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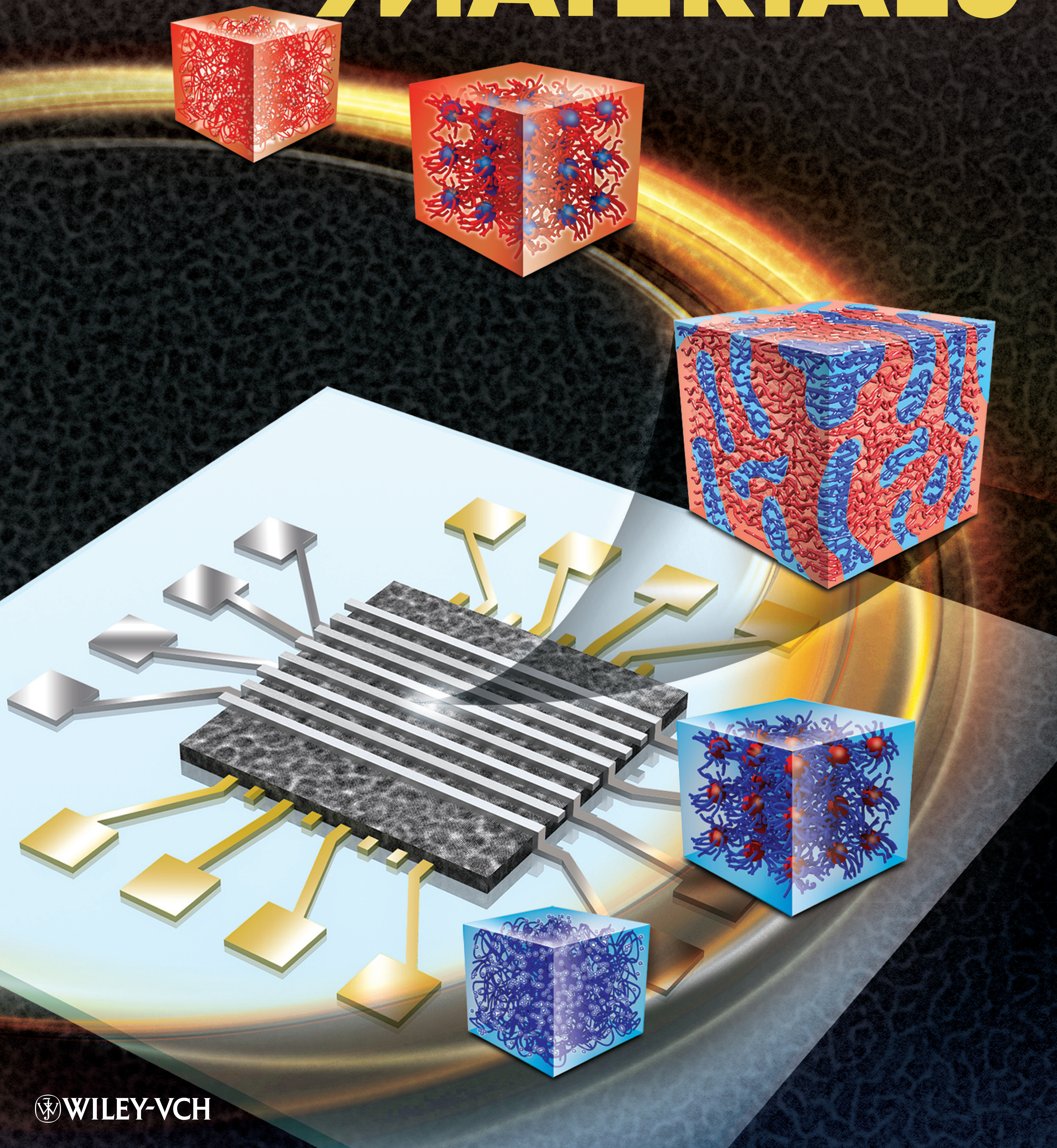

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Structural and Electrical Characterization of a Block Copolymer-Based Unipolar Nonvolatile Memory Device

Nam-Goo Kang, Byungjin Cho, Beom-Goo Kang, Sunghoon Song, Takhee Lee, and Jae-Suk Lee**

