Thermally Stable Nonlinear Optical Polyimide Functionalized by *N,N*-Diallylamino-Substituted Chromophore

WEI WU,[1] DONG WANG,[1] PENG WANG,[2] PEIWANG ZHU,[2] CHENG YE[2]

[1] Center for Molecular Science, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, People's Republic of China

[2] Organic Solids Lab, Center for Molecular Science, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, People's Republic of China

Received 21 September 1999; accepted 8 November 1999

ABSTRACT: Thermally stable nonlinear optical (NLO) polyimide prepolymers with different chromophore-loading levels were prepared through an addition reaction of bismaleimide **1** and *N,N*-diallylamino-substituted chromophore **2.** The prepolymers were used to prepare NLO polyimide in a simultaneously poling/curing process. These prepolymers exhibited excellent film-forming quality and good solubility in low boiling-point solvents. Kinetic parameters (n, ln A, Ea) of their solidification reaction were determined by the dynamic method. Thermal and NLO properties of the cured polyimides with various chromophore-loading levels were studied. With increase of the chromophore-loading level, the glass transition temperature (T_g) and the thermal stability of the polyimides decreased, but the second-order nonlinearity of the poled film increased. The largest d_{33} coefficient obtained was 76.2 pm/V at 1064 nm. The SHG signal was rather stable up to 120°C. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 77: 2939–2947, 2000

Key words: *N,N*-diallylamino chromophore; bismaleimide; prepolymer; poling/curing process; second-order nonlinearity; thermal stability; SHG signal

INTRODUCTION

Extensive studies have recently been focused on a poled polymer for nonlinear optical (NLO) materials since these materials are promising candidates for fabrication of high-performance electrooptical devices.[1] However, most NLO polymers suffer from the decay of electric field-induced second-order optical nonlinearity, arising from the relaxation of the induced noncentrosymmetric alignment of chromophore molecules to a random configuration. Various approaches have been used to stabilize the poling-induced order.[2–5] Significant improvement in the design and synthesis of efficient NLO polymer films have been achieved by covalently linking the NLO chromophore to the backbones of high T_g glassy polymers[6–10] as a side chain or a structural unit of the main chain.

To date, the majority of the high T_g polymers studied in poled polymer systems centers on condensation-type polyimides.[11–15] Although the high T_g of a cured polyimide is desired for the

---

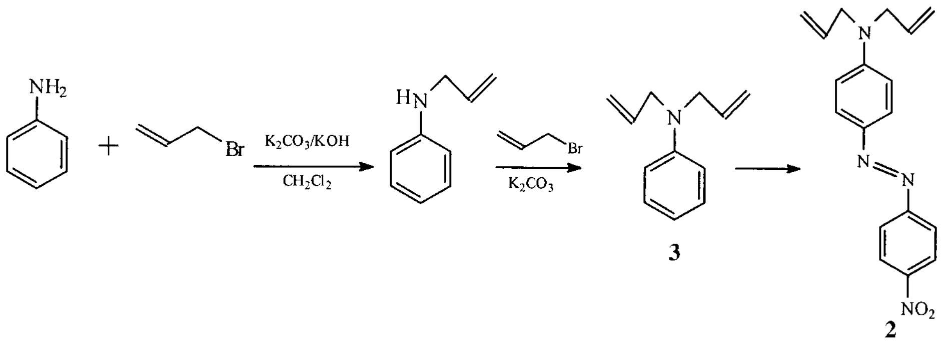
Correspondence to: D. Wang.

Contract grant sponsor: NSFC of China; contract grant number: 59790050.

Contract grant sponsor: The National Advanced Material Committee of China; contract grant number: 863-715-002-0120.

Contract grant sponsor: The Foundation of the Director of the Institute of Chemistry (CAS).

Journal of Applied Polymer Science, Vol. 77, 2939–2947 (2000)



Scheme 1

thermal stability of the poling-induced order, very high poling temperature was required for cured polyimide, thus resulting in unsatisfactory processibility and low poling efficiency due to the stiffness of the main chain of the polyimide molecule. To circumvent this problem, a poly(amic acid) (PAA) solution, which is the initial condensation product of the reaction between an aromatic dianhydride and a diamino chromophore in an aprotic solvent, is usually used to cast films. But another problem will be encountered when the PAA films are poled and cured simultaneously, that is, the water released during the imidization step can increase the conductivity of the film and, hence, then decrease the poling efficiency.[16] In contrast, addition-type polyimide can be cured without evolution of volatile by-products. Furthermore, addition-type polyimide would exhibit the densification effect at lower temperature owing to the crosslink structure of the polymer matrix. These features of addition-type polyimide offer great promise for the fabrication of NLO materials. Among addition-type polyimides, bismaleimide–diamine addition-type polyimide has been extensively studied.[17]

Recently, we developed another addition-type NLO polyimides. *N,N*-Diallylamino-substituted chromophore (DAAC) is utilized as a monomer to copolymerize with bismaleimide (BMI) monomers via a polyaddition reaction of double bonds to obtain the desired prepolymers. The prepolymers have good film-forming quality and withstand the subsequent poling/curing process. Also, they possess certain end-capped functional groups, so that they can be thermally cured into polyimides. During the *in situ* simultaneously poling/curing process, the induced noncentrosymmetric alignment of chromophores is stabilized by the formation of a crosslink structure. The motion of the chromophore moiety in the polymer matrix can be effectively restricted. Such a type of DAAC-functionalized addition-type polyimides could lead to a processable NLO material with good thermal properties and optical characteristics.

