Residual Stress and Mechanical Properties of Polyimide Thin Films

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ABSTRACT: Four different structure polyimide thin films based on 1,4-phenylene diamine (PDA) and 4,4′-oxydianiline (ODA) were synthesized by using two different dianhydrides, pyromellitic dianhydride (PMDA) and 3,3′,4,4′-biphenyltetracarboxylic dianhydride (BPDA), and their residual stress behavior and mechanical properties were investigated by using a thin film stress analyzer and nanoindentation method. The residual stress behavior and mechanical properties were correlated to the morphological structure in polyimide films. The morphological structure of polyimide thin films was characterized by X-ray diffraction patterns and refractive indices. The residual stress was in the range of −5 to 38 MPa and increased in the following order: PMDA-PDA < BPDA-PDA < PMDA-ODA < BPDA-ODA. The hardness of the polyimide films increased in the following order: PMDA-ODA < BPDA-ODA < PMDA-PDA < BPDA-PDA. The PDA-based polyimide films showed relatively lower residual stress and higher hardness than the corresponding ODA-based polyimide films. The in-plane orientation and molecularly ordered phase were enhanced with the increasing order as follows: PMDA-ODA < BPDA-ODA < BPDA-PDA ∼ PMDA-PDA. The PDA-based polyimides, having a rigid structure, showed relatively better-developed morphological structure than the corresponding ODA-based polyimides. The residual stress behavior and mechanical properties were correlated to the morphological structure in polyimide films. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 976–983, 2009

Key words: polyimide; residual stress; hardness; morphological structure; chain mobility

INTRODUCTION

Recently, multichip packing technologies for high-performance electronic devices are expected to benefit from the use of polymers as interlayer dielectrics, passivation layers, and alpha particle barriers. Aromatic polyimide films, for these devices, have received significant attention because of their high thermal stability, relatively low dielectric constant, good mechanical properties, high chemical resistance, and easy processability.[1–3] For the application of poly (amic acid)s in the fabrication of devices, residual stress is one of the most important properties to be investigated because the residual stress causes delamination at the metal/polyimide interface or crack formation in the metal or in the polyimide film associated with reliability problems.[4,5] Therefore, the stress at all interfaces needs to be as low as possible to avoid the stress-associated reliability problems.

Also, many technologies depend, at some critical juncture, on advanced manufacturing processes.[6] As manufacturing operations approach submicron, nanometer, or even at the best atomic scale, efforts are underway in materials and mechanics communities to measure, characterize, and model behaviors at submicron and nanometer scale.[7,8] The need to measure and characterize properties, such as, electronic, physical, and mechanical, etc., have driven the development of instrumented "nanoindentation" hardness testers capable of characterizing the near-surface properties of thin films, microscopic wires, and bulk materials. Surface and near-surface mechanical properties of thin films and coatings are critical to their final performance. The rapidly expanding field of depth-sensing nanoindentation provides a quantitative method for mapping the mechanical properties, such as hardness and elastic modulus, of the surface/near-surface region. In the study of Poilane et al.,[9] nanoindentation tests have been performed with success on thin films of polyurethane (PU)-shaped memory polymer to characterize their mechanical behavior. Cramez et al.[10] have reported that for rotationally molded polyethylene (PE), the values of microhardness depend on the type of pigment.

In this article, we present the residual stress and hardness/elastic modulus of polyimide thin films

with different chemical structures and studied their morphological structure. The residual stress between polyimide film and Si substrate was measured *in situ* using a thin film stress analyzer (TFSA) and hardness/elastic modulus of the surface/near-surface region were investigated using a nanohardness tester.[11] The residual stress behaviors and mechanical properties were interpreted from the morphological structure in polyimide films. The morphological structure of polyimide thin films was characterized by wide angle X-ray diffraction (WAXD) patterns and refractive indices.

EXPERIMENTAL

Material and sample preparation

Diamine and dianhydride monomers were purchased from commercial products (Aldrich and Chriskev, Yongin-City, Korea) and purified by recrystallization in methanol or sublimation under reduced pressure before use. Anhydrous 1-methyl-2-pyrrolidinone (NMP) was purchased from Aldrich (Yongin-City, Korea) and used as received. Poly(1,4-phenylene pyromellitamic acid) (PMDA-PDA PAA) solution was prepared under a nitrogen atmosphere by slowly adding pyromellitic dianhydride (PMDA) to 1,4-phenylene diamine (PDA) in NMP as described in our previous studies.[12] The resulting solution had a solid content of 15 wt %. Other poly(amic acid)s were prepared in the same manner as PMDA-PDA PAA solution was synthesized: PMDA-4,4′-oxydianiline (ODA) PAA, 3,3′,4,4′-biphenyltetracarboxylic dianhydride (BPDA)-PDA PAA, BPDA-ODA PAA (see Fig. 1).