Polymeric memory devices have attracted a great amount of attention due to their low fabrication cost, good scalability, printability, simplicity of device structure, and three-dimensional stackability.[1–7] Furthermore, polymeric materials in particular have unique properties, such as flexibility and easy processability by spin-coating, ink-jet printing, and roll-to-roll.[8] These merits have further accelerated the development of several types of polymer-based memory devices such as rewritable memory,[2–7] write-once read-many times (WORM) memory,[9–11] and dynamic random access memory (DRAM).[12,13] For instance, polythiophene,[14] polyaniline,[15] poly(9-vinylcarbazole),[16] and polyimide[17] have been applied to memory devices as the matrix of a dye, polyelectrolyte, or component of a charge-transfer (CT) complex in a doping or mixing system. Several systems based on polymers and nanoparticles, such as mixtures of fullerenes (C_{60}) and polystyrene,[18] composites of poly(9-vinylcarbazole) and titanium dioxide (TiO_2) nanoparticles,[19] polyaniline nanofibers with Au nanoparticles,[20] and polystyrene with Au nanoparticles,[21] have been proposed to induce bistable switching phenomena. In these pioneering approaches, however, some difficulties of non-uniform dispersion due to poor compatibility between polymers and nanoparticles or small molecules still exist. For example, the aggregation of nanoparticles and irregular phase separation can occur in these non-homogeneous systems, causing poor reproducibility of device performance.[22]

This limitation in controlling the electrical properties of polymeric materials due to irregular dispersion has encouraged the search for new routes of homogeneous dispersion. To this end, various random copolymers, which link nanoparticles or small molecules covalently, such as non-conjugated copolymers containing carbazole, europium complex, and oxadiazole derivatives[4] and homopolymers such as poly(9-vinylcarbazole) and poly(2-(9*H*-carbazol-9-yl)ethyl methacrylate),[23] have been developed to prevent aggregation and thus improve the reproducibility and stability of devices. Nevertheless, it is almost impossible to achieve a thoroughly controlled molecular weight distribution from random copolymers as reported above.[2,4,5] Furthermore, if each segment of a random copolymer possesses completely different electrical properties (e.g., metallic versus insulating), it is generally difficult to control the electrical characteristics of devices based on these materials.

Accordingly, this difficult manipulation of the electrical characteristics of random copolymers requires a new approach to easily control the electrical properties through well-defined nanostructuring of block copolymers. The nanostructure and morphology of self-assembled block copolymers has been applied successfully to organic electronic devices.[24–27,28] However, only some coil-coil block copolymers such as polystyrene-*b*-poly(methyl meth acrylate) (PS-*b*-PMMA) and polystyrene-*b*-poly(4-vinylpyridine) (PS-*b*-P4VP) in organic-based nonvolatile memory devices,[29–31] nanoparticles in ordered coil-coil block copolymer,[32] and rod-coil block copolymers[33,34] have been utilized. Therefore, the design of novel block copolymers with controlled morphologies and electrical properties is required to enable reversible resistive memory switching.

In this study, to improve the non-uniform dispersion caused by mixing or doping as mentioned above, we synthesized new block copolymers of poly(9-(4-vinylphenyl)carbazole)-*b*-poly(2-vinylpyridine) (denoted PVPCz-*b*-P2VP) consisting of two different blocks of carbazole as an electron donor (PVPCz) and pyridine as a very weak electron acceptor (P2VP) moiety by anionic polymerization. Various morphologies, from spherical to lamellar, were obtained by microphase separation derived from different block ratios of PVPCz and P2VP. Furthermore, depending on the block ratios of PVPCz and P2VP, different electrical behaviors—metallic, bistable switching, and insulating—were reproducibly demonstrated. Especially, the devices based on a block copolymer system showing a lamellar structure exhibited excellent memory performances (stable endurance and long retention) with a high ON/OFF ratio of 10^3. The reversible switching is associated with the formation and rupture of highly conductive paths consisting primarily of carbazole segments.

