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Polyimide Derived from 2,2′-Bis(trifluoromethyl)-4,4′-diaminobiphenyl. 1. Synthesis and Characterization of Polyimides Prepared with 2,2-Bis(3,4-dicarboxyphenyl)hexafluoropropane Dianhydride or Pyromellitic Dianhydride

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ABSTRACT: New fluorinated polyimides have been synthesized by the reaction of 2,2′-bis(trifluoromethyl)-4,4′-diaminobiphenyl (TFDB) with 2,2-bis(3,4-dicarboxyphenyl)hexafluoropropane dianhydride (6FDA) or pyromellitic dianhydride (PMDA). The polyimide 6FDA/TFDB from the reaction of TFDB with 6FDA has high optical transparency and high solubilities in several solvents such as *N,N*-dimethylacetamide (DMAc), tetrahydrofuran (THF), acetone, and ethyl acetate. In addition, it has a low dielectric constant of 2.8 at 1 MHz, a low refractive index of 1.556 at 589.3 nm, and a low water absorption rate of 0.2%. The polyimide PMDA/TFDB prepared from the reaction of TFDB with PMDA has not only a low dielectric constant of 3.2 and a low refractive index of 1.647, but also a low coefficient of thermal expansion (CTE) of -5×10^{-6} °C^{-1}.

Introduction

Organic polymers containing imide groups in the main chain have excellent thermal stability. Conventional polyimides like Kapton produced by Du Pont have been applied to microelectronic devices[1,2] and aerospace fields.[3,4] However, these polyimides are generally insoluble in organic solvents, exhibit low optical transparency, and have intense yellow color. Soluble polyimides are needed as coating materials on specific space components such as solar cells and thermal control systems.[5] Polyimides with high optical transparency are also required as optical waveguide materials.[6]

Less colored polyimide films have been reported by St. Clair and co-workers for varying the molecular structure so as to introduce fluoroalkyl groups, to separate chromaphoric groups, to reduce electronic interaction between color-causing centers, and to lower the color intensity of the resulting polymer.[7,8] Gibbs and Breder demonstrated soluble and melt-processable polyimides incorporating versatile fluorinated groups.[9]

In recent years, polyimides, known as engineering plastics, have become more and more important in the microelectronic industry. Applied to interlayer dielectrics, low dielectric constant polyimides are vital for high signal propagation speed.[10] Furthermore, low water absorption polyimides are needed to maintain electric stability and prevent metal corrosion.[11] Low thermal expansion polyimides are also needed because they are used with low thermal expansion substrates such as aluminum, silicon, or silicon dioxide. Low thermal expansion polyimides have been reported in detail by Numata and co-workers.[12]

In applying polyimides to optical use, the refractive index is one of the most important parameters and must be controlled.

We have been investigating the synthesis and characterization of fluorinated polyimides for application to electrical and optical components. We have already reported a new series of polyimides containing fluorinated alkoxy side chains, prepared from novel fluorinated alkoxy diamines. In these polyimides, the dielectric constant, the refractive index, and the water absorption rate decrease with increasing fluorine content.[13]

In addition to the low dielectric constant, the low refractive index, and the low water absorption rate, we have studied the synthesis of low thermal expansion and high optical transparent polyimides and have found that 2,2′-bis(trifluoromethyl)-4,4′-diaminobiphenyl (TFDB) monomer, which is benzidine with two trifluoromethyl groups, is effective for the synthesis of low thermal expansion and high optical transparent polyimides. Rogers and co-workers have reported the synthesis and properties of polyamides using TFDB.[14] For polyimides prepared from TFDB, we reported in detail on the synthesis and properties such as the coefficient of thermal expansion (CTE), dielectric constant, refractive index, water absorption rate, and polymer decomposition temperature.[15–18] Harris and co-workers also reported on the synthesis and properties.[19,20]

This article gives the results of studies on the synthesis and characterization of polyimides based on TFDB.

