# Transient Absorption Spectra of Photoconductive Polyimides and Their Model Compound by Picosecond Pulse Radiolysis

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Transient absorption spectra of an aromatic polyimide, PI(PMDA/ODA), and a polyimide with an alicyclic diamine, PI(PMDA/DCHM), were determined in order to investigate the main active species in the photoconductivity of the polyimide films by picosecond pulse radiolysis method. A model compound, *N,N′*-di-*n*-butylpyromellitic diimide (DBPMDI), was utilized to investigate the active species of pyromellitic imide moieties in solutions of dichloromethane (DCM) and tetrahydrofuran (THF). In the aromatic polyimide film, the radical anions of the electron-accepting pyromellitic imide moieties at 660 and 720 nm and the radical cation of diphenyl ether moieties at 550 nm were determined by electron transfer from the aromatic ring to imide moieties in the polymer backbone. In the polyimide film with the alicyclic diamine, by utilizing the model system, only the radical anions of pyromellitic imide moieties could be assumed at 660 and 720 nm as the active species in the photoconductivity of the polyimide film. These active species, such as radical cations or radical anions, are expected to be the charge carriers of holes or electrons under the applied electric field, and the transport of these charge carriers would result in the photoconductivity of the polyimide films.

## Introduction

Polyimides have been widely used as the materials for microelectronics due to their high thermal stability, solvent insensitivity, and good dielectric characteristics. The photoconductivity of aromatic polyimide films has been reported,[1−6] while organic photoconductors (OPCs) and photoconductive polymers have been investigated for applications to xerographic photoreceptors of photocopying or printing machines.[7−10]

In recent years, the polyimides prepared from alicyclic diamines are attracting much interest because they have good optical spectroscopic and dielectric properties as well as good thermal stability.[11−13] One of the reasons for their high performance is due to the elimination of charge transfer, because the charge-transfer state of aromatic polyimides is formed between the aromatic diamines as electron donors and the aromatic dianhydrides as electron acceptors both intra- and intermolecularly.[14,15] In our previous paper,[16] the photoconductivity of the polyimide film, PI(PMDA/DCHM), prepared from pyromellitic dianhydride (PMDA) and diaminodicyclohexylmethane (DCHM) was investigated, compared with that of an aromatic polyimide, PI(PMDA/ODA), prepared from PMDA and oxydianiline (ODA). We reported that the polyimide film prepared from the alicyclic diamine showed equal photoconductive properties compared with the aromatic polyimide film, even without the addition of any donors. In addition, enhanced photoconductivity compared to that of the aromatic polyimide film was observed for the polyimide film prepared from the alicyclic diamine even though it has no charge-transfer (CT) complex in the polymer backbone. LaFemina[15] reported that the radical anions formed by electron transfer from diphenyl ether to pyromellitic imide moieties resulted in the photocon-

duction of Kapton polyimide film. Takai[2] measured polarity dependence in the photoconductivity of Kapton polyimide film and reported that no polarity dependence was observed. It means that the apparent mobilities of photogenerated electrons and holes are almost the same. Freilich[5] observed the transient absorptions of a donor-loaded polyimide film, PI(PMDA/ODA)/DMA, by the addition of dimethylaniline (DMA) as a donor in order to determine the active species in the photoconductivity of the donor-loaded polyimide film by using the Nd:YAG laser flash photolysis technique.

In the present paper, we measured the transient absorption spectra of polyimide films by picosecond pulse radiolysis technique in order to determine the main active species in the photoconductivity of polyimide films without the addition of donor. There has been no previous work on the picosecond pulse radiolysis of polyimides. Pulse radiolysis is one of the methods for the study of electron-transfer reactions.[17] In solution, this method allows production of many primary solvent radicals and secondary solute radicals and determination of absolute rate constants of their ensuing reactions. Thus, the study of a model system by using solvents of DCM and THF is appropriate to assist in the assignment of transient phenomena. *N,N′*-di-*n*-butylpyromellitic diimide (DBPMDI) is used as a model compound for the pyromellitic imide unit in the polymer backbone. A similar model compound, *N,N′*-di-*n*-pentylpyromellitic diimide (DPPMDI), was investigated as an electron acceptor by Sep et al.,[18] reporting the solvatochromism of charge-transfer complexes formed by DPPMDI and electron donors in strongly hydrogen bonding solvents.

