Perfluorinated polymers for optical waveguides

The goal is a polymer that won't absorb near-IR light. This is achieved by producing fully fluorinated polyimides.

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Along with optical telecommunications systems (see box) (*1–3*), "integrated optics" (*4*) and "optical interconnect" (*5*) technologies are becoming more advanced. The major components of these technologies are photonic integrated circuits (PICs), optoelectronic integrated circuits (OEICs), and optoelectronic multichip modules (OE–MCMs). All the functional devices within these components, including optical and electronic components, are formed or attached on a single planar substrate. Optical signals are transmitted through optical waveguides that connect the components.

To implement these technologies, active optical devices such as light sources, optical switches, and optical detectors are needed. Passive optical components such as beam splitters, beam combiners, star couplers, and optical multiplexers are also required. Various materials, including glasses and polymers, have been considered for the construction of these devices.

Passive optical components have been fabricated using glass optical waveguides prepared by ion-exchange techniques (*6*) or by flame hydrolysis deposition and reactive ion etching (*7*). There are drawbacks to silica-based waveguides, however. First, all the waveguides and passive optical components have to be formed on the substrate before the active optical and electronic devices are attached because of the high processing temperatures needed. For example, temperatures up to 1300 °C are used to consolidate the silica in flame hydrolysis deposition and reactive ion etching. Such temperatures would damage the active optical and electronic devices. Second, glass waveguides are inflexible, making it difficult to process the large waveguides (longer than 30 cm) needed for PICs and OEICs.

The development of optical technologies necessitates the fabrication of waveguides after optical and electronic components have been formed and attached. Thus, materials that are flexible and processable at low temperatures are needed. Polymers are one possibility.

Polymers will soon be used as waveguide materials for interconnecting passive optical components (*8*). For integrated circuits and multichip modules, the manufacturing process includes soldering at 260 °C and brief processing at temperatures up to 400 °C. Therefore, waveguide polymeric materials need to be thermally stable, with a glass transition temperature high enough that it will not be reached during processing. The materials also need to be thermally stable and must have high transparency at the wavelengths of optical communications, 1.3 and 1.55 μm.

Conventional polymeric materials used in plastic optical fibers and waveguides for short-distance optical data links do not have this thermal stability. Ordinary poly(methyl methacrylate), polystyrene, and polycarbonates have low glass transition temperatures. In addition, their optical losses at the preferred wavelengths for optical communications are much higher than their losses at the wavelengths of visible light (0.4–0.8 μm) because C–H bonds harmonically absorb near-IR radiation.

Figure 1 shows the absorption spectrum from the visible to the near-IR for poly(methyl methacrylate) dissolved

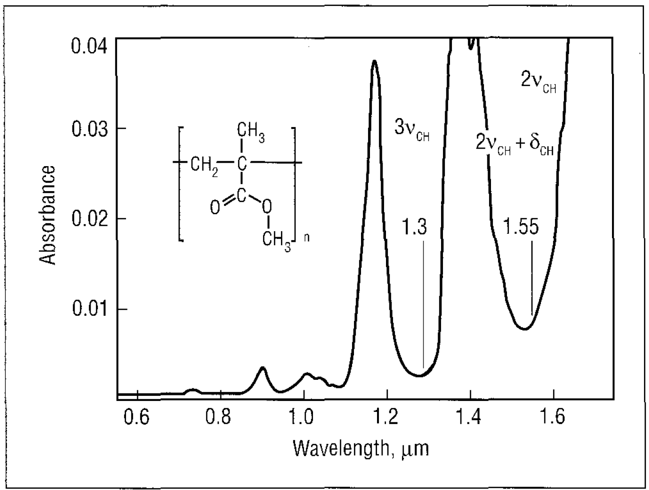

Figure 1. **Absorbing near-IR radiation, the C–H bonds in poly(methyl methacrylate) increase the optical losses at the wavelengths of optical communications.** Although the 1.3- and 1.55-μm wavelengths are in the valleys in this spectrum, the neighboring absorption peaks are strong enough to increase the optical losses.

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Optical telecommunications

Telephone lines are carrying far more information than they once did because of the increase in online computer communications and fax transmissions. Because optical fibers can carry so much more information, they are replacing the copper in telephone wires. Instead of electricity, light in the near-IR part of the spectrum is transmitted through silica-based, single-mode optical fibers.

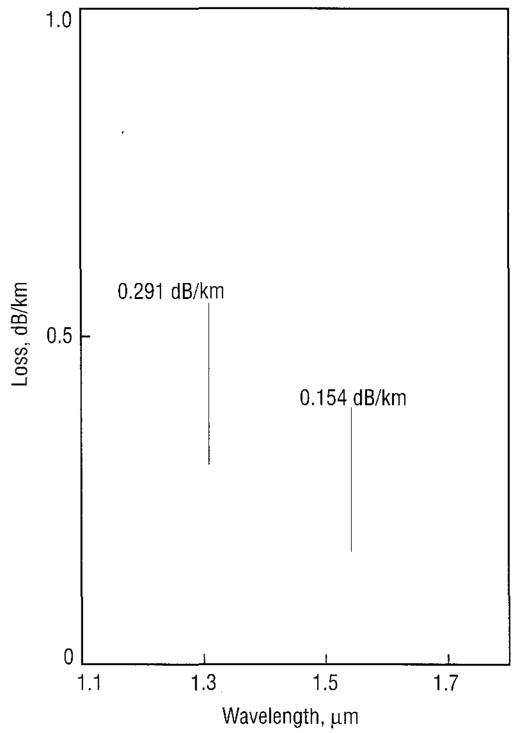
For optical telecommunication systems, a wavelength of 1.3 µm is best for the quality of transmission, allowing the least dispersion. The refractive index dispersion is zero at 1.3 µm, and this wavelength is called the "zero-dispersion" wavelength for these fibers.

