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POLYIMIDES**

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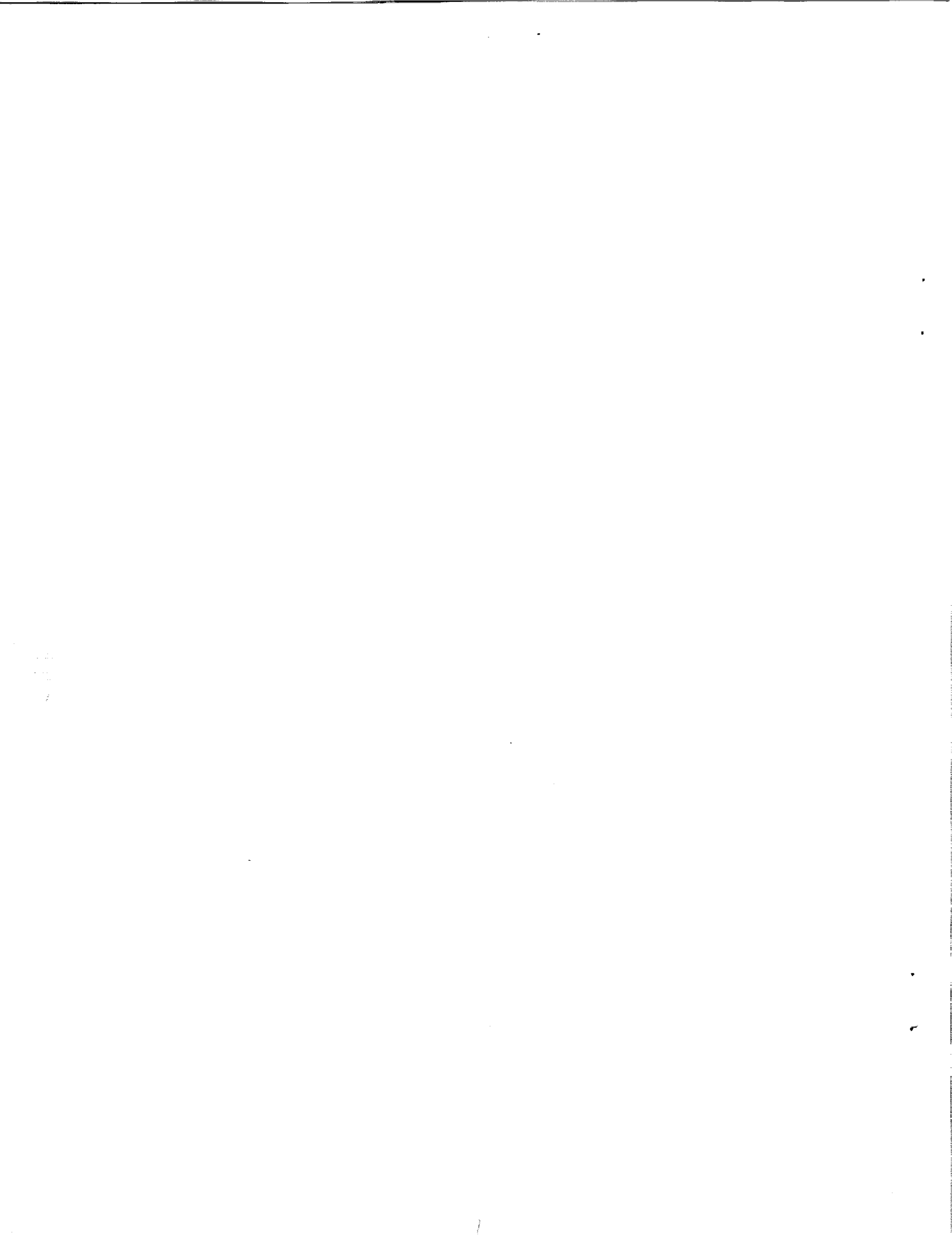
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INTRODUCTION

As part of an on-going effort to develop an understanding of how changes in the chemical structure affect polymer properties, an empirical study was performed on polyimides containing only ether and/or carbonyl connecting groups in the polymer backbone. During the past two decades the structure-property relationships in linear aromatic polyimides have been extensively investigated[1-5]. More recently, work has been performed to study the effect of isomeric attachment of keto-ether polyimides on properties such as glass transition temperature and solubility[6-7]. However, little work has been reported on the relation of polyimide structure to mechanical properties. The purpose of this study was to determine the effect of structural changes in the backbone of keto-ether polyimides on their mechanical properties, specifically, unoriented thin film tensile properties.

This study was conducted in two stages. The purpose of the initial stage was to examine the physical and mechanical properties of a representative group (four) of polyimide systems to determine the optimum solvent and cure cycle requirements. These optimum conditions were then utilized in the second stage to prepare films of keto-ether polyimides which were evaluated for mechanical and physical properties. All of the polyimides were prepared using isomers of oxydianiline (ODA) and diaminobenzophenone (DABP) in combination with 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA) and 4,4'-oxydiphthalic anhydride (ODPA).

