Adhesion Performance and Microscope Morphology of UV-Curable Semi-interpenetrated Dicing Acrylic PSAs in Si-Wafer Manufacture Process for MCP

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Abstract
UV-curable acrylic pressure-sensitive adhesives (acrylic PSAs) have many applications in industry. As the Si-wafers become thinner, the acrylic PSAs for MCP need to show proper adhesion and leave little residue on the Si-wafer after UV irradiation when released from the dicing tapes. Strong adhesion is required in the dicing process to hold the Si-wafer before UV irradiation. On the other hand, weak adhesion strength is required after UV irradiation to prevent damage to the Si-wafers during the pick-up process. This study employed semi-interpenetrating polymer network-structured dicing of acrylic PSAs in the Si-wafer manufacture process. The binder PSAs contained 2-ethylhexyl acrylate (2-EHA) and acrylic acid (AA). The adhesion performance of the peel strength on a Si-wafer was examined as a function of the UV dose. The results showed that the abovementioned two requirements were achieved using semi-IPN dicing acrylic PSAs using a hexafunctional acrylate monomer and a UV-curing system. FE-SEM and XPS revealed little residue on the wafer after removing the tape. This paper suggests the optimal conditions for the curing agent, the additional hexafunctional monomer, photoinitiator and the coating thickness.
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Keywords
Si-wafer, UV-curable PSA, semi-IPN, DPHA, FE-SEM, XPS, peel strength, hexafunctional monomer

1. Introduction

Photo-induced polymerization includes polymerization by UV light, visible light electron beam, or by laser. Photo-curing refers to polymerization that includes un-

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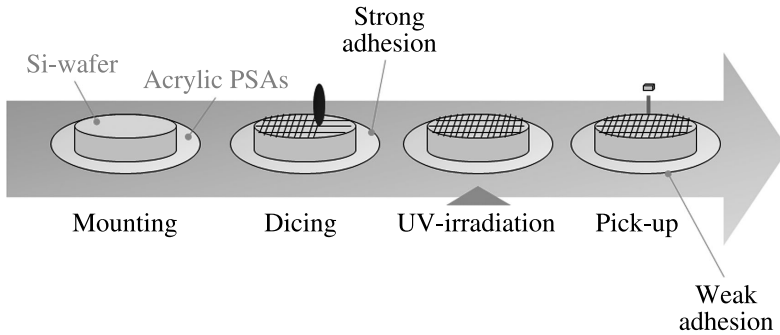
Figure 1. Schematic diagram of the dicing process on thin Si-wafer manufacture applied with acrylic PSAs [9].

saturated inks, coatings and pressure-sensitive adhesives (PSAs) [1, 2]. UV-curing systems involving photo-curing are used widely because of their rapid production rate in a small working place and solvent-free curing at ambient temperatures [2].

Adhesive tape is commonly adopted to tightly hold silicon wafers during the fabrication of semiconductor chips. Such adhesive tapes are called dicing tapes. With the rapid development of integrated semiconductor technology, a higher reliability is needed for the manufacture of electronic devices [3]. An excessive degree of cross-linking in pressure-sensitive adhesives (PSAs) prepared from unsaturated polyester resins by electron beam irradiation results in poor adhesive strength [3, 4]. This observation led us to establish a novel dicing process, as shown in Fig. 1. A piece of silicon wafer was tightly held using dicing tape with strong adhesion strength, which enabled rapid and smooth dicing of the wafer. Subsequently, the adhesive layer was irradiated with UV light through the reverse side of the substrate. After UV irradiation, the adhesion strength decreased to a certain level, where it easily picked up the diced chips for a further die-bonding process. Dicing tapes developed using this process have been reported [3, 5, 6] and similar suggestions have been made [3, 7, 8].

