

Polymer Optical Fiber with High Thermal Stability and Low Optical Losses Based on Novel Densely Crosslinked Polycarbosiloxanes

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ABSTRACT: A multimode step-index polymer optical fiber has been developed based on a highly crosslinked polycarbosiloxane as the core material and a fluorinated polyolefin (poly(fluoroethylene-co-fluoropropylene)—FEP) cladding layer. The combination offered a system with a high thermal stability, both optically and dimensionally, up to at least 200°C. The polymer optical fiber was produced by injection of a poly(phenylmethylvinylhydro)siloxane crosslinkable prepolymer in an FEP tubing, after which the prepolymer was cured *in situ*. From attenuation measurements losses as low as 0.98 dB/m at 780 nm were found. The refractive indices of the core material and the cladding are 1.48 and 1.30, respectively. The large difference in refractive index results in a high value for the numerical aperture of 0.70. The prepolymer is a single-component, self-crosslinking, and conveniently processable system. The combination of a low attenuation and easy processability makes these materials very suitable not only for polymer optical fibers, but for all kinds of optical components. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 67: 2223–2230, 1998

Key words: polymer optical fiber; high dimensional stability; high thermal stability; densely crosslinked polymer

INTRODUCTION

The application of polymer optical fibres (POFs) is a strongly growing market. There is a great demand for low-loss polymer optical fibers for use in, for example, communication systems, replacement of electronic systems, or optical sensors. One demand, which the conventional POFs based on thermoplastic polymers cannot fulfill, however, is a high thermal and dimensional stability. The thermoplastic polymers are limited in their use to maximum temperatures close to their softening

points (T_g s), i.e., $\sim 80^\circ\text{C}$ for polymethylmethacrylate (PMMA) and polystyrene (PS). Polymers of higher T_g are applied such as polycarbonate and polyimids. However, for the latter polymers it is difficult to obtain low optical losses due to relatively high intrinsic molecular absorptions. Moreover, the problem of softening and the consequential loss of properties, at high temperatures, is still relevant here.

An interesting option is the use of crosslinked materials. By crosslinking, the dimensional stability is not limited by a softening point and properties are retained above the T_g . This effect is strongly enhanced for highly crosslinked polymers. The maximum temperature at which the POF can operate is then determined by the thermal stability of the polymer system. As polysiloxanes have a very thermally stable backbone,

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densely crosslinked networks made thereof are superior candidates, which can fulfill all the requirements. An additional positive property of densely crosslinked polymers is a low compressibility of such systems. A low compressibility results in only small or even negligible fluctuations in density and, therefore, in small or negligible contributions to scattering. Of course, the material needs to be homogeneously and isotropically crosslinked to avoid scattering by density fluctuations.

This article describes the application of a new type of densely crosslinked polysiloxanes for polymer optical fibers with high thermal stability. Besides low intrinsic optical losses and a thermally stable backbone, these materials are crosslinked without production of volatile products and without shrinkage, which allows their use for bulk applications such as polymer optical fibers and planar waveguides. In this article the focus is on application in polymer optical fibers.

EXPERIMENTAL

Analysis

Glass transition temperatures were measured with a MDSC (Modulated-DSC) 2920 apparatus from TA Instruments. For these measurements a scan speed of 3°C/min, an amplitude of 1°C and a period of 60 s were used. The glass transition temperature was defined as the midpoint of the DSC curve. The thermal stability was measured with a Perkin-Elmer TGA-7 apparatus operating at a scan speed of 10°C/min in a nitrogen atmosphere. For the measurement of the polycarbosiloxane polymer networks a ceramic pan was used. The conventionally used platinum cups are easily solubilized by the polymer due to a strong complexation of platinum with residual silicon–vinyl groups of the polymer.

The mechanical properties were determined on cylindrically shaped specimens (diameter: 0.76 mm) with an Instron 4301 tensile tester at room temperature (20°C). The crosshead speed was 10 mm/min and the gauge length 25 mm.

Measurement of the bulk density was done in absolute ethanol by measuring the exact volume of the specimen (~ 1 g). The values of the density of ethanol and their dependence on temperature were taken from literature.¹

Atomic Force Microscopy (AFM) on the inner surface of the FEP tubing was performed with a

Topometrix Discoverer AFM. Profile measurements were done with a Sloan DEKTAK 3030ST Surface Texture Analysis System with a needle force of 10 mg.