EXPERIMENTAL

POLYMER SYNTHESIS

The monomers used in this study are shown in Table I and were obtained from commercial sources. The monomers were either used as received after vacuum drying @ 50°C or purified by vacuum sublimation. Fresh N,N-dimethylacetamide (DMAc) and

1-methyl-2-pyrrolidinone (NMP) were used for the polymerizations. Every attempt was made to use monomers of constant purity throughout the study. The keto-ether polyimides were prepared according to the general reaction scheme in Figure 1. The polymerizations were conducted in a closed vessel at room temperature (RT) at a concentration of 15% solids (w/w). A stoichiometric amount of one of the dianhydrides was added to a solution of one of the diamines in the solvent. The container was flushed with nitrogen and the solution mechanically stirred overnight. Initially, four systems were prepared using the two different solvents: 3,3'-DABP/BTDA, 3,3'-DABP/ODPA, 4,4'-ODA/BTDA, and 4,4'-ODA/ODPA.

CHARACTERIZATION

Melting points of the monomers were determined at a heating rate of 10°C/min on a DuPont Model 990 Thermal Analyzer in combination with a standard DTA cell. Inherent viscosities (η_{inh}) were obtained on poly(amide acid) (PAA) solutions with a Cannon-Ubbelohde viscometer at 0.5% (w/v) in DMAc at 35°C. Glass transition temperatures (T_g) were determined by differential scanning calorimetry (DSC) using a DuPont 990 Thermal Analyzer at a heating rate of 20°C/min in static air. Polyimide films were heated to 375°C, quenched, and rerun to obtain the T_g . The T_g s were obtained from the second heating at the inflection midpoint of the ΔT versus temperature curve. Dynamic thermogravimetric analyses (TGA) were obtained using a Seiko Model TG/DTA 220 at a heating rate of 2.5°C/min and an air flow of 15cc/min. Samples were held at 100°C for 30 min and then heated to 650°C. A Model 2000/2 table-top Sintech load frame, equipped with a CompuAdd Model 286 computer was used for measuring unoriented thin film tensile properties. Pneumatically actuated, one inch grips with steel faces were used for clamping the polyimide films.

POLYIMIDE FILMS

The PAA solutions were centrifuged, the decantate cast onto plate glass using a 30 mil doctor blade, and air dried in a dust-free, low humidity chamber until tack-free. Three films of each solution were prepared and cured in a forced-air oven utilizing one of three different imidization cure cycles: 1) *standard* - 1 hr each @ 100, 200, and 300°C; 2) *relative* - 1 hr each @ 150, 200, and ($T_g + 20^\circ\text{C}$); and 3) *extreme* - 1 hr each @ 150, 250, and 350°C. After cooling, the films were removed by soaking in warm tap water, if necessary.

Mechanical properties of the nominal 2.0 mil thick unoriented films were determined @ RT as recommended in ASTM D882 using a minimum of five specimens per

test condition. Each specimen thickness was determined to ± 0.0001 in. with a Testing Machines, Inc. Model 49-70 electronic micrometer. The film specimens were cut to a width of 0.20 in. with a Thwing-Albert JDC Precision Cutter. A gauge length of 2.00 in. and a crosshead speed of 0.20 in/min was used for determination of elastic modulus, tensile strength, and percent elongation at break.

Mechanical properties were also determined on film specimens after thermal aging. Film strips were clamped in a machined stainless steel device under no initial stress and placed uncovered in a forced-air oven @300°C. Specimens were removed after 50 and 100 hours and tested as previously described.

Thermally imidized films were tested for solubility at RT at approximately 0.5% by weight in methylene chloride (CH_2Cl_2), DMAc, *m*-cresol, and NMP. After 96 hours at RT, if the films were insoluble, they were retested again after heating 15 minutes at the boiling point of each solvent.

RESULTS AND DISCUSSION

FIRST STAGE

The first stage of the study involved the synthesis of four representative keto-ether polyimides in two different solvents, DMAc and NMP. The first stage of the synthesis (Figure 1), conducted at RT, resulted in a precursor poly(amide acid) solution which was light yellow to dark amber in color. As seen in Table II, inherent viscosities were higher in three out of the four cases for those PAA solutions prepared in DMAc. The highest viscosity of 1.95 dL/g was obtained on the 4,4'-ODA/ODPA system. After the solvent-cast films were thermally imidized by air curing, they were all flexible and creasable. Films cast from NMP were much more difficult to remove from the glass plates and had to be soaked in water for several days. The film color and glass transition temperatures are also shown in Table II. The imidized films obtained from DMAc solutions were light yellow to light amber in color and those obtained from NMP solutions were dark yellow to dark amber. There was no variation in the T_g of DMAc cast films as compared to NMP cast films. At this point in the study, the best choice of solvent appeared to be DMAc.

