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Self-adhesion of poly(4,4′-oxydiphenylene biphenyltetracarboximide) and its adhesion to substrates

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Abstract

Poly(4,4′-oxydiphenylene biphenyltetracarboxamic acid) was synthesized in *N*-methyl pyrrolidone from biphenyltetracarboxylic dianhydride and 4,4′-oxydiphenylene diamine, and then converted to the polyimide in films by thermal imidization at 400°C. The polyimide exhibits an exceptionally good self-adhesion (814–1187 J/m^2) and also good adhesions (>687 J/m^2) to inorganic substrates such as silicon wafer and glass ceramic. An oxygen plasma treatment enhances the self-adhesion highly, whereas a silane primer treatment improves the adhesions to the substrates. In addition, the morphological structure and some other properties were investigated. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Aromatic polyimide; Interfacial adhesion; Residual stress

1. Introduction

The current high technology in the fabrication of memory chips and integrated circuits demands a high performance interdielectric material to have good self-adhesion and adhesion to substrates, low swelling in solvents, low dielectric constant, high temperature thermal stability, and low internal stress/thermal expansion, in addition to good mechanical properties [1,2]. A representative polymer dielectric is poly(4,4′-oxydiphenylene pyromellitimide) (PMDA-ODA) which has reasonably good properties: 3.0 GPa Young's modulus, 170 MPa ultimate tensile strength, 40–110% strain at break, and ca. 40 ppm/°C thermal expansion coefficient (TEC) in the film plane [3–7]. Another representative polymer dielectric is poly(*p*-phenylene biphenyltetracarboximide) (BPDA-PDA) which has high thermal stability, excellent mechanical properties (10.0 GPa modulus, 500 MPa ultimate tensile strength, and 15–35% strain at break), low residual stress, and low thermal expansion [3,8–13]. For the advantageous properties, these polymers have been widely used in the microelectronics industry. However, both of the polymers have very poor adhesion to the polymer itself as well as to substrates such as silicon wafers and ceramics. Further, PMDA-ODA polyimide has relatively high degree of swelling in organic solvents such as *N*-methyl-2-pyrrolidone (NMP), and relatively high degree of moisture uptake [1,7,14–21].

Compared to the BPDA-PDA polyimide, poly(4,4′-oxydiphenylene biphenyltetracarboximide) (BPDA-ODA) has gained relatively less attention because of its relatively inferior properties [22–24]: 4.1 GPa modulus, 230 MPa ultimate tensile strength, and 73–113% strain at break, and 43 ppm/°C TEC. However, these properties still are relatively better than those of the PMDA-ODA polyimide. Recently, we have investigated the glass transition and imidization behaviors of its soluble precursors using oscillating differential scanning calorimetry [25,26].

In this study, BPDA-ODA polyimide was investigated further in the aspect of adhesion characteristic. In fact, a polyimide coating layer is normally interfaced with the polymer itself, metals (mainly, copper and cobalt), and substrates (ceramics, silicon wafers, and glasses) in its applications in microelectronic devices [1]. For microelectronic devices fabricated with polyimide, their reliability is highly dependent upon the stability at the interfaces [1]. For this reason, the self-adhesion of BPDA-ODA polyimide and its adhesion to substrates, such as glass ceramics and silicon

