Interface-Assisted Room-Temperature Magnetoresistance in Cu-Phenalenyl-Based Magnetic Tunnel Junctions

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ABSTRACT: Delocalized carbon-based radical species with unpaired spin, such as the phenalenyl (PLY) radical, have opened avenues for developing multifunctional organic spintronic devices. Using direct laser writing and in situ deposition, we successfully fabricated Cu-PLY- and Zn-PLY-based organic magnetic tunnel junctions (OMTJs) with improved morphology and a reduced junction area of 3 × 8 μm². The nonlinear and weakly temperature-dependent current−voltage (I−V) characteristics in combination with the low organic barrier height suggest tunneling as the dominant transport mechanism in the structurally and dimensionally optimized OMTJs. Cu-PLY-based OMTJs show significant magnetoresistance up to 14% at room temperature due to the formation of hybrid states at the metal−molecule interfaces called “spinterface”, which reveals the importance of spin-dependent interfacial modification in OMTJs’ design. Additionally, at high bias, in the absence of a magnetic field, OMTJ shows stable voltage-driven resistive switching. Cu-PLY having spin 1/2 with net magnetic moment demonstrates magnetic hardening between the surface molecule at the Co interface and gives rise to stable MR, which suggests its use as a feasible and scalable platform for building molecular-scale quantum memristors and processors.

KEYWORDS: organic magnetic tunnel junctions, spinterface-induced magnetoresistance, resistive switching, magnetic exchange coupling, direct laser writing, molecular spintronics

INTRODUCTION

Organic spintronics, a nascent field at the crossover of spintronics with organic electronics and magnetism, has attracted extensive interest and gradually developed into a new potential platform for future information technologies1−10 due to the intriguing properties of organic materials such as long-spin coherence times,11−13 tunability of the magnetic properties by molecular design, and high chemical diversity.14 Organic magnetic tunnel junctions (OMTJs),15 whereby a thin layer of organic molecules is sandwiched between two ferromagnetic (FM) electrodes, have received immense attention due to their mechanical flexibility, chemically tunable electronic property, and structural fabricability.16−18 After the first report of magnetoresistance (MR) in an organic junction,19 many groups have reported MR measurement by studying the spin injection and transport in OMTJs with different organic materials.2,5,20−24 The performance of such devices depends not only on the properties of the organic molecule as the tunnel barrier and magnetic injecting electrodes but also spectacularly on the interfacial properties in the hybrid region of devices’ so-called “spinterface”.3,25 This region, which arises from orbital hybridization between organic molecules and spin-split bands of the ferromagnet, can drastically influence the spin transport properties of devices. Consequently, the accurate design of the spinterface is crucial and for that some considerations must be made simultaneously, that is, the energy level alignment for facilitating the carrier injection and the spin injection/extraction and modification of the magnetic properties of FM electrode/organic molecule interfaces.26

Extensive investigation has been performed to improve the spinterface and hence the tunnel MR (TMR) value in OMTJs.20,27−35 Despite getting a high TMR signal in OMTJ at 2 K25 and 11 K1, there are very few reports at room temperature,36 the main reason being instability of organic molecules at room temperatures, leading to the formation of a
poor-quality interface between FM and organic molecules. An oxide layer of 1–2 nm is often inserted between the organics and the FM contact to enhance the MR effect,27,37−40 but their transport properties remain unclear. To explore the MR due to the interfacial effect between FM and organic materials, there is a strong need to design organic molecules, which forms a stable interface up to room temperature.

Delocalized carbon-based radical molecules with an unpaired free electron, such as phenalenyl (PLY)-based radicals,41,42 provide novel schemes for building organic spintronic devices43,44 because their spin structure can be manipulated by external stimuli (such as light, electric, and magnetic fields). It has been shown that PLY coordinated with zinc ion (ZMP)-based tunnel junction generates 20% MR near room temperature.43,45,46 These advancements inspired us to explore PLY and its transition metal-based complex molecule-based OMTJ, that is, Cu-PLY. Contrary to Zn-PLY, Cu-PLY is a typical coordination complex; hence, Cu-PLY is air- and moisture-stable, which is one of the main reasons to explore this molecular system. Furthermore, Cu-PLY is paramagnetic (χ > 0) with spin 1/2 net magnetic moment; therefore, the interaction of these molecules with the magnetic field would be different. Therefore, Cu-PLY is expected to modify the interaction at the interface and influence the spin transport.

