied by an electrically conducting “gate” electrode.

The gate electrode is not attached directly to the source or drain electrodes. The electric field associated with a given positive or negative potential applied to the gate electrode passes through a nonconducting material “dielectric” that separates the source/drain electrode material from the gate electrode. The change in conductivity of the active semiconducting source/drain material is, in effect, modified by the “through space” electric field effect.

There are two types of field effect transistors, the junction field effect transistor (JFET) and the metal–oxide–semiconductor field effect transistor (MOSFET). The MOSFET is also referred to as a metal–insulator–semiconductor field effect transistor (MISFET)¹ or insulated-gate field effect transistor (IGFET).²

In 1994, the first of three reports describing completely organic FETs³ appeared. An oligomer of thiophene was used as the active semiconducting material, and a commercial conducting cement composed of graphite powder in an organic polymer was used as the gate material. The dielectric consisted of a thin polyester film.

Subsequent reports describe the use of pentacene,⁴ regio-regular poly(3-hexylthiophene)⁴ and poly(9,9-dioctylfluorene-co-bithiophene)⁵ as the active semiconducting organic material. The organic gate materials used were highly doped conducting polyaniline and PEDOT/PSS.

The dielectric materials used were commercial photoresist (SC100, Olin Hunt)⁴ and polyvinylphenol.⁵

A number of hybrid FET devices have been reported^{6–15} which employed both inorganic and organic materials. Inorganic materials similar to those used for completely inorganic FETs were frequently employed as gate or dielectric materials. Organic polymers such as polythiophene,⁶ polyacetylene,⁷ polyparaphenylene-vinylene,⁸ polyaniline^{9,10} and various oligomers¹¹ have been used as the active semiconducting material. In these hybrid devices, a “global” gate was employed, i.e., the gate consisted of a commercial doped Si wafer having a thin layer of silicon oxide (the dielectric) on its surface upon which a narrow thin film of the organic material was deposited, which acted as the active semiconductor. A partly doped polythiophene derivative was also studied¹² in order to ascertain field effects on its conductivity and mobility of charge carriers.

We have previously reported preliminary studies¹⁶ of the FET characteristics of a completely organic device, where a thin film of a polythiophene derivative, PEDOT, doped to the metallic conductivity regime was used instead of the conventional active semiconductor between the source and drain electrodes. We observed a curious unexpected phenomenon, whereby an electric field applied to the source/drain PEDOT resulted in large changes in conductivity of the source/drain material, also consisting of PEDOT. No change was expected since the conductivity of the source/drain material (~ 2 S/cm) is in the “metallic” conducting regime, not in the semiconducting regime.

In this article, we give further information on this effect and on the fabrication of an all polymer FET type (metallic polymer)–insulator–(metallic polymer) (PIPFET) by our line patterning technique^{16–19} which involves no printing of a conducting polymer and which uses a localized, as distinct from a global, gate electrode. We also describe a hybrid FET device in which a commercial micrometer is used as the adjustable gate electrode to controllably vary the thickness of the dielectric material. In both types of devices we demonstrate that the conductivity of the metallic (doped) conduct-

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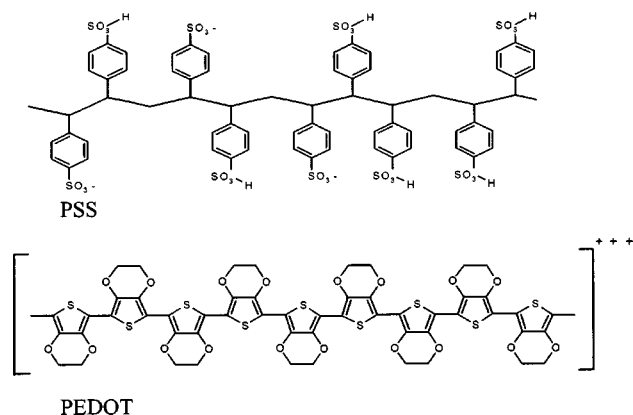