EXPERIMENTAL

Starting Materials

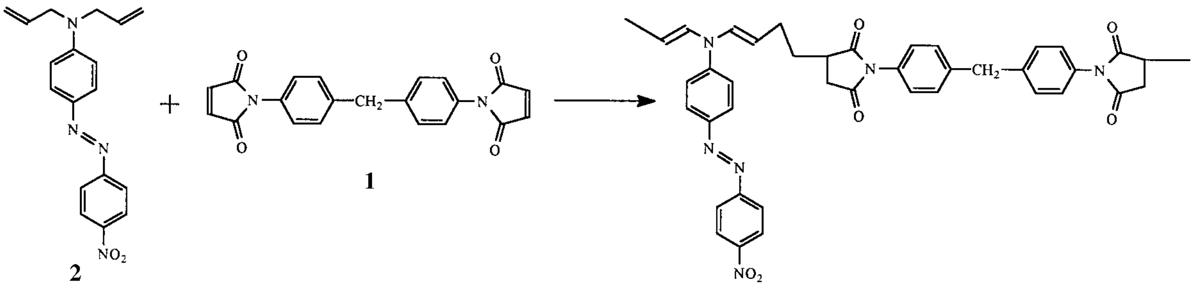
N,N′-Bismaleimido-4,4′-diphenylmethane **1** was selected as the BMI monomer, which is commercially available and purified by recrystallization from methanol/chloroform, followed by chromatography on silica gel. Aniline was distilled over calcium hydride before use. Allyl bromide (J & K Chemica, Beijing) was used as received. All the solvents used were purified by normal methods.

Synthesis of Chromophore (2) (Scheme 1)

N,N-*Diallylaniline* 3 *(ref. 18)*

Fresh aniline (9.3 g, 0.1 mol) and allyl bromide (12.1 g, 0.1 mol) were dissolved in dichloromethane (100 mL) at room temperature. To this solution were added anhydrous potassium carbonate (41.4 g, 0.3 mol) and a small amount of potassium hydroxide. The mixture was stirred at 40°C for 4 h. After cooling, the solid was filtered out. Then, to the filtrate were added additional allyl bromide (12.1 g, 0.1 mol) and potassium carbonate (41.4 g, 0.3 mol). The resulting suspension was refluxed for 4 h. After filtration, and removal of the solvent, the crude product was purified by distillation to give *N,N*-diallyl aniline **3** (12.3 g, 71.6%), bp 96–98°C/5 mmHg.

^{1}H-NMR (200 MHz, $CDCl_3$) δ 7.19 (m, 2H), 6.69 (m, 3H), 5.85 (m, 2H), 5.15 (m, 4H), 3.91 (d, 4H). ^{13}C-NMR ($CDCl_3$) δ 146.6, 133.9, 129.0, 116.3, 116.0, 112.4, 52.7.



Scheme 2

4-(N,N-Diallylamino)-4′-nitro-azobenzene 2

To a stirred ice-cooled suspension of *p*-nitroaniline (2.76 g, 0.02 mol) in 12 mL of 50% aqueous hydrochloride were added 80 g of crushed ice and sodium nitrite (1.6 g, 0.023 mol). The solution became homogeneous after a few minutes and then was added dropwise to the solution of **3** (3.46 g, 0.02 mol) in 20 mL of ethanol at 3–5°C. The resulting mixture was stirred for 1 h at 3–5°C and 1 h at room temperature. The precipitate was filtered and washed with cold water, then dried under a vacuum. Recrystallization from ethanol gave a red solid, **2** 5.92 g (92%); mp 100°C.

IR (KBr, cm^{-1}) ν 3370, 2904, 1600, 1516, 1388, 1341, 1239, 1138, 1105, 926, 860. ^{1}H-NMR (200 MHz, $CDCl_3$) δ 8.33 (d, 2H, J = 9 Hz), 7.92 (d, 2H, J = 9 Hz), 7.88 (d, 2H, J = 9 Hz), 6.77 (d, 2H, J = 9 Hz), 5.88 (m, 2H), 5.21 (m, 4H), 4.05 (d, 4H). ^{13}C-NMR ($CDCl_3$) δ 156.6, 152.4, 147.4, 143.7, 132.3, 126.2, 124.6, 122.5, 116.5, 111.9, 52.9.

