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Power law behavior of magnetoresistance in tris(8-hydroxyquinolinato)aluminum-based organic light-emitting diodes

Hoju Kang [a], Chan Hyuk Park [b], Jongsun Lim [b], Changjin Lee [b], Woun Kang [c], Choon Sup Yoon [a,*]

[a] Department of Physics, KAIST, Daejeon 305-701, Republic of Korea
[b] Advanced Materials Division, Korea Research Institute of Chemical Technology, Daejeon 305-600, Republic of Korea
[c] Department of Physics, Ewha Womans University, Seoul 120-750, Republic of Korea

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ABSTRACT

Long-term current drift and dielectric relaxation in organic thin films of a single-layer structure pose a serious problem for the accurate measurement of magnetoresistance at low magnetic fields. A new measurement scheme was devised to minimize errors and to report that the magnetoresistance in tris(8-hydroxyquinolinato)aluminum obeys a power law on the magnetic field at 300, 100, and 4.2 K in an entire range from 1 to 140 mT. The exponent of the power increases gradually from 0.47 for a bias voltage of 3 V to 0.58 for a bias voltage of 8 V. The magnetoresistance was observable above the threshold voltage only and its sign was always negative.

1. Introduction

Organic magnetoresistance (OMAR) has drawn much attention since it was first reported in 2003 [1] because of its sizable effect of ~10% at room temperature [2] and its potential applications in organic electronic devices [3] such as magnetic field sensors [4], interactive displays using organic light-emitting diodes [5], and magnetic random access memory devices [3]. Although OMAR has been studied intensively [2,6–11], the underlying mechanism remains unexplained [9–11]. To obtain a genuine OMAR effect, having a single-layer structure, in which an organic layer is placed between a cathode and an anode, is ideal. However, the long-term current drift is relatively larger in the single-layer structure than in the multilayer structures [12], and long-term dielectric relaxation is universal in organic systems [13]. Long-term current drift and dielectric relaxation may cause a serious problem in the accurate measurement of OMAR, as pointed out by many workers [10,14–20]. This is particularly true when the magnetic field intensity is weak and/or the applied bias voltage is small, where the effect of the long-term current drift and dielectric relaxation on current is larger than the magnetic field. Therefore, examining the nature of long-term current drift and dielectric relaxation in a single-layer organic device, and creating a proper measurement scheme to overcome the problems are necessary.

A new measurement scheme is proposed to minimize errors caused by the long-term current drift and dielectric relaxation that may cause significant errors at low magnetic fields. The magnetoresistance (MR) of tris(8-hydroxyquinolinato)aluminum (Alq_3) measured by the scheme showed a power law behavior on the magnetic field in an entire range from 1 to 140 mT at 300, 100, and 4.2 K.

2. Experimental

The organic light-emitting diode (OLED) device consists of indium tin oxide (ITO)/poly(3,4-ethylenedioxythio-

* Corresponding author. Tel.: +82 42 350 2532; fax: +82 42 350 8780.
E-mail address: csyoon@kaist.ac.kr (C.S. Yoon).

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phene) poly(styrenesulfonate) (PEDOT:PSS)/Alq_3/LiF/Al as shown in Fig. 1a. It is virtually a single-layer structure considering that PEDOT:PSS may be regarded as an electrode because of its high conductivity [21] and negligible magnetoresistance [22] compared with Alq_3. An ITO glass (Geomatec Co., Ltd.) with a sheet resistance of 15 Ω/□ was pre-cleaned and treated with UV-O_3. PEDOT:PSS (Baytron Ltd.) was spin-coated with a thickness of ~40 nm on top of the ITO in the nitrogen atmosphere. Then 100 nm-thick Alq_3 (Doosan Electro-Materials Co., Ltd. >99.9%), 1 nm-thick LiF (Cerac Inc., 99.999%), and 100 nm-thick Al (Cerac Inc., 99.999%) were evaporated thermally in sequence at a pressure of 10^{-7} Torr. The active area of the device was 4 mm^2. Finally, the device was encapsulated by UV curable epoxy to protect it from moisture and oxygen.