On the other hand, acrylic monomers and oligomers are used widely on account of their superior properties, such as transparency, colorlessness, resistance to yellowing under sunlight and resistance to oxidation due to their saturated structures. However, they have poor thermo-mechanical stability because of their linear structure. Therefore, the cross-linking of multifunctional acrylates is needed to increase their thermo-mechanical stability. Kaczmarek and Decker [2, 10] reported that an adhesive shows fluid-like behavior when it is widely cross-linked. However, the creep resistance increases greatly for a more cross-linked adhesive [2, 11]. Multifunctional acrylates cross-link rapidly by radical and cationic polymerization, and their kinetics and properties have been investigated [2, 12]. Cross-linked multifunctional acrylates produce semi-interpenetrating polymer networks (IPNs). Semi-IPN structured PSAs have advantages, such as improved heat and creep resistance [2,

11, 13]. In addition, Athawale *et al.* [14] reported that photo-polymerization was effective in obtaining networks with a high degree of interpenetration.

In the fabrication of semiconductors, PSA tapes are used for processes other than dicing, wherein the required adhesion strength is different. Accordingly, control of the peel strength by UV curing has the potential to facilitate the procedure and/or broaden the applications of PSA tapes. An acrylic copolymer itself would not change its adhesion strength with UV irradiation due to the absence of a reactive site to UV irradiation [3, 16–18]. In this study, semi-IPN dicing acrylic PSAs in a wafer manufacture process, were prepared using a hexafunctional acrylate monomer with a UV-curing system. Emphasis was placed on the optimal conditions, such as the curing agent contents, hexafunctional acrylate monomer contents, photo-initiator contents and coating thickness, which were examined using the 180° peel strength. The analysis by FE-SEM and XPS showed that there was little residue on the wafer after the release of the dicing tape after UV irradiation.

2. Experimental

2.1. Materials

The 2-ethylhexyl acrylate (2-EHA, 99.0% purity, Samchun Pure Chemical Co., Ltd, Republic of Korea) and acrylic acid (AA, 99.0% purity, Samchun Pure Chemical Co., Ltd, South Korea) were commercially available and used without purification. The ethyl acetate (EAc, Samchun Pure Chemical Co., Ltd, Republic of Korea) and methanol (MeOH, Samchun Pure Chemical Co., Ltd, Republic of Korea) were used as solvents. The chemical 2,2'-azobisisobutyronitrile (AIBN, Junsei Chemical, Japan) was used as a thermal initiator, a methylaziridine derivative (MAZ, DSM Neoresins, USA) was used as the cross-linking agent and dipentaerythritol hexacrylate (DPHA, Miwon Specialty Chemical, Republic of Korea) was used as the diluent monomer. Figure 2 shows the chemical structure of hexafunctional acrylate, which has six C=C double bonds. The compound, 2-hydroxy-2-methyl-1-phenyl-propane-1-one (Miwon Specialty Chemical, Republic of Korea) was used as the photo-initiator.
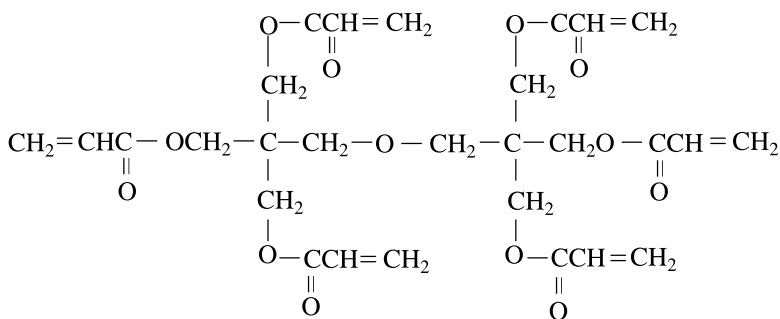

Figure 2. Chemical structure of the dipentaerythritol hexacrylate (DPHA).

2.2. Methods

2.2.1. Synthesis of Binders

Acrylic monomers (2-EHA, AA) were synthesized as 95 and 5 wt% by solution polymerization. The amount of AIBN in the binders was 0.3 phr. The mixture was placed into a 500 ml four-neck flask equipped with a stirrer, condenser and thermometer, and heated to 75°C with constant stirring. At the end of the exothermic reaction, the temperature was maintained for 30 min, and a blend of ethyl acetate and AIBN was added. The reaction was allowed to proceed for 0.5 and 2.5 h. Finally, polymerization was terminated by cooling the mixture to room temperature. The prepared pre-polymers were used as PSAs [13].