For this purpose, we developed a three-dimensional (3D) mask with two-photon absorption lithography (TPL).47 Low-temperature [(LT) ∼ 80 K] in situ UHV angle deposition method combined with a 3D-mask was used to fabricate OMTJs with an effective area of 3 × 8 μm². These devices show an MR ratio ∼ 10−14% without the use of oxide layers between the FM/organic interface at room temperature, which to our knowledge is one of the highest MR ratios reported.48−50

Fabrication and Optimization of OMTJs. We used the TPL technique to fabricate 3D masks. The steps of the fabrication process are shown in Figure 1. The use of 3D mask ensures precise control of the cross-sectional dimensions as well as tuning the overall size of 3D molecular junctions. Using the 3D mask and angle deposition method as explained in the Supporting Information (Figure S3), we fabricated multiple sets of wedge devices (Figure S4) in two different categories. In the first category, wedge devices consist of Co (8 nm)/PLY (Cu, Zn, 2–6 nm)/Co (12 nm)/MgO (4 nm), and in the second, Co (8 nm)/Cu-PLY (2–6 nm)/Cu (12 nm)/MgO (4 nm). In the latter, we replaced top magnetic electrodes with a non-magnetic copper electrode. Here, a thin layer of MgO (∼4 nm) works as a capping layer to protect the devices from degrading due to the interaction with the environment. Co/Cu electrodes and the capping layers were deposited by e-beam evaporation. The organic materials were deposited by thermal evaporation with a substrate temperature of ∼80 K. The optical image of a single OMTJ having two Co electrodes with an organic barrier layer is shown in Figure 1. The area was decreased by a factor of 10³ to 10⁴, through the 3D printing of the mask and the new shadow evaporation technique, demonstrated for the first time.

The structures of PLY and its transition-metal-based complex molecules (Cu-PLY and ZMP) are shown in Figure 2a. To get a working MTJ, a very thin barrier layer is required.
because tunneling is expected to dominate in thin barrier junctions and decays exponentially with increasing thickness.

In Figure 2b, the resistance of the OMTJs are plotted as a function of the thickness of the different organic layers. Accordingly, for barrier layer thickness below ≈2.5 nm, the resistance is low (100−200 Ω) because of short-circuits via pinholes or diffusion of top electrodes in the organic layer. The abrupt resistance jumps from kΩ to MΩ occur at thickness ≈4.4 nm; afterward, by increasing the thickness up to ≈6 nm, the resistance increases linearly in logarithmic scale from kΩ to GΩ. For a thickness more than ≈6 nm, the resistance is in the range of more than 100 GΩ, with almost no detectable current below 1 V bias. With this optimization, the organic barrier thickness of ≈5 nm is selected for the OMTJ device.

In order to get more insights into the origin of spin transport through organic layers, we performed current−voltage (I−V) curves in the low-voltage range at 200−293 K using a home-built four-point probe cryostat setup. In general, the spin-dependent transport phenomenon in OMTJs is governed by tunneling and hopping mechanisms.

Inspection of the data in Figure 2b,c reveals that for organic-layer thickness ranging ≈4.7−5.6 nm, the J−V curves are clearly non-linear and symmetric, and resistance has a weak temperature dependence with negligible resistance variation at 293 and 200 K. This excludes the thermally activated hopping as the dominating transport mechanism. The J−V curves of OMTJ junctions with area A (J = I/A) are well-matched with the theoretical Brinkman model based on the Wentzel−Kramers−Brillouin (WKB) approximation for estimation of barrier height “φ” (eV) and tunneling barrier thickness “d” (nm) (details are shown in Table S1). These results clearly show that the tunneling is the dominating transport mechanism in the optimized OMTJs discussed in this report.

TMR of Cu-PLY-Based OMTJs. To further explore the spin transport properties of our OMTJs, we focus to investigate the TMR with the Cu-PLY molecule being the barrier layer. Cu-PLY synthesis and its characterization is explained in Figures S1 and S2, and Figures S9−S13 and crystallography details are given in Table S2 to Table S5. In such systems, the TMR arises from the magnetization-dependent total density of states of the tunneling electrons at the surface. The transmission electron microscopy (TEM) images of Cu-PLY-based OMTJs show that all the interfaces are sharp and without interdiffusion, see Figure 3a. The nonlinear I−V characteristics with φ = 1.32 eV suggest tunneling as the dominating transport mechanism, see Figure 3b. Moreover, the magnetic properties of the OMTJ were characterized using magneto-optical Kerr effect (MOKE) measurement technique at room temperature. The hysteresis loops for the top and bottom electrodes of the device in Figure 3c shows coercive field of $H_c \sim 10$ mT and $H_c \sim 15$ mT, respectively, although as we showed in Figure S5, for 8 nm Co thin film $H_c \sim 6$ mT. This suggests that addition of the Cu-PLY layer enhances the coercivity/switching field of Co (also with ZMP and PLY shown in Figure S5). A significant change in the MOKE curve is a signature of the molecules anisotropy. The magnetic moment/coercive field modification depend strongly on the hybridization between the $\pi$-electrons of the Cu-PLY molecular layers and the 3d-band electrons of the Co metal. Furthermore, as a result of the hybridization, the electronic states at the interface change, leading to a new hybrid interface called "spinterface". 