Experimental Section

Materials. The diamines and dianhydrides for polyimide synthesis are shown in Figure 1. The TFDB used was synthesized by the method given by Maki and Inukai[21] and was purified carefully by sublimation under reduced pressure. The 2,2-bis(3,4-dicarboxyphenyl)hexafluoropropane dianhydride (6FDA) was obtained from Hoechst Japan Ltd. and was purified by sublimation under reduced pressure. The pyromellitic dianhydride (PMDA) was obtained from Mitsui Toatsu Chemical Inc. and was purified by recrystallization from acetone. The 2,2′-dimethyl-4,4′-diaminobiphenyl (DMDB) was obtained from Wakayama Seika Co. Ltd. and was purified by distillation under reduced pressure. The *N,N*-dimethylacetamide (DMAc), which had a water content below 0.005% and an evaporation residue below 0.0010%, was obtained from Aldrich Chemical Co., Inc., and used without further purification.

Polyimide Preparation. Polyimide Prepared from 6FDA and TFDB (6FDA/TFDB). Preparation of the Poly(amic acid). The polyimide for this study was prepared by the reaction shown in Scheme I. 6FDA (13.3274 g, 30.000 mmol) was dissolved in DMAc (206.4 g) to give a clear, colorless solution. TFDB (9.6072

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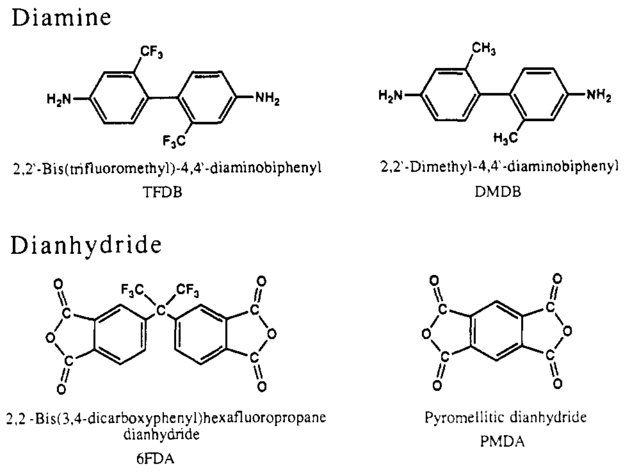
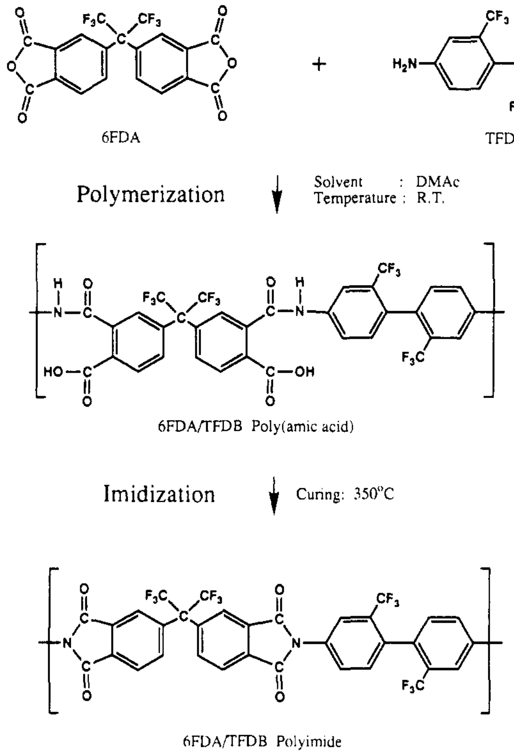


Figure 1. Diamines and dianhydrides.

Scheme I



g, 30.000 mmol) was added to the solution with vigorous stirring. The solution was stirred in a dry nitrogen atmosphere at room temperature for 48 h. The solution became viscous. The solution viscosity was approximately 100 P at low frequency. At this stage, 10 wt % poly(amic acid) solution was obtained. This poly(amic acid) solution was kept at −50 °C in a dry nitrogen atmosphere to prevent decomposition. All preparation of poly(amic acid) was carried out under a dry nitrogen atmosphere.