However, this optical fiber has its minimum transmission loss at 1.55 µm. To transmit the signal at the highest speed with the least loss, the zero-dispersion wavelength and the minimum-loss wavelength should be identical.

Techniques have been developed to shift the zero-dispersion wavelength of silica-based optical fibers to 1.55 µm *(1)*. To confine the optical signal, the refractive index of the core of the fiber is made slightly higher than that of the cladding. A small amount of GeO_2 is doped into the core of the silica-based fiber. Because this doping does not help to decrease the transmission loss, fluorine is doped into the cladding. The fluorine decreases the refractive index of the medium and inhibits water absorption.

The transmission loss spectrum of such a dispersion-shifted fiber shows that the lowest loss, 0.154 dB/km, occurs at 1.55 µm *(2)*. This minimum loss is close to the theoretical limit determined from Rayleigh scattering and the harmonic absorption of oxygen–hydrogen (O–H) and silicon–oxygen (Si–O) bond stretching *(3)*.

The sharp absorption peak at 1.38 µm is due to the vibration of the residual Si–OH groups by the second harmonic of O–H bond stretching. The optical communication wavelengths of 1.3 and 1.55 µm are located in the valleys between the absorption peaks, indicating the low transmission losses at these wavelengths. As a result of the research developments with silica-based optical fibers, the transmission wavelength will soon be shifted from 1.3 to 1.55 µm in the optical telecommunication systems in Japan.



in chloroform at a concentration of 10% by weight. We negated the influence of the C–H bonds of chloroform by using the same concentration of chloroform as a reference. Two types of C–H bonds, those in the methyl and methylene groups, respectively, give broad, strong absorption peaks in the near-IR region. Although the points in the graph for 1.3 and 1.55 µm are located in the valleys, absorption peaks from C–H bonds increase the optical losses at these wavelengths.

Thermally stable polymers

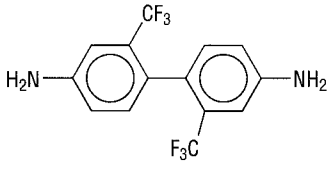
Polyimides and epoxy resins have been considered for use as optical waveguide materials because they have excellent thermal, chemical, and mechanical stabilities. The first attempt to use polyimide for optical waveguides was made by Furuya and co-workers at AT&T (*9*). They fabricated a laser–waveguide integration by using a spin-coated polyimide for the core layer of the waveguide.

Franke and Crow at IBM discussed the influence of the curing process on soluble polyimides. They obtained an optical loss of 0.3 dB/cm at 0.633 µm (*10*). This loss is a measure of the change in intensity of the light signal according to the following expression: $-\mathrm{dB} = 10 \log I/I_o$. In this case, the loss per centimeter is approximately 7% of the incident light. Sullivan at Honeywell described polyimide waveguides fabricated by reactive etching. Measuring 8 µm high by 8–64 µm wide, these waveguides had a 0.3-dB/cm loss at 0.83 µm and excellent, mirror-like, right-angle bends as a result of reflections at the interface with air (*11*). High-density routers, splitters, and combiners are building block components that have been developed in polyimide multimode waveguides (Figure 2).

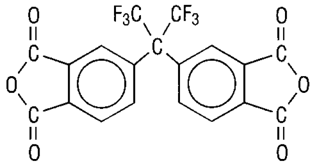
On the other hand, Hagerhorst-Trewhella and colleagues at IBM reported solvent etching, or "wet" etching, processes using UV-curable epoxies and polyimides. They achieved a 0.3-dB/cm loss at 1.3 µm (*12*). Mirrors prepared through wet etching have been demonstrated for out-of-plane, input–output laser interconnections. Finally, Reuter and co-workers at IBM reported that optimally cured, partially fluorinated polyimides can be used to achieve optical losses <0.1 dB/cm at a visible wavelength of 0.63 µm; such losses were stable at temperatures up to 200 °C (*13*).

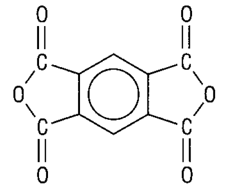
Reducing loss further

We recently demonstrated that single-mode, embedded waveguides can be fabricated with fluorinated polyimides. To fabricate these single-mode waveguides, precise control of the shape and size of the core and of the refractive indices of the core and cladding is needed. The imides we used are copolymers synthesized from TFDB as the diamine



and 6FDA and PMDA as the dianhydrides.





Increasing the PMDA ratio made the refractive index of the core slightly higher than that of the cladding.

In the fabrication process, an undercladding layer and a core layer of the polyimide are formed on a substrate (Figure 3). The undercladding is formed by spin coating of the substrate with poly(amic acid) solution (a prepolymer solution of the anhydride and the diamine) followed by thermal imidization. The difference in refractive index between the core and the undercladding is about 0.4%. The core ridge is then fabricated by photolithographic patterning and reactive ion etching with the use of oxygen. Finally, an overcladding layer is formed.