FIG. 1. Chemical structure of poly(3,4-ethylenedioxythiophene) (PEDOT) doped with poly(styrene sulfonic acid) (PSS) PEDOT/PSS blend (Baytron P), concentration of $\sim 1.3\%$ w/w ($\sim 1:2.5$ PEDOT:PSS); doping level of $\sim 1/3$ ethylenedioxythiophene); molecular weight of PSS of 200 000; and repeat units of PEDOT of 5–10 units (Ref. 22).

ing polymer, PEDOT, can be greatly affected by what appears to be an external electric field. Although doped PEDOT has not been extensively studied it is generally assumed to be a metallic organic conducting polymer in comparison with other doped conducting polymers such as polyaniline “which shows Pauli susceptibility approximately linearly proportional to the degree of protonation in agreement with phase segregation into metallic and nonmetallic phases.”²⁰ Such a phenomenon, which we conjecture, based on the slow response time of the device, is ionic in origin, represents a new type of phenomenon in doped conducting polymers.

II. EXPERIMENT

A. Materials

Ten mL ethyleneglycol (Fisher Scientific Co.) and a drop of dodecylbenzenesulfonic acid (DBSA) (Tokyo Chemical Co.) were mixed in a 20 mL vial and manually shaken. Two mL of this solution was mixed with 10 mL of Baytron P (a water dispersion of poly-3,4-ethylenedioxythiophene/polystyrenesulfonic acid, referred to as PEDT/PSS in publications by Bayer Co., Pittsburgh, PA)²¹ in a 20 mL vial and manually shaken. This solution was used to produce films of PEDOT. The chemical structure is shown in Fig. 1.

Optical adhesive (No. 65) was purchased from Norland Products Inc. (New Brunswick, NJ). Polystyrene micron spacers were purchased from Polyscience Inc. (Warrington, PA). Approximately 8 mg of 15 μm spacers or 5 mg of 3 μm spacers were mixed with one drop of optical adhesive. The mixture was used as the (insulating) dielectric material between the source/drain electrode and the gate electrode. It also served, as described below, as an adhesive to bind the above two electrodes. Silicone oil (S159-500) was purchased from Fisher Scientific (Fair Lawn, NJ), and was also used as a dielectric material.

B. Instruments

Batteries of 9.0 and 1.5 V were purchased from Duracell (Bethel, CT). Two 15XL (Wavetek Corp., San Diego, CA)

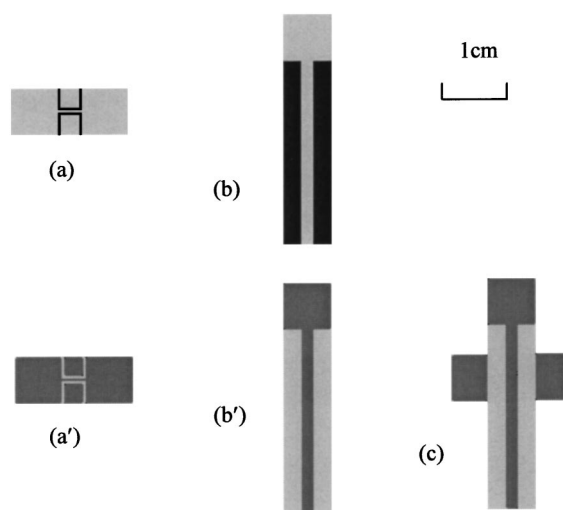


FIG. 2. Line pattern (actual size) of (a) the source–drain electrode and (b) the gate electrode; \square represents clear PET transparency; \blacksquare represents toner deposited on the clear PET transparency; (a') indicates the source–drain electrode and (b') the gate electrode coated with PEDOT after removal of the toner; \blacksquare represents PEDOT material; (c) the fabricated device made by superposition of b' on a' separated by optical adhesive with a polystyrene micron spacer.

multimeters were used to measure the current to within $\sim \pm 0.05 \mu\text{A}$ between the source and drain (I_{S-D}) and leakage current between the gate and source/drain electrodes. Two 77 III multimeters (Fluke Corp., Everett, WA) were used to measure the gate voltage (V_G) and the source/drain voltage (V_{S-D}). The voltage supplied by the batteries was frequently slightly different from the rated value. The actual voltage in any given experiment was measured by a voltmeter as shown in Figs. 3 and 4. A variable high voltage power supply (Gamma High Voltage Research Inc., Ormond Beach, FL) was also used to supply the gate potential.