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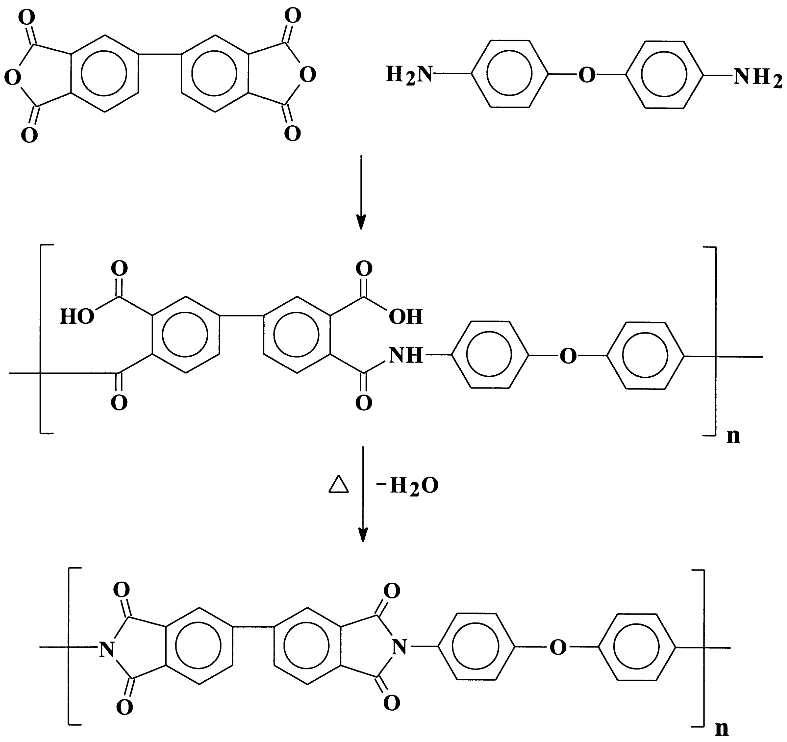

Fig. 1. Synthetic scheme of poly(4,4′-oxydiphenylene biphenyltetracarboxamic acid) and its polyimide.

wafers with oxidized surface, were measured. In addition, the dynamic mechanical properties, residual stress, and swelling in NMP were investigated. The morphological structure was also examined.

2. Experimental

2.1. Polymer and film preparation

Poly(4,4′-oxydiphenylene biphenyltetracarboxamic acid) (BPDA-ODA PAA) precursor was synthesized in NMP from biphenyltetracarboxylic dianhydride and 4,4′-oxydiphenylene diamine according to a conventional polymerization scheme reported previously (see Fig. 1) [25]. For the obtained precursor solution, the solid content was 17.5 wt.% and the intrinsic viscosity was measured to be 0.704 dl/g in NMP at 25.0°C. The BPDA-ODA PAA solution was spin-coated on silicon wafers and glass slides, soft-baked on a hot plate of 80°C for 1 h under nitrogen gas flow, and then thermally imidized in a furnace under ambient nitrogen flow by a step-cure procedure (150°C/30 min, 200°C/30 min, 300°C/30 min and 400°C/1 h). The polyimide films were taken off the substrates by immersion in deionized water, followed by drying in vacuum.

2.2. Self-adhesion and adhesion to substrates

The adhesion characteristics of BPDA-ODA polyimide were investigated at room temperature by 90° peel test using an Instron mechanical tester (Model 1122) equipped with a peel test fixture. Specimens for the peel test were prepared as follows. First, the polyimide precursor solution was spin-coated on substrates such as silicon wafer, glass ceramic, and glass slide, soft-baked on a hot plate of 80°C under slow nitrogen gas flow for 1 h, and then thermally imidized in a furnace under nitrogen atmosphere by the step-cure process as mentioned in the film preparation part. The substrates were cleaned in a Plasmaline 515 asher of Tegal (300 W/5 min and 535 cc/min oxygen flow rate) before the precursor polymer solution was applied. γ-Aminopropyltriethoxysilane (APTES) of 0.1 vol.% in ethanol/water (95/5 in volume) mixture was used for some of substrates as an adhesion promoter. The APTES solution was spin-coated on substrates at 2000 rpm/20 s, followed by baking on a hot plate of 120°C for 20 min in air ambient. The adhesion with APTES primer was compared to that without APTES adhesion primer.

