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Adhesion strength of poly(imide-siloxane) with Alloy 42 lead frame and silicon dioxide

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Abstract

Poly(imide-siloxane) passivation layer was investigated for tapeless lead-on-chip (LOC) packaging. Various molecular weights of siloxane diamine (SDA), α,ω-(aminophenoxypropyl)-poly(dimethylsiloxane) were synthesized and incorporated to synthesize poly(imide-siloxane). Effects of molecular weight and content of SDA on the adhesion strength of poly(imide-siloxane) with Alloy 42 lead frame and silicon dioxide were studied. Poly(imide-siloxane) having high molecular weight of SDA showed good adhesion strength with Alloy 42 lead frame. On the other hand, poly(imide-siloxane) having low molecular weight of SDA showed good adhesion strength with silicon dioxide. Therefore, blends of poly(imide-siloxane)s having high and low molecular weight of SDA showed good adhesion strength with both Alloy 42 lead frame and silicon dioxide. The origin of different adhesion behavior of poly(imide-siloxane) depending upon the molecular weight of SDA with Alloy 42 lead frame and silicon dioxide was examined by contact angle measurement and X-ray photoelectron spectroscopy (XPS). © 2000 Published by Elsevier Science Ltd.

Keywords: Poly(imide-siloxane); Lead-on-chip packaging; Adhesion strength

1. Introduction

Polyimide has been used as a buffer coating layer for semiconductor chips because it has excellent thermal, mechanical and electrical properties [1,2]. There have been many studies on the adhesion between the polyimide passivation layer and other materials (e.g. epoxy molding compounds (EMC), SiO_2, copper, aluminum) [3–9]. Silane coupling agents have been used as adhesion promoters [4,5] and siloxane modified polyimides also have been used to improve the adhesion strength without an adhesion promoter [6–8].

Recently lead-on-chip (LOC) packaging has become popular in semiconductor devices. Adhesion tapes such as polyamide-imide, polyamide, and polyimide tapes have been employed to improve the adhesion strength between the Alloy 42 lead frame and the polyimide passivation layer. The Alloy 42 lead frame is used usually for MOS (metal-oxide-semiconductor) chip and is composed of 42% of Ni and 58% of Fe.

In this study, noble poly(imide-siloxane) was synthesized and investigated as a passivation layer for tapeless LOC packaging to avoid adhesive tape. Poly(imide-siloxane) should adhere to the Alloy 42 lead frame as well as to the silicon wafer. The Alloy 42 lead frame especially has to be adhered to fully imidized poly(imide-siloxane) with normal chip packaging process conditions. Suitable molecular weight and content of SDA were examined to maximize the adhesion strength of poly(imide-siloxane) with an Alloy 42 lead frame as well as a silicon wafer.

2. Experimental

2.1. Synthesis of α,ω-(aminophenoxy)polydimethylsiloxane

α,ω-(aminophenoxypropyl)poly(dimethylsiloxane) (SDA) was synthesized by the ring opening polymerization of octamethylcyclotetrasiloxane (D_4, Petrach Systems Silanes and Silicones) using 1,3-bis(nitrophenoxypropyl)dimethyldisiloxane (BNPPD) as an initiator and end-blocker. BNPPD was synthesized as follows. First, potassium *p*-nitrophenoxide was obtained by reaction with *p*-nitrophenol (Aldrich Chemical Company Inc.) and potassium hydroxide (Aldrich Chemical Company Inc.) at room temperature for 1 h (Fig. 1(I)). Then BNPPD was synthesized with potassium