Contact angle measurements were conducted with a home-built setup. The angle that a droplet of ultrapure water made with the polished surface of the material was determined with a camera.

Refractive indices of the prepolymers were determined with an Abbé-refractometer working with the D-emission line of sodium, being 589.3 nm at 20°C. The refractive index of the cured polymers was measured on a film with a Metricon 2010 prism coupler in two directions (n_o and n_e) at 20°C and at wavelengths of 633, 803, and 1300 nm.

The attenuation of light of the produced POFs was measured via the cutback method for wavelengths from 600 to 1000 nm. The light source was a halogen-tungsten lamp coupled to a grating monochromator. The exit intensity was measured with a silicon-photodiode.

Materials

Vinylmethyldiethoxysilane and methyldiethoxysilane were synthesized from vinylmethyldichlorosilane (purchased from Acros Chimica) and methyldichlorosilane (purchased from ABCR), respectively. The procedure employed has been described in an earlier article.² Phenyltriethoxysilane and dimethyldiethoxysilane were obtained from Acros Chimica. All four monomers were carefully distilled twice via spinning band distillation (plate number = 20). The catalyst was a platinum 1,3-divinyltetramethyldisiloxane complex with 3–3.5 wt % of platinum in vinyl-terminated polydimethylsiloxane. The catalyst system, purchased from ABCR, was dissolved in 1,3-divinyltetramethyldisiloxane (ABCR) to give a 0.1 ± 0.008 wt/wt % platinum concentration. Absolute ethanol (pro analysis) was obtained from Acros Chimica and was used as received. Toluene was distilled freshly from sodium and benzophenone, prior to use. Florisil™ magnesium silicate, 30–60 mesh and pH = 8.5 was obtained from Fluka AG.

FEP tubing was purchased from Polyfluor Holland with an inner diameter of 0.95 mm and an outer diameter of 1.6 mm.

Synthesis of the Poly(phenylmethylvinylhydro)siloxane Prepolymer

The basic synthesis of the particular prepolymers has been described in an earlier article.² A similar

procedure has been employed, but with some adjustments, as reported below.

A round-bottomed flask was charged with 8.0 g (0.033 mol, 25 mol %) of phenyltriethoxysilane, 6.9 g (0.047 mol, 35 mol %) of dimethyldiethoxysilane, 4.3 g (0.027 mol, 20 mol %) of vinylmethyldiethoxysilane, 3.6 g (0.027 mol, 20 mol %) of methyldiethoxysilane and 100 wt %, based on the total weight of the monomers, of absolute ethanol. To the cooled homogeneous solution 34.1 g (150 wt %, based on the total weight of the monomers) of a 20% aqueous hydrochloric acid solution were added dropwise at such a rate that the temperature of the reaction mixture did not exceed 10°C. After the addition was complete, the still homogeneous reaction mixture was stirred for 1 h at room temperature. The mixture was combined with 50 mL of toluene and stirred for 3 h at 60–70°C. Hereafter, the toluene layer was separated and washed with an aqueous saturated sodium bicarbonate solution followed by washing with demineralized water until the pH was neutral. The remaining water left from the washing procedure was azeotroped off at 130–140°C for 2–3 h. The reaction mixture was filtered over a Florisil™ column and toluene was removed *in vacuo* by means of a rotary evaporator. The product was combined with 50 mL of methanol and stirred for 1 h, after which the mixture was filtered through a 0.2 μm Teflon membrane filter. The methanol was removed *in vacuo* by means of a rotary evaporator. Finally, the solvent-free polymer was filtered through a 0.2 μm Teflon membrane filter. The polymer was kept under reduced pressure at room temperature for at least 48 h to remove traces of toluene and possibly, low molecular weight volatile oligomeric components. The yield was approximately 85% of a very clear and transparent poly(phenylmethylvinylhydro)siloxane with a found average composition (¹H-NMR) of [PhSiO_{3/2}]_{0.24}[Me₂SiO]_{0.38}[ViMeSiO]_{0.21}[HMeSiO]_{0.18}.