Conversion of Poly(amic acid) into Polyimide. The poly(amic acid) solution was spin-cast onto a clean dry silicon substrate to give a thin layer of solution. It was then heated to 350 °C in an inert oven in the following steps: 70 °C, 2 h; 160 °C, 1 h; 250 °C, 0.5 h; 350 °C, 1 h.

This heat treatment promoted imidization and converted poly(amic acid) into polyimide. Whereas the IR absorption peak due to carbonyl stretching of the amide group appeared at about 1720 cm^{-1} in the poly(amic acid), the absorption peaks due to carbonyl asymmetric and symmetric stretching of imide groups appeared at 1740 and 1790 cm^{-1} in the polyimide (Figure 2). The polyimide film was elastic and flexible, and had excellent transparency.

Polyimide Prepared from PMDA and TFDB (PMDA/PFDB). PMDA/TFDB poly(amic acid) solution (DMAc 145.4 g, 10 wt %) was prepared in the same manner as the 6FDA/TFDB, but with PMDA (6.5436 g, 30.000 mmol) instead of 6FDA, and was then converted into PMDA/TFDB polyimide in the same way.

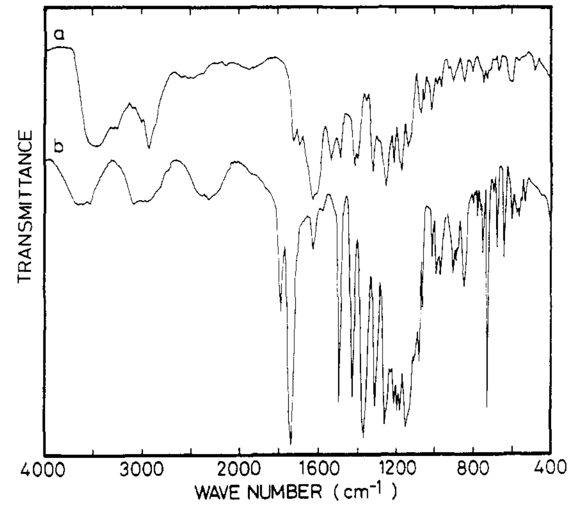


Figure 2. IR spectra of 6FDA/TFDB poly(amic acid) (a) and polyimide (b).

Polyimide Prepared from PMDA and DMDB (PMDA/DMDB). PMDA/DMDB poly(amic acid) solutions (DMAc 116.2, 10 wt %) was prepared in the same manner as the PMDA/TFDB, but with DMDB (6.3690 g, 30.000 mmol) instead of TFDB, and was then converted into PMDA/DMDB polyimide in the same way.

Measurements. The IR spectra were measured with a Hitachi 270-30 IR spectrometer. The ^1H NMR spectra were measured with a Bruker MSL-400 NMR spectrometer, using dimethyl-d_6 sulfoxide as a solvent. The ^1H chemical shifts were calibrated by using tetramethylsilane (TMS). The UV-visible spectra were measured with a Hitachi U-3400 photometer.

The intrinsic viscosities of the poly(amic acid)s in DMAc solutions were measured at 30 °C with a Ubbelohde viscometer setting in a Lauda Viscotimer S.

The polymer decomposition temperatures were measured by thermogravimetry analysis (TGA) with a Perkin-Elmer TGA-7 thermogravimetric analyzer. The glass transition temperatures were measured by differential scanning calorimetry (DSC) with a Perkin-Elmer DSC-7 differential scanning calorimeter. The TGA and DSC measurements were conducted with a heating rate of 10 °C/min in a nitrogen atmosphere.