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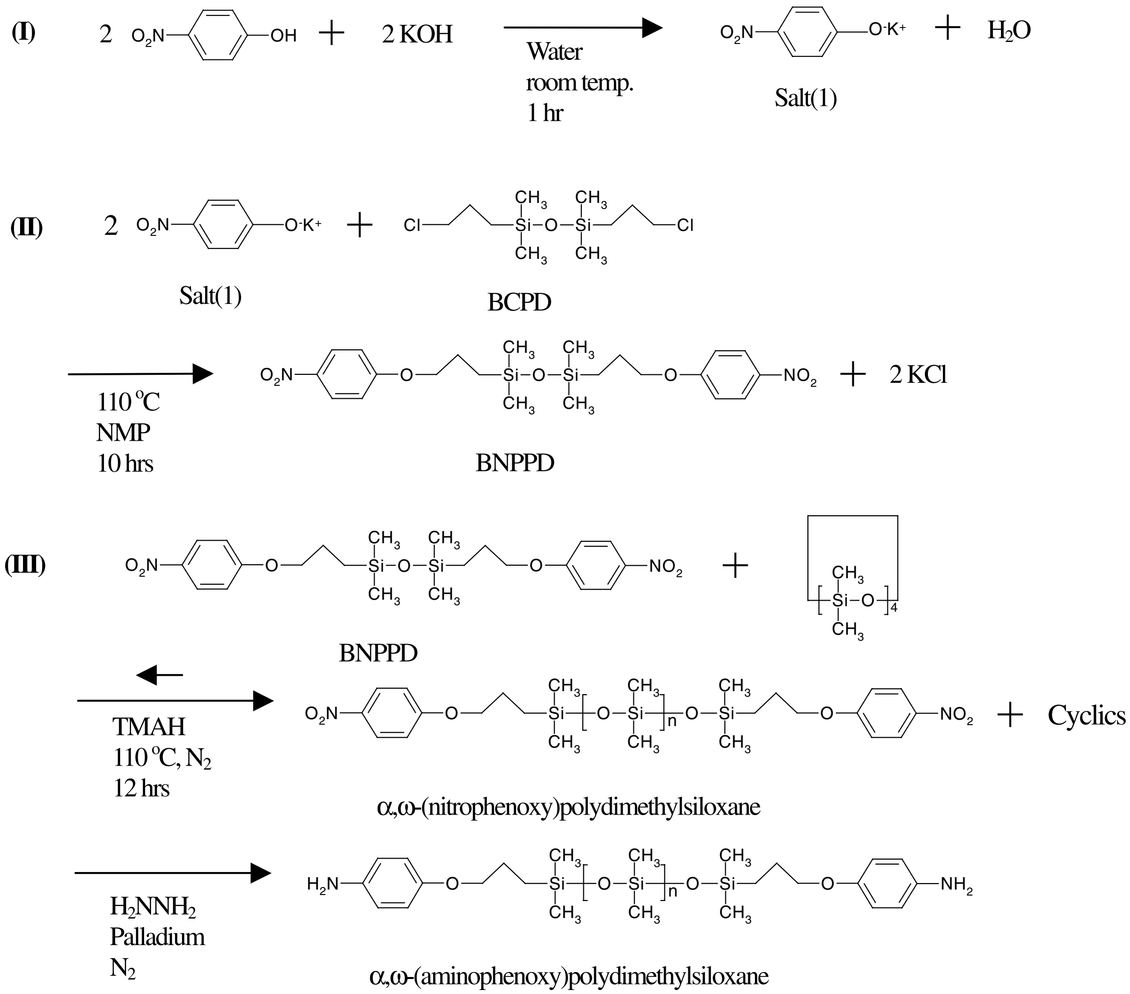


Fig. 1. Synthesis of α,ω-(aminophenoxy)polydimethylsiloxane.

p-nitrophenoxide and 1,3-bis-(3-chloropropyl)tetramethyldisiloxane (BCPD, Petrach Systems Silanes and Silicones) in *N*-methylpyrrolidone (NMP Aldrich Chemical Company Inc.) at 110°C for 10 h (Fig. 1(II)). The completion of the reaction was checked using thin layer chromatography. BNPPD was recrystallized in 0.3 mol KOH aqueous solution three to four times.

SDA was synthesized with D_4 and BNPPD at 110°C for 12 h (Fig. 1(III)). Tetramethylammonium hydroxide (TMAH, 25 wt% in methanol, Aldrich Chemical Company Inc.) used as a catalyst was azeotropically distilled. The unused catalyst was decomposed thermally at 150°C for 5 h. Then the unused catalyst and non-reacted cyclics were vacuum-stripped under a pressure of 0.5 mmHg at 160°C for 5 h. The product was reduced with hydrazine (Aldrich Chemical Company Inc.) and palladium (Aldrich Chemical Company Inc.) to have amine end groups. Molecular weight of SDA was controlled by a ratio of BNPPD and D_4 and four different molecular weights of SDA (433, 1500, 5000, and 10 000 g/mol) were synthesized. The SDAs were analyzed by using NMR, FT-IR, and GPC.

