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Linkage and donor–acceptor effects on resistive switching memory devices of 4-(*N*-carbazolyl) triphenylamine-based polymers†

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In order to gain deeper insight into the linkage effect and donor–acceptor effect on memory behavior (from DRAM to WORM), 4-(*N*-carbazolyl)triphenylamine-based polyimides and polyamides *t*-butyl-6FPI, *t*-butyl-DSPI, OMe-6FPI, OMe-DSPI, *t*-butyl-6FPA, *t*-butyl-DSPA, OMe-6FPA, and OMe-DSPA were synthesized and their memory behaviours were investigated.

In recent years, polymer materials have been widely used in organic electronics, such as light-emitting devices,¹ transistors,² solar cells,³ and electrochromic devices.⁴ Beside these applications, polymeric memory materials have attracted increasing attention since the first one was reported by Silva *et al.* in 1970.⁵ Polymeric memory materials have the advantages of rich structural flexibility, low-cost, solution processability, and three-dimensional stacking capability.⁶ They can store information in the form of high (ON) and low (OFF) current state in place of the amount of charges stored in a cell of the traditional inorganic memory devices. Hence, they are likely to be predominant materials for memory devices. The design of the polymer structures that can provide expected memory properties within a single polymer chain is an important issue.

The resistor-type memory devices offer simplicity in fabrication and allow possibility for high data storage density *via* two-dimensional or even three-dimensional (stacking) cross-bar arrays.⁷ Electron donor–acceptor polymers are considered as suitable resistor-type memory materials because charge transfer (CT) between the donor and acceptor moieties can give rise to a highly conductive state. There are numbers of application of donor–acceptor type polymers including conjugated polymers,⁸ non-conjugated polymers with pendent electroactive chromophore,⁹ functional polyimides,¹⁰ and hybrid composites.¹¹ Among the polymer systems, aromatic polyimides (PIs) and polyamides (PAs) are promising candidates for memory device applications due to their excellent thermal stability, chemical resistance, mechanical properties, and high ON/OFF current ratio, resulting

from the low conductivity in OFF state. In addition, triphenylamine (TPA) and its derivatives are candidates for hole-transporting materials in organic optoelectronic devices due to the resulting stable radical cations and high hole-mobility.¹² TPA could act as a donor and facilitate the CT behaviour of polymer. Besides, the introduction of TPA units into the polymers could enhance the glass transition temperature (T_g) and the solution processability due to its high aromatic content and its packing-disruptive propeller-like structure. In 2006, Kang *et al.* first found the TPA-based polyimide 6F-TPA PI, which had a volatile dynamic random access memory (DRAM) characteristic.¹³ As demonstrated in our previous studies,¹⁴ TPA-based polymers might reveal different volatile memory properties due to the donor, acceptor or linkage effect. The polymers with the stronger electron donating/withdrawing units or non-planar linkage structure generally showed a longer retention time. It is well known that carbazole derivatives have high triplet energy and good hole-transporting ability, and can be used as host materials for phosphorescent organic light-emitting diodes.¹⁵ Carbazole can be easily functionalized at its (3,6-), (2,7-), or *N*-positions, and then covalently linked into polymeric systems, both in the main chain as building blocks and in a side chain as subunit. Carbazole-containing polymers are considered as a very important class of electroactive and photoactive materials.¹⁶ For example, the excellent hole-transporting properties of poly(2,7-carbazole)s make them highly promising materials for p-type transistors, photovoltaic devices, and light-emitting diodes.¹⁷ The behaviour of memory devices based on carbazole-containing polyimides were also reported.¹⁸

In this study, we investigate the memory properties of the electron donating 4-(*N*-carbazolyl)triphenylamine (CzTPA) unit containing polyimides (PIs), *t*-butyl-6FPI, OMe-6FPI, *t*-butyl-DSPI and OMe-DSPI, and poly-amides (PAs), *t*-butyl-6FPA, OMe-6FPA, *t*-butyl-DSPA and OMe-DSPA and demonstrate the effects of different linkages, donors and acceptors within these CzTPA based polymers on the memory behaviours systematically. The