## Experimental Section

**Materials and Solvents.** Pyromellitic dianhydride (PMDA) supplied by Tokyo Kasei Co. Ltd. was purified by recrystallization in distilled acetic anhydride and dried at high temperature (150 °C) under vacuum for 12 h. 4,4′-Diaminodicyclohexylmethane (mixture of isomers, DCHM) was supplied by Wako Pure Chemical Co., which was used without further purification.

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**PI(PMDA/DCHM)**
(Pyromellitic dianhydride / Diaminodicyclohexylmethane)

**PI(PMDA/ODA)** (Pyromellitic dianhydride / Oxydianiline)

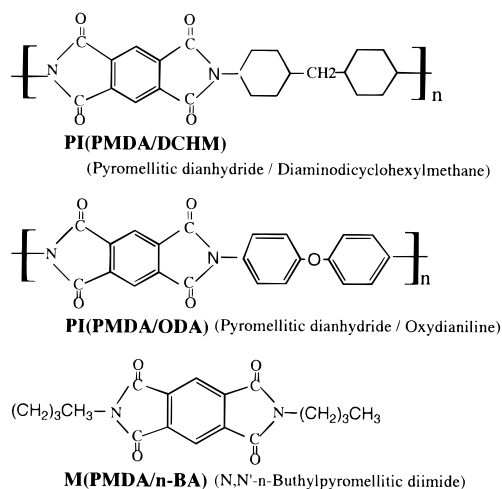**M(PMDA/n-BA)** (N,N'-n-Buthylpyromellitic diimide)

**Figure 1.** Chemical structures of an aromatic polyimide, PI(PMDA/ODA), a polyimide with an alicyclic diamine, PI(PMDA/DCHM), and a model compound of the latter DBPMDI.

4,4′-Diaminodiphenyl ether (ODA) and *n*-butylamine (BA) were supplied by Tokyo Kasei Co. Ltd. and used without further purification. Distilled *N,N′*-dimethylacetamide (DMAc) and *N,N′*-dimethylformamide (DMF), provided by Tokyo Kasei Co., were used as solvents for preparing poly(amide acid) (PAA) and a model compound, respectively. Pyrene (Tokyo Kasei Co. Ltd.) was subject to chromatographic purification, which was used as a cation scavenger. Solvents for the transient absorption measurements, dichloromethane (DCM) and tetrahydrofuran (THF), were supplied by Tokyo Kasei Co. Ltd. and used without further purification.

**Preparation of Polyimides and Its Model Compound.** The poly(amide acid)s, PAA(PMDA/ODA) and PAA(PMDA/DCHM), were prepared at room temperature by 24 h stirring of stoichiometric PMDA with ODA and PMDA with DCHM, respectively, in 5% distilled DMAc solution. The values of intrinsic viscosity were measured to be 1.21 d*L*/g for PAA-(PMDA/ODA) and 1.02 dL/g for PAA(PMDA/DCHM) in DMAc at 25 °C. The 5% poly(amide acid) solutions in DMAc were cast on glass plates and then heated to the imidization temperature region at 50 °C for 12 h and for 1 h each at 160, 180, and 200 °C, resulting in the corresponding polyimides, PI-(PMDA/ODA) and PI(PMDA/DCHM). The almost complete imidization was ascertained with IR spectra. Experimental details of preparation and the characteristics of PI(PMDA/DCHM) have been given by Qin et al.[11,12] *N,N′*-Di-*n*-butylpy-romellitic diimide (DBPMDI) was prepared as a model compound by the reaction of BA with PMDA in DMF and recrystallized in carbon tetrachloride. These chemical structures are shown in Figure 1.