![Figure 3](image-url)
Figure 4. (a) Resistance variation with applied magnetic field for OMTJ Co/Cu-PLY/Cu/MgO at room temperature showing an IMR effect. (b) Schematic of two layers of Cu-PLY adsorption (top image) on the Co surface (side view) and the bottom image shows the top view of the Cu-PLY molecular structure adsorbed on the Co surface.

Variation of the resistance as a function of the in-plane magnetic field amplitude is shown in Figure 3d. The TMR can be defined as TMR = \( \frac{(R_{AP} - R_P)}{R_P} \times 100 \), where \( R_P \) and \( R_{AP} \) are the tunneling resistance when magnetization of the two electrodes are aligned in parallel (p) and antiparallel (AP), respectively. We found TMR \( \sim 14\% \) for Cu-PLY-based OMTJ, which lies among the highest value obtained until now in a device without adding any oxide tunnel barrier between the FM/organic interface at room temperature (to compare MR results, PLY- and ZMP-based OMTJs are shown in Figure S7). This is a promising finding for the development of future logic devices by increasing the material choice as it is very limited at present.

To understand the mechanism behind TMR signal with Cu-PLY OMTJs, we used a model similar to the one discussed in Raman et al.\(^5\) as the Cu-PLY molecule is planar molecular (crystallographic picture see in Figure S10) like ZMP. The origin of the MR signal can be explained based on the spin selective transport through the hybrid interface (see Figure 3e). Figure 3e shows schematically the Co Fermi level \( E_F \) and the lowest unoccupied molecular orbitals (LUMOs), highest occupied molecular orbitals (HOMOs), and semi-occupied molecular orbitals (SOMOs) of the radical state of the Cu-PLY molecule. As the HOMO is fully occupied, the SOMO of the molecule with a free electron with spin up or down participates in transport. It is very well established that the SOMO of the odd alternant hydrocarbon PLY-based radical is formally a nonbonding molecular orbital (NBMO) and hence during transport, it experiences a minimal change in reorganization energy.\(^5\)\(^4\) Due to the strong exchange bias coupling of the Cu-PLY molecule to the Co surface (as we see in Figure 3c) a new hybrid interface is formed. When the spin of Co and the SOMO level of this hybrid molecular interface are aligned antiparallel (Figure 3e left), the spin-up electron from the metal can be injected into the SOMO hence showing lower resistance. On the other hand, when the Co layer and the interface layer are aligned parallel (Figure 3e right), absence of required empty spin state in the SOMO level of the hybrid molecular interface requires tunneling via LUMO, which leads to a higher resistive state. Theoretical calculations and atomic scale experiments (STM measurements)\(^5\)\(^5\)\(^5\) demonstrated that by chemisorbing organic molecules containing metal atoms onto a magnetic substrate a spin unbalanced interaction is induced into the \( \pi \)-orbitals molecular orbitals and metal d-states. As a consequence, the chemisorbed molecule acts as an efficient spin-filter.\(^4\)\(^5\)\(^5\)\(^5\)\(^6\) Consequently, the Cu-PLY layer can be a spin filter with a high MR effect at room temperature.

To further explore the spinterface properties, we replace the top magnetic electrode with a non-magnet Cu electrode which gives interface-induced MR. Figure 4a shows independent switching of the bottom Co with respect to the hybridized interface layer, giving rise to the interface MR (IMR) effect. This happens due to the strong \( \pi \)-d hybridization between molecular \( \pi \)-orbitals and the Co d-states which alter magnetic properties of Co surface.\(^3\) IMR ratio IMR = \( \frac{(R_{AP} - R_P)}{R_P} \times 100 \sim 14\% \), where \( R_P \) (as see in Figure 4a, \( R_P = \frac{R_{AP} + R_{UP}}{2} \)) and \( R_{AP} \) are the resistance when magnetization of Co electrode and interface are aligned in P and AP states, respectively.