J–V–L curve was measured to evaluate the characteristics of the OLED device using a sourcemeter (Keithley 2400), a spectrometer (PR650) and a photomultiplier tube (PDS-1). Prior to conducting the magnetoresistance (MR) measurement, the long-term current drift was measured while applying constant bias voltages of 4 V at 300 K, 20.5 V at 100 K, and 24.5 V at 4.2 K for 5000 s. Thus, the current density at 1 s would be the same value of 0.23 mA/cm^2 after applying the bias voltages at each temperature (Fig. 2a). To make a comparison, the current drift at a constant bias voltage of 3 V was also measured at 300 K, with the current density at 1 s after applying the bias voltage at 0.04 mA/cm^2. The dielectric relaxation was examined at a constant bias voltage of 5 V at 300 K (Fig. 2b).

The OLED device was placed between the poles of an electromagnet in such a manner that the direction of the current was perpendicular to the magnetic field. However,

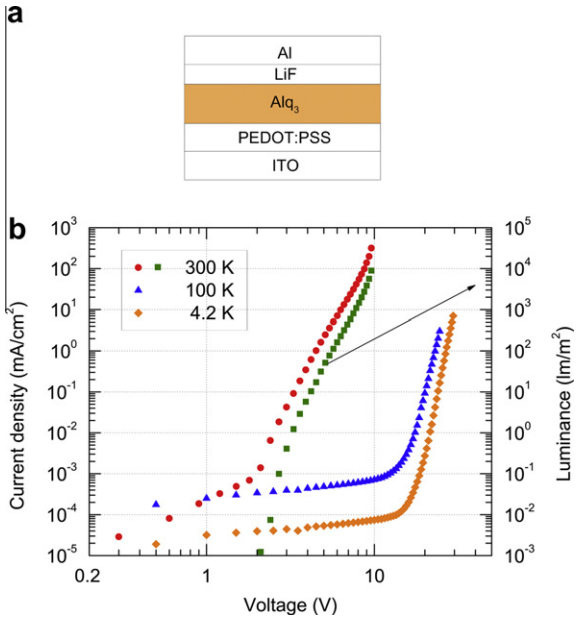

Fig. 1. (a) Schematic structure of a quasi single-layer OLED device based on Alq_3. (b) *J*—*V* curves (left) at 300, 100, and 4.2 K, and *L*—*V* curves (right) at 300 K of the OLED device in (a).

