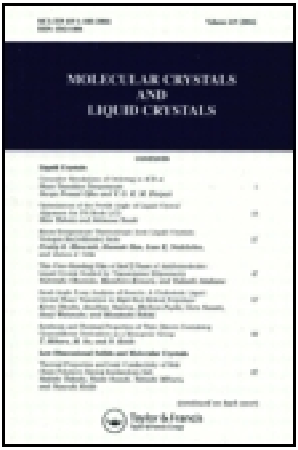

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Polarized Phthalocyanine Electroluminescence Diodes Prepared on Polysilane Films Treated with Polarized UV Light

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Photoalignment ability of poly[methyl(phenyl)silylene] (PMPSi) films makes possible to use them as hole transporting substrates for the preparation of organic oriented films. PMPSi layer prepared by spin coating was after drying irradiated with linearly polarized UV light. Then, water soluble hydroxyaluminium phthalocyaninesulfonate sodium salt [Al(OH)Pc(SO_3Na)$_{1\text{–}2}$] was deposited by casting. The cell ITO/PMPSi/Al(OH)Pc(SO_3Na)$_{1\text{–}2}$/Al showed non-linear current-voltage characteristics. For applied voltages higher than 20 V, a polarized electroluminescence was observed. Its polarizing anisotropy $R_{EL} = \phi_{\perp}/\phi_{\parallel}$ was ca. 2 and 2.1 for phthalocyanine film prepared from solution and by vacuum evaporation, respectively.

Keywords: hydroxyaluminium phthalocyaninesulfonate; photoalignment; polarized electroluminescence; poly[methyl(phenyl)silylene]

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INTRODUCTION

Organic light emitting diodes (OLEDs), as one of the most promising candidates for the next generation of flat panel displays, have been extensively studied. In the past decade many kinds of diodes with high quantum efficiency and long lifetime have been developed [1]. In such devices, light emission occurs due to the formation of singlet excitons generated by interactions of negative and positive charges which are injected from electrodes and subsequently drift toward each other under the applied electric field. Obviously, it is important to achieve well balanced charge injection and good charge carrier transport in order to obtain equal numbers of positive and negative charge carriers in the emissive region. Since the first demonstration of OLEDs the combination of electron transporting layer and/or hole transporting layer together with an emissive layer has been shown to be essential for improved device performance.

Several attempts have been made to prepare polarized OLEDs [2–5]. They can be used in many electro-optical applications. For example, the application of polarized OLED to a backlight for a liquid crystal display could save energy because such a device would be free from any additional polarization optics. The fabrication of polarized LEDs needs a special treatment (orientation) of the electroluminescent (EL) material. One way represents a uniaxially oriented film of the EL material. In the case of conjugated polymers the light is polarized along the polymer backbones due to their delocalized electronic states. Polarized EL from uniaxially oriented polymer films was obtained with devices where emissive polymer films were oriented by stretching [2] or rubbing [3] or by applying the Langmuir-Blodgett technique [4]. Large dichroic ratio was obtained by employing liquid crystalline conjugated polymers [5]. Recently, polarized EL was observed on spin-coated polysilane film deposited on friction-transferred insoluble polysilane substrate [6]. It indicates that the soluble polymer chains were aligned uniaxially onto the highly oriented friction-transferred film as a ‘template’ layer. Such an epitaxial-like fabrication process should be useful to obtain sufficient thickness for the device application. Another possibility how to obtain an oriented polymer surface is a rubbing technique. However, the development of new rubbing-less technique has been of a great interest during last years. Some of these techniques are based on photosensitive polymers.

Light sensitive poly[methyl(phenyl)silylene] (PMPSi) with significant electron delocalization along the chain [7] seems to be suitable substrate material for the preparation of oriented template layer. If it is irradiated with linearly polarized light, it shows an angular-dependent photoinduced cleavage of Si−Si bonds, formation of polysiloxane structures, and quasi-stable photogenerated ion-pairs (dipoles) preferentially within segments oriented along the light polarization [8]. Liquid crystals deposited on in this way treated PMPSi surface are oriented [9]. Thus, we can simply prepare electroluminescent diodes of the type ITO glass|hole transporting PMPSi film|oriented EL material|Al electrode, which can produce polarized electroluminescence. In this paper we report the fabrication and properties of polarized OLEDs based on bilayer structure consisting of PMPSi and phthalocyanine.

