Synthesis and Properties of Novel Polyimides from 3-(4-Aminophenylthio)-N-aminophthalimide

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ABSTRACT: A novel, asymmetric diamine, 3-(4-amino-phenylthio)-N-aminophthalimide, was prepared from 3-chloro-N-aminophthalimide and 4-aminobenzenethiol. The structure of the diamine was determined via IR and ^{1}H-NMR spectroscopy and elemental analysis. A series of polyimides were synthesized from 3-(4-aminophenylthio)-N-aminophthalimide and aromatic dianhydrides by a conventional two-step method in N,N-dimethylacetamide and by a one-step method in phenols. These polyimides showed good solubility in 1-methyl-2-pyrrolidinone, m-cresol, and p-chlorophenol, except polyimide from pyromellitic dianhydride, which was only soluble in p-chlorophenol. The 5% weight loss temperatures of these polyimides ranged from 460 to 498°C in air. Dynamic mechanical thermal analysis indicated that the glass-transition temperatures of the polyimides were in the range 278–395°C. The tensile strengths at break, moduli, and elongations of these polyimides were 146–178 MPa, 1.95–2.58 GPa, and 9.1–13.3%, respectively. Compared with corresponding polyimides from 4,4'-diamiodiphenyl ether, these polymers showed enhanced solubility and higher glass-transition temperatures. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 706–711, 2008

Key words: polyimides; structure-property relations; synthesis

INTRODUCTION

Wholly aromatic polyimides, as a class of high-performance polymers, have unique thermal, mechanical, and electrical properties as well as outstanding chemical resistances. Therefore, they have been used widely as films, wire enamels, coatings, molding resins, and matrix resins in composites in the aerospace, transportation, electrical, and electronics industries. However, because of their chain rigidity and strong interchain interaction, most polyimides are insoluble in common organic solvents and intractable in their imide forms, which limit their applications.[1–3] To overcome these shortcomings, different structural modifications have been made to improve the processability of polyimides while maintaining their other excellent properties. Typical approaches include the introduction of flexible or kinked links, bulky lateral substituents, asymmetric or alicyclic structures, and noncoplanar or cardo groups into the polymer backbone.[4–6]

Hydrazine is the simplest diamine,[7,8] but only a few reports on hydrazine-based polyimides have appeared in the literature. Dine-Hart[9] described the synthesis and properties of hydrazine-based polyimides and their model compounds from N,N'-diaminonaphthalene-1,4,5,8-tetracarboxydi-imide and N,N'-diamino pyromellitimide; however, the molecular weights of the resulting polymers were very low.[9] Hay and coworkers[10–16] obtained high-molecular-weight polyimides from six-membered bis(N-aminoimide)s and aromatic dianhydrides. These polyimides had very high glass-transition temperature (T_g) values, excellent thermooxidative stability, and good solubility in organic solvents. Most of the studies mentioned previously centered on polyimides based on six-membered bis(N-aminoimide)s because they are easy to synthesize and are more stable. Moreover, a one-step polymerization in phenols is the only method for obtaining high-molecular-weight polyimides because of the lower reactivity and solubility of bis(N-aminoimide)s. In this article, we present the synthesis and properties of a series of polyimides derived from an asymmetric diamine, 3-(4-aminophenylthio)-N-aminophthalimide, which has enough activity and solubility for a conventional two-step polymerization. These polyimides, with

five-membered ring *N,N'*-biphthalimide segments, had very high T_g values and enhanced solubility.