ANAL. Calcd for $C_{18}H_{18}N_4O_2$: C, 67.06%; H, 5.63%; N, 17.38%. Found: C, 67.03%; H, 5.92%; N, 17.45%.

Prepolymers (Scheme 2)

BMI–DAAC prepolymers were prepared by the following procedure: DAAC **2** was charged into a 50-mL three-necked flask fitted with a paddle stirrer and nitrogen inlet; the flask was maintained in a oil bath. After **2** was melted into a deep red liquid, **1** was added under nitrogen. Then, the mixture was stirred at 110–120°C for 10 min. The resulting red liquid was poured into a shallow disk under nitrogen; after cooling to room temperature, the formed solid was ground to get a red powder. The prepolymers prepared with molar ratios of **1** to **2** (1 : 1, 2 : 1, and 3 : 1) were denoted by *Samples* **1, 2,** and **3,** respectively.

Film Casting

The 15% solutions of the prepolymer *Samples* **1–3** in tetrahydrofuran (THF) were cast onto glass slides. The solvent was evaporated in a convection oven at 60°C overnight. Then, the samples were thermally treated by gradually increasing the temperature by the following program: 150°C for 2 h, 200°C for 2 h, and 250°C for 2 h. After heating, the films, *Samples* **1a–3a,** corresponding to prepolymers *Samples* **1–3,** respectively, were removed from the glass slides by soaking them in cold water, then dried in a vacuum. The thickness of the cured films was about 1–2 μm.

To prepare thin films for *in situ* second harmonic generation (SHG) measurements, the THF solutions of the prepolymer *Samples* **1–3** were filtered through a 0.2-μm syringe filter and then spun-cast onto glass slides. The films were dried in an oven at 60°C for 2 days in a vacuum, giving *Samples* **1b–3b.** The thickness of the films was about 0.5–1 μm.

Measurements

The ^{1}H- and ^{13}C-NMR spectra were collected on a Varian XL-200 NMR spectrometer. The FTIR spectra were recorded on a PE FTIR 1760X spectrometer. Differential scanning colorimetry (DSC) and thermogravimetric analysis (TGA) were performed using a PE-7 series thermal analysis system. The second-order NLO coefficient d_{33} of the film samples was measured by the SHG technique at 1064 nm, with a Y-cut quartz crystal as the reference. The instrument was equipped with a heating and corona-poling apparatus to allow the *in situ* measurements of the SHG intensity at different temperatures during the poling process. The poling voltage was 7 kV at the needle point. The poling and thermal curing of the film *Samples* **1b–3b** were simultaneously carried out with a tip-to-plane distance of 1.0 cm in multistepped increasing temperatures from room temperature to 210°C.

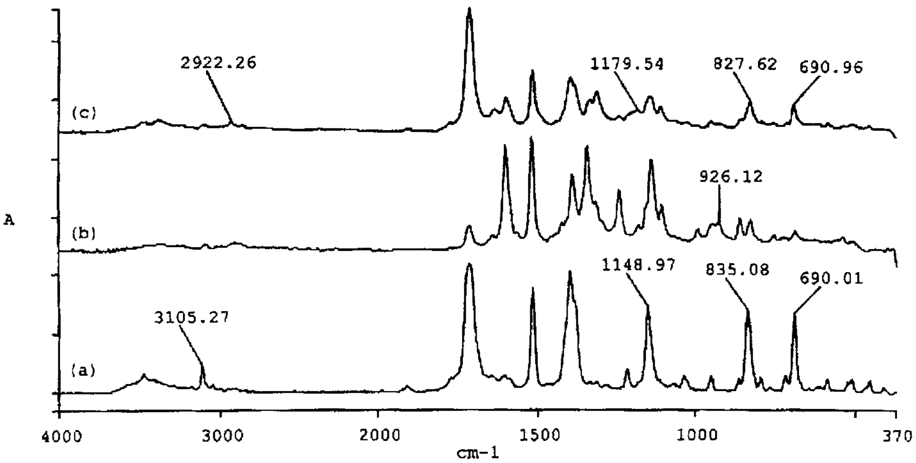


Figure 1 FTIR spectra of the monomers and prepolymer (1 : 1): (a) monomer 1; (b) monomer 2; (c) prepolymer.

RESULTS AND DISCUSSION

Prepolymers

DAAC **2** is structurally similar to Disperse Red 1 (DR 1) but with a *N,N*-diallyl moiety. The double bond of the allyl moiety can be covalently incorporated into the polymer backbone through an addition reaction. The first hyperpolarizability ($\beta_0 = 51 \times 10^{-48}$ esu) and the ground-state dipole moment ($\mu_g = 9.6$ D) of **2** were estimated by a semiempirical computational calculation AM1 program with a finite-field (FF) method.[19] Soluble prepolymers were prepared by a melt reaction of **1** with **2.** The prepolymers exhibited good solubility in low boiling-point solvents, such as acetone and THF and had an excellent film-forming quality.

