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Recent Optical Applications of Perfluorocyclobutyl Aryl Ether Polymers

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Summary: Perfluorocyclobutyl (PFCB) aryl ether polymers have various applications in the field of optics due to their synthetic versatility and availability, solution and melt processability, high temperature and photo stability, excellent optical transparency. Nanoparticle matrix materials, encapsulants, and chemical sensors are just a few examples of recent developments.

Keywords: cyclopolymerization; electro-optics; fluoropolymers; high performance polymers; nanocomposites; optics; perfluorocyclobutyl (PFCB) aryl ether polymer; polymer light emitting diode (PLED)

Introduction

The outstanding thermal and mechanical properties of perfluorocyclobutyl (PFCB) aryl ether polymers combined with a highly amorphous character and excellent solution processability are the reasons for their success in a multitude of commercial technologies.[1–6] The synthesis of aryl trifluorovinyl ether (TFVE) monomers can be tailored for a wide variety of thermoplastic and thermoset materials employing a versatile intermediate strategy using commercial aryl TFVE monomers. These applications include photonics, polymer light emitting diodes (PLEDs), proton exchange membranes (PEMs) for fuel cells, atomic oxygen (AO) resistant coatings, and hybrid composites. The combination of thermal stability, solution processabillity, and excellent optical transparency is important for applications in the field of optics. This article highlights the most recent contributions to optical applications of PFCB aryl ether polymers.

Polymer Synthesis and Properties

Perfluorocyclobutyl (PFCB) aryl ether polymers are prepared via thermal [2+2] cyclodimerization of aryl TFVE monomers (Scheme 1). The thermally initiated, step-growth polymerization is carried out either as a melt or in high boiling solvents without initiator and is condensate-free. Polymerization gives primarily head-to-head, stereo-random PFCB aryl ether polymers as an isomeric mixture. As a result, most PFCB aryl ether polymers are highly amorphous materials with highly desirable optical clarity and solution processability.

Aryl TFVE monomers are synthesized in two steps starting from phenols. Alkylation with 1,2-dibromotetrafluoroethane[7] followed by dehalogenation with zinc in acetonitrile delivers the desired aryl TFVE functionality (Scheme 2). Different mono-, bis-, and tris-functional aryl TFVE monomers are available by using appropriate phenolic starting materials for the synthesis. Different degrees of fluorination can be achieved and provides added utility in order to modularly tailor the optical properties of the resulting polymer.

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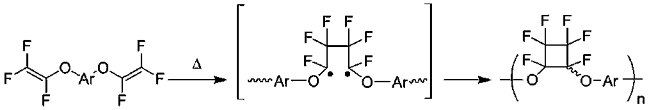


Scheme 1.
Thermal [2+2] cyclodimerization of aryl TFVE monomers to PFCB aryl ether polymers.

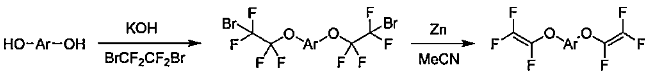
A variety of functional groups can be introduced starting from *p*-bromo(trifluorovinyloxy) benzene; examples include carboxylic acid, acid chloride, aldehyde, alcohol, and amino groups. These different functionalities open possibilities for further reactions such as coupling or condensation steps. Boronic acid and stannyl aryl TFVE monomers are often employed for Pd-catalyzed coupling reactions such as Suzuki, Stille and Sonogashira. The bromo(trifluorovinyloxy) benzene manifold gives the possibility aryl TFVE monomer system tailored for specific polymer applications.[5,8,9] Tetramer Technologies, L.L.C. has commercialized many of the aryl TFVE monomers and they are distributed by Oakwood Chemicals.[10]

These semi-fluorinated amorphous polymers are solution and melt processable and produce optically transparent materials that are compatible for optical applications. Furthermore, PFCB aryl ether polymers possess a low dielectric constant, low surface energy, thermal oxidative stability, photo stability and high temperature resistance. These properties are summarized in Table 1. Decomposition occurs for most polymers between 450–500 °C and glass transition temperatures range from 150–300 °C. Low optical loss at telecommunication wavelengths makes them particularly suitable for the application in waveguides. Because of their tunable optical and thermal properties PFCB aryl ether polymers possess properties utilized in various technologies in the field of optics.