All precursor solutions were spin-coated on silicon wafers and soft-baked at 80°C for 30 min. The ramping rate was 2.0°C/min and the cooling rate was 1.0°C/min. The film thickness was controlled within the range of 10–14 μm to exclude the thickness effect and measured with a surface profiler (KLA-Tencor Alpha Step 500). The one-side polished Si (100) wafer as substrate was 76.2 mm. For the WAXD and dynamic mechanical thermal analysis (DMTA) measurements, thermally cured polyimide thin films were removed from the substrate after soaking in distilled water for 24 h. They were washed with distilled water several times and dried.

Measurement

The residual stress of polyimide thin film on a Si (100) substrate was measured during thermal imidization with cure process and subsequent cooling, using a double He–Ne laser beam using a home-made TFSA.[13] The residual stress was calculated from the measured radii of curvature using the following equation:

$$\sigma = \frac{E_s}{6(1 - \nu_s)} \frac{t_s^2}{t_f} \left(\frac{1}{R_2} - \frac{1}{R_1} \right) \quad (1)$$

where σ is the residual stress in the polyimide film. The subscripts f and s denote the polyimide film and the substrate, respectively. E, v, and t are Young's modulus, Poisson's ratio, and the thickness of the substrate, respectively. R_1 and R_2 are the wafer curvature measured before and after the film deposition. For Si (100) wafer, $E_s/(1 - \nu_s)$ is 180,500 MPa.[14] The details are described in our previous studies.[15,16]

The nanohardness tester (MTS XP System) was used to measure the mechanical properties of the polyimide thin films. Berkovich (3-faced pyramid) indenter was used in this study. Multiple indentations were made at different locations of the film surface at a fixed and applied load. Both the indentation loading and unloading rates were set equal as 0.06 mN/s. At different locations, the load versus displacement curve was recorded, from which the effective modulus and hardness can be calculated using standard formulae.[17,18] In addition, the actual plastic (permanent) deformation was determined at the end of each loading cycle.

The out-of-plane and in-plane refractive indices of the polyimide thin films were measured using a prism coupler (Metricon Model 2010) equipped with a He–Ne laser light source (wavelength = 632.8 nm), which was controlled by a personal computer. Measurements of the refractive indices were carried out in transverse electric (TE) and transverse magnetic (TM) mode with the appropriate polarization of the incident laser beam as described elsewhere.[12] The TE measurement in which the electric field was in the film plane provides the in-plane refractive index (n_{TE}), whereas the TM measurement in which the electric field is out-of-plane gives the out-of-plane refractive index (n_{TM}). All measurements were performed using a cubic zirconia prism of $n_{TE} = n_{TM} = 2.1677$.

WAXD measurements were conducted in the $\theta/2\theta$ method over 3.5°–60° using a Rigaku horizontal X-ray diffractometer (Model D/Max-200B) with Cu Kα (λ = 1.54 Å) radiation source. Step and count data were taken at 0.02° intervals with a scan speed of 0.4°/min. The DMTA measurements were performed with PL series (Polymer Lab, MKIII) from 50 to 500°C at a heating rate 5.0°C/min and at a frequency of 1 Hz, respectively.