EXPERIMENTAL

Materials

3-Chlorophthalic anhydride (97%) and 1,4-bis(3,4-dicarboxyphenoxyl)benzene dianhydride (HQPDA) were prepared in our laboratory. 4-Amino-benzenethiol, pyromellitic dianhydride (PMDA), 3,3′,4,4′-biphenyltetracarboxylic acid dianhydride (BPDA), 3,3′,4,4′-benzophenonetetracarboxylic acid dianhydride (BTDA), 3,3′,4,4′-oxy(diphthalicanhydride) (ODPA), and hydrazine hydrate (80)% were purchased from Aldrich Chemical Co. (St. Louis, MO). All other reagents were purchased from Shanghai Chemical Reagent Plant (Shanghai, China). PMDA, BPDA, BTDA, and ODPA were purified by sublimation *in vacuo*. *N,N*-Dimethylacetamide (DMAc) was dried over phosphorus pentoxide, distilled under reduced pressure, and then stored over 4-Å molecular sieves. *m*-Cresol and *p*-chlorophenol were used after distillation. 3-Chlorophthalimide was prepared according to a method in the literature.[17] Other reagents were analytical grade and were used as received.

Characterization

Fourier transform infrared (FTIR) spectra were determined with a Bio-Rad Digilab Division FTS-80 spectrometer (Hercules, CA). ^{1}H-NMR spectra were recorded on a Varian Unity spectrometer (Palo Alto, CA) at 400 Hz with tetramethylsilane as an internal standard. Elemental analyses were performed on an elemental analyzer MOD-1106 (Milan, Italy). Melting points were determined on an XT-4 melting point apparatus (Beijing Taike Apparatus, Inc., Beijing, China) and were uncorrected. Inherent viscosities were determined at 30°C with an Ubbelohde viscometer (Shanghai, China), and the concentration was 0.5 g/dL in *p*-chlorophenol. Thermogravimetric analyses (TGAs) were obtained at a heating rate of 10°C/min in air with a Perkin-Elmer TGA-2 thermogravimetric analyzer (Waltham, MA). Dynamic mechanical thermal analysis (DMTA) was performed on a dynamic mechanical thermal analyzer (Rheometric Scientific, Inc., Piscataway, NJ) with film samples (length = 10 mm) in a tensile mode (strain = 0.1%, initial static force = 0.2 N, static > dynamic force by 10.0%, minimum static force = 0.01 N, maximum autotension displacement = 3.0 mm) at a heating rate of 3°C/min and a frequency of 1 Hz from room temperature to 300°C. Wide-angle X-ray diffraction (WAXD) measurements were undertaken on a Rigaku max 2500

V PC X-ray diffractometer (Tokyo, Japan) with Cu Kα radiation (40 kV, 200 mA) with a scanning rate of 2°C/min from 2 to 50°C. The tensile measurements were carried out on an Instron model 1122 instrument (Norwood, MA) at room temperature.

Monomer synthesis

3-Chloro-*N*-aminophthalimide

3-Chloro-*N*-aminophthalimide was prepared according to the procedure in the literature.[8]

mp = 194–195°C (194–195°C[8]). IR (KBr): 3341 (asymmetric N—H stretching), 3203 (symmetric N—H stretching), 1779 (asymmetric C=O stretching), 1727 (symmetric C=O stretching), 1403 (C—N stretching), 1100 (N—N stretching), 730 (C=O bending). ^{1}H-NMR [400 MHz, dimethyl sulfoxide (DMSO), δ, ppm]: 7.85–7.79 (m, 3H), 4.95 (s, 2H).

ANAL. Calcd for $C_8H_5ClN_2O_2$: C, 48.88%; H, 2.56%; Cl, 18.03%; N, 14.25%. Found: C, 48.80%; H, 2.59%; Cl, 17.99%; N, 14.12%.

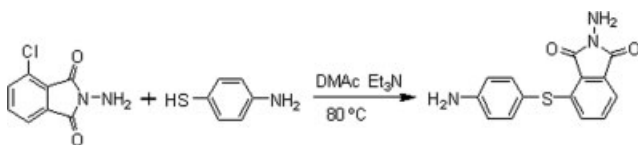#### 3-(4-Aminophenylthio)-*N*-aminophthalimide