Fabrication of Polymer Optical Fibers

The FEP tubing was thoroughly cleaned before use by ultrasonic rinsing with 20% HNO₃ for 30 min. Thereafter, the tubing was flushed several times with demineralized water, filtered (0.2 μm) ethanol and finally with filtered (0.2 μm) pentane. After each flushing step, the tube was dried with pure nitrogen gas. The tube was dried at 80°C *in vacuo* for at least 24 h.

The polymer optical fibres were fabricated by injecting a poly(phenylmethylvinylhydro)silox-

ane prepolymer, with an average composition of [PhSiO_{3/2}]_{0.24}[Me₂SiO]_{0.38}[ViMeSiO]_{0.21}[HMeSiO]_{0.18}, into an FEP tube. The prepolymer was filtered over a 0.2 μm Teflon membrane filter and deaerated ultrasonically, prior to injection. The curing of the prepolymer was accomplished *in situ* with 1 ppm of platinum catalyst (Karstedt) at 150°C for 24 h, producing a step-index polymer optical fiber.

RESULTS AND DISCUSSION

Synthesis of the Prepolymers and the Polymer Networks

The synthesized prepolymers are based on four different types of monomers, viz. phenyltriethoxysilane, dimethyldiethoxysilane, vinylmethyldiethoxysilane, and methyldiethoxysilane. For the nomenclature of the polymer the same monomer sequence has been used resulting in poly(phenylmethylvinylhydro)siloxane. Before synthesis, the monomers were distilled twice via a spinning band distillation with a plate number of 20, to obtain a maximum purity.

The prepolymers were produced via cohydrolysis and condensation reactions (Fig. 1). The trifunctional monomer, phenyltriethoxysilane (PTES), was used to introduce branching of the polymer backbone. The branching points will turn into network junctions after the curing reaction has been accomplished. Because they are present in a large amount before the curing step, the polymer system will show only a minute or even a negligible degree of shrinkage. This is a very important issue because the prepolymer is injected into a tubing and for a good contact with the wall of the tubing shrinkage of the core as a consequence of the curing reaction may not occur. If the contact is not adequate, this undoubtedly forms a source of scattering. Generally, shrinkage is a large problem when applying polymer networks, however.

The prepolymers are highly soluble in common organic solvents and gelation during the synthesis was prevented by cyclization reactions. These cyclization reactions are well known for siloxane polymerization. It was found that the ring structures have a molecular structure similar to that of the nonring structures and are able to crosslink as well.² The incorporation of ring-like structures is favorable for the thermal stability of the cured polymer.³

A final step during workup is fractionation of

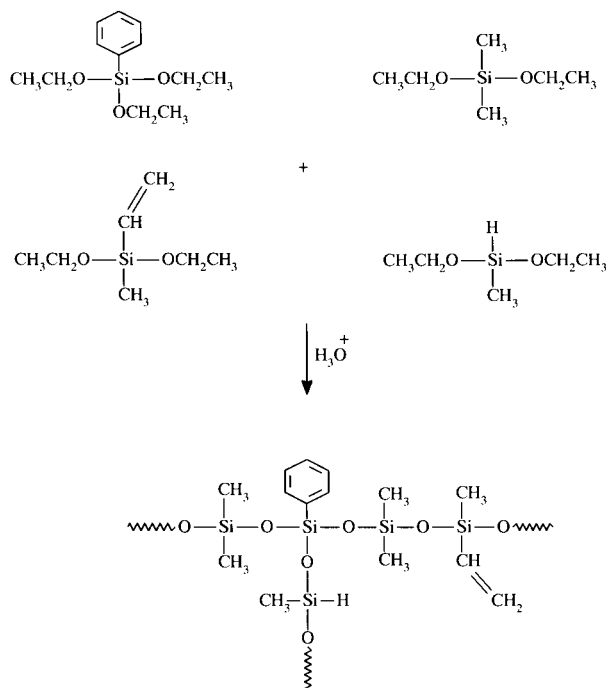


Figure 1 The reaction scheme for the synthesis of the prepolymer.

