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Haoliang Liu, Matthew Groesbeck, Evan Lafalce, Xiaojie Liu, Zeev Valy Vardeny, "Study of spin transport in polyfluorene films," *J. Photon. Energy* **8**(3), 032212 (2018), doi: 10.1117/1.JPE.8.032212.

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Study of spin transport in polyfluorene films

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Abstract. We have investigated spin transport in films of polyfluorene, a π -conjugated polymer, using the technique of "spin pumping" from a ferromagnet substrate, namely Ni₈₀Fe₂₀ in a NiFe/ polyfluorene/Pt trilayer device at room temperature. Pure spin current (without carrier injection) is generated in the polymer film by microwave excitation of the NiFe magnetic moment at ferromagnetic resonance conditions. The induced spin current through the ferromagnet/polymer interface crosses the polymer layer and is detected by a Pt overlayer in the device, where it is converted into electric current via the inverse spin Hall effect. We have successfully determined the spin diffusion length, λ_S , in the polyfluorene film by varying the polymer thickness in the trilayer structure, and found $\lambda_S = 118 \pm 9$ nm. We also measured the charge-carrier mobility, μ in polyfluorene film using the time-of-flight technique and found it to be affected by dispersive transport. From the obtained λ_S and μ values, we estimated the spin relaxation time in polyfluorene to be ~5 μ s at room temperature. © 2018 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.JPE.8.032212]

Keywords: polymer; spin transport; spin pumping; organic spintronics.

Paper 18026SS received Feb. 7, 2018; accepted for publication Apr. 19, 2018; published online May 8, 2018.

1 Introduction

Organic semiconductors (OSC) have attracted great attention not only because of their wide applications in optoelectronics,^{1,2} such as organic light-emitting diodes (OLEDs),³ but also due to their potential use in spintronic devices.⁴ OSCs have very weak spin orbit coupling (SOC) and hyperfine interaction (HFI), and therefore, are expected to have long spin relaxation times. These unique properties assure that OSCs are ideal for the research of spin injection, transport, and detection. Spin-polarized carrier transport in OSCs is based on the giant magnetoresistance effect found in Co/Alq₃/La_{0.7}Sr_{0.3}MnO₃ (LSMO) spin valve structure, which was first demonstrated in 2004⁵ and accompanied with their spintronic applications in magnetic random access memories.⁶ Spin diffusion lengths of OSCs have been determined by the OSCs thickness dependence of the magnetoresistance. In addition, the combination of spin and OLEDs has been realized by integrating ferromagnets (FMs) as electrodes in OLED device structures.^{7,8} The OLED device with deuterated poly(dioctyloxy)phenylvinylene as organic interlayer, Co and LSMO as bipolar electrodes, showed a spin valve characteristic magnetoelectroluminescence (MEL) response on the order of $\sim 1\%$ at low temperature.⁸ The magnetic fringe field effect on MEL has been observed in OLED with a unipolar FM electrode.⁹ Spin properties have been well explored in various OSC-based spintronic devices, such as organic spin valve,^{5,10,11} organic spin transistor,¹² spin OLEDs,^{8,9,13} etc.¹⁴ These studies indicate that a new research field—organic spintronics—has emerged, which mainly focuses on the spin-related phenomena in OSCs.

Recently, pure spin current transport has been achieved using the "spin pumping" process. The first observation of pure spin current in OSC generated by spin pumping was in a bilayer structure of the ferrimagnetic insulator, $Y_3Fe_5O_{12}$ and conducting polymer polyethylenedioxythiophene/polystyrene sulphonate (PEDOT:PSS).¹⁵ The inverse spin Hall effect (ISHE) voltage

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could be directly measured in the PEDOT:PSS layer. By using the spin pumping technique with pulsed microwave (MW) excitation in FM/OSCs bilayer structures, OSCs with variable SOC strengths, ranging from π -conjugated polymers with strong SOC (that contain intrachain platinum atoms) to weak SOC polymers and to C₆₀ films, were shown to also act as tunable efficient converters of spin current to charge current via the ISHE.¹⁶ Another structure was also used to study spin current transport in OSC; this is an FM /polymer/nonmagnet (NM) trilayer structure. In such structure, the "magnonic current" excited at the ferromagnetic resonance (FMR) conditions of the FM layer generates pure spin current in the polymer through the FM/polymer interface, which is detected as electric current in the NM layer (i.e., Pt) having large SOC, via the ISHE.^{17,18} Compared with studies of spin-polarized charge-carrier transport using spin valve devices, the spin pumping technique has two advantages for spin transport investigations in OSCs. First, it is free of the well-known hurdle of the conductivity mismatch at the FM/NM interface, which significantly lowers the spin injection efficiency from the FM metals into the OSC film that has low conductivity. Second, it can be measured at various temperatures rather than being limited to low temperatures.¹⁹