3-Chloro-*N*-aminophthalimide (19.66 g, 0.10 mol), 4-amino-benzenethiol (12.50 g, 0.10 mol), and triethylamine (11.31 g, 0.11 mol) were placed in a 250-mL, three-necked, round-bottom flask containing 150 mL of DMAc. The mixture was heated to 90°C and stirred for 12 h in a nitrogen atmosphere. After it was cooled to room temperature, the resulting yellow mixture was poured into 1 L of water. The resulting yellow precipitate was filtered and washed with dilute ammonium (0.25 mol/L) three times and was then washed with water. After recrystallization from acetonitrile, a light-yellow needle crystal (19.38 g, yield = 68%) was obtained.

mp = 231–233°C. IR (KBr): 3405 (asymmetric N—H stretching), 3333 (symmetric N—H stretching), 1777 (asymmetric C=O stretching), 1717 (symmetric C=O stretching), 1399 (C—N stretching), 1100 (N—N stretching), 733 (C=O bending). ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 7.57–7.54 (t, 1H), 7.49–7.48 (d, 1H), 7.23–7.21 (dt, 2H), 6.86–6.84 (d, 1H), 6.70–6.68 (dt, 2H), 5.70 (s, 2H), 4.91 (s, 2H).

ANAL. Calcd for $C_{14}H_{11}N_3O_2S$: C, 58.93%; H, 3.89%; N, 14.73%; S, 11.24%. Found: C, 58.97%; H, 3.80%; N, 14.79%; S, 11.32%.

Polymerization

The polyimides were synthesized by two different methods: a two-step method in DMAc and a one-step method in phenolic solvents. An equimolar amount of dianhydride and diamine monomers were used in all cases. The representative polymerization procedures are described in the following text.

Scheme 1 Synthesis route of the monomer.

Two-step method in DMAc

3-(4-Aminophenylthio)-N-aminophthalimide (0.5706 g, 0.002 mol) and DMAC (5 mL) were charged into a 50-mL, three-necked flask equipped with a mechanical stirrer. After the diamine was dissolved, PMDA (0.4362 g, 0.002 mol) was added in one pot. The mixture was stirred at room temperature for 72 h to afford a viscous solution of poly(amic acid) (PAA). The PAA solution was cast onto a glass plate and dried at 80°C for 12 h; it was then heated at 150°C for 1 h, 200°C for 1 h, 250°C for 1 h, and 300°C for 1 h to afford the film.

One-step method in phenolic solvents

3-(4-Aminophenylthio)-N-aminophthalimide (0.5706 g, 0.002 mol), PMDA (0.4362 g, 0.002 mol), and p-chlorophenol (5 g) were placed in a 50-mL, three-necked flask equipped with a mechanical stirrer. The mixture was heated to 100°C for 1 h in a nitrogen atmosphere with stirring and was then heated at 190°C for 24 h. The water formed during imidization was removed with a slow stream of nitrogen passed through the solvent. The yellow viscous mixture was cooled and poured slowly into 100 mL of ethanol. The precipitate was collected by filtration and extracted with ethanol in a Soxhlet extractor for 24 h and dried *in vacuo* at 200°C for 4 h to afford the polyimide powder.

Yield = 93%. IR (KBr): 1781 [asymmetric C=O (two imide groups) stretching], 1742 [C=O (hydrazine-based imide group) stretching], 1717 [symmetric C=O (conventional imide group) stretching], 1370 [C—N (conventional imide group) stretching], 1350 [C—N (hydrazine-based imide group) stretching], 1080 (N—N stretching), 714 (C=O bending).

Other polymides

Polyimide from BPDA. Yield = 92%. IR (KBr): 1778 [asymmetric C=O (two imide groups) stretching], 1740 [C=O (hydrazine-based imide group) stretching], 1717 [symmetric C=O (conventional imide group) stretching], 1370 [C—N (conventional imide group) stretching], 1350 [C—N (hydrazine-based imide group) stretching], 1080 (N—N stretching), 715 (C=O bending).

Polyimide from BTDA. Yield = 93%. IR (KBr): 1783 [asymmetric C=O (two imide groups) stretching], 1742 [C=O (hydrazine-based imide group) stretching], 1719 [symmetric C=O (conventional imide group) stretching], 1671 [C=O (diaryl ketone) stretching], 1370 [C—N (conventional imide group) stretching], 1350 [C—N (hydrazine-based imide group) stretching], 1080 (N—N stretching), 713 (C=O bending).