As indicated by Phelan and Sung,[20] the reaction of BMI with diallylbisphenol A is very complicated, in which ene, Diels–Alder, homopolymerization, rearomatization, and alternating copolymerization are involved. In all these transformations, maleimide moieties are converted to succinimide groups. In the reaction of **1** with **2,** such conversions of the maleimide moieties to the succinimide group were also observed. The FTIR spectra of **1, 2,** and *Sample* 1 (1 : 1 molar ratios of two monomers) are shown in Figure 1. Compared with the FTIR spectrum of **1,** several distinct changes occur in the spectrum of the prepolymer *Sample* **1.** The intensities of the band at 1150 cm^{-1} associated with $\nu_{C—N—C}$ of the maleimide ring and the band at 830 cm^{-1} (strong), due to the out-of-plane hydrogen deformation of a double band of the maleimide ring, decrease. On the other hand, the band at 1180 cm^{-1}, attributed to $\nu_{C—N—C}$ of the succinimide ring, occurs. The relative ratio of the band intensities of 1150 and 1180 cm^{-1} could be used for monitoring the polyaddition reaction process. In comparison with the FTIR spectrum of **2,** the band at 940 cm^{-1} in the spectrum of the prepolymer *Sample* **1,** due to the allyl group of **2,** decreases noticeably.

The ^{1}H-NMR data of the prepolymer *Sample* **1** include a quartet at 8.2 and 7.9 ppm (AA′BB′) assigned to the phenyl protons of the *p*-nitrophenyl moiety; a quartet at 7.8 and 6.8 ppm (AA′BB′), to the phenyl protons of the 4-substituted aniline moiety; and a multiplet at 7.3 ppm, to the phenyl protons of the BMI moiety. Absorption at 7.2 ppm can be assigned to the cyclic olefin proton of the terminal maleimide ring. The signals at 5.9, 5.2, and 4.1 ppm are due to the olefin protons of the terminal *N,N*-diallyl group. The signals at upfield are due to the protons of the alkyl units formed in the prepolymerization reaction.

From these results, it is concluded that the BMI monomer **1** and the chromophore **2** are able to carry out a polyaddition reaction under melt-reaction conditions. The produced prepolymers have not only good film-forming quality, but also terminal olefin bonds (either maleimide or allylic moieties) for subsequent chain propagation and/or crosslink reaction under thermally curing conditions.

Curing Behavior

Thermal scans of the prepolymers *Samples* **1–3** (Fig. 2) were performed using a DSC instrument.

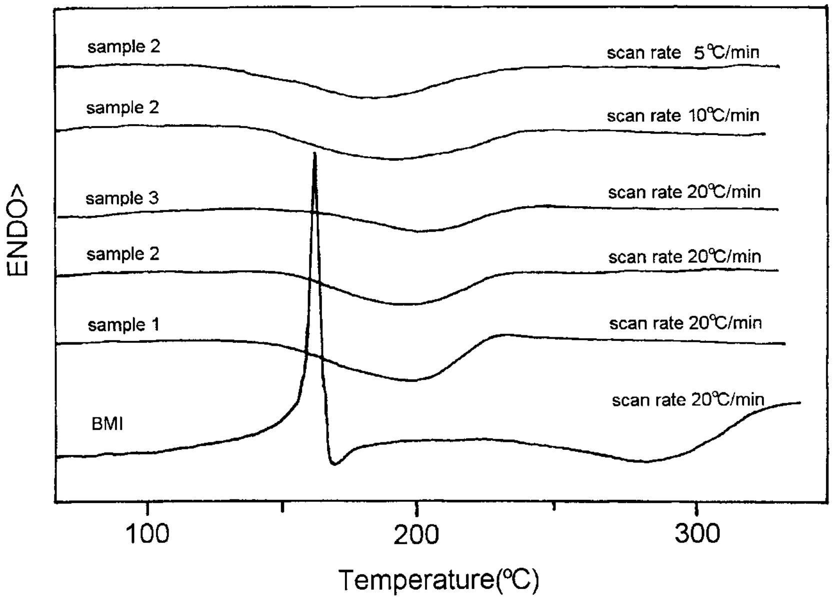


Figure 2 DSC traces of BMI monomer **1** and prepolymers.

