The Effect of Curing History on the Residual Stress Behavior of Polyimide Thin Films

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ABSTRACT: The effect of curing history on the residual stress behaviors in semiflexible structure poly(4,4′-oxydiphenylene pyromellitimide) (PMDA–ODA) and rigid structure poly(*p*-phenylene biphenyltetracarboximide) (BPDA–PDA) polyimide was investigated. Depending upon the curing history and different structures of polyimide, the residual stress behaviors and the morphology of polyimide thin films were detected *in situ* by using a wafer bending technique and wide angle X-ray diffraction (WAXD), respectively. For the rigid structure BPDA–PDA polyimide, the residual stress and the slope decreased from 11.7 MPa and 0.058 MPa/°C to 4.2 MPa and 0.007 MPa/°C as the curing temperature increased, and the annealing process is done. However, for the semiflexible structure PMDA–ODA, the change of the residual stress and the slope was relatively not significant. In addition, it was found that the cured polyimide prepared at a higher temperature with a multistep curing process showed a higher order of chain in-plain orientation and packing order than does the polyimide film prepared at a lower temperature with a one-step curing process. These residual stress behaviors of polyimide thin films show good agreement with WAXD results, such as polyimide chain order, orientation, and intermolecular packing order, due to curing history. Specifically, it shows that the effect of curing history on residual stress as well as morphological change was significant in rigid BPDA–PDA polyimide but, not in semiflexible PMDA–ODA polyimide. Therefore, it suggests that the morphological structure depends upon curing history, and the polyimide backbone structure might be one of important factors to lead the low residual stress in polyimide thin films. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 74: 3287–3298, 1999

Key words: residual stress; curing history; morphology; the degree of orientation

INTRODUCTION

High-temperature polyimides are widely used in the microelectronics industry as interlayer dielectrics and passivation layers, owing to excellent properties, such as good mechanical properties, high thermal stability, low dielectric constant, and high chemical resistance.[1–3] Despite their inherent attractiveness, there are many problems associated with polyimide usage. It is commonly known that for the multilayered device, there are often reliability problems, such as displacement, cracks, and delamination at the interface.[4–6] Interfacial stress generation is linked to potential reliability problems in integrated circuits, such as loss of adhesion and dimensional stability due to the mismatch of physical properties between layers. Thus, it is desirable to have low stress level materials that are inert to ambient fluctuations in conditions such as temperature and humidity.

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It is well known that imidization of polyimides under thermal imidization process is completed by about 250°C.[7] However, the optimal properties of the polyimide thin films do not develop until the cure has reached 400°C. In addition, mechanical drawing and thermal annealing can substantially modify the thermal expansion properties and geometrical distribution of the amorphous component. In both of these, there is a tendency to orient and arrange the fully cured polyimide thin films. Although the structure is developed in the annealing process, it is a difficult phenomenon to observe and understand because it combines the effect of development of order with the problem of molecular relaxation in a glassy and near-glassy system. The changes are slow and often not reversible. In addition, the curing of polyimide films involves the removal of solvent and chemical reaction, both the solvent and the reaction products of the imidization process.

In multilayer structure, residual stress is primarily dependent upon the thermal expansion coefficient and process history of the layers. Therefore, the matching of these properties for the interlayers is necessary to minimize the generation of stress. The thermal expansion coefficient of a polymer film is strongly dependent on the polymer chain rigidity as well as molecular orientation. Here, controlling the thermal expansion coefficient of the polymer through the modification of the polymer backbone chemistry and molecular orientation can minimize the stress at interface of polymer and substrate.

The degree of orientation in the spin-cast films is suspected to depend on curing conditions, solvent systems, and polyimide chain characteristics. An important question raised in many applications concerns the interrelationships between polyimide chemistry, microstructure, and residual stress behavior. In order to predict the behavior of interlayer materials and to optimize their fabrication, it is important to understand this relationship between polyimide structures and properties as they related to residual stress because environmental parameters and curing conditions, including annealing step, would very significantly affect to the morphology of polyimide.

In this work, hinged poly(4,4′-oxydiphenylene pyromellitimide) (PMDA–ODA) and linear poly(*p*-phenylene biphenyltetracarboximide) (BPDA–PDA) polyimide were prepared by different thermal imidization history in the range of 25–400°C. The idea is to develop different degree of packing, chain orientation, or crystallinity in fully cured polyimide thin films. The developed structure may have a significant impact on residual stress behavior in polyimide films. The residual stress behaviors induced by thermal mismatch with substrate were investigated in detail by using a thin film stress analyzer (TFSA). Molecular order and chain orientation in the films was characterized by Wide Angle X-ray Diffraction (WAXD). These are understood, together with morphological change, such as polymer chain rigidity and chain order and curing history.

EXPERIMENTAL

Materials and Sample Preparation

The poly(amic acid)s (PAAs) were synthesized in normal methyl pyrollidone (NMP) from the following respective dianhydrides and diamines: poly(4,4-oxydiphenylene pyromellitamic acid) (PMDA–ODA PAA) and poly(*p*-phenylene biphenyltetracarboxamic acid) (BPDA–PDA PAA).[7,8] For all the one-side polished Si (100) wafer of 3-in diameter used in the study, the curvatures were measured with the aid of a thin film stress analyzer (TFSA). Then, adhesion promoter solution, *r*-aminopropyltriethoxysilane solution (0.1 vol %) in ethanol–deionized water, was used prior to the spin-coating of polyimide on the Si (100) substrates and baked at 100°C for 30 min on the hot plate. The thin films of polyimide were prepared by spin-coating and soft-baked at 80°C in a convection oven. The dynamic residual stress behaviors of polyimide thin films were investigated by using the TFSA.

Residual Stress Measurement

Dynamic residual stress measurements were performed during thermal imidization of the soft-baked precursor films on Si wafers by using the TFSA. Here, the imidization was performed with a one-step and multistep curing process with a ramping rate of 2.0°C/min as shown in Figure 1 and Table I. The schematic bending apparatus, TFSA, was set up and used, as shown in Figure 2. Polyimide thin films (PMDA–ODA and BPDA–PDA) were in the thickness range from 9–11 μm to exclude the effect of thickness in polyimide.