C. Device fabrication

The source (S)/drain (D) electrode and the gate (G) electrode (Fig. 2) were fabricated on 100 μm thick poly(ethylene terephthalate) (PET) transparency film (Nashua XF-20) using a 600 dpi office laser printer, Hewlett-Packard LaserJet 5000N, and Hewlett-Packard Laser Jet Toner Cartridge C4129X. The laser jet lines and the PET transparency were coated three to five times with the PEDOT dispersion using a simple, glass roller consisting of a glass rod inside a glass tube followed by a 2–3 min drying with a hot air gun between each coating. The total thickness of the PEDOT films was ~ 300 –400 nm. The PEDOT dispersion wets the plastic transparency, but not the lines printed on it by the office laser printer. The printed lines were removed by sonication in toluene for 1 min followed by methylethylketone for 30 s, leaving only the conducting polymer on the transparency (see Fig. 2).

The field effect device (type 1) was constructed by covering the narrow bridge of the source/drain electrode with the insulating dielectric material as described above, upon which the gate electrode is placed at 90° . The two electrodes are placed between two microscope slides and are conveniently

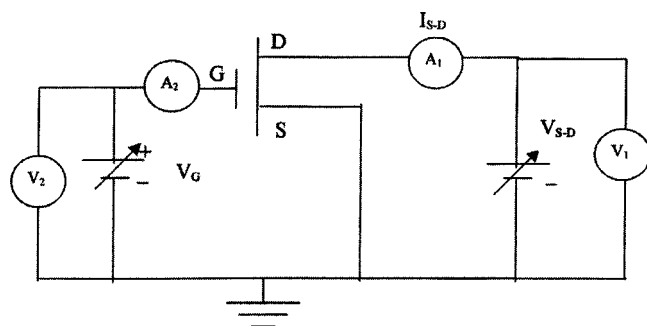


FIG. 3. Schematic of the electrical circuit for monitoring the effect of electric field strength on the conductivity of the conducting PEDOT source/drain. V is the voltmeter, A is the ammeter, G is the gate, S and D are connections to the source-drain polymer, V_G is the gate voltage, and V_{S-D} is the source-drain voltage.

held firmly in place by clamping between the movable barrel and fixed base of a micrometer (Mitutoyo Co., Japan, model No. 293-765, accuracy of $\pm 0.5 \mu\text{m}$). The insulating dielectric material was then cured from different directions by UV light from a portable UV lamp (UVGL-25, UVP Inc.), maximum wavelength at 366 nm for ~ 45 min.

The field effect device (type 2) was constructed by placing the source/drain electrode on a microscope slide which was attached to the fixed base of the micrometer by adhesive tape. The movable barrel of the micrometer was used as the adjustable gate electrode and could be controllably moved towards the source-drain electrode. Air served as the insulating dielectric material between the source-drain electrode and the adjustable micrometer gate electrode.

D. Measurement setup

After fabrication, the device was incorporated into the circuit shown in Fig. 3. Figure 4 shows the dc power supply used in two typical experiments: (1) constant S-D and G potential; (2) variable S-D and G voltages. In both of these experiments, an ammeter, A_1 , was used to measure the current passing between the source and drain (I_{S-D}) as a function of a variable gate voltage V_2 . Another ammeter, A_2 , was placed between the gate electrode and the power supply in order to ascertain if any current could possibly flow between the gate and source-drain electrode. Unless noted to the contrary, no significant current, i.e., "leakage" current, was observed in any of our studies. All measurements were carried out at ambient temperature.