The DSC scan of **1** is also presented for reference, which shows an exothermic peak corresponding to the homopolymerization superimposed on the melting endotherm. In most cases, the melt endotherm and polymerization exotherm are unable to be resolved. Homopolymerization of BMI (**1**) appears to be complete by about 170°C. A second much more diffuse exotherm starts at about 220°C and is continuous up to 330°C, which was due to a further crosslinking reaction. Therefore, a higher curing temperature is necessary to fulfill the crosslink reaction. For all the prepolymers, *Samples* **1–3,** there is no melting endotherm or exotherm at lower temperatures, but a broad exotherm at the range of 150–225°C corresponding to a crosslinking reaction, which is much lower than the curing temperature of the BMI monomer **1.** It is clear from these results that the introduction of the chromophore moiety to the molecular backbone of the prepolymer could decrease the curing temperature required for fulfilling the crosslink reaction, leading to improved processibility to some extent. Evidently, the chromophore plays a role as an effective modifier to reduce the curing temperature. The influence of the chromophore-loading level in prepolymer molecules on the curing behavior of the prepolymers was observed. With increase of the chromophore loading level, the maximum endotherm peak temperature decreased but the enthalpy (ΔH) increased from 117 to 159 and then to 234 kJ/mol for *Samples* **3, 2,** and **1,** respectively (Table I).

The FTIR spectra of the prepolymer *Sample* **2,** treated at different temperatures, are shown in Figure 3. When the sample was treated at 150°C for 2 h, the band at 1180 cm^{-1} associated with the succinimide ring became very noticeable, while the band at 1139 cm^{-1} due to the maleimide rings just exhibited a small shoulder, indicating that chain propagation/crosslink reactions proceeded with the formation of succinimide groups. After the sample was further treated at 200°C for 2 h, the bands at 1139, 830, and 690 cm^{-1} decreased markedly. At the subsequent curing step (up to 250°C), the increase of the band intensity at 1180 cm^{-1} and the disappearance of the bands at 934 and 1139 cm^{-1} demonstrated that, after the programmed heat treatment, the olefin groups in the prepolymer molecules were consumed nearly completely.

Cure Reaction Kinetics

A kinetic study can provide a valuable reference for optimization of the curing/poling process and for a better understanding of structure–property relationship in these crosslink materials. A few attempts to study the kinetics of the cure reaction for the bismaleimide homopolymer or copolymers with diamine monomers were reported.[21,22] Due to the complex nature of thermosetting reactions, a phenomenological method based on the equation below is usually used:

Table I Results of DSC and TGA Analysis of Prepolymers and Polyimides

Samples	ΔH (kJ/mol)[b]	Kinetic Parameters: $\ln A$(s^{-1})[c]	Kinetic Parameters: Ea(kJ/mol)[c]	n	T_g[a] (°C)	T_d[a] (°C)	Y_c[a] (%)	d_{33} (pm/V)
1	234	22	109	0.8	205	394	60	76.2
2	159	2535	123.5	1.04	234	413	52	53.6
3	117	28.2	133.5	1.12	277	436	57	30.0

[a] Data for finally cured samples **1a–3a.**
[b] Extroplated value (to heating rate = 0°C).
[c] At heating rate = 20°C.

$$\beta\frac{d\alpha}{dt} = Kf(\alpha) \tag{1}$$

where β is the heating rate and K is a constant, depending on temperature in an Arrhenius form, and $f(\alpha)$ expresses a function of the conversion of reactive group, where α is the extent of the reaction in most cases:

$$f(\alpha) = (1 - \alpha)^n \tag{2}$$

The reaction rate eq. (3) can be deduced by combining eqs. (1) and (2):

$$\beta\frac{d\alpha}{dt} = Ae^{-Ea/RT}(1 - \alpha)^n \tag{3}$$

Based on this reaction rate eq. (3) and DSC dynamic experiments, the active energy Ea, preexponential factor A, and order parameter n for the cure reaction at different heating rates can be directly obtained by a PE-7 series thermal analysis system (Table I). It can be found from the DSC curves of the prepolymer (Fig. 2) that, with increase of the heating rate, the exotherm peak moves to a higher-temperature region. In addition, the activation energy Ea and the preexponential factor A also depend on the heating rate. So, the kinetic parameters were finally obtained as extrapolation values (to $\beta = 0$). As results, $Ea = 109–133$ kJ/mol and $\ln A = 22.0–28.2$ s^{-1}. Figure 4 includes plots of both the Ea and $\ln A$ versus β, which exhibit fairly good linearity with correlation coefficients of 0.995 and 0.998.