Charge transport in π -conjugated polymers has been well understood; it is dominated by the hopping process of polarons formed by strong electron–phonon coupling in these soft compounds.^{20,21} In contrast, the spin relaxation mechanism in π -conjugated polymers has been investigated to a lesser degree. In general, there are two possible mechanisms for the spin relaxation in OSC: these are the HFI^{10,22} and SOC.^{17,23} For the HFI, spin relaxation mainly occurs when the spin half polarons are localized on a molecular trapping site; whereas SOC may induce spin flips during the hopping events.

A series of pioneering works in organic spintronics, including organic spin valves,^{5,10} spin OLEDs,⁸ and spin pumping in OSCs,^{16,24} has been done in Vardeny's group at the University of Utah in the last dozen years. In the Festschrift for Vardeny's 70th birthday, we herein study the room temperature spin transport in polyfluorene (PFO) polymer films using spin pumping in NiFe/PFO/Pt trilayer structures. We have determined the spin diffusion length in this polymer from the decay of the ISHE electrical voltage in the Pt layer of the device with varying PFO interlayer thicknesses. The spin relaxation time of ~5 μ s at room temperature is estimated in PFO based on simultaneous characterization of spin and charge transport, where charge transport is monitored using the time-of-flight (TOF) technique.

The chemical structure of PFO is shown in Fig. 1(a). Although the PFO polymer has been intensively investigated for its potential applications in OLED,²⁵ field effect transistors,²⁶ and plastic solar cells,²⁷ the spin transport properties of this polymer have not been investigated so far. The excited magnetic moment of the NiFe substrate in FMR conditions generates pure spin current into the PFO film through the NiFe/PFO interface, which crosses the PFO layer and is detected by the Pt adjacent layer as electrical voltage (or current) via the ISHE. As a result, the ISHE voltage in the Pt film decreases with increasing the PFO interlayer thickness, and the spin diffusion length can be thus determined.



Fig. 1 Schematics of pure spin pumping and transport in the PFO polymer film. (a) Chemical structure of the PFO polymer. (b) Illustration of pure spin transport in the PFO polymer generated by the ISHE. The MW-excited FMR of the NiFe substrate generates magnon current that, in turn, generates pure spin current at the NiFe/PFO interface via spin pumping. The spin current diffuses through the PFO layer reaching the Pt overlayer. The Pt film acts as spin sink that converts the spin current into electric current via the ISHE mechanism.

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2 Experimental Method

The NiFe/PFO/Pt trilayer devices for the ISHE measurements [Fig. 1(b)] were fabricated with 15-nm Ni₈₀Fe₂₀ (NiFe) films on glass substrates, followed by a PFO (9,9-dioctylfluorene) polymer layer, and capped with 7-nm Pt connected with Cu wires to a lock-in amplifier for measuring the ISHE current induced by the FMR in the NiFe film. The spin cast of the PFO polymer film was performed at room temperature in a glove box which was full of nitrogen where the oxygen level was below 0.5 ppm. The PFO film was deposited by spin casting a 5-g/L solution of the polymer in toluene on NiFe substrates at various speeds ranging from 1000 to 5000 rpm. The sample was then annealed at 100°C for 30 min. Under these deposition conditions, the polymer structure is expected to be the α phase.²⁸ Other details of the device structures can be found elsewhere.¹⁶ The devices were protected by a 100-nm SiO₂ capping layer. The NiFe, Pt, and SiO₂ films were deposited by electron beam evaporation at a base pressure of 1×10^{-7} mbar. The MW excitation for the ISHE measurements was generated by an Agilent N5173B signal generator working in the frequency range of 9 kHz to 20 GHz. The MW frequency was fixed at 3 GHz. The samples were put on top of a coplanar MW guide of 250 μ m wide transmission line with the radio frequency magnetic field perpendicular to the DC magnetic field. The ISHE voltage generated in the Pt layer in the device was detected by phase-sensitive lock-in technique, in which the MW intensity was modulated by a square wave at 17 kHz.

