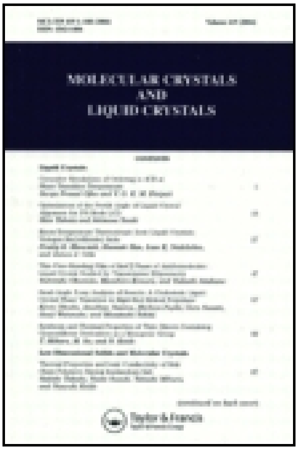

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Charge Transport in Liquid Crystalline Semiconductor and Crosslinked Polymer Composite

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CHARGE TRANSPORT IN LIQUID CRYSTALLINE SEMICONDUCTOR AND CROSSLINKED POLYMER COMPOSITE

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We have fabricated a novel charge carrier transport material composed of a liquid crystalline molecule and a cross-linked polymer. This composite system exhibited high hole mobility comparable with that of the pure liquid crystalline material. The charge carrier transport properties were influenced by the polymerization temperature, i.e., by the mesophase during polymerization. The hole mobility did not depend on temperature and electric field in the case of polymerized samples in the mesophase. We discussed the carrier transport properties in the polymer composites in comparison with those of the pure liquid crystalline material and the conventional polymer composite of the molecularly doped polymers, in addition to the resulting domain structures in the polymer composites with reference to the carrier transport properties in smectic mesophase as well as in the isotropic phase.

Keywords: charge carrier transport; crosslinked polymer; liquid crystal; organic semiconductor; phase separation

INTRODUCTION

The organic semiconductors recently have attracted much attention to organic devices such as organic light emitting diodes (OLEDs) [1] and thin film transistors (TFTs) [2]. In particular, single crystals of the organic aromatics such as pentacene [3] and hexithiophene (α-6T) [4] have extensively studied because of their high mobility originated from ordered molecular alignment. Single crystals exhibit very high mobility over 10^{-1}–$10\,\mathrm{cm^2/Vs}$ indeed, but there remains unsolved problem of

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how to prepare large-area thin films for the device applications. Recently it was discovered that some type of liquid crystals, such as the discotic [5–8] and the smectic [9–12] having an aromatic π-conjugate moiety as a core part, exhibit very fast electronic conduction characterized by high mobility over $10^{-2}\,\mathrm{cm}^2/\mathrm{Vs}$ [11] and up to $0.1\,\mathrm{cm}^2/\mathrm{Vs}$ [6]. Now, liquid crystals are being recognized as a new class of organic semiconductors, *i.e. self-organizing molecular semiconductor*. For liquid crystals, indeed, the cell technologies in display devices will provide us with a good basis for practical applications in term of large area uniformity and protection of the material in ambient atmosphere, but it limits the flexible design of the devices and the minimum thickness of material over 1 μm. This is a big draw back in terms of difficulty in a low voltage operation of the devices.

In order to expend the present potential of the liquid crystalline semiconductor, we have fabricated a new material composite prepared with the liquid crystalline semiconductor and crosslinked polymer for feasibility of film preparation and fixing the molecular alignment, and characterized its liquid crystalline behavior and charge carrier transport properties [13]. The carrier transport properties in the molecularly doped polymer, which is a conventional polymer composite and is used for the carrier transport layer in the xerographic photoreceptors, are subject to the homogeneous solubility of the low-mass organic semiconductors in binding polymers. The liquid crystalline composite system, however, can be different from the conventional homogeneous films, due to their self-organization and microscopic phase separation. Polymer dispersed liquid crystals (PDLCs) and polymer stabilized liquid crystals (PSLCs) are typical examples [14,15]. In these hybrid systems, the liquid crystal is the major component and exhibit heterogeneous structure consisting of liquid crystal domains and polymer networks. Taking into account of electrically inactive boundaries in the liquid crystal domains, the liquid crystalline semiconductor composites could be a possible solution to the problem describe above.

In this paper, we report here that a novel hybrid system comprised of a smectic liquid crystalline semiconductor and a crosslinked polymer and characterized its charge transport properties by a time-of-flight (TOF) technique and discuss the charge transport properties of these hybrid films and discuss them in comparison with those with the conventional homogeneous film described above. In addition, this composite system is expected to increase “macroscopic” viscosity by the resulting polymer network, which suppress the ionic conduction. Generally, The ionic charge transport is affected by viscosity of solvent as known Walden rule [16]. Thus, we also have investigated the effect of the polymer network on the ionic conduction with the aid of impurity doping.