For given solid-state reactions with the same cure mechanism, a linear relationship between the logarithm of the preexponential factor A and the apparent activation energy Ea should meet

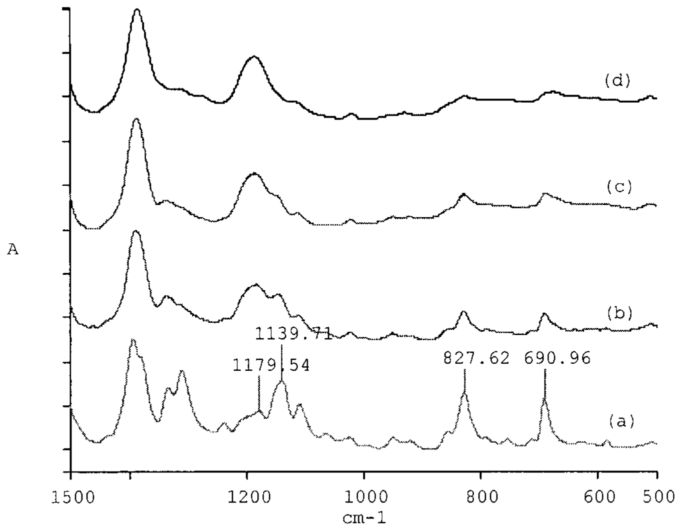


Figure 3 FTIR spectra of *Sample* **1** at different curing stages: (a) before heat treatment; (b) 150°C, 2 h; (c) 200°C, 2 h; (d) 250°C, 2 h.

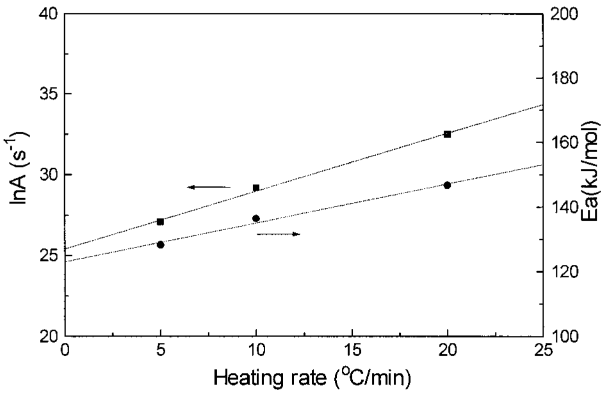


Figure 4 Dependencies of ln A and Ea on heating rate for *Sample* **2.**

the kinetic compensation effect[23] (KEC). In the case of prepolymer *Sample* **2,** the linear relationship is shown in Figure 5 with a linear correlation coefficient of 0.999.

Glass Transition Temperature and Thermal Stability of Cured Polyimides

The glass transition temperatures, T_g's, of polyimides *Samples* **1a–3a** were determined by DSC (Table I). With the increase of the chromophore-loading level, the T_g of the finally cured polyimide obviously decreased. Considering the higher T_g of the homopolymer of the BMI monomer **1** (390–400°C),[24] the lower T_g's of the BMI/DAAC copolymers are attributed to the introduction of flexible alkyl moieties of chromophore **2.** Although the introduction of the chromophore could reduce the curing temperature, the presence of flexible moieties in the molecules of the cured polyimide would be harmful to the stability of the poling-induced order. So, the chromophore-loading level should be optimized in the design and synthesis of the BMI/DAAC NLO materials for a balance of thermal and optical properties.

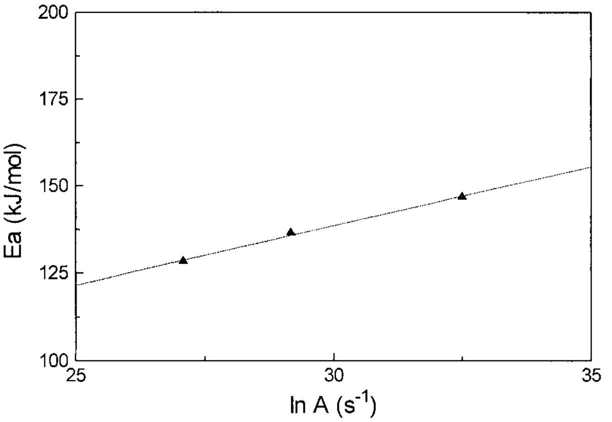


Figure 5 Plot of Ea versus ln A for prepolymer *Sample* **2.**

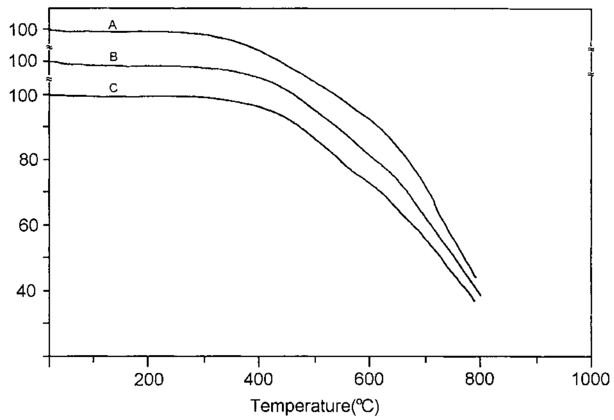


Figure 6 TGA traces of cured samples: (A) *Sample* **1;** (B) *Sample* **2;** (C) *Sample* **3.**

The thermal properties of cured polyimide *Samples* **1a–3a** were evaluated by TGA. Figure 6 shows the TGA curves and Table I summarizes the decomposition temperature (Td_1 and Td_2) and the char yield at 700°C (Yc_1). It can be found that cured polyimides are stable up to 300°C and then start losing weight. Unlike the TGA curves of homopolymer of **1,** these addition-type polyimides degrade through a two-step process: The first step occurs between 330 and 450°C with an ~ 10% weight loss. The second step begins at ~ 630°C and ~ 65% residue is obtained. It is worth noting that, with increase of the chromophore-loading level, the thermal stabilities of the cured polyimides turned out to be slightly worse.