2.2.2. Formation of PSA Films

All acrylic PSAs were coated onto the corona treated polyethylene terephthalate (PET, SK Chemical, S. Korea) film using coating bars, kept at room temperature for 1 h and then dried in an oven at 80°C for 20 min. These dried films were kept at 22 ± 2°C and 60 ± 5% RH for 24 h before performing the tests [19].

2.2.3. Preparation of Cured Acrylic PSAs

The acrylic PSAs were cured using two methods: the addition of a curing agent followed by UV-curing. The curing agent was the multifunctional methylaziridine. The cross-linking of the PSAs with multifunctional methylaziridine is based mainly on the carboxyl groups of the vinyl carbonic acids within the polymeric chain. The oxygen of the nucleophilic carboxyl group causes ring opening of the tense aziridine rings, whereas the hydrogen atoms accompanying the carboxyl groups protonate the nitrogen atoms (Fig. 3) [20]. UV-curable PSAs were prepared by blending the polymerized binders with a photo-initiator and hexafunctional acrylic monomer. The UV-curable PSAs were coated onto polyester (PET) films and cured using conveyor belt type apparatus fitted with a 100 J/s high pressure mercury lamp (main wavelength: 365 nm). The UV doses were measured using an IL 390C Light Bug UV radiometer (International Light, USA). Despite low molecular weights, the hexafunctional monomers in the PSAs can be photo-polymerized after a UV dose to form semi-IPN structures (Fig. 4) [13]. The cured PSA samples were weighed and immersed in toluene for 3 days at 50°C, and then screened and dried at 80°C to a constant weight. The gel fraction of the samples was calculated using the following equation:

$$\text{Gel fraction (\%)} = W_t / W_0 \times 100, \tag{1}$$

where W_0 and W_t are the PSA weights before and after immersion, respectively [19]. The gel content provides information on the degree of cross-linking [21, 22].

2.2.4. Adhesion Performance

The peel strength was measured using a texture analyzer (Micro Stable Systems, TA-XT2i). The specimens were 25 mm in width. The specimens were pressed onto the Si-wafer substrate by 2 passes of a 2 kg rubber roller and stored at room temperature for more than 12 h. The peel strength was determined at a 180° angle with
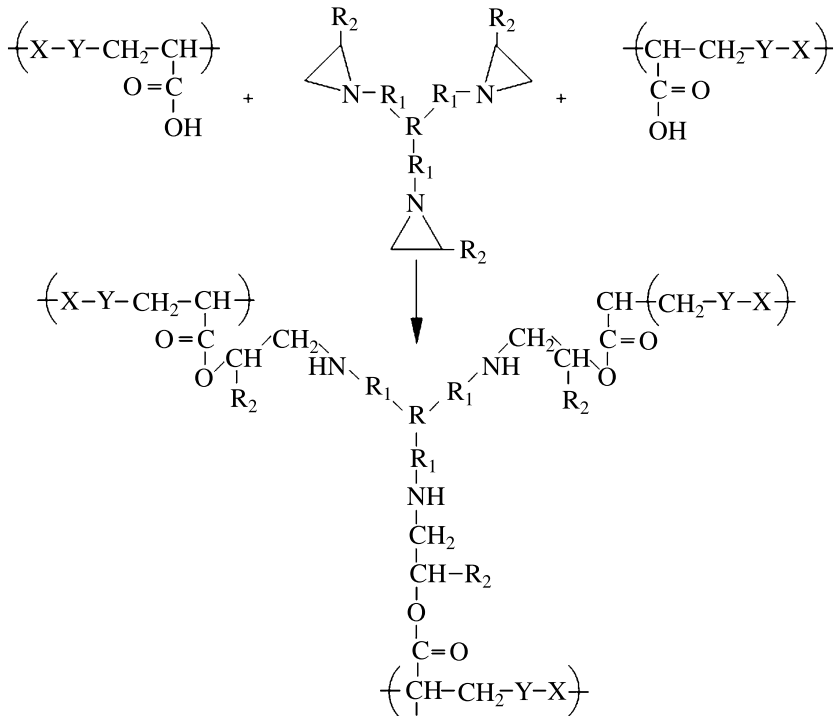
Figure 3. Crosslinking of carboxylated PSA with methylaziridine [20].