For the synthesis of block copolymers of poly(9-(4-vinylphenyl)carbazole)-*b*-poly(2-vinylpyridne) (PVPCz-*b*-P2VP), as shown in **Figure 1**b, each condition for the anionic polymerization of 9-(4-vinylphenyl)carbazole (VPCz) and 2-vinylpyridine (2VP) was optimized (see Table S1 in the Supporting Information). First, VPCz was synthesized as an electron donor by modifying the structure of *N*-vinylcarbazole (NVC) by inserting a phenyl group between the vinyl group and carbazole. This structure makes the anionic polymerization of VPCz possible. In particular, it overcomes the limits of anionic polymerization

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N.-G. Kang,[+,‡] B. Cho,[+] B.-G. Kang, S. Song,
Prof. T. Lee,[†] Prof. J.-S. Lee
Department of Nanobio Materials and Electronics
School of Materials Science and Engineering
Gwangju Institute of Science and Technology
Oryong-Dong, Buk-Gu,Gwangju 500-712, Korea
E-mail: tlee@snu.ac.kr; jslee@gist.ac.kr

[†] Present Adress: Department of Physics and Astronomy, Seoul National University, Seoul 151-747, Korea

[‡] Present Address: Polymer Science Program, Department of Chemistry, University of Massachusetts Lowell, 1 University Avenue, Lowell, Massachusetts 01854, USA

[+] These two authors contributed equally to this work.

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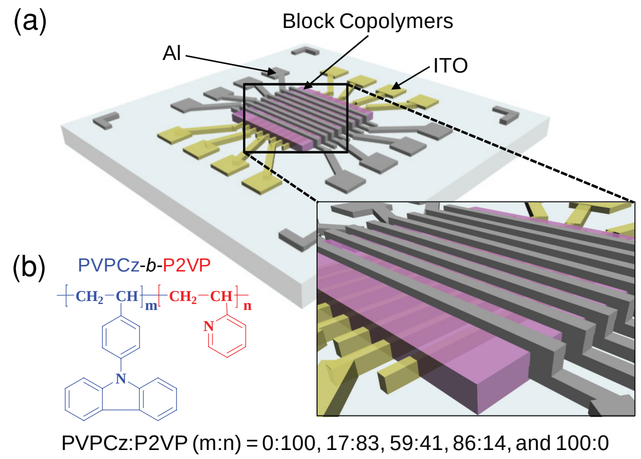

Figure 1. (a) A schematic diagram of the organic memory devices consisting of Al/polymer/ITO junctions in an 8 × 8 array structure. (b) Structure of the block copolymer of PVPCz-*b*-P2VP. Various block ratios (mol%) of PVPCz:P2VP (m:n) = 0:100, 17:83, 59:41, 86:14, and 100:0 were used in this study. m and n indicate block ratio of PVPCz and P2VP in block copolymer, respectively

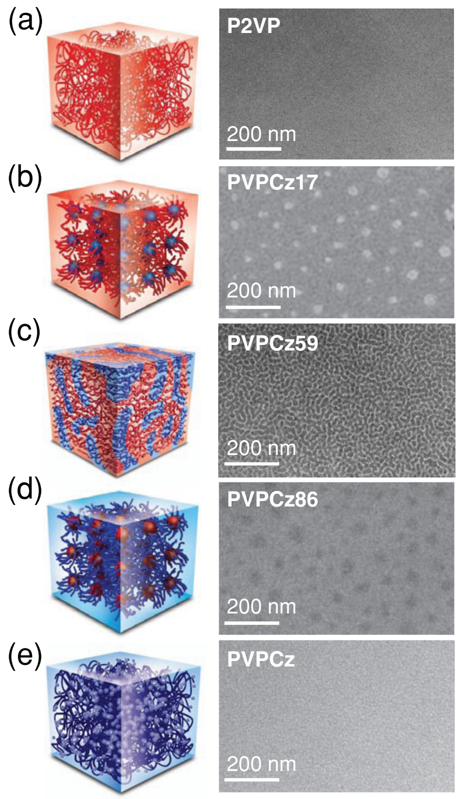

Figure 2. Schematic illustrations (left) and TEM images (right) of PVPCz-*b*-P2VP block copolymers of (a) P2VP (PVPCz:P2VP = 0:100), (b) PVPCz17 (PVPCz:P2VP = 17:83), (c) PVPCz59 (PVPCz:P2VP = 59:41), (d) PVPCz86 (PVPCz:P2VP = 86:14), and (e) PVPCz (PVPCz:P2VP = 100:0). The scale bars on TEM images are 200 nm.