To determine the residual stress from the measured radii of curvature, a well-known eq. (1) by

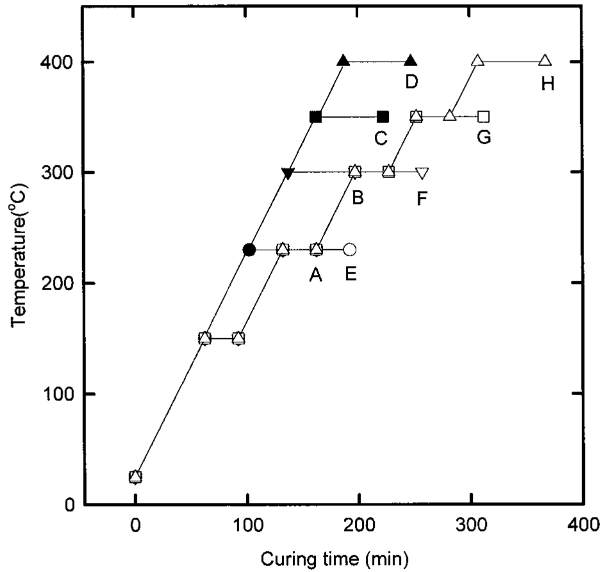

Figure 1 Curing sequences for the residual stress experiment.

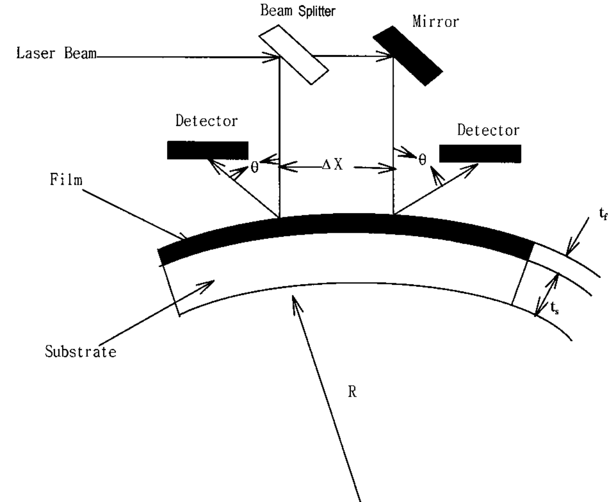

Figure 2 The schematic of the bending wafer apparatus.

Timoshenko was employed.[9] The residual stress was calculated from the measured radii of curvature using the following equation:

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

where σ is the residual stress in the polyimide film. The subscripts f and s denote the polyimide film and the substrate. E, v, and t are Young's modulus, Poisson's ratio, and the thickness, respectively. R_1 and R_2 are the wafer curvatures measured before and after the film deposition. For Si (100) wafer, $E_s/(1 - \nu_s)$ is 180500 MPa.[10] Before use, the initial curvature and the thickness of wafers were measured by using the TFSA and the thickness gauge (SM1201, Teclock Co., Japan), respectively. In addition, the residual stress of a polymer film on a wafer may relax due to moisture absorption over range of 25–60°C in cooling process.[11] Thus, the residual stress behavior due to thermal history after cure was considered from final cure temperature to 60°C to exclude the effect of stress relaxation by the water molecules.

The thermal mismatch between polyimide films and Si substrate were estimated during cooling process from final cure temperature to 25°C by the curvature change of substrate by using the TFSA. On thermal curing cycling for the polyimide film, the difference of the film stresses at any two given temperatures can be attributed to the mismatch in the thermal expansion coefficients between the polymer film and the silicon

Table I Temperature–Time Curing Cycles for Polyimide Thin Films

Thermal Imidization Process	Imidization Schedule with 2.0°C/min Ramping Rate per Step[a]
A	230°C/1 h
B	300°C/1 h
C	350°C/1 h
D	400°C/1 h
E	150°C/30 min + 230°C/1 h
F	150°C/30 min + 230°C/30 min + 300°C/1 h
G	150°C/30 min + 230°C/30 min + 300°C/30 min + 350°C/1 h
H	150°C/30 min + 230°C/30 min + 300°C/30 min + 400°C/1 h

[a] After imidization, the cooling rate of each process was 1°C/min.

Table II Residual Stress for Polyimide Thin Films Prepared by Different Curing Processes

	Curing Process	Thickness (μm)	Final Cure Temperature (°C)	Slope in Cooling Curve (MPA/°C)	Residual Stress (MPA) at 50°C
BPDA–PDA	A	10	230	0.058	11.7
BPDA–PDA	B	10	300	0.032	7.5
BPDA–PDA	C	10	350	0.019	6.3
BPDA–PDA	D	9	400	0.015	6.0
BPDA–PDA	E	10	230	0.045	9.8
BPDA–PDA	F	10	300	0.024	5.3
BPDA–PDA	G	10	350	0.015	5.2
BPDA–PDA	H	9	400	0.007	4.2
PMDA–ODA	A	11	230	0.123	37.7
PMDA–ODA	B	9	300	0.089	31.1
PMDA–ODA	C	10	350	0.086	30.5
PMDA–ODA	D	10	400	0.081	29.5
PMDA–ODA	E	11	230	0.112	37.5
PMDA–ODA	F	11	300	0.087	30.8
PMDA–ODA	G	11	350	0.082	29.9
PMDA–ODA	H	10	400	0.079	29.3

substrate.[12,13] It can be expressed as follows in eq. (2):

$$\Delta\sigma_t/(T_f - T) = \frac{E_f}{(1 - v_f)}(\alpha_s - \alpha_t) \qquad (2)$$

Where, $\Delta\sigma/(T_f - T)$ is the slope of the cooling process curve, which means the residual stress change in the polymer film per temperature variation. E_f and v_f are the relaxation modulus and Poisson's ratio of the polymer film, respectively. T_f and T are the final temperature of fully cured polyimide film and the temperature when radius is measured, respectively. When the biaxial modulus of the film is known, its thermal mismatch between polymer film and silicon wafer due to different thermal expansion coefficient can be obtained from the slope of the resulting curve of stress versus temperature by eq. (2). In addition, WAXD was used for characteristics of molecular order and orientation in these polyimide films. WAXD patterns were collected using the θ–2θ method in the reflection and transmission mode over $3.5° < 2\theta < 60°$. The measured X-ray diffraction (XRD) intensities were corrected by the background run and then normalized for the film samples by matching the integrated intensity over the range of 58–60° (2θ).[14,15] The X-ray generator, CuKα radiation sources (λ = 1.54 Å) was run at 35 kV and 40 mA.