These fluorinated polyimide waveguides have optical losses of <0.3 dB/cm for light polarized parallel to the substrate at 1.3 µm. The increase in optics loss was <5% in the two experiments after heating at 300 °C for 1 h and at 85 °C with a relative humidity of 85% for 24 h.

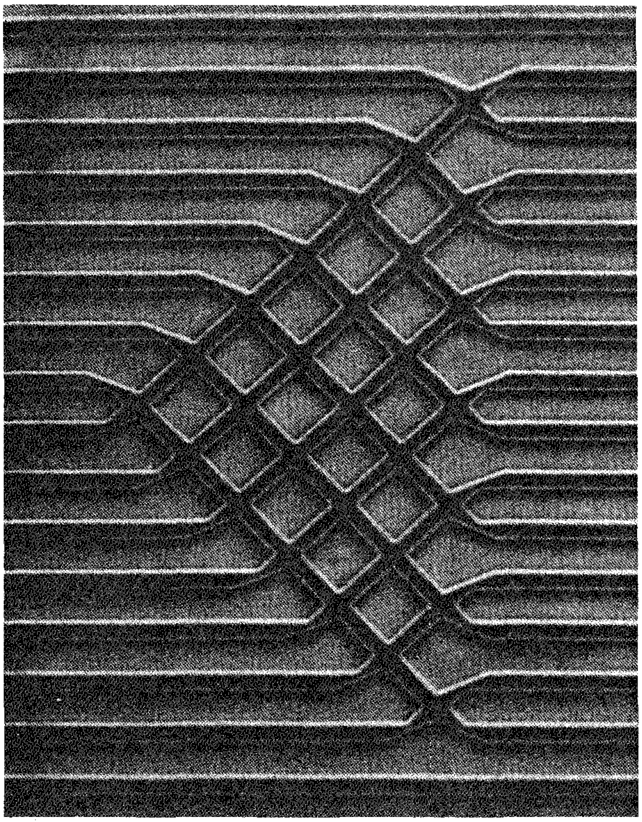


Figure 2. **This polyimide waveguide has 16 channels, 14 of which are interlaced in what is called a "shuffle."** The shuffle pictured here is linearly perfect. The channels are 16 µm wide, separated by spacers that are 25 µm wide. Used wih permission from reference *11*.

These materials also show high transparency at visible wavelengths as well as low dielectric constants, low refractive indices, and low water absorption (*15*). In these properties, the materials are superior to conventional nonfluorinated polyimides and epoxies used for waveguides. Nonfluorinated polyimides have high water absorption of about 2–3% by weight, which causes substantial optical losses at 1.3 and 1.55 µm.

However, fluorinated polyimides also have some absorption peaks in the near-IR region as a result of C–H bonds in their phenyl groups. Figure 4 shows the fundamental stretching bands and their harmonic absorption wavelengths for the carbon–hydrogen (C–H), carbon–deuterium (C–D), and carbon–fluorine (C–F) bonds. The wavelengths were measured for benzene, hexadeuterobenzene, and hexafluorobenzene with a near-IR spectrophotometer. For simplicity, we do not show the absorptions arising from the fourth and fifth harmonics of the stretching vibration and from the combinations of the harmonics with the deformation vibration.

The harmonics of C–D and C–F bonds are displaced to longer wavelengths than are those of the C–H bond (Figure 4). The wavelengths for the fundamental stretching vibrations of the C–D bond are about 1.4 times longer than those of the C–H bond. The corresponding wavelengths for the C–F bonds are about 2.8 times longer than those of the C–H bond.

The absorption band strength decreases about 1 order of magnitude with an increase in the order of harmonics (i.e., the vibrational quantum number). As a result, the losses at visible and near-IR wavelengths can be reduced appreciably by the substitution of deuterium or fluorine for hydrogen atoms (*16*). Kaino, Jinguji, and Nara have produced low-loss optical fibers from deuterated poly(methyl methacrylate)s and fluorodeuterated polystyrenes (*17*). Imamura, Yoshimura, and Izawa at NTT recently fabricated low-loss waveguides of <0.1 dB/cm at 1.3 µm by using deuterated and fluorodeuterated poly(methyl methacrylate)s (*18*). As shown in Figure 5, this substitution greatly reduces the optical loss at 1.3 µm. However, the strength of absorption arising from the harmonics of

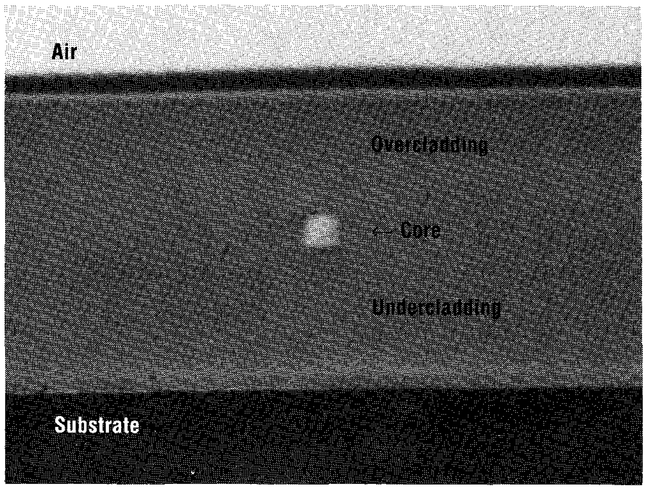


Figure 3. **The embedded waveguide in this cross section was formed from partially fluorinated polyimide.** At the bottom is the substrate. The undercladding layer is the dense dark layer under the clear core.

