Temperature dependence of transport mechanisms in organic multiferroic tunnel junctions

Can Xiao\textsuperscript{1}, Huawei Sun\textsuperscript{1}, Luming Cheng\textsuperscript{1}, Xavier Devaux\textsuperscript{2}, Anthony Ferri\textsuperscript{3}, Weichuan Huang\textsuperscript{4}, Rachel Desfeux\textsuperscript{3}, Xiao-Guang Li\textsuperscript{4}, Sylvie Migot\textsuperscript{2}, Mairbek Chshiev\textsuperscript{5}, Sajid Rauf\textsuperscript{1}, Yajun Qi\textsuperscript{1}, Ruilong Wang\textsuperscript{1}, Tianjin Zhang\textsuperscript{1}, Changping Yang\textsuperscript{1}, Shiheng Liang\textsuperscript{4}\textsuperscript{2}, and Yuan Lu\textsuperscript{2}\textsuperscript{\dagger}

\textsuperscript{1} Faculty of Physics and Electronic Science, Hubei University, Wuhan 430062, People’s Republic of China

\textsuperscript{2} Université de Lorraine, CNRS, Institut Jean Lamour, UMR 7198, Campus ARTEM, 2 Allée André Guinier, BP 50840, Nancy 54011, France

\textsuperscript{3} University Artois, CNRS, Centrale Lille, ENSCL, University Lille, UMR 8181, Unité de Catalyse et Chimie du Solide (UCCS), Lens F-62300, France

\textsuperscript{4} Hefei National Laboratory for Physical Sciences at Microscale, Department of Physics, University of Science and Technology of China, Hefei 230026, People’s Republic of China

\textsuperscript{5} University Grenoble Alpes, CEA, CNRS, Spintec, Grenoble 38000, France

E-mail: yuan.lu@univ-lorraine.fr and shihengliang@hubu.edu.cn

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Abstract

Organic multiferroic tunnel junctions (OMFTJs) with multi-resistance states have been proposed and have drawn intensive interests due to their potential applications, for example in memristor and spintronics based synapse devices. The ferroelectric control of spin polarization at a ferromagnet/ferroelectric organic (FE-Org) interface by electrically switching the ferroelectric polarization of the FE-Org has been recently realized. However, there is still a lack of understanding of the transport properties in OMFTJs, especially the interplay between the ferroelectric domain structure in the organic barrier and the spin polarized electron tunneling through the barrier. Here, we report on a systematic study of the temperature dependent transport behavior in La\textsubscript{0.6}Sr\textsubscript{0.4}MnO\textsubscript{3}/PVDF/Co OMFTJs. It is found that thermal fluctuation of the ferroelectric domains play an important role on the transport properties. When T > 120 K, the opposite temperature dependence of resistance for up and down ferroelectric polarization states results in a rapid diminishing of the tunneling electroresistance. These results contribute to the understanding of the transport properties for designing high performance OMFTJs for memristor and spintronics applications.

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(Some figures may appear in colour only in the online journal)

1. Introduction

Organic multiferroic tunnel junctions (OMFTJs) [1–5] have recently attracted much attention due to their synergetic advantages for spintronics, organic electronics, and ferroelectric electronics. In comparison to inorganic based devices, organic materials are appealing because of the long spin lifetime of charge carriers [6], low cost and chemical diversity.
[7, 8]. In an OMFTJ, the core structure comprises a ferroelectric organic (FE-Org) tunnel barrier layer sandwiched between two ferromagnetic (FM) electrodes. Owing to the combined tunneling magnetoresistance (TMR) and tunneling electroresistance (TER) effects [9–11], it shows a performance of a multi-resistance switching behavior. Therefore, the magnetic and ferroelectric dependent nonvolatile multi-states properties can be utilized to develop spintronics-based artificial intelligence devices, such as synapses [11, 12]. More interestingly, it has been demonstrated that the spin polarization at the FM/FE-Org interface, such as ‘spinterface’, can be inverted by electrical switching of the ferroelectric polarization of the FE-Org [1]. This discovery emphasizes the critical role of ‘spinterface’ [13, 14] on the determination of the spin polarization at the organic/ferromagnetic interface and opens up new functionality in controlling the injection of spin polarization into organic materials via modulating the ferroelectric polarization of the barrier.