2.2. Synthesis of poly(imide-siloxane)

Poly(imide-siloxane) was synthesized from 4,4′-oxydianiline (ODA) and SDA as diamines and pyromellitic dianhydride (PMDA) and benzophenone tetracarboxylic dianhydiride (BTDA) as dianhydrides. ODA (Wakayama Seika Kogyo Co. Ltd, Japan), PMDA (Daicel Co. Ltd, Japan), and BTDA (Daicel Co. Ltd, Japan) were taken out of bottles in a glove box in argon atmosphere and used immediately without any refining. Anhydrous NMP (Aldrich Chemical Company Inc.) was used as a solvent without further purification and cyclohexanone (Aldrich Chemical Company Inc.), the co-solvent for SDA, was vacuum-distilled with magnesium sulfate as a dehydrating agent.

In order to obtain a homogeneous poly(amic acid-siloxane), monomers were dissolved in NMP/cyclohexanone at a ratio of 1/2. The reaction proceeded for 8 h with stirring at about 10°C in N_2 atmosphere (Fig. 2). In order to control the molecular weight of poly(amic acid-siloxane), the poly(amic acid-siloxane) was kept in a convection oven at 80°C until about 25 000 cP of viscosity was obtained.

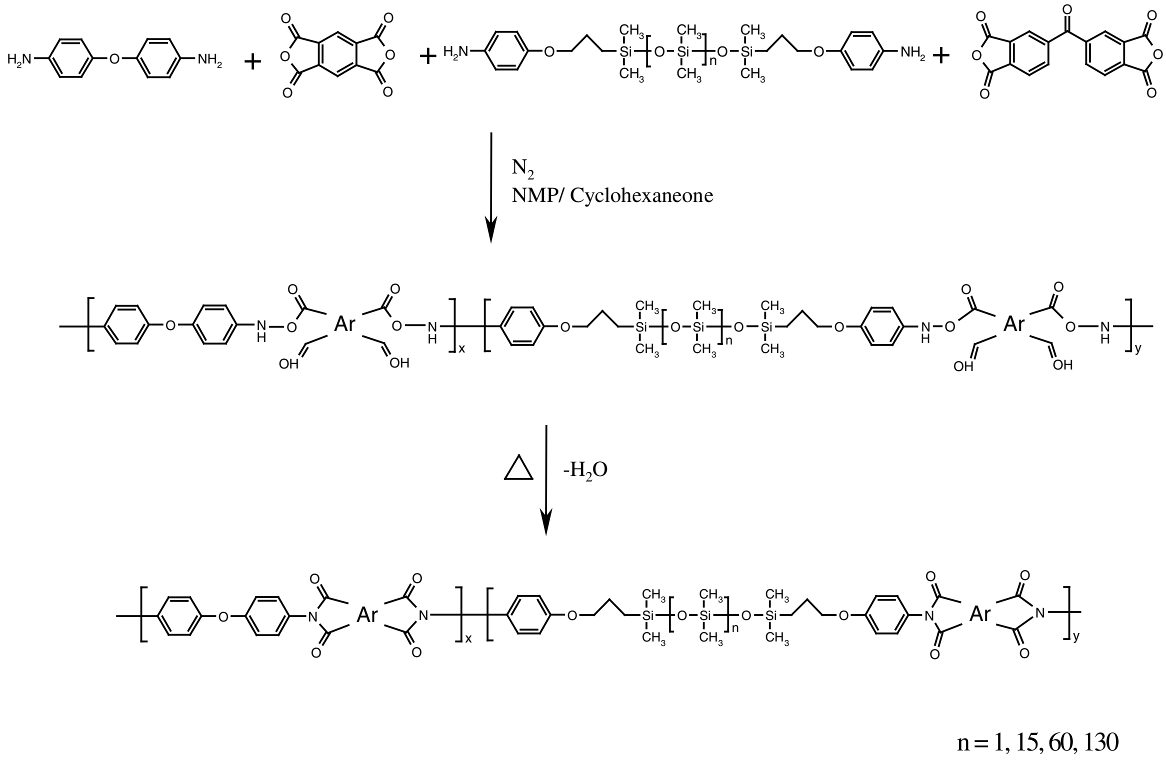


Fig. 2. Synthesis of poly(imide-siloxane) from ODA, PMDA, SDA, and BTDA.

Four different molecular weights of SDA (433, 1500, 5000, and 10 000 g/mol) were incorporated into the polyimide backbone with a concentration of 0–20 wt% among the diamines. All the poly(amic acid-siloxane) had a PMDA/BTDA ratio of 0.45/0.55 and 20 wt% of solid content.

