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Corrosion protection and adhesion promotion for polyimide/copper system using silane-modified polymeric materials

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Abstract

The copolymers of vinyl imidazole (VI) and vinyl trimethoxy silane (VTS) were applied as corrosion inhibitors and as adhesion promoters for polyimide/copper system at elevated temperatures. The mole ratios of VI to VTS were 100:0, 70:30, 30:70, and 0:100. Peel test was performed to evaluate the adhesion strength of polyimide/primer/copper system after heat treatment at 400°C in nitrogen atmosphere. The effect of the copolymer composition on corrosion protection and adhesion promotion was investigated by Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), and scanning electron microscopy (SEM). The adhesion strength of polyimide/primer/copper system depended on the thermal stability of the primer and the chemical interaction between polyimide and the primer. It showed the highest value when the mole ratio of VI to VTS is 30:70. The primer reduced or suppressed copper oxide formation in the polyimide layer. The degree of corrosion protection by the primer was affected by its thermal stability and its reactivity with copper. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Polyimide/copper system; Adhesion promotion; Corrosion protection

1. Introduction

Polyimides have been widely utilized for the micro-electronic and composite applications. This is attributed to the fact that polyimides offer high thermal stability, good mechanical property, low moisture absorption, low dielectric constant, and good chemical resistance [1,2]. A good adhesion of polyimide to the metal is essential for micro-electronic applications. As a result, numerous studies have been performed to improve the adhesion of polyimide to the metal [3–13]. Especially, polyimide/metal interface, which can be formed by spin-coating poly(amic acid) solution onto metal surface, has been intensively studied [14–20]. Polyimide/metal interface involves the interaction of metal with poly(amic acid).

In multilevel thin film packaging, poly(amic acid) is spin-coated onto the metal substrate, and then cured at high temperature (~400°C). When poly(amic acid) is coated onto copper, its acid functionality was reported to react with copper surface to form carboxylate that decomposes upon curing [19,21]. Solvent plays a major role in the aggregation of copper-rich precipitates and their diffusion away from the interface. Accordingly, copper oxides are left in the cured polyimide layer, which deteriorates the dielectric property and the material property of polyimide. Transmission electron microscopy (TEM) confirmed the existence of very fine copper oxide particles in the polyimide layer [6]. Polyimide film with copper oxide particles had almost twice the dielectric constants in comparison with the pure materials [21].

Poly(amic ethyl ester) was prepared to suppress the formation of copper oxide particles in the polyimide layer [6]. It did not form copper oxide particles in the polyimide layer, but its adhesion strength (below 100 J/m^2) showed a relatively weak value in micro-electronic applications.

Therefore, the modification of polyimide/copper system has been attempted, to provide polyimide with both an adhesive property to copper and an electrical property. A thin passivation/adhesion promoting layer of metals as Cr, Ni, and Ta was coated onto copper to improve the adhesion of polyimide to copper as well as to prevent the copper oxide formation in the polyimide layer. However, this process is not simple and cost-effective because it contains metal capping step by the deposition of metal onto polyimide. To simplify the manufacturing process, a new process using polymer as a passivation/adhesion-promoting layer was proposed [22].

Imidazole and its derivatives have been reported to be the corrosion inhibitors for copper at elevated temperatures

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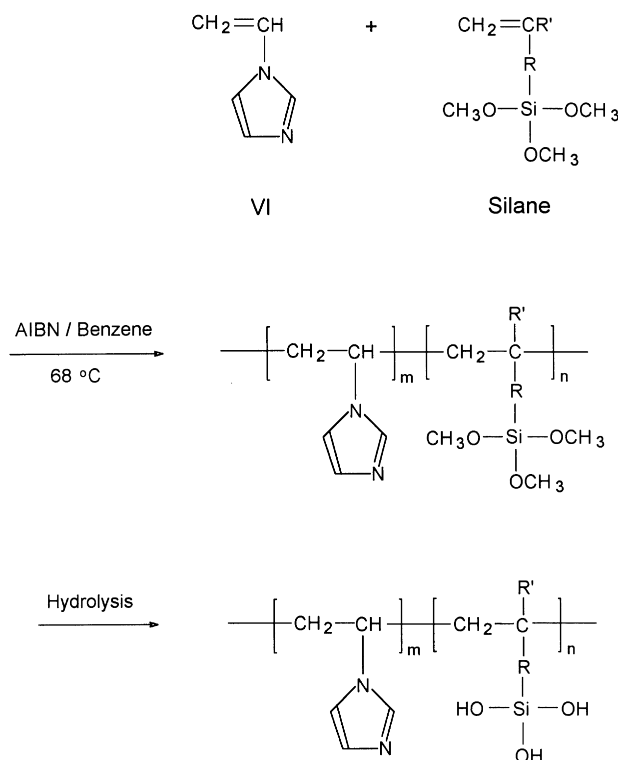

Fig. 1. Schematic diagram of copolymerization and hydrolysis.