In the inorganic all-perovskite multiferroic tunnel junction (MFTJ), the temperature dependence of transport properties and mechanisms have been studied [15, 16]. The structural defects such as oxygen vacancies in the oxide ferroelectric barriers were found to lead to a thermally activated inelastic hopping through chains of localized states in thicker barriers and at higher temperatures. This conduction channel is not sensitive to the ferroelectric orientation and cannot carry spin polarization information, which reduces the TMR and TER effects significantly at higher temperatures [17]. For OMFTJ, the replacement of an inorganic ferroelectric barrier by organic polymer material should introduce different mechanisms to influence the temperature dependent transport behavior. However, up to now, there is still a lack of illumination on this issue. In this work, we have measured temperature dependence of transport behavior in OMFTJs, which shows that different mechanisms dominate the transport properties at different temperature ranges. The understanding of the transport properties is essential for designing advanced OMFTJs for memristor and spintronics applications.

2. Experimental methods

In our experiments, OMFTJ samples based on La$_{0.6}$Sr$_{0.4}$MnO$_3$ (LSMO)/Polyvinylidene fluoride (PVDF)/Co/Au structure were fabricated. The sample structure is shown in figure 1(a). The LSMO film with thickness of ~85 nm was firstly epitaxial grown on <100> oriented SrTiO$_3$ (STO) substrates at 750°C using DC magnetron sputtering. The film was subsequently annealed at 800°C for two hours in O$_2$ atmosphere before being slowly cooled down to room temperature. For the devices prepared for transport measurements, the LSMO layer was etched by using hydrogen chloride (37%) to pattern 200 µm width bars as the bottom electrodes. The PVDF film was prepared by spin-coating method. The solution was obtained by dissolving PVDF powders (purchased from Sigma-Aldrich) into N,N-dimethylformamide (DMF). The solution was then spin-coated onto the LSMO/STO(001) substrate with a speed of 3000 RPM for 1 min. Subsequently, the as-coated film was annealed at 150°C in air for two hours to improve the crystallinity of the ferroelectric β-phase. The thickness of the PVDF film was controlled by adjusting the concentration of the DMF:PVDF solution. With the concentration of 20 mg ml$^{-1}$, the thickness of PVDF is estimated to be about 20 nm. After spin-coating the PVDF layer, the 10 nm Co/10 nm Au was deposited by molecular beam epitaxy (MBE) with a shadow mask to form the top electrode. In the purpose of minimizing the metal diffusion into organic material, the temperature during the growth of the top electrode was maintained at ~80 K by cooling the substrate with liquid nitrogen. The final junction, which is schematically shown in figure 1(a), has a typical size of about 200 × 200 µm$^2$.

High-resolution scanning transmission electron microscopy (HR-STEM) was performed by using a probe-corrected microscope JEOL ARM200F (cold FEG) equipped with a GATAN GIF Quantum energy filter to reveal the structure and element distribution in the FM/FE-Org interface. Thin lamella were prepared by conventional focused ion beam milling with an FEI Helios Nanolab dual beam microscope. Pt-C deposit was used as a capping layer. Due to the strong sensitivity of polymers to electron-beam damages, the microscope was operated at 80 kV and the temperature of the sample was held at 103 K using a Gatan double-tilt liquid nitrogen cooling sample holder. Electron energy loss spectroscopy spectrum images (EELS-SI) were denoised using a principal component analysis before being processed. Before observation, the lamella were plasma-cleaned under Ar-O$_2$ atmosphere for 2 min.

Piezoresponse force microscopy (PFM) was used to check the ferroelectric properties of the PVDF film. For this study, the PVDF film was spin-coated on a thermal oxidized SiO$_2$/n-Si substrate covered with 100 nm Au. The surface morphology was studied using an atomic force microscopy (AFM) (Asylum Research/Oxford Instruments, MFP-3D, USA) working in contact mode under environmental conditions. Local electrical experiments were performed by using dual AC resonance tracking (DART) PFM.