NLO Properties of Polyimide

Since the addition-type polyimide *Samples* **1–3** can be cured without evolution of volatiles, effective poling can be performed. To achieve optimum poling efficiency, the films were poled and thermally cured simultaneously. This process was monitored by *in situ* SHG intensity measurements. The multistepped thermal history and the SHG intensity profiles are shown in Figure 7. For all three prepolymers (*Samples* **1–3**), the SHG signals began to appear at 150°C and then rose at a rather fast rate. After the films were poled at 150°C for about 1.5 h, their SHG intensities reached the maximum values. With increasing temperature to 180°C, the SHG intensities decreased markedly at first and then increased slowly in the subsequent homothermic process, but could reach maximum values again. It can be found that *Sample* **1b,** with the highest chromophore-loading level, exhibited the biggest signal drop between the two stages. Although the SHG intensity would decrease slightly in the further heat treatment, keeping the samples at higher temperature (such as 210°C) for a longer time is necessary for completing the crosslink reaction—hence, improving the orientation stability. If, during the process of cooling to room temperature, the electric field was employed on the samples throughout, the SHG signals were nearly unchanged.

The calculation of the second-order nonlinear coefficient d_{33} of the poled polymers is based on the method suggested by Meredith et al.[25] as follows:

$$\frac{d_{33,s}}{d_{11,q}} = \frac{\chi_s^{(2)}}{\chi_q^{(2)}} = \sqrt{\frac{I_s}{I_q}}\,\frac{l_{c,q}}{l_s}F \qquad (4)$$

where $d_{11,q}$ is d_{11} of quartz crystal, which is 0.45 pm/V; I_s and I_q, SHG intensities of the sample and quartz, respectively; $l_{c,q}$, the coherence length of quartz; l_s, the thickness of the sample; and F, a correction factor, which is equal to 1.2 approximately when $l_s \gg l_c$. Using eq. (4), the d_{33}'s of the three poled polymer films, *Samples* **1b–3b,** obtained are in the range of 30.0–76.2 pm/V at a 1064 nm fundamental wavelength (Table I).

The dynamic stability of the SHG signal was studied by heating the poled films of *Samples* **1b–3b** at a constant rate of 2°C/min over the temperature range of 20–200°C (Fig. 8), accompanying monitoring of the change of the SHG signals. For *Sample* **1b,** with the highest chromophore-loading level, the SHG signal was quite stable until the temperature reached 120°C. A fast decay of the signal was observed when the temperature was up to 150°C. Its half-decay temperature is close to 210°C. Meanwhile, with decrease of the chromophore-loading level, the orientation stability of the poled film was improved. In comparison with the results that the addition-type polyimide, based on BMI and diamine chromophore, has good thermal stability and moderate optical nonlinearity (r_{33} = 7.8 pm/V at λ = 632 nm),[26] the addition-type polyimides of BMI/DAAC provide great promise in the preparation of a good performance NLO material.

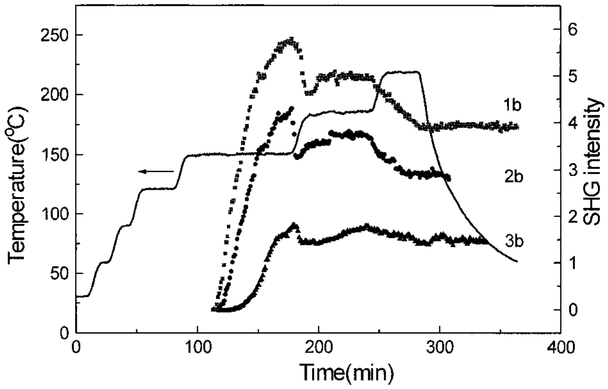


Figure 7 Profiles of SHG and temperature in the corona poling of the three samples.