Optical Waveguides

Polymeric optical devices have begun to replace traditional inorganic materials in communication fields as optical tele/datacom networks. Fluoropolymers exhibit superior properties compared to non-fluorinated polymers due to their extended strength and chemical, temperature, and oxidation resistance, and most important because of their low optical attenuation at telecom wavelengths in the near infrared (NIR) region. The optical behavior of different PFCB aryl ether homopolymers was analyzed for the wavelengths between 200–3200 nm.[11] PFCB aryl ether polymers show excellent optical transparency in addition to their superior properties such as excellent processability, low dielectric constant, low moisture absorption, and mechanical stability.[12–14] The solution and melt processability allows the application of PFCB aryl ether polymers for coatings, molds, and stamps. Due to their structure–property tailorability, these polymers can be successfully used for low loss waveguides[15] as thermoplastics or thermosets[16] and other high performance photonic devices that can be fabricated by various lithographic methods.[17,18]

Recently, low attenuation waveguides based on the propagation of long-range surface plasmon polaritons (LRSPPs) were reported, which showed a new low in propagation loss of < 2.0 dB/cm for a 4 μm wide waveguide.[19] The high quality of the multilayer LRSPP waveguide structures with low intrinsic absorption and



Scheme 2.
Preparation of aryl TFVE monomers from functionalized bisphenols.

Table 1.
Selected physical properties of PFCB aryl ether polymers.[2]

Property	Range
Mechanical	
Glass transition (DSC or DMA)	110–350 °C
Thermal decomposition (TGA in N_2 & air)	$>$ 450 °C
Tensile strength	50.3–66.0 MPa
Tensile modulus	1770–2270 MPa
Flexural strength	74.0–92.4 MPa
Flexural modulus	1779–2320 MPa
Percent elongation (break)	4.1–12.5%
Interfacial shear	123–163 MPa
Hardness	175–653 MPa
Processing	
Cure temperature/time (°C)	150–220/0.1–3.0 h
Molecular weight (GPC)	1,200–30,000 M_w
Solution viscosity (RMS)	0.02–100 Pa.s
Crystallinity (WAXD)	0–35%
Solid content	50–90%
Patterning technique	μ-molding, RIE, O_2 plasma
Spin coat thickness (μm, single)	1–30
Percent water absorption (wt %) (24 h)	0.021
Optical	
Loss (1550 nm)	$<$ 0.25 dB/cm
Birefringence	$<$ 0.003
Refractive index (1550 nm)	1.442–1.505
dn/dt (1550 nm)	-0.7 to -1.5 (x10^{-4})
Insulation	
Dielectric constant (10 kHz)	2.4–2.45
Dissipation factor (10 kHz)	0.0003–0.0004

highly homogeneous refractive index profiles could be demonstrated.

Thin gold stripes were embedded in a low absorption PFCB aryl ether polymer which was copolymerized via thermally induced cyclopolymerization of bifunctional and trifunctional aryl TFVEs. The resulting copolymerization formed the symmetric dielectric surrounding material in the LRSPP waveguide structure. The excellent processability and optical properties of PFCB materials used in simple LRSPP structures should lead to low-cost photonic devices for a range of applications.

New compact arrayed waveguide grating (AWG) demultiplexer for wavelength-division multiplexing (WDM) have been designed and fabricated with a PFCB core on a poly(imide) substrate.[20] Using air-trench bends the new design shrank the required chip area by a factor of 20 and completely avoids curved waveguide bends in order to realize the smallest possible AWG device. The 8×8 AWG was imbedded in a PFCB aryl ether polymer waveguide material system[21–23] with a 1.3% refractive index contrast on polyimide substrates.

Due to the combination of low loss in the NIR and its excellent processability, PFCB aryl ether polymer substantially contributes to the success of reducing the AWG size through use of air-trench bends. The ability to etch smooth, highly vertical interfaces that are precisely positioned with respect to each waveguide bend intersection was essential to realize this new device (Figure 1).