The dielectric constants were measured by the bridge method with a YHP 4278 capacitance meter at a frequency of 1 MHz and a temperature of 23 °C. Specimens for dry samples were subjected to vacuum pretreatment of 1 Torr at 120 °C for 2 h to eliminate absorbed water. Specimens for wet samples were subjected to a 50% relative humidity (RH) air atmosphere at 23 °C for 24 h after the measurement as a dry sample.

The refractive indexes were measured by Abbe's method with an Atago 4T-Type refractometer. The measurement conditions were a temperature of 20 °C and a light wavelength of 589.3 nm from the sodium D line.

The water absorption rates were measured. Specimens were immersed in 23 °C water, and the weight differences after aging were measured.

The CTEs were measured by thermomechanical analysis (TMA) with a Sinku-Riko TMA-7000 thermomechanical analyzer. Specimens were made with 5-mm width, 15-mm length, and 15-μm thickness. The measurements were carried out three times during elongation with a heating rate of 5 °C/min in a nitrogen atmosphere at a load of 3 g. After the first measurement (first run), the sample was cooled gradually to 25 °C in a nitrogen atmosphere. Then the second measurement (second run) was made. The same operation was carried out between the second run and the third run. The CTE values were determined as the mean at 50–300 °C.

Table I
Elemental Analysis of Fluorinated Polyimides

element	6FDA/TFDB calcd	6FDA/TFDB found	PMDA/TFDB calcd	PMDA/TFDB found
C	54.41	54.43	57.39	57.17
H	1.66	1.57	1.61	1.57
N	3.85	3.92	5.58	5.64

Table II
Characteristics of Fluorinated Polyimides

	6FDA/TFDB	PMDA/TFDB
fluorine content, %	31.3	23.0
intrinsic viscos.[a]	1.00	1.79
polymer decompstn temp,[b] °C	569	610
glass transition temp,[c] °C	335	>400
dielectric constant[d]		
dry	2.8	3.2
wet (50% RH atmos)	3.0	3.6
refractive index[e]	1.556	1.647
water absorptn rate,[f] %	0.2	0.7
coeff of thermal expansn,[g] °C^{-1}		
first run	4.8×10^{-5}	3×10^{-6}
second run	8.2×10^{-5}	-5×10^{-6}

[a] Measured in poly(amic acid) solution. [b] 10% weight loss in N_2 atmosphere. [c] Measured by DSC. [d] At 1 MHz. [e] λ = 589.6 nm, 20 °C. [f] After 3 days. [g] Temperature range 50–300 °C.

Results and Discussion

Characterization of Polymers. Table I shows the elemental analysis of the fluorinated polyimides, 6FDA/TFDB and PMDA/TFDB. The experimental values from both polyimides agree with the calculated values. Therefore, the polymerization and imidization reactions are completed without byproducts.

Table II shows the characteristics of the fluorinated polyimides. As the intrinsic viscosities of both poly(amic acid)s are high, polymerization to high molecular weight proceeds easily. The viscosity of PMDA/TFDB is higher than that of 6FDA/TFDB because of its rigid-rod main chains and/or its higher molecular weight.

The polymer decomposition temperature of 6FDA/TFDB is 569 °C, and nearly as high as PMDA/TFDB and conventional polyimides; however, the glass transition temperature of 6FDA/TFDB, 335 °C, is lower than that of PMDA/TFDB. 6FDA/TFDB has flexible hexafluoroisopropylidene groups between the rigid phenyl rings in the main chains and a smaller imide content.

The dielectric constant of dry 6FDA/TFDB is very low (2.8 at 1 MHz) because of its four trifluoromethyl groups (fluorine content 31.3%). The low dielectric constants of fluorinated polyimides result from the reduced chain–chain electronic interaction, due to the low electronic polarizability of the fluorine. The dielectric constant of wet 6FDA/TFDB (50% RH atmosphere), 3.0, is a little larger than that of dry 6FDA/TFDB, 2.8. It exhibits little increase in dielectric constant even after being kept in the wet condition. The stability of the dielectric constant is related to the water absorption. The water absorption rate of 6FDA/TFDB is very small, 0.2%, because of the waterproofing effect of the fluorine atoms. It is smaller than that of PMDA/TFDB, 0.7%. The stability of the dielectric constant is one of the most important properties for interlayer dielectrics in microelectronic devices.