[23–31]. They suppressed copper corrosion through complex formation with copper [32]. On the other hand, aminofunctional silanes have been utilized as an effective adhesion promoter of polyimide/inorganic interface [33–35]. Aminosilane forms imide bond with poly(amic acid) to enhance the adhesion of the polyimide/metal interface [35]. However, aminosilane lost its effectiveness as an adhesion promoter above 370°C in nitrogen atmosphere due to its thermal degradation. For the purpose of corrosion protection and adhesion promotion for polyimide/copper system above 370°C, therefore, silane-based imidazole copolymer with high thermal stability was synthesized.

In this study, the copolymer of vinylimidazole (VI) and vinyltrimethoxy silane (VTS) was applied as an adhesion promoter as well as a corrosion inhibitor for polyimide/copper system. The mole ratios of VI to VTS were 100:0, 70:30, 30:70, and 0:100. The effect of the copolymer composition on adhesion promotion and corrosion protection for polyimide/copper was investigated by the measurement of peel strength and interfacial analyses. For effective interfacial analyses, copper side and polymer side after peel test were investigated with Fourier transform infrared reflection and absorption spectroscopy (FTIR-RAS), micro-attenuated total reflectance (micro-ATR), and X-ray photoelectron spectroscopy (XPS).

2. Experimental

VI and VTS were purchased from Aldrich Chemical Co. and distilled in vacuo to yield pure and colourless liquids. Azobis(isobutyronitrile) (AIBN) from Wako Pure Chemical Industries, Ltd was dissolved in warm methanol (35°C), recrystallized in an ice bath, and then dried in a vacuum oven at room temperature for 2 days.

VI and VTS were copolymerized in benzene at 68°C with stirring in an argon atmosphere using AIBN as initiator. The total monomer concentration was 2 M, and the initiator concentration was fixed at 2×10^{-3} M. The mole ratios of VI to VTS were 100:0, 70:30, 30:70, and 0:100. The copolymerization scheme of VI and VTS is shown in Fig. 1.

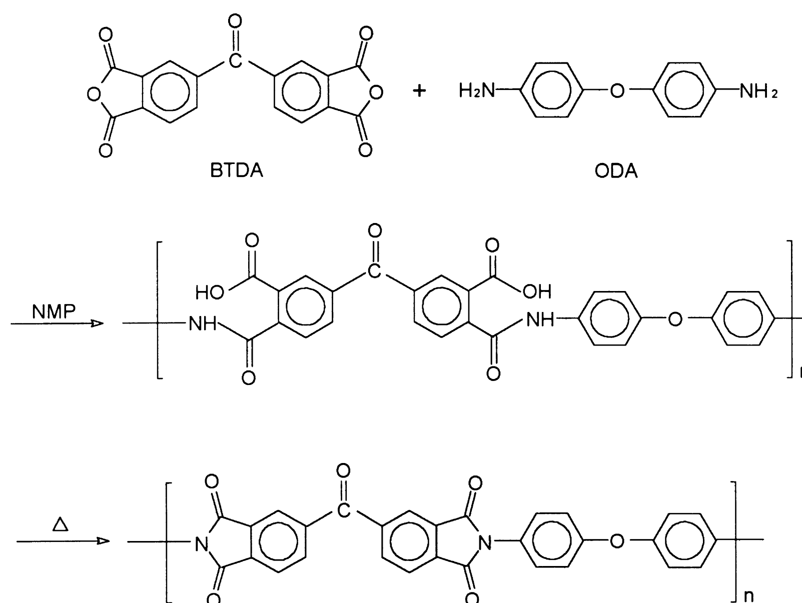

Fig. 2. Synthetic scheme of BTDA–ODA polyimide.

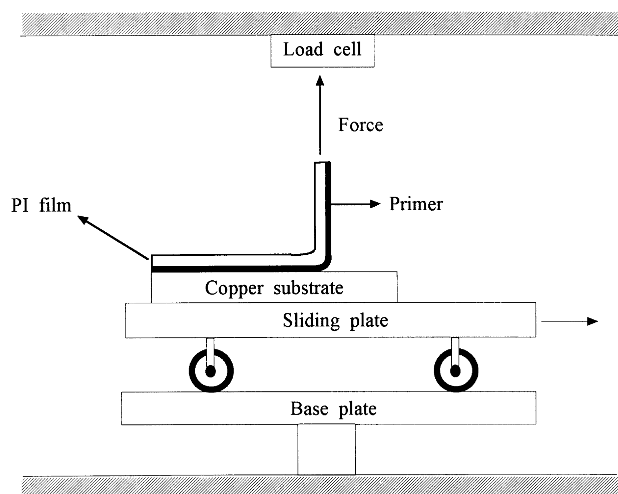

Fig. 3. Schematic diagram of measurement of peel strength.

BTDA–ODA poly(amic acid) was prepared by reacting stoichiometric amounts of BTDA and ODA. *N*-methyl-2-pyrrolidinone (NMP) was purchased from Aldrich Chemical Co., dehydrated with phosphorous pentoxide, and distilled under reduced pressure. 3,3′,4,4′-Benzophenonetetracarboxylic dianhydride (BTDA) and oxydianiline (ODA) were purchased from Aldrich Chemical Co. and recrystallized in acetic anhydride and ethanol, respectively.