The interaction of the PLY part of the molecule with the Co substrate is rather similar like when the Zn-PLY system interacts with the Co surface.\(^5\) In Figure 4b, top schematic shows the adsorption of the Cu-PLY molecule (which is planar molecules) on the Co surface. The PLY ligand chemisorbs on the magnetic substrate due to the strong hybridization between the \( \pi \)-orbitals of the PLY-ligand and d-states of Co surface atoms shown. More precisely, in the case of other \( \pi \)-conjugated molecules or graphene flakes that interact with magnetic surfaces and magnetic exchange interactions drastically increase and giving rise an intra-layer magnetic hardening\(^5\)\(^7\)\(^9\) leading to the formation of nanomagnets embedded in the magnetic substrates. Interestingly, this hybrid molecule–surface nanomagnets can switch independently with respect to the surrounding magnetic substrate\(^5\)\(^3\)\(^5\)\(^7\)\(^6\) and also represents a key effect for the new devices discussed in this work. It is worth mentioning that by controlling the Co/Cu-PLY hybrid interface, spinterface effect higher IMR could be accessible. We found evidence that multiple 100% IMR could potentially be realized, consistent with the spinterface having a spin blockade effect.

Additionally, with the MR effect, as shown in Figure 5, the \( I-V \) characteristics of the Co/Cu-PLY/Co junction demonstrate a novel preliminary electric memory effect. By increasing the bias voltage from zero to positive voltages, it can be seen that the device is in a low conductivity state with less than half of the resistance. The current sharply increases from 20 to 50 mA. Within a hysteresis, when the voltage is decreased back to zero, the device returns to the high conductance state. A similar process occurs at negative voltages. This \( I-V \) behavior at high bias, indicates that the device resistance can increase or
represent an ideal material platform to develop high-quality magnitude of MR by engineering the interfacial properties and potential for controlling parameters such as the sign and molecule. Most importantly, our results demonstrate its and memristor devices.

development of room-temperature molecular memory logic spinterface effects. These findings pave the way for the among the highest values reported so far in devices without an interface coupling, and magnetic anisotropy is observed. We of the molecule, that is, with Cu-PLY, evidently changes the mechanism in optimized OMTJs. Tuning the function of the shown tunneling as the dominant electron/spin transport tunnel barrier thickness of ∼14% for OMTJs, which lies and in situ LT OMTJ growth; initial electrical and magnetic properties; and resistive switching of PLY and ZMP.

**CONCLUSIONS**

Using a 3D mask and LT in situ deposition, we successfully fabricated PLY-, Cu-PLY-, and Zn-PLY-based OMTJs without adding any additional interface separation. With an optimal tunnel barrier thickness of ∼5 nm, our studies (I–V) have shown tunneling as the dominant electron/spin transport mechanism in optimized OMTJs. Tuning the function of the PLY-molecule by their chemistry, by adding Cu at the center of the molecule, that is, with Cu-PLY, evidently changes the interface coupling, and magnetic anisotropy is observed. We found room-temperature TMR ∼ 14% for OMTJs, which lies among the highest values reported so far in devices without an oxide layer at the metal/molecule interface, and OMTJ with the Cu top electrode shows IMR ∼ 10%. We demonstrated a memristive behavior at higher voltages, allowing the manipulation of the interface charge in future works. The memristive aspect is a completely new and very exciting result and is directly related to the properties of the interfacial nature of the molecule. Most importantly, our results demonstrate its potential for controlling parameters such as the sign and magnitude of MR by engineering the interfacial properties and represent an ideal material platform to develop high-quality spininterface effects. These findings pave the way for the development of room-temperature molecular memory logic and memristor devices.

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsaelm.2c01428.

Cu-PLY synthesis methods, crystal structure, and characterization data; fabrication method of 3D mask and in situ LT OMTJ growth; initial electrical and magnetic properties; and resistive switching of PLY and ZMP.

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**Author Contributions**
M.M and S.K.M designed the original research approach of this work. M.M, C.D, and N.J conceived the idea to study spin-dependent transport on OMTJ. C.D and N.J designed the experiment and fabrication of devices with 3D mask technology. N.J carried out the experiments. N.J, C.D, and J.S.M analyzed the data. N.J, M.M, and T.S.P wrote the manuscript including contribution from all the authors. A.P synthesized the Cu-PLY molecule, G.V. and P.K.V characterized the Cu-PLY molecule by single-crystal X-rays, and

![Figure 5. I–V room-temperature characteristics at H = 0. The irreversibility is clearly visible at both positive and negative voltages (bias). Inset: I–V characteristics from 3 to 8 V reflect that at low voltage range there is no hysteresis appeared.](https://pubs.acs.org/acsaelm/2023/5/1471/acsaelm.2c01428)
P.K.V prepared PLY, Zn-PLY, and Cu-PLY molecules. N.A provides supporting qualitative theory for the Co/Cu-PLY hybrid system. S.K.M supervised the molecular materials preparation and characterization. A.A and T.M. and M.S. carried out TEM measurements.

Notes
The authors declare no competing financial interest.

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