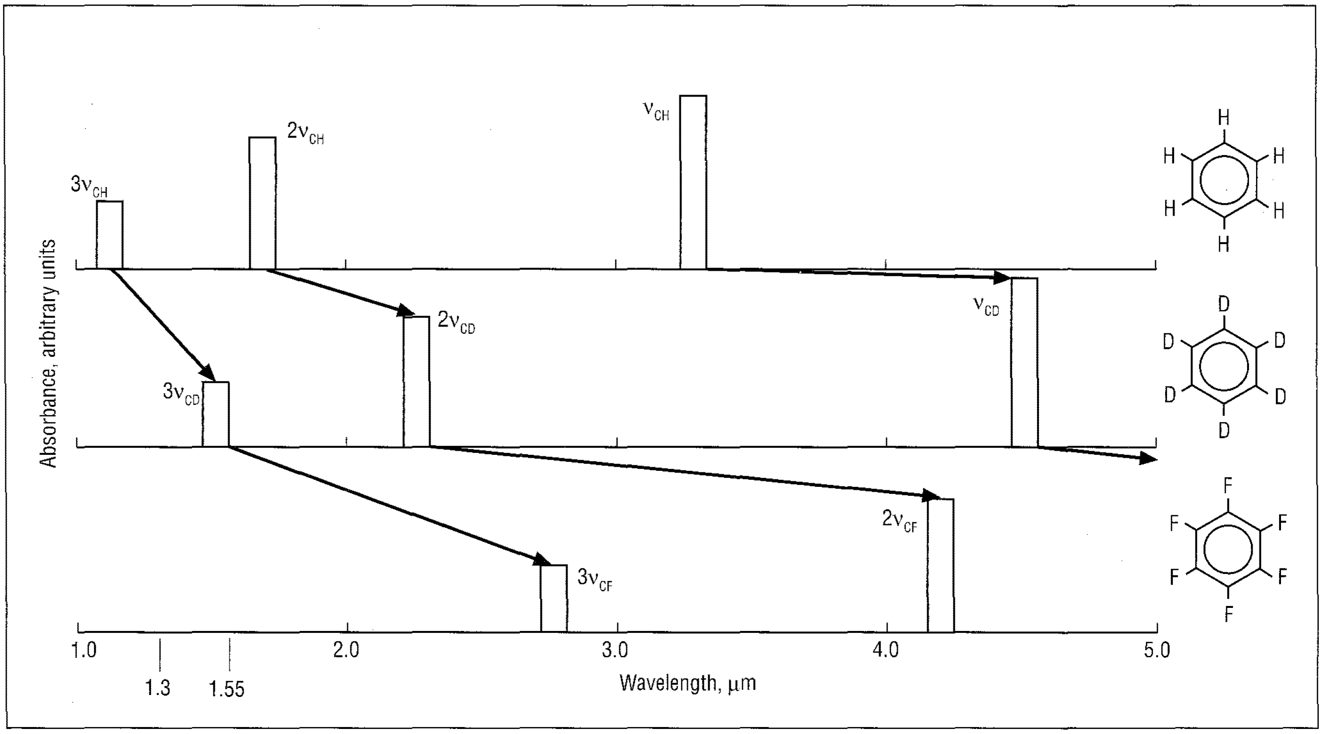


Figure 4. **The harmonics of the C–D and C–F bonds are displaced to longer wavelengths than those of the C–H bonds.** These harmonic absorption wavelengths are for the fundamental stretching bands ν_{CH}, $2\nu_{CH}$, and $3\nu_{CH}$ for the C–H bonds of benzene, the C–D bonds of hexadeuterobenzene, and the C–F bonds of hexafluorobenzene, respectively. Adapted with permission from reference *18*.

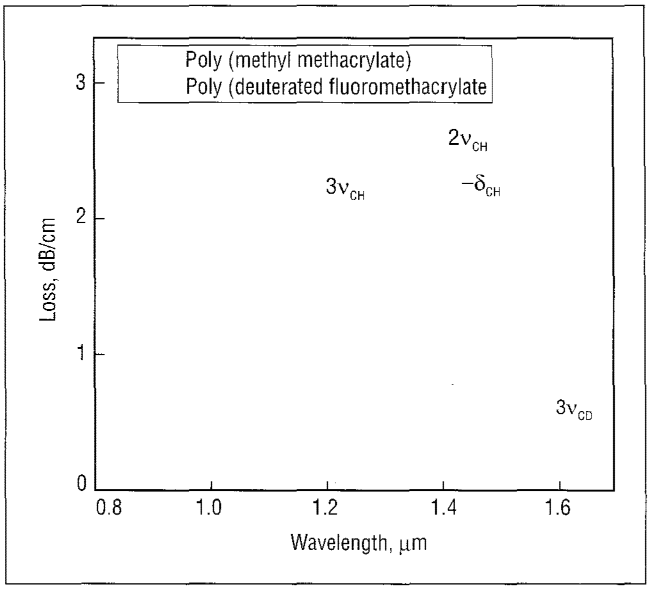


Figure 5. **Poly(deuterated fluoromethacrylate) compares favorably with poly(methyl methacrylate) in its reduction of optical losses at 1.3 µm.** For the wavelength of 1.55 µm, however, strong absorption arises from the harmonics of C–D bond stretching between 1.4 and 1.6 µm *(18)*.