The magneto-transport measurements were performed in a closed cycle cryostat by varying temperature from 10 K to 300 K in the presence of magnetic field up to 4 kOe. I–V measurements were performed using Keithley-2400 as a voltage source and a Keithley-6487 picoammeter to measure the current. Since junction resistance is much larger than electrode resistance, a two terminal configuration has been used for I–V characterization. To polarize the PVDF barrier, different amplitudes of voltage pulses with a ramp of 0.1 V s$^{-1}$ and a duration of 1 s were applied to the junction. It was verified with conductive AFM that the polarizing voltage around ±3 V range does not induce damages to the organic barrier with the formation of pinholes inducing leakage currents.

3. Results and discussion

The surface morphology of the PVDF barrier layer has been investigated by AFM observation. It reveals a smooth surface
with root mean square (RMS) roughness <3.5 nm in 1 × 1 μm² scanning range, as shown in figure 1(b). The surface morphology exhibits a feature of grains composed by needle-like crystallites with a width of ~30 nm and length of ~100 nm, which is characteristic of the ferroelectric β-phase of PVDF [18]. The ferroelectric properties of the PVDF barrier have been characterized by PFM to check the homogeneity in ferroelectric polarization switching. The PFM phase map was recorded over 10 × 10 μm² PVDF surface area, as shown in figure 1(c). The film was initially polarized with a biased tip at positive voltage (+3 V), then an opposite poling bias (~3 V) over an inside area of 5 × 5 μm². As observed, the clear switching of the polarization is obtained by applying positive and negative bias voltages, suggesting a homogeneous polarization switching.

Figure 2(a) shows a bright-field STEM image of the sample. The bottom LSMO magnetic electrode revealed a good homogeneity with a thickness of ~85 nm and low roughness of about 3 nm. The spacer layer PVDF appears homogenous without pinholes with a thickness of ~20 nm. This also validates the homogeneous ferroelectric switching measured by PFM phase mapping (figure 1(c)). In addition, we have performed the chemical analysis by using electron energy loss spectroscopy (EELS) to check the Co diffusion in the organic barrier. Figure 2(b) shows an high angle annular dark-field (HAADF) STEM image where EELS spectrum image was recorded. For the three regions marked with orange, red and blue squares on the STEM image, EELS spectra are collected and presented in figure 2(c). Although the Co signal is well distinguished in the region close to Co/PVDF interface (orange zone), we cannot find any Co signal in the middle of PVDF barrier (red zone). There are also no La and Mn signals in PVDF barrier compared to the strong signals from the region close to PVDF/LSMO interface (blue zone). The chemical EELS maps of Co/PVDF/LSMO interface is presented in figure 2(d). The maps of chemical elements Co (yellow), F (red) and La (sky-blue) are extracted from the EELS spectrum images by using signals of CoL3 (~779 eV), FK (685 eV) and LaMs (~820 eV) edges, respectively. It also appears that there is very small Co diffusion inside the organic barrier and the interface PVDF/LSMO is quite sharp. All these results prove that there is very limited Co diffusion into the PVDF organic barrier when Au/Co electrode is deposited at low temperature, which agrees well with our previous report in P(VDF-TrFE) based OMFTJ [4].

Figure 3(a) shows the I–V characteristic of the device measured at different temperatures under a magnetic field of 2 kOe (in the parallel state). The device has been only initially polarized by +1.5 V at 15 K. It can be seen that the I–V curves show non-linear properties which indicate a tunneling behavior. We have also plotted the bias dependent differential resistance (dV/dI) as a function of temperature in figure 3(b). It is found that the resistance decreases at larger bias, which can be understood due to the deformation of barrier height at larger bias. It is interesting to find that the temperature dependent behavior is different for small and large bias. Figure 3(c) displays the differential resistance as a function of temperature for zero bias and +0.5 V bias. At zero bias, the resistance varies in three temperature regions: firstly decreasing from 15 K to 40 K, and then increasing between 40 K and 120 K, followed by a decrease up to 300 K. However, the resistance at +0.5 V bias shows an almost monotonous increase with temperature. This could be due to the influence of large bias on the ferroelectric domain in PVDF barriers, and this effect could be more pronounced at high temperature [19]. In the following part, we will discuss more details for the temperature dependent resistance.
Figure 2. (a) Large scale bright-field STEM image of the LSMO/PVDF/Co structure. The PtC layer deposited during the lamella preparation appears on the top of the image. (b) HAADF STEM image of the sample. The red frame indicates where an EELS-SI was recorded. The colored squares shows the area where EELS spectra in (c) were extracted. (c) Typical EELS spectra of the Co/PVDF interface, the PVDF layer and the PVDF/LSMO interface. (d) Elemental map drawn from quantitative analysis of EELS-SI.