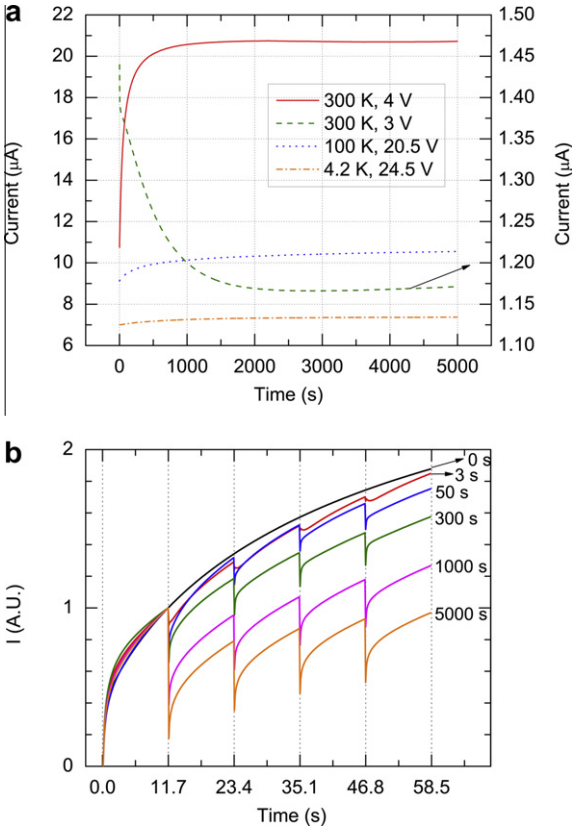

Fig. 2. (a) Current drift in a single-layer OLED device based on Alq_3 at bias voltages of 3 and 4 V at 300 K, 20.5 V at 100 K, and 24.5 V at 4.2 K. The bias voltages were controlled so that the current density at 1 s would have the same value of 0.23 mA/cm^2 after applying the bias voltages at each temperature, except the case of 3 V at which the current density at 1 s was set to 0.04 mA/cm^2 after applying the bias voltage. (b) Dielectric relaxation in the Alq_3-based OLED device during various off-times (0, 3, 50, 300, 1000 and 5000 s) of the bias voltage after applying a pulse-type bias voltage of 5 V for 11.7 s. The relaxation of the currents during the off-times of the bias voltage is not shown.

it was reported that OMAR did not depend on the direction of the magnetic field in amorphous organic semiconductor films [2]. Low-temperature measurements were carried out using a variable temperature insert (1.5–300 K) and a superconducting magnet. Magnet current was controlled within 0.1 mA and the magnetic field was directly measured with a cryogenic Hall effect sensor.

3. Results and discussions

The *J–V* curves of the OLED device based on Alq_3 measured at 300, 100, and 4.2 K and the *L–V* curve at 300 K are shown in Fig. 1b. The *J–V* curve at 300 K was ohmic below 2.1 V, at which the luminance was ~0.001 cd/m^2. The turn-on voltage (1 cd/m^2) was 3.2 V. A luminance of 321.1 cd/m^2 (commercial) was achieved at 6.6 V (13.30 mA/cm^2), at which the power efficiency was 1.15 lm/W. The external quantum efficiency and current efficiency were 1% and 2.4 cd/A at 10 mA/cm^2, respectively, which indicate that the quality of the device is comparable to that used in

literature [23]. As shown in Fig. 1b, the ohmic range of the J–V curves extended from 2.1 V at 300 K to 11.5 V at 100 K and 14.0 V at 4.2 K. Electrical resistance in the ohmic region also increased at low temperatures, which merely indicates that the charge transport is governed by hopping.

Fig. 2a shows the long-term current drift in the OLED device based on Alq_3. For the bias voltage of 4 V at 300 K, the current increased rapidly from 10.7 to 19.3 μA during the transition period of ~250 s and then increased gradually afterwards until it reached a constant value of 20.67 μA. On the other hand, for the bias voltage of 3 V at 300 K, the current decreased from 1.40 to 1.175 μA until ~2000 s and remained constant thereafter. At low temperatures, the long-term current drift decreased significantly. The current drifts at 100 and 4.2 K were 14.3% and 3.7% of that at 300 K for the bias voltage of 4 V, respectively. A similar long-term current drift was also observed in quasi-single-layer devices based on copper phthalocyanine (CuPc), poly(*N*-vinylcarbazole) (PVK) and 4,4-*N*,*N*-dicarbazole-biphenyl (CBP), but the drift currents in PVK, and CBP always decreased regardless of the magnitude of the bias voltage. To examine the dielectric relaxation, a pulse-type bias voltage of 5 V was applied with an on-time of 11.7 s, followed by various off-times of 3, 50, 300, 1000, and 5000 s, and the on- and off-times were repeated. Fig. 2b shows the long-term dielectric relaxation at 300 K in the OLED device based on Alq_3. The relaxations of the current during the off-times are not shown because the off-times are too long to be indicated on the x-axis. The shorter the off-time, the larger the current remnant is. A current remnant of 17.7% remained even after an off-time of 5000 s. The relaxation time of the current was estimated to be 383 s using the simple Debye model. This rather long relaxation time may be attributed to trapping and detrapping of electrons at the grain boundaries in the polycrystalline Alq_3 layer [24] and/or orientational relaxation of Alq_3 molecules [25], which has a dipole moment of 5.3 debye [26].