**Preparation of Sample Solutions and Films, Irradiation, and Measurements of Transient Absorption Spectra by Picosecond Pulse Radiolysis.** The 100 mM DBPMDI solutions in THF and DCM and the solution of 100 mM DBPMDI with 5 mM pyrene in DCM were prepared in 2 cm fused silica cells and degassed by the argon bubbling before the measurements. Poly(amide acid) films of about 1 mm thickness were prepared from the poly(amide acid) solutions by the solvent cast method and then transformed to polyimide films by using the thermal imidization technique. The picosecond pulse radiolysis by using an electron linear accelerator, LINAC, was used for the observation of the transient species through the spectrophoto-metric recording of the transient optical absorption spectra. Electron beams of 35 MeV energy with 20 ps pulse duration were used for all experiments. A pulsed flash Xe lamp (EG&G
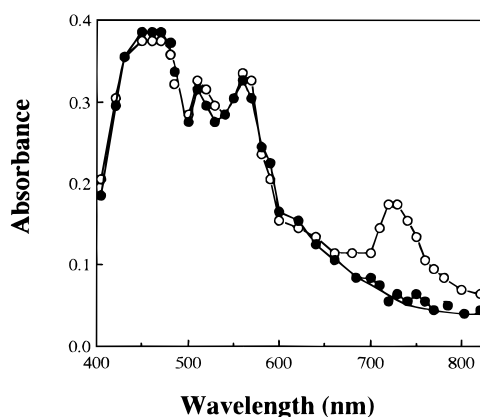
**Figure 2.** Transient absorption spectra of 100 mM DBPMDI solution in DCM measured by using a 20 ps electron pulse. Transient absorptions are observed at the end of the pulse (○) and 80 ns after the pulse (●).
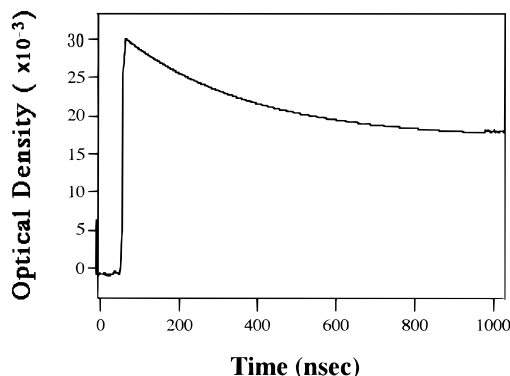
**Figure 3.** Kinetic profile of the transient absorption spectra of 100 mM DBPMDI solution in DCM measured by using a 20 ps electron pulse monitored at 560 nm.

FX279-U) and a PIN photodiode (Hamamatsu Photonics, 1722-01) were used as a monitoring light source and a detector, respectively. The absorption was recorded by using a transient digitizer (Techtronics, SCD-1000). This apparatus is equipped in the Nuclear Engineering Research Laboratory of our University.[19]

**Results and Discussion**

**Transient Absorption Spectra of DBPMDI in Dichlo-romethane.** Aromatic radical cations have been prepared most commonly by pulse radiolysis in a halogenated hydrocarbon.[20−22] These solvents react rapidly with the electrons produced by the radiolysis, and thus only the positive holes react with the aromatic solute to form the radical cation. A model compound, DBPMDI, was prepared for the measurement of the cation radical species of pyromellitic imide moieties using dichlo-romethane (DCM) as a solvent. Figure 2 shows the transient absorption spectra of 100 mM DBPMDI solution in DCM by using a 20 ps electron pulse. A broad absorption at around 450 nm, two stable absorption bands at 510 and 560 nm, and a rapid decay at 720 nm are observed in Figure 2. Both two absorption bands at 510 and 560 nm show a slow single-exponential decay with the same lifetime of 325 ns as is exemplified in Figure 3. These transient absorptions at 510 and 560 nm are assigned differently from the other transient absorptions of 450 and 720 nm and would be attributed to a radical cation of pyromellitic imide moieties. Strong absorption at around 450 nm may be assigned as the transient absorption of charge-transfer (CT) complex formed between the chloride radical anion and the radical cation of pyromellitic imide because this absorption is broad and very stable. Kira et al.[22] have
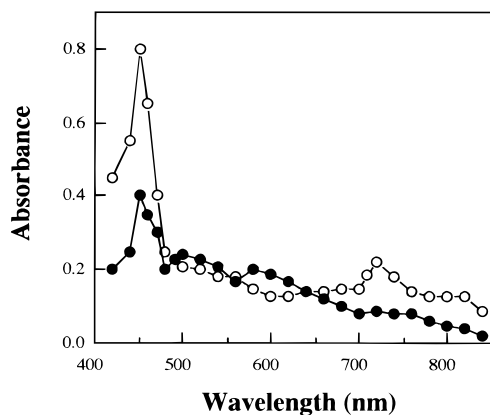
**Figure 4.** Transient absorption spectra of 100 mM DBPMDI and 5 mM pyrene solution in DCM measured by using a 20 ps electron pulse. Pyrene was used as a cation scavenger. Transient absorptions are observed at the end of the pulse (○) and 80 ns after the pulse (●).
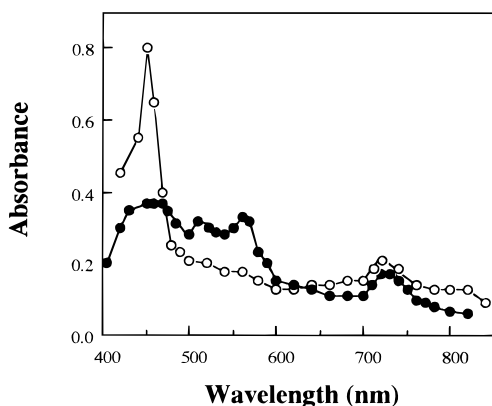