Add curing agent   Add hexafunctional monomer

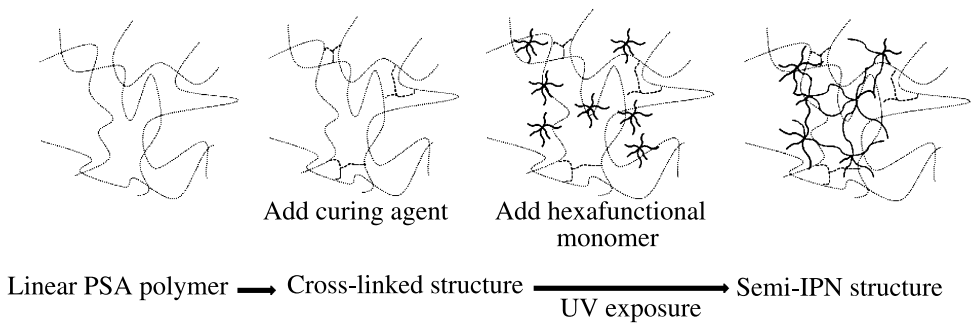Linear PSA polymer ⟶ Cross-linked structure ⟶ Semi-IPN structure
UV exposure

Figure 4. Process of producing a semi-IPN structure in UV-cured dicing acrylic PSAs [13].

a crosshead speed of 300 mm/min at 20°C based on the ASTM D3330. The peel strength was the average force on the de-bonding process. The force was recorded in g units for 5 different runs and the average force was reported as g/25 mm [9, 13].

2.2.5. Field-Emission Scanning Electron Microscopy Observation

The morphology of each sample after the peel strength test was measured by field-emission scanning electron microscopy (SUPRA 55VP, NICEM at Seoul National

University). The fractured samples were coated with a thin layer of gold (purity 99.99%) prior to the FE-SEM examination to prevent electron charging [23].

2.2.6. X-Ray Photoelectron Spectroscopy (XPS) Analysis

X-ray photoelectron spectroscopy (XPS, PHI 5000 Versa Probe, Ulvac-PHI) was performed using Al Kα radiation (1486.6 eV, Anode (25 W, 15 kV)). The binding energies were calibrated with reference to the C (1s) peak at 284.6 eV. For the measurement, the samples were placed into an ultra high vacuum chamber and data analysis of the sensitivity factors of each element present, was performed [21, 24, 25].

3. Results and Discussion

3.1. Adhesion Performance

The adhesion performance of the acrylic PSAs was determined by measuring the 180° peel strength. The peel strength decreased sharply to approximately 150 g/25 mm through the use of 1 phr (in binder) aziridine, as shown in Fig. 5. Interfacial failure, which means that there was no residue, was observed on all samples. This was attributed to networking formation of the acrylic PSAs due to the aziridine derivatives. Furthermore, the cross-linked structure indicated increased cohesion force within the acrylic PSAs. Using cross-linking agents, the increase in cohesion might be accomplished during drying of the PSA coating in the drying step. Logically, the tackifying properties of the PSA coating and peel adhesion should decrease [9, 20, 26]. Two different types of substrates were used to measure the 180° peel strength. The peel strength on a Si-wafer was lower than that on SUS when less than 2 phr (in binder) aziridine was used because the surface free energy of a Si-wafer is considered to be lower than one of SUS [27]. However, the peel strength was similar on both substrates (30–50 g/25 mm) when more than 2 phr (in binder) aziridine was used. At more than 2 phr (in binder) aziridine, all cross-linked samples affected the peel strength to a greater extent regardless of the substrate. In
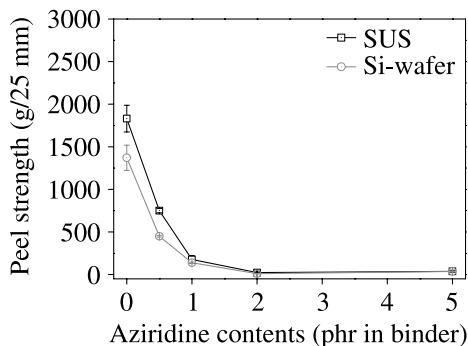