Considering that the long-term current drift and the dielectric relaxation are inevitable in a quasi-single-layer structure of organic devices, a new measurement scheme was devised to minimize the errors, as shown in Fig. 3a. To circumvent the problem with the long-term drift and dielectric relaxation of the bias current, a constant bias voltage was applied all the time, then a pulse-type magnetic field with increasing strength was applied long after the transient period of the current drift, which is about 250 s. As the pulse-type magnetic field was applied for 50 s, the magnetocurrents (MCs) grew and became saturated within ~6 s on top of the drift current that was caused by the bias voltage. The saturated MCs were relaxed completely to null-field currents during a pulse-off time of 50 s; the relaxation time of the MCs was estimated to be approximately 3 s. The null-field currents were interpolated and depicted as a dotted line in Fig. 3a, which is equivalent to the current drift curve in Fig. 2a. The MCs, $\Delta I(B)$, at different magnetic field intensities were obtained by subtracting the interpolated null-field currents, I, from the total currents at the end of the pulse duration in the pulse train, which are marked by double-sided arrows in Fig. 3a. The scheme thus enables measuring the MCs

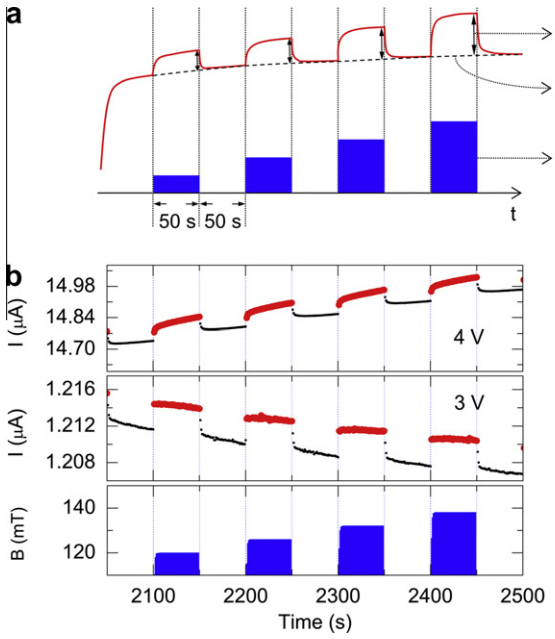

Fig. 3. (a) A measurement scheme for magnetocurrents. The pulse-type magnetic field of 50 s on- and off-time with increasing strength is depicted as a histogram at the bottom. The magnetocurrents (ΔI) are superposed on the drift current which is represented by interpolating the null-field currents (dotted curve). The magnetocurrents at different magnetic fields are marked by the double-sided arrows at the end of each pulse in the pulse train.

accurately even when the magnetic field intensity is weak and/or the applied bias voltage is small, where the magnitude of MCs is small. The raw data of MCs for the bias voltages of 3 and 4 V are shown in Fig. 3b. Although the current drift for the bias voltage of 4 V increases with time while that for 3 V decreases, as shown in Fig. 2a, the MCs for both bias voltages are positive irrespective of the sign of the slope of the current drift curve.

The MCs at 300 K were observable only above the threshold voltage of 2.1 V, at which the current density started to deviate from the ohmic dependence ($J \propto V$) and the luminance started to increase suddenly (Fig. 1b). The threshold voltage agrees well with the calculated built-in potential and may be regarded as the onset of hole injection [23]. This suggests that there exists a strong correlation between OMAR and exciton formation, which agrees with the results of Desai et al. [10] and Wang et al. [27]. The judgment about whether excitons are the sole contributors to OMAR is premature but it can be assumed that excitons are at least one of the major contributors. The MCs at 300 K for the bias voltages of 3–8 V are shown in Fig. 4a. The higher the bias voltage, the larger the magnitude of the MCs is. The MCs for the bias voltages of 3, 4, 6, and 8 V are all positive, and are 0.00392, 0.082, 1.39, and 9.19 μA at 100 mT, respectively. The relative MCs, $\Delta I/I$, follow the power law in an entire range of magnetic field from 1 to 140 mT (Fig. 4b), which shows a marked contrast with other results [2,9,28] at magnetic fields less than ~10 mT. The exponent of the power increases gradually from 0.47 for the bias voltage of 3 V to