ODA was dissolved in NMP and BTDA was added into the ODA/NMP solution at room temperature. The total solid content was 15 wt%. The reactor was stirred in an ice-water bath for 12 h. Viscous poly(amic acid) (PAA) solution was obtained and refrigerated until use. Fig. 2 shows the synthetic scheme for BTDA–ODA polyimide.

Copper plates (1.2 mm thick, Poongsan Co. in Korea) were mechanically polished with No. 5 chromic oxide, washed with *n*-hexane and acetone in an ultrasonic bath. They were rinsed with 1% HCl/distilled water solution, distilled water, and acetone, and then dried with a stream of nitrogen gas. Copolymer solution was cast onto copper plates with microsyringe as the primer and the samples were dried at 60°C for 12 h to remove the residual solvent in the copolymer film. Film thickness was calculated based on the concentration of the copolymer solution, copolymer density and the area of the copper surface. In this study, 1 μm thick films were used. Primer-coated copper plates were coated with diluted PAA solution (7 wt%). Imidization was performed at 100°C for 30 min, at 150°C for 30 min, and 250°C for 1 h. After imidization the coating thickness was ca. 15 μm.

The imidized samples were heat-treated at 400°C in a furnace with a continuous flow of nitrogen. After heat treatment, the whole polymer layers (5 mm wide) were peeled from the copper substrates. The peel strengths were measured by the 90° peel test, using Lloyd Universal Testing Machine. Fig. 3 shows schematic diagram of peel strength measurements. The peel rate was 1.5 mm/min and at least three measurements were performed. Peel energy (G_a) was calculated by the following equation:

$$G_a = F/W$$

where F is the peel force and W the width of the sample. After peeling off the polymer layer from the copper substrate, both the copper side and the polymer side were analysed with FTIR-RAS, micro-ATR, and XPS, in order to characterize the locus of failure.

XPS analysis was carried out with Escalab 220i-XL system from *FISONS* instruments to characterize PI/primer/copper system after peel test. Survey scan and narrow scan was performed and AlK_{α} radiation (1486.6 eV) was used as the X-ray source. The sample was positioned at a take-off angle of 45° with respect to the detector. The X-ray source was operated at a base pressure of $\sim 10^{-8}$ Torr.

FTIR-RAS was used to characterize copper surfaces after adhesion test. The spectrometer was continuously purged with dry nitrogen gas to remove water vapour and atmospheric CO_2. Absorbance spectra were obtained using Bomem MB-100 spectrometer at a resolution of 4 cm^{-1} and 100 scans were collected. Graseby Specac P/N 19650 monolayer/grazing angle accessory was used. The angle of incidence was 78° and a freshly polished copper coupon was used to obtain the reference spectrum.

Micro-ATR analysis was performed to characterize the polymer film peeled off the copper substrate. ATR attachment from Harrick Co. were used. A germanium IRE with endface angle of 45° were utilized and its dimension was $10 \times 5 \times 1$ mm^3. The reflection number in the germanium IRE was 10 and 100 scans were collected.

Scanning electron microscopy (SEM) was used to observe the fractured surfaces of PI/silane-modified PVI systems. The imidized samples were fractured in liquid N_2 to observe the fractured surfaces. The instrument used in this experimental was Jeol JSM 840A microscope and specimens were coated with a thin layer of gold to eliminate charging effects.

3. Results and discussion

Adhesion strengths of polyimide/primer/copper systems were estimated by 90° peel test to investigate the effects of primer composition on adhesion strength. The samples were heated in a furnace with a continuous nitrogen flow at 400°C for 1 h. Four primers were used to investigate the effect of mole ratio in the copolymer on the adhesion strength: polyvinylimidazole (PVI), 7030, 3070, and polyvinyltrimethoxysilane (PVTS). For the brief notation, 7030 and 3070 represent the copolymer where the mole ratio of VI to VTS are 70:30 and 30:70, respectively. Fig. 4 represents the peel strength values of polyimide/primer/copper samples with various mole fractions in the copolymer. The peel strength value is the highest in polyimide/3070/copper system.