of NVC caused by the high electron density of nitrogen atoms, which act as strong electron donors adjoining the vinyl group.[35] Moreover, the inserted phenyl group in VPCz extends the conjugation length with respect to the structure of NVC to increase the conductivity. The successful living anionic polymerization of 2VP as a very weak electron acceptor[36–38] using an additive of 1,1-diphenylethylene (DPE)[39] was performed without the branching and crosslinking caused by the nucleophilic attack of another carbanion on the pyridine.[40,41] Based on these optimum conditions for the anionic polymerization of VPCz and 2VP, the block copolymerization of VPCz and 2VP was carried out sequentially using DPE after the synthesis of living PVPCz.

In this work, we prepared a total of five different polymer materials (two homopolymers and three block copolymers). The molecular weight and polydispersity of the resulting homopolymers (with block ratios (mol%) of PVPCz:P2VP = 0:100 and 100:0) and block copolymers (with block ratios (mol%) of PVPCz:P2VP = 17:83, 59:41, and 86:14) were measured by gel permeation chromatography (GPC) (see Table S1 in the Supporting Information). For simplicity, we denote the materials with block ratios (mol%) of PVPCz:P2VP (m:n) = 0:100, 17:83, 59:41, 86:14, and 100:0 as P2VP, PVPCz17, PVPCz59, PVPCz86, and PVPCz, respectively.(Figure 1b) The thermal properties of the block copolymers were measured by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). It was verified that the block copolymer PVPCz59 exhibited good thermal stability (see Figure S5 in the Supporting Information). In addition, depending on their block ratios, the block copolymers are soluble in various organic solvents such as tetrahydrofuran, toluene, chloroform, dichloromethane, and 1,2-dichloroethane. For example, 1,2-dichloroethane was used to dissolve the P2VP homopolymer and PVPCz17 block copolymer, toluene was used to dissolve the PVPCz homopolymer and PVPCz86 block copolymer, and a mixed solvent of toluene/1,2-dichloroethane (50/50 v/v) to dissolve the PVPCz59 block copolymer were used to cast transparent and uniform thin films by spin-coating at 2000 rpm for 40 s. Then, five different polymer materials (two homopolymers and three block copolymers) were used as an active layer for the electronic devices as Al/polymer/indium-tin-oxide (ITO) junctions. Figure 1a shows a schematic diagram of the organic electronic devices in an 8 × 8 array structure.

The phase separation of the block copolymers was generally controlled by changing the molecular weight of the polymers, the block composition, and the kind of solvent used for film casting.[42–47] The three block copolymer solutions dissolved in chlorobenzene (40 mg/ml, 4.0 wt%) were drop-casted onto carbon-coated copper grids and annealed at 230 °C under reduced pressure (10^{-1} mmHg) for 3 days. The annealed sample was stained with I_2 vapor for 8 hours, and the excess I_2 was removed under vacuum at 25 °C for 24 h. Micro-phase separations of two homopolymers (PVPCz and P2VP) and three block copolymers (PVPCz17, PVPCz59, and PVPCz86) were confirmed using an energy-filtering transmission electron microscope (EF-TEM), as shown in **Figure 2**.