Figure 5. Peel strength of PSAs with the curing agent contents at the stainless steel (SUS) and Si-wafer.
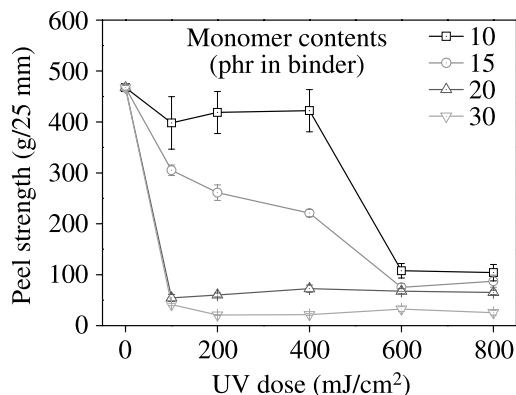
Figure 6. Peel strength of the PSAs at different hexafunctional monomer contents.

the dicing process of Si-wafer manufacture, the initial adhesion strength is an important factor because of the loss of the fitted thin Si-wafers [9]. The optimum peel strength value was attained at approximately 0.5 phr (in binder) aziridine, as shown in Fig. 5.

Figure 6 shows the effect of the UV dose on the peel strength at different hexafunctional acrylic monomer content. Soluble linear binders turned into UV-cured insoluble semi-IPN structured polymers by blending the hexafunctional acrylic monomer with the photo-initiator [13]. The hexafunctional acrylic monomer contents were 10, 15, 20 and 30 wt% (in binder). The photo-initiator content was 2 phr (in the hexafunctional monomer) in all samples. The peel strength decreased with increasing hexafunctional monomer content for all UV doses (mJ/cm^2). After UV exposure, the hexafunctional monomer formed a tightly cross-linked semi-IPN structure in the PSAs because of its short chain length. In addition, the PSA molecular mobility could be decreased because of the over-cured hexafunctional monomer, which overly increased the cohesion of the PSAs and decreased the peel strength [13]. The peel strength of the blends with monomer contents of 20 and 30 phr (in binder) decreased sharply to approximately 50 g/25 mm using an approximately 100 mJ/cm^2 UV dose and almost leveled off. However, the peel strength of the blend with a hexafunctional monomer content of 30 phr (in binder) was lower than that with a content of 20 phr (in binder). The optimum peel strength was attained using a hexafunctional monomer content of approximately 30 phr (in binder).

The influence of the photo-initiator contents on the peel strength of acrylic pressure-sensitive adhesives after UV-irradiation was examined. UV light is absorbed mainly by the photo-initiator and is attenuated progressively as it passes through the sample. The penetration of the incident photons is related directly to the photo-initiator content. Therefore, photo-initiated radical polymerization is an effective process for readily cross-linking functionalized polymers [28]. Figure 7 shows the effect of the amount of photo-initiator on the peel strength of the acrylic pressure-sensitive adhesives with the UV dose. A low peel strength was obtained at
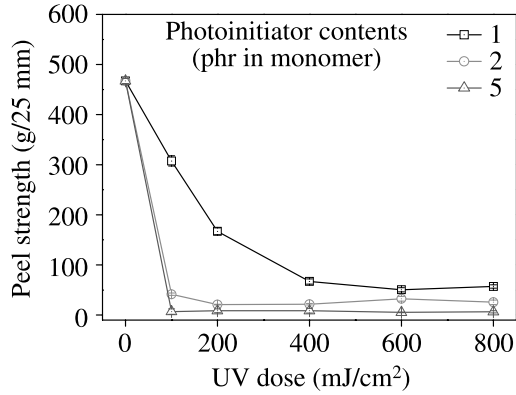
Figure 7. Peel strength of PSAs at different photoinitiator contents.
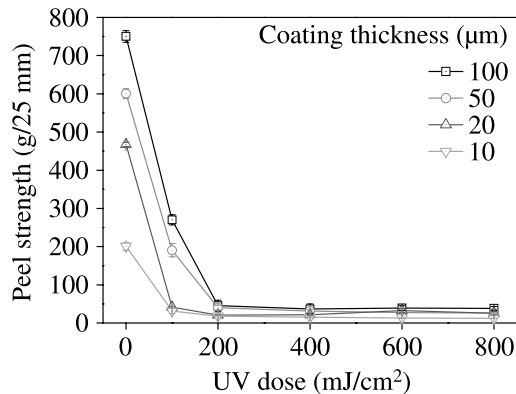