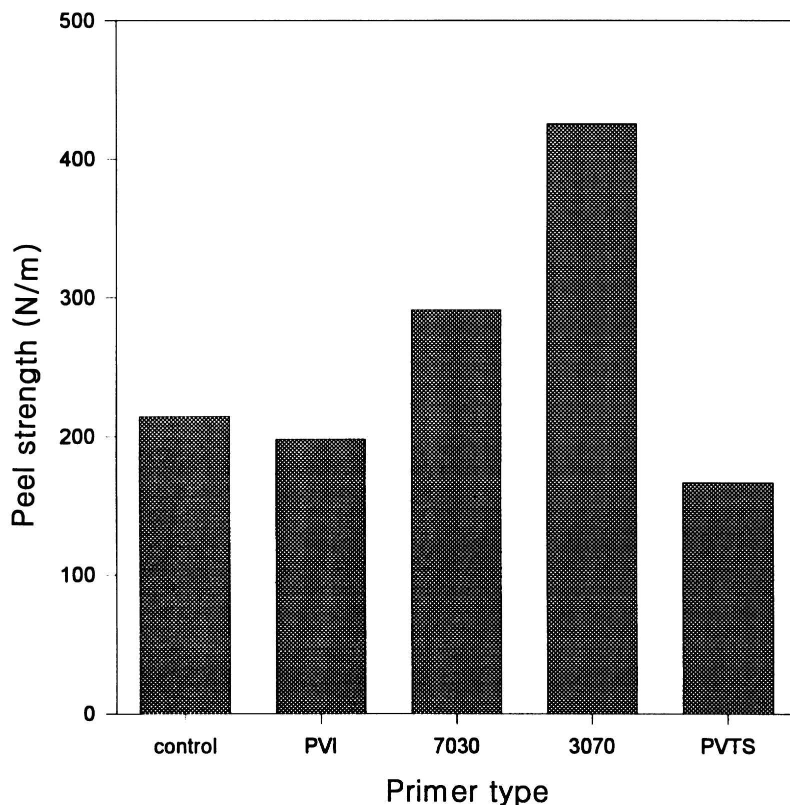

Fig. 4. The peel energy of PI/Cu and PI/primer/Cu systems with different primers.

Since the peel strength of 300 N/m or greater is acceptable to most micro-electronic applications, the adhesion of polyimide/3070/copper is considered to be strong enough [22]. The peel strengths of the other primer systems are lower than 300 N/m. In polyimide/primer/copper samples, the adhesion strength is deeply related to the interfacial phenomena. The interfacial phenomena include chemical bond formation, weak boundary layer formation, and mutual interdiffusion at the interface, among others.

To investigate the interfacial phenomena in polyimide/primer/copper systems, both the copper surface and the polymer film peeled from the copper surface were analysed with FT-IR, XPS, and SEM. Fig. 5 shows ATR and R–A spectra of polyimide/copper system after peel test. Considering the penetration depth of IR beam, ATR spectrum gives information about the film 0.8 μm below the film surface [36]. In spectrum B, the characteristic peaks of polyimide are not shown, but cupurous oxide peak at 655 cm^{-1} is observed [37]. The broad band in the 1250–900 cm^{-1} region may be due to the oxidation of polyimide at the polyimide/copper interface. Spectrum A is similar to ATR spectrum of BTDA–ODA polyimide before heat treatment. The characteristic peaks of polyimide appear at 1778, 1723, and 1665 cm^{-1} [38,39]. This suggests that the bulk property of polyimide is maintained without a significant thermal degradation in the penetration depth of IR beam. Judging from this phenomenon, thermal degradation of polyimide mainly occurred in the polyimide/copper interface.

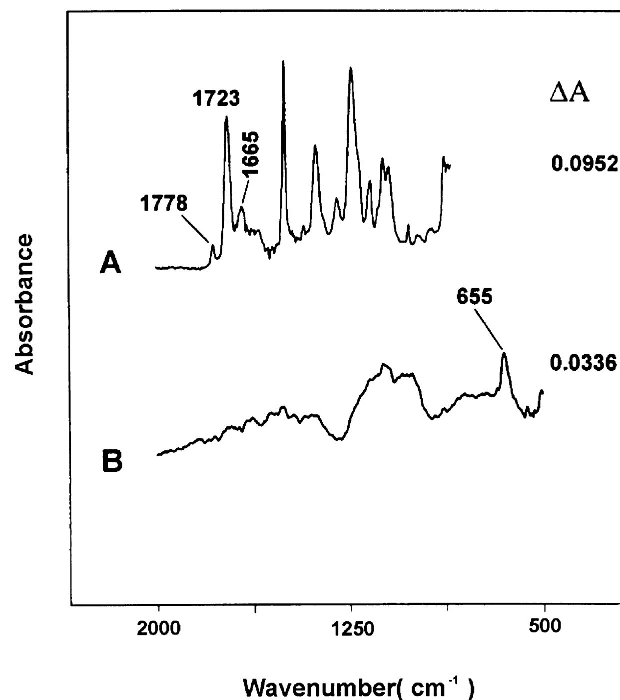

Fig. 5. IR spectra of PI/Cu system after peel test: (A) ATR spectrum and (B) R–A spectrum.

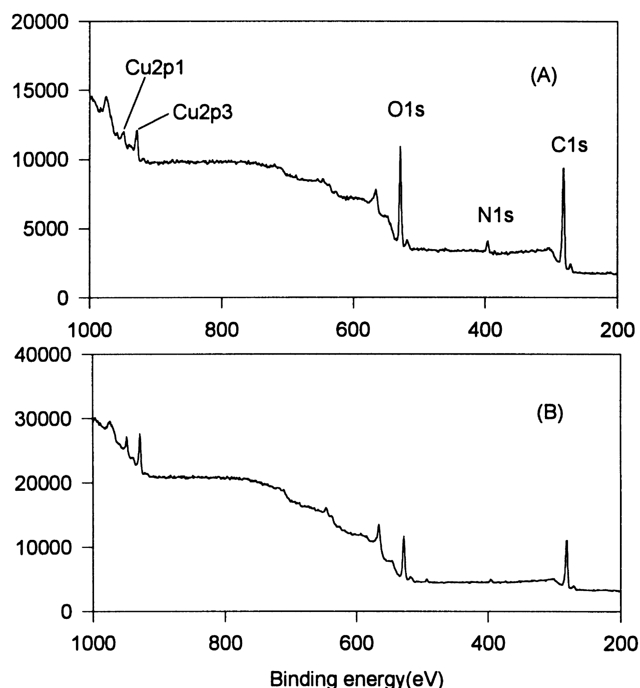

Fig. 6. XPS spectra of PI/Cu sample after peel test: (A) polymer surface and (B) copper surface.

