## Curing Behaviors of UV-Curable Temporary Adhesives for a 3D Multichip Package Process

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Temporary bonding adhesives for a three-dimensional (3D) multichip package process have been synthesized. To enhance the thermal stability, the adhesives used a fluorinated silicon urethane acrylic binder and ultraviolet (UV) curing for crosslinked network structures. Focusing on different photoinitiator contents and UV doses, the UV-curing behaviors and thermal stability were studied using Fourier-transform infrared spectroscopy attenuated total reflectance, gel fraction, swelling ratio, shrinkage, and thermogravimetric analyses.

**Key words:** Temporary bonding adhesives, curing behaviors, 3D multichip package process

## **INTRODUCTION**

Bonding of two substrates or wafers has long been an important process in the fabrication of both microelectronic systems and microelectromechanical systems (MEMS). Wafer bonding enables fabrication and packaging of complex three-dimensional (3D) microcomponents. Historically, some of the earliest uses of wafer bonding were in fabrication and packaging of pressure sensors.<sup>1–3</sup> The main commercial applications of wafer bonding are in the fabrication of silicon-on-insulator (SOI) substrates and the packaging of inertia and pressure sensors for automotive and consumer products.<sup>4–7</sup> The wide variety of wafer bonding techniques include direct bonding,<sup>5–7</sup> anodic bonding,<sup>5–7</sup> solder bonding,<sup>8</sup> eutectic bonding,<sup>9</sup> thermocompression bonding,<sup>10</sup> direct metal-to-metal bonding,<sup>11</sup> ultrasonic bonding,<sup>12</sup> low-temperature melting glass bonding, and adhesive bonding,<sup>13</sup> as presented in Table I.

In adhesive bonding, an intermediate adhesive layer is used to create a bond between two surfaces to hold them together. Although successfully used in many industries, including the airplane, aerospace, and car manufacturing industries, to join various similar and dissimilar materials, adhesive bonding did not play a significant role during the initial research on semiconductor wafer bonding. There are many applications of adhesive bonding for microelectronic and photonic components, mainly related to relatively small bonding areas of chip-level bonding. In contrast to these applications, recent research on adhesive wafer bonding involves bonding of large-area substrates using well-defined and defect-free intermediate adhesive layers. In some applications, precise wafer-to-wafer alignment of the bonded wafer pairs is required. Recent developments of reliable and high-yield adhesive bonding processes have made adhesive wafer bonding a generic and in some cases enabling wafer bonding technique for a variety of applications.<sup>1</sup>

In the most commonly used adhesive wafer bonding processes, a polymer adhesive is applied to one or both of the wafer surfaces to be bonded. After joining the wafer surfaces that are covered with the polymer adhesive, pressure is applied to force the wafer surfaces into intimate contact. The polymer adhesive is then converted from a liquid or viscoelastic state into a solid state, typically by exposing

<sup>(</sup>Received March 2, 2014; accepted July 26, 2014; published online August 27, 2014)

Table I. Commonly used wafe	ır bonding techniques <sup>1-12</sup>		
Wafer bonding technique	<b>Typical bonding Conditions</b>	Advantages and disadvantages	Application areas
Direct bonding	600°C to 1200°C Room-temperature schemes have been reported Low or no bond pressure	+High bond strength +Hermetic +Resistant to high temperatures -High surface flatness required -High bond temperatures, not always	SOI wafer fabrication
Anodic bonding	150°C to 500°C 200 V to 1500 V No bond pressure	compatible with electronic wafers +High bond strength +Hermetic +Resistant to high temperatures -Bond temperatures in combination with voltage not always compatible with elec-	Sensor packaging
Solder bonding	150°C to 450°C Low bond pressure	tronic wafers +High bond strength +Hermetic +Compatible with electronic wafers	Bump and flip chip bonding
Eutectic bonding	200°C to 400°C Low bond pressure	+High bond strength +Hermetic +Compatible with electronic wafers	Hermetic packaging Bump and flip chip bonding
Thermocompression bonding, direct metal-to-me- tal bonding	350°C to 600°C 100 MPa to 800 MPa (high bond pressure)	-Delisitive to harve oxues at surfaces +Hermetic +Compatible with electronic wafers -Very high net forces required for full wafer bonding	Wire bonding, bump, and flip chip bonding
Ultrasonic bonding	Room temperature up to 250°C High bond pressure	-High surface flatness required +Compatible with electronic wafers -Only demonstrated for small bond areas	Wire bonding
Low-temperature melting glass bonding	400-C to 1100-C Low to moderate bond pressure	<ul> <li>+fign bond strengtn</li> <li>+Hermetic</li> <li>Bond temperatures in combination with voltage not always compatible with electronic wrefver</li> </ul>	Sensor packaging
Adhesive bonding	Room temperature up to 400°C Low to moderate bond pressure	<ul> <li>High bond strength</li> <li>+High bond strength</li> <li>+Low bond temperature</li> <li>+Works practically with any substrate</li> <li>material, including electronic wafers</li> <li>-No hermetic bonds</li> <li>-Limited temperature stability</li> </ul>	MEMS, sensor packaging, 3D ICs, temporary bonds