Secondly, for self-adhesion study, the first polymide layer was prepared on the APTES primer-coated glass slide in accordance to the same procedure as in the preparation of samples for the polyimide/substrate adhesion study. The second polyimide layers were prepared in three different ways. That is, the second layer coating of the precursor solution was applied on (i) the first polyimide layer without any further surface treatment, (ii) the first layer ashed at 150 W/4 min and 535 cc/min oxygen flow, and (iii) the first polyimide layer ashed and subsequently treated with the APTES primer solution. The overcoated precursor layers were thermally imidized in the same way as the first polyimide layer was imidized.

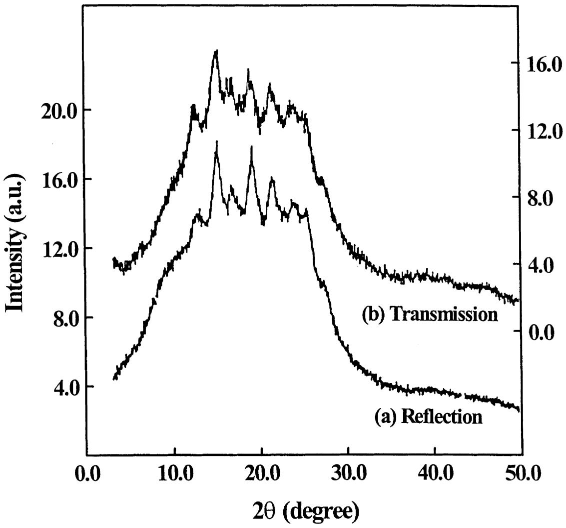

Fig. 2. Wide angle X-ray diffraction patterns of a BPDA-ODA polyimide film thermally imidized at 400°C.

In addition, another adhesion specimen was prepared with the aid of poly(4,4′-oxydiphenylene pyromellitamic acid) (PMDA-ODA PAA) in order to compare with the three different samples prepared above. Namely, the BPDA-ODA PAA solution was first spin-coated over the first BPDA-ODA polyimide layer without any surface treatment, soft-baked at 80°C for 30 min, and partially cured at 150°C for 30 min. Then, the overcoating of PMDA-ODA PAA solution was subsequently performed and soft-baked at 80°C for 1 h. Finally, the whole layers of BPDA-ODA PAA and PMDA-ODA PAA were imidized together by the same imidization process described above. The prepared adhesion specimen has a layer structure of PMDA-ODA(18 μm)/BPDA-ODA(2 μm)//BPDA-ODA/APTES/glass slide. The films on glass slide substrates were diced on a dicer in 2.0–5.0 mm width for the peel test. In the peel test, the cross-head speed was 0.05 to 5 mm/min. The total thickness of peeling polyimide layer was 20–24 μm.

2.3. Measurements

The wide angle X-ray diffraction (WAXD) study was performed for the thermally imidized BPDA-ODA polyimide films at room temperature, using a diffractometer (Model D-500, Siemens). The X-ray source was Cu K_α radiation which was generated at 40 kV and 30 mA. The WAXD measurement was carried out in both transmission and reflection geometry. The 2θ scan was performed at 0.05° step interval and 12 s collecting time over the range 3–50° in the $\theta/2\theta$ mode. In the measurement, several layers of films, rather than a single layer film, were mounted on a film sample holder. The total film thickness was ca. 200 μm. The measured WAXD patterns were corrected to the background runs and then deconvoluted. Here, an interactive curve-fitting technique based on a nonlinear-least-squares estimation algorithm was used to fit a collection of pseudo-Voight functions and one linear baseline to the WAXD profile corrected [27,28]. From integrated areas of the deconvoluted peaks, a crystallinity was estimated.

The dynamic mechanical properties (storage modulus E', loss modulus E'', and tan δ) were measured as a function of temperature using a dynamic mechanical thermal analyzer with a tension probe (Mark-I, Polymer Laboratories). The measurement was conducted with a nitrogen gas flow over the range of room temperature to 500°C. The heating rate and frequency were 10.0 K/min and 10 Hz, respectively. The width of the film strips and grip gauge length were 6.350 and 15 mm, respectively. The thickness of films was 20–25 μm.