In addition, the refractive index of 6FDA/TFDB, 1.556, is much lower than that of the low fluorine content PMDA/TFDB, 1.647, because of the low electronic polarizability as well as the dielectric constant.

The CTE (second run) of 6FDA/TFDB is higher than that of the rigid-rod structure PMDA/TFDB and is similar to that of conventional polyimides such as PMDA/ODA prepared from PMDA and oxydianiline (ODA), (PMDA/ODA has the same structure as Kapton, produced by Du Pont). The reason 6FDA/TFDB and PMDA/ODA have high CTEs is that their main chains have bent hexafluoroisopropylidene units, and bent ether units, respectively. These bent units loosen the molecular packing in the film. In the 6FDA/TFDB film, the CTE in the first run of 4.8×10^{-5} °C is lower than that of the second run of 8.2×10^{-5} °C. The CTE of the 6FDA/TFDB film is larger than that of the silicon substrate. Therefore, this film, prepared on a silicon wafer at 350 °C, is in an elongated state during cooling to room temperature. Upon removal from the substrate, the film is under stress. Therefore, at the first TMA measurement the stress is released, and the CTE is lower than after the second run. On the other hand, with the PMDA/TFDB film, the CTE of the first run of 3×10^{-6} °C is higher than that of the second run of -5×10^{-6} °C. The CTE of PMDA/TFDB film is lower than that of the silicon substrate, and this film shrinks during cooling to room temperature while on the substrate. Therefore, at the first TMA measurement, the stress is released in the form of expansion, and the CTE is higher than after the second run. In the third TMA measurement in both films, the CTEs are almost the same as those of the second run. A polyimide film was prepared by copolymerization of 6FDA/TFDB and PMDA/TFDB so as to have the same CTE as that of silicon; its CTE in the first run was equal to that of the second run.[17]

Table III
Solubility of Fluorinated Polyimides[a]

solvent	6FDA/TFDB 70 °C	6FDA/TFDB 200 °C	6FDA/TFDB 350 °C	PMDA/TFDB 70 °C	PMDA/TFDB 200 °C	PMDA/TFDB 350 °C[b]
N,*N*-dimethylacetamide	++	++	++	++	–	–
acetone	++	++	++	–	–	–
tetrahydrofuran	++	++	++	–	–	–
ethyl acetate	++	++	++	–	–	–
methanol	++	+	–	–	–	–
chloroform	+	+	–	–	–	–
benzene	–	–	–	–	–	–
n-hexane	–	–	–	–	–	–

[a] Key: ++, easy to dissolve; +, soluble; –, insoluble. [b] Final cure temperature.

Solubility Studies. The most significant characteristic of 6FDA/TFDB is its excellent solubility. Table III shows the dependence of 6FDA/TFDB and PMDA/TFDB solubilities in various solvents on cure temperature. Poly(amic acid) solution on the silicon wafer was heated to 70 °C at 1 Torr over 12 h to remove the solvent. Then, it was heated to the given temperature over 1 h. After curing, the polymer film was peeled from the silicon wafer. Although the low fluorine content polyimide, PMDA/TFDB, and conventional nonfluorinated polyimides such as PMDA/ODA are insoluble in all solvents, 6FDA/TFDB polyimide film is still soluble in DMAc, tetrahydrofuran (THF), acetone, and ethyl acetate after curing at 350 °C. After curing at up to 200 °C, 6FDA/TFDB is also soluble in methanol and chloroform.