RESULTS AND DISCUSSION

Residual stress

The residual stress–temperature profiles of four different backbone structure polyimide films on Si (100) substrate were studied at different temperatures and
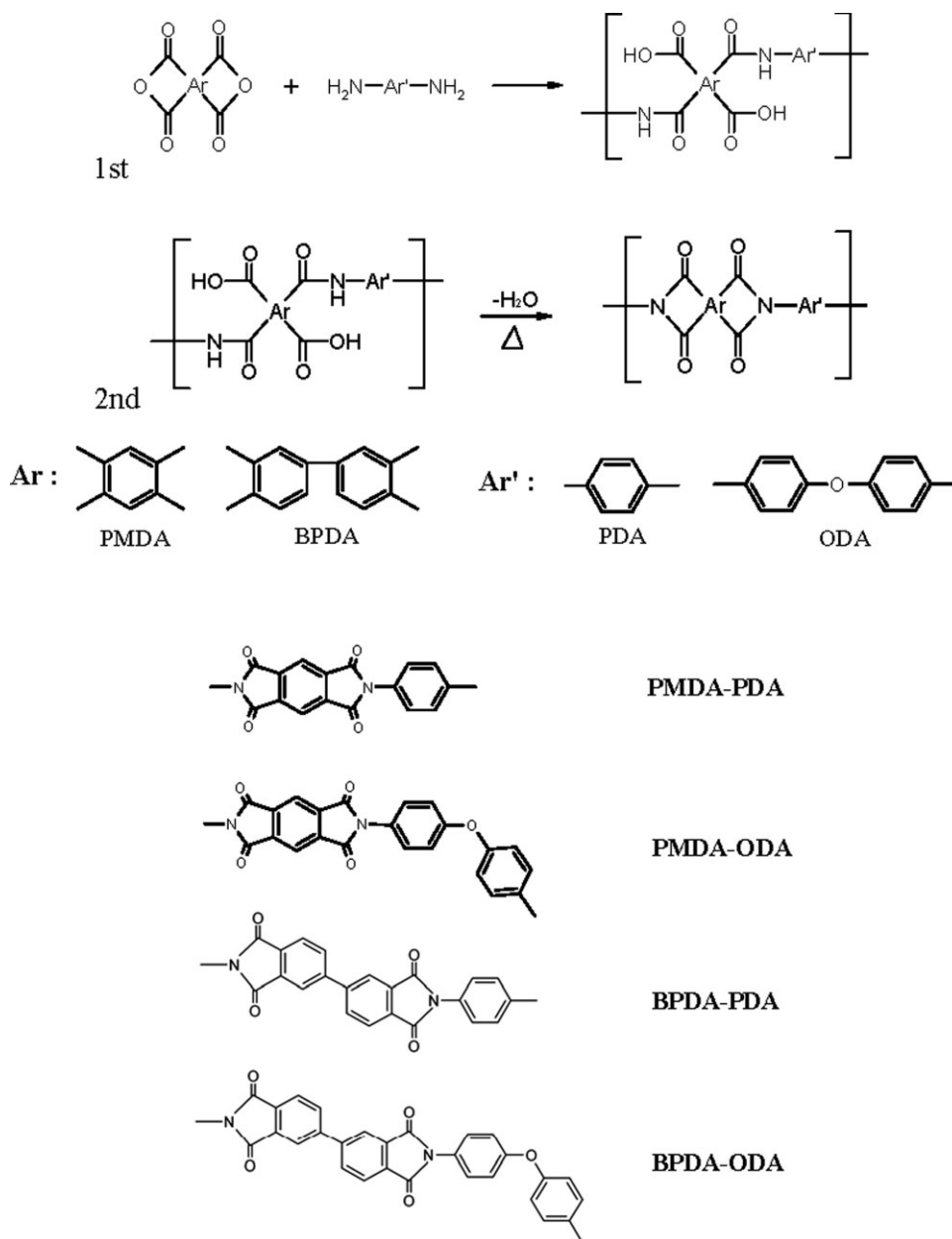
Figure 1 Synthetic scheme of poly(amic acid) precursor and its polyimide.

depicted in Figure 2. The stress behavior of polyimide films with different backbone structures was quite different and strongly depends on the polyimide backbone structure. During curing process (i.e., heating) and cooling process of polyimide film, residual stress can be attributed to the mismatch in the thermal expansion coefficients between polymer film and the silicon substrate and the modulus of polymer film. When the biaxial modulus of the film is relatively invariable, the slope of the cooling process curve means the change in the residual stress of polyimide film with temperature variation.[16] In our study, the residual stress was calculated from the measured radii of curvature using eq. (1). Here, the positive and negative signs denote the residual stress