**Figure 5.** Transient absorption spectra of 100 mM DBPMDI in DCM (●) and 100 mM DBPMDI with 5 mM pyrene (○) solutions in DCM measured by using a 20 ps electron pulse. Both transient absorptions are plotted only at the end of the pulse.

reported that radical cations produced by pulse radiolysis method may decay by forming the CT complex with radical anions present in solution.

To investigate the radical cation of pyromellitic imide moieties, a 10 mM DCM solution of pyrene as a cation scavenger was added to an equal amount of 200 mM DBPMDI solution in DCM. It is expected that electron transfer occurs from pyrene to the radical cation of pyromellitic imide moieties, and the radical cation of pyromellitic imide moieties is converted to the radical cation of pyrene. Figure 4shows the transient absorption spectra of 100 mM DBPMDI and 5 mM pyrene in DCM measured by using a 20 ps electron pulse. As shown in Figure 4, the two transient absorptions at 510 and 560 nm disappeared with the addition of 5 mM pyrene. An absorption at 720 nm with a rapid decay is observed at the end of the 20 ps electron pulse. A newly observed sharp transient absorption at 450 nm decays single exponentially with a lifetime of 30 ns, which agrees with that of the monomer radical cation of pyrene reported by Shida et al.[23] and Aalbersberg et al.[24] At 80 ns after the pulse, we can observe weak and broad absorptions at 500, 580, and 750 nm, which agree with those of the dimer radical cation of pyrene reported by Kira et al.[22] The dimer radical cation is generated by the decay process of the monomer radical cation of pyrene.

Figure 5 shows the comparison of the transient absorption spectra of 100 mM DBPMDI in DCM with 100 mM DBPMDI and 5 mM pyrene in DCM observed at the end of the 20 ps electron pulse. These spectra suggest the formation of the
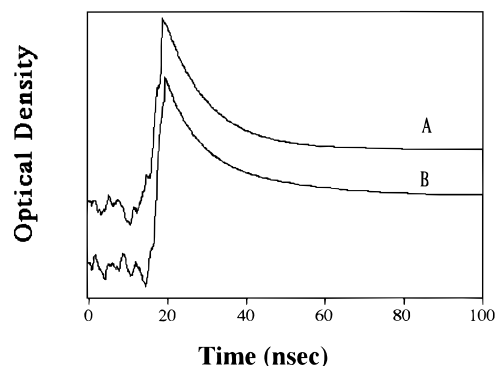