The residual stress of BPDA-ODA polyimide film was measured by means of silicon wafer bending technique using a specially designed X-ray diffractometer [29]. The curvature of both side polished Si(1 0 0) wafers (397.5 μm thick) was measured before applying the polymer solution. Then, the APTES primer solution was applied on the calibrated silicon wafers at 2000 rpm for 20 s and baked on a hot plate of 120°C for 20 min in ambient air. Subsequently, the precursor solution was spin-coated on the primer-coated wafers, soft-baked on a hot plate of 80°C for 1 h, and thermally imidized in an oven with nitrogen flow by the step-cure process. Then, the curvature of the polymer-coated wafer was again measured to determine the stress of the polyimide film.

For the polyimide films, the swelling measurements were performed in NMP by means of gravimetry at both room temperature and 80°C. The thickness of films was ca. 20 μm.

3. Results and discussion

3.1. Structure

Fig. 2 shows WAXD patterns obtained for the BPDA-ODA polyimide film. The transmission pattern shows a typical crystalline polymer WAXD spectra as well as a large amorphous halo. A similar WAXD pattern was obtained in the reflection geometry, indicating that the BPDA-ODA polyimide chains in the film are aligned randomly rather than in the film plane. The (0 0 2) and (0 0 4) diffraction are presumably assigned to peaks at ca. 6.3° (14.02 Å) and 12.5° (7.08 Å), respectively. The peak at 15.3° (5.79 Å) is too sharp to be an amorphous halo. Thus, this peak may be assigned by the (1 1 0) diffraction. Besides these peaks, several sharp and broad peaks appeared in the high angle region of >16.3°: the peaks are expected to be diffraction peaks from (0 0 *l*) and (*h k l*) diffraction. This WAXD result indicates that BPDA-ODA polyimide exhibits the molecular chain orientation along the chain axis as well as relatively good lateral packing order. However, the amorphous halo is relatively very big. Crystallinity is estimated to be only ca. 17% from the X-ray diffraction

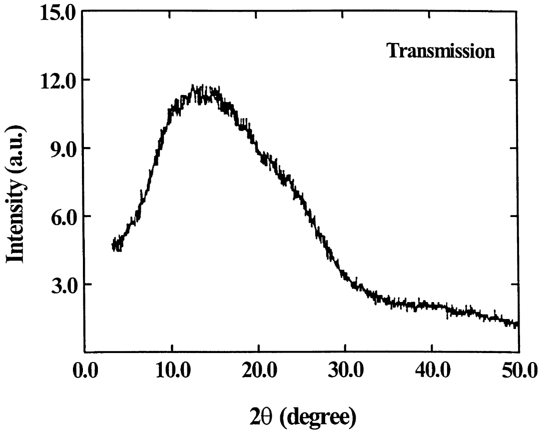

Fig. 3. Wide angle X-ray diffraction pattern of a Upilex-R® film, 75 μm thick.

patterns. The crystal structure has not been determined yet but its structural refinement analysis is in progress in our laboratory so that the result may be published in future.

In addition, a Upilex-R® film, 75 μm thick, which is a BPDA-ODA polyimide film supplied from Ube Chemicals in Japan, was characterized by X-ray diffraction. In general, it has been well known that commercial polyimide films are chemically imidized and subsequently stretched biaxially during heat treatment [22,30,31], perhaps leading to a different morphological structure in the Upilex-R® film. A typical X-ray pattern of Upilex-R® films is shown in Fig. 3. This X-ray pattern is quite different from that of the thermally imidized film aforementioned. The film shows only amorphous halos: that is, peaks from any molecular orientation and lateral order do not appear. Here, an average intermolecular distance is estimated to be ca. 6.4 Å from the peak maximum position of the big amorphous halo. The WAXD results indicate that the polymer chains in the Upilex-R® film are in the amorphous state rather than an ordered state.