EXPERIMENTAL

Materials

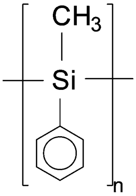
Poly[methyl(phenyl)silylene] (PMPSi, see Scheme 1a) was prepared by the Wurtz synthesis from the corresponding dichlorosilane. The monomer was reacted with a sodium dispersion in boiling toluene. Details of the synthesis are reported in Ref. [10].

Hydroxyaluminium phthalocyaninesulfonate sodium salt [Al(OH)Pc-$(SO_3Na)_{1\text{–}2}$], [see Scheme 1(b)] was prepared from hydroxyaluminium phthalocyanine [Al(OH)Pc] dissolved in 10% fuming sulfuric acid and subsequently heated (85°C, 6 h). The reaction mixture was purred into the mixture of water and ice, filtered and washed. The filter cake was dispersed in water and pH adjusted with NaOH to the value of about 11. Thus, [Al(OH)Pc] was converted into dark blue water soluble sodium salt, which was isolated by evaporation of water by using a water bath. The product was analyzed and purified by high pressure liquid chromatography method.

Copper phthalocyanine [CuPc, see Scheme 1(c)] was obtained from Tokyo Kasi and purified by sublimation.

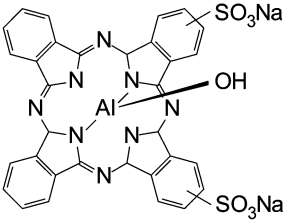
Samples and Measurements

Thin films of PMPSi thickness from 300 to 500 nm were prepared from a toluene solution by casting on conductive ITO glasses. Before the deposition of the films the polymer was three times reprecipitated from a toluene solution with methanol and centrifuged (12 000 rpm, 15 min). After the deposition the films were dried at the vacuum of 0.1 Pa at 330 K for at least 4 h. Then, the optical treatment of PMPSi layer was performed using linearly polarized light of He–Cd laser ($\lambda = 325\,\mathrm{nm}$, light intensity $P = 10\,\mathrm{mW/cm^2}$). The degradation process was checked by UV-Vis spectroscopy (Hitachi U300). On this template



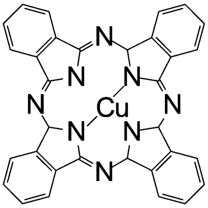
(a)

Poly[methyl(phenyl)silylene], (PMPSi)



(b)

Hydroxyaluminium phthalocyaninesulfonate sodium salt, Al(OH)Pc$(SO_3Na)_{1\text{-}2}$



(c)

Copper phthalocyanine, (CuPc)

SCHEME 1 Materials used in this work.

layer a [Al(OH)Pc$(SO_3Na)_{1-2}$] film was deposited by spin coating or CuPc film was vacuum evaporated. For the measurements of electroluminescence and current-voltage characteristics a top Al electrode, 40–60 nm thick, was prepared by the vacuum evaporation.

Electroluminescence was measured on sandwich samples of the type glass|ITO|PMPSi|Al(OH)Pc$(SO_3)_{1-2}$|Al. The emitted light was detected by Hamamatsu C4877 photomultiplier, current-voltage characteristics were measured using Keithley 6517 A electrometer. The samples were kept in a vacuum cryostat (10^{-3} Pa) or under helium or argon during the measurements. In all cases the same experimental results were obtained.

RESULTS AND DISCUSSION

The device under study, glass|ITO|PMPSi|Al(OH)Pc$(SO_3)_{1-2}$|Al, consisted of hole injection ITO electrode, hole transporting PMPSi film, emissive phthalocyanine layer and Al electrode. The aim of the paper is to present a new way of the preparation of polarized OLED; no detail study was carried out to get a maximum emission efficiency.