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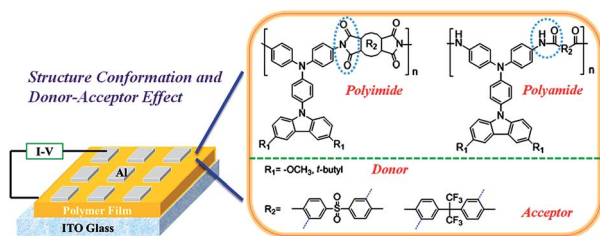


Fig. 1 Chemical structures of CzTPA-based aromatic polymers and the schematic diagram of the memory device consisting of a polymer thin film sandwiched between an ITO bottom electrode and an Al top electrode.

different linkers (imide and amide linkages), donors (methoxy and *t*-butyl groups), and acceptors (hexafluoroisopropylidene (6F) and sulfonyl groups) are expected to have different conformation, dipole moment, HOMO and LUMO energy levels, and retention time. Chemical structures of the polymers and the schematic diagram of the memory device are illustrated in Fig. 1.

These polyimides and polyamides were readily synthesized by the polycondensation reactions of CzTPA-based diamines with commercially aromatic dianhydrides and dicarboxylic acids, respectively. The synthetic details and characterization data, such as FT-IR, proton NMR, GPC, inherent viscosities, UV-visible absorption spectra, cyclic voltammetric diagrams, solubility behaviours and thermal properties of the polymers are included in the ESI.† All the polymers could afford flexible and strong films, indicating they exhibited high molecular weights.

UV-vis absorption spectra of these polymers are depicted in Fig. S8 (ESI†), and the onset wavelength of optical absorption was utilized to obtain the optical energy band gap (E_g). The electrochemical properties of these polymers were investigated by cyclic voltammetry (CV) using a 0.1 M tetrabutylammonium perchlorate (TBAP) as the supporting electrolyte under a nitrogen atmosphere. The typical CV diagrams of these polymers are depicted in Fig. S9 (ESI†), and the onset oxidation is used to calculate the HOMO energy level. The redox potentials of the polymers and their respective HOMO and LUMO were calculated

and summarized in Table 1. The HOMO energy levels of the polyamides are higher than the corresponding polyimides.

The memory behaviours of these polymers were depicted by the current–voltage (I – V) curves of an ITO/polymer/Al sandwich device as shown in Fig. 2, and Al was used as the electrode for applying voltage during the sweep. Polymer film thickness was optimized around 50 nm and was mentioned the effect of the polymer film thickness on the obtained memory behavior in our previous studies.²⁰ Fig. 2a reveals I – V curves of ***t*-butyl-6FPi**. The device based on ***t*-butyl-6FPi** could not switch to the ON state and stayed in the OFF state with a current range 10^{-13} to 10^{-15} A in the positive sweep up to 6 V. However, a sharp increase in the current could be observed at -4.3 V during the negative sweep, indicating that the device undergoes an electrical transition from the OFF state to the ON state (writing process). The device also remained in the ON state during the subsequent negative (the third sweep) and positive scans (the fourth sweep). Thus, the ***t*-butyl-6FPi** memory device could not be reset to the initial OFF state by applying a reverse electric field exhibiting non-erasable behaviour. The fifth sweep was conducted after turning off the power for about 1 min and it was found that the ON state had relaxed to the steady OFF state without an erasing process, and the device could be switched to the ON state again at the threshold voltage of -4.0 V. Thus, the device behaves short retention time and rewritable property which are indicative of DRAM characteristics.

The memory devices of **OMe-6FPi** and ***t*-butyl-DSPI** switched from 10^{-13} to 10^{-5} A at the threshold voltage of -4.1 V (**OMe-6FPi**) and -3.9 V (***t*-butyl-DSPI**) in the negative sweep, and the ON state could be read by the subsequent negative (the third sweep) and positive scan (the fourth sweep) as shown in Fig. 2b and c. The ON state would return to the OFF state in 6 min (**OMe-6FPi**) and 30 min (***t*-butyl-DSPI**) after removing the applied voltage, and could subsequently switch to ON state again at the threshold voltage of -4.0 V (**OMe-6FPi**) and -3.6 V (***t*-butyl-DSPI**), respectively, implying a volatile SRAM like behaviour. Comparing to volatile DRAM and SRAM behaviour, the ON state of **OMe-DSPI** could be retained even after turning off the power for 1 h or a longer time since it has switched on.