**Figure 6.** Kinetic profiles of the transient absorption spectra of 100 mM DBPMDI (A) and 100 mM DBPMDI with 5 mM pyrene (B) solutions in DCM measured by using a 20 ps electron pulse monitored at 720 nm.
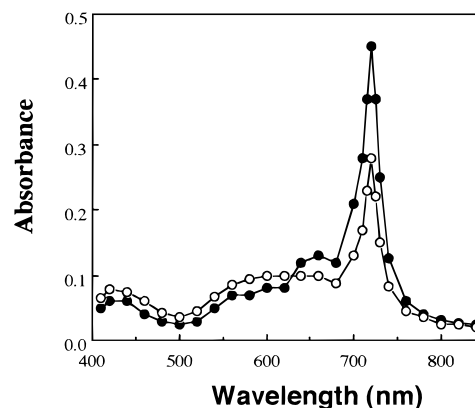


**Figure 7.** Transient absorption spectra of 100 mM DBPMDI solution in THF measured by using a 20 ps electron pulse. Transient absorptions are observed at the end of the pulse (○), and 80 ns after the pulse (●).

radical cation of pyromellitic imide moieties and its conversion to the monomer radical cation of pyrene used as a cation scavenger. Therefore, the transient absorptions at 510 and 560 nm observed by picosecond pulse radiolysis are assigned as the radical cation of pyromellitic imide moieties.

As shown above in Figures 2 and 4, both transient absorption spectra of 100 mM DBPMDI in DCM and 100 mM DBPMDI with 5 mM pyrene in DCM show a rapid decay at 720 nm. As shown in Figure 6, these absorptions, observed at 720 nm by using the 20 ps electron pulse, decay single exponentially with similar lifetimes of 10.7 or 13 ns without or with pyrene, respectively. We can suppose that these absorptions are attributed to the same species, because their kinetic profiles are nearly identical. Therefore, this species observed at 720 nm is assigned to an anionic species of pyromellitic imide moieties by considering the strong electron-accepting properties of imide portion of the molecule. This supposition is supported by the fact that the transient absorption at 720 nm was observed, although, by the addition of pyrene as a cation scavenger, under conditions where the cation species of pyromellitic imide moieties were converted to the radical cation of pyrene moieties by electron transfer (Figure 4).

**Transient Absorption Spectra of DBPMDI in Tetrahydrofuran.** Aromatic radical anions can be produced in ether solution by pulse radiolysis.[25−27] In the radiolysis of ether solution, the solvated electrons, e$^-_{solv}$, react rapidly with aromatic compounds to produce radical anions of aromatic compounds, Ar$^-$. Thus, tetrahydrofuran (THF) is used as a solvent in order to investigate the radical anion species of pyromellitic imide moieties. Figure 7shows the transient absorption spectra of 100 mM DBPMDI solution in THF
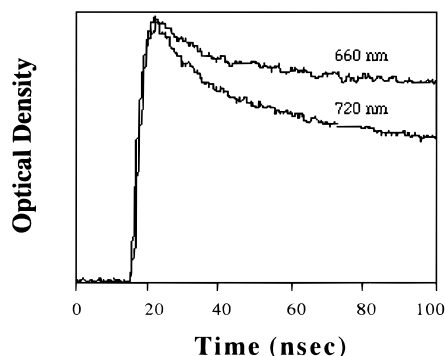
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**Figure 8.** Kinetic profiles of the transient absorption spectra of 100 mM DBPMDI solution in THF measured by using a 20 ps electron pulse, monitored at 660 and 720 nm.



**Figure 9.** Transient absorption spectra of film, PI(PMDA/ODA), measured by using a 20 ps electron pulse. Transient absorptions are observed at the end of the pulse (●), 10 ns after the pulse (○), and 80 ns after the pulse (▲).