Because pyridine-containing P2VP block is more polar than carbazole-containing PVPCz block in PVPCz-*b*-P2VP block copolymers, P2VP is soluble in high polar solvent but PVPCz is not. Therefore, the polarity of PVPCz is different from that of P2VP in the selected solvent of chlorobenzene. Each block volume fraction can be different depending on the composition of block copolymers. Annealing temperature is also one of the critical factors to affect the morphology of block copolymer. Accordingly, these critical parameters such as the polarity of PVPCz and P2VP phase in chlorobenzene, volume fraction of each PVPCz and P2VP phase, and annealing temperature of 230 °C under reduced pressure of 10^{-1} mmHg have an important effect on the morphology (lamellar and spherical structure) of the block copolymer. In TEM images (Figures 2b–Figure 2d) of these block copolymers (PVPCz17, PVPCz59, and PVPCz86), the iodine-stained P2VP domains appear as black whereas non-stained PVPCz domains appear as white. These differences in the film morphology can influence dominant charge conduction paths in the devices (see details in the Supporting Information). In contrast, there was no phase separation in the homopolymers (Figures 2a and Figure 2e). Then, using these five different polymer materials, we fabricated memory devices and characterized their electrical properties. Note that we also found that the films made using chlorobenzene and the films made using 1,2-dichloroethane/toluene exhibited similar results for both morphology and device characteristics (see details in the Supporting Information, Figures S8 and S9).

Figures 3a and Figure 3b show the representative current-voltage (I-V) characteristics of devices fabricated from the following materials: P2VP, PVPCz17, PVPCz59, PVPCz86, and PVPCz. The voltage was applied to the top electrode (Al) while the bottom electrode (ITO) was grounded. It was observed that the electrical properties were strongly dependent on the block ratio between PVPCz and P2VP in the block copolymers. Carbazole-containing polymers are one of the most important materials in organic electronics because the carbazole units have stable holes, high charge-carrier mobilities, and a high thermal and photochemical stability.[48] P2VP-containing pyridines used as π-electron-deficient moieties, such as quinoxaline and thiadiazole with an electron-withdrawing imine (–C=N–) structure,[49] can be utilized as n-type materials.[50] At the same time, P2VP is a versatile and well-known functional polymer due to the unshared electron in the pyridine ring. In general, P2VP and poly(4-vinylpyridine) (P4VP) have very low conductivities (almost insulating).[51] However, some groups have reported that P2VP, P4VP, and block copolymers containing pyridine units have some conductivity by the quaternization of pyridine units with methyl iodide (CH_3I), iodine (I_2), and HCl.[52]

As shown in Figure 3a, device-P2VP and device-PVPCz17 maintained low current levels without switching behavior, although device-PVPCz17 containing 17% carbazole showed a slightly higher current level than that of device-P2VP. Therefore, the P2VP in block copolymer act as insulating matrix, so that its contribution to the charge conduction is negligible. On the contrary, device-PVPCz86 and device-PVPCz exhibited high current levels, which are mainly induced by the high-conducting properties of the carbazole unit occupying most of the block copolymer and homopolymer of PVPCz.

An important finding was that device-PVPCz59 exhibited two stable resistance states, depending on the magnitude of the applied voltage, as shown in Figure 3b. The electrical property of the device-PVPCz59 changed from an initial high resistance