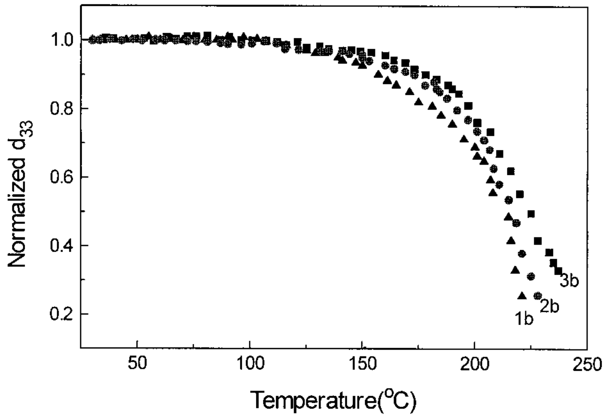


Figure 8 Decay plots of the normalized SHG coefficient d_{33}^t/d_{33}^0 as a function of temperature for *Samples* **1–3.**

The work was supported by the NSFC of China (59790050). The National Advanced Material Committee of China (863-715-002-0120), and The Foundation of the Director of the Institute of Chemistry, CAS.

REFERENCES

1. Eaton, D. F. Science 1991, 253, 281; Marks, T. J.; Ratner, M. A. Angrew Chem Int Ed Engl 1995, 34, 155, and references cited there.
2. Jen, A. K. Y.; Liu, Y. J.; Cai, Y.; Rao, V. P.; Dalton, L. R. J Chem Soc Chem Commun 1994, 2711.
3. Verbiest, T.; Samyn, C.; Van Beylen, M.; Persoons, A. Macromol Rapid Commun 1998, 19, 349.
4. Sakai, Y.; Haba, O.; Kato, S.; Ueda, M.; Fukuda, T.; Matsuda, H. J Photopolym Sci Technol 1998, 11, 217.

5. Jiang, H.; Kakkar, A. K. Macromolecules 1998, 31, 4170.
6. Mao, S. S. H.; Ra, Y.; Guo, L.; Zhang, C.; Dalton, L. R.; Chen, A.; Garner, S.; Steier, W. H. Chem Mater 1998, 10, 146.
7. Chen, T. A.; Jen, A. K. Y.; Cai, Y. J Am Chem Soc 1995, 117, 7295.
8. Verbiest, T.; Burland, D. M.; Jurich, M. C.; Lee, V. Y.; Miller, R. D.; Volksen, W. Macromolecules 1995, 28, 3005.
9. Marks, T. J.; Ratner, M. A. Angew Chem Int Ed Engl 1995, 34, 155, and references therein.
10. Chen, T. A.; Jen, A. K. Y.; Cai, Y. Macromolecules 1996, 29, 535.
11. Yu, D.; Gharavi, A.; Yu, L. P. Macromolecules 1995, 28, 784.
12. Yu, D.; Gharavi, A.; Yu, L. P. J Am Chem Soc 1995, 117, 11680.
13. Verbiest, T.; Burland, D. M.; Jurich, M. C.; Lee, V. Y.; Miller, R. D.; Volksen, W. Science 1995, 268, 1604.
14. Verbiest, T.; Burland, D. M.; Jurich, M. C.; Lee, V. Y.; Miller, R. D.; Volksen, W. Macromolecules 1995, 28, 3005.
15. Peng, Z. H.; Yu, L. P. Macromolecules 1994, 27, 2638.
16. Wu, W. J.; Valley, J. F.; Ermer, S.; Binkley; Kenney, J. T.; Lipscomb, G. F.; Lytel, R. Appl Phys Lett 1991, 58, 225.
17. Bell, V. L.; Young, P. R. J Polym Sci Polym Chem Ed 1986, 24, 2649; Tangare, A. V.; Martin, G. C. J Appl Polym Sci 1992, 46, 1125; Grennier-Loustalot, M. F.; Gouarders, F.; Joubert, F.; Grenier, P. Polymer 1993, 34, 3848; Wu, W.; Wang, D.; Ye, C. J Appl Polym Sci 1998, 70, 2471.
18. Carnahan, F. L.; Hurd, C. D. J Am Chem Soc 1930, 52, 4586.
19. Frich, M. J.; Trucks, G. W.; Head-Gordon, M. Gaussian-92; Gaussian Inc.: Pittsburgh, PA, 1992.
20. Phelan, J. C.; Sung, C. S. P. Macromolecules 1997, 30, 6837; Phelan, J. C.; Sung, C. S. P. Macromolecules 1997, 30, 6845.
21. Ciulio, C. D.; Gautiier, M.; Jasse, B. J Appl Polym Sci 1984, 29, 1771.
22. Acevedo, M.; Abajo, J. D.; Campa, J. G. Polymer 1990, 31, 1955.
23. Varma, I. K.; Sangita; Varma, D. S. J Polym Sci Polym Chem Ed 1984, 22, 1419.
24. Koga, N.; Tanaka, H. J Therm Anal 1991, 37, 347.
25. Meredith, G. R.; VanDusen, J. G.; Williams, D. J. Macromolecules 1982, 15, 1385.
26. Wang, J. F.; Marks, T. J.; Liu, W. P.; Zhou, H. T.; Wong, G. K. Polym Prepr 1995, 36, 308.