RESULTS AND DISCUSSION

The effect of curing history on the residual stress behaviors of the linear structure BPDA–PDA and hinged structure PMDA–ODA polyimides was investigated in this study. The thickness of polyimide films was controlled by approximately by 10 μm (9–11 μm) to minimize the effect of thickness deviation. Eight different imidization protocols for the polyimide curing process are given in Table I and Figure 1.

The residual stress behaviors with curing temperatures were significantly different depending upon the linear structure BPDA–PDA and the hinged structure PMDA–ODA polyimides, as shown in Table II.

The curing steps were reflected in the stress–temperature profile. The resulting residual stresses, as well as the slopes of cooling curve, mean the thermal mismatch between polyimide thin film and substrate during curing process. As shown in Figures 3 and 4, the residual stress behaviors of BPDA–PDA polyimide thin film are investigated, depending upon the various curing schedules.

For the BPDA–PDA polyimide films imidized by one-step curing processes, the residual stress and the slope of cooling curve were decreased with increasing curing temperature from 11.7 MPa and 0.058 MPa/°C at 230°C to 5.15 MPa and 0.015 MPa/°C at 400°C, as shown in Table II. Depending upon multistep curing processes, the

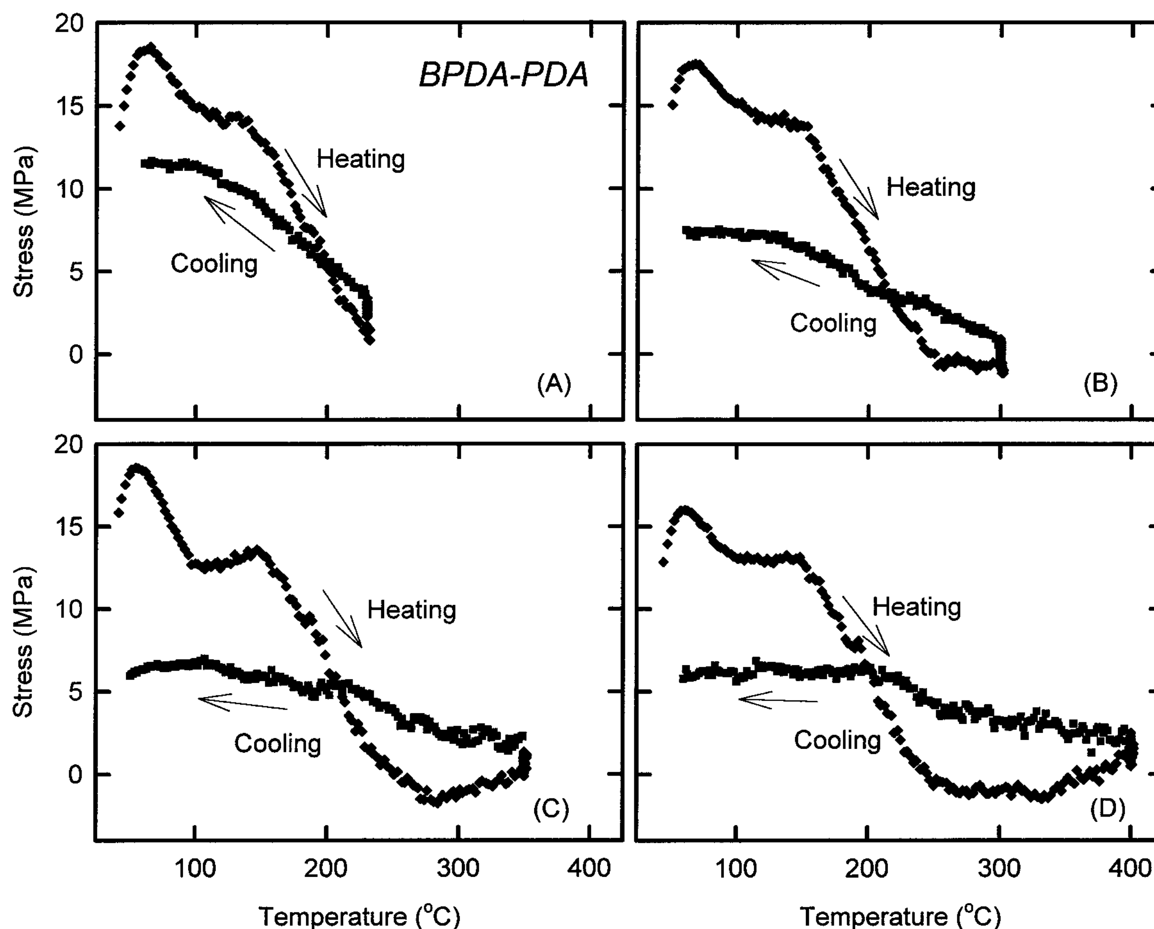

Figure 3 Dynamic residual stress behaviors for the BPDA–PDA polyimide with one-step curing processes.

residual stress and the slope of cooling curve were decreased with increasing curing temperature from 7.5 MPa and 0.032 MPa/°C at 230°C to 4.2 MPa and 0.007 MPa/°C at 400°C.

As shown in Figure 3, the highest residual stress and slope at 230°C was observed. It may show that the polyamic acid precursor (PAA) is initially a relatively random coil polyelectrolyte dissolved in a solvent NMP. During curing step, imide groups appear along the chain backbone, transforming the initial polyamic acid homopolymer first into an imide–amic acid copolymer and then ultimately into an imide homopolymer as the curing temperature increase. With conversion, the polymer chain significantly stiffens and the random coil appearance diminishes. It may show that the residual stress and the slope are decreased as the cure temperature increases. It means that the final residual stresses of polyimide thin films were remarkably dependent to final curing temperatures for the imidization of polyimide.