in the tension mode and in the compression mode, respectively. As summarized in Table I, the residual stress was in the range of −5 to 38 MPa and increased in the following order: PMDA-PDA < BPDA-PDA < PMDA-ODA < BPDA-ODA. In addition, the PDA-based polyimides, PMDA-PDA and BPDA-PDA, showed relatively lower residual stress and slope in cooling curve than the corresponding ODA-based polyimides, PMDA-ODA and BPDA-ODA. In particular, for the PMDA-PDA, the residual stress decreased with the cooling temperature in compression mode and varied to –5 MPa at 25°C. The negative value in compression mode means the thermal expansion coefficient of PMDA-PDA thin films attached on Si wafer is lower than that of Si (100).
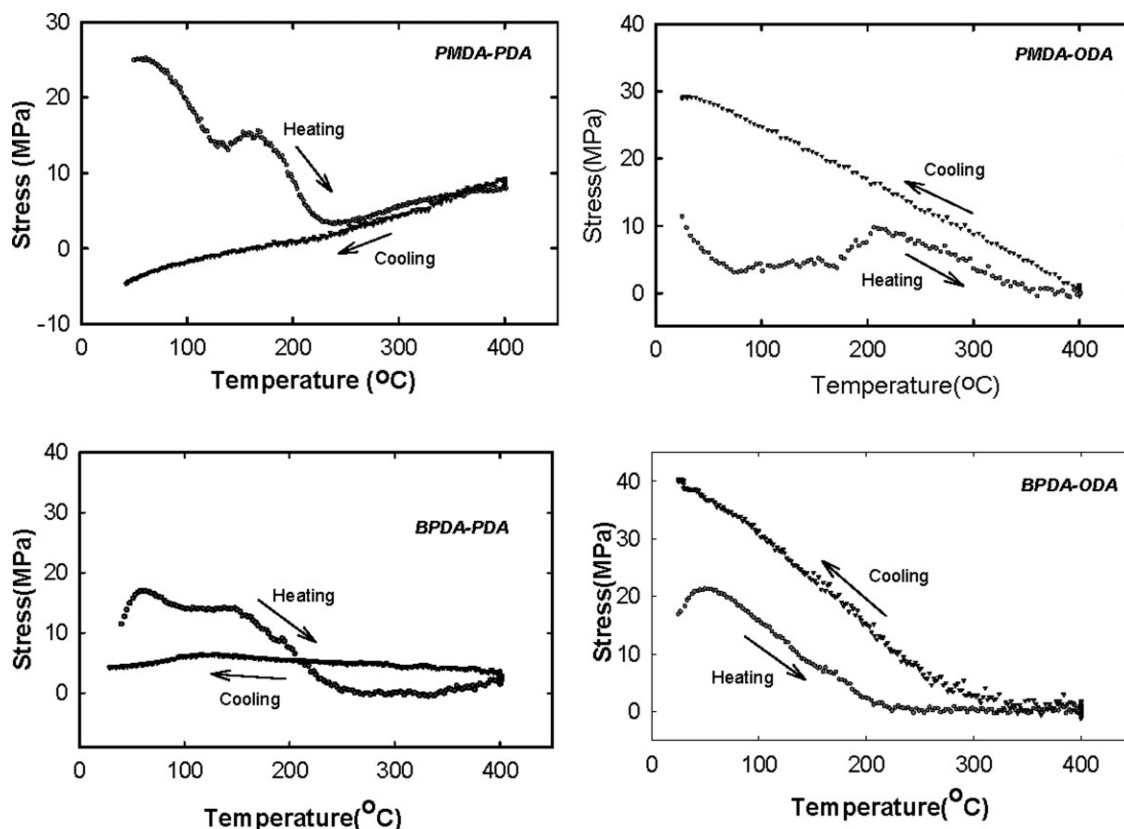
Figure 2 Residual stress of polyimide thin films with different backbone structures.

The film stress is mainly contributed to the thermal stress component, which is proportional to several factors, such as mismatched thermal expansion coefficient (α_s, α_f) and modulus (E). For Si (100), the α_s is 3.0 ppm/°C over 25–400°C. Therefore, the film stress mainly depends on the factors, α_f (thermal expansion coefficient of film) and modulus. Generally, the incorporation of flexible unit into the polyimide backbone will increase the thermal expansion coefficient and chain mobility, whereas it lowers the modulus in film. Comparing chemical structures of PDA, ODA has an ether linkage (—O—) per one repeating unit (see Fig. 1), which can be treated as a relatively flexible unit in polyimide chain. This flexible ODA unit may induce relatively easy chain mobility, high thermal expansion coefficient, and lower modulus, which may result in high residual stress in the ODA-based polyimide film.[15,16]

Nanoindentation

For the different structure polyimide films, the mechanical properties were calculated from load versus displacement curve using the Berkovich nanoindenter. The elastic–plastic behavior of the polyimide thin films with various backbone structures is best illustrated in Figure 3. For the different structure polyimide films, the characteristic kickback

(elbow) appears in the unloading curves, which indicates that all the polyimide films exhibit an elastic–plastic response.[9,10,19–21] Figure 3 clearly shows that with an increase in the applied load, both the maximum indentation depth and the plastic response of the polymer increased. Their results are summarized in Table II.