Polyimide from ODPA. Yield = 95%. IR (KBr): 1782 [asymmetric C=O (two imide groups) stretching], 1742 [C=O (hydrazine-based imide group) stretching], 1718 [symmetric C=O (conventional imide group) stretching], 1370 [C—N (conventional imide group) stretching], 1350 [C—N (hydrazine-based imide group) stretching], 1080 (N—N stretching), 727 (C=O bending).

Polyimide from HQPDA. Yield = 92%. IR (KBr): 1780 [asymmetric C=O (two imide groups) stretching], 1743 [C=O (hydrazine-based imide group) stretching], 1718 [symmetric C=O (conventional imide group) stretching], 1370 [C—N (conventional imide group) stretching], 1350 [C—N (hydrazine-based imide group) stretching], 1080 (N—N stretching), 720 (C=O bending).
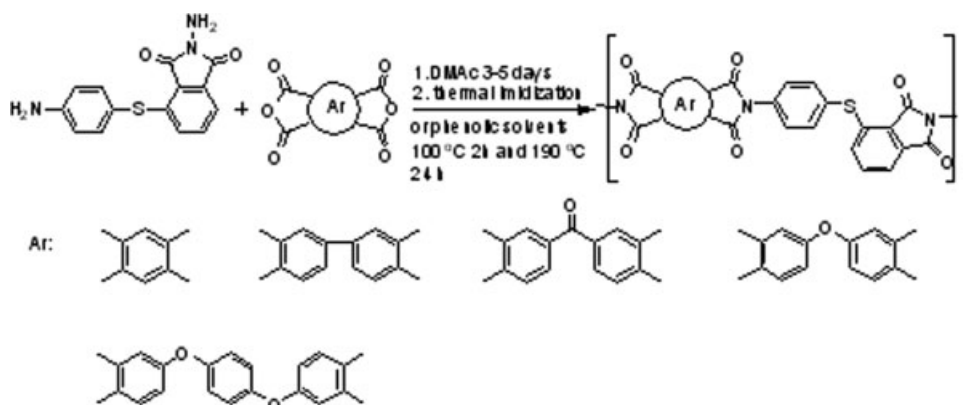
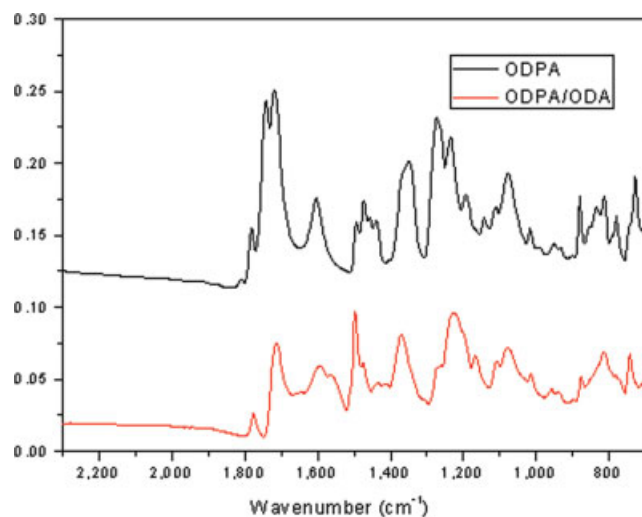**Scheme 2** Synthesis routes of the polyimides.