III. RESULTS

This is the first time to the best of our knowledge, that a field effect has been observed in a metallic doped conducting polymer (conducting PEDOT polymer). Here we describe two new devices based on PIPFET construction: type 1 is a flexible all-polymer transistor that uses an optical adhesive plus spacer as the insulating dielectric material; type 2 uses the micrometer as the adjustable gate electrode and air as the insulating dielectric material.

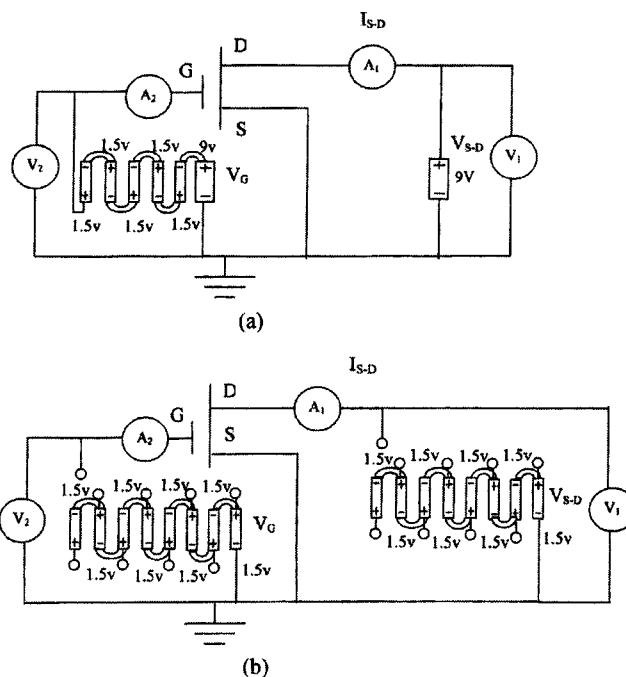


FIG. 4. Diagrams of the dry cell battery dc power supply for the electrical circuit in Fig. 3. Appropriate V_G and V_{S-D} potentials could easily be obtained by appropriate selection of battery combinations: (a) constant G voltage ~ 15 V and S-D voltage ~ 9 V; (b) varying positive G and S-D voltages of ~ 1.5 , 3, 4.5, 6, 7.5, 9, 10.5 and 12 V.

A. Type 1: Flexible all-polymer transistor

The type 1 all-polymer transistor device exhibits the general reversible characteristics of a field effect transistor (see Fig. 5). By applying positive 9.1 V potential to the S-D electrode without applying gate voltage, the transistor is in an "on" state, which shows high S-D current ($\sim 106.6 \mu\text{A}$). We keep the S-D current stable for 40 min, then apply positive 15.7 V gate potential. The S-D current is reduced to $\sim 0.4 \mu\text{A}$ at a rate of $\sim 17 \mu\text{A}/\text{min}$ at the steepest portion of the curve, which is called the "off" state of the transistor. In other studies, the I_{S-D} frequently decreases to lower values. After removing the gate voltage, i.e., the $V_G = 0$ V, the S-D

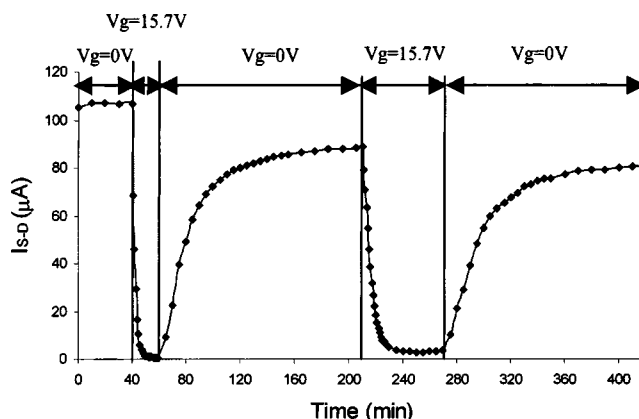


FIG. 5. I_{S-D} vs time for the type 1 PEDOT device shown in Fig. 2(c). The voltage applied to the gate terminal (V_G) is indicated and is held constant at the value shown between any two consecutive vertical lines. The V_{S-D} was held constant at 9.1 V.