Also, the effect of the multistep curing process on the residual stress and slope of cooling cycle of polyimide was observed, as shown in Figure 4. The residual stress and slope of cooling cycle of polyimide with the multistep curing process was more or less lower than that the polyimide with one-step curing. The residual stress and slope of cooling cycle of BPDA–PDA polyimide decreased remarkably with annealing step process. It may suggest that there is some equilibrium reached in a dynamic process associated with the dwelling temperature. When a higher temperature dwells, certainly the chain mobility may exist, and further molecular rearrangements may be possible during step process. In addition, a higher final curing temperature shows a more significant effect on slope of the cooling curve, as well as residual stress after cures, then the annealing step, as

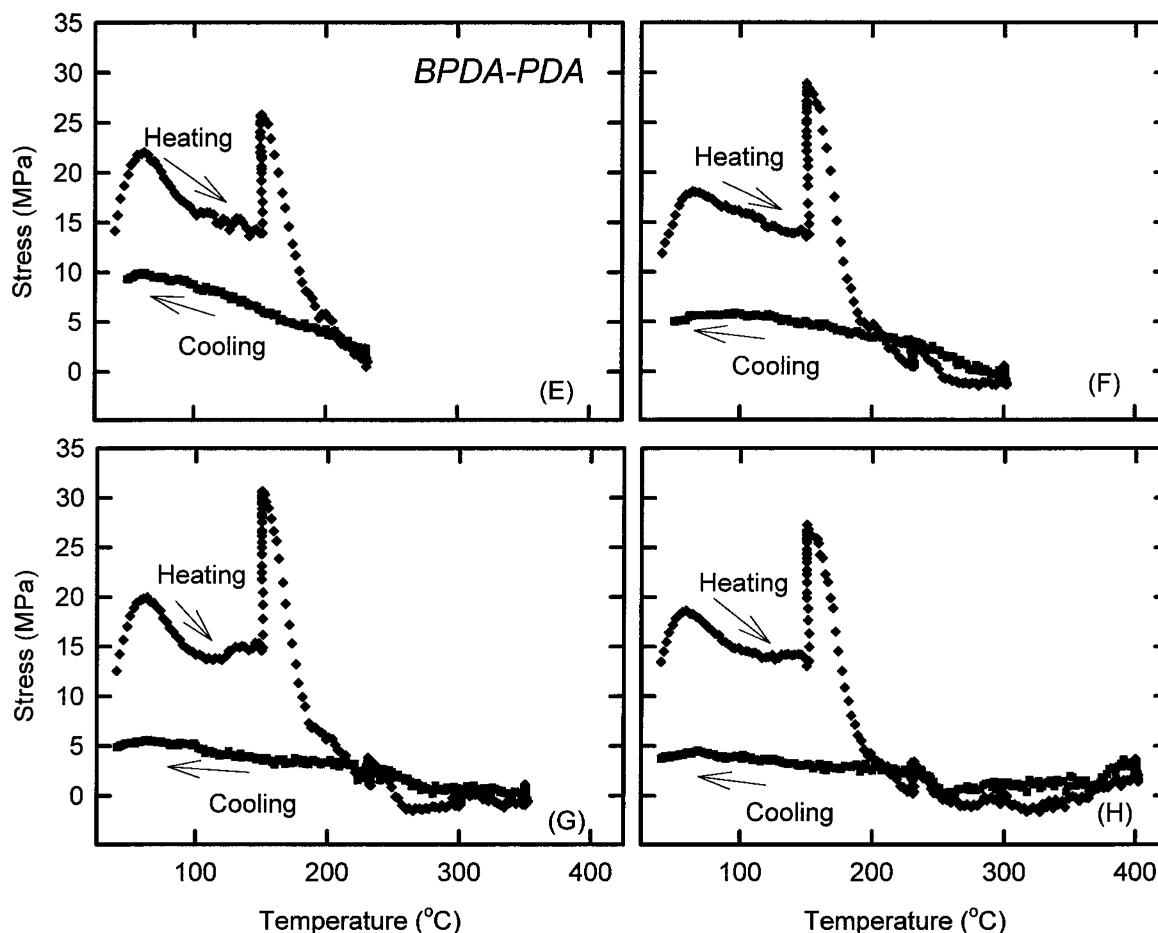

Figure 4 Dynamic residual stress behaviors for the BPDA–PDA polyimide with multistep curing processes.

shown in Figure 5. It suggests that a curing temperature may be one of important factors to lead to a low stress in polyimide thin film, as shown in Figure 5.

In addition, it may suggest that the morphological structure, degree of chain orientation, packing, and crystallinity, are significantly related to the residual stress in polyimide thin film.

The effect of the thermal imidization history on the residual stress behaviors was investigated. WAXD measurement was performed for the BPDA–PDA polyimide film in both transmission and reflection pattern, which can give structural information in the film plane and in the out-of-film plane, respectively. WAXD clearly shows the effect of cure temperature and multistep curing on the degree of chain order in the films, as shown in Figure 6.

The transmission data in Figure 6 show that diffraction peaks, which reflect from the polymer chain order along the chain axis, increase in sharpness as curing temperature increase. The intensity of a diffraction peak is indication of the fraction of the ordered state in a material. These are improved through imidizing at a higher temperature and step curing. Thus, as the cure temperature increases, the increased intensity signifies that more of the BPDA–PDA chains are lining up in an ordered state. For (004) diffraction, the coherence length was estimated from Scherrer equation.[16,17] The coherence length was 105–125 Å, depending on the curing history for the films over imidized over 230–400°C, as shown in Table III. The enhancement in the intensity and shape of the (001) peaks by imidization at higher temperature for step curing might result from an increase in the molecular in plane orientation, as shown in Figure 6.

In contrast to the transmission experiments, the reflection diffraction spectra depend on the