The poly(imide-siloxane) film was prepared as follows. Poly(amic acid-siloxane) was spin-coated on slide glass (No. 2947, Corning Co.) and pre-baked at 100°C for 1 h and cured at 150, 200, and 300°C for 1 h each. The heating rate was 5°C/min up to 200°C and then changed to 2°C/min above 200°C. After delaminating in a steam cooker (119°C, 0.9 kgf/m^2), the film was dried in a convection oven at 120°C for more than 24 h. The film thickness was 37 ± 3 μm with micrometer (1 μm resolution, Mitutoyo Co.). The notation representing poly(imide-siloxane) having various molecular weights and content of SDA is, for example, as follows: PI 433-10 represents poly(imide-siloxane) having a molecular weight of 433 g/mol and 10 wt% of SDA.

2.3. Sample preparation for 90° peel test

Adhesion test specimens of Alloy 42 lead frame/poly(imide-siloxane) were prepared by hot press molding at 350°C with 50 psi for 70 s, where silicon rubber was put on joints of Alloy 42 lead/poly(imide-siloxane) for intimate contact. In order to remove grease and contaminants on the Alloy 42 lead frame plate, Alloy 42 lead frame plates were cleaned with dichloromethane in an ultrasonic bath for 1 h, rinsed with acetone, and dried in a convection oven at 70°C for 3 h. The specimen was backed up with adhesive tape (Scotch tape™, 68 μm thickness, 3M Company Inc.) to minimize film deformation. Adhesion strength was measured with an Instron 4206 Universal Testing Machine (UTM) by the 90° peel test with a cross-head speed of 5 mm/min (Fig. 3).

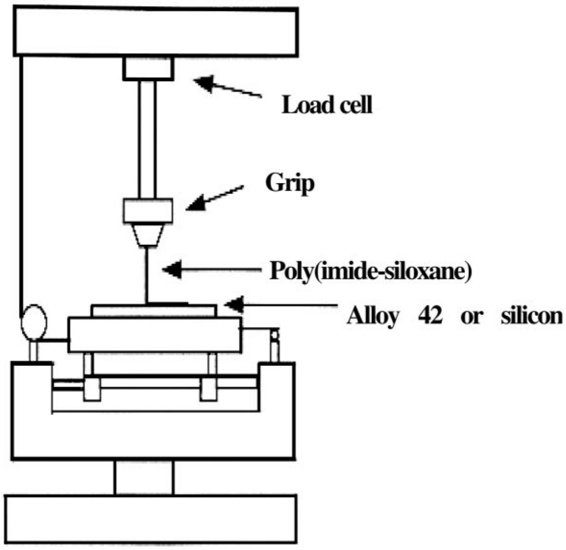


Fig. 3. Schematic diagram of 90° peel test.

Adhesion strength of the poly(imide-siloxane)/SiO_2 interface was also measured by the 90° peel test after spin-casting of poly(amic acid-siloxane) on silicon wafer and imidization at 150, 200 and 300°C for 1 h each. The silicon wafer was cleaned in Piranhya solution (H_2SO_4:$H_2O_2 = 4:1$) for 30 min and rinsed three times with distilled water.

2.4. X-ray photoelectron spectroscopy (XPS)

The surface composition of poly(imide-siloxane) and the locus of failure of Alloy 42 lead frame/poly(imide-siloxane) were investigated with an X-ray photoelectron spectrometer (Mg K_α X-ray source, KRATOS XSAM 800 pci). Take-off angles between the sample surface and the detector to examine the atomic composition depending on the depth from the surface were 30 and 90°.

2.5. Surface energies

The surface energies of poly(imide-siloxane) were determined by measuring the contact angle with a contact angle goniometer (Model CA-A, Kyowa Surface Science Co. Ltd, Tokyo, Japan). The sampling depth is within 5 Å thickness by this method [10]. The following simultaneous equations [11] were solved to obtain the dispersive (γ_s^d) and polar (γ_s^p) components of the surface energies of poly(imide-siloxane) from the contact angles using two test liquids, distilled water(1) and diiodomethane(2):

$$(1 + \cos\theta_1)\gamma_1 = 2[(\gamma_1^d\gamma_s^d)^{1/2} + (\gamma_1^p\gamma_s^p)^{1/2}] \qquad (1)$$

$$(1 + \cos\theta_2)\gamma_2 = 2[(\gamma_2^d\gamma_s^d)^{1/2} + (\gamma_2^p\gamma_s^p)^{1/2}] \qquad (2)$$

$$\gamma_s = \gamma_s^d + \gamma_s^p \qquad (3)$$

The surface energy γ, the dispersive component γ^d, and the polar component γ^p values used in these calculations were 72.2, 22.0, and 50.2 mJ/m^2 for water, and 50.8, 48.5, and 2.3 mJ/m^2 for diiodomethane [11].