To obtain the carrier mobility in the PFO polymer, we carried out the TOF measurement²⁹ on a typical photovoltaic device. The device structure was indium tin oxide (ITO) semitransparent anode, ~40-nm spin-cast PEDOT:PSS hole transport layer, ~5- μ m-thick PFO interlayer deposited by spin coating, 20-nm Ca as electron transport layer, that was capped with ~100-nm aluminum layer that served as a cathode. The device structure was thus ITO/PEDOT:PSS/PFO/Ca/ Al with an active area of 2 × 2 mm². A laser pulse (7 ns, 532 nm) of 2 W was directed on the Al electrode. The laser pulse generates a thin layer of photoexcited species, including excitons and both positive and negative free carriers (polarons). A strong positive electric field (~200 kV/cm) was applied pointing from the Al electrode toward the ITO electrode that caused holes to drift through the film. Photogenerated electrons were extracted on a much shorter time scale due to their proximity to the Ca/Al electrode, while hole-injection is blocked at the Ca interface due to the relatively high work function of Ca and similarly for electron-injection at the PEDOT:PSS interface. Thus, the photogenerated holes are solely responsible for the transient current during the TOF measurement.

3 Results and Discussions

Figure 2(a) shows the derivative of the MW absorption at FMR resonance conditions obtained for the NiFe (15 nm)/PFO (140 nm)/Pt (7 nm) device. The field response is recorded at 3 GHz while sweeping the external magnetic field. The resonance field value of ~ 10.8 mT can be well interpreted using the Kittel equation $f = \gamma \sqrt{H_R(H_R + 4\pi M_S)}$, where γ is the gyromagnetic ratio, and H_R and M_S are the resonance field and saturation magnetization of NiFe, respectively.³⁰ Figure 2(b) shows the magnetic field dependence of the electromotive force measured in the Pt layer generated by the ISHE process. The magnetic field direction is perpendicular to the electric field direction in the Pt layer. The pure spin current generated by the FMR in the NiFe substrate induces aligned spins into PFO through the NiFe/PFO interface. Subsequently, the pure spin current diffuses in the PFO layer until it reaches the PFO/Pt interface, where it is converted into electric current by the ISHE mechanism. The induced electric field (or current) direction is perpendicular to both the spin current and spin polarization directions, since $\vec{E}_{\text{ISHE}} \propto \vec{J}_S \times \vec{\sigma}$, where \vec{J}_S and $\vec{\sigma}$ are the spin current and spin polarization, respectively.³¹ Pt is adopted here as the cap detection layer since it has strong SOC and therefore generates very strong ISHE electric current from the spin current. The field peak position of the V_{ISHE} response is the same as H_R in the FMR as shown in Fig. 2(a). In addition, we also checked that V_{ISHE} changes polarity when reversing the external magnetic field direction from +H to -H, which is consistent with the symmetry of the ISHE process according to the theoretical prediction.³¹



Fig. 2 Observation of spin transport in the PFO polymer interlayer in a trilayer structure NiFe/PFO/ Pt. (a) Magnetic field response of the FMR absorption derivative in the NiFe film. (b) The induced voltage, V_{ISHE} , measured in the Pt overlayer in the trilayer structure NiFe (15 nm)/PFO (140 nm)/Pt (7 nm) due to the reaching spin current in the PFO interlayer. The spin current in the Pt layer is converted into electric current (voltage) via the ISHE mechanism.

Spin pumping into materials with long spin relaxation times, such as π -conjugated polymers, may involve a strong spin backflow process.³² However, the Pt layer with strong SOC can act as a perfect spin sink that minimizes the spin backflow. Consequently, by increasing the PFO polymer thickness in the trilayer structure, the spin relaxation probability is enhanced before the spin current reaches the PFO/Pt interface. The ISHE voltage detected in the Pt layer is thus proportional to the pure spin current that reaches the Pt layer after its diffusion and transport through PFO polymer layer. Figure 3(a) shows the detected V_{ISHE} in the Pt adjacent layer of the trilayer device at various PFO interlayer thicknesses, d. The induced voltage decays with increasing PFO layer thickness, which is approximated by an exponential decay, $V_{\rm ISHE} \propto e^{-d/\lambda_s}$,¹⁷ as seen in Fig. 3(b), where λ_s is the spin diffusion length of the PFO polymer. It should be noted that a strong ISHE signal is still clearly observed with PFO thickness up to a few hundred nanometers. From the ISHE(d) fitting, we can determine the spin diffusion length of the PFO polymer at room temperature to be $\lambda_s = 118 \pm 9$ nm. In addition, this exponential decay also provides solid evidence that the measured voltage is generated by the ISHE process in the Pt layer, rather than other artifacts generated in the NiFe substrate.¹⁷ We note that the resonance field (~10.8 mT) is much larger than the effective HFI strength, so we attribute the main spin relaxation process during the spin transport in the PFO layer as due to the SOC in the polymer.