As the applied load increases from 0.08 to 0.162 mN, the penetration depth of the polyimide thin films varied from 86 to 123 nm for BPDA-PDA, from 88 to 127 nm for PMDA-PDA, from 103 to 151 nm for BPDA-ODA, and from 130 to 183 nm for PMDA-ODA, respectively. The hardness varied from 0.749 to 0.687 GPa for BPDA-PDA, from 0.681 to 0.618 GPa for PMDA-PDA, from 0.453 to 0.386 GPa for BPDA-ODA, and from 0.273 to 0.248 GPa for PMDA-ODA, respectively. The variation in the hardness with the

TABLE I
Residual Stress of Polyimide Thin Films with Different Backbone Structures

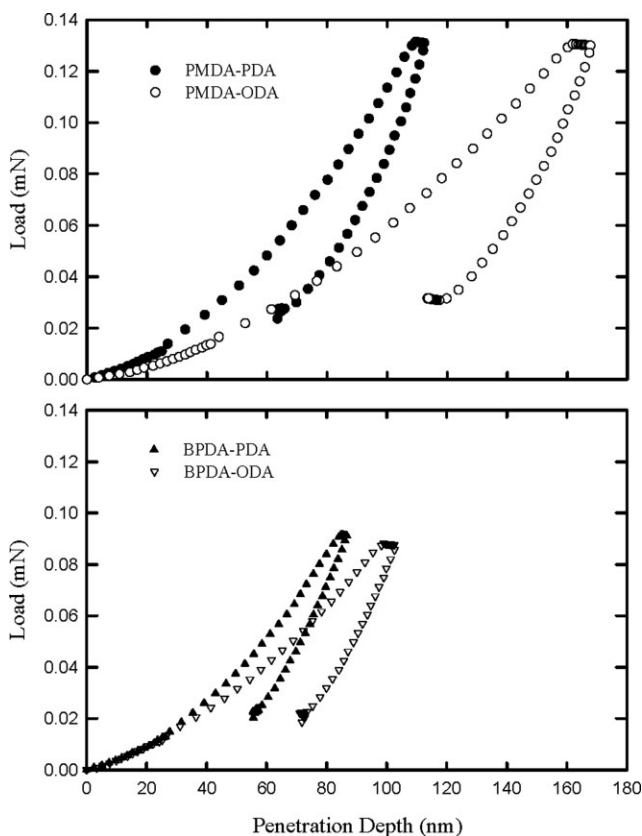| Polymer	Residual Stress at 25°C (MPa)	Slope in cooling curve (MPa/°C)	Thickness (μm)
PMDA-PDA	−5	−0.052	14
PMDA-ODA	30	0.081	14
BPDA-PDA	7	0.015	10
BPDA-ODA	38	0.094	12

Figure 3 Indentation response of polyimide thin films.

applied load may be due to the change in the crystallinity of the polymer films.[22] For all the polyimide films in this study, the hardness decreased as the penetration depth increased. It indicates that the polyimide films were more crystalline at the surface than in the bulk and become more and more disordered and less crystalline in the bulk in comparison with the surface of polyimide film.

The hardness of the polyimide films was increased in the following order: PMDA-ODA < BPDA-ODA < PMDA-PDA < BPDA-PDA; and the penetration depth, on the other hand, increased in the following order: BPDA-PDA ∼ PMDA-PDA < BPDA-ODA < PMDA-ODA. The hardness of the PDA-based polyimide films is almost twice that of the corresponding ODA-based polyimide films. Also, the PDA-based polyimide films showed relatively higher modulus than the corresponding ODA-based polyimide films. It may indicate that the PDA-based polyimide films have more ordered and crystalline structure than the ODA-based polyimide films.

In general, the film properties were sensitive to the molecular order (packing order) and orientation.[23,24] From a structural point of view, BPDA-PDA and PMDA-PDA have a rodlike structure in repeating unit, whereas BPDA-ODA and PMDA-ODA have hinged and flexible ether group (—O—). The PDA-based polyimides with the rodlike structure are line-

arly connected without a bent structure and a conformation change, and so this could lead to large chain orientation along the polymer chain. In contrast, the ether group of the ODA-based polyimides will impart flexibility and bent structure in polymer chains, leading to change in their morphology.

It is well known that residual stress and mechanical properties of a polymer film are largely determined by its chemical and morphological structures.[15,16] Generally, when a repeating unit of a polymer has a rodlike structure, the toughness and strength should be high because such a structure accompanies large chain orientation along the polymer chain. In contrast, the bent or hinged structure may weaken the intermolecular interactions among polymer chains and prevent effective molecular packing.