measured by using a 20 ps electron pulse. A strong transient absorption at 720 nm and a shoulder at 660 nm are observed at the end of the pulse. This transient absorption observed at 720 nm decays with a shorter lifetime of 8.5 ns and a longer one which cannot be determined in this experiment. A shoulder observed at around 660 nm decays single exponentially with a lifetime of 20 ns. These two absorptions at 660 and 720 nm are regarded as those of anion species of pyromellitic imide moieties because the solvated electrons rapidly react with pyromellitic imide moieties. Shida et al.[27] reported that the strong absorption of a radical anion of PMDA was observed at 666 nm by using pulse radiolysis in THF. Freilich[5] has reported the transient absorptions of radical anions of pyromellitic imide moieties at 659 and 728 nm by using a model system, *N*,*N*′-di-*n*-pentylpyromellitic diimide (DPPMDI), with a Nd:AG laser flash photolysis apparatus. Therefore, the transient absorptions at 660 and 720 nm, obtained by picosecond pulse radiolysis using a 20 ps electron pulse, are attributed to the radical anions of pyromellitic imide moieties. Figure 8 shows the kinetic profiles of the transient absorption spectra monitored at 660 and 720 nm. They show the different decay behavior with single exponential at 660 nm and double exponential at 720 nm, although they are the same species of the radical anion of the pyromellitic imide moieties in the polymer backbone. It would be supposed that the faster decay component observed at 720 nm with a lifetime of 8.5 ns would be due to the absorption of the radical anion of pyromellitic moieties, while the shoulder at 660 nm with a lifetime of 20 ns is due to the imide radical anion conjugated with the pyromellitic moieties.[27]

**Transient Absorption Spectra of Polyimide Films.** To investigate the main active species in the photoconductivity of the polyimide films, transient absorption spectra of PI(PMDA/ODA) film were measured by picosecond pulse radiolysis. As shown in Figure 9, complex spectra with weak and broad absorptions are observed from below 500 nm to above 850 nm in the transient absorption spectra of PI(PMDA/ODA) film. From these complex transient absorption spectra, it can be supposed that various transient active species such as radical cation, radical anion, excited state, etc., are formed because the sample is in the bulk film state. In solution, the transient absorption spectra by picosecond pulse radiolysis are observed clearly and sharply because electron transfer occurs rapidly between pyromellitic imide moieties and solvent. In the spectra of PI(PMDA/ODA) film in Figure 9, we can observe a strong absorption band at 720 nm with a shoulder at 660 nm and a rapid decay at 550 nm in comparison with those of the model systems. The shoulder at 660 nm decays single exponentially with a lifetime of 20 ns, while the transient absorption at 720
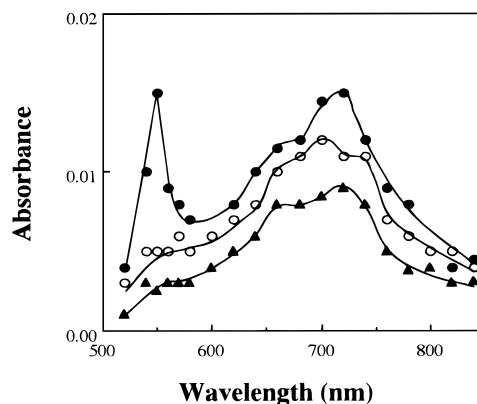
nm decays double exponentially with a faster lifetime of 7.5 ns and a slower one, which cannot be determined in the present experiment. The species for these absorptions would be the same as the radical anions of the model system, 100 mM DBPMDI in THF as above shown in Figure 7. In addition, this assignment agrees with that for the radical anions of pyromellitic imide moieties of the polymer backbone in donor-doped Kapton film measured by Freilich[5,29] with laser transient spectroscopy and is supported the spectra of radical anion of Kapton polyimide film produced by Krause et al.[28] with the chemical reduction technique. Therefore, the transient absorptions at 660 and 720 nm formed in the picosecond pulse radiolysis of the PI(PMDA/ODA) film are attributed to the radical anions of pyromellitic imide moieties of the polymer backbone.

As shown in Figure 9, we can observe a rapid decay at 550 nm determined only at the end of the 20 ps electron pulse whose single exponential lifetime is about 2.2 ns. It may be attributed to the radical cation of diphenyl ether moieties because diaminodiphenyl ether (ODA) was used as an aromatic diamine, though we did not try to measure the transient absorption of diphenyl ether moieties. Washio et al.[30] reported that the radical cation of diphenylmethane (DPM) were observed at around 480 nm by using DCM as a solvent. In the case of the polyimide film prepared from diphenyl ether as an aromatic diamine, it would be assumed that electrons of diphenyl ether moieties are excited by a 20 ps electron pulse to give the radical cation of diphenyl ether moieties, and these electrons transfer to pyromellitic imide moieties to give the radical anion of pyromellitic imide moieties in the polymer backbone. In other words, electron transfer occurs from the aromatic ring to the electron acceptor imide moieties and forms the radical anions of pyromellitic imide moieties at 660 and 720 nm and radical cation of diphenyl ether moieties at 560 nm, as is observed by picosecond pulse radiolysis as shown in Figure 9.