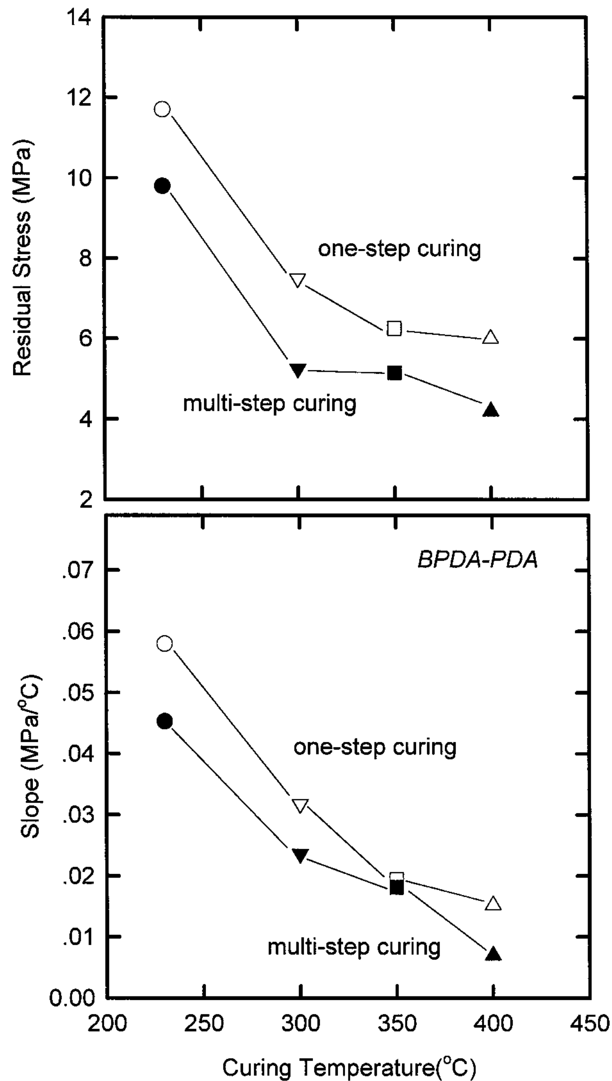

Figure 5 Residual stress behaviors for the BPDA–PDA polyimide films with different curing history.

degree of order in the a and b directions. The dependency of cure temperature and step curing on the molecular packing is clearly evident in the reflection pattern. At low temperatures of curing, there is a broad peak at $2\theta = 20°$ that appear to be a fusion of the (110), (200), and (210). These are very broad and ill-defined peaks in polyimide films with low temperature curing process. It means that the degree of chain order was relatively low in polyimide thin film with low temperature of curing process. As the curing temperature increases, the total area of the diffraction pattern in the reflection mode increases slightly. These diffraction peaks were very much enhanced in intensity and shape for the samples cured with high temperatures above 300°C. The (210) and (200) peaks appeared for the samples imidized above 350°C. Furthermore, for the (110) peak, the position of the peak maximum was slightly shifted in the high 2θ region. It indicates that the molecular packing order in the BPDA–PDA polyimide film has significantly improved by curing at a higher temperature. In addition, the samples that were cured by the multistep process, showed increased intensity of peak in the range of 10–30°, indicating enhancement of intermolecular packing order. Thus, there is increase in the mass of material that is ordered with respect to the a and b directions of the crystal structure. The (110), (200), and (210) reflection peaks are all

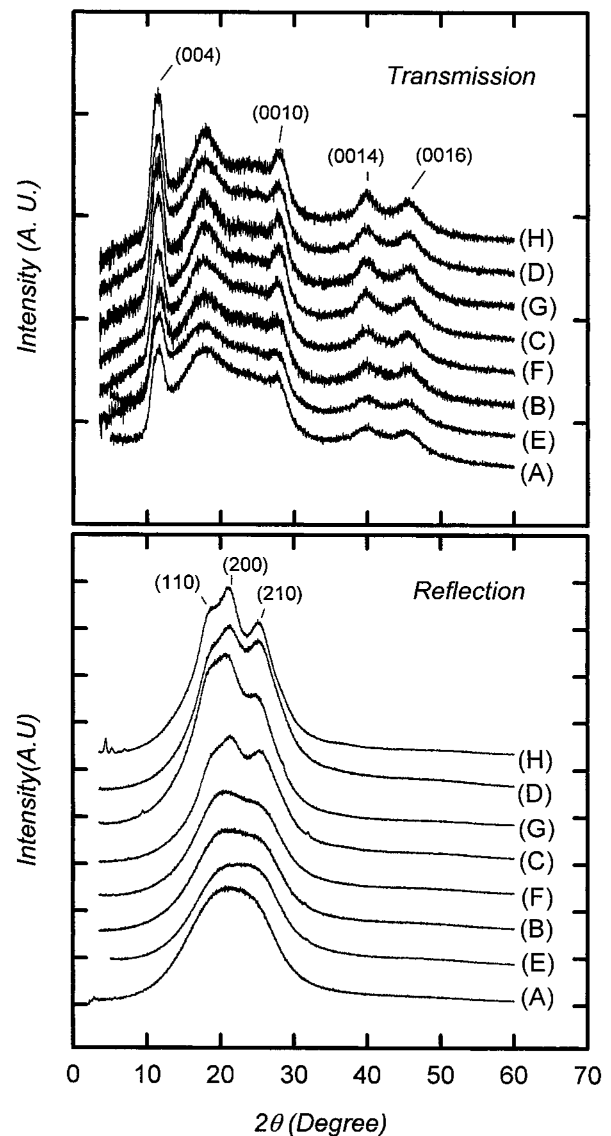

Figure 6 WAXD patterns of the BPDA–PDA polyimide films depending upon different curing histories.

Table III Change in the (001) Peak of Polyimides with Different Curing Histories

	Curing Process	Final Cure Temperature (°C)	d-Spacing (Å)	Coherence Length (Å)
BPDA–PDA	A	230	7.46	105
BPDA–PDA	B	300	7.60	115
BPDA–PDA	C	350	7.80	118
BPDA–PDA	D	400	7.87	123
BPDA–PDA	E	230	7.51	106
BPDA–PDA	F	300	7.72	118
BPDA–PDA	G	350	7.87	120
BPDA–PDA	H	400	7.96	125
PMDA–ODA	A	230	14.48	71
PMDA–ODA	B	300	14.72	72
PMDA–ODA	C	350	14.78	74
PMDA–ODA	D	400	15.12	75
PMDA–ODA	E	230	14.48	71
PMDA–ODA	F	300	14.75	72
PMDA–ODA	G	350	15.85	74
PMDA–ODA	H	400	15.12	75