3.2. Properties

Fig. 4 shows dynamic mechanical and thermal spectra obtained for the BPDA-ODA polyimide film. These spectra are very similar to those reported previously [22,23]. The storage modulus (E') gradually decreases from room temperature to 290°C, drops sharply through the first phase transition, and then gradually decreases again with further elevating temperature. The E' sharply drops again through the second phase transition at >450°C. That is, different from other conventional aromatic polyimides, BPDA-ODA polyimide exhibits two phase transitions. Through the first transition, the modulus was dropped from 2.15 GPa at 290°C to 0.531 GPa at 365°C. The modulus decrease at this transition seems like a big drop relatively to provide enough molecular chain mobility. This kind of chain mobility is reflected directly to the self-adhesion. For the bilayer film specimens prepared at $T_1 = T_2 = 400$°C (here, T_1 and T_2 are imidization temperatures for the first and second polyimide layers), an excellent adhesion was observed in the peel test. This means that a reasonable degree of chain mobility has taken place at 400°C to allow molecular inter-diffusion through the first and second polymer layers and has resulted in good self-adhesion. The detailed adhesion behavior is discussed later.

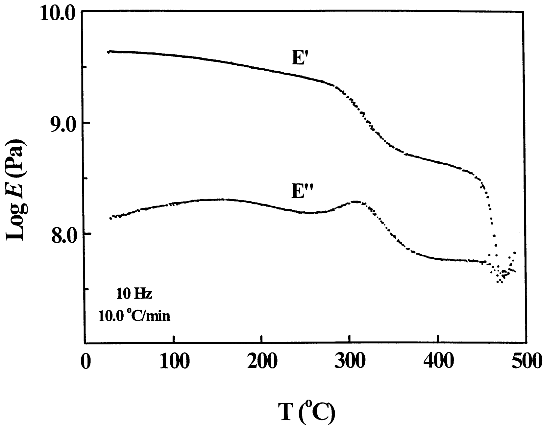

Fig. 4. Variations of dynamic moduli (storage modulus E' and loss modulus E'') of BPDA-ODA polyimide film as a function of temperature. The employed frequency and heating rate were 10 Hz and 10.0 K/min, respectively.

After the first transition, the modulus leveled off and then gradually decreased up to 445°C. This behavior indicates that the polymer chains are in an ordered phase over the range 365–445°C. This result indicates that the dimensional stability is well maintained up to 445°C. For further elevation of temperature, the modulus decreased sharply again from 316 MPa at 445°C to 34.8 MPa at 473°C. This second transition is much sharper than the first transition. From these transition behaviors, it is suggested that the first transition at the range 290–365°C corresponds to a glass transition while the second transition in the higher temperature region corresponds to an order–disorder transition.

The loss modulus (E'') profile with temperature reveals three relaxation peaks. The first broad peak over 25–275°C might correspond to the β-relaxation, which results from rotational motions of phenyl moieties as well as of phenylene imide rings on the polymer backbone as observed in most aromatic polyimides [9,32]. The β-relaxation is usually overlapped with the β'-relaxation due to moisture absorbed in films [9,32]. The second peak at 275–380°C is the relaxation due to the polymer chain motions through the glass transition. Finally, the third peak over 450–480°C may result from motions of polymer chains through the order–disorder transition.