Figure 1 FTIR spectra of the polyimides from ODPA and 3-(4-aminophenylthio)-*N*-aminophthalimide. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

RESULTS AND DISCUSSION

Monomer synthesis

3-Chloro-*N*-aminophthalimide was prepared by a method reported in the literature.[8] 3-Chloro-*N*-aminophthalimide was converted into its six-membered ring isomer, 5-chlorophthalaz-1:4-dione, on exposure to a strong base. We tried to prepare 3-(4-aminophenoxyl)-*N*-aminophthalimide from the sodium of 4-aminophenol and 3-chloro-*N*-aminophthalimide but failed because of the poor stability of 3-chloro-*N*-aminophthalimide. When 4-amino-benzenethiol was used instead of 4-aminophenol, a weak organic base, triethylamine, completed the nucleophilic displacement smoothly without the isomerization of 3-chloro-*N*-aminophthalimide.[18,19] As shown in Scheme 1, 3-(4-aminophenylthio)-*N*-aminophthalimide was suc-

cessfully synthesized from 4-aminobenzenethiol and 3-chloro-*N*-aminophthalimide via nucleophilic displacement. The structure of 3-(4-aminophenylthio)-*N*-aminophthalimide was confirmed by IR spectra, ^{1}H-NMR spectra, and elemental analysis. The ^{1}H-NMR spectra exhibited the peaks assignable to the two different amino groups around 5.70 and 4.91 ppm. The difference in chemical shift meant that each amino group should have had a different reactivity. The elemental analysis data also confirmed the structure of the monomer.

Polymerization

As shown in Scheme 2, the polyimides were prepared by two methods: a two-step method in DMAc and a one-step method in phenolic solvents. Because the amino group attached to the imide group had a lower reactivity, the time in the two-step synthesis in DMAc should have been longer than usual, about 3–5 days according to the reactivity of the dianhydrides. When HQPDA was used, a high-molecular-weight polyimide could not be obtained via the two-step method. However, high-molecular-weight polymers could be prepared in all cases via the one-step method in phenolic solvents. When PMDA was used as the dianhydride, *p*-chlorophenol was necessary to prevent the formation of a precipitate, although *m*-cresol could maintain the homophase of the reaction mixture when the other dianhydrides were used. The inherent viscosities of the polymers were in the range 1.50–2.12 dL/g in *p*-chlorophenol at 30°C. These polymers could be cast into tough, flexible, and transparent films, which confirmed their high molecular weights. The structures of the polyimides were confirmed by IR spectra and elemental analysis. Figure 1 displays the representative IR spectra of the polyimides based on 3-(4-aminophenylthio)-*N*-aminophthalimide and the polyimides from ODPA/

TABLE I
Solubilities of the Polyimides in Different Solvents

Polymer	Inherent viscosity (dL/g)[a]	CHCl$_3$	TCE	THF	DMAc	DMSO	NMP	*m*-Cresol	*p*-Chlorophenol
PMDA (H)	1.50	−	−	−	−	−	−	−	+
PMDA (O)	1.80	−	−	−	−	−	−	−	+
BPDA (H)	1.29	−	−	−	−	−	±	+	+
BPDA (O)	1.42	−	−	−	−	±	+	+	+
BTDA (H)	1.38	−	−	−	−	±	+	+	+
BTDA (O)	1.72	−	−	−	±	+	+	+	+
ODPA (H)	1.80	−	−	−	−	−	±	+	+
ODPA (O)	2.12	−	−	−	−	±	+	+	+
HQPDA (O)	1.72	−	±	−	+	+	+	+	+

TCE = 1,1,2,2-terachloroethane; THF = tetrahydrofuran; H = polyimide films obtained by thermal imidization; O = polyimide films obtained in one-step in phenolic solvents; + = fully soluble at room temperature; ± = partially soluble on heating; − = insoluble on heating.
[a] Measured at 0.5 g/dL and 30°C in *p*-chlorophenol.

TABLE II
Thermal and Mechanical Properties of the Polyimides

Polymer	T_g (°C)[a]	$T_{5\%}$ (°C)[b]	Tensile strength (MPa)	Modulus (MPa)	Elongation (%)
PMDA	395 (399[1])	480	171	2497	10.3
BPDA	346 (285[1])	494	178	2585	11.5
BTDA	336 (306[21])	490	158	2266	9.1
ODPA	326 (272[24])	498	178	1958	13.3
HQPDA	278 (243[22])	460	146	1949	10.8

[a] Obtained from DMTA at a heating rate of 3°C/min at 1 Hz. The values in parentheses are for polymers based on ODA as reported in the literature.
[b] Obtained from TGA at a heating rate of 10°C/min in air.