EXPERIMENTAL

Chemical structures of the materials used here are shown in Figure 1. In order to establish a uniform mixture of a liquid crystal of 6-(4'-octylphenyl)-2-dodecyloxynaphthalene (8-PNP-O12) and 1,6-hexanediol diacrylate (HDA) as a crosslinker, these materials were dissolved into distilled toluene. The resulting solution was evaporated *in vacro* at room temperature. Concentration of HDA was ranged from 5 wt% (10.4 mol%) to 20 wt% (35.6 mol%) on a liquid crystal basis. The mixture was sandwiched between two indium-tin oxide (ITO) coated glass plates. The cell gap was adjusted to be 9 or 15 µm with the aid of coloidal-silica spacers. The cell was irradiated with Xenon lamp to achieve the photopolymerization, in which the exposure energy was of 4.0 Jcm^{-2} at each liquid crystalline phase of the mixtures such as the SmA phase at 93°C and the SmB phase at 70°C and isotropic phase at 125°C, which were slightly lower than those of corresponding temperature of the pure 8-PNP-O12. The resulting polymerized cell was cooled at the cooling rate of 5°C/min. The conversion for polymerization was monitored by Fourier transfer infrared (FT-IR) spectroscopy. The phase transition behavior was studied by polarized microscope and differential scanning calorimetry (DSC). We selected a hexyldodecylterthiophene derivative (6-TTP-12) as a chemical trap, which causes a deep trap state for hole in 8-PNP-O12, as judged from the energy levels of the highest occupied molecular orbital (HOMO) of 5.1 eV and 5.6 eV in 6-TTP-12 and 8-PNP-O12, respectively. Concentration of 6-TTP-12 was ranged from 0.001 wt% to 0.05 wt%. Charge transport properties were studied by transient photocurrent measurements with a conventional time-of-flight set-up, in which the composite cell was illuminated by a light pulse from N_2 laser (600 ps in pulse width).

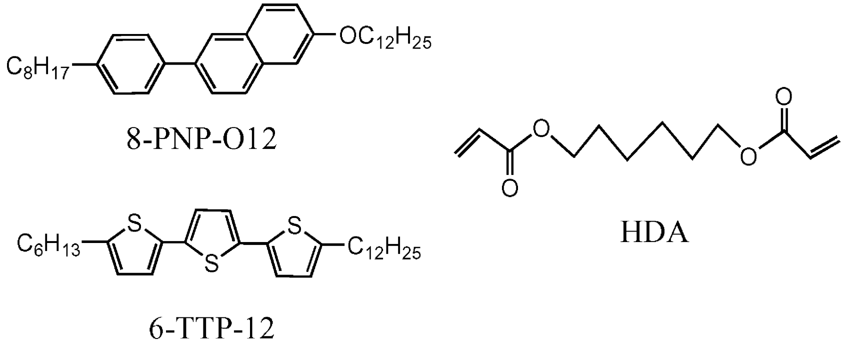

FIGURE 1 Chemical structures of compounds used in this study.

The induced displacement current was monitored by a digital oscilloscope after its amplification. Transit times were determined from double logarithmic plots of transient photocurrent curves. The charge mobility was estimated from a slope of $1/t_T$-V/d^2 plots where t_T is the transit time at a given applied voltage, V and d a cell thickness.

RESULTS AND DISCUSSION

Phase Separated Structures with Various Photopolymerization Conditions

The polymerized cell showed the phase transition temperature lower than that of the pure 8-PNP-O12 by only 1–2°C in each condition. This is due to because the complete polymerization reaction without decomposed products or unreacted monomers. The textures of this composite fabricated in various photopolymerization conditions, *i.e.*, at different phases are shown in Figure 2. The composite cells polymerized in the isotropic phase of 125°C exhibited a pattern indicating microscopic phase separation, judging from the characteristic phase separated structures and the transient temperatures comparable with pure 8-PNP-O12 as shown in Figure 2(a) and (b). The homogeneous textures were obtained in the cell polymerized in the isotropic phase, and the domain sizes were dramatically changed according to the concentration of containing monomer: the mesophase texture was of several hundred μm in domain size in the cell containing 5 wt% HDA while the homogeneous texture of sub μm in the cell containing 20 wt% HDA or more. When the photopolymerization reaction was carried out in the isotropic phase, anisotropic alignment of liquid crystal could not be reflected in the resulting polymer network. In general, liquid crystal composite materials such as PDLC or polymer network liquid crystal (PNLC), the domain size is determined exclusively by a ratio of polymer or monomer [14]. Therefore, it is plausible that the domain sizes in this composite system are mainly determined by concentration of crosslinker.