Residual stress of the BPDA-ODA films imidized at 400°C was measured at room temperature. The radii of curvature of the calibrated wafer without and with the polymer film were measured to be 669.82 and 3.88 m, respectively, giving a residual stress of 48 MPa. This stress value is relatively larger than those of PMDA-ODA and

Table 1
Self-adhesion of BPDA-ODA polyimide

Specimen structure[a] (bottom to top layer)	Peel strength (J/m^2) with peeling rate (mm/min)						
	0.05	0.1	0.2	0.5	1.0	2.0	5.0
BPDA-ODA//BPDA-ODA	1060	1089	1118	1148	1187	–	–
BPDA-ODA//BPDA-ODA/PMDA-ODA	814	873	893	912	922	942	971
BPDA-ODA/Ash //BPDA-ODA[b]	Cannot peel						
BPDA-ODA/Ash/Primer//BPDA-ODA[c]	1069	1079	1138	1236	–	–	–

[a] Thermal imidization was conducted at $T_1 = T_2 = 400$°C: T_i is an imidization temperature of polyimide layer *i*.

[b] Ashing was carried out at 300 W/5 min and 535 cc/min oxygen flow rate.

[c] A primer, γ-aminopropyltriethoxysilane of 0.1 vol.% in ethanol/water (95/5 in volume) mixture was spin-coated at 2000 rpm/20 s, followed by baking at 120°C for 20 min in ambient air.

BPDA-PDA polyimide films: 30 MPa for the PMDA-ODA polyimide film and 12 MPa for the BPDA-PDA polyimide film [3]. The stress value is relatively high in spite of some degree of molecular chain orientation and intermolecular packing order in the polyimide film. The high stress might be attributed to some degree of chain flexibility caused by losing chain linearity due to biphenyl and ether linkages on the backbone. In addition, the low degree of crystallinity may contribute positively to the residual stress to increase.

The residual stress of a polyimide film is dependent upon the physical parameters, such as TEC, modulus, Poisson's ratio, and thermal history [3]. Among these parameters, the TEC mismatched from that of substrate (3 ppm/°C for silicon wafer [5,33]) is the primary factor to cause residual stress. As aforementioned, the BPDA-ODA film has a TEC of 43 ppm/°C. This value is very close to that (40 ppm/°C) of PMDA-ODA but much higher than that (ca. 5 ppm/°C) of BPDA-PDA. Therefore, the high residual stress in the BPDA-ODA, compared to that of the PMDA-ODA, might be attributed to the relatively high modulus.

In addition, the swelling behavior in NMP was investigated at both room temperature and 80°C. The degree of swelling was ca. 0.5 wt.% after a day at room temperature. Also, a similar amount of swelling in NMP was obtained after 2 h at 80°C. This value is comparable to that of BPDA-PDA polyimide [13], but relatively much lower than that of PMDA-ODA polyimide [24]. In addition, BPDA-ODA polyimide has been reported to exhibit a relatively low moisture uptake as observed for BPDA-PDA polyimide [17]: 1.3–1.4 wt.%. Instead, PMDA-ODA polyimide is known to absorb moisture in 2.5 wt.% [17]. Conclusively, the BPDA-ODA polyimide has a relatively high solvent resistance to NMP as well as water.

In comparison, the BPDA unit in the polymer backbones of BPDA-ODA and BPDA-PDA polyimide seems likely to provide enough solvent resistance to NMP and water. Another contributing factor for the low degree of swelling might be the intermolecular lateral crystalline like packing even though the overall crystallinity is low.

3.3. Self-adhesion and adhesion to substrates

Adhesion measurements were performed with various peeling rates for the specimens in which all the polyimide layers were prepared by thermal imidization at 400°C which is higher than its T_g. The measured peel strengths are summarized in Tables 1 and 2.

To measure self-adhesion of BPDA-ODA polyimide, specimens were prepared in four different ways (see Table 1). In the first case, the bottom polyimide layer was prepared in a conventional way, and the top polyimide layer was overcoated on the bottom layer, giving an adhesion specimen, BPDA-ODA//BPDA-ODA. For this specimen, peel strength was measured to be 1060–1187 J/m^2 over the peeling rate of 0.05–1.0 mm/min. In the second case, a peel test was performed with the aid of PMDA-ODA

Table 2
Adhesion of BPDA-ODA polyimide to substrates: silicon wafer with oxidized surface and glass ceramic (GC) substrate

Specimen structure[a] (bottom to top layer)	Peel strength (J/m^2) with peeling rate (mm/min)						
	0.05	0.1	0.2	0.5	1.0	2.0	5.0
Si wafer/Ash//BPDA-ODA[b]	687	746	795	834	893	961	1040
Si wafer/Ash/Primer//BPDA-ODA[c]	1010	1050	1099	1138	1207	1256	1364
G.C./Ash/Primer//BPDA-ODA	–	–	–	922	–	–	–

[a] Thermal imidization was conducted at 400°C.