3. Results and discussion

3.1. Adhesion strength between Alloy 42 lead frame and poly(imide-siloxane)

Fig. 4 shows the effects of molecular weight and content of SDA on the adhesion strength between an Alloy 42 lead frame and poly(imide-siloxane). Homo polyimide without SDA had little adhesion strength, about 100 N/m, to the Alloy 42 lead frame. The adhesion strength of the Alloy 42 lead frame/poly(imide-siloxane) joints having low molecular weight of SDA such as PI 433 and PI 1500 increased with the increase of SDA content up to 20 wt%. The adhesion strength of the Alloy 42 lead frame/PI 5000 joints was much higher than those of Alloy 42 lead frame/PI 433 and Alloy 42 lead frame/PI 1500 joints and increased more rapidly up to 10 wt% of SDA content. The adhesion strength of the Alloy 42 lead frame/PI 5000-10 joints appeared to be higher than 600 N/m because the poly(imide-siloxane) film was torn at 600 N/m before peeling. However, a higher content of SDA decreased the adhesion strength of the Alloy 42 lead frame/PI 5000 joints as shown by the Alloy 42 lead frame/PI 5000-20 joint. For the case of Alloy 42 lead frame/PI 10000 joints, good adhesion strength (about 320 N/m) was obtained with only 1 wt% of SDA, but the adhesion strength decreased with more than 1 wt% of SDA to less than that of homo polyimide.

3.2. Adhesion strength between poly(imide-siloxane) and silicon dioxide

Table 1 shows the adhesion strength between poly(imide-siloxane) and SiO_2 having various molecular weights and contents of SDA. Homo polyimide without SDA had little adhesion strength to SiO_2. However, the adhesion strength of PI 433/SiO_2 joints was so great that the poly(imide-siloxane) film could not be peeled. Therefore, the PI 433/SiO_2 joints were stored in the autoclave at 120°C with 1.0 kgf/m^2 to reduce the adhesion strength of PI 433/SiO_2 joints: PI 433-1/SiO_2 joint was delaminated and PI 433-5/SiO_2 joint was peeled with a peel force of 940 N/m after storage for 6 h in the autoclave. However, PI 433-10/SiO_2 and PI 433-20/SiO_2 joints could not be peeled even after storage for 48 h in the autoclave.

For PI 1500/SiO_2 joints, the adhesion strength was near zero with 1 and 5 wt% of SDA content and increased to 950 N/m with 10 wt% of SDA content. PI 1500-20/SiO_2 could not be peeled even after storing it for 48 h in the autoclave. For PI 5000/SiO_2 and PI 10000/SiO_2 joints, the adhesion strength was near zero even with 20 wt% of SDA content. From these results, it can be concluded that a lower molecular weight of SDA with a larger amount is effective in improving the adhesion strength of SiO_2/poly(imide-siloxane) joints.

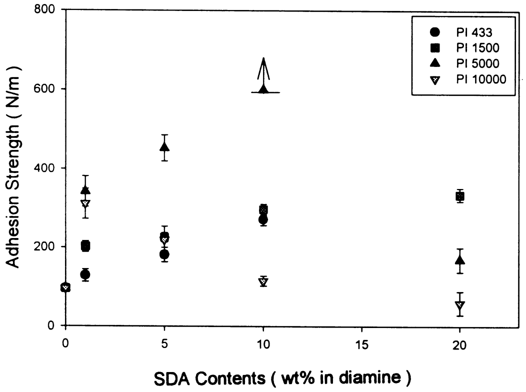


Fig. 4. Adhesion strength between Alloy 42 lead frame and poly(imide-siloxane).

Table 1
Adhesion strength between SiO_2 and poly(imide-siloxane)

Mn of SDA	SDA content in diamine (wt%)			
	1	5	10	20
433	Cannot peel (delamination)[a]	Cannot peel (940)[a]	Cannot peel[b]	Cannot peel[b]
1500	Near zero	Near zero	950	Cannot peel[b]
5000	Near zero	Near zero	Near zero	Near zero
10 000	Near zero	Near zero	Near zero	Near zero

[a] After treating at 120°C with 1.0 kgf/m^2 in an autoclave for 6 h.