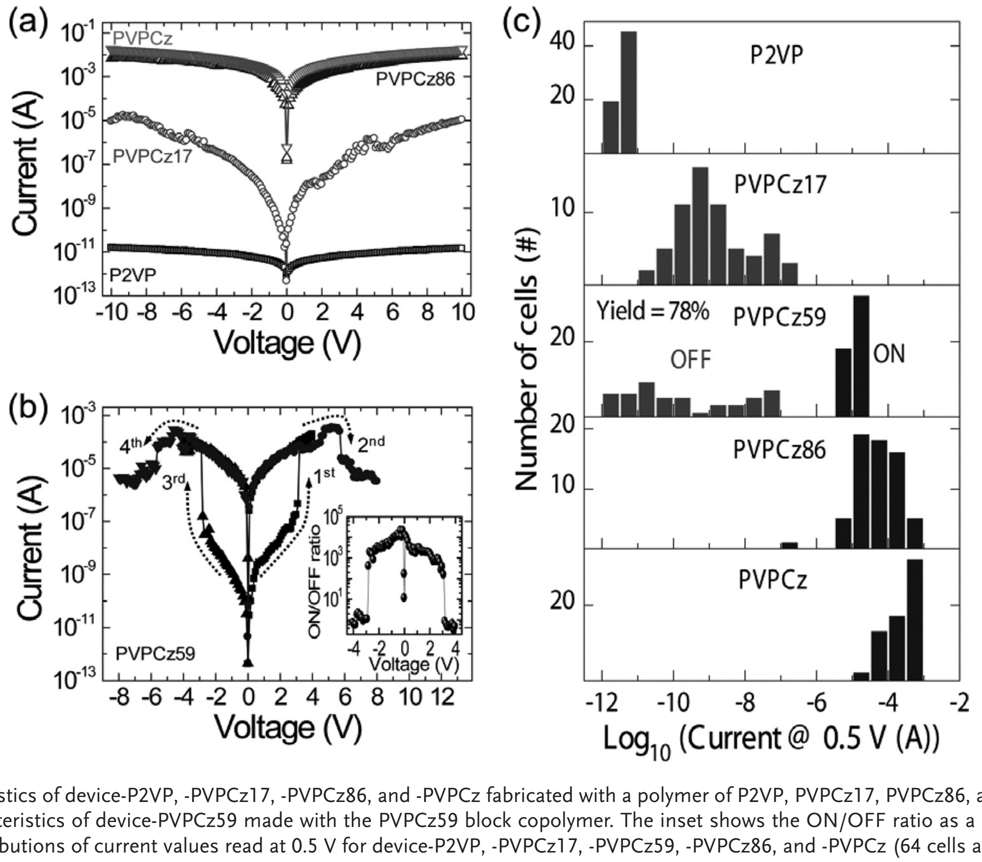

Figure 3. (a) *I–V* characteristics of device-P2VP, -PVPCz17, -PVPCz86, and -PVPCz fabricated with a polymer of P2VP, PVPCz17, PVPCz86, and PVPCz, respectively. (b) *I–V* characteristics of device-PVPCz59 made with the PVPCz59 block copolymer. The inset shows the ON/OFF ratio as a function of voltage. (c) Statistical distributions of current values read at 0.5 V for device-P2VP, -PVPCz17, -PVPCz59, -PVPCz86, and -PVPCz (64 cells at each kind of device; total of 320 cells).

state (HRS, OFF state) to a low resistance state (LRS, ON state) through a double sweep (0 to 4 to 0 V; first sweep), which is associated with the writing process of the memory device. When the higher voltage was applied to the device (second sweep), it remained in the ON state in the low-voltage region and then current decreased rapidly near 6 V. This represents the erasing process. Based on these I-V characteristics, the operating voltages for writing, erasing, and reading were determined to be ~4, 8, and 0.5 V, respectively. The bistability was also observed in the negative voltage region (third and fourth sweeps). As shown in the inset of Figure 3b, the ON/OFF ratio was found to be over 10^3 in the low-voltage range, which is sufficient to distinguish each resistance state.

To investigate the role of phase demixing of the block copolymers in the device characteristics, we investigated the *I–V* characteristics of devices made with a blending system of homo polymer PVPCz and P2VP and devices made with a random copolymer of PVPCz59-*r*-P2VP41 containing PVPCz and P2VP. Both cases did not show any switching behaviors, which indicates the arrangement as well as the amount of each segment in block copolymer is important in exhibiting the resistive switching (see details in the Supporting Information, Figure S10). In addition, to study the thickness dependence of polymer on the switching property, polymer films with different thickness (15, 45, 80, and 155 nm) were prepared. From the I–V characterization, we observed that all the devices made with block copolymer with different thickness exhibited memory switching characteristics. However, OFF current was reduced with increasing polymer thickness and the case with very thick polymer (155 nm) showed somewhat unstable electrical property (see details in the Supporting Information, Figures S11).

Device-PVPCz59 in particular showed typical unipolar switching behavior and nonvolatile memory characteristics (Figure 3b). These properties are essential to realize more practical memory applications involved with a one-diode and one-resistor (1D-1R) memory device architecture, which can prevent the cross-talk leakage path problem.[7,53–55] In general, organic resistive memory devices are categorized into two types of switching behaviors: unipolar and bipolar switching. Unipolar switching operates within a range of voltages that provides the same polarity for the writing and erasing processes[19] while bipolar switching requires both positive and negative voltages.[56] The 1D-1R memory devices, including the bipolar switching memory components, cannot be operated correctly because the erase process is not available due to the suppressed current in the voltage region with negative polarity. To overcome this limitation, the introduction of unipolar switching memory devices is necessary. Our device-PVPCz59 showed unipolar memory behavior, so it can be used in 1D-1R memory devices, and the cross-talk leakage path problem can be prevented.