Charge Carrier Transport in PMPSi

Polysilanes consist of a chain of silicon atoms with interacting three sp^3 hybrid orbitals. The resonance integral between two sp^3 orbitals located on adjacent silicon atoms and pointing to each other, β_{vic}, is responsible for the formation of a Si−Si σ-bond. Electron delocalization along the silicon backbone results from the mutual interaction of sp^3 orbitals of adjacent silicon atoms [11]. The degree of electron delocalization in the backbone is a function of the $\beta_{\mathrm{vic}}/\beta_{\mathrm{gem}}$ ratio, where β_{gem} is the resonance integral between two sp^3 orbitals localized on the same silicon atom. Electron delocalization is perfect if the ratio equals unity. Thus, the linear Si backbone behaves as a molecular wire due to the σ-conjugation. The "on-chain" hole mobility in PMPSi was found to amount to $\mu \sim 2 \times 10^{-6}\,\mathrm{m^2V^{-1}s^{-1}}$ using time resolved microwave photoconductivity (TRMP) [12,13]. The long-time decay process in TRMP can be related to the intrachain recombination (Si chain cation radicals with phenyl anion radicals) and/or to the interchain electron-hole recombination. From the measurements of microwave conductivity the value of the "long-time" mobility was obtained as $2 \times 10^{-9}\,\mathrm{m^2V^{-1}s^{-1}}$ [14]). This value must be close to the zero-field ($F \rightarrow 0$) drift mobility measured on 3D samples as was indeed observed (at room temperature $\mu\,(F \rightarrow 0) \approx 10^{-9}\,\mathrm{m^2V^{-1}s^{-1}}$ [15].

Thus, the charge carrier movement in 3D samples of PMPSi consists of "on-chain" transport combined with hopping among the chains. The charge mobility μ fulfills the relation

$$\mu = \mu_0 \exp\left(-\frac{E_a}{kT}\right) \tag{1}$$

where μ_0 is the "on-chain" mobility, E_a is the activation energy of the mobility, k is the Boltzmann constant and T is the temperature.

Figure 1 illustrates the dependences of the charge carrier drift mobility, μ, of a 3D PMPSi sandwich sample (thickness ca. 2 µm) on F, the electric field strength, at several temperatures. In all cases, the mobility can be described by an exp $(\beta F^{1/2})$ dependence for $F > 10^7\,\mathrm{V\,m^{-1}}$. At lower field strengths, $\mu(F)$ becomes constant or increases slightly upon reducing F. This behaviour [16] is in agreement with the data published by Bässler *et al.* [17], but at variance with the data of Abkowitz and Stolka [18] who reported a ln μ vs. $\beta F^{1/2}$ dependence extending over two decades to $F = 10^6\,\mathrm{V\,m^{-1}}$. These types of dependences are usually treated in the framework of the hopping disorder concept.

However, the charge carrier transport in PMPSi and in polymers generally is strongly influenced by electron-phonon coupling. This interaction causes carrier self-trapping and creates a quasiparticle, a polaron, which can move only by carrying along the associated molecular deformation. The motion of such a charge carrier, dressed into a cloud of local deformation of the nuclear subsystem, can

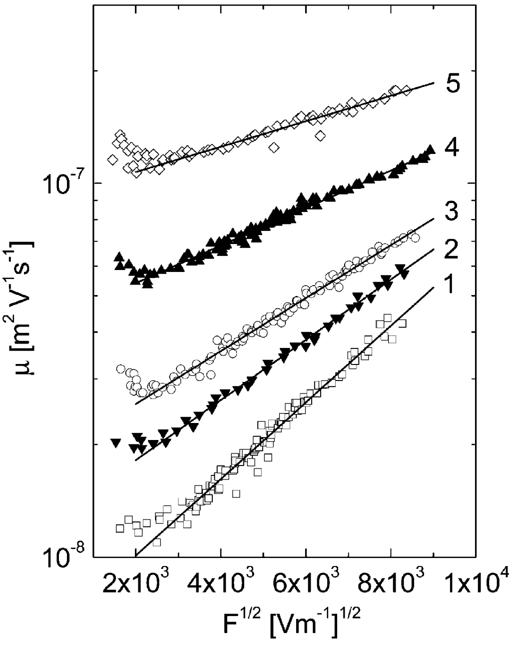


FIGURE 1 Electric field dependence of the mobility of PMPSi parametric in temperature: $T = 295\,\mathrm{K}$ (curve 1), 312 K (curve 2), 325 K (curve 3), 355 K (curve 4), and 358 K (curve 5).

be phenomenologically described by introducing a temperature-dependent effective mass which is higher than the electron mass. Another feature of the polymer chain is its deformation if a charge carrier is present. A significant distortion of the PMPSi chain was recently found by Kim *et al.* [19] by measuring the migration rate of the excitation energy along the polymer chain. The results of quantum-chemical calculations demonstrate that the presence of the charge in the macromolecule indeed deforms the chain [20].

