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Invited Paper

# POLYSILANES – ADVANCED MATERIALS FOR OPTOELECTRONICS

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The electronic structures, electrical and photoelectrical properties and possible electronic applications of polysilanes are discussed. The electronic properties are influenced by electron delocalization along the polymer silicon backbone. Attached  $\pi$ -conjugated chromophores improve the photostability of the silicon main chain, and influence the band gap energy and electrical behaviour. Optical excitations lead to the formation of excitons, electron-hole pairs and charge transfer states (ion-pairs). Under the influence of an external electric field, these can dissociate into free charge carriers. Charge carrier transport can be described by a model of disordered polarons.

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# 1. Introduction

Polysilanes (sometimes called polysilylenes, polyorganosilanes, poly(silanediyl)s, organopolysilanes, or catena-silicon polymers) are of considerable research interest, because of their electronic, photoelectrical, and non-linear optical properties, and the effect of  $\sigma$ -electron delocalization along the chain [1]. Their optical and electrical properties differ significantly from structurally analogous carbon-based  $\sigma$ -bound systems, e.g. polystyrene and polyethylene, resembling rather fully  $\pi$ -conjugated systems like polyacetylenes. They are good charge transport materials. The carrier mobility is quite high, and can be influenced by additives and dipolar species. The "on-chain" mobility, ~ 10<sup>-6</sup> m<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, allows their use as molecular wires. The physical properties are strongly influenced by the chemical structure of the polymer side groups; various polymers with photoconductive, ionic, thermochromic, piezochromic, photorefractive, electrooptical, non-linear optical, and liquid-crystalline properties can be synthesized [2]. These properties can be utilized in electronic devices like photorefractive memories, photodetectors, light emitting diodes and displays.

## 2. Electronic structure

Polysilanes are usually amorphous materials which can also contain crystalline phases, amorphous silicon, silicon clusters and silane oligomers. Because of their mainly amorphous character one can start considering their electronic structure with the knowledge of that of amorphous silicon. The s and p states of the isolated atom combine to form the sp<sup>3</sup> hybrid orbitals of the tetrahedral silicon bonding. In a solid, the electron interaction splits the valence states into bonding and

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antibonding levels. These are split by bonding interactions to form the valence and conduction bands, separated by a band gap. Material disorder also results in the creation of band tail states.

Polysilane chains, with two organic groups on each silicon atom, behave as one-dimensional systems with weak intermolecular interactions. The resonance integral between two sp<sup>3</sup> orbitals located on adjacent silicon atoms and pointing towards each other,  $\beta_{vic}$ , is responsible for the formation of a Si–Si  $\sigma$ -bond. The degree of electron delocalization is a function of the ratio  $\beta_{vic}/\beta_{gem}$ , where  $\beta_{gem}$  is the resonance integral between two sp<sup>3</sup> orbitals located on the same silicon atom. Electron delocalization is perfect if the ratio is unity. Side groups attached to the Si backbone strongly influence the electronic properties of the polymer, as demonstrated by, e.g., fluorescence spectra [3]. The photostability of the silicon backbone is similarly influenced. Attached  $\pi$ -conjugated chromophores reduce the chain degradation.



Fig. 1. Chemical structures of materials in this paper, and the energy diagram of PMPSi: (a) free macromolecule, (b) neutral states in the solid state, (c) ionized states in the solid state. (i) Optical absorption, (ii) luminescence,  $(\sigma, \pi^*)^{CT}$  – charge transfer state,  $A_c$  – electron affinity,  $I_c$  – ionization potential,  $E_g$  – energy gap, P – polarization energy,  $E_{BP}$  – energy of branching points, ET – electron traps, HT – hole traps, MES – metastable electron states,

#### MHS - metastable hole states.

The energy structure of polysilanes will be discussed in detail on a model material, poly[methyl(phenyl)silylene] (PMPSi, see Fig. 1). Three bands have been detected in its absorption spectrum [4]. The dielectric function provides a reasonable fit of the reflectance throughout the measurement range. While the high-energy bands have single broad Gauss-Lorentz profiles, the lowest-energy band seems to feature a cluster of several components. It has been shown [5] that the longest wavelength absorption band ( $\lambda_{max} = 338$  nm for solid PMPSi films) is formed mainly by delocalized ( $\sigma$ - $\sigma^*$ ) transitions. The energy of these transitions is conformation-dependent and depends strongly on the effective conjugation length of the molecule. The contribution of ( $\pi$ - $\pi^*$ ) transitions to the longest wavelength band is weak in PMPSi. In polymers with fused-ring aryl substituents (e.g., naphthyl, biphenylyl), this absorption is better characterized as a localized ( $\pi$ - $\pi^*$ ) excitation with