4,4′-diamiodiphenyl ether (ODA) as a reference. The characteristic absorption bonds of hydrazine-based imide groups were different from those of the conventional imide groups, which was discussed in our earlier article.[20] The elemental analysis data agreed well with the proposed structures in general.

Properties of the polyimides

Solubility

The solubility of the polyimides is summarized in Table I. It must be pointed out that all solubility tests were carried out with high-molecular-weight polyimide films that had poorer solubility compared with the corresponding powders. Except that polyimides from PMDA could only be soluble in *p*-chlorophenol, the polyimides had good solubility in 1-methyl-2-pyrrolidinone (NMP), *m*-cresol, and *p*-chlorophenol. Polyimide films from thermal imidization showed poorer solubility than those formed from the one-step method. Compared with the corresponding polyimides derived from ODA, the solubility of the polymers was enhanced.[1,21,22] There were two possible reasons responsible for the enhanced solubility. The first was the presence of the *N,N*′-bisphthalimide groups in the main chain. Because of the steric repulsion of the C=O groups, two imide rings were twisted and forced into noncoplanar conformation,[23] which inhibited chain packing, broke up the conjunction along the backbone, and hindered the formation of intermolecular change-transfer complexes. The second, 3-(4-aminophenylthio)-*N*-aminophthalimide, was asymmetric and bent, which caused the polymer chains to become more disordered.

Thermal and mechanical properties

Table II illustrates the thermal and mechanical properties of the polyimides based on 3-(4-aminophenylthio)-*N*-aminophthalimide. All data were obtained from the transparent, flexible, and tough films that were cast from corresponding PAA solutions and cured at 150°C for 1 h, 200°C for 1 h, 250°C for 1 h, and 300°C for1 h, except the film from HQPDA,

which was cast from NMP solution obtained from the one-step synthesis. The 5% weight loss temperature ($T_{5\%}$) values were in the range 460–498°C in air. The TGA curves of the polymides from 3-(4-aminophenylthio)-*N*-aminophthalimide, as well as polymides based on ODPA and ODA, are shown in Figure 2. All polymers showed two weight-loss stages, which indicated that the two types of thermooxidative decomposition occurred in these polymers. We assumed that the first weight loss stage was the decomposition of aromatic sulfide segments, which were not as stable as the aromatic polyimide segments.

The tensile strengths at break, moduli, and elongations of the polyimides based on 3-(4-aminophenylthio)-*N*-aminophthalimide were 146–178 MPa, 1.95–2.58 GPa, and 9.1–13.3%, respectively. These values were similar or slight higher compared with those of ODA.[1,21,22,24]

The DMTA curves of the polymides from 3-(4-aminophenylthio)-*N*-aminophthalimide, as well as the polyimides based on ODPA and ODA, are
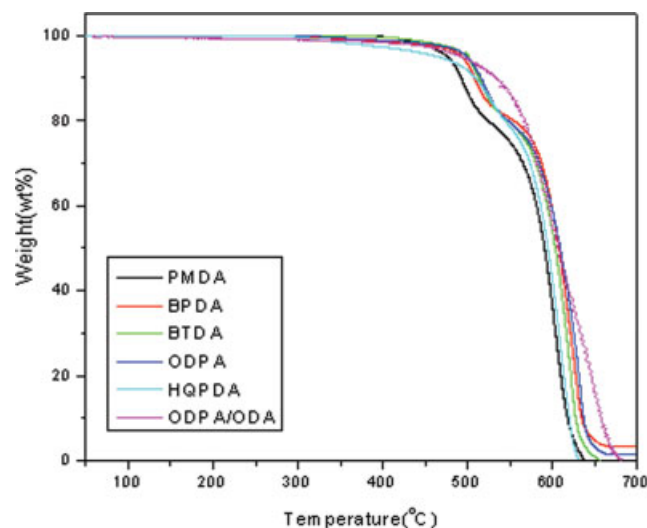