The essential difference between the polaron and disorder models is that the latter, at variance with the former, implies a sufficiently weak electron-phonon coupling and the activation energy of charge transport reflects the static energy disorder of the hopping sites. In contrast, the polaron model suggests a strong electron-phonon coupling and a negligible contribution of energy disorder to the activation energy of the carrier mobility. Since the structural distortion is an intramolecular process, polaron binding energy E_p is not subjected to meaningful variation. Concomitantly, the polaronic charge transport must obey the Gaussian statistics and the photocurrent transients should neither feature a long tail nor become dispersive at any temperature. However, these predictions were not confirmed experimentally in polymers. In addition, the small polaron model fails to account for the observed Poole-Frenkel-type and non-Arrhenius field and temperature dependences of the thermal equilibrium carrier mobility. It was suggested [15,17,21] that the zero-field activation energy of the mobility, E_a $(F \to 0)$, can be approximated by the sum of the disorder and polaron contribution as

$$E_a(F \to 0) = E_a^{pol} + E_a^{dis} = \frac{E_p}{2} + \frac{4}{9}\frac{\sigma^2}{(kT)^2} \tag{2}$$

where E_a^{pol} and E_a^{dis} are the polaronic and disorder contributions, respectively, σ is the energy width of the density of states (DOS) distribution, and E_p is the polaron binding energy. Usually it is difficult to distinguish from experimental data between μ $(1/T^2)$ and μ $(1/T)$ dependences. Then, the second term of Eq. (2) can be treated as an apparent (effective) activation energy which can be expressed as $(8/9)$ σ^2/kT. The polaron binding energy was determined for PMPSi [17] as $E_p = 0.16\,\mathrm{eV}$. Recently, Pan *et al.* [22] reported a smaller value of $E_p = 0.08\,\mathrm{eV}$. The E_p values are still an open question and need more measurements and discussions. For PMPSi, the zero-field value of the activation energy of the mobility was measured as E_a $(F \to 0) = 0.29\,\mathrm{eV}$. Using Eq. (2) the value of the width of the distribution of hopping sites σ was determined as $\sigma = 0.078\,\mathrm{eV}$.

Preparation of Template Layer from PMPSi

Irradiation of the PMPSi film with linearly polarized light of He–Cd laser ($\lambda_{\max} = 325\,\text{nm}$) under ambient conditions produced changes of the UV spectra of the film. The changes (see Fig. 2) were found to be independent of the power density of the exciting light, but dependent on the exposure dose. The photoinduced changes were found to be angular dependent with respect to the polarization of the actinic light. The long-wavelength absorption band with the maximum at $\lambda_{\max} = 333\,\text{nm}$ (absorption coefficient $\alpha = 7.6 \times 10^4\,\text{cm}^{-1}$), which is characterized mainly by the delocalized (σ–σ^*) transitions in the Si backbone, is formed by the all-trans main chain segments separated by gauche conformations. Thus, a polysilane film is an assembly of energetically different, weakly coupled Si–Si chain segments. The exposure with the linearly polarized light results in the angular dependent excitation of the main chain segments, which are oriented parallel to the electric field vector of the exciting light.

The change in absorbance at 333 nm during the UV exposure is presented in Figure 3. The decrease in the absorbance was not very sensitive to light polarization. The absorbance parallel and perpendicular to the polarization of the actinic light decrease almost simultaneously. From

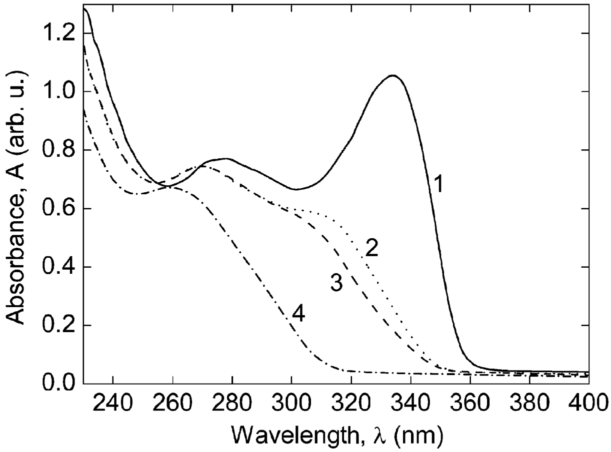


FIGURE 2 Changes in polarized UV spectra of PMPSi film on irradiation with linearly polarized light of He–Cd laser ($\lambda = 325\,\text{nm}$, $P = 10\,\text{mV}\,\text{cm}^{-2}$): (1) spectrum of as-prepared film; (2) spectrum parallel to the polarization of the exciting light after the exposure dose $0.2\,\text{J}\,\text{cm}^{-2}$; (3) spectrum perpendicular to the polarization of the exciting light after the exposure dose $0.2\,\text{J}\,\text{cm}^{-2}$; (4) spectrum measured after the exposure dose $12.0\,\text{J}\,\text{cm}^{-2}$.