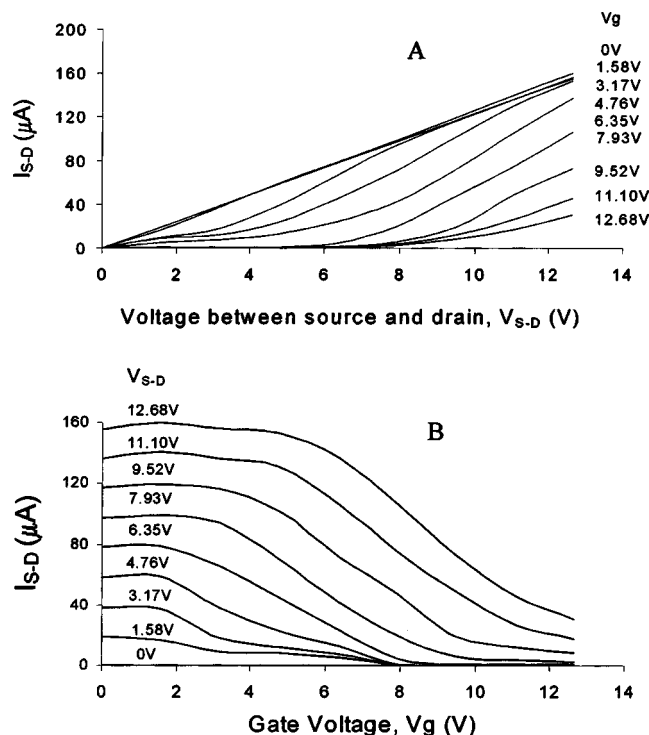


FIG. 6. (A) I_{S-D} vs V_{S-D} under different gate potentials (V_G) and (B) I_{S-D} vs V_G under different V_{S-D} characteristics of a type 1 PEDOT transistor measured at room temperature.

current recovers at a rate of $\sim 12.2 \mu A/min$ at the steepest portion of the curve and approaches some final undetermined value asymptotically. This field effect is reproducible, however, with a slower response time as seen (in Fig. 5) in the second cycle with a rate of decrease of I_{S-D} of $\sim 8.4 \mu A/min$ and a rate of increase of I_{S-D} of $\sim 1.8 \mu A/min$ at the steepest portion of the curve. The on/off ratio of this device is approximately 1×10^3 .

We also studied the field effect of different V_G values on the current between the source and drain, I_{S-D} , using the electronic circuit shown in Fig. 4(b). Since the response time is slow, we only compare the data dynamically. We fix the source-drain potential and apply different continuously increasing gate voltages of 1.58, 3.17, 4.76, 6.35, 7.93, 9.52, 11.10 and 12.68 V. We record the source-drain current 5 min after applying a different gate voltage. The same experimental results are given in both Figs. 6(a) and 6(b). The decrease in current (I_{S-D}) curves at constant V_{S-D} with an increase in positive V_G is qualitatively consistent with a depletion effect as expected for the p -type doped PEDOT employed as the source/drain electrode; however, the curves have a different shape from those of, for example, α , ω -di(hexyl)sexithiophene.³ This is not surprising, since (i) the source/drain material is a metallic doped polythiophene derivative; and (ii) the curves are obtained over a limited range of V_{S-D} under nonequilibrium conditions.