the prepolymer. In this step methanol is used to separate the major low molecular weight part from the minor high molecular weight part. This provides a very clear prepolymer. Namely, the higher molecular weight part tends to phase separate from the lower molecular weight part, which can result in a slightly turbid prepolymer. The number-average molecular weight of the prepolymer under investigation (poly(phenylmethylvinylhydro)siloxane, 25/35/20/20 mol %), is typically 1200 g/mol, resulting in a moderate viscosity.²

For the synthesis, a solvent like THF or ethanol is used to homogenize the reaction mixture. If such a solvent is not used, and the hydrochloric acid is added to the pure monomer system, then after a short while a phase separation occurs. It is obvious that the higher molecular weight molecules become less soluble in the monomer/water mixture and will phase separate. These molecules become less available for the catalyst system and cannot grow easily and homogeneously to higher molecular weights. If, on the other hand, a solvent is used that is able to solubilize both the monomer/water/hydrochloric acid phase and the polymer phase formed, polymerization can take place more homogeneously in the complete system. This results in a smaller distribution of the molecular

weight, and above all, in a very clear prepolymer. Nonetheless, a fractionation step is applied, but the yield of highly transparent polymer is high.

The polysiloxane prepolymer is a single-component system and has a moderate viscosity, which results in a conveniently processable system. Moreover, this makes a thorough purification of the solvent-free prepolymer (filtering and deaeration), prior to the processing, feasible. The prepolymer possesses both functional groups needed for the curing reaction. Crosslinking occurs intermolecularly via hydrosilylation reactions, which is a clean crosslinking reaction without the production of volatiles, if proper conditions are chosen (Fig. 2). No problems with volatile crosslinking agents or inhomogeneous mixing of nonvolatile crosslinkers arise. If volatilization of crosslinking agents would occur during processing, the crosslinking could become insufficient. The second problem may occur if a high molecular weight polymeric crosslinker is applied. An inhomogeneous mixing can give rise to density fluctuations on a nanometer or micrometer scale with a consequential scattering of light.

The functional groups were not present in exactly stoichiometric amounts. During workup, a small portion of the relatively sensitive silicon-hydro groups is defunctionalized through a reaction with acid, ethanol or methanol. The best-known side reaction is an alcoholysis of the silicon-hydro group to a silicon-hydroxyl group. Such a silicon-hydroxyl group can react with methanol during the fractionation step and forms a silicon-methoxy group or it takes part in the cocondensation reactions and produces a branching point. The slight distortion of the stoichiometry does not inhibit the curing reaction to a significant extent, however. By measuring the conversion of the silicon-vinyl groups with infrared spectroscopy it was found that up to 50% of the vinyl groups react.² It was also found that the number of reacted silicon-hydro groups was somewhat higher, up to 65%. For the curing reaction enough silicon-hydro groups are present. The higher conversion of these groups is likely due to reactions with silanol groups or silicon-methoxy and residual silicon-ethoxy groups. Reactions with atmospheric moisture are not likely, because the reacting system is protected from the environment through the FEP sheathing (cladding).

Curing took place under the influence of 1 ppm platinum catalyst for 24 h at 150°C. The relatively slow curing process can be accelerated by adding more catalyst. This introduces a dramatic increase of the optical losses, however.⁴

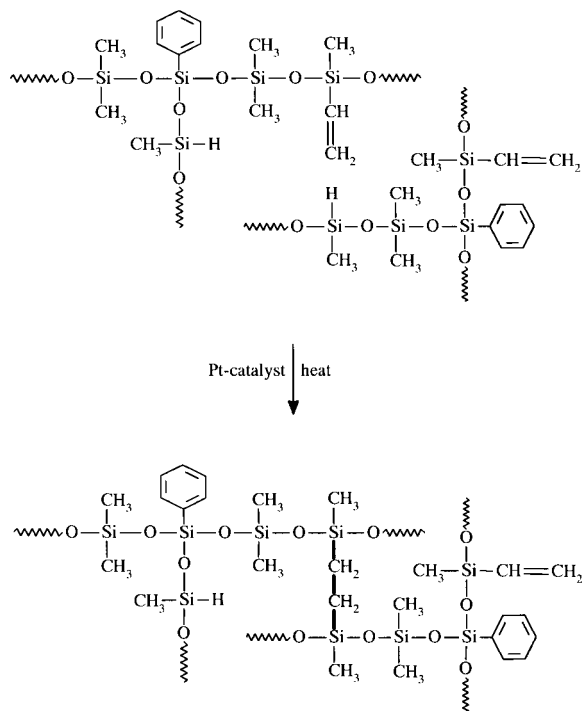


Figure 2 The formation a polymer network by intermolecular hydrosilylation crosslinking reactions.