[b] Ashing was carried out at 300 W/5 min and 535 cc/min oxygen flow rate.

[c] A primer, γ-aminopropyltriethoxysilane of 0.1 vol.% in ethanol/water (95/5 in volume) mixture was spin-coated at 2000 rpm/20 s, followed by baking at 120°C for 20 min in air ambient.

polyimide layer. It is well known that the peel strength is dependent upon properties of the peeled polyimide layer, such as Young's modulus, yield strength, Poisson's ratio, and stiffness [5,34,35]. Numerous adhesion studies have been done with the aid of PMDA-ODA polyimide as a backing layer in the peel test because the polymer has been used widely in the microelectronic industry for the last decade. For this reason, in the present study the top polymer layer was overcoated on the bottom BPDA-ODA polyimide layer with the aid of PMDA-ODA polyimide. That is, a BPDA-ODA polyimide layer of ca. 2 μm thick was overcoated first on the bottom layer and followed by overcoating of PMDA-ODA polyimide layer to ca. 18 μm thickness. The peel strength was measured to be 814–971 J/m^2 over the peeling rate of 0.05–5.0 mm/min. These peel strength values are relatively lower than those obtained for the BPDA-ODA//BPDA-ODA bilayer specimen. The difference might result mainly from the different mechanical properties between the BPDA-ODA and PMDA-ODA backing layers as aforementioned.

Overall, the self-adhesion of BPDA-ODA polyimide is relatively very high in spite of some variations with the backing layer polymer in the peel test: in general, a peel strength of >500 J/m^2 is required for all the interfaces in microelectronic devices [1]. The high peel strength values indicate that enough inter-diffusion of the polyimide molecules take place between the bottom and top layers through the conventional imidization process, leading to an excellent self-adhesion. In comparison, both PMDA-ODA and BPDA-PDA polyimides are known to exhibit a very low peel strength of ≤ 30 J/m^2, respectively [1,13,15,16,24]. In fact, bilayer films of these two polyimides are often delaminated natually without any external stress because of their poor self-adhesion characteristics.

In general, a plasma ashing is always used for each process step in the fabrication of microelectronic devices, in order to clean the surface of devices being fabricated. For this reason, the bottom polyimide layer was prepared and its surface was ashed at 150 W/4 min and 535 cc/min oxygen flow. Then, the precursor polymer solution was overcoated on the ashed layer, followed by thermal imidization. For this specimen, the peeling could not be achieved, indicating very strong adhesion between the layers. That is, the O_2 plasma ashing of the bottom layer surface improves highly the self-adhesion of BPDA-ODA polyimide.

In addition to the plasma ashing, a primer treatment was performed. That is, the bottom polyimide layer was ashed and subsequently treated with a primer, APTES. The precursor polymer solution was overcoated on the primer-coated polyimide layer, followed by thermal imidization. The specimen exhibited a peel strength of 1069–1236 J/m^2 over the peeling rate of 0.05–0.5 mm/min in the peel test. These self-adhesion values are very slightly higher than those obtained for the bilayer specimen without ashing and primer treatments. This result indicates that the APTES primer treatment does not contribute effectively to improve the self-adhesion.