On the other hand, the composite polymerized in the mesophases exhibited the characteristic textures different from that polymerized in the isotropic phase. When the mixture of a liquid crystalline molecule and a crosslinker was cooled from the isotropic phase to the mesophases, the resulting textures was of islands rich in liquid crystals as shown in Figure 2(c) and (e). These islands structure were caused by characteristic phase separation of liquid crystal and crosslinker [17,18], and observed in the mixture of a smectic liquid crystal and an alcohol with a long alkyl chain [19] as well. These textures were maintained at a fixed temperature because of thermal equilibrium. After the photopolymerization in the mesophases, the resulting textures of the cells exhibited the phase-separated

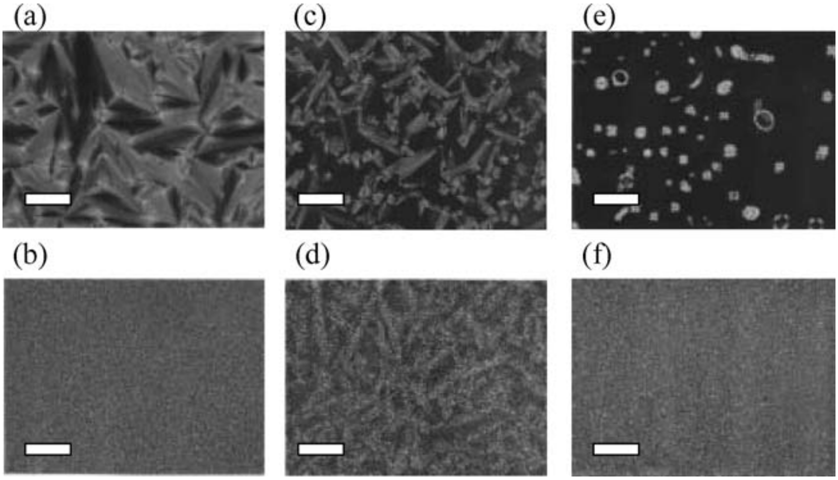

FIGURE 2 Micrographic textures of 8-PNP-O12 and crosslinked polymer composite. The white bar represents 200 mm.: (a) containing 5 wt% of HDA after polymerization in the isotropic phase (observation in the SmA phase at 110°C); (b) containing HDA 20 wt% after polymerization in the isotropic phase (observation in the SmA phase at 110°C); (c) containing 20 wt% of HDA before polymerization in the SmB phase (observation in the SmB phase at 70°C); (d) containing 20 wt% of HDA after polymerization in the SmB phase (observation in the SmA phase at 110°C); (e) containing 20 wt% of HDA before polymerization in the SmA phase (observation in the SmA phase at 93°C); (f) containing 20 wt% of HDA after polymerization in the SmA phase (observation in the SmA phase at 110°C).

structures reflecting the texture before polymerization, i.e. the liquid crystal rich islands in the SmB phase as shown in Figure 2(d). However, the homogeneous phase separated structures were obtained in the cell polymerized in the SmA phase as shown in Figure 3(f). This is probably due to the fluidity of liquid crystal in the SmA phase. The domain size of this composite fabricated in the mesophases was several μm when polymerized in the SmA phase to several hundreds μm when in the SmB phase even in the high concentration of HDA of 20 wt% crosslinker. In this case, the domain sizes were affected by the self-organization of the liquid crystalline molecules in addition to a concentration of crosslinker.

Charge Transport in Liquid Crystalline Semiconductor-crosslinked Polymer Composite

Figure 3 shows typical transient photocurrent curves of the composite cells polymerized in different phases. In the composite polymerized in the