Considering the fact that the bistability phenomenon in organic devices is sometimes non-reproducible and unreliable, it is important to investigate the cell-to-cell uniformity in devices. Figure 3c shows the statistical data obtained from 64 array cells for each kind of device (a total 320 cells from devices-P2VP to device-PVPCz). With the increase in the carbazole ratio of the block copolymers, the current flowing through the polymers also gradually increased, which is consistent with the *I–V* results shown in Figures 3a and Figure 3b. Although the OFF states of device-PVPCz59 were somewhat widely distributed comparing to the ON states, only device-PVPCz59 exhibited bistable current states. The switching probability of device-PVPCz59 was 50 out of 64 cells (device yield of 78%), which is reasonably acceptable for memory applications. However, we did not observe any bistable switching effect in the other device types (Figures 3a and Figure 3c).

From the statistical results (Figure 3c), we observed that the number of carbazole units as electron donor domains within the block copolymers containing weak electron acceptor domains is an important factor in exhibiting bistable switching. Even though it is possible that the charge transfer effect between carbazole donor and weak pyridine acceptor can occur in the charge complex system, the contribution of the charge transfer on the electrical property might be negligible because of very weak acceptor property of pyridine. The strong insulating property of the pyridine homopolymer supports this expectation. A brief explanation of the potential mechanism for our memory devices is as follows. Resistive switching has often been

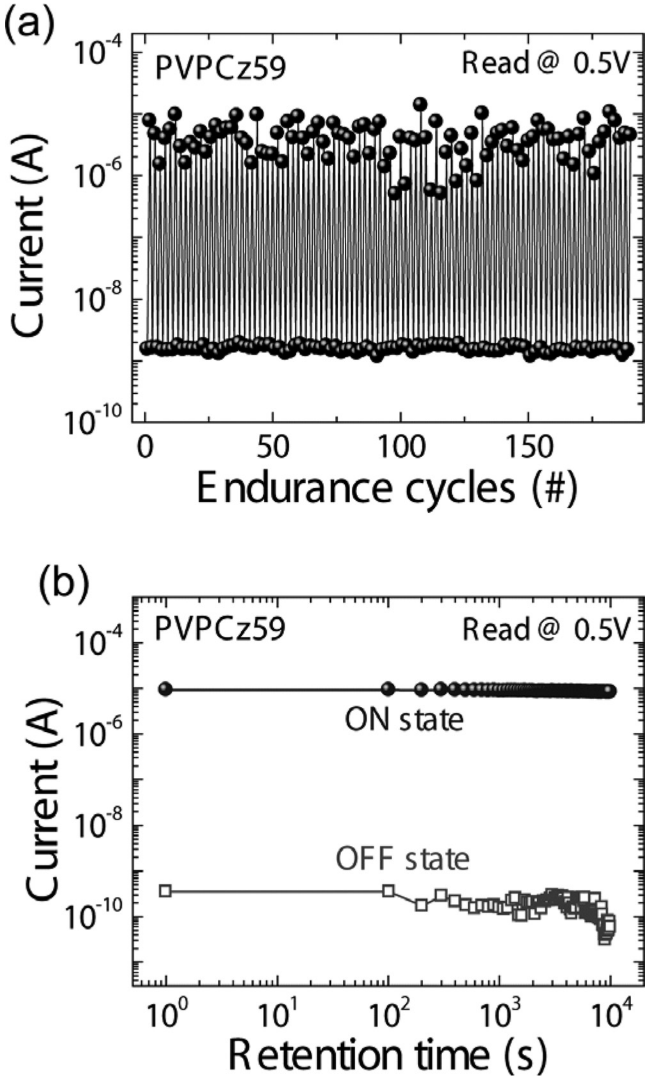

Figure 4. Memory performance of device-PVPCz59 in terms of (a) endurance cycles by repetitive sweeps of a single cell and (b) retention time.