participation of a weaker  $(\sigma - \sigma^*)$  one. The  $(\pi - \pi^*)$  aryl-like excitations are responsible for the shorter wavelength absorption (maximum at ~ 276 nm for solid PMPSi). Using a  $(h\nu \times \alpha_0)^2$  vs.  $h\nu$  plot  $(\alpha_0$  is the absorption coefficient), the band gap energy of PMPSi has been estimated as  $E_g = 3.5$  eV. Its lowering compared with the value of 4.5 eV for poly(dialkylsilylene)s was ascribed to a  $(\sigma, \pi)$  band mixing at the valence band edge state between skeleton Si 3p and side-chain phenyl  $\pi$  HOMO states. The energy diagram is given schematically in Fig. 1.

From fluorescence emission measurements, the photoluminescence of PMPSi consists of a relatively sharp band with a maximum at 357 nm, plus a broad emission in the visible spectrum. The latter is enhanced at low temperatures and during excitation by short wavelength light  $((\pi - \pi^*)$  absorption). At least four sub-bands were detected at low temperatures: (i) charge transfer  ${}^{1}(\sigma, \pi^*)^{CT}$  states (410–500 nm); (ii) branching points (energy  $E_{BP}$ , see Fig. 1), (iii) tail state transitions ET $\rightarrow$ HT, and (iv) defect metastable states formed by backbone deformation or scission ( $\lambda = 520-540$  nm),. From the excitation spectrum, it follows that direct radiationless deactivation processes from both the  ${}^{1}(\pi, \pi^*)$  and the  ${}^{1}(\sigma, \sigma^*)$  state to the charge transfer  ${}^{1}(\sigma, \pi^*)^{CT}$  state can be performed.

# 3. Experimental details

Poly[methyl(phenyl)silylene] (PMPSi, - Fig. 1) and poly[biphenyl-4-yl(methyl)silylene] (PBMSi) have been prepared by sodium-mediated Wurtz-coupling polymerization in boiling toluene [6,7]. Chemical functionalization is a potential route for synthesizing polysilanes containing functional groups attached to some of the pending substituents. Wurtz coupling remains the dominant synthetic route, but due to the harsh conditions, sensitive functional groups must very often be introduced after the polymerization. Using, e.g., chloromethylation, quaternization, the Kroehnke reaction and acid hydrolysis, the poly[methyl(phenyl)silylene/(3(4)-{[(2,4-dinitrophenyl) hydrazono]methyl}phenyl)methylsilylene] (PMPSi-DNPH) copolymer was prepared [8].

Thin  $(0.3-2 \ \mu\text{m})$  films were prepared from a toluene solution by casting onto stainless steel substrates and ITO-coated glass for xerographic discharge, time-of-flight (carrier mobility [7]) or steady state photoconductivity measurements. In the latter cases, top Al electrodes, 40-60 nm thick, were prepared by vacuum evaporation. Before deposition, the polymer was purified three times by precipitation into methanol and centrifuging (12,000 rpm, 15 min). After deposition, the films were dried at 0.1 Pa at 330 K for at least 4 h. The quantum efficiency of carrier photogeneration was measured by the xerographic discharge technique. The electrical conductivity and the electroluminescence were measured at  $10^{-3}$  Pa on sandwich samples.

### 4. Results

# 4.1. Electrical conductivity

The activation energy of the dark current was 0.9 eV. Taking the density of states as  $N_b = 4 \times 10^{27}$  m<sup>-3</sup>, the thermodynamic charge carrier concentration was expected to be  $n_f = 3 \times 10^{12}$  m<sup>-3</sup> at room temperature. Thus, PMPSi is a large band gap semiconductor. The electrical conductivity is  $\sigma = en_f \mu = 4 \times 10^{-15}$  S m<sup>-1</sup>, taking the zero-field mobility as  $8 \times 10^{-9}$  m<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> [7].