Table 1 Redox potentials and energy levels of polymers

Polymer	UV-vis absorption (nm)	Oxidation potential (V) (vs. Ag/AgCl in CH ₃ CN)			
	λ_{onset}^a	E_{onset}^a	E_g^b (eV)	HOMO ^c (eV)	LUMO (eV)
<i>t</i>-Butyl-6FPi	405	0.81	3.06	5.25	2.19
<i>t</i>-Butyl-DSPI	394	0.79	3.15	5.23	2.08
OMe-6FPi	396	0.91	3.13	5.35	2.22
OMe-DSPI	387	0.93	3.20	5.37	2.17
<i>t</i>-Butyl-6FPA	466	0.65	2.72	5.09	2.37
<i>t</i>-Butyl-DSPA	406	0.66	3.05	5.10	2.05
OMe-6FPA	369	0.7	2.64	5.21	2.57
OMe-DSPA	395	0.69	3.14	5.22	2.08

^a Data from ref. 19. ^b The data were calculated from absorption edges of the polymer films by the equation: $E_g = 1240/\lambda_{\text{onset}}$ (energy gap between HOMO and LUMO). ^c The HOMO energy levels were calculated from CV and were referenced to ferrocene (4.8 eV; $E_{\text{onset}} = 0.36$ V).

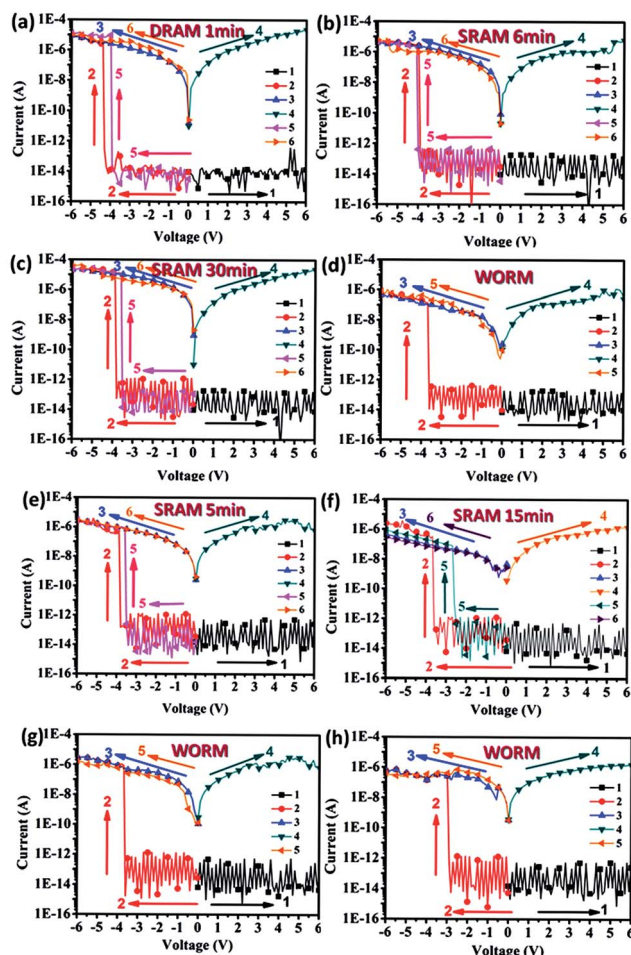


Fig. 2 Current–voltage (I – V) characteristics of the ITO/polymer (50 ± 3 nm)/Al memory devices. (a) *t*-Butyl-6FPI, (b) OMe-6FPI, (c) *t*-butyl-DSPI, (d) OMe-DSPI, (e) *t*-butyl-6FPA, (f) OMe-6FPA, (g) *t*-butyl-DSPA, and (h) OMe-DSPA.