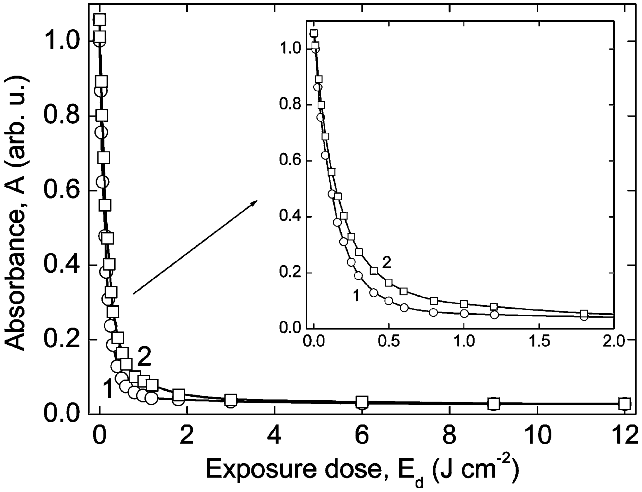


FIGURE 3 Changes of absorbance of PMPSi film at 333 nm on irradiation with linearly polarized light of He–Cd laser ($\lambda = 325\,\text{nm}$, $P = 10\,\text{mV}\,\text{cm}^{-2}$): (1) parallel to the polarization of the exciting light; (2) perpendicular to the polarization of the exciting light. Inset: Detail for low-exposure doses.

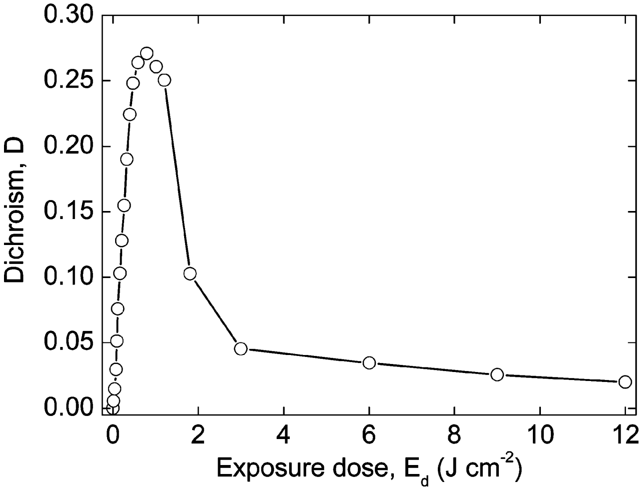


FIGURE 4 Changes of dichroism calculated at 333 nm on irradiation of PMPSi film with linearly polarized light of He–Cd laser ($\lambda = 325\,\text{nm}$, $P = 10\,\text{mV}\,\text{cm}^{-2}$).

the changes in absorbance at this wavelength the appropriate change of the dichroism $D = (A_{\max} - A_{\min})/(A_{\max} + A_{\min})$ was calculated (Fig. 4). Initially the dichroism increases with the conversion of PMPSi when long all-trans chain segments absorb. The dichroism reaches the maximum value ca. 0.27 at the exposure dose of about $1\,\mathrm{J\,cm^{-2}}$ when the vast majority of chain segments are photodegraded (the absorbance is less than 10% of the initial value) and on subsequent irradiation it practically goes to zero. The dichroism is related to the remaining, still intact chain segments perpendicular to the electric field vector of the incident light.

The polysilane segments oriented parallel to the electric field vector undergo photoinduced scission of the Si–Si backbones (quantum yield for films is 0.016 bond scission per photon). The photodegradation results in a decrease in molar mass of the polymer with the irradiation dose and in the shift of the absorbance maximum to shorter wavelengths (Fig. 2). During the photodegradation, polysiloxanes, polymer networks and metastable electronic states are also formed [8].