Unoriented thin film mechanical properties were obtained @ RT on films of each polyimide after curing at one of three cycles: *standard*(to 300°C), *relative*(to 20°C above T_g), and *extreme*(to 350°C). Some of the data that was generated on standard cure films are listed in Table III. The thermal stability, as determined by dynamic TGA, did not vary

significantly for each pair of films. However, there was a significant variation in tensile modulus. For the first three polyimides, the modulus was higher for the NMP cast films than for the DMAc ones, and for the fourth they were very similar. The percent elongation was unchanged for the two diaminobenzophenone polymers. In comparison, the 4,4'-ODA/BTDA elongation was higher when prepared in NMP and the 4,4'-ODA/ODPA elongation was higher when prepared in DMAc.

Table IV lists some of the data generated on extreme cured films. The variation in properties due to the difference in reaction solvent followed the same pattern found in the standard cure data. There was no significant variation in thermal stability, as there was in tensile properties. The elongations followed the same pattern as in the previous cure condition.

Some of the data collected on relative cure films are shown in Table V. The purpose of the relative cure cycle was to see if optimum film properties could be obtained by only imidizing the polymer 20°C above its T_g. There was not as much variation in tensile modulus for the relative cured films as there was for the other two curing conditions. But, in this case there was a slight advantage to using DMAc as the reaction solvent. Again, the elongations followed the same pattern as in the previous two cure situations.

The tensile modulus results for the NMP prepared films are plotted in Figure 2 and those on DMAc prepared films are plotted in Figure 3 for comparison. Tensile modulus was used as the main criteria for determining optimum conditions because it is not affected by flaws or defects in the films like tensile strength and elongation are. In examining the modulus results of polymers prepared in NMP, there was no distinct advantage in using one cure cycle over another. The standard cure gave unusually high results for the 3,3'-DABP/BTDA system and poor results for the 4,4'-ODA/ODPA system. On the other hand, examining the data on those polymers prepared in DMAc, the relative cure cycle resulted in the best modulus values overall. For the four keto-ether polyimides investigated, the optimum results were obtained using DMAc as the reaction solvent along with the *relative* imidization cure cycle.

SECOND STAGE

Six additional polymers were prepared using combinations of the monomers from Table I in DMAc and the films were cured using only the *relative* imidization cure cycle, in which the films were heated to 20°C above their T_g, as determined by DSC. Some of the physical properties for these polyimides are shown in Table VI. For the entire series, the lowest η_{inh} of 0.56 dL/g was obtained on the 4,4'-DABP/BTDA system and the highest

value of 1.90 dL/g on the 4,4'-ODA/ODPA system. For each pair of systems with the same diamine moiety, the one with the ether bridge in the dianhydride portion generally had the higher η_{inh} . For both oxydianiline series, the η_{inh} was observed to increase as one went from the 3,3'-isomer to the 4,4'-isomer. All of the polyimide films were flexible and creasable, but varied in color. The diaminobenzophenone films were always amber in color, whereas the oxydianiline films were usually yellow. Thermal stability data as determined by dynamic TGA was inconclusive since there was not a significant difference between the weight loss of these polyimides. The glass transition temperatures ranged from a low of 202°C for the 3,3'-ODA/ODPA system to high of 290°C for the 4,4'-DABP/BTDA system. The BTDA based polyimides always had a higher Tg than their ODPA based counterparts. For each series with the same dianhydride portion, the Tg increased in relation to the isomeric attachment in the diamine as follows: 3,3'- < 3,4'- < 4,4'-. As has been observed in previous studies, the increased chain flexibility of the meta-linkage causes a decrease in Tg, whereas the highly dipolar nature of the carbonyl bridging group causes an increase in Tg over those with the ether bridge[7-8].

The solubility characteristics of the keto-ether polyimides were compared at the boiling point of four organic solvents and the results are shown in Table VII. The most soluble system was the 3,3'-ODA/ODPA, which was presumably due to a combination of increased flexibility and the presence of ether bridges. The least soluble systems were those made with the 4,4'-DABP. In general, the ODPA polyimides appeared to be more soluble than the BTDA counterparts.

Unoriented thin film tensile properties were determined on each of the keto-ether films and are reported in Table VIII. The tensile strengths of the films were approximately 20 ksi. The data is listed in order of increasing tensile modulus, which ranged from a low of 419 ksi for 4,4'-ODA/ODPA to a high of 552 ksi for 3,3'-DABP/BTDA. In general, the ODPA based polyimides exhibited lower tensile moduli and higher elongations at break than the BTDA based polyimides. Also it appeared that meta catenation in the polyimide backbone resulted in higher moduli than the corresponding polyimides with para catenation. These results were unexpected.

Mechanical properties at RT were determined on polyimide films thermally aged at 300°C in air for 50 and 100 hours. The aging temperature was anywhere from 10 to 100°C above the Tg of these polymers. Figure 4 is a comparison of tensile moduli before and after aging. After 50 hours at temperature, most of the values dropped 10 to 20%, and there did not appear to be any further degradation after 100 hours. The exception to this rule was the two 3,4'-ODA systems, which retained their properties. The 3,3'-ODA/ODPA system, which had the lowest Tg, exhibited the most dramatic decrease in properties. The trend

was much different when examining the percent elongation data before and after aging (Figure 5). The polyimides that had elongations of about 10% or less were unaffected by the aging conditions. Whereas, those systems with high values initially dropped off significantly after aging, some as much as 80%. This could possibly be attributed to the existence of defects in the films, such as dust particles and pin holes, which were sites for small crack development during aging, which could cause the films to fail prematurely. There was no structural related trends apparent from this mechanical data.