described by a filament model.[57,58] Conductive paths mainly consisting of carbazole segments can be formed or ruptured by an electric field, which results in a reversible resistance change. In particular, filamentary paths are most favorably grown and then easily broken up in lamellar structures with a conducting and insulating layer. Generally, filamentary conduction occurs when the charge conduction is limited to highly localized regions of a junction area. Although the kinds of elements constituting high conductive paths are not known clearly, it is likely that the conductive paths are mainly comprised of carbazole segments and they can be formed or ruptured by the electric field. In particular, the effect of top metal diffusion into block copolymer on resistive switching seems to be negligible because both PVPCz86 and PVPCz17 in our memory structure of ITO/block copolymer/Al did not show resistive switching behavior.

The memory performance of device-PVPCz59 was evaluated in terms of endurance cycles and retention time. The endurance cycling test was performed by repetitive sweeps of a single cell as shown in **Figure 4**a. After writing (4 V) and erasing (8 V) voltages were alternately applied to the top electrode, the two different current states were read at 0.5 V. During 190 sweep cycles, the ON and OFF states of the memory device remained stable with a high ON/OFF ratio of ~4 orders of magnitude without any significant electrical degradation. To evaluate the devices' abilities to retain information, we measured the retention characteristics of the ON and OFF states of the memory device, as shown in Figure 4b. Two current values were also read at 0.5 V after writing at 4 V or erasing at 8 V. The memory device showed a long retention time of 10^4 s, indicating a stable information storage capability.

In summary, we fabricated electronic devices based on a series of synthesized block copolymers (two homopolymers and three block copolymers with different block ratios). Depending on the block ratio, different current-voltage characteristics (metallic, memory, or insulating) were observed. Among these, a block copolymer system exhibiting a lamellar structure (device-PVPCz59, PVPCz:P2VP = 59:41) showed unipolar switching behavior with nonvolatile memory characteristics. In terms of memory performance, reasonably good endurance cycling (>190 cycles) and retention characteristics (>10^4 s) were observed with a high ON/OFF ratio (>10^4) in these devices. Our study provides a simple strategy based on the adjustment of the block ratio in block copolymers to control the polymer morphology and therefore the electrical and switching properties of polymer-based memory devices.

Experimental Section

Synthesis of 9-(4-vinylphenyl)carbazole (VPCz): The synthesis and characterization of 9-(4-vinylphenyl)carbazole are shown in Scheme S1 and Figures S1 and S2 in the Supporting Information.

Synthesis of block copolymers by anionic polymerization: The details of the synthesis of block copolymers, poly(9-(4-vinylphenyl)carbazole)-*b*-poly(2-vinylpyridne) (PVPCz-*b*-P2VP), with various block ratios (mol%) (PVPCz:P2VP = 0:100, 17:83, 59:41, 86:14, and 100:0) by anionic polymerization are summarized in Table S1 and Figures S3 and S4 in the Supporting Information. A series of characterization results for these block copolymers are provided in the Supporting Information, which include molecular weight and molecular weight distribution as determined by GPC (Table S1); thermal properties as determined by TGA (Figure S5a) and DSC (Figure S5b); UV-Vis spectra (Figure S6); and cyclic voltammograms (Figure S7a).

Fabrication and characterization of an organic memory device: Al/PVPCz-*b*-P2VP/indium tin oxide (ITO) devices in an 8 × 8 cross-bar array structure were fabricated. ITO (200 nm) was used as a bottom electrode and was patterned with 8 lines on a glass substrate by conventional photolithography and an etching process. The polymer solution was spin-coated onto the clean ITO-coated glass substrate at 2000 rpm for 40 s. To promote micro-phase separation, it was annealed at 230 °C under reduced pressure (10^{-1} mm Hg) for 3 days. An Al layer 100 nm in thickness was used as a top electrode and was deposited by an e-beam evaporator at a pressure of ~10^{-6} Torr. All electrical measurements were carried out using a semiconductor characterization system (Keithley 4200 SCS) at room temperature in a N_2-filled glove box.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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