[b] After treating at 120°C with 1.0 kgf/m^2 in an autoclave for 48 h.

3.3. Effect of SDA on the adhesion strength of poly(imide-siloxane) with Alloy 42 lead frame and SiO_2

3.3.1. Surface composition of poly(imide-siloxane)

Fig. 5 shows that the surface energy of poly(imide-siloxane) decreased with the increase of SDA content. The decrease of surface energy was pronounced with the increase of molecular weight of SDA, especially with larger than 5000 g/mol of SDA, which indicates that SDA migrates to the air surface of poly(imide-siloxane) because of low surface energy, and high molecular weight of SDA has better mobility to air surface than low molecular weight of SDA [12]. With low molecular weight of SDA, the mobility of the SDA molecules was much restricted by the rigid imide group owing to the short chain length of SDA (Fig. 6(b)). SDA with 5000 and 10 000 g/mol, unlike the low molecular weight of SDA, was likely to have more freedom to migrate to the surface and not be affected by the rigid imide group connected to both ends of SDA because of the long chain length of the SDA molecules (Fig. 6(a)). Since poly(imide-siloxane) was spin-coated on the substrate, poly(imide-siloxane) molecules could be oriented parallel to the substrate. Coburn et al. have already reported that the in-plane molecular orientation of spin-coated polyimide film was confirmed with birefringence [13]. The surface energies of PI 5000-20, PI 10000-10 and PI 10000-20 were all equal to 24 mJ/m^2, which was the surface energy of pure polydimethylsiloxane (PDMS) [14]. This means that their surfaces were covered with SDA.

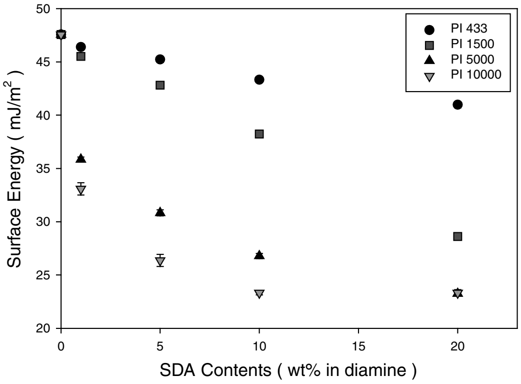


Fig. 5. Surface energy of poly(imide-siloxane) depending on the molecular weight and the content of SDA.

Table 2 shows the atomic composition of poly(imide-siloxane). The sampling depth was about 40 Å with 30° and 90 Å with 90° as take-off angles [15,16]. The silicon concentration was higher near the surface and increased with the increase of molecular weight of SDA. This is consistent with the results of surface energy (Fig. 5).

3.3.2. Distribution of SDA in Alloy 42 lead frame/poly(imide-siloxane) joints

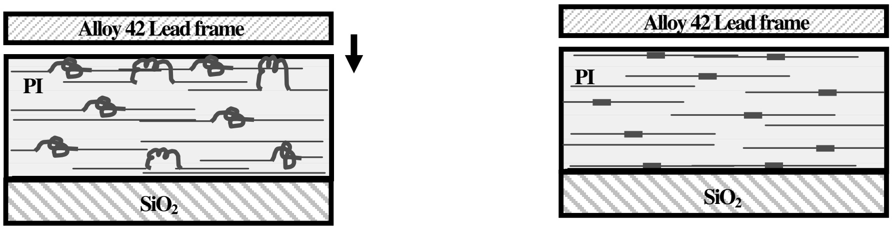
Since high molecular weight of SDA has better mobility to the air surface of poly(imide-siloxane) than low molecular weight of SDA, the concentration of SDA on the surface of poly(imide-siloxane) having high molecular weight of SDA is high as shown in Fig. 6(a). Therefore, the adhesion strength of PI 5000 with an Alloy 42 lead frame was higher than those of PI 433 and PI 1500 with an Alloy 42 lead frame (Fig. 4). Since the glass transition temperature of SDA is −123°C, the glass transition temperature of the surface of poly(imide-siloxane) appears to be very low [17] and wettability to the Alloy 42 lead frame should be good enough to have inter-molecular interactions such as electrostatic interaction and van der Waals force.

However, too much SDA on the surface of poly(imide-siloxane) reduced the adhesion strength by forming a weak boundary layer. Although PI 5000-10 showed very good adhesion strength, PI 5000-20 showed poor adhesion strength because of too much SDA on the surface of poly-(imide-siloxane).