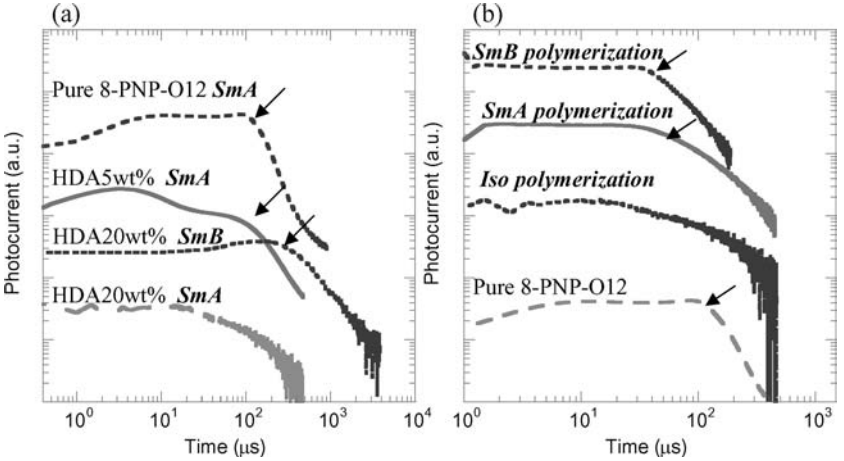

FIGURE 3 Typical double logarithm transient photocurrent curves in this composite and the pure 8-PNP-O12. The cell thickness and applied voltage are 9 mm and 100 V, respectively.: (a) transient photocurrent curves at the various phases in the case of the isotropic polymerization; (b) transient photocurrent curves at the SmA phase in the case of various phases polymerization.

isotropic phase, the shape of transient hole photocurrents depended on the concentration of crosslinker [13]: the transient photocurrent curves were non-dispersive at the HDA concentration of 5 wt% (see Fig. 2(a)), while the composite cells containing 20 wt% of crosslinker (see Fig. 2(b)) exhibited quite dispersive transient photocurrent curves in the SmA phase. Not only the photocurrent shape but also the mobility was affected by the HDA concentration. For example, in the SmB phase the hole mobility was one order of magnitude lower than that of the pure 8-PNP-O12, *i.e.*, $1.3 \times 10^{-4}\,\mathrm{cm}^2/\mathrm{Vs}$. In fact, the incorporation of crosslinked polymeric products in 8-PNP-O12 does not cause any deep defects states for holes because the crosslinked polymers derived from HDA have low HOMO levels relative to that of the 8-PNP-O12 molecule, but the resulting random polymer networks results in the domain size in the mesophases, which depends on the concentration of crosslinker. These results indicate the formation of shallow trap states associated with the resulting domain texture. However, it should be noted that the effect of the shallow trap is very small judging from the mobility reduction of one order of magnitude in spite of the big change in the domain size from several hundred μm to sub-μm [13].

On the other hand, when the composite cell was polymerized in mesophases, clear non-dispersive transient photocurrent curves were obtained even in the high HDA concentration over 20 wt%. The hole mobilities for the composite polymerized in the SmA phases at 93°C (Fig. 2(f)) and the SmB phase at 70°C (Fig. 2(d)) were very similar in the SmA phase at 110°C, *i.e.*, $1.8 \times 10^{-4}\,\mathrm{cm^2/Vs}$ and $1.7 \times 10^{-4}\,\mathrm{cm^2/Vs}$, respectively, which were increased in the SmB phase at 90°C up to $1.0 \times 10^{-3}\,\mathrm{cm^2/Vs}$. These mobilities were very similar to those in corresponding smectic mesophases of the pure 8-PNP-O12 [10]. As described above, the homogeneously polymerized networks in the isotropic phase affect seriously the charge transport even in the mesophases where the liquid crystalline molecules align in layers, while the polymer network formed in the smectic mesophases where the crosslinker and liquid crystalline molecule are phase-separated does not affect the charge transport at all. This indicates that the phase-separated structures maintained the self-organization of liquid crystalline molecules after the polymerization when polymerized in the mesophases and play a very important role not so as to degrade the charge transport properties in the resulting composite. In addition, when the polymerization takes place in the smectic mesophases, the domain sizes were larger than those in the case of isotropic phase polymerization. This fact also supports the fast charge mobility similar to those in the pure 8-PNP-O12. Judging from the clear non-dispersive transient photocurrent curves, the self-organization of liquid crystal in this composite perhaps assists the connection each domain boundaries.

On the contrary, the transient photocurrent curves in the crystalline phase were very dispersive in irrespective of polymerized conditions, probably because of serious defects formation at grain boundaries in the crystalline phase of 8-PNP-O12 as in the case of the pure 8-PNP-O12. Insides, we found that the electron transport was very much degraded after the polymerization irrespective of polymerization condition: the slow transit corresponding to the mobility on the order of $10^{-6}\,\mathrm{cm^2/Vs}$ in the SmB was observed and so on the order of $10^{-5}\,\mathrm{cm^2/Vs}$ in the SmA, indicating the reduced mobility due to the formation of shallow trapping states or the ionic conduction. Those results were discussed in the separate publication [20].

