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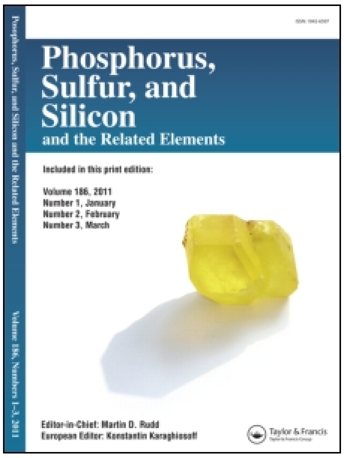

## Phosphorus, Sulfur, and Silicon and the Related Elements

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# Applications of Inorganic Polymeric Materials II: Polysilanes

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# Applications of Inorganic Polymeric Materials II: Polysilanes

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*Polysilanes which cover one of the most attractive and challenging fields, are high molecular weight polymers with inorganic elements in their backbone. These materials have silicon atoms in their main chain and exhibit unique properties resulting from the easy delocalization of sigma electrons in the Si—Si bonds. There are many technical uses as well as applications of these materials such as precursors of silicon carbide ceramics; a strengthening agent in porous ceramic; imaging materials in microlithography; photoiniting in radical reactions; and photoconducting, conducting, and semiconducting which are due to the unusual mobility of sigma electrons. In this work, the main structural features, properties, and some other important and recent applications of polysilanes are discussed.*

**Keywords** Inorganic polymers; novel applications; properties; structural features; polysilanes

## INTRODUCTION

Inorganic polymer materials comprise a very unique field of polymer science. Recent growing interest in this field is related to the unusual properties of these compounds. Most of these polymers, which have inorganic elements in their backbone, are based on silicon, e.g., polysiloxanes, polysilanes, polysilazanes, and polycarbosilanes. A significant amount of works that deal with totally or partially inorganic polymers have been published in recent years.[1–3]

Polysilanes is the popular term used to describe a whole family of organo-silicon compounds based on a backbone or molecular chain of silicon atoms. They show unique properties resulting from the easy delocalization of sigma electrons in the Si—Si bonds.[4–8] Many of their

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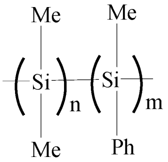
applications and unusual properties result from this unusual mobility. For example, strong electronic absorption, conductivity, photoconductivity, photosensitivity, and special physical properties are due to sigma electron delocalization.[9–14] In this work, we briefly will discuss some more important structural features and properties, synthesis methods, and general applications of polysilanes, and then focus on some more novel applications that have been explored recently.

## DISCUSSION

Structurally, polysilanes or polysilylenes are closely related to the homoatomic organic polymers such as polyolefins. They have a linear backbone of silicon atoms with two organic side groups attached to each silicon atom. The structure of materials may include chains, rings, or a three-dimensional network of silicon atoms that are joined by covalent bonds.[15] The chemical structures of some of the polysilanes are shown in Scheme 1.

poly (dimethyl silane)

poly (methyl pheny silane)



poly (trimethyl phenyl silane)

**SCHEME 1** The chemical structures of some of polysilanes.

The first polysilane derivatives were prepared in the 1920s by Kipping via the Wurtz-type coupling of arylsubstituted dichlorosilanes. These materials were intractable and poorly characterized. But the first soluble high molecular weight materials were synthesized in late 1970s and early 1980s.[15]

Several synthetic methods of polysilanes have been reported that include the conventional Wurts-type coupling of halosilanes, the ring opening polymerization of cyclic oligosilanes,[15] the anionic ring opening polymerization of cyclic oligosilanes,[16] the catalytic dehydrogenative coupling of hydrosilanes,[17] electrochemical reduction of halosilanes (electrosynthesis),[18] photochemical vapor deposition (photo-CVD method) of small cyclic oligosilanes,[19] and the catalytic disproporationation reaction of alkyoxydisilanes for the preparation of polysilanes' network.[20,21] Polysilanes are usually synthesized by the following methods:

- condensation reactions of both homopolymers and or copolymers, and

$$RR'SiCl_2 \xrightarrow[\text{Toluene}]{\text{Na, heat}} (RR'Si)_n \tag{1}$$

$$R_1R_2SiCl_2 + R_3R_4SiCl_2 \xrightarrow[\text{Toluene}]{\text{Na, heat}} (R_1R_2Si)_n(R_3R_4Si)_n \tag{2}$$

- the dehydrogenation of diorganosilanes.

$$nRR'SiH_2 \xrightarrow{\text{Transition metal}} H(SiRR')_nH + (n-1)H_2 \tag{3}$$

Physical properties of the polysilanes such as solubility and m.p. depend greatly on the molecular weight and also on the nature of the organic groups attached to the silicon atoms. The molecular weight of polysilanes depends on the methods of synthesis.[22] Polysilanes cover all the range of properties from highly crystalline; and insoluble, to partially crystalline; and flexible solid, glassy amorphous materials; and rubbery elastomers.[23] Polysilanes such as $(Ph_2Si)_n$ are crystalline, but polysilanes with less symmetrical structures have lower crystallinity. Linear polysilanes are thermoplastic, and soluble in organic solvents like toluene, ethers, and chlorinated hydrocarbons but are insoluble in alcohols.[24]

The glass transition temperature ($T_g$) of soluble polysilanes ranges $\sim -50 > 120^\circ C$ depending on the nature of organic substituents bonded to the polymer backbone.[22,25] For example, polymers with a methyl or long-chain alky groups have a glass transition temperature well below 0°C and therefore are elastomers. Polysilanes are stable in air and moisture and are not attacked by mild reagents such as dilute acids, but solvolysis of the Si–Si bond is rapid in strong bases or solvents such as THF. Polysilane polymers are stable up to 300°C, but however, are degraded on exposure to UV light. They react with strong oxidizing agents such as *meta*-chloroperbenzoic acid, and oxygen atoms are inserted between the silicons. Pure polysilanes are normally insulators, but the addition of oxidizing agents such as $AsF_5$, $SbF_5$, and $H_2SO_4$ can change them to semiconducting materials.[22,24,25] Polysilanes are photoactive and used in applications in which photosensitivity is required.[25]

All polysilanes show strong electronic absorption bands in the ultraviolet region between 300 and 400 nm. The electronic transitions are of $\sigma \rightarrow \sigma^*$ type, result from the extensive delocalization of $\sigma$-electrons in the catenated silicon atoms. The absorption maximum depends on the nature of organic substituents.[24]

Thermochromism is one of the most unusual optical properties of polysilanes, which is dependent on the nature of the side chain. The thermochromic changes result from equilibrium between different conformations. Poly (di-n-hexysilane) is one of the most studied polysilanes that undergoes a dramatic change in UV absorption at 42°C. Above this temperature it absorbs at 317 nm and below it at $\lambda_{\max} = 372$ nm. Thermochromism of polysilanes has been discussed on the basis of the phase transition of the main chain, which is induced by side chain ordering.[23,26]

The unique electronic, optical, photophysical, and chemical properties of polysilanes are from the extensive delocalization of $\sigma$-electrons along the silicon backbone and are extremely sensitive to the nature of organic substituents attached to the polymer backbone. Some of the potential applications of these materials are as follows:

- precursors of silicon carbide ceramics ($\beta$-SiC fibers),[27]
- protective coatings,[28,29]
- photoinitiators in radical reactions,[30,31]
- photoconductors in photography and nonlinear optical materials,[32]
- production of conducting and semiconducting electronic devices,[33,34]
- photoresists in microelectronics,[35] and
- high resolution UV resists in microlithography.[36]

Some of the more important classes of their technical uses and novel applications will be presented in more details in the following sections.