C–D bond stretching that appears between 1.4 and 1.6 µm is not negligible.

The effect of perfluorination

For the reduction of optical loss at near-IR wavelengths, perfluorination is, theoretically, superior to perdeuteration. Table 1 (p. 24) shows the molecular structures and properties of three kinds of perfluoropolymers: Teflon AF (DuPont); Cytop (Asahi Glass); and poly(tetrafluoroethylene) (PTFE), a semicrystalline polymer that cannot be used to transmit light. Cytop has been reported to have no absorption peaks between 1.0 and 2.5 µm (*19*). However, its glass transition temperature is so low that it cannot be used in waveguides for integrated circuits, and its refractive index cannot be controlled.

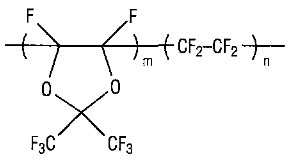
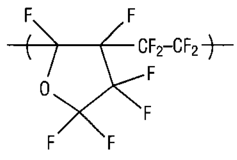
With Teflon AF, however, the glass transition temperature and the refractive index can be controlled by changes in the copolymer ratio. Decreasing the tetrafluoroethylene content can increase the glass transition temperature to >300 °C. In addition, Teflon AF has a low dielectric constant and a high chemical stability. Because these characteristics are similar to those of optical waveguides, which are used as insulating layers of electronic circuits in OEICs and multichip modules, Teflon AF may be used as a new waveguide material.

Synthesis of perfluorinated polyimides

By perfluorinating polyimides (*20*), we expected to achieve not only low optical losses at 1.3 and 1.55 µm but also high thermal, chemical, and mechanical stabilities. Figure 6 (p. 24) presents the structures of one perfluorinated dianhydride and five perfluorinated diamines. These compounds, which are commercially available or at least well known, can be used to synthesize perfluorinated polyimides.

Fluorine is highly electronegative, which means that substituting it for hydrogen decreases the acylation reactivity of diamine monomers and increases the reactivity of dianhydride monomers. To generate high molecular weight perfluorinated polyimides, it is necessary to determine exactly how fluorine affects the reactivities of the monomers, particularly those of the diamines. Kinetic studies of the acylation of conventional monomers have revealed that acylation rate constants can differ by a factor of 10^2 between different dianhydrides and by a factor of 10^5 between different diamines (*21*).

***Table 1.* Molecular structure and properties of amorphous and semicrystalline perfluoropolymers**

			$-(\text{CF}_2\text{-CF}_2)-$
Property	**Teflon AF**	**Cytop**	**PTFE**
Morphology	Amorphous	Amorphous	Semicrystalline
Fluorine content, wt %	67.1, 65.0	67.9	76.0
Glass transition temp., °C	160, 240[a]	108	—
Melting point, °C	—	—	327
Dielectric constant	1.89–1.93	2.1–2.2	2.1
Refractive index	1.29–1.31	1.35	1.38
Optical transmittance, % (visible region)	>95	95	Opaque

[a] The glass transition temperature with no TFE content is higher than 300 °C.

We first determined the reactivities of the diamines shown in Figure 6 by synthesizing poly(amic acid)s using 6FDA as the dianhydride. Note that 6FDA is not perfluorinated. The end-group contents of the poly(amic acid)s that were determined from ^{19}F NMR are as follows:

■ 4FMPD	tetrafluoro-*m*-phenylenediamine	15%
■ 4FPPD	tetrafluoro-*p*-phenylenediamine	42%
■ 8PODA	bis(2,3,5,6-tetrafluoro-4-aminophenyl)ether	75%
■ 8FSDA	bis(2,3,5,6-tetrafluoro-4-aminophenyl)sulfide	91%
■ 8FBZ	4,4′-diaminooctofluorobiphenyl	>99%

Of these, 4FMPD shows the lowest end-group content, or the highest reactivity, and 4FPPD shows the next lowest end-group content.

However, for all the diamines, the acylations were not complete, and end-group contents were still high even after 6 days of reaction at room temperature. When 8FBZ diamine, the least reactive one, was reacted with 6FDA, no NMR signal for the poly(amic acid) was detected. The electron-donating properties of the diamines, which determine the reactivity of diamines for acylation, matched the end-group contents of the poly(amic acid)s (*20*). We estimated the electron-donating properties from ^{15}N NMR chemical shifts and from electron-related parameters such as ionization potential, electronic affinity, and molecular orbital energy.