The charge-carrier (polaron) mobility of the PFO polymer was determined by the TOF measurement, as schematically shown in Fig. 4(a). The charge carriers are photogenerated by a pulse laser excitation of the PFO at the Al/Ca electrode interface. By applying a strong external electric field, the positive charge carriers (holes polaron) drift toward the ITO electrode resulting in a time-dependent current, while the electrons are quickly swept by the cathode. Due to highly dispersive transport for the holes in PFO, the transient photocurrent measured after the laser pulse follows a slow power-law decay, with a marked transition to a steeper decay rate once the initial excited carriers begin to reach the opposite electrode. The transition point or "kink" between the two power-law time-domain regions represents the transit time, t_{tr} of the holes



Fig. 3 Spin diffusion length determination in PFO. (a) The ISHE voltage, V_{ISHE} , measured in the Pt overlayer in the trilayer device NiFe/PFO/Pt with various PFO thicknesses, as denoted. (b) PFO thickness dependence of V_{ISHE} induced in the Pt layer. The $V_{\text{ISHE}}(d)$ decay is fitted by an exponential function, from which we obtain the spin diffusion length, λ_S in PFO at room temperature as denoted.



Fig. 4 Hole mobility in the PFO polymer measured by the TOF method. (a) Schematics of the TOF technique in a device of structure Al/Ca/PFO/PEDOT:PSS/ITO. The photogenerated holes drift from the Al/Ca side toward the ITO-collecting electrode under the application of a bias, *V*. The time-dependent current is monitored in the device. The transit time, t_{tr} , for the arrival of the charge-carrier packet is directly related to the hole mobility, μ_h . (b) Transient TOF photocurrent in PFO at room temperature. The transit time t_{tr} and calculated hole mobility μ_h are indicated.

across the film,³³ as shown in Fig. 4(b). The hole mobility μ can readily be calculated by the relation: $\mu = d^2/t_{tr}V$, where V is the electrical voltage (100 V) and d (5.6 μ m) is the OSC layer thickness. The obtained hole mobility at room temperature was found to be $\mu = 3.1 \times 10^{-4}$ cm² (Vs)⁻¹. It should be noted that this relatively high hole mobility in the PFO film shows very weak electric field dependence and assures its broad application as an electroluminescent polymer.²⁹

In π -conjugated polymers with hopping transport, the spin diffusion process may be described using the relationship: $\lambda_S = \sqrt{D\tau_S}$, which connects the spin diffusion length and spin relaxation time via the carrier diffusion subjected to the Einstein relationship. Here, τ_S is the spin relaxation time, and $D = \mu k_B T/e$ is the diffusion constant, where k_B is the Boltzmann constant. Therefore, τ_S may be extracted from the obtained values of λ_S and μ . Using this procedure, we estimated the spin $\frac{1}{2}\tau_S$ for holes in PFO to be ~5 μ s at room temperature. We note that this value is much larger than the spin coherence time of ~300 ns obtained by electron spin resonance measurements in various π -conjugated polymers.^{34,35}

It should be noted that we only consider spin diffusion as the main relaxation mechanism for spin transport, where spin flip happens during the polaron hopping from one trap state to another. In addition, the exchange coupling between the localized polarons may also contribute to spin transport in π -conjugated polymers.³⁶ A pure spin current may therefore propagate due to exchange coupling between two neighboring polarons with opposite spin directions without involving carrier hopping. Therefore, in contrast to polaron hopping with simultaneous charge and spin transport, only pure spin current is induced in the exchange coupling mechanism, and consequently, this leads to a much longer estimate for the effective spin relaxation time. Clearly, the origin of this large spin relaxation time calls for more future theoretical analysis and experimental measurements.

4 Summary and Conclusions

In summary, we have investigated pure spin current transport in PFO polymer, which is induced by the spin pumping mechanism. The pure spin current was generated by MW-excited FMR of a NiFe substrate, and after transmission through the PFO polymer interlayer, was detected as a dc electric voltage in the Pt overlayer generated by the ISHE mechanism. We have shown that PFO polymer can act as an efficient spin transport layer with spin diffusion length of $\lambda_s = 118 \pm$ 9 nm at room temperature. Based on the mobility value obtained by TOF measurements, we estimated the spin relaxation time of ~5 μ s in PFO at room temperature in the framework of spin transport via polaron hopping. Our work paves the way toward the application of PFO polymer in spintronic devices.

Disclosures

The authors declare no conflicts of interest.

Acknowledgments

M.G. and X.L. were supported by the NSF Grant No. DMR-1701427. H.L. and E.L were also supported by the AFOSR through MURI Grant RA 9550-14-1-0037.

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