## Novel Applications of Polysilanes

### *Precursors of Silicon Carbide*

Porous SiC preforms have been prepared for the preparation of SiC-glass composites, via pyrolysis of a mixture of polysilanes and polycarbosilanes at 1397°C, and three types of SiC preforms with open porosity between 60 and 70% were obtained. Some samples of SiC preforms were oxidized at 797°C in air to promote wetting by the glass melt. These preforms were then used with an alumosilicate glass melt at 1,397°C in 3 MPa argon for gas pressure infiltration. The SiC-glass composites were evaluated by light microscopy, scanning electron microscopy, and electron scanning microanalysis.[37]

SiC with 20% residual carbon was prepared by pyrolysis of a sample of vinylic polysilane in argon at 1,600°C. A $SiC/Si_3N_4$ composite was also prepared by the addition of excess Si and pyrolysis of the mixture in $N_2$ at 1,395°C. All products showed a fine-grained microstructure and excellent homogeneity.[38]

Pyrolysis of a mixture of polysilanes and polycarbo-silanes under argon at 650°C resulted in the formation of sponge-like polycarbosilanes with an open porosity of 50%. The pyrolyzed preforms were oxidized in air at 600°C and then infiltrated with aqueous $Al_2O_3$ slurry for the preparation of porous SiC-$Al_2O_3$ composites. Finally sintering was carried out at 1,600°C and in 1.5 MPa argon. The SiC-$Al_2O_3$ composites characterized by infrared spectroscopy, scanning electron microscopy microanalysis, and electron scanning microanalysis.[39]

Three different difunctional monomers, dimethyl-dichlorosilane, diphenyl-dichlorosilane, and methyl-phenyl-dichlorosilane, and two trifunctional monomers, phenyltrichlorosilane and ethyltrichlorosilane, were used to prepare a series of polysilanes. The obtained products were characterized via thermogravimetry, Gel Permeation Chromatography (GPC), and thermo-mechanical analysis. These polysilane precursors were investigated for silicon carbide fibers.[40]

Polycarbosilane (PCSi), a carbon-rich SiC precursor, and polyborazin-modified polymethylsilane (M-PMSi), a silicon-rich SiC precursor, were blended at a ratio of (PCSi: M-PMSi) 0.25:1.00 by weight for the preparation of a hybrid polymer as a near-stoichiometric SiC precursor. The hybrid polymer exhibited a very high ceramic yield of 82 wt% and a higher molecular weight than what was produced using a single precursor. The hybrid precursor showed good processibility, and by dry-spining it was converted into green fibers. It was found from XRD, $^{13}C$ MAS-NMR, and $^{29}Si$ MAS-NMR data that the SiC ceramic derived from the hybrid polymer at 1,400°C exhibited no excess Si and little residual carbon. The chemical compositions of 98.08 wt% SiC and 1.92% wt% $SiO_2$ were observed by SEM-EDX analysis.[41]

## Coatings

Polysilanes were used to prepare the Deep-UV (DUV) bottom antireflactive coating. This coating has the real component of refractive index, n, equals 2.00 and the imaginary component, k, equals 0.23 at 248 nm. This new coating is immiscible with a chemically amplified photoresist and is not removable during the normal wet development of photoresist. The etching rate of the polysilane is greater than that of DUV resist and less than that of DUV resist by using $O_2$ gas process. The polysilane layer is easily removed by ashing.[42]

One of the important class of polysilanes is organic-inorganic hybrid materials, which have very intersting properties and diverse applications such as coatings. Organic-inorganic hybrid coatings based on epoxy resin and sol gel materials (Tetraethoxysilane [TEOS]) for the corrosion protection of aluminum alloy substrats have been prepared.[43–45] The formation of an inorganic phase from TEOS by a sol-gel process, organic phase, and hybrid network were characterized by FTIR and $^{29}$Si NMR spectroscopy and the TGA technique. TGA results showed that the cross-linking between the epoxy resin and silica increases the thermal stability of the sample.[46,47] These networks show excellent optical transparency and nanoscale microphase separation. The epoxy silica hybrid films were coated on a 1050 aluminum alloy by dip coating and spray brushing methods at r.t. and were post cured at 130°C for 1 h. The cured hybrid film exhibited high adhesion properties to the underlying metal substrate as well as corrosion protection in a salt spray test.[48] Corrosion resistance properties of hybrid sol-gel coatings were studied by electhrochemical methods. The results indicate the excellent protection performance of the coatings.[49]