Figure 3. (a) Temperature dependence of $I$–$V$ characteristics under the range between $-0.5$ V to $+0.5$ V. (b) Differential resistance as a function of bias at different temperatures. (c) Temperature dependence of differential resistance at zero bias and $+0.5$ V.

Figure 4 shows the magneto-response loops measured at 10 K after different polarizations ($+1.2$ V for positive polarization and $-1.5$ V for negative polarization, respectively). When scanning the magnetic field, the magnetization of LSMO and Co are switched separately to get a parallel or antiparallel magnetization configurations and results in the TMR curves. The TMR is defined by the following relations: $TMR = \frac{R_{AP} - R_P}{R_P} \times 100\%$, where $R_P$ and $R_{AP}$ are junction resistance where the magnetizations of two electrodes are parallel and antiparallel, respectively. It is clear that the junction shows a positive TMR of $+8.3\%$ when the PVDF is positively polarized and a negative TMR of $-15.3\%$ when the PVDF is negatively polarized. At the same time, a clear difference between the two parallel resistances ($R_P$) for the two polarizations results in a TER about $65\%$. The TER is defined as: $TER = \frac{R_{Down} - R_{Up}}{R_{Up}} \times 100\%$, where $R_{Down}$ and $R_{Up}$ are parallel resistance in PVDF ‘down’ and ‘up’ polarization states, respectively. The four resistance states associated with different magnetization and ferroelectric configurations are identified in the inset of figure 4, which clearly demonstrates the MFTJ function of our LSMO/PVDF/Co organic tunnel junction. To validate that the measured TMR is not due to the anisotropic magnetoresistance (AMR) of FM electrodes, we have checked the AMR of LSMO and Co electrodes, which is
much smaller than the observed TMR (see supplementary S1 (stacks.iop.org/JPhysD/53/325301/mmedia)).

The change of TMR sign with different PVDF polarizations can be understood as due to the sign change of the spin polarization at the organic/ferromagnetic ‘spinterface’ depending on the ferroelectric polarization of the organic barrier [1]. Due to the strong hybridization at organic/ferromagnetic interface, there exist strong spin-dependent metal induced gap states (MIGS) inside PVDF at Co/PVDF interface. The decay rate of spin up and spin down MIGS can be changed upon the ferroelectric polarization states, which results in a sign change of spin polarization in different PVDF layer. As shown in the schematics in figures 1(d) and (e), the sign of the spin polarization of the second C layer can be changed due to the switching of PVDF polarization, which acts as a final spin-filter for electrons tunneling through the PVDF barrier by considering Jullière’s model [20]. We have verified the stability of ferroelectric repetition and the reproductivity of TMR sign change with another device with a thinner PVDF thickness (15 mg m\(^{-1}\)) (see supplementary S2).

Figure 5(a) shows the magneto-response loops at different temperature for both polarization states. We have firstly polarized PVDF in both polarization states at 10 K and then increased the temperature separately in each polarization state. It is found that the TMR values in both polarization states decrease monotonously when increasing the temperature (figure 5(b)). The positive TMR completely vanishes at temperature higher than 150 K while the negative TMR can persist even at 250 K even though becoming small after 200 K. This behavior generally occurs for the organic tunneling junction using the LSMO electrode [1, 14, 21]. The reduction of TMR at a high temperature can be understood by the reduction of LSMO interface spin polarization in addition with the decrease of PVDF spin diffusion length at high temperature [4]. Figure 5(c) shows the variation of TER with temperature. It is found that the TER reveals a much more complicated temperature dependent behavior. It firstly increases from 65% at 10 K to a maximum of 80% at 100 K, then is followed by a rapid decrease to 5% up to 300 K.