SUMMARY

A series of polyimides were synthesized containing only carbonyl and/or oxygen linkages in the backbone. These polymers were characterized as films after curing under different conditions, which included heating up to 20°C above their glass transition temperature. Unoriented thin film mechanical properties were greatly affected by differences in reaction solvent and which imidization cycle was used. Optimum physical and mechanical properties were obtained by using a combination of DMAc as the reaction solvent and a relative imidization cure cycle. Inherent viscosities were usually higher for the ether bridged systems and appeared to increase as one went from the 3,3'-isomer to the 4,4'-isomer of the diamine. Glass transition temperatures were generally higher for keto-bridged polyimides over the corresponding ether-bridged system and increased in relation to the isomeric attachment in the diamine portion of the backbone. The solubility of the polyimides were dependent on several factors. A combination of meta catenation and ether linkages yielded the most soluble systems. Mechanical properties were related to the isomeric attachment in the diamine portion of the keto-ether polyimides. Meta catenation in the polymer backbone seemed to result in higher tensile moduli than those polyimides with para catenation. Polyimides with an ether linkage in the dianhydride portion of the backbone generally exhibited lower tensile moduli and higher elongations than those with a carbonyl linkage.

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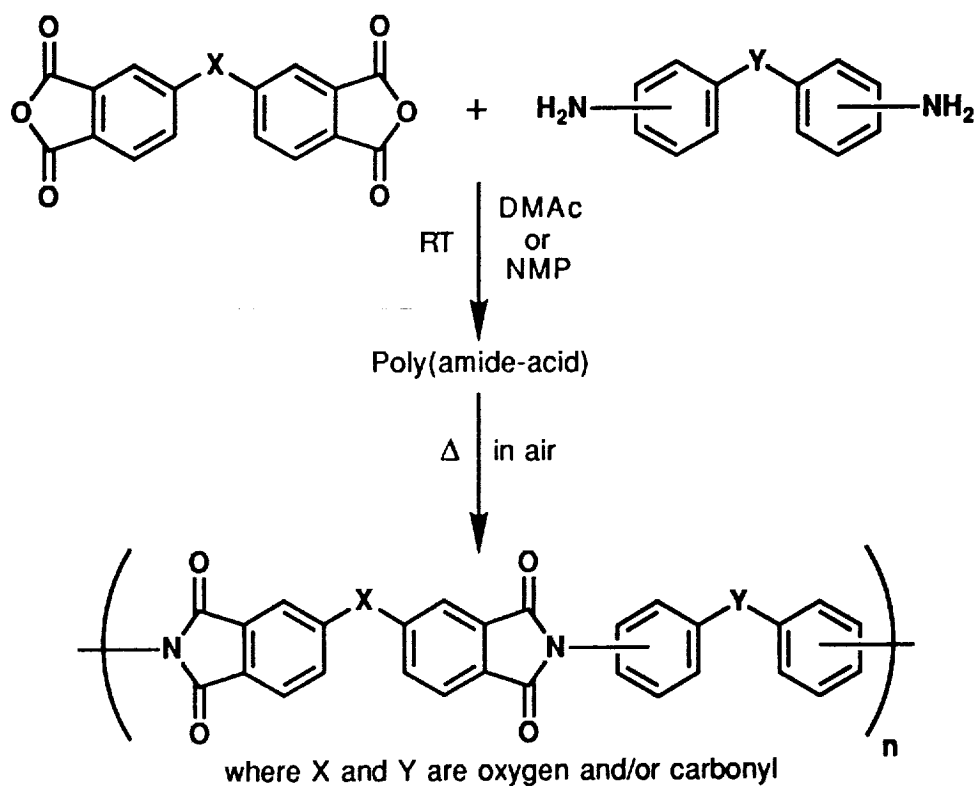


Figure 1. Keto-ether polyimide reaction scheme.