According to Cassie [18], when there are two types of surface compositions, the equilibrium contact angle can be expressed as

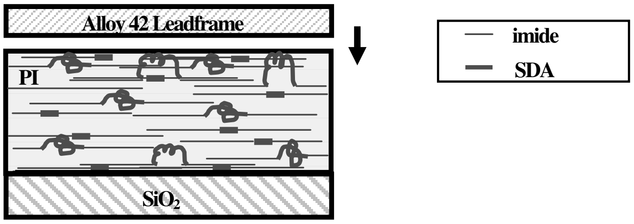
$$\cos \theta_{\mathrm{E}} = f_1 \cos \theta_{\mathrm{E}}^1 + f_2 \cos \theta_{\mathrm{E}}^2$$

where θ_{E} is the equilibrium contact angle, and f_1 and f_2 are the fractions of the surface occupied by surface types having contact angles θ_{E}^1 and θ_{E}^2, respectively. Since the contact angle is closely related with the surface energy, SDA surface fraction in poly(imide-siloxane) can be calculated



(a) Poly(imide-siloxane) having high molecular weight of SDA

(b) Poly(imide-siloxane) having low molecular weight of SDA



(c) Blend system of above poly(imide-siloxane)s

Fig. 6. Schematic diagrams of Alloy 42 lead frame/poly(imide-siloxane)/SiO_2 joints showing the distribution of SDA in the interfaces with Alloy 42 lead frame and SiO_2.

by the following equation:

$$\gamma_t = \gamma_s f + \gamma_h(1 - f)$$

where γ_t is the surface energy of a poly(imide-siloxane) film, γ_s is the surface energy of pure SDA (24 mJ/m^2), γ_h is the surface energy of homo PI (48 mJ/m^2), and f is the fraction of surface area covered with SDA. From this equation, we can estimate that about 91% of the surface area of PI 5000-10 (surface energy 26 mJ/m^2) showing the best adhesion strength with the Alloy 42 lead frame is covered with SDA.

Table 3 shows the atomic compositions of the peeled surface of the Alloy 42 lead frame/poly(imide-siloxane) joints by XPS. Although the Alloy 42 lead frame is only composed of 59% of Fe and 41% of Ni, XPS data of the Alloy 42 lead frame show significant amounts of C, O, and N. These appear to come from the contamination of the Alloy 42 lead frame. Since the polyimide side had Fe atomic concentration and the Alloy 42 lead frame side had Si atomic concentration, the locus of the failure of all the samples was mainly cohesive failure in the polydimethylsiloxane-rich layer of poly(imide-siloxane). Higher Si atomic concentration on both the poly(imide-siloxane) and Alloy 42 lead frame sides with higher molecular weight of SDA indicates that a higher molecular weight of SDA has better mobility and moves more easily toward the air surface.

Table 2
Atomic composition of poly(imide-siloxane) by XPS

Sample	Take-off angle	Atomic concentration (%)			
		O	N	C	Si
1500-10	30°	17.41	3.69	75.89	3.00
	90°	17.05	4.73	76.58	1.64
	Theoretical concentration	16.86	5.98	76.60	0.56
5000-10	30°	20.45	3.33	68.64	7.58
	90°	18.93	4.46	72.05	4.56
	Theoretical concentration	16.89	5.79	76.49	0.84
10000-10	30°	20.63	2.52	66.18	10.68
	90°	19.17	3.74	70.50	6.59
	Theoretical concentration	16.90	5.77	76.44	0.89

3.3.3. Distribution of SDA in SiO_2/poly(imide-siloxane) joints

The adhesion strength of SiO_2/poly(imide-siloxane) joints decreased with the increase of molecular weight of SDA as shown in Table 1. Since a low molecular weight of SDA has poor mobility, it stays in the interface of SiO_2/poly(imide-siloxane) joints as shown in Fig. 6(b). On the other hand, a high molecular weight of SDA moves toward the air surface of poly(imide-siloxane) and there is no SDA in the SiO_2 interface (Fig. 6(a)). Therefore, only the poly(imide-siloxane) having a low molecular weight of SDA can enhance the adhesion strength of SiO_2/poly(imide-siloxane) joint.