However, Hougham and co-workers at IBM have prepared continuous films using 6FDA as the dianhydride and 4FPPD and 8FBZ as diamines (*22*). They showed that two

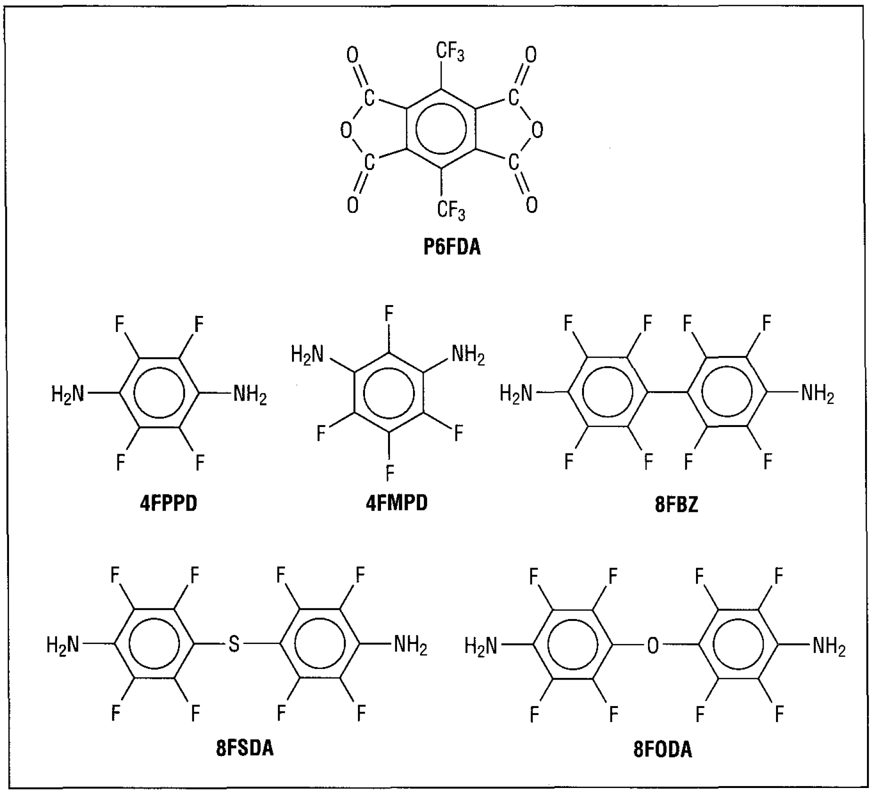


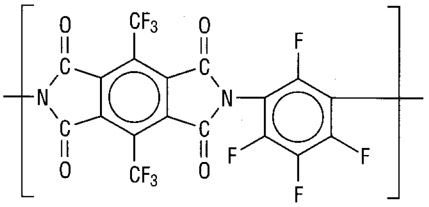
***Figure 6.* These structures are used to synthesize perfluorinated polyimides.** This perfluorinated dianhydride and the five perfluorinated diamines may be purchased or prepared by known procedures.

stages of polymerization are needed to obtain high molecular weight polyimides from the perfluorinated diamines: a solution polycondensation at temperatures of 130 °C–150 °C followed by a high-temperature, solid state chain extension.

A novel perfluorinated dianhydride is needed

The introduction of fluorine or fluorinated groups into dianhydrides increases their reactivity. The ^{13}C NMR chemical shift of carbonyl carbons (δC) of P6FDA suggests a considerable increase in reactivity (*20*). Therefore, P6FDA should compensate for the low reactivity of perfluorinated diamines.

However, the end-group content of the poly(amic acid) synthesized from 4FMPD and P6FDA was 36% (just higher than that from 4FMPD and 6FDA). Furthermore, the resultant perfluorinated polyimide P6FDA–4FMPD was cracked and brittle and did not form a continuous film. For the other four diamines, the polyimides prepared with P6FDA were coarse powders or films that had many cracks.

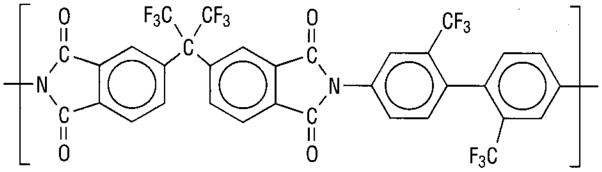


The main reason the films are not continuous is that the high reactivity of the perfluorinated dianhydride probably could not compensate for the low reactivities of perfluorinated diamines. However, the rigidity of the polymer chain cannot be neglected in the cases of P6FDA and one-benzene-ring diamines. In this situation, bond rotation is permitted only at the imide linkage (nitrogen aromatic carbon bond). However, this rotation is restricted by steric hindrance between the fluorine atoms and carbonyl oxygens. The rigidity of the polymer chain must be improved by the introduction of flexible linkage groups into the dianhydride component. Accordingly, we would expect to make continuous and flexible films of perfluorinated polyimides by combining diamines, which have high reactivities, with dianhydrides, which have flexible molecular structures.

We synthesized a novel perfluorinated dianhydride, 1,4-bis(3,4-dicarboxytrifluorophenoxy)tetrafluorobenzene dianhydride (10FEDA), as in the first three steps of the scheme in Figure 7. This molecule has two ether linkages that give flexibility to the molecular structure. In addition, the chemical shift of carbonyl carbons of 10FEDA was almost the same as that of P6FDA. Therefore, this dianhydride should have a reactivity that is higher than those of unfluorinated and partially fluorinated dianhydrides.

The end-group content of the poly(amic acid) prepared from 10FEDA and 4FMPD was 6%, which is much less than that of poly(amic acid) prepared from P6FDA and 4FMPD (36%). This result can be attributed to the considerable increase in flexibility of the dianhydride structure. The resultant perfluorinated polyimide (10FEDA–4FMPD) was a 9.5-μm-thick, strong, flexible film that had a pale yellow color like DuPont's Kapton. The structure of the 10FEDA–4FMPD polyimide was confirmed by elemental analysis, IR spectroscopy, and ^{19}F NMR (*23*).