Properties of the Core Material and the Polymer Optical Fiber Manufactured Thereof

The glass transition temperature of the densely crosslinked system was determined to be 14°C. This is not a high value when it is compared to the more conventional polymers, but due to the dense network structure this system is dimensionally stable above its T_g and the optical properties are retained. That the system is very densely crosslinked is reflected in the value of the molecular weight between crosslinks, M_c , which was found to be 300 g/mol.⁵

The mechanical properties were measured at room temperature (20°C) and showed a stress at break of 16 MPa, an elasticity modulus of 350 MPa, and an elongation at break of 22%. The polycarbosiloxane core is surrounded by a very tough FEP sheathing (tubing). The mechanical properties of the tube were 120 MPa in tensile strength and an elasticity modulus of 1.7 GPa. The strain at break was 500%. The combination of these mechanical properties results in a very tough material (a toughness of 440 MPa). In combination, therefore, the core and the cladding materials produce a strong and tough polymer optical fiber. To do so, a good contact of the core with the cladding is important. In the case of a good contact, the

very tough thermoplastic lining can dissipate energy from the core material, which results in a mechanically stable fiber. Strong contact can be based on two contributions, a chemical bond type interaction (covalent or physical, i.e., Van der Waals interactions), and a mechanical interaction by anchorage at a rough surface. In synergism they add to the total adhesive interaction. The inner surface of the FEP tubing is very smooth, as has been demonstrated by atomic force microscopy (AFM) and profilometry. From an AFM topography image (Fig. 3) an average roughness of 11.2 nm was calculated. The maximum change in height was 200 nm, which was also found in the profile measurements. Because of such a smoothness, adhesion is predominantly based on a chemical interaction. A prerequisite for a good contact is, above all, a good wetting of the inner surface of the tubing by the prepolymer. The fluorinated tubing material has a low surface tension (contact angle with water is 107°).⁶ For a good wetting the contact angles of both substrate (cladding) and polymer (core) need to be in the same range. Polysiloxanes are known to have low surface tensions. From contact angle measurements an angle of 103° was found for the crosslinked material, which is very close to the value of FEP.

Generally, crosslinking leads to a shrinkage of the polymer. In the case of highly crosslinked polymers this especially can be a severe problem. In a confined volume like a tube, this means a decrease of the diameter of the core and thereby the contact with the cladding is lost. The curing of the poly(phenylmethylvinylhydro)siloxane prepolymer was found not to be accompanied by any measurable extent of shrinkage. This resulted in a nicely fabricated optical fiber without any signs of release of the core from the cladding. A picture of the cross section of the optical fiber in (Fig. 4) indicates a good contact of the core and cladding. A good contact is not only important for good mechanical stability, but for low optical losses as well. The roughness of the cladding layer is significantly smaller than the wavelength of the propagating light and does not contribute strongly to the total loss mechanism.

The refractive index of the prepolymer was found to be 1.464 (at 589.3 nm), which is rather high for polysiloxane-based systems. The presence of the phenyl groups, which possess a high degree of polarizability, are responsible for this high value. Upon curing, a refractive index of 1.483 at 633 nm is reached. Due to the crosslink-