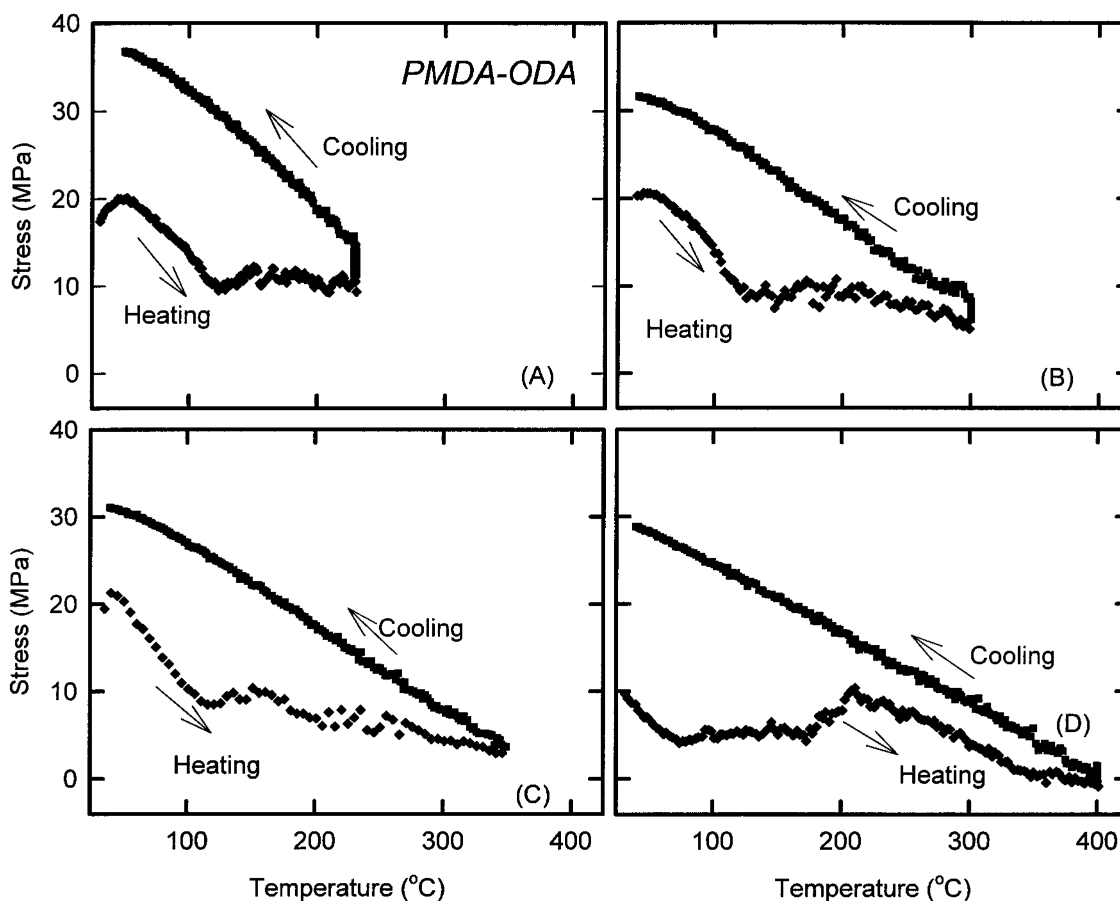

Figure 7 Dynamic residual stress behaviors for the PMDA–ODA polyimide with one-step curing processes.

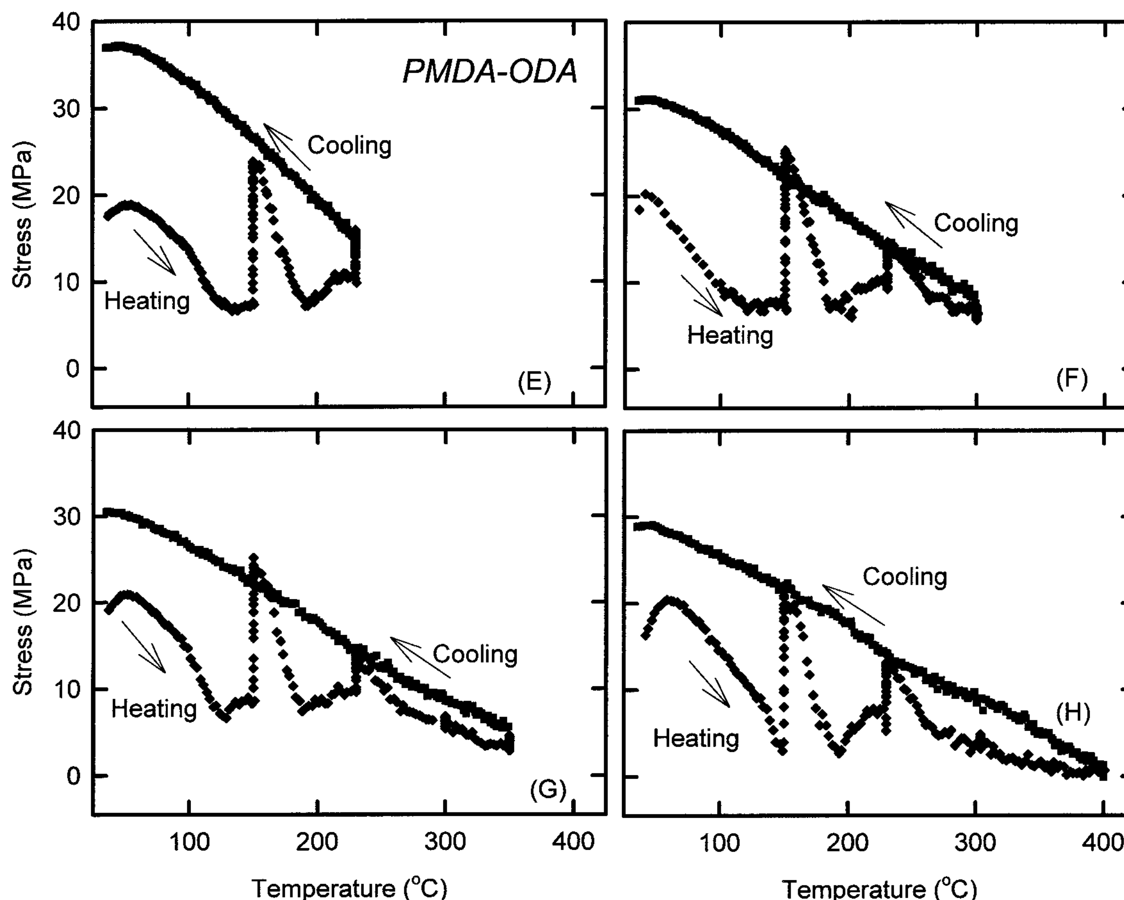

Figure 8 Dynamic residual stress behaviors for the PMDA–ODA polyimide with multi-step curing processes.

indicative of interchain packing density and order. It shows that at a given temperature, the packing order apparently was improved by step curing.