Polyimides are generally insoluble because they have many aromatic rings in the main chain. However, the poly(amic acid), the precursor of the polyimide, dissolves because of the electronic polarizability of its amide groups and carboxyl groups. Even in this case, the poly(amic acid) is only soluble in particular polar solvents such as DMAc. However, 6FDA/TFDB is soluble in many solvents because the hexafluoroisopropylidene groups separate the aromatic rings, and trifluoromethyl groups hinder the interaction between neighboring molecules.

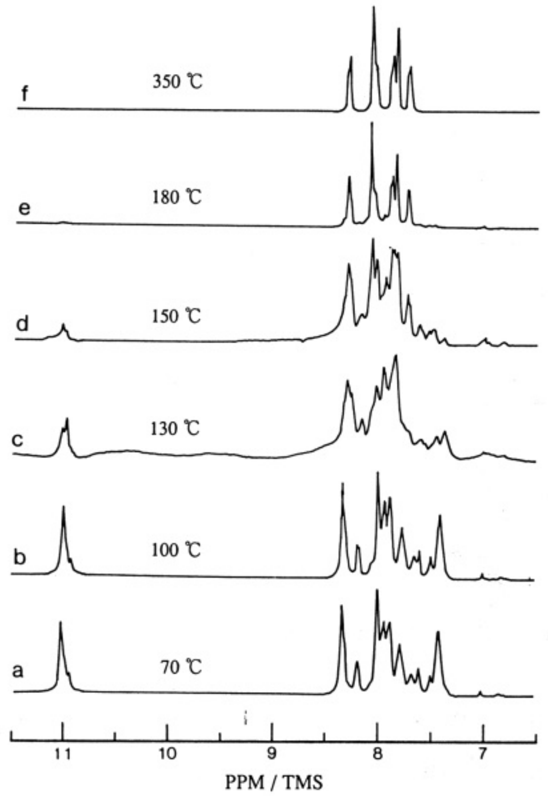


Figure 3. ^{1}H NMR spectra of 6FDA/TFDB poly(amic acid) during imidization.

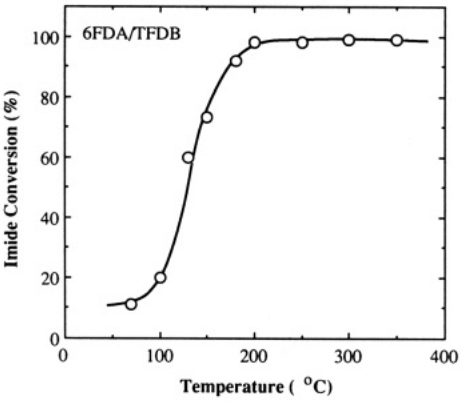


Figure 4. Dependence of imide conversion on curing temperature.

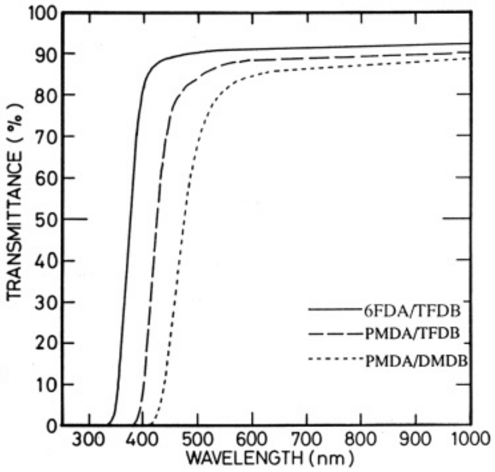


Figure 5. UV–visible spectra of polyimide films (film thickness 20 μm).

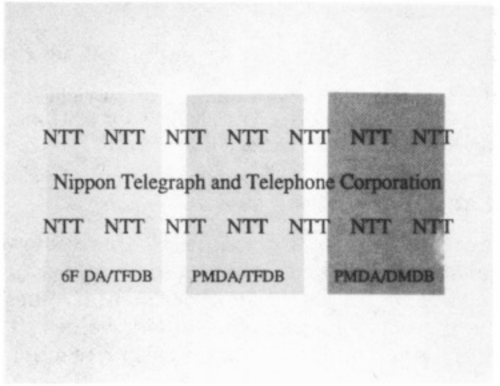


Figure 6. Comparison of color intensity.