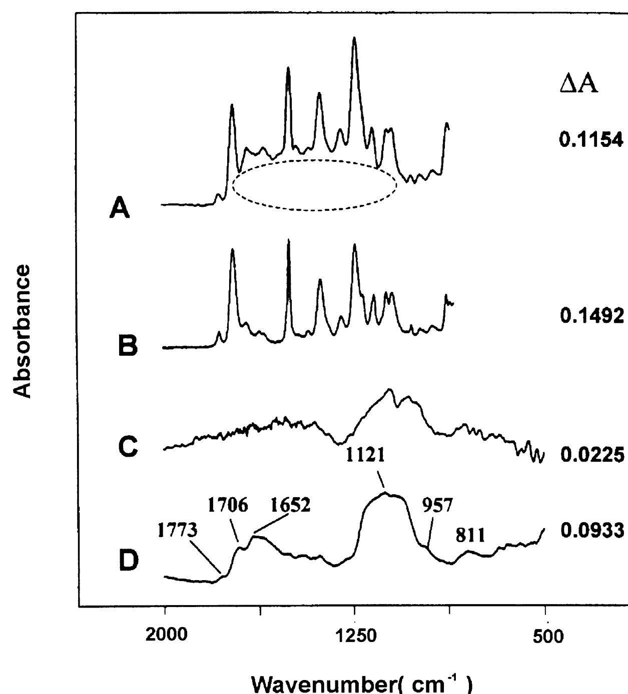

Fig. 7. IR spectra of PI/PVI/Cu system and PI/7030/Cu after peel test: (A) ATR of PVI system; (B) ATR of 7030 system; (C) R–A of PVI system; and (D) R–A of 7030 system.

XPS analysis was performed to investigate chemical composition within a few top layers from locus of failure. XPS spectra of both copper side and polymer side of polyimide/copper sample after peel test are shown in Fig. 6. Strong copper peaks are observed in the spectrum of polymer side as well as on the copper side. This means that copper ions on copper surfaces migrate into polyimide layer during heat treatment. Copper oxides in the polyimide layer deteriorate the electrical properties of polyimide due to their semiconducting properties, and decrease the mechanical properties of polyimide due to their catalytic action on thermal degradation of polyimide.

In polyimide/copper system, the copper oxide formation and thermal degradation of polyimide in the polyimide/copper interface region occurred during heat treatment. When peel test is carried out, the locus of failure of polyimide/copper system is a weak boundary region neighbouring copper substrate, which consists of copper oxides and degraded polyimide layer.

The loci of failure of polyimide/PVI/copper and polyimide/7030/copper systems were also investigated. The IR spectra of polyimide/PVI/copper and polyimide/7030/copper systems after peel test are represented in Fig. 7. In polyimide/PVI/copper system, spectrum A shows the characteristic peaks of polyimide, which are based on the broad background band designated by a dotted ellipse. This background band originates from the oxidation products of PVI during heat treatment [38,39]. In spectrum C, neither the polyimide peak nor a distinct corrosion peak is observed. The broad band in the 1300–900 cm^{-1} region appears, which is due to the thermo-oxidative products of PVI. From spectra A and C, it can be inferred that the locus of failure in polyimide/PVI/copper is the degraded PVI layer.

In the polyimide/7030/copper system, spectrum D shows both the primer and the polyimide peaks. However, no corrosion peak is observed in spectrum. This is attributed to the fact that the VTS unit in the 7030 copolymer prevents corrosion formation due to both strong chemical interaction with copper surface and thermally stable siloxane network formation [30,31]. The primer peaks at 1121, 957, and 811 cm^{-1} are related to Si–O–Si linkages [38,39]. The peaks from polyimide are observed at 1773, 1706, and 1652 cm^{-1}. The peak at 1652 cm^{-1} also overlaps with the peaks from oxidation products of the copolymer [38,39]. In contrast to spectrum D, spectrum B represents polyimide peaks only. Accordingly, a portion of polyimide and primer remains on the copper surface.

The XPS spectra of the polymer sides in polyimide/PVI/copper and polyimide/7030/copper systems after peel test are shown in Fig. 8. In the case of polyimide/PVI/copper system, the copper peak intensities are reduced in comparison with polyimide/copper system. Considering that the sensitivity factor of Cu2p3 peak is 15.87 times larger than that of C1s peak, it can be inferred that PVI layer suppresses copper diffusion into polyimide layer considerably.