The changes in I_{S-D} (Fig. 7) of a type 1 device with V_G were measured simultaneously as described in Sec. II D. The changes in I_{S-D} with changes in $V_G \geq 0$ V (Fig. 7) are in qualitative agreement with analogous data shown in Fig. 5. However, when a negative V_G (-9.2 V) was applied, as

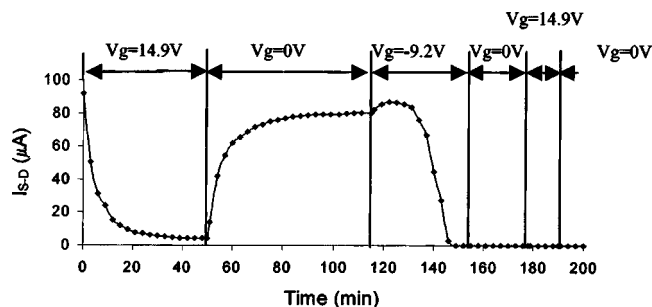


FIG. 7. I_{S-D} vs time for the type 1 PEDOT device shown in Fig. 2(c). The voltage applied to the gate terminal (V_G) is indicated and is held constant at the value shown between any two consecutive vertical lines. The V_{S-D} was held constant at 9.14 V.

shown in Fig. 7, a small increase in I_{S-D} was observed followed by a continuous decrease in I_{S-D} to $0 \mu A$. A subsequent increase in the value of V_G to 0 V followed by an increase in V_G to 14.9 V followed by a decrease in V_G to 0 V had no effect on I_{S-D} which remained at zero.

It is apparent from Fig. 7 that application of a negative V_G during the time period studied destroys the capability of the PEDOT source-drain electrode to respond further to a change in V_G .

B. Type 2: Transistor using a micrometer as the gate electrode

An adjustable gate electrode, described in Sec. II C, adds an additional method by which to study field effects. It permits the study of I_{S-D} at adjustable field strengths (F), where $F = V_G/d$ (d is the distance between the gate electrode and source/drain electrode); the dielectric constant of air is approximately that of vacuum, i.e., 1. The field strength can be controllably varied by this method by two very simple ways using exactly the same source/drain electrode sample:

- by keeping d constant and varying V_G .
- by keeping V_G constant and varying d .

Preliminary studies of each of these two types are described below.

(i) Keep d constant and vary V_G . For example, the adjustable micrometer barrel was set $1 \mu m$ above the PEDOT source/drain electrode with no V_G applied. A stable source/drain current of $108 \mu A$ was observed over a 10 min period (Fig. 8). A potential of 22.4 V was then applied to the gate electrode, i.e., the movable micrometer barrel. In these studies, in Fig. 8 the I_{S-D} decreases within $\sim 1-2$ s judged qualitatively by eye to a value of $0.7 \mu A$. The gate voltage applied was then immediately disconnected and gives $V_G = 0$ V. Within $\sim 1-2$ s, the I_{S-D} increases to $75.6 \mu A$ and then slowly increases further as shown in Fig. 8, stabilizing after $2-3$ min at $I_{S-D} = 101.0 \mu A$. A repetition of the experiment using instead a $V_G = 41.3$ V followed by $V_G = 0$ V also gave a response time of $\sim 1-2$ s and qualitatively analogous changes of I_{S-D} . These were the fastest response times observed with a type 2 setup. No observable leakage current was detected. In a different, but identical experiment, a response time of ≤ 4 s and a leakage current of $1.5 \mu A$ were

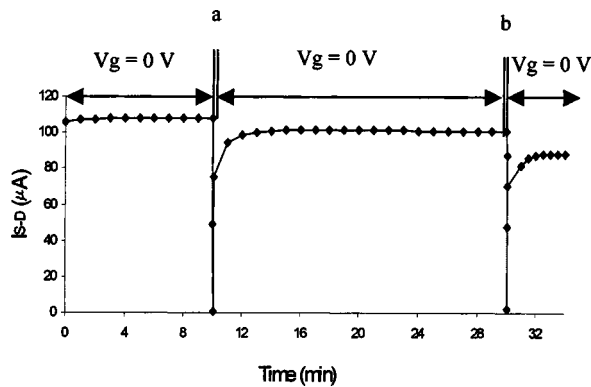


FIG. 8. At $d=1\text{ }\mu\text{m}$, I_{S-D} vs time for the type 2 PEDOT device. The source-drain voltage was held constant at 8.7 V. The voltage applied to the gate terminal is indicated and is held constant as shown between any two consecutive vertical solid lines. At point (a), V_G was changed from 0 to 22.4 V, and then after $\sim 2\text{ s}$ V_G was changed back to 0 V. At point (b), V_G was changed from 0 to 41.3 V, and then after $\sim 2\text{ s}$ V_G was then changed back to 0 V.