The current-voltage characteristic is given in Fig. 2a. Two main parts can be distinguished. The low-voltage regions with linear and superlinear dependences is followed by a strong current jump to the  $j \sim U^2$  behaviour. Here, the current value  $j = 10^4$  A at 5.1 V agrees well with the predicted space-charge-limited injection current (if the carrier mobility is taken as  $2 \times 10^{-8}$  m<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> at an electric field  $F = 2.5 \times 10^7$  V m<sup>-1</sup> [7]). However, the profile of the low-voltage part of the characteristic is more complex. One can assume a model of emission-limited current, with the participation of traps. These influence the probability of a carrier crossing the barrier formed by the superposition of the image Coulombic and external potentials. The current can be expressed as [9]:

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

where *a* and *b* are constants. According to Eq. (1), the current is driven by two processes: lowering of the Schottky barrier which is proportional to  $U^{1/2}$  (thus  $j \sim \exp(aU^{1/2})$  and trap control of the number of carriers arriving at the barrier with sufficient energy to cross it ( $j \sim \exp(-bU^{1/2})$ ). The plot ln *I* vs.  $(aU^{1/2} - bU^{-1/2})$  is given in Fig. 2b. The low-voltage part of the characteristic gives a good agreement with the proposed model. For high voltages, we assume that the traps are filled and the transition into the trap-free regime is realized.



Fig. 2. (a) Current-voltage characteristic ( $\Box$ ) and the dependences of the electroluminescence intensity on applied voltage ( $\circ$ ) for a PMPSi film. (b) Current-voltage characteristic replotted as log *I* vs.  $aU^{1/2} - bU^{-1/2}$ .

The formation of the energy barrier near the contact allows one to construct diodes. For higher voltages, holes are injected into the sample, and they recombine with electrons near the Al electrode. In this way, electroluminescent diodes can be constructed [10]. Figure 2a gives the dependences of the current and light emission on the applied voltage for this type of the device.

The low conductivity of PMPSi films can be increased by acceptor doping. For example, doping in iodine vapor gives  $\sigma = 10^{-4}$  S m<sup>-1</sup>. This value is much lower than that of  $\pi$ -conjugated polymers. The main reason is that during doping, polaron formation results in the deformation and scission of the Si backbone - the effective conjugation length is reduced and the mobility decreases.

The  $j\sim U$  dependences under illumination show a similar character to the dark ones. From the kinetic curve in Fig. 3a, the transient photo-response consists of a sharp increase, followed by a slower component and saturation. The slow decay after the light is switched off is influenced by the formation of metastable localized states due to the backbone deformation during exciton and polaron formation. This effect is evident in the case of strong UV irradiation, where no saturation part is present during illumination (see Fig. 3b) and the photocurrent immediately decreases due to space-charge effects and trap formation. The intensity dependence of the photocurrent suggests a single-photon process. The spectral response follows the absorption spectrum.



Fig. 3. Photocurrent kinetics of a PMPSi film under illumination by light of  $\lambda = 340$  nm. (a) Light intensity 0.3 mW cm<sup>-2</sup>, (b) light intensity 20 mW cm<sup>-2</sup>.

#### 4.2. Charge carrier photogeneration

The electric field (F) dependences of the photogeneration efficiencies, as measured by the xerographic discharge method (gas contact illuminated) are shown in Fig. 4. They can be fitted using the Onsager theory of geminate recombination (full line) [11]. This allows one to describe one step in the photogeneration process: the thermal dissociation of ion-pairs formed by light into free charge carriers under the influence of an external electric field. The best theoretical fit to the experimental data in Fig. 4 was for a Gaussian distribution of the radii of the ion-pairs in the CT state  $g(r) = (\pi \bar{r})$  $^{3/2}\alpha^3$ ) exp( $-r^2/\alpha^2$ ) with parameters  $\alpha = 1.3$  nm and a high-field photogeneration efficiency (fraction of ion-pairs generated per photon) of  $\eta_0 = 0.45$ . The activation energy of  $\eta_0$  was determined as  $E_n = 0.05$ eV. The temperature dependence of the photogeneration efficiency is also in good agreement with the Onsager dissociation theory. Note that large  $\pi$ -conjugated electronic systems in side groups lead to higher photogeneration efficiencies. This can be demonstrated with PBMSi, for which  $\alpha = 1.6$  nm and  $\eta_0 = 0.85$ . A similar effect can be obtained with polysilanes containing strong-acceptor side groups. The parameters for the copolymer PMPSi-DNPH were  $\alpha = 1.6$  nm,  $\eta_0 = 0.56$ . This copolymer also exhibits photoconductivity in the visible region. The electron acceptor groups in the side polymer chains effectively increase the ion-pair separations, limiting geminate recombination and increasing the quantum efficiency. The present results were obtained by discharge measurements using the gas type of the illuminated contact. However, due to the contact barrier in sandwich samples, the characteristics of the quantum efficiency vs. applied voltage are usually deformed due to the barrier internal field. This is demonstrated in Fig. 4, for an ITO/PMPSi/Al sandwich sample. In this case it is very difficult to get the exact value of the photogeneration efficiency.