In the polyimide/7030/copper system, Cu2p3 peak is marginally observed because the primer suppressed the diffusion of copper into polyimide layer. Si peaks emerge

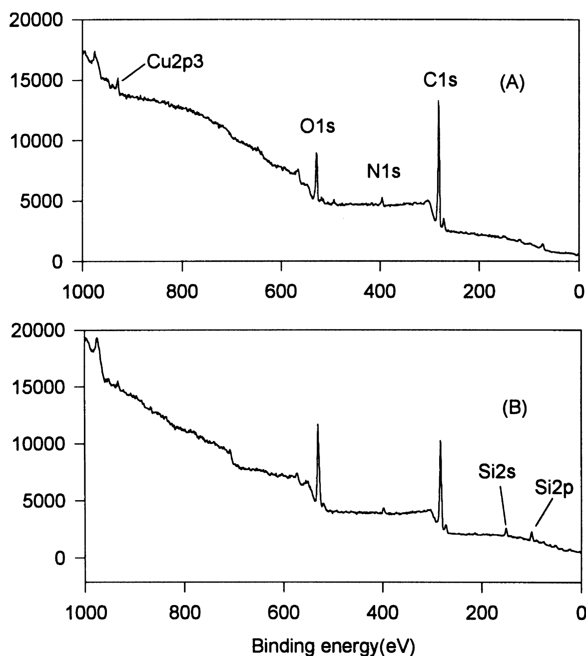

Fig. 8. XPS spectra of polymer surfaces in PI/PVI/Cu and PI/7030/Cu system after peel test: (A) PVI and (B) 7030.

at 149 and 99.5 eV as a result of the existence of the primer in the peeled-off polymer film.

Fig. 9 represents the scanning electron micrographs of fractured surfaces of polyimide/7030/copper and polyimide/PVI/copper systems before peel test. In both systems, distinct phase separation is not observed. Polyimide is compatible with both PVI and 7030 copolymer, and interphase region is formed by mutual interdiffusion at the interface.

From these FT-IR, ESCA, and SEM analyses, it can be concluded that locus of failure in polyimide/PVI/copper system is in the PVI layer neighbouring copper substrate. This is attributed to the fact that mechanical property of PVI is weakened due to thermal degradation at the copper–PVI interface. In polyimide/7030/copper system, the peeling failure mainly occurred in the interfacial region between polyimide and 7030 copolymer because the mechanical property of 7030 copolymer was improved by the introduction of thermally stable VTS unit into the primer layer. This means that the adhesion promotion in polyimide/7030/copper system is mainly due to the strengthened mechanical property of the primer layer.

The loci of failure of polyimide/3070/copper and polyimide/PVTS/copper systems were also investigated. Fig. 10 represents the IR spectra of polyimide/3070/copper system and polyimide/PVTS/copper after peel test. IR spectra of polyimide/3070/copper system are similar to those of polyimide/7030/copper system. Polymer side shows polyimide characteristic peaks only. Copper side represents some polyimide peaks at 1772, 1706, and 1652 cm^{-1}, as well as the primer peaks at 1144, 1056, and 964 cm^{-1}. No corrosion

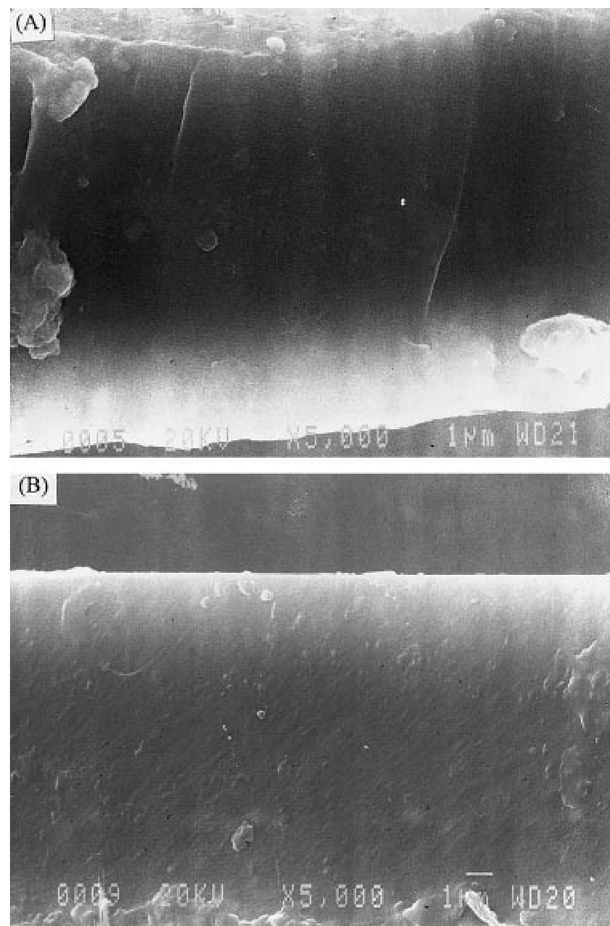

Fig. 9. Scanning electron micrographs of the fractured surface of PI/PVI film and PI/7030 film heated at 400°C: (A) PI/PVI film and (B) PI/7030 film.

peak is observed on the copper side. This suggests that 3070 copolymer not only prevents corrosion formation on copper surface, but also forms thermally stable siloxane network structure.