the polymer adhesive to heat or ultraviolet light. The main advantages of adhesive wafer bonding include relatively low bonding temperatures (between room temperature and 450°C, depending on the polymer material), insensitivity to the topology of the wafer surfaces, compatibility with standard complementary metal-oxide-semiconductor (CMOS) wafers, and the ability to join practically any wafer materials. Adhesive wafer bonding does not require special wafer surface treatments such as planarization or excessive cleaning. Structures and particles at the wafer surfaces can be tolerated and offset to some extent by the polymer adhesive. While adhesive wafer bonding is a comparably simple, robust, and low-cost process, concerns such as the limited temperature stability and limited data about the longterm stability of many polymer adhesives in demanding environments need to be considered. Also, adhesive wafer bonding does not provide hermetically sealed bonds towards gasses and moisture.<sup>1,13</sup>

The process for temporary wafer bonding is shown in Fig. 1.<sup>14</sup> The front side of the carrier wafer is coated with the adhesive, and the wafer undergoes an initial bake to remove the solvent. The device wafer (possibly after application of a protective layer) is brought into contact with the adhesivecoated carrier wafer under vacuum and pressure. The adhesives used for the bonding process must adhere to a variety of semiconductor substrates, such as silicon, gallium arsenide, and indium phosphide, as well as different metals, insulators, and dielectrics. The adhesive layer between the device and the carrier wafer provides the mechanical strength required for handling thin wafers. Adhesives should possess flow properties to flow into structures on the front side of the device wafer to provide good bonding properties. In addition, the adhesives must be easy to apply, have suitable mechanical strength, thermal stability, and chemical resistance, and exhibit thickness variation across large wafers. Also, temporary wafer bonding requires the adhesive to be easily removed without damaging the features on the active side of the device wafer over a short debonding time. At present, only a few adhesives are available for this type of application. The tapes currently used for temporary wafer bonding have limited thermal stability at higher temperatures and can be used only up to 170°C to 200°C.  $^{14-18}$ 

UV-curing technology has been considered as an alternative to traditional solvent-borne coatings, due to its ecocompatible process and excellent properties resulting from its high crosslink density. On the other hand, acrylic monomers and oligomers are used widely. However, they have poor thermomechanical stability. Therefore, crosslinking of multifunctional acrylates is needed to increase their thermomechanical stability.<sup>19–21</sup> Athawale<sup>2</sup> reported that photopolymerization was effective for obtaining networks with a high degree of interpenetration. The curing kinetics and peel strength of dual-curable adhesives based on acrylic resins have been reported.<sup>23,24</sup> To combine the functionalities of UV and heat curing in an epoxy acrylate oligomerbased system, a thermal curing agent should be introduced.<sup>2</sup>

Polyorganosiloxane contains Si–O bonds, which allow flexibility of the chain as well as good chemical resistance, corrosion resistance, water resistance, and thermal and oxidative stability.<sup>30</sup> The interesting and useful properties of siloxane polymers are a result of the somewhat unusual molecular structure of the polymer chains, which consist of alternating silicon and oxygen atoms.<sup>31</sup> Polyurethane (PU) chemistry is involved in isocyanate reactions with macrodiols, diisocyanate, and chain extenders.<sup>32</sup>