Electroluminescence

The current-voltage ($j \sim U$) characteristic of the cells ITO|PMPSi|Al (optically untreated PMPSi) is given in Figure 5, curve 1. Three main

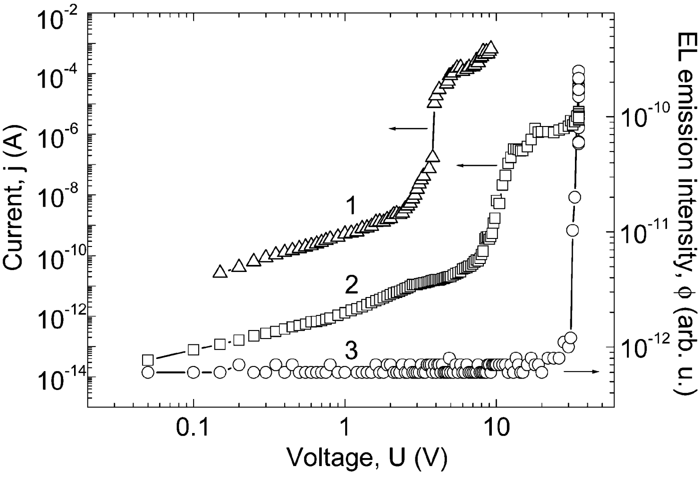


FIGURE 5 Current-voltage characteristics (curves 1 and 2) and electroluminescence emission intensity vs. applied voltage (curve 3) of the studied cells at room temperature. Curve 1–cell ITO|PMPSi (optically untreated)|Al, curve 2–cell ITO|PMPSi (optically treated) |Al(OH)Pc(SO_3Na)$_{1-2}$|Al, curve 3–cell ITO|PMPSi (optically treated) |Al(OH)Pc(SO_3Na)$_{1-2}$|Al.

parts can be distinguished. Low voltage part with linear and superlinear behaviour is followed by strong current jump. For voltages higher than 4 V a $j \sim U^2$ dependence was observed; in this part the current value $j = 1 \times 10^{-4}$ A for $U = 5.1$ V agrees well with the prediction of the space-charge-limited current (SCLC),

$$\frac{j}{S} = \frac{9}{8}\varepsilon\varepsilon_0\mu\frac{U^2}{d^3} \tag{3}$$

where S is the sample area, $\varepsilon\varepsilon_0$ is the electric permittivity and d is the sample thickness, if the charge carrier mobility is taken as $2 \times 10^{-8}\,\mathrm{m}^2\,\mathrm{V}^{-1}\,\mathrm{s}^{-1}$ (this value was obtained by time-of-flight experiments [15]). The strong current increase is influenced by charge injection.

The low voltage part of the $j \sim U$ characteristic can be described by the model based on emission-limited current with the participation of traps in the precontact region [23]. The traps influence the probability of a carrier to cross the barrier formed by the superposition of the image Coulombic and external potential. The current is influenced by thermoionic emission [24] and can be written in the form [25]

$$j \approx j_0 \exp(aU^{1/2} - bU^{-1/2}) \tag{4}$$

where

$$a = \frac{e}{kT}\left(\frac{e}{4\pi\varepsilon\varepsilon_0 d}\right)^{1/2}, \quad b = e\left(\frac{dH\nu_t\Sigma}{16\pi\varepsilon\varepsilon_0\mu kT}\right)^{1/2} \tag{5}$$

Here, H is the trap concentration, e is the unit charge, ν_t stands for the thermal velocity of the carriers and Σ for the cross section for the carrier trapping. According to Eq. (4) the current is driven by two processes; the lowering of the Schottky barrier which is propotional to $U^{1/2}$ (thus $j \sim \exp\,(aU^{1/2})$) and the trap control of the number of carriers arriving at the barrier with sufficient energy to cross it ($j \sim \exp\,(bU^{1/2})$).