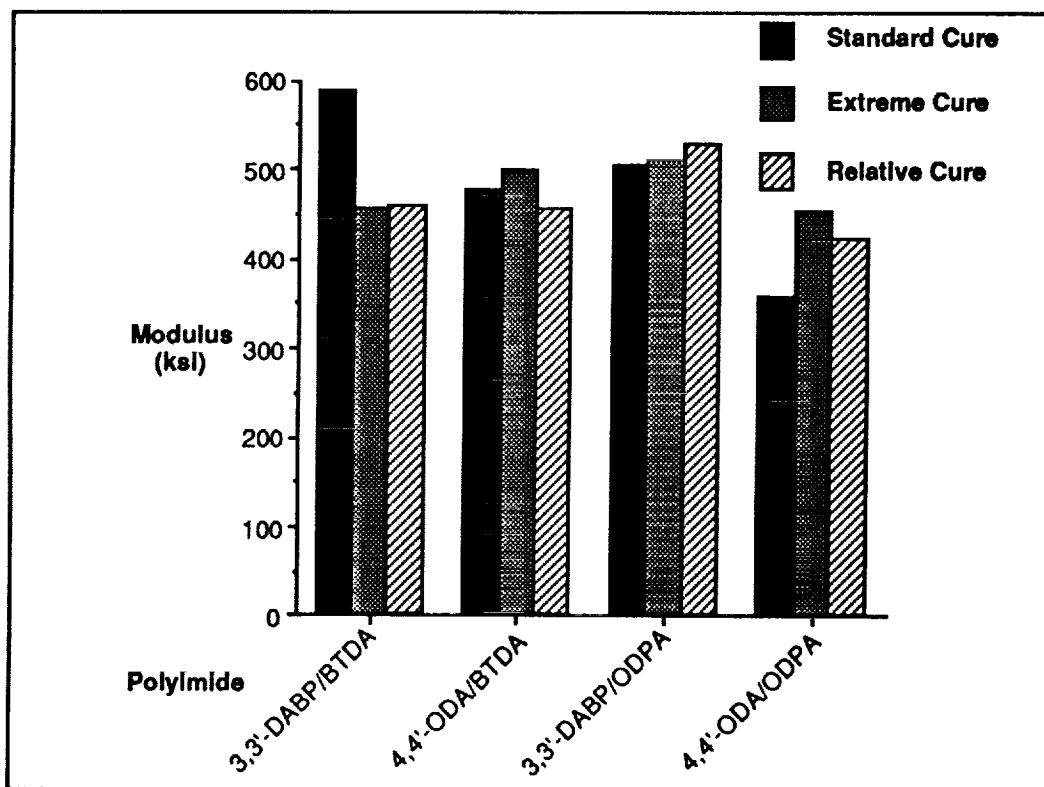


Figure 2. Tensile properties of keto-ether polyimides prepared in NMP.

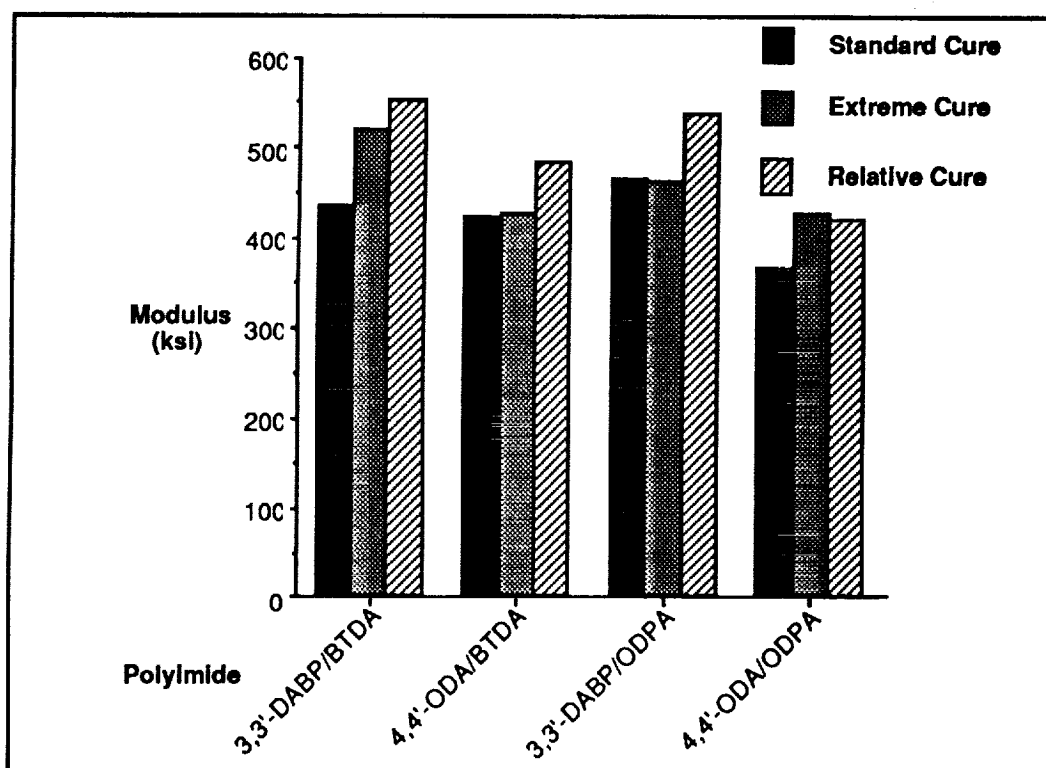


Figure 3. Tensile properties of keto-ether polyimides prepared in DMAc.