Morphological structure

Polyimide films with different chemical fragments may have different conformational freedom in main chains, consequently leading to the inherent change in the microstructure or morphology of the polyimide films. For morphological structures of polyimide thin films, WAXD and refractive index measurements were performed. Transmission and reflection WAXD results are shown in Figure 4. Here, the transmission pattern gives a structural information for the film plane, whereas the reflection pattern does a structural information for the out-of-film plane.[12,24]

The PDA-based polyimide thin films showed (00*l*) diffraction peaks in the transmission pattern corresponding to the molecular order along the chain axis. In contrast, (00*l*) diffraction peaks in the transmission patterns were not apparent for ODA-based polyimide thin films. PMDA-ODA showed only one sharp (002) diffraction peak at a low angle of $2\theta = 5.74°$ and BPDA-ODA showed multiple diffraction peaks on the big amorphous halo peak in the diffraction patterns over the angle range of 10° to 30°.

TABLE II
Nanoindentation Results of Polyimide Thin Films

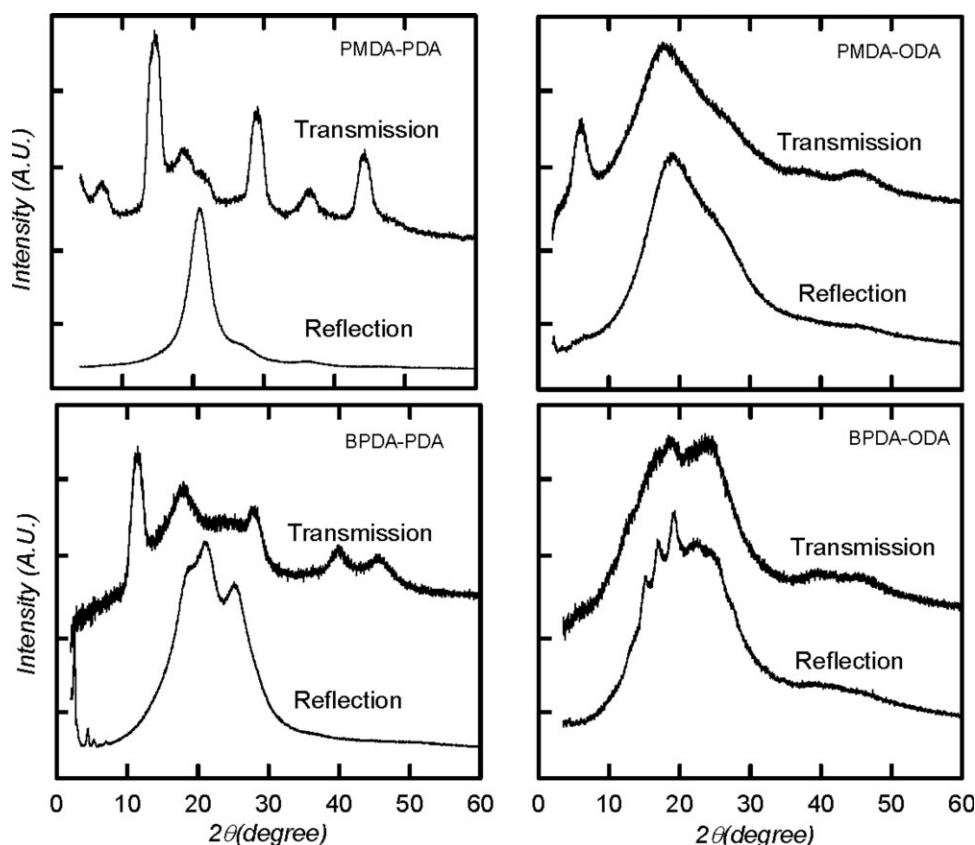| Polymer	Load (m*N*)	Penetration depth (nm)	Modulus (GPa)	Hardness (GPa)
PMDA-PDA	0.087	88	6.87	0.681
	0.127	112	6.75	0.623
	0.162	127	6.82	0.618
PMDA-ODA	0.087	130	3.82	0.273
	0.127	167	3.78	0.268
	0.162	183	3.76	0.248
BPDA-PDA	0.089	86	6.75	0.749
	0.126	107	6.68	0.709
	0.162	123	6.67	0.687
BPDA-ODA	0.086	103	5.77	0.453
	0.124	129	5.68	0.420
	0.159	151	5.63	0.386

Figure 4 WAXD patterns of the different backbone structures of polyimide thin films.