Figure 8 (p. 26) shows the visible-to-near-IR absorption spectrum of 10FEDA–4FMPD, cured at 200 °C and dissolved in acetone-d$_6$ (10 wt %). The same amount of acetone-d$_6$ was used as a reference. The solid line indicates the absorbance of 10FEDA–4FMPD, and the dashed line indicates the absorbance of partially fluorinated polyimide (6FDA–TFDB).



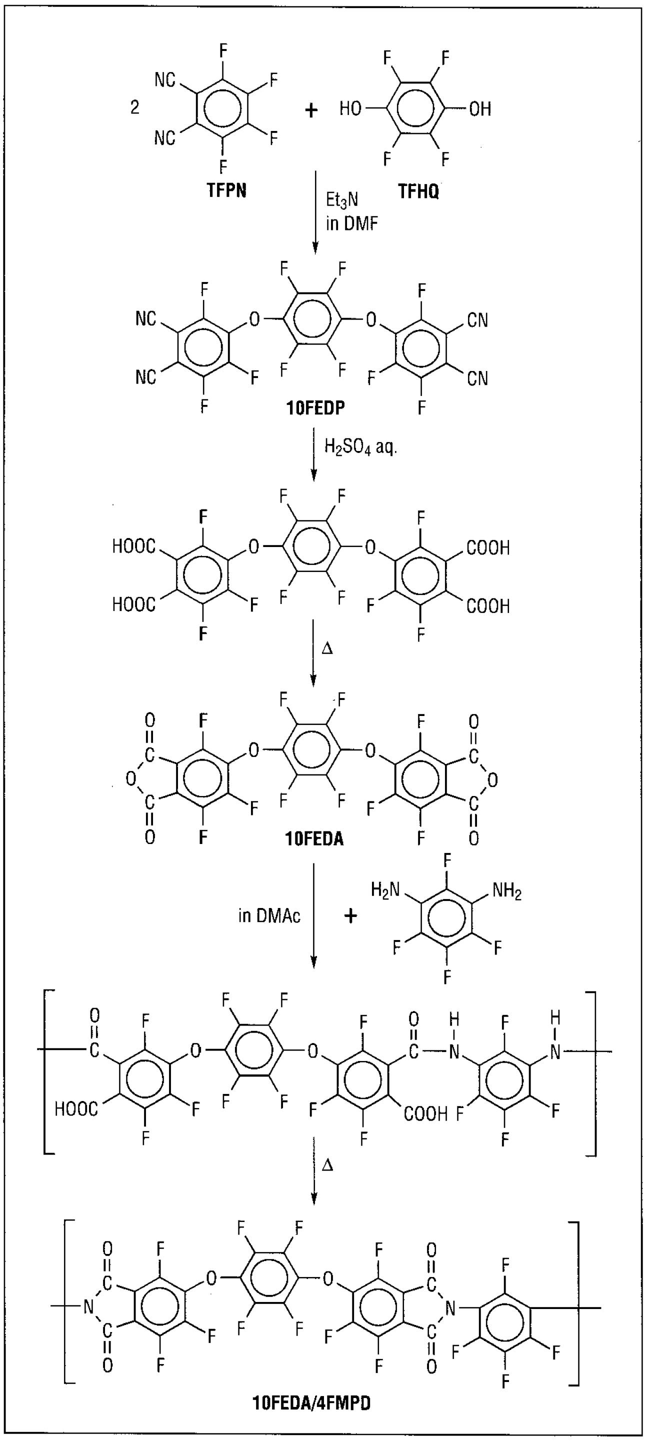


Figure 7. **This scheme shows the steps for synthesizing a new perfluorinated dianhydride, 10FEDA.** From this anhydride, a poly(amic acid) is prepared from which the perfluorinated polyimide is derived.

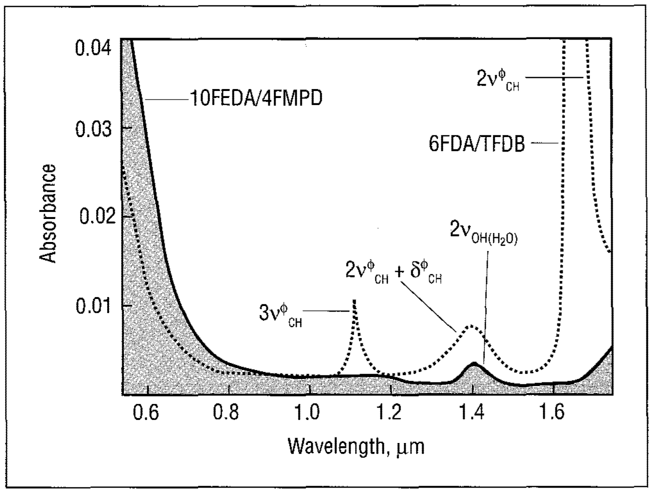


Figure 8. **The perfluorinated polyimide 10FEDA–4FMPD does not have a substantial absorption peak over the wavelengths for optical communication.** However, the partially fluorinated polyimide 6FDA–TFDB does have one. For 10FEDA–4FMPD, indicated by the solid line over the shaded area, the small peak at 1.4 µm is caused by moisture, and the line below it shows what the absorbance would be without the moisture.