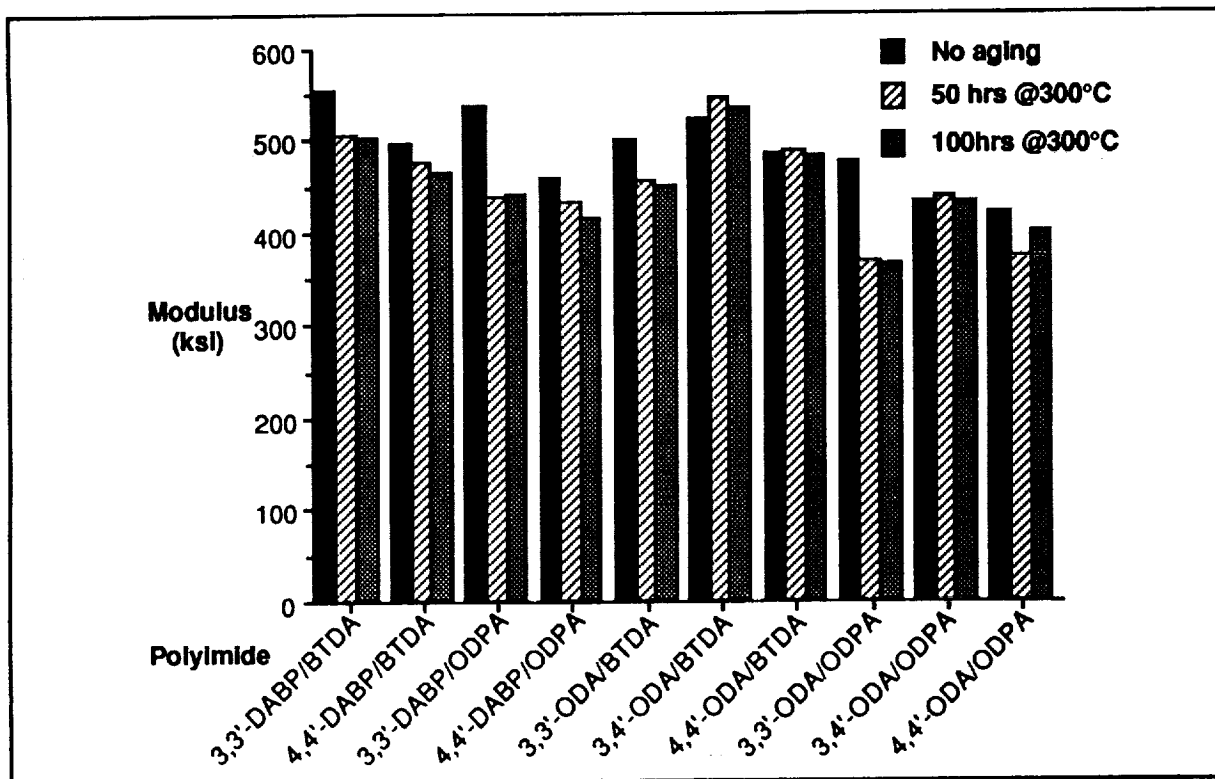


Figure 4. Tensile modulus of keto-ether films before and after aging.