Figure 8. Effect of the UV dose on the peel strength at different coating thicknesses.

a photo-initiator content >2 phr (hexafunctional monomer) regardless of UV irradiation. The optimum peel strength was obtained with a photo-initiator content and UV dose of approximately 2 phr (in hexafunctional monomer) and 200 mJ/cm^2, respectively.

The coating thickness of the acrylic PSAs is a very important factor affecting the performance, including the peel strength. As the Si-wafers become thinner, dicing acrylic PSAs whose thickness is different from 10∼20 μm can be considered [9]. Figure 8 shows that the increase in UV dose from 0 to 200 mJ/cm^2 decreased the peel strength from approximately 450 to 20 g/25 mm at a coating thickness of 20 μm. A low peel strength was obtained at approximately <20 μm despite the UV dose being more than 200 mJ/cm^2.
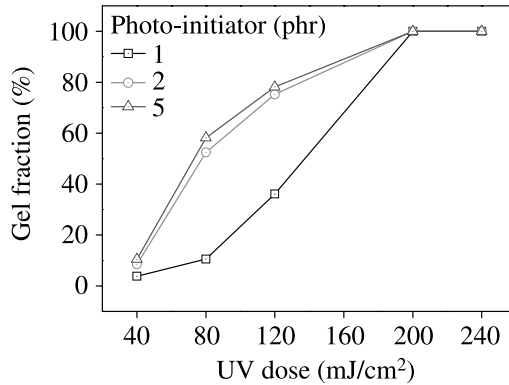
Figure 9. Gel fraction of the PSAs with hexa-functional monomers at different photoinitiator contents.

3.2. Gel Fraction of the PSAs with the Hexafunctional Monomer at Different Photo-Initiator Contents

The photo-initiator plays a key role in controlling both the rate of initiation and the penetration of incident light [21, 29]. An increase in photo-initiator concentration will accelerate the cross-linking reaction, but a small excess of organic photo-initiator can be poisonous and harmful cleavage products can form during the application [21]. Figure 9 shows the gel fraction of the PSAs with hexafunctional monomers at different photo-initiator contents (1/2/5 phr in binder). The gel content provides information on the degree of cross-linking. The gel fraction was calculated using equation (1). The gel fraction of cross-linked PSAs increased with increasing UV dose and photo-initiator content. However, the gel fraction of the PSAs was similar when more than 2 phr of the photo-initiator was used. This shows that the cross-linking reaction accelerates more than 2 phr of the photo-initiator contents. This means that the gel fraction can be controlled facilely by the UV dose and photo-initiator content [30].

3.3. FE-SEM Observations

The second aim of this study was to observe the morphology using FE-SEM (Fig. 10) and to characterize contaminants of the polymers on the surface of the Si-wafers by XPS (Fig. 11). PSAs might remain on the Si-wafers after peeling after the UV dose during the dicing process. Figure 10 shows the effect of the UV dose on the PSA remaining after peeling. In the Si-wafer manufacturing process, it is important to check whether there is any PSA remaining as the Si-wafer becomes thinner [9, 31, 32]. The increase in UV-irradiation indicates more cross-linking reactions [9, 28]. The proper UV dose in this study was found to be ∼200 mJ/cm^2.