The multiple (00*l*) peaks in the PDA-based polyimide films indicate that these polyimide films have more enhanced molecularly ordered phase than the corresponding ODA-based polyimide film.

The reflection patterns are quite different from the transmission patterns as shown in Figure 4. In the reflection WAXD patterns, PMDA-PDA and PMDA-ODA exhibited only one amorphous halo peak, whereas BPDA-PDA and BPDA-ODA showed several peaks in the reflection pattern. BPDA-PDA showed three (*hkl*) peaks, such as (110), (200), and (210), and BPDA-ODA showed multiple diffraction peaks on the big amorphous halo peak in diffraction patterns over the angle range of 10° to 30° (2θ). It indicates that PMDA-PDA and PMDA-ODA have poor packing order.

The mean intermolecular distance in the polyimide films was calculated from the characteristic transmission/reflection peaks as shown in Table III. The mean intermolecular distance for the transmission pattern varied in the range of 4.68–4.72 Å and those for the reflection pattern did in the range of 4.33–4.66 Å, respectively. The mean intermolecular distance was relatively higher in PMDA-ODA than in PMDA-PDA, indicating that polymer chains of PMDA-PDA polyimide film are more or less well oriented in film plane. From the WAXD patterns and intermolecular dis-

tance, PMDA-PDA with the higher chain rigidity exhibits higher molecular in-plane orientation in film despite its poor packing order.

As described earlier, the molecularly ordered phase was enhanced with increasing order PMDA-ODA < BPDA-ODA < BPDA-PDA ~ PMDA-PDA. That is, the PDA-based polyimide films with relatively rigid phenyl structure have higher population of more ordered phase than do the corresponding ODA-based polyimide films with flexible ether linkage. From these results, it is suggested that the variation in the molecular order and orientation, which were caused by the chain rigidity, are one of the major factors to residual stress behaviors and mechanical properties.

The molecular in-plane orientation of the polyimide thin films was further investigated using the prism coupler. As shown in Table III, all the polyimide thin films showed larger in-plane refractive index (n_{xy}) than out-of-plane refractive index (n_z), regardless of the chemical structure. This indicates that the polyimide thin films had positive birefringence and the polymer chains are preferentially aligned in the film plane. In comparison, birefringence (= $n_{xy} - n_z$), indicating the in-plane orientation of polymer films increases in the following order: PMDA-PDA ~ BPDA-PDA > PMDA-ODA ~

Mean Intermolecular Distances and Refractive Indices of Polyimide Thin Films

Polymer	Mean intermolecular distance[a] (Å)		Optical properties at 632.8 nm			
	In-plane[b]	Out-of-plane[c]	In-plane refractive index (n_{xy})	Out-of-plane refractive index (n_z)	Average refractive index (n_{av})[d]	Birefringence[e]
PMDA-PDA	4.68	4.33	1.8242	1.5826	1.7437	0.2416
PMDA-ODA	4.71	4.66	1.7311	1.6302	1.6974	0.1009
BPDA-PDA	4.72	4.66	1.8507	1.6147	1.7720	0.2360
BPDA-ODA	4.72	4.66	1.7550	1.6720	1.7273	0.0830

[a] Calculated by Bragg's law.
[b] Calculated from the peak maximum of amorphous halos in the transmission WAXD patterns.
[c] Calculated from the peak maximum of amorphous halos in the reflection WAXD patterns.
[d] $n_{av} = (2n_{xy} + n_z)/3$.
[e] Birefringence $= n_{xy} - n_z$.

BPDA-ODA. The PDA-based polyimide films showed relatively higher n_{xy} and n_{av} than the corresponding ODA-based polyimide films, which indicates that the PDA-based polyimide films are preferentially aligned in the film plane. These results are well consistent with the WAXD results.

Thermal properties

In addition to the molecular order and orientation, the chain mobility should be considered as a key factor to influencing in the stress development induced by thermal mismatch according to imidization temperature.[15,16] The chain mobility is directly related to the glass transition temperature, which is a measure of chain flexibility. The higher chain flexibility leads to the lower glass transition temperature, providing the higher chain mobility. In other words, the temperature and magnitude of the mechanical relaxation are critically dependent on the chain rigidity, chain order, and cooperativity of the segmental motion. For the different structure polyimide thin films, the dynamic mechanical properties (storage modulus and tan δ) are depicted in Figure 5 and compared them over the temperature range of 25–500°C.