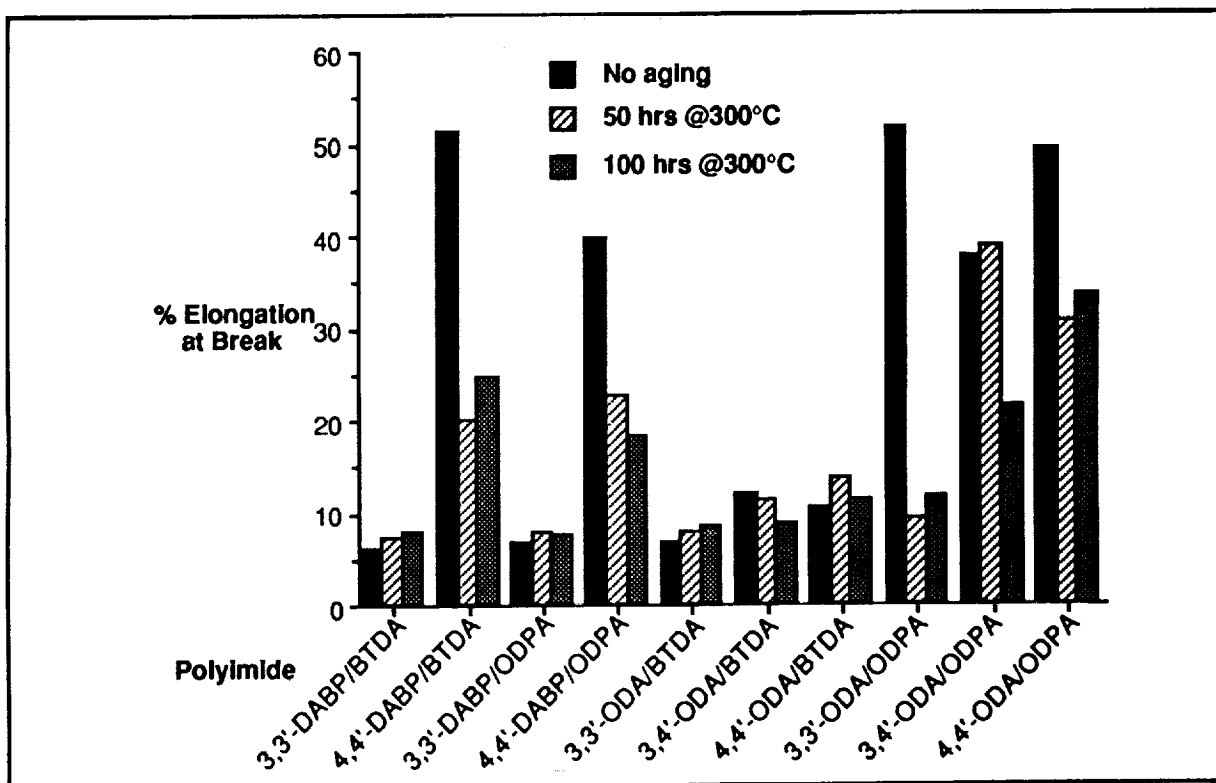


Figure 5. Percent elongation of keto-ether films before and after aging.

TABLE I - MONOMERS USED TO PREPARE KETO-ETHER POLYIMIDES

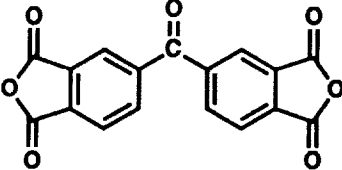
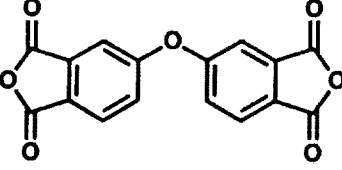
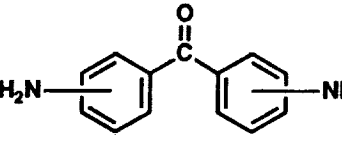
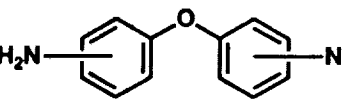
STRUCTURE	NAME and ACRONYM	SOURCE and PURIFICATION	m.p. ONSET
	3,3',4,4'-benzophenone-tetracarboxylic dianhydride BTDA	Allco Chemical; sublimed @ 200°C	228°C
	3,3',4,4'-oxydiphthalic anhydride ODPA	Occidental Chemical; sublimed @ 220°C	224°C
	3,3'-diaminobenzophenone 3,3'-DABP	Mitsui Toatsu; used as received	145°C
	4,4'-diaminobenzophenone 4,4'-DABP	Ash Stevens; sublimed @ 240°C	243°C
	3,3'-oxydianiline 3,3'-ODA	Daychem Laboratories; used as received	76°C
	3,4'-oxydianiline 3,4'-ODA	Kennedy & Klim; sublimed @ 90°C	72°C
	4,4'-oxydianiline 4,4'-ODA	Kennedy & Klim; sublimed @ 190°C	189°C

TABLE II - PHYSICAL PROPERTIES OF KETO-ETHER POLYIMIDES

Polymer	Solvent	η_{inh} , dL/g ¹	Film Color	Tg, °C ²
3,3'-DABP/BTDA	DMAc	1.49	amber	255
3,3'-DABP/BTDA	NMP	1.22	dark amber	256
4,4'-ODA/BTDA	DMAc	1.67	yellow	277
4,4'-ODA/BTDA	NMP	1.59	dark yellow	276
3,3'-DABP/ODPA	DMAc	1.32	light amber	239
3,3'-DABP/ODPA	NMP	1.18	dark amber	238
4,4'-ODA/ODPA	DMAc	1.90	light yellow	267
4,4'-ODA/ODPA	NMP	1.95	yellow	269

¹0.5% wt/vol in DMAc @ 35°C ²DSC, 20°C/min, after initial heating to 375°C in air

TABLE III - MECHANICAL PROPERTIES OF STANDARD CURED POLYIMIDES

Polymer	Solvent	Temp. at 5% Wt. Loss, °C	Tensile Modulus, ksi	% Elongation at Break
3,3'-DABP/BTDA	DMAc	465	435	6.5
3,3'-DABP/BTDA	NMP	470	586	6.6
4,4'-ODA/BTDA	DMAc	516	422	17.9
4,4'-ODA/BTDA	NMP	481	479	31.2
3,3'-DABP/ODPA	DMAc	460	465	7.7
3,3'-DABP/ODPA	NMP	477	503	7.3
4,4'-ODA/ODPA	DMAc	508	366	56.3
4,4'-ODA/ODPA	NMP	499	356	36.9

Films cured 1 hr ea. @ 100, 200, and 300°C in air

TABLE IV - MECHANICAL PROPERTIES OF EXTREME CURED POLYIMIDES

Polymer	Solvent	Temp. at 5% Wt. Loss, °C	Tensile Modulus, ksi	% Elongation at Break
3,3'-DABP/BTDA	DMAc	480	517	7.4
3,3'-DABP/BTDA	NMP	481	455	6.4
4,4'-ODA/BTDA	DMAc	476	425	14.8
4,4'-ODA/BTDA	NMP	480	498	26.5
3,3'-DABP/ODPA	DMAc	464	461	7.6
3,3'-DABP/ODPA	NMP	480	510	7.0
4,4'-ODA/ODPA	DMAc	495	425	38.5
4,4'-ODA/ODPA	NMP	509	453	18.2