observed. The difference in response time was presumably caused by larger dielectric air space between the adjustable gate electrode and the source/drain material. The response time is presumed to be sensitive to the relatively coarse micrometer setting attainable.

(ii) Keep V_G constant and vary d . An example of preliminary studies using this approach is summarized in Fig. 9. The data in Fig. 9 were obtained using exactly the same experimental setup of gate and source/drain used for the data given in Fig. 8. The initial I_{S-D} curve in Fig. 9 is simply a continuation of the final I_{S-D} data curve given in Fig. 8. V_G is first held constant at 0 V at $d=1\text{ }\mu\text{m}$; d is then increased by adjusting the micrometer setting to $\sim 3\text{ }\mu\text{m}$ where I_{S-D} stabilized at a constant value of $88.4\text{ }\mu\text{A}$. Figure 9 shows I_{S-D} values for a change in d for two different constant V_G values. As can be seen, when $V_G=0\text{ V}$, i.e., the gate has no applied potential, i.e., it is at earth potential ($F=0$), there is, as expected, no change in I_{S-D} when d is changed from ~ 1

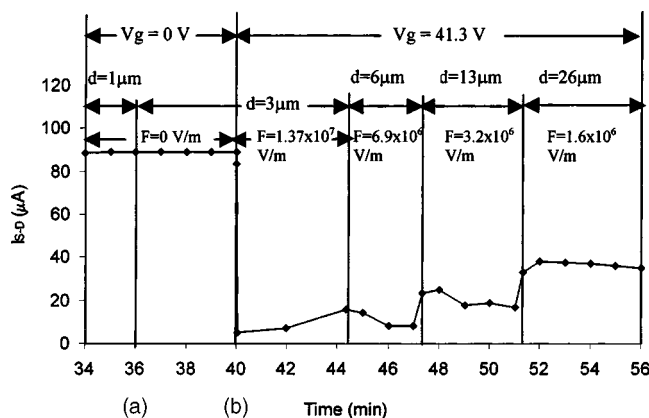


FIG. 9. At $V_G=0$ and then 41.3 V, I_{S-D} vs time for the type 2 PEDOT device. The source-drain voltage was held constant at 8.7 V, however, the distance between the movable gate electrode and source/drain electrode was adjusted. Between two consecutive vertical lines the insulating air gap was maintained at a constant value of d . The voltage applied to the gate electrode is held constant between any two consecutive dashed vertical lines as shown by the values given. The field strength (F) is shown by the values.

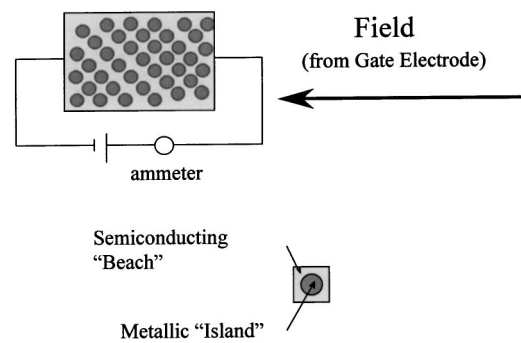


FIG. 10. Percolation field effect in doped conducting polymers. Metallic islands separated by beaches of non- or lowly conducting (semiconducting) polymer. The field changes the conductivity of the semiconducting beaches but not that of the metallic islands, hence the field changes the extent of electrical percolation between the metallic islands, and therefore changes the bulk conductivity of the material.