To understand the TER temperature dependence and transport mechanisms, we have studied the resistances in both polarization states as a function of temperature, as shown in figure 6(a). To avoid the influence of large bias on the ferroelectricity of PVDF, all resistances were measured with a small bias of +10 mV without performing \(I–V\) measurement. It can be observed that after positive polarization at 10 K, the resistance firstly decreases slightly with temperature varying from 10 K to 60 K (green region) and then increases with the temperature between 60 K and 120 K (yellow region), followed by a decrease up to 290 K (grey region). This behavior correlates with the tendency of \(dV/dI\) measured at zero bias shown in figure 3(c). For the case after negative polarization at 10 K, the resistance firstly decreases to a minimum at 60 K and then increases monotonously with temperature until 290 K. Below 120 K, the resistance in both polarization states varies with the same tendency with the temperature and there is small variation in TER. However, above 120 K, the opposite variation tendency of resistance in both polarizations induces a rapid decrease of TER for the OMFTJ device.

To explain the temperature dependence of transport behavior, we have defined three temperature ranges. For the first range from 10 K to 60 K (green region), the decrease of resistance as a function of temperature could be caused by the activation of the hopping process due to the inelastic tunneling which reduces the effective tunneling resistance [22]. Since the thickness of PVDF organic barrier is about 20 nm, it is not possible for electrons to directly tunnel through the barrier. The hopping process through the defect states close to Fermi level inside the barrier is indispensable. Thermally activated phonons can help electron hopping through the defect sites and effectively reduce the tunneling resistance [22, 23]. The Kondo effect due to the magnetic impurity scattering can also effectively increase the junction resistance at low temperature [24]. However, this mechanism can be excluded in our case since this effect would also manifest in zero-bias anomaly (ZBA) in the I–V curves at low temperature that we do not observe in our measurements, as shown in figure 3(b).

The increase of resistance with temperature between 60 K and 120 K (yellow region) for both polarization states can be due to several mechanisms. First of all, the metallic behavior induced by pinholes inside the barrier can result in such behavior [25]. However, as observed in our STEM image, the barrier is almost homogenous and no pinhole is observed. To definitively confirm this point, we have performed conductive AFM characterization. No conducting path is obtained under applied bias of +5 V, as demonstrated by the uniform contrast associated to insignificant background current (∼30 pA) as shown in supplementary figure S3. The second possibility is that PVDF has a semi-crystalline nature which is known for the ubiquitous presence of amorphous phase in addition to the ferroelectric crystalline phase [26]. When temperature increases, thermal energy can result in a structural disorder in the PVDF film, as it has already been demonstrated that in some organic superconductors the structure transition exists around 80 K [27]. The thermal activation of structural disorder could induce an increase of electron scattering and result in the increase of resistance. However, this hypothesis can also be excluded because the structural disorder will also induce a disorder in ferromagnetic domains, which should manifest a decrease of resistance in the positive polarization state with the increase of temperature. The third possibility could be due to the thermal strain effect. Supplementary figure S4 displays the saturation magnetization (\(M_s\)) as a function of temperature for the PVDF/Co bilayers and pure Co. We have observed about 10% increase of magnetization from 10 K to 300 K for the Co on PVDF. This behavior is completely different from the normal decrease of \(M_s\) with temperature for the pure Co due to the magnetization disorder generated by the limited Curie temperature. In fact, the increase of Co \(M_s\) is related to the magnetostriction effect of Co since PVDF has an anisotropic thermal expansion, which can generate a uniaxial strain on Co when changing the temperature. The thermal strain can then effectively influence the junction resistance [28], as discussed in supplementary S4.
Figure 4. Magneto-response curves measured under a bias of $+10 \text{ mV}$ at $10 \text{ K}$ after $+1.2 \text{ V}$ and $-1.5 \text{ V}$ polarizing. The inset schematics show four resistance states associated with different polarization and magnetization orientations.