Nanoparticle Dispersion Matrix

Recently, PFCB aryl ether polymers were also found useful for nanocomposites and as encapsulants. Nanocrystals and clusters such as quantum dots (QDs) feature unique optical and spectroscopic properties like broad absorption, narrow and tunable emission, photo stability, efficient and long lifetime

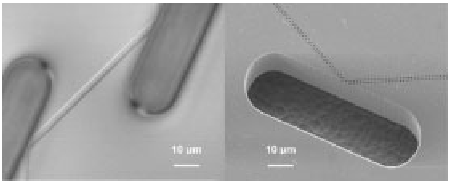


Figure 1.
Etch results for air trenches in PFCB aryl ether polymer on a polyimide substrate using a step by step etch process. Microscope image taken with a DIC filter through a 350 objective focused at the waveguide plane of single air interface bends (SAIBs) in good alignment (left). Scanning electron micrograph of finished SAIB (right). The rounded edges were introduced to reduce stress. The dotted line shows the waveguide core location. (From S. Kim, et al. Proc SPIE 2005, 5729, 264–276).

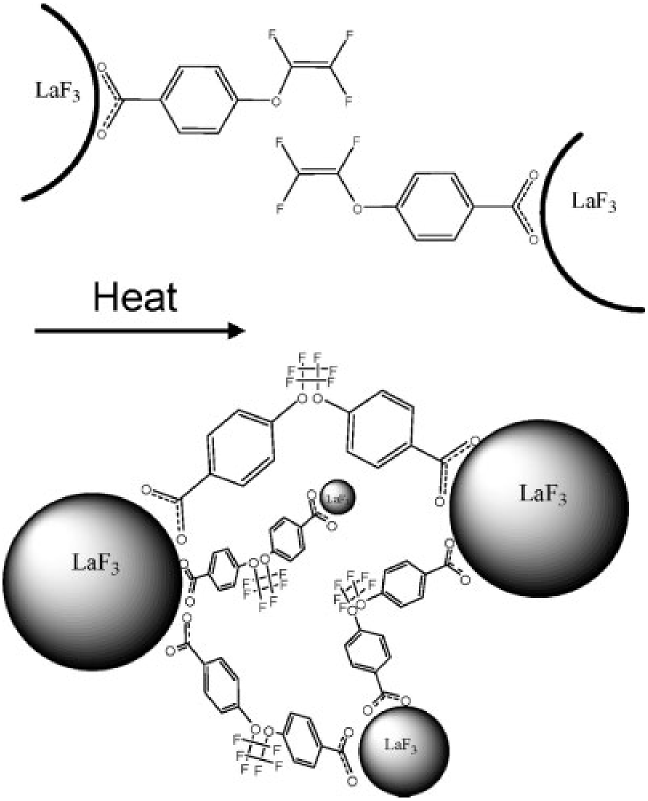
luminescence.[24] QDs are chemically synthesized semiconducting nanometer-sized crystalline particles with various photonic and imaging applications.[25,26] These particles can easily be dispersed in many solvents. For practical applications in photonic systems, it is more useful to incorporate the nanocrystals into a matrix or encapsulate them to increase the stability (Scheme 3). PFCB aryl ether polymer nanocomposites with imbedded infrared-emitting nanoparticles such as PbSe, InAs or CdSe/ZnS were used as planar optical amplifier.[27–29] The dispersion of the QDs into the PFCB aryl ether matrix was achieved by solvent blending. Loading levels up to 20 wt % are possible, when the chemical compatibility of the QD ligands is adapted to the chemical structure of the polymer matrix.[30]

Recently, a new strategy for forming bulk nanostructured materials was published, which allows the fabrication of optical materials with loading levels up to 80 wt % of nanoparticles.[31] These omni-composites take advantage of the unique optical properties of nanoparticles and ultimately rare earth doped nanocomposites.[32,33] Lanthanide emissions can be narrow line and possess near unity quantum efficiency. Problems such as aggregation at high loading levels cause undesirable scattering of light. In general, the dispersion of nanometer-sized materials is difficult with traditional organic polymer host materials. An additional benefit for optical applications of nanocomposites is the ability to match refractive indices thereby minimizing scattering.[34]

For traditional organic ligands that can create dispersible nanoparticles, loading levels are usually limited as well as potentially compromising overall bulk mechanical properties of the host material. DiMaio et al.[31] successfully developed a new method of forming bulk nanostructured materials using crosslinkable ligands to form optical materials with very high loading levels of nanoparticles and decreased scattering. Every LaF_3 nanoparticle is shrouded by aryl TFVE ligands. Thermal polymerization produced a crosslinked, thermally and chemically stable material (Scheme 3).[35] The degree of crosslinking was controlled through the addition of bi-functional monomer. Due to the employment of deprotonated TFVE acid precursors as crosslinkable ligands there is no need for a traditional host matrix. The PFCB aryl ether polymer matrix formed a processable nanocomposite with excellent dispersion (Figure 2).