to $\sim 3\text{ }\mu\text{m}$ at point (a), i.e., $t=36\text{ min}$, since there is no change in F , which remains zero. At point (b), i.e., $t=40\text{ min}$, a potential of 41.3 V was applied to the gate electrode ($F=1.38\times 10^7\text{ V/m}$) with a concomitant decrease in I_{S-D} as expected. Successive increases in the d values set by the micrometer to $d=6, 13$, and $26\text{ }\mu\text{m}$, i.e., F values of $6.9\times 10^6, 3.2\times 10^6$ and $1.6\times 10^6\text{ V/m}$, respectively, resulted, as might be expected, in successive increases in I_{S-D} values. The relatively small changes in I_{S-D} following a decrease in F values are undoubtedly related to the time dependent adjustment of I_{S-D} to the new electric field strength. It is of interest to note that I_{S-D} is sensitive to changes in field strength, even at large gate and source/drain electrode separations of, for example, $d=13\text{ }\mu\text{m}$.

In a preliminary experiment, it was demonstrated that the effect of an electric field ($F=1.67\times 10^5\text{ V/m}$) on the conductivity of a PEDOT source/drain electrode could be observed over a distance of $\sim 18\text{ cm}$ in air or vacuum when a metal gate electrode ($V_G=30\text{ 000 V}$) was employed. The initial gate voltage was 0 V, and I_{S-D} was $\sim 139.1\text{ }\mu\text{A}$. Application of V_G of 30 000 V resulted in a decrease of I_{S-D} to $\sim 127.6\text{ }\mu\text{A}$ during $\sim 28\text{ min}$. This longer response time is qualitatively consistent with the smaller field strength used in this study compared to in the previous studies described.

In another experiment, a drop of silicone oil was placed in the center of the source-drain PEDOT electrode and the adjustable micrometer gate barrel at $V_G=0\text{ V}$ was screwed down onto the PEDOT surface ($V_{S-D}=18\text{ V}$) until the adjustable torque screw turned freely, i.e., the barrel stopped descending, leaving a thin layer of unknown thickness of silicone oil (dielectric) between the gate and source/drain PEDOT. A potential of $V_G=40.6\text{ V}$ was applied, and within 30 s the I_{S-D} fell from ~ 86.8 to $\sim 0.4\text{ }\mu\text{A}$. Upon reducing V_G to 0 V, the I_{S-D} slowly increased to $\sim 56.6\text{ }\mu\text{A}$ within 5 min.

IV. SUMMARY

The results described represent a type of phenomenon not previously observed in either inorganic or organic source/drain material in a field effect configuration. The

source/drain material is a conducting polymer doped to the metallic conductive regime, the conductivity of which is changed by many orders of magnitude under the influence of a modest electric field in which a slow response time is observed. The slow response time (≥ 2 s) is presumably caused by some type of relatively slow ionic diffusion effect. The effect is apparently independent of the dielectric material since it is observed when the dielectric material is an organic polymer adhesive, air, vacuum or silicone oil. It is still not clear why complete reversibility is not observed, although several working hypotheses can be considered. Additional experimental work is underway to obtain evidence of a possible valid interpretation of the results.

The doped metallic PEDOT film ($\sigma \approx 2 \text{ S cm}^{-1}$ at room temperature) would not be expected to show a change in conductivity by exposure to a field of this type under the configuration used. We believe that this effect presents an entirely different method for ascertaining the nature of a highly doped metallic conducting polymer. It has frequently been postulated that a doped conducting polymer consists of metallic "islands" surrounded by lowly conducting "beaches" as shown in Fig. 10.^{16–18} Assuming that the metallic islands act in a similar way to an electric field as a conventional inorganic metal, which may not necessarily be the case, we postulate that in the effect we have observed only the lowly conducting beaches and not the metallic islands respond to the field applied. The application of an electric field changes the conductivity of the beaches and hence the extent of electrical percolation in the source/drain PEDOT electrode between the metallic islands, hence changing the bulk conductivity of the material.

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