The charge mobility as a function of temperature in the present composite is shown in Figure 4. The mobilities of the comprised cell including 5 wt% HDA, when polymerized in the isotropic phase at 125°C, were almost constant within the temperature range for each mesophase as in the case of the pure 8-PNP-O12. And the hole mobility when the HDA concentration was 20 wt% was on the order of $10^{-4}\,\mathrm{cm^2/Vs}$, which is on order of magnitude smaller than that of the pure 8-PNP-O12 as discussed. Contrary to the cells polymerized in the isotropic phase, the hole mobilities in the cells polymerized in the mesophases do not depend on the temperature even in the high HDA concentration of 20 wt% as shown in Figure 4(b). It is worth noting that the charge transport in the composite containing as high as 20 wt%

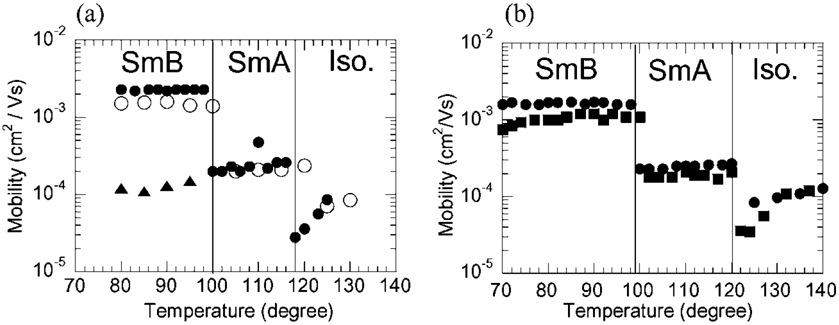

FIGURE 4 Mobilities in the different phases of 8-PNP-O12 and crosslinked polymer composite as a function of temperature. (a) in the case of the isotropic polymerization: HDA 5 wt%: filled circles, HDA 20 wt%: filled triangles (only the SmB phase), pure 8-PNP-O12: opened circles. (b) in the case of mesophase polymerized samples containing 20 wt% of HDA. The SmA phase polymerization: filled squares, the SmB phase polymerization: opened squares.

of crosslinker still keeps the excellent charge transport properties similar to that of the pure 8-PNP-O12 as a function of temperature.

In the molecularly doped polymers such as triphenylamine(TPA)-bisphenol A polycarbonate system [21], which is a solid solution or a homogeneous polymer composite, the charge carrier mobility is subject to the following Eq. (1):

$$\mu \propto r^2 \exp\left(-\frac{r}{2\gamma}\right) \tag{1}$$

where r is the average hopping distance and γ is a decay constant of molecular orbital [22]. This is due to the hopping charge carrier transport among the molecularly doped TPA molecules. Therefore, the molecularly doped polymers require usually a high concentration of charge transport molecules over 50 wt% and its mobility is limited by the highest concentration of TPA without crystallization because of deep defects formation associated with the crystallization, as we see those in polycrystals. Thus, in the molecularly doped polymers, the compatibility of charge transport molecules with the binding polymers is very important for those reasons. In the present composite, however, there is no such the concentration limitation because of the phase separation, and it keeps its own charge transport properties of 8-PNP-O12. This is owing to the self-organization of liquid crystalline semiconductors into the mesophase whose domain boundaries do not result in deep trapping states. In other words, the high mobility comparable to the pure transport material can be fabricated by the phase separation structures of liquid crystalline semiconductor and crosslinked polymer. The phase separated structures are generally investigated by SEM observation in PDLC and PSLC [14], however the SEM observation is necessary to wash up including liquid crystalline molecules by organic solvents so that it can not be done *in situ*.

Ionic Conduction in Liquid Crystalline Semiconductor-Crosslinked Polymer Composite

In general, the charge transport in the materials containing trapped states is generally explained by trap-controlled hopping process where the carriers are transported repeating trapping and detrapping process in and from trapped states. In the fluid media such as liquid crystal, however, the trapped carriers that are ionized impurity molecules can move as ions before being detrapped to the liquid crystalline molecule when the trap states is deep in energy. Thus, in the liquid crystalline materials, the ionic conduction takes place not in the isotropic phase but also the less-ordered smectic mesophase when contaminated with impurity materials responsible for deep trap states. Ionic transport depends on viscosity of the media and size of charged molecules, which is well-known as Walden rule:

$$\mu \cdot \eta^{m} = \frac{e}{6\pi r} \tag{2}$$

where η and r are the viscosity of media and the diameter of charged molecules, respectively and m is a parameter ($m = \tilde{1}2$) [16].