In a comparison of reflection and transmission patterns, the multiple (001) peaks, which present a high degree of the polymer chain order along the chain axis, appeared on the transmission pattern, indicating that polymer chains are highly aligned in the film plane. In transmission pattern, the intensity of the (004) peak, which is a probe of in-plane orientation, was slightly varied by annealing at a chosen step process and, however, significantly increased by the elevation of curing temperature. This result suggests that in-plane orientation could be controlled by imidization temperature than step curing process. Conclusively, the degree of in-plane orientation and the molecular packing order were enhanced by thermal imidization at higher temperature with annealing step process. It shows that there is a good agreement with results of residual stress behaviors of polyimide thin films, as shown in Figure 5.

The flexible PMDA–ODA polyimide was also investigated, depending upon the same curing process. The dynamic residual stress behaviors are analyzed with the various curing conditions, as shown in Table II and Figures 7 and 8.

For the PMDA–ODA polyimide film imidized by one-step curing, the residual stress and the slope of cooling curve decreased from 37.7 MPa and 0.123 MPa/°C at 230°C to 29.5 MPa and 0.081 MPa/°C at 400°C, respectively. For the PMDA–ODA polyimide imidized by multistep curing, the residual stress and slope of cooling curve also decreased from 37.5 MPa and 0.112 MPa/°C at 230°C to 29.3 MPa and 0.079 MPa/°C at 400°C, respectively. It is also clear that the polyimide films imidized at a higher temperature with the step cure showed a lower slope and residual stress than did the polyimide film at imidized lower temperature with one step.

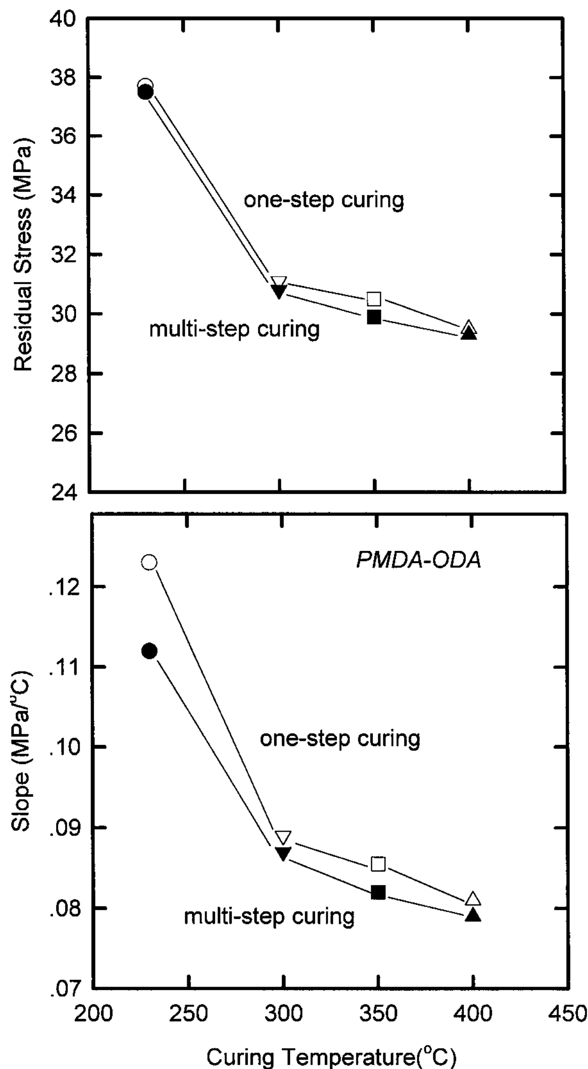

Figure 9 Residual stress behaviors for the PMDA–ODA polyimide film with different curing history.

Figure 9 shows that the residual stress of PMDA–ODA polyimide thin films is continuously decreasing as the curing temperature increase up to 400°C. However, the residual stress behavior of semiflexible PMDA–ODA polyimide thin film with the multistep curing process was not significantly different with that of the PMDA–ODA polyimide with the one-step curing process, as shown in Figure 9. Also, there is a slight but not very significant decrease in the slope of cooling curve and the residual stress with the curing process above 230°C in contrast to the residual stress behavior of BPDA–PDA polyimide. It may indicate that the ODA structure in PMDA–ODA polyimide is inherently more flexible because of its hinged character. Thus, it may not easily align the chain order or alter its morphological change of polyimide by high temperature curing.

Thus, WAXD measurements were performed for the PMDA–ODA polyimide films. As shown in Figure 10 and Table III, the transmission and the reflection diffraction results are presented. The transmission diffraction peak at $2\theta = 8°$ resulted from the polymer chain-order, and its intensity is increased as the curing temperature increase. However, unlike the case of linear structure BPDA–PDA, the intensity and the shape of peaks for the semiflexible PMDA–ODA polyimide does not significantly depend upon the curing history. Furthermore, there is little evidence of specific dif-

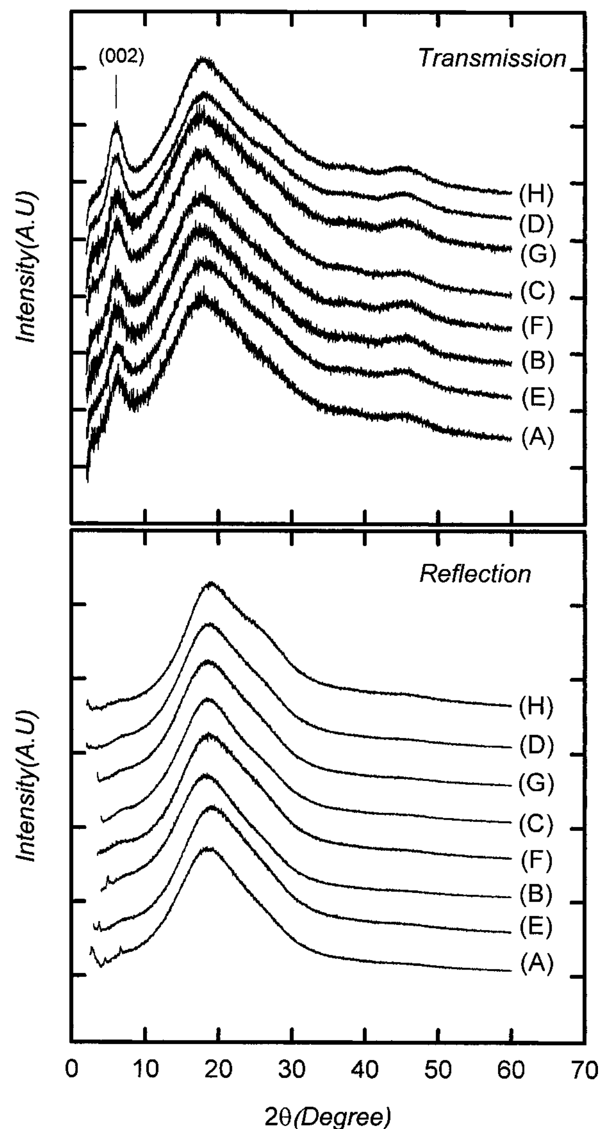

Figure 10 WAXD patterns of the PMDA–ODA polyimide film depending upon different curing history.