After the optical treatment of the PMPSi film by UV light the current was generally lower. The strong increase of the current, caused by charge injection, was shifted to higher voltages. It was caused by the formation of metastable electron states, charge carrier traps and polysiloxane species [8]. Deposition of $\mathrm{Al(OH)Pc(SO_3Na)_{1\text{–}2}}$ thin film on optically treated PMPSi surface did not change the $j \sim U$ characteristic (curve 2 in Fig. 5) too much. The linear part of the characteristic in the

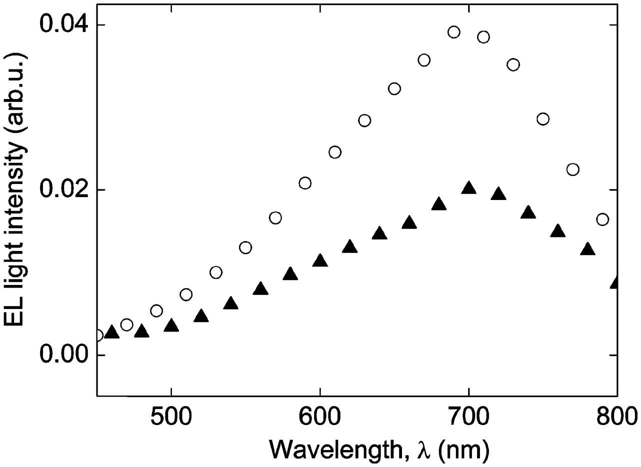


FIGURE 6 Polarized EL spectra of the cell ITO|PMPSi (optically treated) |AlOHPc(SO_3Na)$_{1-2}$|Al measured at 80 V. The polarizing directions are parallel (solid triangle) or perpendicular (open circle) to the electric vector of the photoaligning light.

voltage region 3–6 V, which follows the superlinear part of the dependence, suggests the formation of potential barrier. It is assumed that the barrier is formed on the interface PMPSi|Al(OH)Pc(SO_3Na)$_{1-2}$ because the ionization potential of phthalocyanine is lower than the ionization potential of PMPSi (5.6 eV) and hole traps can be formed. From the high-voltage ($U > 12$ V) part of the $j \sim U^2$ dependence follows that the charge carrier mobility in the device ITO|PMPSi (phototreated)|Al(OH)Pc-SO_3Na)$_{1-2}$|Al is about three orders of magnitude lower than in the cell with nontreated PMPSi and without Al(OH)Pc-(SO_3Na)$_{1-2}$ film. Curve 3 represents the dependence of the electroluminescent (EL) intensity on applied voltage. The emission starts at voltages higher than 20 V where strong charge injection can be detected. It could be pointed out that the EL was polarized. The polarized spectra for soluble Al(OH)Pc(SO_3Na)$_{1-2}$ are shown in Figure 6. The spectra are typical for the phthalocyanine films [6] with maxima at about 700 nm; their polarizing anisotropy $R_{EL} = \Phi_{\perp}/\Phi_{\parallel}$ was about 2.

It could be pointed out that the oriented electroluminescent films can be deposited on optically treated PMPSi surface not only from solution but also by vacuum evaporation. It was demonstrated with CuPc films. The polar diagram of EL of the cell glass |ITO|PMPSi|CuPc|Al is shown in Figure 7 (the thickness of CuPc film was 80 nm). The polarizing ratio R_{EL} was about 2.1.

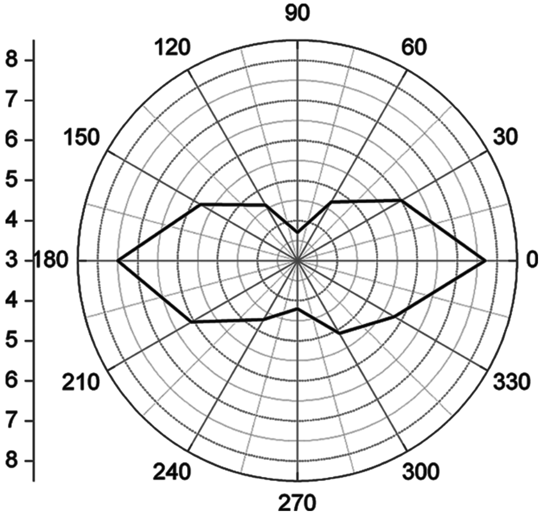


FIGURE 7 Polar diagram of EL of the cell ITO|PMPSi (optically treated) |CuPc|Al.

CONCLUSION

Polarized organic light emitting diodes of the type ITO glass|photodegraded PMPSi|phthalocyanines|Al can be prepared using PMPSi films treated by polarized UV light. The EL films of phthalocyanines deposited on the treated PMPSi surfaces from solution or by vacuum evaporation show oriented structures, which produce polarized electroluminescence. It seems that the method can be generalized: many others electroluminescent materials deposited on the surface of PMPSi treated by polarized light both from solution and by vacuum evaporation showed polarized electroluminescent characteristics.

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