Imidization. The imide conversion from poly(amic acid) to polyimide can be observed with NMR spectrometry, as 6FDA/TFDB shows excellent solubility. Figure 3 shows high-resolution ^{1}H NMR spectra of poly(amic acid)s during imidization. Each sample was dissolved in dimethyl-d_6 sulfoxide. At 70 °C, the large peaks at 11 ppm in spectrum a corresponds to the COOH proton in the poly(amic acid). As the curing temperature rises, the intensity of the COOH proton peaks reduces, because the poly(amic acid) converts to the polyimide. When the curing temperature is 180 °C (spectrum e), the COOH proton peaks almost disappear. Figure 4 shows the dependence of imide conversion on curing temperature. The imide conversion is calculated by integrating the intensity ratio between the COOH proton peaks and the aromatic proton peaks from 7.9 to 8.4 ppm. This figure shows that imidization begins at just over 70 °C and finishes at about 200 °C. At curing temperatures over 200 °C, poly(amic acid) is completely converted into polyimide. This result is consistent with the solubility results in Table III. 6FDA/TFDB is soluble in DMAc, acetone, methanol, THF, ethyl acetate, methanol, and chloroform until 200 °C (where it is partially converted to polyimide), but it does not dissolve easily in methanol and chloroform at 200 °C and is insoluble in these two solvents at 300 °C (where it is completely converted).

UV–Visible Spectroscopic Studies. The fluorinated polyimides are transparent, and the 6FDA/TFDB film has greater transparency than the PMDA/TFDB film, because electron conjugation is prohibited by the fluoroalkyl unit in the 6FDA/TFDB molecular main chain and the fluorine content is large. Transmission UV–visible spectra of 20-μm-thick polyimide films are shown in Figure 5. The 6FDA/TFDB film shows strong absorption, with a cutoff between 300 and 400 nm, and no distinct peak maxima are obtainable. The cutoff wavelength of the absorption in 6FDA/TFDB is lower than that of the other polyimides (PMDA/TFDB and PMDA/DMDB). Transmittances of 6FDA/TFDB are higher than those of the other films and are directly related to the color intensity of the film. The color intensity of the 6FDA/TFDB film is compared to those of PMDA/TFDB and PMDA/DMDB films of the same thickness in Figure 6. St. Clair and co-workers demonstrated that polyimides prepared with 6FDA had UV cutoffs at low wavelengths and correspondingly had high optical transparency over a broad range.[7] The structure of PMDA/TFDB is similar to that of PMDA/DMDB, and optical transparency increases by changing the diamine monomer from nonfluorinated DMDB to fluorinated TFDB. This indicates clearly that the substitution for side methyl groups by side trifluoromethyl groups increases optical transparency. The trifluoromethyl groups in TFDB diamine units separate the chromaphoric centers and cut the electronic conjugations.

In addition, the optical transparency of 6FDA/TFDB becomes excellent by changing the dianhydride monomer from nonfluorinated PMDA to fluorinated 6FDA. The fluorine content of 6FDA/TFDB is the highest in these polyimides, and fluoroalkyl groups in the 6FDA dianhydride units cut the intramolecular conjugation.

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Registry No. (6FDA)(TFD) (copolymer), 129197-26-8; (6FDA)(TFD) amic acid (SRU), 134628-79-8; (6FDA)(TFD) imide (SRU), 129219-42-7; (PMDA)(TFDB) (copolymer), 129197-24-6; (PMDA)(TFDB) amic acid (SRU), 134904-53-3; (PMDA)-(TFDB) imide (SRU), 129219-40-5.