In the polyimide/PVTS/copper system, polymer side is made up of polyimide peaks. Copper side gives a strong Si–O–Si peak around at 1128 cm^{-1} from the primer, but does not show any polyimide peaks. The peak at 1623 cm^{-1} is assigned to the peak of the water trapped in siloxane network structure [40]. These spectra indicate the interfacial failure between polyimide and the primer.

Fig. 11 illustrates the XPS spectra of the polymer sides in polyimide/3070/copper and polyimide/PVTS/copper systems after the peel test. In polyimide/3070/copper system, no copper peak emerges and Si peaks are observed. Copper diffusion into polyimide layer is completely suppressed by 3070 copolymer. The N1s peak from polyimide and Si peaks from PVTS appear in polyimide/PVTS/copper system. The existence of copper peaks is attributed to the fact that copper migrates towards the polyimide layer through the film defects during heat treatment. PVTS film is more sensitive to defect formation in the film during heat

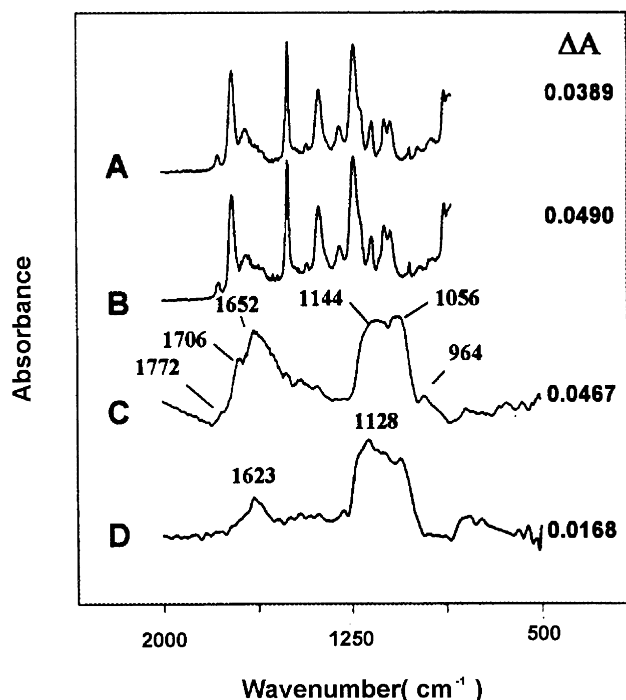

Fig. 10. IR spectra of PI/3070/Cu and PI/PVTS/Cu system after peel test: (A) ATR of 3070 system; (B) ATR of PVTS system; (C) R–A of 3070 system; and (D) R–A of PVTS system.

treatment than the other primers, and sometimes contains film defects within itself.

The scanning electron micrographs of the fractured surfaces of polyimide/3070/copper and polyimide/PVTS/copper systems before peel test are shown in Fig. 12. Compared with PVI and 7030 copolymer, mutual interdiffusion at the interface is decreased due to the poor compatibility of the primer with poly(amic acid). Especially, PVTS system shows the distinct boundary between polyimide and PVTS.

From FT-IR, XPS, and SEM analyses, it can be concluded that the adhesion promotion in polyimide/3070/copper system is attributed to the improved mechanical property of both the primer layer itself and the primer–polyimide interface region. In polyimide/PVTS/copper system, however, the adhesion is weakened due to the poor compatibility between the primer layer and polyimide layer.

Peel strengths in polyimide/primer/copper systems are deeply related to the locus of failure. The results for locus of failure and corrosion formation in polyimide/primer/copper system are summarized in Table 1. These differences in the locus of failure are attributed to the thermal stability of the primer and the compatibility of the primer with polyimide. PVI is compatible with polyimide and sensitive to thermal degradation. In the case of polyimide/PVI/copper system, the mechanical property of PVI is deteriorated during heat treatment due to its thermal degradation, and thermally degraded PVI layer acts as a weak boundary layer.

Table 1
Locus of failure and corrosion formation in polyimide/primer/copper system

Primers	Locus of failure	Corrosion formation
PVI	PVI layer	A little
7030	Primer–polyimide interface	No
3070	Primer–polyimide interface	No
PVTS	Primer–polyimide interface	A little

On the other hand, the introduction of VTS into the primer layer improves the thermal stability and the reactivity on copper of the primer, but it decreases the compatibility of the primer with polyimide. Especially, silane unit of the copolymer after heat treatment forms a thermally and mechanically stable siloxane network structure. Therefore, the locus of failure is shifted from the primer layer to the polyimide–primer interface due to the introduction of VTS into the primer layer. The chemical interaction between polyimide and the primer and the mechanical strength of the interface determines the adhesion strength of the system. In both 7030 and 3070 copolymer, failure occurs in the interface, but peel strength value is higher in 3070 copolymer. The 3070 copolymer forms mechanically and thermally stronger interface due to its higher VTS content. In the case of PVTS, it has a poor chemical interaction with polyimide in comparison with the other primers. Adhesive failure mainly occurs at the polyimide/primer interface due to the decreased compatibility of the primer with polyimide.