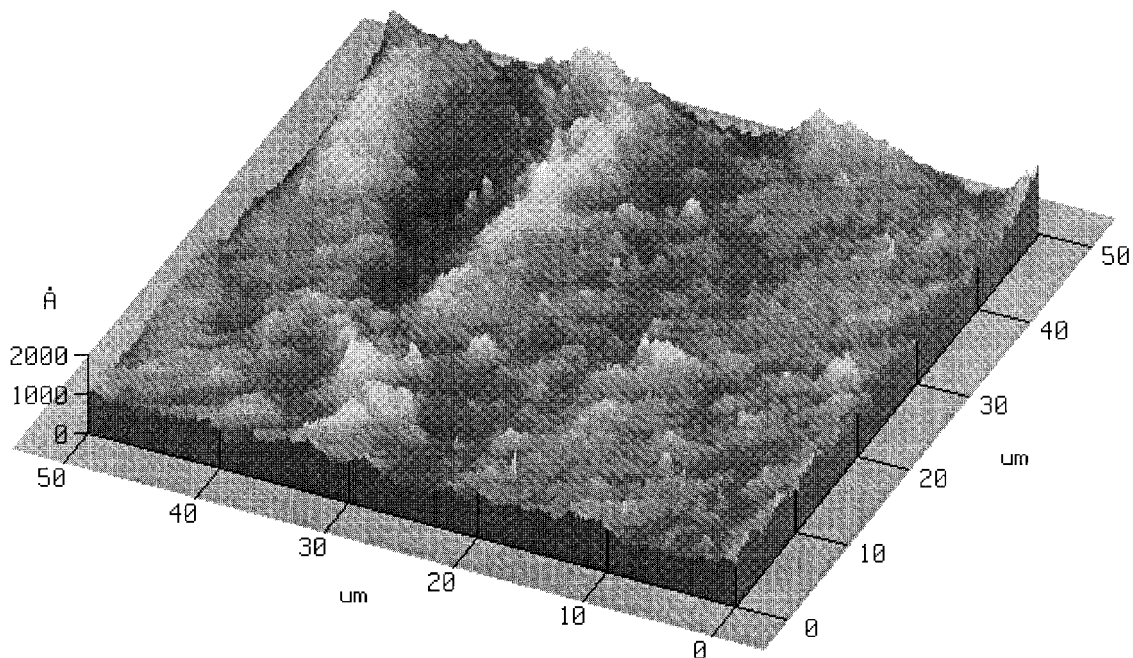


Figure 3 An AFM topography image of the inner surface of the FEP tube used.

ing a densification of the polymer system takes place accompanied with a rise in refractive index. As has been shown above, the densification did not give rise to a measurable extent of shrinkage in diameter of the core, however. In Table I, the

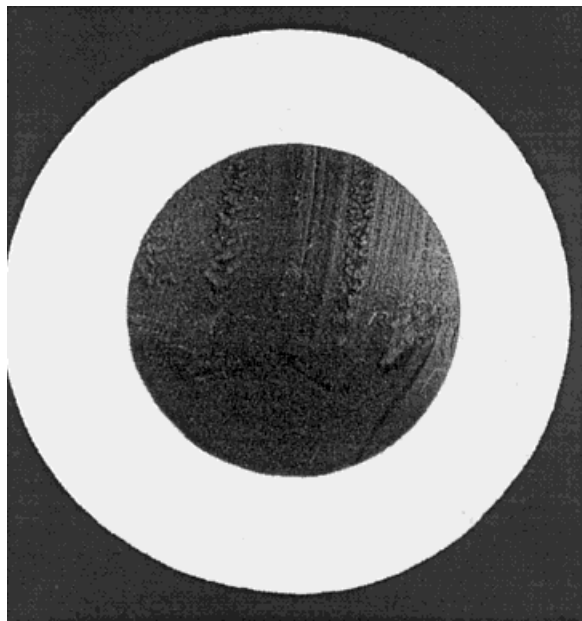


Figure 4 A picture of the cross section of the polymer optical fiber. The surface has not been polished. The actual size is 0.95 mm for the core and 1.6 mm for the outer diameter of the fiber.

refractive indices are listed for three different wavelengths. The refractive index was measured on a planar film in two directions, parallel to the plane (n_o) and perpendicular to the plane (n_e). The table shows that in both directions exactly the same refractive indices are found. This indicates that the material is entirely isotropic in terms of light refraction and thus does not show any signs of birefringence. From the table one can also see the wavelength dependence of the refractive index. For higher wavelengths a lower refraction of light is caused by the material, because of a smaller interaction between the light and the material. For the optical fiber similar conclusions

Table I Refractive Indices at Three Different Wavelengths and in Two Directions (parallel, n_o , and perpendicular, n_e , to the plane) for the Cured Poly(phenylmethylvinylhydro)siloxane with 25 mol % Phenyltriethoxysilane

Wavelength (nm)	n_o^{20}	n_e^{20}
Prepolymer (589.3)	1.464	
633	1.483	1.483
830	1.475	1.475
1300	1.472	1.472

The refractive index of the prepolymer is given for 589.3 nm, measured parallel to the plane. The measurements were performed at 20°C.