## Photoinitiators in Radical Reactions

On exposure to UV light, polysilanes undergo a main-chain scission due to photolysis and involve the homolytic cleavage of Si–Si bonds and formation of the free silyle radicals. The silyle radicals react with olefinic monomers, which initiate free radical polymerization. Poly (Methylphenylsilane), PMPSi, poly (hexylmethylsilane), PhxMSi, or poly(dihexylsilane), PdHxSi, are used for polymerization of methyl methacrylate (MMA) and styrene (St) in Ar-saturated THF solution at $\lambda = 313$ and 346 nm at 30°C. The rate of monomer conversion and quantum yield of initiation ($\phi_i$) were measured ($\phi_i$ is the number of kinetic chains saturated per absorbed photon). The $\phi_i$ values of the polysilanes are two orders of magnitude larger than the reported values for the St/PMPSi system. These results prove that the mentioned polysilanes are efficient photo-initiators for the free-radical polymerization of MMA and St.[50]

Polysilane-acrylamide block copolymers were prepared with various compositions by photo-polymerization. PMPSi, used as a macro-photo-radical initiator in photo-polymerization of monomers of acrylamide-type. The Hydrophilic property of PMPSi was improved by an acrylamide block in the copolymers. The resulting copolymers were used to prepare PMPSi-silica hybride thin films using a sol-gel reaction. Homogeneous and transparent hybrid thin films were prepared from a few PMPSi-acrylamide block copolymers.[51]

Block copolymers with polystyrene were incorporated into polysilanes via metal-catalyzed Atom Transfer Radical Polymerization (ATRP). An ABA triblock copolymer was synthesized using a macroinitiator of PMPSi with styrene by ATRP. These results confirm that copolymers with improved mechanical properties and processing could be prepared with this approach. Future developments in synthetic methods of novel hybrid nanocomposites such as interpenetrating polymer networks, nanoobjects, and biomimetic materials are possible.[52]

## The Production of Conducting and Semiconducting Electronic Devices

A new hybrid polysilane polymer was prepared by a catalytic addition of poly[methyl(H)silane-*co*-methylphenylsilane] to an *N*-(allyl) cycloiminium salt. The chemical structure of the resulting polymer was investigated by spectroscopic analysis, TGA, and GPC techniques. The formation of molecular dipoles inside the macromolecular chain was due to a strong charge-delocalization of the pendant cycloiminium salt segments. This new polymer can be use develop organic semiconductors.[53]

$SiO_2$ thin films were prepared from polysilanes membranes when treated with oxygen plasma below 60°C. The ratio of silicon to oxygen in these films is about 1/1.99. This new type of $SiO_2$ thin film may find a promising application in microelectronic technology.[54] Polysilanes doped with phthalocyanines were prepared, and the behavior of the quantum yield versus the polarization field was evaluated. The result shows that the doped polymer is suitable for photoconductive applications.[55]

UV irradiation of Poly (Ferrocenylmethylphenylsilane) (PFMPSi) films that are casted from chloroform solution leads to the photooxidation of these materials. The photooxidized PFMPSi conductivity is three orders of magnitude greater than unoxidized material. The resulting film is also photoconductive. The photooxidized film can be chemically reduced by hydrazine ($N_2H_4$) or decamethylferrocene ($FeCp_2^*$) to regenerate the neutral polymer. These ferrocene-containing conducting polymers can be used in photovoltaic applications.[56]

## The Fabrication of Light-Emitting Diodes and Electroluminescence Devices

Polysilanes are suitable for the fabrication of Light-Emitting Diodes (LED)s due to the specific chemical, optical, and electronic properties. The LEDs were fabricated by using three types of dialky, monoalkylaryl, and diaryl polysilanes as an emissive material. These LEDs made from polysilanes show Electroluminescence (EL) in the Near-Ultraviolet (NUV) or ultraviolet region, which is related to their

$\sigma$-conjugation. The single-layer LEDs can be made using emissive polysilanes that emit NUV light at 407 nm (3.05eV) with a quantum efficiency of 0.1% of photons/electron at r.t.[57] The spectroscopic, electronic, and structural properties of di-arly polysilane and poly[bis(p-n-buthylphenyl)silane] were examined, and improvements were observed in the EL characteristics compared with LEDs based on conventional polysilanes.[58]