Rosenthal et al.[36] used PFCB aryl ether polymers as encapsulants for white-light emitting CdSe nanocrystals for solid-state lighting (SSL). The term refers to a type of lighting that utilizes light-emitting diodes (LEDs), organic light-emitting diodes (OLED), or polymer light-emitting diodes (PLED) as light sources instead of traditional electrical filaments, plasma, or gas. SSL creates visible light with reduced heat generation and energy consumption, similar to that of fluorescent lighting.

Additionally, its solid-state nature provides advantages like resistance to shock, vibration, and wear, which significantly increases lifespan. Different polymers for white-light emitting ultra-small CdSe nanocrystals were tested as potential encapsulants. Extensive aggregation of the nanocrystals was observed even at low loading levels (<0.5% w/w) due to the solubility difference between polymer structure and the nanocrystals ligands. The most robust, color stable, and homogeneous encapsulation was obtained using a biphenyl PFCB



Scheme 3.
Thermally initiated cyclodimerization of aryl TFVE ligands forms a bulk crosslinked material.

polymer, which allowed loading level as high as 20 wt%. The encapsulated nanocrystals were coated on various UV-LEDs creating a white light source with chromaticity coordinates of (0.324, 0.322) and high color-rendering index of 93, which successfully meets two of the three U. S. Department of Energy (DOE) goals for general illumination.

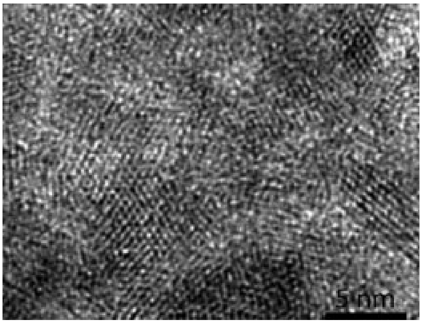


Figure 2.
High-resolution TEM image of the omni-composite shows the density of particles.

Polymer Light Emitting Diodes

PFCB aryl ether polymers offer various advantages as emissive materials for PLEDs. High thermal stability, high glass transition temperature (T_g), superior optical clarity, and a broad tailorability are an excellent base for the development of efficient devices with long emission lifetime.[37] PFCB aryl ether polymers with polyaromatic core shells show a unique photoluminescence.[38] Due to various possibilities of introducing chromophore segments such as fluorene, thiophene, and *p*-phenylvinylene into light-emissive PFCB aryl ether polymers, the photoluminescence can be controlled.[39] Photoluminescence studies showed that the fluorinated ether linkage did not affect the chromophore emission wavelength or solution quantum yields.[40] These mixed chromophore PFCB aryl ether copolymers are easily prepared in one step from commercial starting materials.

The facile, metal-free condensation polymerization of bisphenols with bis(trifluorovinyloxy) biphenyl gives a new class of fluorinated arylene vinylene ether (FAVE) polymers (Scheme 4).

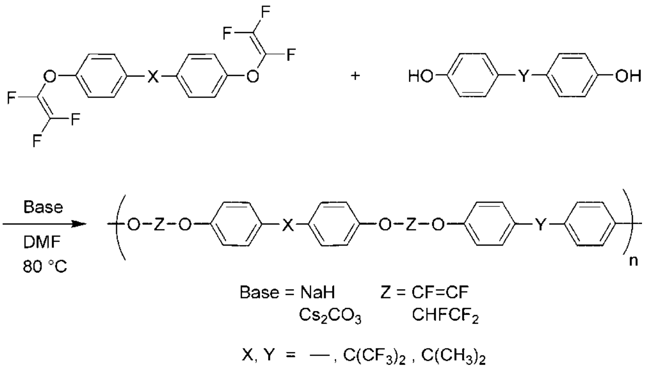
Due to excellent solubility in organic solvents, FAVE polymers can be solution or spin-casted producing transparent, flexible films. Furthermore, by controlling monomer feedstock using excess bis(trifluorovinyloxy)biphenyl, FAVE telechelomers with thermally labile aryl TFVE groups were synthesized with up to 95% 1,2-difluoroethylene (–CF=CF–) enchainment. Studies showed that the 1,2-difluoroethylene moieties are capable of undergoing latent thermal crosslinking at > 250 °C after initiating linear PFCB aryl ether chain extension of the telechelomers.