Table 3
Atomic composition of the peeled surfaces of Alloy 42 lead frame/poly(imide-siloxane) joints by XPS

Sample	Peel side	Take-off angle	Atomic concentration (%)					
			O	N	C	Si	Fe	Ni
1500-10/Alloy 42 lead frame	PI	30°	16.97	4.12	76.72	2.11	0.09	–
		90°	17.80	4.35	75.85	1.86	0.15	–
	Theoretical conc.		16.86	5.98	76.60	0.56	0	0
	Alloy 42	30°	17.58	4.07	76.46	1.65	0.25	–
		90°	18.76	4.00	75.52	1.39	0.34	–
	Theoretical conc.		0	0	0	0	59.21	40.79
5000-10/Alloy 42 lead frame	PI	30°	17.06	4.14	75.72	2.94	0.14	–
		90°	17.30	4.44	75.56	2.52	0.17	–
	Theoretical conc.		16.89	5.79	76.49	0.84	0	0
	Alloy 42	30°	16.28	4.08	77.23	2.30	0.11	–
		90°	16.44	4.18	77.55	1.68	0.15	–
	Theoretical conc.		0	0	0	0	59.21	40.79
10000-10/Alloy 42 lead frame	PI	30°	18.06	3.65	72.70	5.37	0.23	–
		90°	18.57	4.14	72.75	4.22	0.31	–
	Theoretical conc.		16.90	5.77	76.44	0.89	0	0
	Alloy 42	30°	17.42	3.77	74.96	3.61	0.24	–
		90°	17.97	3.77	75.10	2.81	0.36	–
	Theoretical conc.		0	0	0	0	59.21	40.79
Alloy 42 lead frame	30°		35.55	1.73	56.39	0	5.35	0.97
	90°		42.66	1.02	36.54	0	15.56	4.23
	Theoretical conc.		0	0	0	0	59.21	40.79

3.4. Effect of PI blends having high and low molecular weights on adhesion strength

From Section 3.3, it was rationalized that high molecular weight of SDA was favorable for Alloy 42 lead frame/poly(imide-siloxane) joints, and low molecular weight of SDA was favorable for SiO_2/poly(imide-siloxane) joints. Therefore, poly(imide-siloxane)s having a high molecular weight of SDA as well as a low molecular weight of SDA were made to have good adhesion strength to both the Alloy 42 lead frame and SiO_2. In this way, we can have SDA in the interface of both the Alloy 42 lead frame and SiO_2 as shown in Fig. 6(c). PI 433-10 with PI 5000-5, and PI 433-10 with PI 5000-10 were blended at the ratio of 1:1 at 10°C for 3 h. All the polyimide blends had good adhesion strength to the Alloy 42 lead frame as well as SiO_2 (Table 4).

Table 4
Effect of PI blends having high and low molecular weight of SDA on the adhesion strength with Alloy 42 lead frame and SiO_2

PI blends	Adhesion strength (N/m)	
	Alloy 42 lead frame[a]	SiO_2
PI 5000-5 and PI 433-10	Cannot peel	Cannot peel
PI 5000-10 and PI 433-10	Cannot peel	Cannot peel

[a] Alloy 42 lead frame/PI blend joints were prepared at 350°C with 50 psi for 20 s.

4. Conclusions

Poly(imide-siloxane)s having siloxane diamine (SDA) were synthesized as a buffer coating layer for tapeless lead-on-chip (LOC) packaging semiconductors. As a siloxane diamine, various molecular weights of α,ω-(aminophenoxypropyl)poly(dimethylsiloxane) were synthesized. Poly(imide-siloxane) having a high molecular weight of SDA showed good adhesion strength with the Alloy 42 lead frame. Since a high molecular weight of SDA has a good mobility, it migrates toward the air interface of poly(imide-siloxane), which enhances the adhesion strength of poly(imide-siloxane) with the Alloy 42 lead frame. There was an optimum content of SDA for maximizing the adhesion strength of poly(imide-siloxane) with the Alloy 42 lead frame, around 10 wt% among the diamines. Higher than an optimum content of SDA reduced the adhesion strength by forming the weak boundary layer of SDA.

In the case of adhesion between poly(imide-siloxane) and a silicon wafer (SiO_2), poly(imide-siloxane) having low molecular weight of SDA showed good adhesion strength. Since low molecular weight of SDA has poor mobility, it stays in the interface with SiO_2, which increases the adhesion strength of poly(imide-siloxane) with SiO_2. Therefore, blends of poly(imide-siloxane)s containing both high and low molecular weights of poly(imide-siloxane)s showed the good adhesion strength with both Alloy 42 lead frame and SiO_2 for tapeless LOC packaging.

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