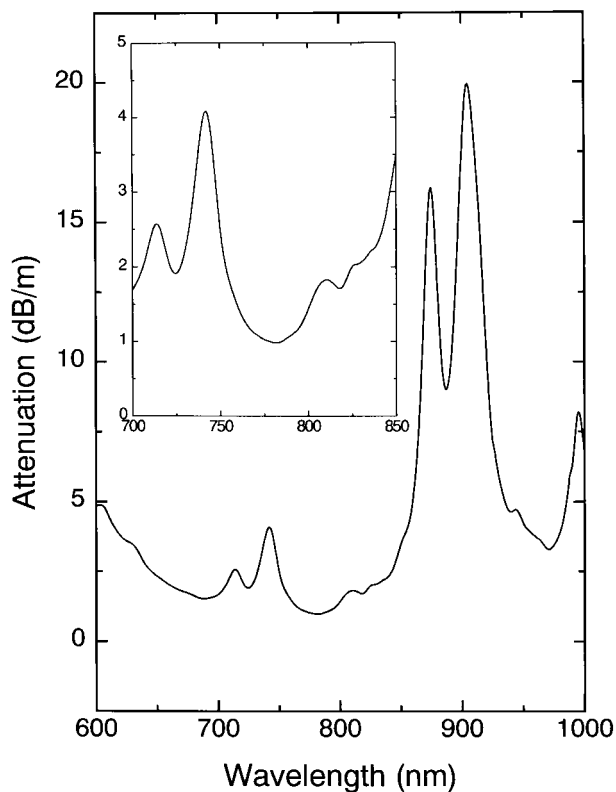


Figure 5 An attenuation spectrum of the polycarbosiloxane-based polymer optical fiber. The optical loss has been determined from 600 to 1000 nm. The lowest loss has been registered at 780 nm being 0.98 dB/m.

can be drawn. Upon injection of the prepolymer into the tubing an orientation of the polymer in the direction of the fiber axis might occur. However, because curing is a relatively slow process here, there is enough time for the polymer system to relax to an unperturbed, nonbirefringent state, before gelation constrains the conformation. This is an advantage over the high- T_g thermoplastic polymers, for which often an orientation of polymer chains in the direction of the fiber axis, i.e., extrusion direction, leads to birefringence. Scattering of light because of birefringence is an important extrinsic loss factor of such polymer systems.

Figure 5 shows a typical attenuation spectrum of an optical fiber based on poly(phenylmethylvinylhydro)siloxane polymer with 25 mol % of PTES. The lowest attenuation is experienced in a window at 780 nm and amounts to 0.98 dB/m. The spectrum shows the higher harmonics of the fundamental C—H stretching vibrations and bending vibrations, the combination of which results in doublet absorption peaks in the visible

part of the spectrum. The number of C—H bonds present can be calculated with eq. (1).

$$N = \rho n_{\text{C-H}} / M_{ru} \quad (1)$$

where N is the number of C—H bonds in mol/cm³, ρ is the density of the bulk in g/cm³, $n_{\text{C-H}}$ is the number of C—H bonds per repeat unit, and M_{ru} is the molecular weight of a single repeat unit.

For the polycarbosiloxane under investigation, the density was found to be 1.145 g/cm³, which results in an intrinsic number of C—H bonds of 0.068. The value for PMMA is 0.095, which is significantly higher. PMMA is extensively used as optical fiber core material, and is known as the (protonated) material with the lowest optical losses in the visible. In the near infrared (1000–1500 nm) PMMA has very high intrinsic losses due to the C—H overtone absorptions, however. The polycarbosiloxanes, on the contrary, have a dramatically lower number of C—H bonds in the backbone structure and are, therefore, very promising materials for use in the near infrared region. The losses in the near infrared have not been measured so far, but losses far below 10 dB/m at 1000 nm indicate good optical transparency in this part of the spectrum. With this advantage, these materials may become very suitable for planar waveguiding (short optical path lengths) of near infrared light. As the conventional optical glass fibers, which are widely used for telecommunications, operate at 1300 and 1550 nm, low-loss and high-NA planar waveguides based on polymers would offer prospects of a more wide-spread, easy, and low-costs application of optical fibers for local area networks.