Copper oxide formation and the migration of copper

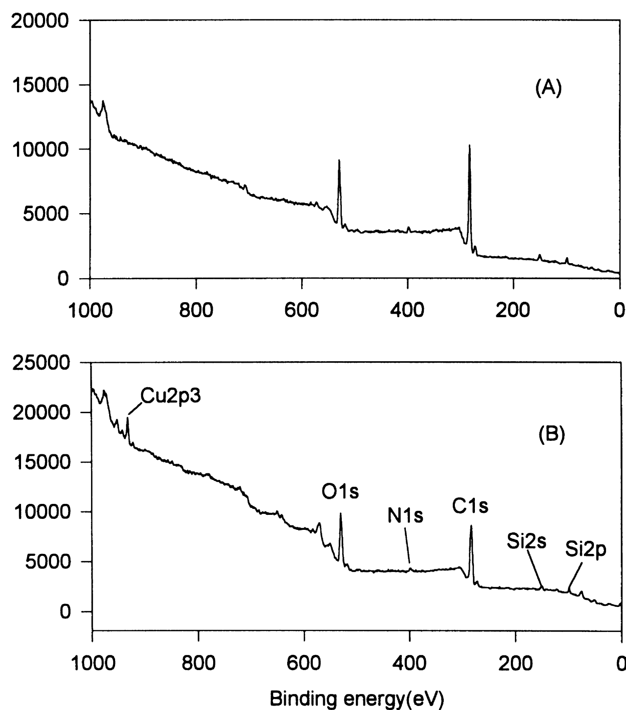

Fig. 11. XPS spectra of polymer surfaces in PI/3070/Cu and PI/PVTS/Cu system after peel test: (A) 3070 and (B) PVTS.

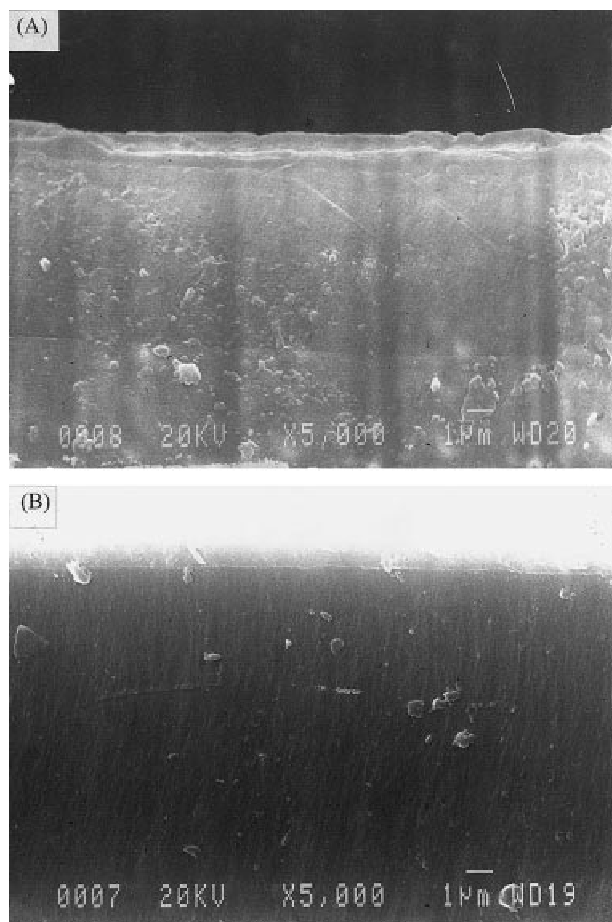

Fig. 12. Scanning electron micrographs of the fractured surface of PI/7030 film and PI/PVTS film heated at 400°C: (A) PI/3070 film and (B) PI/PVTS film.

oxide into the polyimide layer are inhibited by VTS unit of the copolymer. The 7030 copolymer and the 3070 copolymer suppressed copper diffusion, but PVTS could not due to film defect formation during heat treatment.

4. Conclusions

PVI, poly(VI-*co*-VTS), and PVTS were applied as the primer of polyimide/copper system. After heat treatment at 400°C in nitrogen atmosphere, the adhesion strengths of polyimide/primer/copper systems was the highest in the 3070 system. From FTIR, XPS, and SEM analyses, the adhesion strengths of the systems depended on the thermal stability of the primer and the chemical interaction of the primer with polyimide. PVI has good compatibility with polyimide. However, the mechanical property of PVI is deteriorated during heat treatment due to its thermal degradation, and thermally degraded PVI layer acts as a weak boundary layer. The introduction of VTS into the primer layer improves the thermal and mechanical properties of the primer layer through the siloxane network formation during heat treatment. In both 7030 and 3070 copolymers, failure occurs in the primer–polyimide interface, but peel strength value is the higher in 3070 copolymer. The 3070 copolymer forms mechanically and thermally stronger interphase due to its higher VTS. In the case of PVTS, it has a poor chemical interaction with polyimide in comparison with the other primers. Adhesive failure mainly occurs at the polyimide/primer interface due to the decreased compatibility of the primer with polyimide.

On the other hand, VTS unit played a role in the migration of copper oxide into the polyimide layer. The 7030 copolymer and the 3070 copolymer suppressed copper diffusion into the polyimide layer, but PVTS could not due to the film defect during heat treatment.

Acknowledgements

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