The Si-related materials named oxygen-cross-linked polysilanes were prepared by thermal annealing of the precursor polysilane-containing alkoxy group. Two photoluminescence peaks containing a broad visible band at 440 nm and a sharp band at 360 nm were observed at r.t. The relative intensity of visible emission depends strongly on the steric hindrance of the alkoxy groups of the precursor. The visible EL was also observed from the Al/oxygen-cross-linked polysilan/ ITO cell. The current densities of the EL cell increased with an increase of the oxygen-cross-linking.[59]

## As Coupling Agents

Silane coupling agents play an important role in improving the degree of adhesion at an interface in a composite system.[60,61] Silanated a polymer coupling agents were prepared by attaching alkoxysilane groups to a polysulfone. These results show that a dramatic improvement in strain at the failure, strength, and elastic modulus occurred when the interface was treated with the silane and then reacted with the sulfonated polysulfone. This new material was used as an interfacial agent in glass particle reinforced polymer composites.[62]

## The Preparation of Nanoporous Thin Films

Hybrid thin filmes of polysilane-titania were prepared using titanium alkoxides with poly (methyl-phenylsilane)/3-methacyloxypropyltriethoxysilane block copolymer (P(MPSi-*co*-MPTESi)) and titanium alkoxides using a sol-gel reaction. The titania component bonded to a polysilane block copolymer through Si—O—Ti derived from the hydrolysis//condensation of P(MPSi-*co*-MPTESi) and $Ti(O\text{-}n\text{-}C_4H_9)_4$. It was shown by a tapping-mode atomic force microscope that the polysilane segments disperse in a titania componet in a range of several 10 nm. The resulting films can be used to prepare nanoporous titania thin films.[63]

Mesoporous, Nanostructured inorganic-organic hybrid xerogels were prepared in presence of surfactants via a sol-gel process. The n-alkyl substituted trialkoxysilanes were co-condensed with tetraethoxysilane as a cross-linker and n-hexadecyclamine as template. The properties of these novel hybrid xerogels are suitable for application in HPLC as stationary phases. The produced inorganic polymers exhibit a separation performance similar to that of the analogous surface of modified silica. However, the particle size is smaller than the conventional stationary phases that have been used in HPLC.[64]

### The Formation of Patterns

Laser-induced hydrogen desorption from polysilanes was used for silicon pattern formation. Polysilane thin films containing $(SiH_2)_n$ chains terminated with $SiH_3$ have been prepared using a silane plasma at 110°C and evaluated as a UV-sensitive material. The desorption of bonded hydrogen from film occurs at r.t. through excimer laser irradiation. The crystallization and Si network formation occur in the irradiated area. Because of easy oxidation of a nonirradiated part of polysilane at air, the exposure of the film to the laser lead to the selective oxidation of nonirradiated area. A fine polycrystalline Si pattern has been generated via diluted hydrofluoric acid (HF) etching of the oxidized regions.[65]

The deposition of gold, silver, and palladium colloids on polysilane films is a novel method that was carried out via reducing the corresponding metal salt dissolved in alcohol without the use of any other reductants. This methodology used to metal pattern formation such as nickel and copper by palladium-catalyzed electroless metallization on the surface of poly (methylphenylsilane) film and UV-light lithography at 254 nm without the use of any other photoresiste.[66]

### The Fabrication of Microlens Arrays

Microlens arrays are used in many optical applications and digital optical imaging systems. The arrays of refractive microlens are applied to condense light and illuminate and process images. Recently a convex microlens array was fabricated using polysilanes as photoresist materials via the adhesion of silicasol on a polysilane film patterned by UV-light irradiation. This method is extremely simple and low-cost for the fabrication of microlens arrays.[67] Another type of the refractive microlenes array was developed via the electrodeposition of a polymer emulsion on transparent indium-tin-oxide covered with a polysilane film patterned by UV-light irradiation.[68]

## SUMMARY

Inorganic polymers are high molecular weight polymers with inorganic elements in their backbone. In general, inorganic polymers offer unique property profiles not available with conventional organic polymers. Polysilane is an inorganic polymer with a linear backbone of silicon atoms and two organic side groups attached to each silicon atom, which

shows unique properties resulting from the easy delocalization of sigma electrons in the Si—Si bonds. Polysilanes are commercially important inorganic high polymers, which have found application in almost every major industry.

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