Based on the substitution of the bisphenol segment, the properties of the aryl ether polymer were tailored for specific applications such as curing additives,[41] proton exchange membranes (PEMs) for fuel cells,[42] light emitting polymers for PLEDs,[43] and chemical sensors.[44] These copolymers showed similar thermal stability and processability as PFCB aryl ether polymers obtained by classical thermal [2+2] cyclodimerization of aryl TFVEs. Recently, chromophores (e.g., fluorene, thiophene, and *p*-phenylvinylene, bipyridyl) were encapsulated in semiconjugated FAVE polymers (Scheme 5).[45] A vast pool of bisphenols possessing chromophore segments are either commercially available or easy to prepare.

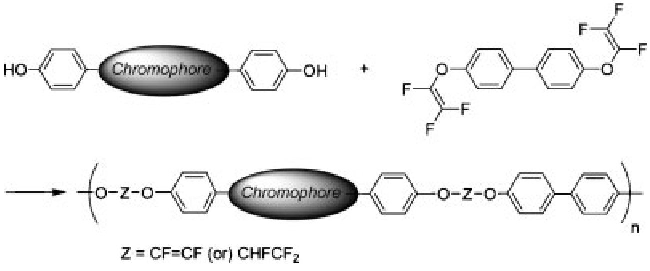
The PFCB aryl ether polymers improve thermal stability and overcome solubility limitations in solvent casting techniques for device fabrication. Studies showed that these polymers are useful for optical sensing.[44,46] Fluoride and cyanide ions induce a highly selective emission enhancement for a metallated bipyridyl-modified FAVE polymer. These materials show a strong ionochromic response to metal ions, including highly selective turn-on fluorescence to Zn^{2+} ions.

The polymers had entirely amorphous character and produced solution processable optically transparent, flexible films, which demonstrated tailorable photoluminescence (PL) that were capable of hole or electron transport.[47–54]

Intermediates such as 4-bromo(trifluorovinyloxy)benzene or 4-trifluorovinyloxyphenyl-boronic acid pinacol ester undergo Pd catalyzed Suzuki coupling reactions and are useful for the synthesis of new aryl TFVE chromophore monomers.[40] Polymerization of these monomers gives chromophore containing PFCB aryl ether polymers with high molecular weights



Scheme 4.
Step-growth polymerization of bisphenols and bis(trifluorovinyloxy)biphenyl.



Scheme 5.
Step-growth polymerizarion of chromophore containing bisphenols and aryl TFVE.

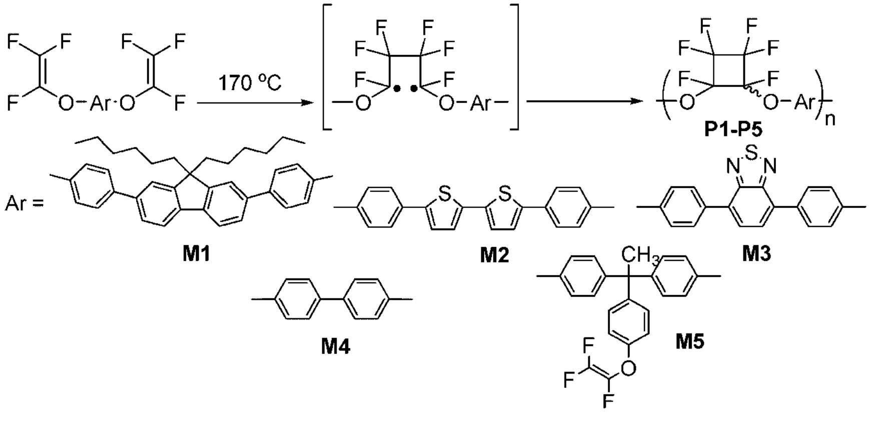
($M_n = 16700–22000$) and high temperature thermal stability ($T_d > 450\,°C$).