Taking into consideration the experimental facts, one can summarize as follows. The illumination of polysilanes leads to several species. In the first step, an exciton is formed. The reverse reaction leads to the photoluminescence. An electron from the excited Si-Si bond is transferred to the antibonding state. Due to the  $\sigma$ -conjugation, it can then move through the polymer chain. In this way, on-chain ion-pairs (electron-hole pairs) are formed. The antibonding electrons move back and recombine due to electrostatic interactions, jump to another chain to form interchain charge transfer (CT) states, or jump to a side group (phenyl in our case) to form intrachain charge-transfer ( $\sigma$ ,  $\pi^*$ )<sup>CT</sup> excitons. Electron transfer from the main chain to the phenyl is quite possible because the ground state is predominantly  $\sigma_{Si-Si}$ , i.e. the ionization potential is  $I_p(Si-Si) < I_p(benzene)$ . At higher light intensities and long exposure times, silyl and silylene radicals are formed, and Si-Si bond scission can occur. The formation of the polar on-chain ion-pairs was detected by time-resolved microwave photoconductivity (TRMP) [12]. This technique is very sensitive to correlated charges (i.e. dipoles). Thus, the decay curve of the microwave signal reflects the geminate recombination kinetics of the pairs generated on the main chain of the polymer. From the half-life, the value of the "on-chain" mobility of holes (cation-radicals) was determined as  $\mu_c = 2 \times 10^{-6} \text{ m}^2 \text{ V}^{-1} \text{ s}^{-1}$ . The fast recombination was followed by a slow process. Its kinetics could be fitted to a stretched exponential function, up to the millisecond range. This process could be related to intrachain recombination (Si chain cation-radicals with phenyl anion-radicals) and/or to interchain electron-hole recombination.

#### 4.3. Charge carrier transport

In the previous section, we presented the value of the "on-chain" charge carrier mobility. A question arises as to why the value is so low. A possible reason is the formation of polarons. The strong electron-phonon coupling 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 in a cloud of local deformation of the nuclear subsystem, can be

phenomenologically described by introducing a temperature-dependent effective mass which is higher than the electron mass. A significant distortion of the PMPSi chain was found by Kim *et al.* [13] by measuring the migration rate of the excitation energy along the polymer chain. Quantum-chemical calculations demonstrate that the presence of the charge in the molecule also deforms the chain [12]. Experimentally, polarons can be detected by the thermostimulated luminescence method [14].





Fig. 4. Electric field dependences of the photogeneration efficiencies of PMPSi, PBMSi, PMPSi-DNPH (gas contact) and a sandwich sample ITO/PMPSi/Al.

Fig. 5. Electric field dependences of the charge mobilities. PMPSi, curves 1-5, T = 295, 312, 325, 355, 358 K. PBMSi, curve 6, T = 295 K. PMPSi-

DNPH, curve 7, T = 293 K.

Fig. 5 illustrates the dependences of the carrier drift mobility of a 3D PMPSi sandwich sample (thickness  $\sim 2 \mu m$ ) on the electric field strength, at several temperatures. For comparison, the dependences for PBMSi and PMPSi-DNPH are included. In all cases, the mobility can be described by an  $\exp(\beta F^{1/2})$  dependence for  $F > 10^7$  V m<sup>-1</sup>. Such dependences are usually treated within the framework of the hopping disorder concept. The essential difference between the polaron and disorder models is that the latter implies a sufficiently weak electron-phonon coupling, and the activation energy of the 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 mobility activation energy. Since structural distortion is an intramolecular process, the polaron binding energy  $E_p$  is not subjected to meaningful variation. Concomitantly, the polaronic charge transport must obey 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 for 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 has been suggested [7,15] that the zero-field mobility,  $\mu$  (T, F  $\rightarrow$  0), can be approximated by the sum of the polaron and disorder contributions as

$$\mu(T, F \to 0) = \mu_0 \exp\left[-\left(\frac{E_p}{2kT} + \frac{4}{9}\frac{\sigma^2}{(kT)^2}\right)\right]$$
(2)

where  $\sigma$  is the energy width of the density-of-states (DOS) distribution and  $\mu_0$  is the mobility prefactor. Usually, it is difficult to distinguish from experimental data between  $\mu$  (1/*T*) and  $\mu$  (1/*T*<sup>2</sup>) dependences. Then, the exponential term in Eq. (2) can be treated as a temperature-dependent contribution to the apparent (effective) activation energy  $E_a^{\text{eff}} = E_p/2 + (8/9) \sigma^2/kT$ .