Figure 5. (a) Magneto-resistance loops measured at different temperatures for positive and negative polarization states. (b) Temperature dependence of TMR in both polarization states. (c) Temperature dependence of TER.
Figure 6. (a) Temperature dependence of $R_P$ in both polarization states. The resistance is measured with small bias of 10 mV. (b) Schematics of polarized states in the PVDF barrier at temperature range between 120 K and 300 K (grey zone). The schematic of energy barrier between two ferroelectric polarization states under thermal fluctuation is also shown.

From 120 K to room temperature (grey region), the temperature dependence can be explained by thermal fluctuation of ferroelectric domains, as schematically shown in figure 6(b). In fact, every needle-like PVDF crystallite can act as an individual ferroelectric domain [4]. When thermal energy is high enough to overcome the energy barrier ($E_b$) between two polarization states [29], the molecular chain starts to rotate to another polarization state. With the increase of temperature, more and more domains with opposite polarization nucleate in both polarization states, which results in a reduction of resistance for the positive polarization state while an increase of resistance for the negative polarization state. Since negligible electric field was applied during the increase of the temperature, the evolution of ferroelectric domain of PVDF could be described by the Kolmogorov–Avrami–Ishibashi (KAI) model [19, 30, 31], which assumes the statistical formation of a large number of nucleation sites and homogeneous domain growth. The transition temperature ($T_{Tr}$) at 120 K can allow us to roughly estimate the energy barrier between two polarization states to be about $E_b = k_B T_{Tr} = 10$ meV. This value is one order smaller than the reported nucleation energy barrier for P(VDF-TrFE) thin film (0.4 eV) grown by the Langmuir–Blodgett method [32]. The smaller energy barrier could be related to the crystal inhomogeneities, lattice defects, interface defects or small residual nuclei [33–35]. As the temperature increases, the opposite variation tendency of resistance in both polarizations results in the disappearing of the contrast of the two polarization states, thus vanishing the TER effect.

In addition, we have measured the temperature dependent magneto-transport behavior for another thin device of LSMO/PVDF (13 nm)/Co (see supplementary S5), which has been polarized by two different methods during the increase of temperature: (1) states only polarized at 20 K and (2) states polarized at each measuring temperature. As shown in supplementary figure S5,
the second polarizing method can indeed achieve better polarization states at higher temperature. The TER can be much enhanced at RT compared to the first polarizing method. This validates that the polarization relaxation by thermal fluctuation dominates the transport properties in the range $T > 120$ K in the first polarizing method. In comparison, the influence of thermal activation of domain switching plays a negligible role in the temperature dependent behavior in inorganic MFTJs based on BaTiO$_3$ [15] due to high nucleation barrier energy (40k$_B$T) [36].

4. Conclusions

In summary, we have investigated the temperature dependent transport behavior in La$_{1-x}$Sr$_x$MnO$_3$/PVDF/Co OMFTJs. We have found that different mechanisms can dominate the transport properties in different temperature ranges. Below 60 K, the transport is dominated by electron hopping through the thick organic barrier, which shows a decrease of resistance with the increase of temperature in both polarization states. Between 60 K and 120 K, the thermal strain could induce an increase of resistance in both polarization states when increasing the temperature. When $T > 120$ K, it is found that thermal fluctuations of the ferroelectric domains play an important role on the transport properties. The opposite temperature dependence of resistance for both ferroelectric polarization states results in a rapid diminishing of tunneling electroresistance. However, polarizing the junction at high temperature can recover a better polarization degree to enhance TER at room temperature. These results pave the way to develop high performance OMFTJs for memristor and spintronics applications.

Acknowledgments

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ORCID IDs

Xavier Devaux  https://orcid.org/0000-0001-5694-464X
Anthony Ferri  https://orcid.org/0000-0002-9162-2715
Rachel Desfeux  https://orcid.org/0000-0002-0629-3671
Xiao-Guang Li  https://orcid.org/0000-0003-4016-4483
Mairbek Chshiev  https://orcid.org/0000-0001-9232-7622
Sajjeng Rauf  https://orcid.org/0000-0003-2343-9334
Shiheng Liang  https://orcid.org/0000-0002-2133-2659
Yuan Lu  https://orcid.org/0000-0003-3337-8205

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