Starting from 4-(trifluorovinyloxy)benzaldehyde the Wittig reaction was shown to be an efficient way to synthesize novel phenylene vinylene-bistrifluorovinyl PFCB aryl ether polymers (Scheme 6).[55] Two different strategies are available for the synthesis of these PFCB aryl ether polymers. One way starts with the Wittig reaction to form a difunctional aryl TFVE monomer, which can then undergo cyclodimerization in a second step. However, this route gave an insoluble material, possessing no photoluminescence. The alternative route, starting with the cyclodimerization of a monofunctional TFVE monomer followed by Wittig reaction of two functionalities successfully delivered readily soluble and highly fluorescent materials with quantum yields of 70% in solution.

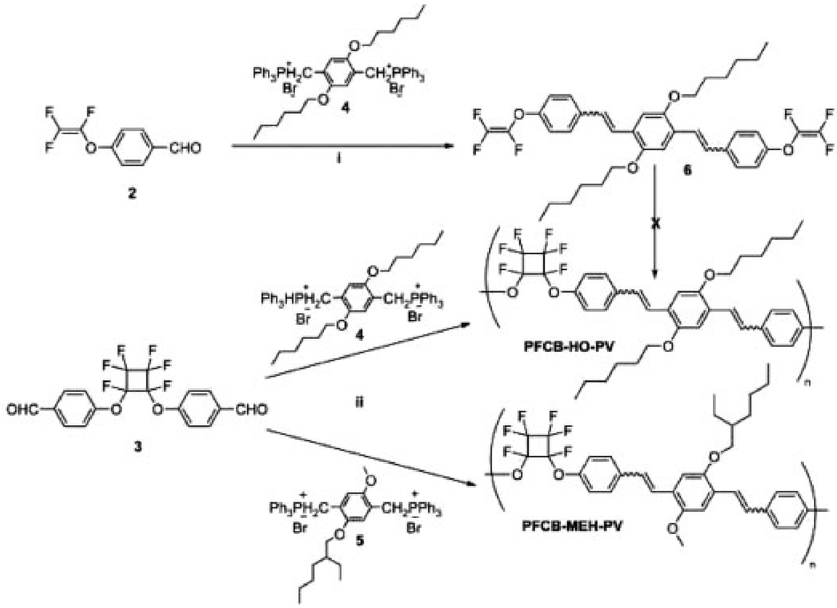
Electro-Optic Polymers

Recently, the synthesis of push-pull PFCB aryl ether chromophores for electro-optic (EO) applications containing a bis(4-methoxyphenyl) amine donor, thiophene acceptor bridges and ring-locked polyene was reported.[56] These materials offer excellent properties for the use in EO devices due to their large optical nonlinearities, and good absorption characteristics besides the high thermal stability, excellent solubility in organic solvents and compatibility with various polymeric materials possessing high hyperpolarizability values.

Jen et al.[57] reported the synthesis of a highly fluorinated and crosslinkable dendritic polymer for photonic applications. The properties of the resulting dendritic PFCB polymer like excellent processability, high thermal stability, high solvent resistance,



Scheme 6.
Polymerization of chromophore aryl TFVE monomers.



Scheme 7.
Synthesis of PFCB aryl ether-*co*-phenylenevinlyenes. (i) Potassium *t*-butoxide in EtOH, THF, 24 h, 70 °C. (ii) Potassium *t*-butoxide in EtOH, THF, 48 h, 70 °C.

low surface roughness, and low optical loss was achieved through thermal polymerization of the trifluorovinyl ether moieties on the periphery of a highly fluorinated dendrimer.

Another novel class of high-performance PFCB containing polymers for second-order nonlinear optics[58] was reported. Described is a novel synthetic approach for incorporating second-order NLO chromophores into PFCB polymer for EO applications. Due to the well-defined nature and the unique combination of properties offered by PFCB polymers, a broad variety of NLO chromophores could be easily incorporated into PFCB polymers without decomposing sensitive chromophores such as the tricyanovinyl acceptor, or inhibiting polymerization.

PFCB aryl ether polymers have also applications as specialty high performance coatings for optical fiber applications.[59] Present materials for optical fiber coatings have limited thermal stabilities and/or are difficult to process in order to achieve the desired film thickness via multiple depositions. PFCB aryl ether polymer-based fiber coatings showed improved properties such as a 150 °C usage temperature of coating without affecting the mechanical properties of the silica-based optical fiber.

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