Films cured 1 hr ea. @ 150, 250, and 350°C in air

TABLE V - MECHANICAL PROPERTIES OF RELATIVE CURED POLYIMIDES

Polymer	Solvent	Temp. at 5% Wt. Loss, °C	Tensile Modulus, ksi	% Elongation at Break
3,3'-DABP/BTDA	DMAc	475	552	6.0
3,3'-DABP/BTDA	NMP	460	547	6.5
4,4'-ODA/BTDA	DMAc	497	484	10.4
4,4'-ODA/BTDA	NMP	508	463	21.0
3,3'-DABP/ODPA	DMAc	481	535	6.9
3,3'-DABP/ODPA	NMP	452	524	6.0
4,4'-ODA/ODPA	DMAc	500	419	49.4
4,4'-ODA/ODPA	NMP	509	414	37.1

Films cured 1 hr ea. @ 150, 200, and (T_g + 20°C) in air

TABLE VI - PHYSICAL PROPERTIES OF KETO-ETHER POLYIMIDES

Polyimide	$\eta_{inh}, dL/g^1$	Film Color	T _g , °C ²	Temp. at 20% Wt. Loss, °C ³
4,4'-DABP/BTDA	0.56	dark amber	290	546
4,4'-DABP/ODPA	0.70	dark yellow	274	541
3,3'-ODA/BTDA	1.09	dark yellow	221	539
3,4'-ODA/BTDA	1.14	dark yellow	252	540
3,3'-ODA/ODPA	1.15	light yellow	202	543
3,3'-DABP/ODPA	1.32	light amber	239	547
3,3'-DABP/BTDA	1.49	amber	255	545
4,4'-ODA/BTDA	1.67	yellow	277	540
3,4'-ODA/ODPA	1.82	yellow	237	539
4,4'-ODA/ODPA	1.90	light yellow	267	542

¹0.5% wt/vol in DMAc @ 35°C ²DSC, 20°C/min, air ³TGA, 2.5°C/min, air

TABLE VII - SOLUBILITY OF KETO-ETHER POLYIMIDE FILMS

Polyimide	DMAc	NMP	CH ₂ Cl ₂	<i>m</i> -Cresol
3,3'-DABP/ODPA	PS	SH	IH	SH
4,4'-DABP/ODPA	IH	IH	IH	IH
3,3'-ODA/ODPA	SH	SH	PS	SH
3,4'-ODA/ODPA	IH	PS	IH	SH
4,4'-ODA/ODPA	PS	SH	IH	SH
3,3'-ODA/BTDA	IH	IH	IH	IH
3,4'-ODA/BTDA	IH	IH	IH	IH
4,4'-ODA/BTDA	IH	IH	IH	IH
3,3'-DABP/BTDA	IH	SH	IH	IH
4,4'-DABP/BTDA	IH	IH	IH	IH

SH - Soluble @ bp of solvent
 PS - Partially soluble @ bp of solvent
 IH - Insoluble @ bp of solvent

TABLE VIII - MECHANICAL PROPERTIES OF KETO-ETHER POLYIMIDE FILMS @ RT

Polyimide	Tensile Strength, ksi	Tensile Modulus, ksi	% Elongation at Break
4,4'-ODA/ODPA	19.6	419	49.4
3,4'-ODA/ODPA	16.6	431	37.8
4,4'-DABP/ODPA	20.0	458	39.6
3,3'-ODA/ODPA	17.5	476	51.7
4,4'-ODA/BTDA	19.8	484	10.4
4,4'-DABP/BTDA	22.9	497	51.2
3,3'-ODA/BTDA	19.3	498	6.6
3,4'-ODA/BTDA	18.7	521	12.1
3,3'-DABP/ODPA	22.2	535	6.9
3,3'-DABP/BTDA	21.8	552	6.0

Nominal thickness: 2 mils, width: 0.2 in, gauge length: 2.0 in.

REPORT DOCUMENTATION PAGE

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13. ABSTRACT (Maximum 200 words) Linear aromatic polyimides are a class of high performance polymers noted for their thermal stability, adhesive properties, and solvent resistance. As part of an on-going effort to develop an understanding of how changes in the chemical structure affect polymer properties, a study was performed on polyimides containing only ether and/or carbonyl connecting groups. The purpose of the initial stage of the study was to examine the properties of a small group of polyimide systems to determine the optimum solvent and cure cycle requirements. These optimum conditions were then utilized to prepare films of keto-ether polyimides which were evaluated for mechanical and thermal properties. These polyimides were prepared using isomers of oxydianiline (ODA) and diaminobenzophenone (DABP) in combination with 3,3', 4,4'-benzophenone-tetracarboxylic dianhydride (BTDA) and 4,4'-oxydiphthalic anhydride (ODPA).				
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