The polaron binding energy has been determined for PMPSi [16] as 0.16 eV, and for PBMSi as 0.22 eV [17]. These values are still questionable, and need more attention. It is interesting to compare the transport properties of mixed PMPSi and PBMSi systems or the copolymers PMPSi-

PBMSi. The mobilities for these systems cover the interval between those for PMPSi and PBMSi. Whereas the concentration dependence of the mobilities of the copolymers on the concentration ratio (PBMSi:PMPSi) is nearly a linear function (see Fig. 6), the dependence for mixtures is more complex. This can follow from the formation of clusters of particular macromolecules.

For PMPSi, the zero-field value of the activation energy of the mobility has been measured as  $E_a^*(F \to 0) = 0.29 \text{ eV}$  [7]. The effective value of the half-width of the Gaussian distribution of the hopping states  $\sigma^*$  was determined from the equation  $E_a^*(F \to 0) = (8/9) \sigma^{*2}/kT$  as 0.093 eV (at room temperature). From TSL studies, the value  $\sigma^* = 0.096$  eV was obtained, in good agreement with the transport data [18]. The real value of the width of the distribution of hopping sites,  $\sigma$ , would be lowered by the polaron contribution ( $E_a^* - E_p/2 = 8 \sigma^2/9 kT$ ); this yields  $\sigma = 0.078$  eV. Both aromatic and polar groups in the side chains decrease the charge carrier mobility.



Fig. 6. Dependence of the charge mobility on the PBMSi:PMPSi ratio for copolymers (0) and mixtures (1).

Detailed analysis of the latter case suggests that charge-dipole interactions are important. Two effects must be mentioned in this context: (1) Broadening of the energy distribution of the hopping states [17]; the mobility decreases with increasing dipole moment), and on the molecular dimensions of the additive and the additive concentration. (2) Creation of new local states in the vicinity of dipolar species, even though these molecules themselves do not necessarily act as trapping sites. In contrast to the situation encountered in the case of both chemical and structural traps, the electron and hole traps are formed in different molecules: those close to the negative pole of the dipole should be hole traps, whereas those close to its positive pole should trap electrons.

### 5. Conclusions

Polysilanes are of considerable research interest, because of their electronic and photoelectrical properties and the effect of  $\sigma$ -electron delocalization along the chain. They are good charge carrier transport materials with quite high charge carrier mobilities. They behave as large band gap semiconductors, with band gaps ~ 3.5 eV. After doping with strong acceptors, the conductivity increases by 11 orders of magnitude, so that polysilanes can be used for antistatic covers. The photoconductivity follows the absorption spectrum, and the kinetic behaviour is influenced by space-charge effects. The formation of excitons and polarons results in a deformation of the backbone, and metastable electronic states are formed. After strong UV illumination, silyl radicals are formed and Si–Si bond scission can be observed. This effect can be utilized in resist technologies and for the preparation of oriented substrates for liquid crystal alignment.

Electronic transitions in polysilanes are influenced by the chain one-dimensionality and weak intermolecular interactions. Besides the delocalized  ${}^{1}(\sigma,\sigma^{*})$  (main chain) and  ${}^{1}(\pi,\pi^{*})$  (side

groups) excited states, the charge transfer  ${}^{1}(\sigma,\pi^{*})^{CT}$  states play an important role in the photoconductivity, luminescence and two-photon processes. The electron-hole pairs or ion-pairs created by light can dissociate under the influence of an external electric field, and free charge carriers can be formed. Their transport through the sample is strongly influenced by disorder and polaron formation. The pre-contact barrier, which arises at the metal/polysilane interface, allows the fabrication of diodes. Simultaneous injection of electrons and holes, and their subsequent recombination, allows the fabrication of UV light emitting diodes.

Even though polysilanes are not very stable under UV illumination – some progress has recently been made in their photostabilization – good solubility and the ability to form thin films of good optical quality, optical non-linearity, efficient luminescence, high carrier photogeneration efficiency and mobility suggest the future applicability of this group of polymers in optoelectronics.

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