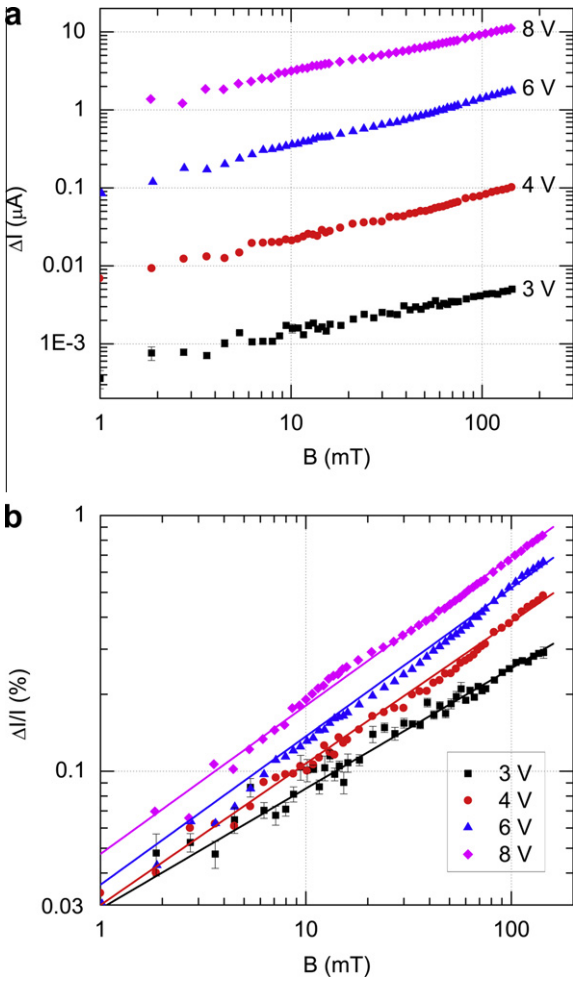

Fig. 4. Log–log plots of (a) magnetocurrents and (b) relative magnetocurrents for the bias voltages of 3, 4, 6 and 8 V in an Alq_3-based single-layer OLED device. The relative magnetocurrents in (b) fit to B^n, where n ranges from 0.47 for the bias voltage of 3 V to 0.58 for 8 V.