3.4. XPS Analysis

XPS was used to determine the surface compositions of the Si-wafer after peeling at different UV doses. According to XPS (Fig. 11), the C$_{1s}$ core-level spectrum was
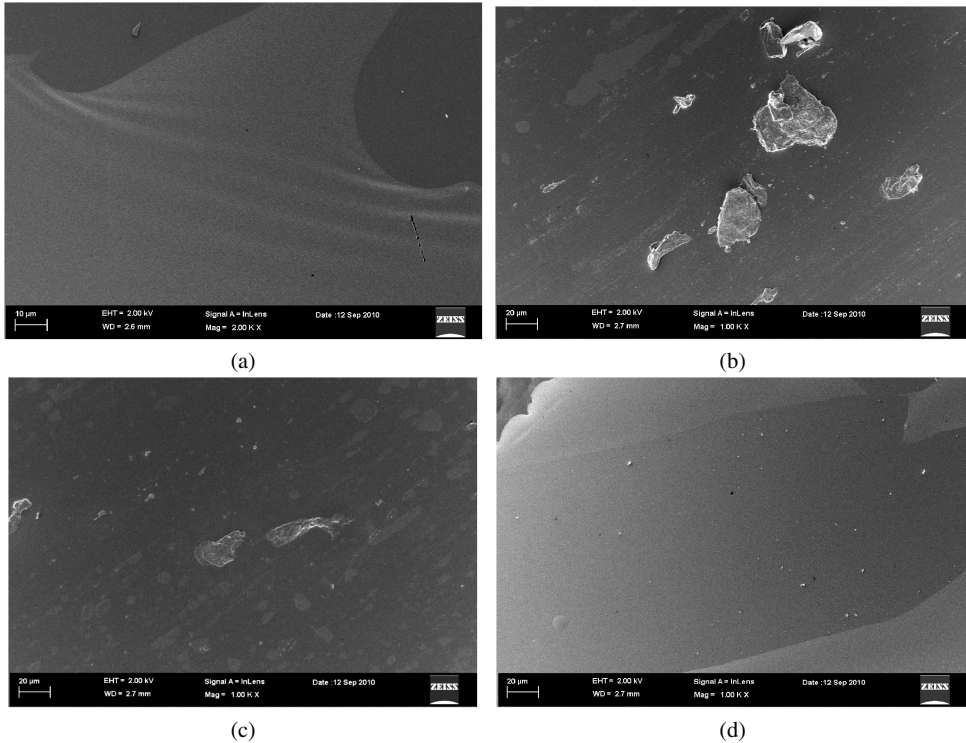
Figure 10. Field-emission scanning electron images of UV-cured acrylic PSAs at UV dose with 0 mJ/cm^2 (a), 50 mJ/cm^2 (b), 100 mJ/cm^2 (c), 200 mJ/cm^2 (d).

curve-fitted with two peak components with binding energies at 284.6 and 288.5 eV, corresponding to the $\underline{C}$–C/$\underline{C}$–H and O–$\underline{C}$=O functional groups, respectively [24, 33–35]. In addition, a peak was observed at 283.9 eV in the C_1 spectra, which was assigned to inhomogeneous charging effect on the polymer surface due to its insulating nature [36]. The above C atoms were derived from the acrylic base polymer [25]. As shown in Fig. 10, the peak at 284.6 eV, which was related directly to the characteristic of the polymer (C–C and C–H), decreased with increasing UV dose. On the other hand, the Si_{2p} peaks at approximately 100 eV and 103 eV (SiO_2) and increased with increasing UV dose. These results are in agreement with the reference values [37–39]. As listed in Table 1, the amount of carbon on the Si-wafer surface decreased with increasing UV dose with a concomitant increase in the amount of the silicon, as a result of the cross-linking of acrylic PSAs.