In fact, APTES has often been used as an adhesion primer for the interface of ploymer/substrate as well as of polymer/polymer in the fabrication of microelectronic devices. However, the role of APTES primer is not clear for the adhesion improvement in polymer/polymer interfaces because APTES primer has originally designed for the interface of polymer with silicon wafers, glasses and ceramic substrates. There is a controversy in the role of APTES primer for the improvement of adhesion at polyimide/polyimide interfaces. In the present study, a merit of APTES, as an adhesion primer, was not observed in the self-adhesion of BPDA-ODA polyimide layer.

In addition to the self-adhesion, the adhesion of BPDA-ODA polyimide was investigated further for the interfaces of silicon wafers. With silicon wafers with oxidized surface, two different kinds of adhesion specimens were prepared. Firstly, some silicon wafers were cleaned by ashing. Then, the BPDA-ODA PAA solution was overcoated on the ashed silicon wafers, followed by drying and imidization. For the specimens, the peel strength was measured to be 687–1040 J/m^2, depending upon the peeling rate (see Table 2). These peel strength values are relatively very high, indicating that the BPDA-ODA polyimide has an excellent adhesion to the silicon wafer. Secondly, the other silicon wafers were ashed and subsequently prime-coated with APTES. The precursor polymer solution was overcoated on the primed silicon wafers, followed by drying and imidization. The specimens exhibited 1010–1364 J/m^2 over the peeling rate of 0.05–5.0 mm/min. These peel strength values are relatively larger than those obtained for the polyimide/silicon wafer without using APTES primer. In conclusion, these results are evident that the APTES primer enhances the adhesion of BPDA-ODA polyimide to the silicon wafer.

The adhesion study was extended to the polyimide/glass ceramic substrate specimens. Glass ceramic substrates were cleaned by ashing and prime-coated with APTES. The precursor polymer solution was applied on the primed ceramic substrates and dried, followed by thermal imidization. For these ceramic specimens, peel strength was 922 J/m^2 at a peeling rate of 0.5 mm/min. This peel strength is slightly lower than that (1138 J/m^2) of the polyimide/silicon wafer, but still reasonably high enough for applications in microelectronic devices based on glass ceramic substrates.

4. Conclusions

The BPDA-ODA polyimide in films exhibits a typical crystal-like structure but its overall crystallinity was relatively small, only ca. 17%. The polymer film reveals two phase transitions: the first at 313°C is the glass transition and the second at 465°C is the order–disorder transition. The residual stress was 48 MPa at room temperature, while the TEC, which is a primary factor to control the residual stress, was 43 ppm/°C over the range 100–200°C. These high stress and TEC might be due to the low degree of crystallinity, some degree of chain flexibility due to the ether linkage, and the loss of chain linearity due to biphenyl and ether linkages on the polymer backbone. Instead, the polyimide shows an excellent solvent resistance to NMP and moisture.

The polyimide imidized at 400°C exhibits an excellent self-adhesion strength of 1060–1187 J/m^2, depending upon the peel rate. The excellent self-adhesion is correlated to the glass transition at 313°C which is lower than the final imidization temperature (400°C) employed. Namely, a high molecular chain mobility is generated through the thermal imidization process, allowing enough molecular inter-diffusion between the bottom and top layers and consequently leading to the excellent self-adhesion. An oxygen plasma treatment of the polyimide surface improved the self-adhesion highly, whereas a silane primer treatment apparently did not enhance the self-adhesion. This is an evidence that silane primer is not a good adhesion promoter for polymer/polymer interfaces.

The polyimide also reveals a good adhesion to silicon wafers and glass ceramic substrates. Different from the self-adhesion, the adhesion of the polyimide to the substrates was further improved by an APTES primer treatment. This indicates that the silane primer is a good adhesion promoter for interfaces of a polymer to silicon and ceramic substrates.

In conclusion, the BPDA-ODA polyimide has excellent properties suitable for applications in the fabrication of microelectronic devices as interdielectric layers, passivation layers, alpha particle protection layers, and high temperature adhesives.

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