Toward lower wavelengths, one observes an increase of the absorption by UV band edge excitations, predominantly caused by the presence of phenyl groups. The residual vinyl groups may take part in the absorption as well.

The numerical aperture (NA) of the step-index polycarbosiloxane-based optical fiber can be calculated using eq. (2).

$$\text{NA} = (n_1^2 - n_2^2)^{1/2} \quad (2)$$

where n_1 is the refractive index of the core, and n_2 is the refractive index of the cladding.

The refractive index of the cladding is 1.30, and that of the core is 1.475 to 1.483, depending on the wavelength. This results in a high value of the NA of 0.70–0.71 and corresponds with an angle of

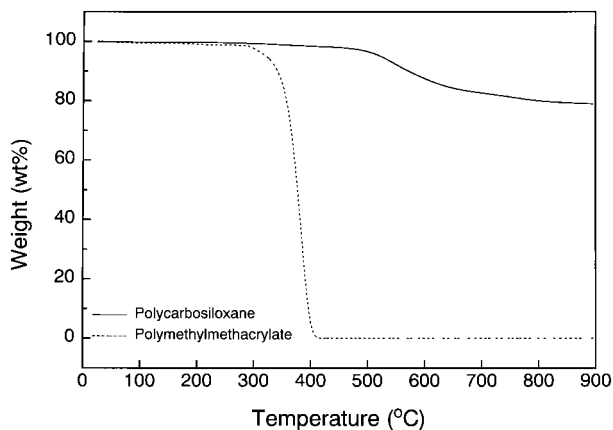


Figure 6 A thermal gravimetric analysis (TGA) plot showing the high thermal stability of the crosslinked polycarbosiloxane with 25 mol % phenyltriethoxysilane (PTES) incorporated. To compare this thermal stability with that of PMMA, conventionally used for polymer optical fibers, a TGA curve of PMMA is shown as well.

acceptance of 90°. The high NA, together with a large core diameter, enable easy coupling of light into the optical fibers. This results in easy handling and a low-cost application of such polymer optical fibers.

The polycarbosiloxane networks are highly thermally stable. Figure 6 shows a thermal gravimetric analysis plot of the polycarbosiloxane network based on 25 mol % PTES. It is known for polysiloxanes in general, that they have a high thermal stability. Crosslinking improves the thermal stability even further. At 200°C a slight lowering of the mass was observed (~ 1 wt %), which proved to be due to lower molecular weight, uncrosslinked material. As the core is protected by the cladding layer, release of the polymer is not possible. So far we have not experienced any influence of this low molecular weight part on the thermal stability of the optical properties.

The cladding material is fully fluorinated, and fluorinated polymers are known to be very thermally stable, as well. Therefore, the POF under investigation offers an outstanding thermal stability. The POF can be used at relatively severe thermal conditions, up to 200°C, without losing its optical properties.

CONCLUSION

We have presented a new thermoset multimode step-index polymer optical fiber with a high ther-

mal stability. The thermoset was produced from a poly(phenylmethylvinylhydro)siloxane prepolymer based on 25 mol % phenyltriethoxysilane (PTES). The prepolymer with moderate viscosity was injected into a FEP tubing [FEP: poly(fluoroethylene-co-fluoropropylene)] and thereafter curing was accomplished at 150°C for 24 h under the influence of 1 ppm platinum catalyst (Karsstedt). The combination of a relatively high refractive index core material (1.483) with a low refractive index cladding (1.30) offers a good light guiding polymer optical fiber with a high numerical aperture. Optical losses as low as 0.98 dB/m at 780 nm were registered. As the material is densely crosslinked the dimensions are stabilized over a broad temperature range, at least up to 200°C. Besides polymer optical fibers, the polycarbosiloxanes can be easily applied for planar waveguides. As these polymers possess a low intrinsic number of C—H bonds they are also promising for light guiding applications in the near infrared region.

The poly(phenylmethylvinylhydro)siloxane is a single-component, self-crosslinking system that offers easy processing, and the crosslinking reaction takes place without production of volatiles.

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