PMDA-PDA did not show glass transition over 50–500°C, indicating that its glass transition temperature is higher than 500°C,[12] whereas the PMDA-ODA showed a relatively sharp glass transition over the range of 350–400°C. BPDA-PDA showed relatively broad glass transition at a wide temperature range of 320–400°C, whereas BPDA-ODA did show a relatively sharp T_g transition around 310°C. Because of the hinged nature, BPDA-ODA and PMDA-ODA are more flexible than the other two linear polyimides. The ether groups impart more flexibility to the system when compared with rodlike linear groups and hence PMDA-PDA has the highest tan δ value. The glass transition temperature increased in the order of BPDA-ODA < PMDA-ODA ~ BPDA-PDA <

PMDA-PDA. However, the magnitude of tan δ is the same order and higher in the ODA-based polyimides than in the corresponding PDA-based polyimides. It indicates that the ODA-based polyimides are more mobile, which may come from the flexible ether group in the ODA-based polyimides.

From the WAXD patterns, the in-plane orientation and molecularly ordered phase was enhanced with increasing order PMDA-ODA < BPDA-ODA < BPDA-PDA ~ PMDA-PDA. The PDA-based polyimides, PMDA-PDA and BPDA-PDA, showed relatively well-developed morphological structure than the corresponding ODA-based polyimide films,
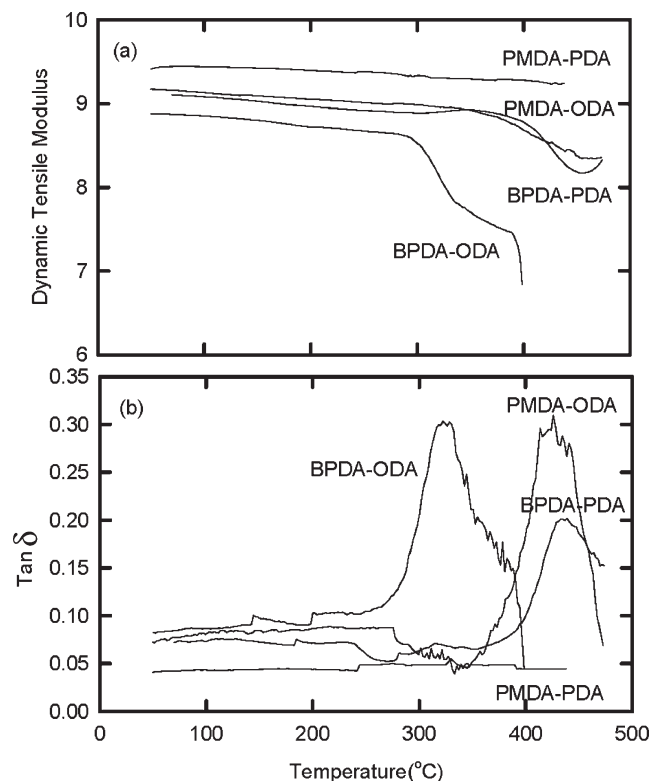

Figure 5 DMTA data of polyimide thin films: (a) storage modulus and (b) tan δ.

which gives less change in temperature variation and applied load. The PDA-based polyimides, having relatively rigid structure, showed relatively well-developed morphological structure (i.e., higher in-plane orientation and higher degree of crystalline structure) than the corresponding ODA-based polyimides. This may cause relatively less change (i.e., lower residual stress and higher hardness) in the PDA-based polyimide films.

CONCLUSIONS

For four different backbone structure polyimide thin films, PMDA-PDA, PMDA-ODA, BPDA-PDA, BPDA-ODA, their residual stress behaviors and mechanical properties were studied and interpreted with the morphological structure in polyimide films. The residual stress was in the range of −5 to 38 MPa. The hardness of the polyimide films was increased in the following order: PMDA-ODA < BPDA-ODA < PMDA-PDA < BPDA-PDA. The PDA-based polyimides, PMDA-PDA and BPDA-PDA, showed relatively lower residual stress and higher hardness than the corresponding ODA-based polyimides, PMDA-ODA and BPDA-ODA. The PDA-based polyimides, having a rigid structure, showed relatively better-developed morphological structure than the corresponding ODA-based polyimides. In conclusion, from the WAXD patterns, refractive indices, and thermal properties, it was concluded that the morphological structure (i.e., film orientation, packing order, and crystallinity) of polyimide films is strongly dependent upon the chemical backbone structure, which may impart the residual stress behaviors and hardness/modulus in polyimide thin films.

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