Figure 10 shows the kinetic profiles of the transient absorptions of the PI(PMDA/ODA) film by using a 20 ps electron pulse monitored at 550 and 720 nm. The kinetic profiles show the difference in their decay behavior, i.e., the radical cation of diphenyl ether moieties at 550 nm decays faster with a lifetime of 2.2 ns, while the radical anion of pyromellitic imide moieties at 720 nm decays double exponentially with a faster lifetime of 7.5 ns and a slower one whose lifetime could not be determined. Therefore, it can be supposed that the radical anions of pyromellitic imide moieties at 660 and 720 nm and the radical cation of diphenyl ether moieties at 550 nm are the main carriers in the photoconductivity of the aromatic polyimide film without the addition of donor.
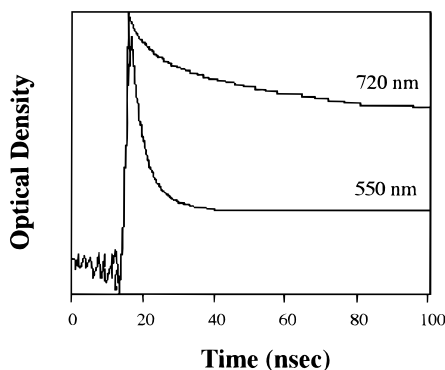
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**Figure 10.** Kinetic profiles of the transient absorption spectra of the aromatic polyimide film, PI(PMDA/ODA), measured by using a 20 ps electron pulse monitored at 550 and 720 nm.

The transient absorption spectra of the polyimide film with an alicyclic diamine, PI(PMDA/DCHM), could not be observed due to the difficulty of sample preparation of about 1 mm thick film, and then its model system, DBPMDI, was utilized instead of the polyimide film. The transient absorption spectra of the polyimide with the alicyclic diamine, PI(PMDA/DCHM), can be assumed to be similar to those of the model system, DBPMDI, for several reasons. One reason is that DCHM and *n*-butylamine (BA) have similar electronic properties in the ground and excited states based on the UV and fluorescence observation reported in our previous work.[16] Another reason is that ionization potentials for these aliphatic compounds are very high and similar to each other, which eliminates the formation of the charge-transfer (CT) complexes in the ground state. Thus, the model system, DBPMDI, was utilized for picosecond pulse radiolysis in order to investigate the main active species in the photoconductivity of the polyimide film with the alicyclic diamine. By referring to Figures 7 and 9, we would say that the strong radical anions of pyromellitic imide moieties observed at 660 and 720 nm by picosecond pulse radiolysis would act as the active species in the photoconductivity of the polyimide with the alicyclic diamine, because it has only the electron acceptor imide portion in the polymer backbone.

## Conclusion

By the measurement of transient absorption spectra with picosecond pulse radiolysis, the active species in the photoconductivity of the polyimide film, PI(PMDA/ODA), were suggested. In the aromatic polyimide film, the radical anions of the electron acceptor pyromellitic imide moieties at 660 and 720 nm and the radical cation of diphenyl ether moieties at 550 nm, formed by electron transfer from aromatic ring to imide portion in the polymer backbone, were observed. The radical cation of diphenyl ether moieties decayed faster than the radical anions of pyromellitic imide moieties in the polyimide film. In the polyimide film with the alicyclic diamine, only the strong radical anions of pyromellitic imide moieties at 660 and 720 nm could be assumed as the main active species in the photoconductivity of the polyimide film by utilizing the model system, DBPMDI. These active species, such as radical cation or radical anions, are expected to be the free carriers of electrons or holes under the applied electric field, and transport of these charge carriers of electrons or holes would result in the photoconductivity of the polyimide films by a hopping mechanism.

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