fraction patterns characteristic of scattering planes in the in-plane direction, and there is no change in the nature of this broad peak with curing temperature increase, as shown in Figure 10. It means that there is no significant evidence in the reflection spectra of any identifiable diffraction feature that anticipate significant development of a or b axis cell order.

In addition, the low-angle diffraction peak becomes strong and sharp in the transmission pattern of PMDA–ODA polyimide film imidized at higher temperature with a multistep cure. The diffraction peak in the region of 10–40° (2θ) improves slightly but is still relatively broad. For the low-angle peak, the d-space, which corresponds to the projected length of the chemical repeat unit ordered along the chain axis increased from 14.72 Å at 230°C to 15.12 Å at 400°C. Whereas the coherence length determined from its half-width increased from 71 Å at 230°C to 75 Å at 400°C. The reflection patterns, which give structural information in the out-of-film plane, showed a broad peak, regardless of thermal imidization history, as shown in Figure 10. The broad peak is slightly sharpened as the curing temperature and step cure, but the effect was small. It seems that although the chain order in PMDA–ODA can be improved somewhat by annealing at higher temperature, no major departure from the order in polyimide films occurs regardless of curing temperature and annealing step. It may show that the chain rigidity of hinged PMDA–ODA was relatively not sensitive to curing history in comparison to rigid structure BPDA–PDA.

It means that the residual stress behavior of PMDA–ODA was not significantly dependent upon curing history due to its relatively flexible structure. It shows a good agreement with results of residual stress behaviors of PMDA–ODA polyimide thin film, as shown in Figure 9.

CONCLUSIONS

The effect of curing history on the residual stress in semiflexible poly(4,4′-oxydiphenylene pyromellitimide) (PMDA–ODA) and rigid poly(p-phenylene biphenyltetra carboximide) (BPDA–PDA) polyimides was investigated. The residual stress and the slope the rigid structure BPDA–PDA polyimide with increasing curing temperature and the multistep process decreased from 11.7 MPa and 0.058 MPa/°C to 4.23 MPa and 0.007 MPa/°C. The semiflexible structure PMDA–ODA polyimide with increasing curing temperature and multistep process decreased from 37.6 MPa and 0.123 MPa/°C to 29.3 MPa and 0.079 MPa/°C. It is clear that the cured polyimide films at a higher temperature with the multistep curing process show lower residual stress than that does the imidized polyimide film at lower temperature with one-step process.

With WAXD results, the polyimide prepared at high curing temperature and with the multistep process shows a high order of molecular orientation in the plain of the film and a higher packing order than the polyimide film prepared at a lower-temperature curing temperature and with the one-step process. In addition, it shows that the morphological change (chain order, intermolecular packing, and coherence length) of rigid-structure BPDA–PDA was influenced significantly by thermal curing history, whereas that of semiflexible-structure PMDA–ODA was not relatively sensitive. These residual stress behaviors of polyimide thin films show a good agreement with WAXD results, such as polyimide chain order, orientation, and intermolecular packing order, due to curing history.

Therefore, it was found that the effect of curing history on the residual stress behavior is dependent upon the following polyimide backbone chemistry: semiflexible PMDA–ODA and rigid BPDA–PDA. However, the effect of curing history on the residual stress as well as the morphological change was significant in rigid BPDA–PDA polyimide, but not in semi-flexible PMDA–ODA polyimide. It suggests that the morphological structure depending upon the curing history and the polyimide backbone structure might be one of important factors leading to the low residual stress in polyimide thin films.

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REFERENCES

1. Mittal, K. L. Polyimides: Synthesis, Characterization and Application; Plenum: New York, 1984.
2. Wilson, D.; Stenzenberger, H. D.; Hergenrother, P. M. Polyimides; Chapman & Hall: New York, 1990.
3. Bessonove, M. I.; Koton, M. M.; Kudryyavtsev, V. V.; Laius, L. A. Polyimides: Thermally Stable Polymer; Consultants Bureau: New York, 1987; p. 1.

4. Brekner, J.; Feger, C. J Polym Sci: Part A: Polym Chem 1987, 25, 2005.
5. Coburn, J. C.; Pottiger, M. T.; Noe, S. C.; Senturia, S. D. Polymer 1993, 32, 1271.
6. Tong, H. M.; Hu, C. K.; Feger, C.; Ho, P. S. Polym Eng Sci 1970, 26, 1213.
7. Bessonov, M. I.; Koton, M. M.; Kudryavtsev, V. V.; Laius, L. A. Polyimides—Thermally Stable Polymers, Plenum: New York, 1987.
8. Ree, M.; Nunes, T. L.; Chen, K. J.; Czornyj, G. Mater Res Soc Symp Proc 1991, 229, 211.
9. Timoshenko, S. P. J Opt Soc Am 1926, 11, 223.
10. Wortman, J. J.; Evans, R. A. J Appl Phys 1965, 36, 136.
11. Ree, M.; Nunes, T. L.; Czornyj, G.; Volksen, W. Polymer 1992, 33, 1228.
12. Joe, J. H.; Huang, P. T. Macromolecules 1991, 24, 3796.
13. Ree, M.; Chu, C. W.; Goldberg, M. J. J Appl Phys 1994, 75, 1410.
14. Takahashi, N.; Yoon, D. Y.; Parrish, W. Macromolecules 1994, 17, 2583.
15. Han, H.; Gryte, C. C.; Ree, M. Polymer 1995, 36, 1663.
16. Scherrer, P. Nachr Fottinger Gesell 1918, 2, 98.
17. Cullity, B. D. Elements of X-ray Diffraction, 2nd Ed.; Addison-Wesley: Reading, MA, 1978.