Fluorinated polymers have several important properties; For instance, they are highly resistant to different thermal, chemical, and weather conditions. The presence of fluorine in the side groups of the polymer has a profound effect on nearly all the properties of these materials.<sup>33–35</sup> This effect is so striking that fluoropolymers are often treated as a separate and remarkable class of polymers with properties unmatched by any other system. Fluorine confers extreme hydrophobicity and water insolubility to polymers. It raises the thermal and oxidative stability. In some instances, the element generates an interchain interaction so strong that it prevents the polymer from dissolving in any solvent. It is the C-F bond that grants such properties, rather than the element itself.<sup>36</sup> Therefore,

Table II. Raw mat	erials used for synthesis of f	fluorinated silico	on urethane acrylic adhesives		
Function	Material	Abbreviation	Chemical structure	Molecular weight (g/mol)	Supplier
Polyol	Hydroxy-terminated carbinol polysiloxane	1	HO Contraction of the second s	940	Shin-Etsu Co., Ltd.
Isocyanate	Isophorone diisocyanate	ICAI	H <sub>5</sub> C OH <sub>4</sub> NCO	228	Bayer Material Science
End-capping agent	1 <i>H</i> ,1 <i>H</i> ,7 <i>H</i> -Dodecafluoro-1- heptanol	DFH	L L L L L L L L L L L L L L L L L L L	332	Tokyo Chemical Industry Co., Ltd.
End-capping agent	Pentaerythritol triacrylate	PETA		298	Sigma Aldrich
Acrylic monomer	Glycidyl methacrylate	GMA		142	Junsei Chemicals
Acrylic monomer	Acrylic acid	AA	o P	72	Samchun Chemicals



fluorine-containing polymers have widespread applications in modern technologies ranging from the building, automotive, and aerospace industries to optics and microelectronics.<sup>37–39</sup>

In this work, UV-curable temporary bonding adhesives were synthesized with a hydroxy-terminated carbinol siloxane for a urethane reaction and with a hydroxy-terminated fluoro-monomer to improve the thermal stability. The incorporation of carbinol siloxane into the soft segments of the PU backbone was carried out by a reaction between the NCO and OH groups in the diisocyanates and hydroxy-terminated carbinol siloxane, respectively. The curing behavior and thermal stability were investigated after the synthesis and curing process.

#### EXPERIMENTAL PROCEDURES

## Materials

Table II presents the chemical structures and properties of the raw materials used for the synthesis process. Hydroxy-terminated carbinol polysiloxane (Shin-Etsu Co., Ltd.) and isophorone diisocyanate (IPDI; Bayer Material Science) were dried at 100°C. 1H,1H,7H-dodecafluoro-1-heptanol (Tokyo Chemical Industry Co., Ltd.) and pentaerythritol triacrylate were used as end-capping materials without pretreatment. Approximately 0.1 wt.% dibutyltin dilaurate was added as a catalyst to cause the reaction to take place at a rapid rate in a urethane reaction. Hydroxydimethyl acetophenone (Micure HP-8; Miwon Specialty Chemical) was used as the photoinitiator for UV curing, as shown in Fig. 2.40 Glycidyl methacrylate (GMA; Junsei Chemicals, Japan) was used as donating epoxy functional group with the carboxyl group of acrylic acid (AA; Samchun Chemicals).

# Synthesis of Fluorinated Urethane Acrylic Binders

Figure 3 shows the synthesis process scheme of the dual-curable hydroxy-terminated carbinol siloxane-modified fluorinated urethane acrylic adhesives. The reaction time was determined by observing changes in the Fourier-transform infrared (FT-IR) peak at 2265 cm<sup>-1</sup> (NCO peak), which decreased with the PU reaction. Initially, IPDI was charged into a dried 300-mL round-bottomed flask equipped with a four-necked separable flask with a mechanical stirrer, thermometer, and condenser with a drying tube and an N<sub>2</sub> inlet. The temperature was maintained at room temperature with constant stirring. The hydroxy-terminated carbinol polysiloxane with some of the catalyst (dibutyltin dilaurate) was then added dropwise over a period of 5 h and was maintained for a further 1 h. The reaction temperature was increased to  $50^{\circ}$ C using a constant-temperature heating mantle with constant stirring. A mixture of 1H, 1H, 7H-dodecafluoro-1heptanol and pentaerythritol triacrylate was added dropwise over a period of 1 h and was reacted for 3 h until the NCO peak almost disappeared. After cooling to ambient temperature, GMA and AA were mixed. Finally, a photoinitiator was blended to initiate UV curing.