Figure 2 TGA curves of the polyimides from 3-(4-aminophenylthio)-*N*-aminophthalimide. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

shown in Figure 3. If the peak temperature in the tan δ curves is regarded as the T_g, the polyimide based on PMDA exhibited a T_g at 395°C, the polyamide based on BPDA exhibited a T_g at 346°C, the polyamide based on BTDA exhibited a T_g at 336°C, the polyamide based on ODPA exhibited a T_g at 326°C, and the polyamide based on HQPDA exhibited a T_g at 278°C. Compared with the corresponding polyimides derived from ODA, the T_g values of these polyimides were 30–60°C higher, except for that from the polyamide based on PMDA. The higher T_g values and excellent mechanical properties may have contributed to the rigid but rotation-restricted structure of N,N'-bisphthalimide and the higher imide contents.

X-ray diffraction

As shown in Figure 4, the crystallinity of the isomeric polyimides was examined by WAXD. All of the polymers showed amorphous patterns.

CONCLUSIONS

A novel diamine, 3-(4-aminophenylthio)-N-aminophthalimide, was prepared from 3-chloro-N-aminophthalimide and 4-aminobenzenethiol. A series of high-molecular-weight polyimides was synthesized from this novel diamine and aromatic dianhydrides. The asymmetric structure of the monomer and the introduction of a rigid but noncoplanar N,N'-bisphthalimide segment greatly affected the properties of the resulting polyimides. These polyimides showed enhanced solubility and were soluble in NMP, m-cresol, and p-chlorophenol, except for the polyimide from PMDA. The T_g values of the polyimides ranged from 278 to 395°C, which were 30–60°C higher compared with the responding polyimides based on
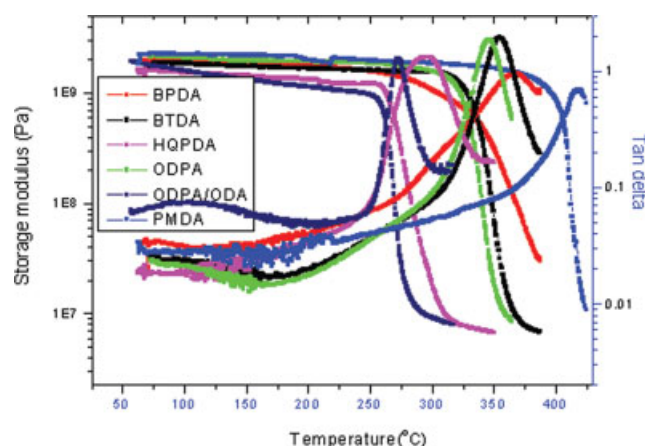

Figure 3 DMTA curves of the polyimides from 3-(4-aminophenylthio)-N-aminophthalimide. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 4 WAXD diagram of the polyimides from 3-(4-aminophenylthio)-N-aminophthalimide. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
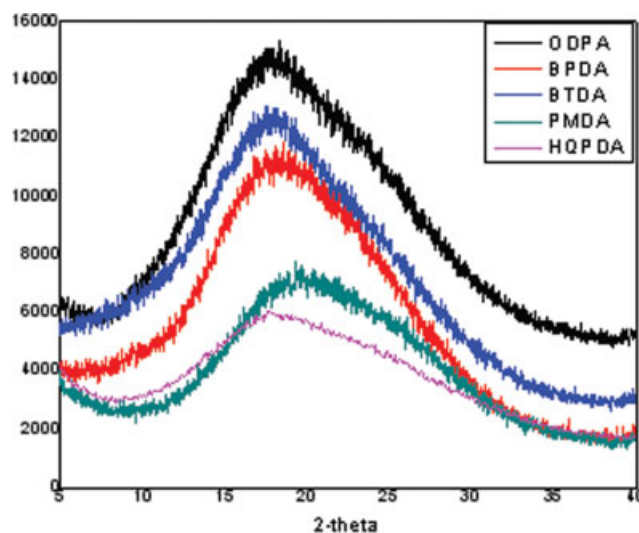
ODA. These polyimides also had excellent mechanical properties.

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