4. Conclusion

In general, "Multi-Chip Packages" or MCP means a packaging configuration containing at most five chips, connected *via* wire bonds to a multilayer circuit board. It is as if all the chips were integrated into one single die and packaged as such, since the same form factor and footprint are kept to facilitate subsequent board assem-
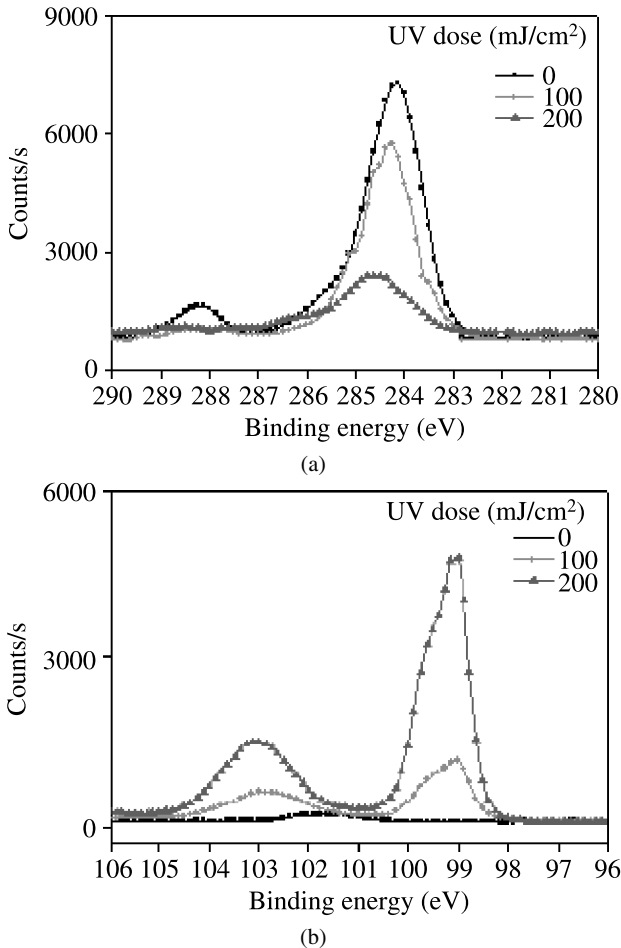
Figure 11. C_{1s} (a) and Si_{2p} (b) XPS spectra of Si-wafer surface after peeling at varying UV dose.

bly operations. For MCP, UV-curable acrylic pressure-sensitive adhesives (Acrylic PSAs) can be used in the Si-wafer handling including both dicing and pick-up process. The coating thickness has been about 40 μm so far. But as Si-wafers become ultra thin (100 → 20 μm), acrylic PSAs have to satisfy the requirements such as coating thickness below about 20 μm because of difficult Si-wafer handling.

This study examined the adhesion performance of polymers formed on Si-wafers from a hexafunctional acrylate monomer and photo-initiator using a UV-curing system. The semi-IPN structured acrylic PSAs were prepared using different UV doses. The residue on the Si-wafer was also observed by FE-SEM and XPS surface analysis after peeling, according to the UV dose. In UV-curing system of acrylic PSAs, the cross-linking reaction is influenced by: the UV dose; the diluent monomers; photo-initiators; and the coating thicknesses of the PSAs etc. In this work the effects of the curing agent, hexafunctional monomer and photo-initiator at

Table 1.

Atomic concentration (%) of C_{1s} and Si_{2p} determined by XPS according to the UV dose

UV dose (mJ/cm^2)	C_{1s}	Si_{2p}
0	81.55	1.18
100	67.32	11.72
200	19.49	39.07

a constant binder concentration, were examined. The effect of the coating thickness was assessed. A strong peel strength is needed in the dicing process of Si-wafer manufacture whereas a weak peel strength is required after UV irradiation to prevent damaging the Si-wafers. Therefore, UV-curable semi-IPN acrylic PSAs for MCP need to show two opposite adhesion properties before and after UV irradiation. The following conclusions were made:

- The optimum peel strength was attained at approximately 0.5 phr (binder) aziridine (curing agent) and 30 phr (binder) hexafunctional monomer content.

- A low peel strength was obtained at photo-initiator contents >2 phr (additional monomer) with UV exposure.

- The optimal coating thickness to achieve strong initial adhesion and weak adhesion after UV-irradiation was 20 μm.

- SEM and XPS revealed that the proper UV dose for achieving minimal residue was approximately 200 J/cm^2.

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