Except for a small absorption peak at 1.4 µm caused by moisture absorbed in the solvent or adsorbed onto the polyimide film, the perfluorinated polyimide has no substantial absorption peak over the wavelengths for optical communication. However, partially fluorinated polyimide has an absorption peak that arises from the third harmonic of the stretching vibration of the C–H bond (1.1 µm), a peak that results from the combination of the second harmonic of stretching vibration and the deformation vibration of the C–H bond (1.4 µm), and a peak that is attributable to the second harmonic of the stretching vibration of the C–H bond (1.65 µm). In the visible spectrum, the electronic transition absorption of 10FEDA–4FMPD is slightly shifted to longer wavelengths than that of 6FDA–TFDB. This shift causes the yellowish color of 10FEDA–4FMPD but does not affect the transparency at near-IR wavelengths.

Table 2 lists the strengths and flexibilities of perfluorinated polyimide films synthesized from the two dianhydrides and five diamines. Polymerizing 10FEDA with 8FODA or 8FSDA produced continuous, flexible films, but the films are slightly brittle compared with 10FEDA–4FMPD. As already mentioned, P6FDA did not give any continuous films.

Table 2. **Strengths and flexibilities of perfluorinated polyimide films**

	Dianhydride	
Diamine	**10FEDA**	**P6FDA**
4FPPD	—	No film
4FMPD	Strong and flexible	Brittle and cracked
8FODA	Flexible	No film
8FSDA	Flexible	No film
8FBZ	No film	No film

The properties of perfluorinated polyimides, along with those of partially fluorinated and unfluorinated polyimides, are listed in Table 3. The structure of PMDA–ODA is the same as that of Kapton. Because the 10FEDA component has a flexible structure, the polymer decomposition temperatures and glass transition temperatures (T_gs) of perfluorinated polyimides are slightly lower than those of conventional polyimides. Nonetheless, the thermal stability of these films is considered high enough to be able to withstand the manufacture of integrated circuits and multichip modules.

In addition, the direct introduction of fluorines into the aromatic rings does not cause a substantial increase in fluorine content. The adhesiveness of the perfluoropolyimides is thus equivalent to that of the partially fluorinated polyimides used to fabricate single-mode waveguides. Their dielectric constants (ϵ) at 1 kHz and their average refractive indices are as low as those of the partially fluorinated polyimides. The reason for these similarities is that the fluorine content of perfluorinated polyimides is comparable to that of partially fluorinated polyimides.

However, the birefringence of perfluorinated polyimides is lower than that of partially fluorinated polyimides. This difference is caused by the steric effect between perfluorinated aromatic rings and bent structures such as ether, thio-ether, and meta linkages. The low birefringence is convenient for designing waveguide structures in the interconnections of OEICs and multichip modules.

The refractive indices of perfluorinated polyimides can be controlled by copolymerization in the same manner as those of partially fluorinated polyimides (*14*). These characteristics show that perfluorinated polyimides are promising materials for waveguides in integrated optics and optical interconnect technology.

Table 3. **Properties of perfluorinated polyimides, partially fluorinated polyimides, and unfluorinated polyimides**

	Fluorine content, %	**Decomposition temp., °C**	**T_g, °C**	**Dielectric constant, ϵ**	**Average refractive index, $\bar{n}$**	**Birefringence, $n_{TE} - n_{TM}$**
10FEDA–4FMPD	36.6	501	309	2.8	1.562	0.004
10FEDA–8FODA	38.4	485	300	2.6	1.552	0.004
10FEDA–8FSDA	37.7	488	278	2.6	1.560	0.006
10FEDA–TFDB	35.1	543	312	2.8	1.569	0.009
6FDA–TFDB	31.3	553	327	2.8	1.548	0.006
PMDA–TFDB	22.7	613	>400	3.2	1.608	0.136
PMDA–ODA	0	608	>400	3.5	1.714	0.088

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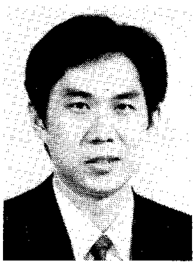

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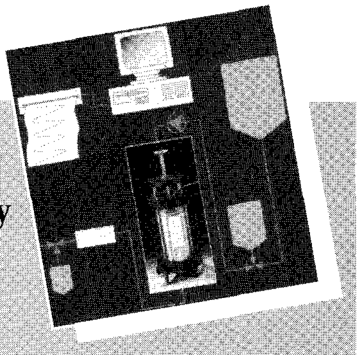

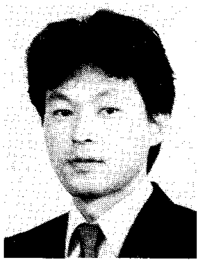

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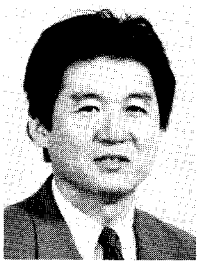

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EDUCATION: AN INDUSTRIAL IMPERATIVE

Our educational system does not appear to be capable of producing persons with the flexibility necessary to deal with the changes that American industry feels are imperative if it is to be competitive. Neither does our educational system seem to be capable of undergoing the changes necessitated by today's societal pressures.

Conventional wisdom currently holds that future workers should be able to acquire the flexibility necessary to respond to changing needs by learning new skills in an appropriate educational system. It's not as if the American educational system is lacking resources; rather, it appears that the system does not provide those elements of education that infuse students with the ability to learn new skills.

J. J. Lagowski
Editor, *Journal of Chemical Education*