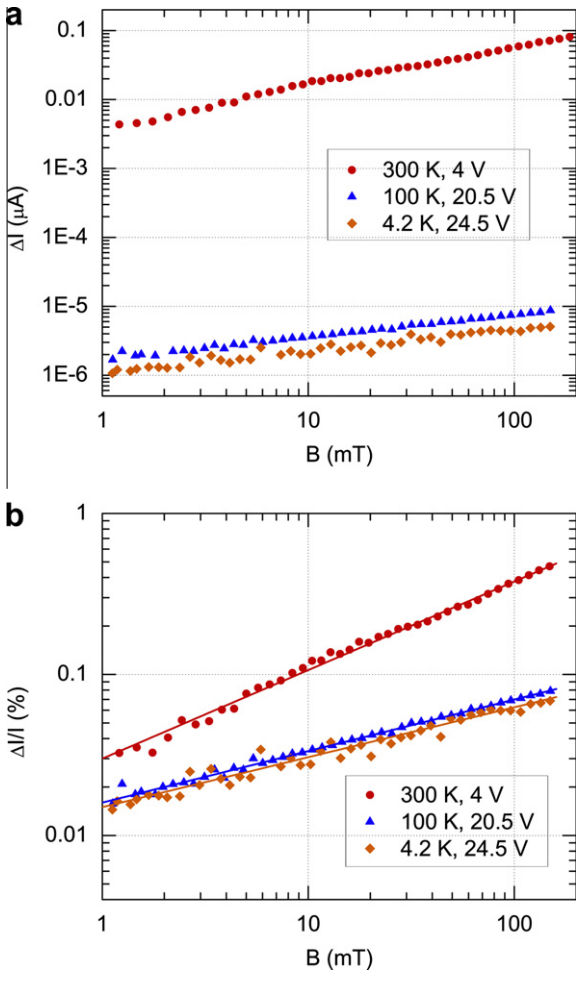

Fig. 5. Log–log plots of (a) magnetocurrents and (b) relative magnetocurrents at 300, 100, and 4.2 K in an Alq_3-based single-layer OLED device. The relative magnetocurrents in (b) fit to B^n, where n is 0.55 at 300 K, 0.32 at 100 K, and 0.31 at 4.2 K.

0.58 for the bias voltage of 8 V. The relative MCs for the bias voltages of 3, 4, 6, and 8 V at 100 mT are 0.25%, 0.38%, 0.53%, and 0.68%, respectively. The corresponding MR values have the same magnitude as the relative MCs up to two decimal places with opposite signs. To ensure that the MC values observed at 300 K using the new measurement scheme truly reflect the OMAR effect, low-temperature measurements were made at 100 and 4.2 K, at which organic devices are much more stable, and the current drift is very small, as shown in Fig. 2a. Fig. 5 shows the MCs and relative MCs at 300, 100 and 4.2 K, which were measured using a different sample from the one used in Fig. 4. The bias voltage at each temperature was controlled in such a way that the current density at 1 s had the same value of 0.23 mA/cm^2 after applying the bias at each temperature. As shown in Fig. 5a, the MCs at low temperatures decrease drastically from those at 300 K. The MCs at 4.2 K is about four orders of magnitude smaller than those are at 300 K. Although the current drift is very small at 100 and 4.2 K, the relative MCs still exhibit power law behavior at low temperatures (Fig. 5b). However, the exponent decreases from 0.55 at 300 K to 0.32 at 100 K and 0.31 at 4.2 K. The fact that the MCs obey the power law even at 4.2 K, at which the current drift is very small and the OLED device is very stable, suggests that the OMAR effect is mostly responsible for the power law behavior of MCs at 300 K because the proposed measurement scheme can effectively avoid the effect of the current drift. In the present study dielectric relaxation did not affect the OMAR measurements because a constant bias voltage was applied all the time. To examine whether the proposed measurement scheme produces the same field dependence irrespective of the direction of magnetic field intensity variation, the relative MCs were measured with both increasing and decreasing amplitudes of the magnetic pulses. Fig. 6 shows that the relative MCs for the increasing and decreasing amplitudes indicate the same dependence on the magnetic field.

Many research groups [10,14–20] have recognized that the time-dependent current drift seriously affects the

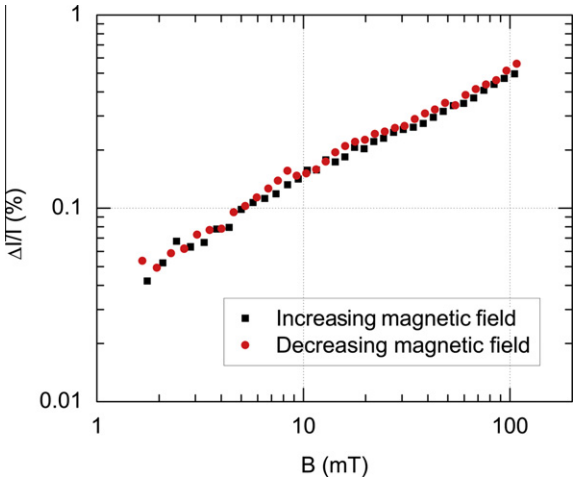

Fig. 6. Log–log plots of relative magnetocurrents at 300 K for the increasing amplitude of magnetic pulses (■) and the decreasing one (●).