Thus, the I – V curves indicate the memory device based on **OMe-DSPI** film in Fig. 2d revealed non-volatile write-once-read-many times (WORM) memory properties. In addition, our group¹⁴ has also demonstrated the effect of the planarity of the linkage between donor and acceptor and found that the polyamide with non-planar linkage exhibited longer retention time. In addition, Fig. 2e–h depict the I – V curves of *t*-butyl-6FPA, **OMe-6FPA**, *t*-butyl-DSPA and **OMe-DSPA** that exhibited longer retention time than the analogous PIs. The devices of *t*-butyl-6FPA and **OMe-6FPA** showed similar SRAM memory properties to **OMe-6FPI** at the ON state, and could be kept for 5 min (*t*-butyl-6FPA) and 15 min (**OMe-6FPA**) after turning off the power. While, the devices derived from *t*-butyl-DSPA and **OMe-DSPA** showed WORM memory behaviour after switching on at voltage of –3.7 V (*t*-butyl-DSPA) and –3.0 V (**OMe-DSPA**), respectively. The retention times on the ON, and OFF states are depicted in Fig. S12 (ESI[†]).

In order to get deeper insight into the different memory behaviours of these CzTPA based polymers, a molecular simulation of the basic unit was carried out by DFT/B3LYP/6-31G(d) with the Gaussian 09 program as shown in Fig. S10 and S11

(ESI[†]). The HOMO and LUMO energy levels calculated by molecular simulation were in agreement with the experimental values tendency and the charge density isosurfaces of the basic unit were summarized. According to a previous literature,¹³ when the applied electric field reaches the switching-on voltage, some electrons at the HOMO accumulate energy and transit to the LUMO, forming a charge transfer complex (ON state) by different ways. For these CzTPA-based polymers, the HOMO is located mainly at the electron-donating CzTPA moieties, while LUMO is distributed around the electron-withdrawing hexafluoroisopropylene (6F) group and sulfonyl group. Using *t*-butyl-6FPI as an example, when the applied electric field reaches the switching-on voltage, some electrons at the HOMO accumulate energy and transit to the LUMO5 with the highest probability because of overlapping of the HOMO and LUMO5 resulting in an excited state. Nevertheless, electrons at the HOMO may also be excited to the other intermediate LUMOs with a lower energy barrier belonging to the acceptor units. Thus, CT occurs through several courses to form the conductive CT complexes, including indirectly drop from LUMO5 through intermediate LUMOs to LUMO, from the intermediate LUMOs to LUMO, or directly be excited from HOMO to LUMO. When the intra- or intermolecular CT occurred by the applied electric field, the generating holes can delocalize to the CzTPA moieties forming an open channel in the HOMO for the charge carriers (holes) to migrate through. Therefore, the current increases rapidly and the memory device can be switched to the high conductivity state (ON state).

Furthermore, the sulfonyl-containing and methoxy-containing polymers generally show a longer retention time than 6F-containing and *t*-butyl-containing polymers with the same linkages due to the stronger donor–acceptor effect. Therefore, these CzTPA-based polyamides and polyimides exhibited different memory behaviors due to the planarity, donor–acceptor effect, and dipole moments. Moreover, the polyamide system revealed higher dipole moment than the corresponding polyimides resulting in longer retention time of the memory devices.

Conclusions

In summary, new CzTPA-based polyamides and polyimides which containing different donor and acceptor groups have been successfully prepared for memory device application. All sulfonyl-containing polymers exhibited WORM properties except *t*-butyl-DSPI, while **OMe-6FPA**, **OMe-6FPI** and *t*-butyl-6FPA exhibited SRAM properties with tunable retention times of 15 min, 6 min, and 5 min. The device of *t*-butyl-6FPI showed DRAM properties with retention only 1 min. Furthermore, sulfonyl-containing polymers generally have a longer retention time than 6F-containing polymers with the same linkage due to the stronger acceptor effect, and methoxy-containing polymers generally have a longer retention time than *t*-butyl-containing analogs with the same linkage due to the stronger donor effect. In addition, the polyamides exhibited the longer retention time than the corresponding polyimides due to the linkage planarity and dipole moment effects.

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