In this polymer composite, there exists the elastic interaction between liquid crystalline molecules and polymer network, so that we expect to suppress the ionic conduction. Therefore, we may expect that the electrical conduction in the SmA phase of contaminated composite with impurity shifts from ionic conduction to the electronic one while governed by the trap-controlled hopping conduction. In order to check this effect, we investigated the carrier transport properties of the polymer composites doped with a terthiophene derivative (6-TTP-12). Figure 5 shows the transient photocurrent curves of this composite containing the 6-TTP-12 molecule in the isotropic phase (Fig. 5(a)) and the SmA phase (Fig. 5(b)). In isotropic phase, the kink point indicating a transit time of the carriers was shifted to a longer time region clearly in the cell polymerized in the isotropic phase the charge mobility was one half of that of the pure 8-PNP-O12 doped with 6-TTP-12. The reduced mobility in this composite is due to enhanced macroscopic viscosity resulted from the elastic interaction between liquid crystal and polymer. However, the composite polymerized in the SmA (93°C) phase exhibited the kink point at the time region comparable to that of the pure 8-PNP-O12 doped with 6-TTP-12. This suggests the conduction in the SmA phase of the doped composite is still governed

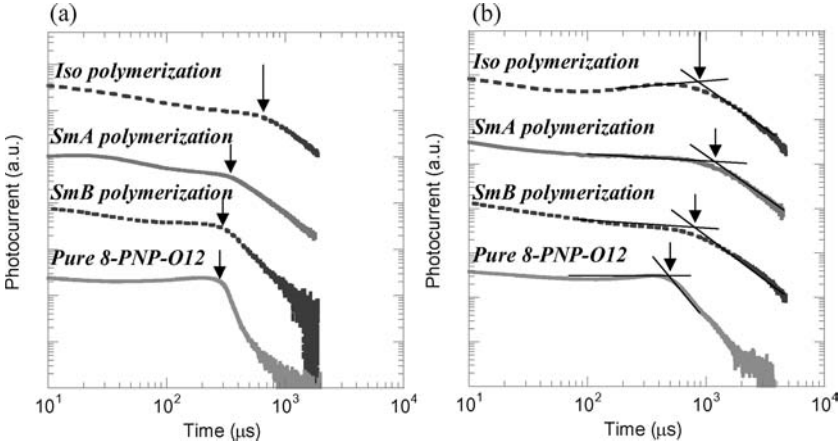

FIGURE 5 Transient photocurrent curves in the case of doping 0.001 wt% of 6-TTP-12. Cell thickness and applied voltage are 9 mm and 100 V, respectively. The kink points are shown by allow.: (a) measurements in the isotropic phase (125°C), (b) measurements in the SmA phase (110°C).

by ionic conduction. In the cells polymerized in the mesophases, liquid crystalline molecules can be less affected by the polymer network than the composite polymerized in the isotropic phase because of more sophisticated self-organized. This is probably the reason why the carrier transport in the SmA phase of the doped composite exhibits ionic behaviors contrary to pour expectation as described. Thus, it is concluded that the polymer network does increase the macroscopic viscosity by elastic interaction in the isotropic phase, but the well phase separated structure of the SmA phase minimizes the effect of the polymer network, resulting in the ionic conduction comparable to that in the SmA phase of the pure 8-PNP-O12 doped with 6-TTP-12.

CONCLUSIONS

A novel liquid crystalline semiconductor and crosslinked polymer composite has been fabricated and their charge transport properties were investigated. The charge transport properties of the composites was essentially different from the homogeneously or heterogeneously dispersed polymer films with organic semiconductors such as molecularly doped polymers. And they are comparable to those of a pure liquid crystalline semiconductor when polymerized in the smectic mesophases even in a high polymer concentration of 20 wt%, probably due to the phase sepatated structures in the composites. Furthermore, the mobility of ionic conduction, which is takes place in the isotropic phase and in the less ordered smectic mesophase when contaminated with impurity, was reduced by the elastic interaction between polymer and liquid crystals. This new polymer composite exhibits fixed molecular alignment and feasibility of film fabrication in addition to the excellent carrier transport properties and is promising for opto-electronics devices.

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