OMAR measurements. To overcome the problem Bloom et al. [29], Wagemans et al. [16,30], and Wang et al. [31] employed an ac-field modulation technique in which a small amplitude ac magnetic field is added while applying a dc magnetic field of varying strengths at a constant dc bias field. The amplitude of the ac magnetic field was less than a few mT, and the strength of the dc magnetic field was up to a couple of 100 mT. This method is very useful to avoid the effect of the long-term current drift on the condition that dI/dB is independent of time. However, at low magnetic fields, where the strength of the dc magnetic field has the same order of magnitude as the amplitude of the ac magnetic field, the observed OMAR values may not be reliable because $\Delta I_{ac}/\Delta B_{ac}$ cannot be approximated as dI/dB. This condition may cause the disagreement between the line shapes of OMAR presented in this work and those obtained using the ac-field modulation technique at low magnetic fields. The other cause may be attributed to the different scan speeds ($\Delta B/\Delta t$) of the magnetic field. Majumdar et al. [18] reported that the magnitude and line shape of OMAR depend considerably on the scan speed of the magnetic field based on the observation of OMAR hysteresis in regio regular poly(3-hexylthiophene) (RRP3HT). At a scan speed of 20 $\mu T\,s^{-1}$, OMAR showed a perfect power law behavior in the range of 0.1–150 mT. However, at scan speeds faster than 0.2 $mT\,s^{-1}$, the line shape of OMAR departed from the power law at magnetic fields less than ~10 mT, and the magnitude of OMAR became significantly smaller. Considering that the scan speed used in the present work ranged from 1.3 $\mu T\,s^{-1}$ to 0.15 $mT\,s^{-1}$ in the magnetic field range of 1–140 mT, the power law behavior observed in Alq_3 could be largely related to the low scan speed, as observed by Majumdar et al. in RRP3HT [18,32].

The observed power law behavior of OMAR in Alq_3 is contrasted with the empirical Lorentzian, $B^2/(B^2+B_0^2)$, or non-Lorentzian, $B^2/(B+|B_0|)^2$, function behavior reported by others [2,33]. Sheng et al. showed that Hamiltonian based on the hyperfine interaction could lead to the Lorentzian, $B^2/(B^2+B_0^2)$, function behavior [33]. Linear combinations of two non-Lorentzian functions [28] or exponential decay functions [34] were also used to describe the behavior of OMAR in Alq_3.

The power law behavior of MR with an exponent of ~0.5 at high magnetic fields is a well-known phenomenon in colossal magnetoresistance materials and is indicative of a highly disordered electronic system with strong electron–electron interaction effects [35]. The power law behavior of MR had been reported in amorphous Si and Ge in the hopping regime [36]. Movaghar and Schweitzer [37] explained the MR mechanism in terms of the modification of electron spin-flip relaxation time in the presence of an external magnetic field using the random walk hopping model. The similarity in highly disordered electronic systems between organics and amorphous Si and Ge thin films suggests that the MR mechanism governing amorphous inorganic semiconductors may also apply to disordered organic semiconductor systems. However, the magnitude of MR in organic semiconductors is larger by two orders than amorphous Si and Ge at a magnetic field of ~100 mT at room temperature. Futher study is needed to verify the mechanism of OMAR in relation to the power law behavior.

4. Conclusions

The long-term current drift and dielectric relaxation in an Alq_3-based quasi-single-layer OLED device was examined, and a new measurement scheme for OMAR that can minimize the errors, especially at low magnetic fields less than ~10 mT and/or at small bias voltages, was presented. The MR in Alq_3 showed a power law behavior at 300, 100 and 4.2 K in an entire range of magnetic field from 1 to 140 mT with an exponent of 0.47 at a bias voltage of 3 V to 0.58 at a bias voltage of 8 V. The MR was observable only above the threshold voltage of 2.1 V, and the sign was always negative, suggesting that the MR is related to exciton formation.

Acknowledgements

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