#### **Curing Process**

## Fourier-Transform Infrared (FT-IR) Spectroscopy

IR spectra were obtained using a JASCO FTIR-6100 (Japan) equipped with an attenuated total reflectance (ATR) accessory. To obtain the IR spectra of dual-curable adhesives, the cured adhesive samples were cut into 0.5 cm  $\times$  0.5 cm pieces. The ATR prism was a diamond with refractive index at 1000 cm<sup>-1</sup> of 2.4 and transmission range from 4000 cm<sup>-1</sup> to 650 cm<sup>-1</sup>. The resolution of the recorded spectra was 4 cm<sup>-1</sup>. The curing behavior of the dual-curable adhesives was analyzed by observing the changes in the C=C bonding bands. All FTIR ATR spectra were modified by baseline correction. The conversion (%) was calculated by the equation<sup>41</sup>

Conversion (%) = 
$$\left[ (A_{810}/A_{1730})_0 - (A_{810}/A_{1730})_t \right] / (A_{810}/A_{1730})_0 \times 100,$$
 (1)

where  $(A_{810}/A_{1730})_0$  and  $(A_{810}/A_{1730})_t$  denote the relative absorbance of the C=C bonds before curing and at given curing time *t*, respectively.

#### Gel Fraction and Swelling Ratio

Gel fraction determination is a convenient method of measuring insoluble fractions, such as the fractions of crosslinked or network polymers.<sup>23</sup> The gel fraction is the insoluble part, while the swelling ratio is the fraction of the polymer swollen by the solvent. Both are significant factors when investigating curing behaviors. The gel fraction of the dual-curable adhesives after UV curing was determined by soaking in toluene at 50°C for 24 h. The insoluble part was removed by filtration and



Fig. 3. Synthesis process of dual-curable fluorinated silicon urethane acrylic adhesives.

dried at 50°C to constant weight. The gel fraction and swelling ratio were calculated by applying the following equations, respectively:

$$Gel fraction = (W_1/W_0), \tag{2}$$

Swelling ratio = 
$$1 + [(W_s - W_1)/(W) \times p/p_0],$$
 (3)

where  $W_0$  is the initial weight of the sample,  $W_s$  is the insoluble swollen part, W is the weight of the dry insoluble part, p is the density of the solvent, and  $p_0$ is the density of polymer.<sup>42</sup> The test was replicated three times.

#### Shrinkage

To investigate the degree of linear shrinkage during the UV-curing process, 0.05 g of the dualcurable adhesive material was placed on a round stainless-steel plate ( $\emptyset$ 0.4 mm) and covered with a glass slide (75 mm × 25 mm × 1 mm), after which UV light was irradiated for about 600 s using spotcuring equipment. The average UV light intensity at the sample was approximately  $10 \text{ mW/cm}^2$  over a wavelength range of 300 nm to 400 nm. The displacement of the samples during the UV-curing process was recorded by software.

#### **Thermogravimetric Analysis**

The thermal stability and decomposition profile of the dual-curable adhesives were measured using a thermogravimetric analyzer (Thermogravimetric Pyris 1 TGA; PerkinElmer). The samples were loaded into a ceramic pan, and heated from 30°C to 500°C at constant heating rate of 10°C/min in an inert nitrogen atmosphere.

## **RESULTS AND DISCUSSION**

## FTIR ATR Spectroscopy

After photoinitiation of the adhesives by UV irradiation, the specific bands of the functional monomers and/or oligomers indicated that polymerization had occurred. The curing reaction can be measured using FTIR ATR spectroscopy because the C=C bonds in the functional monomers participate in the crosslinking



Fig. 4. UV-curing profiles of UV-curable adhesives with different photoinitiator contents by FTIR ATR spectra.



Fig. 5. Gel fraction of UV-curable adhesives with different photoinitiator contents after UV curing.

reaction by photopolymerization.<sup>43,44</sup> Figure 4 shows the conversion of acrylate bonds according to the FTIR ATR spectra. It can be seen that, as the UV dose was increased, the conversion of acrylate bonds increased rapidly then became constant regardless of the photoinitiator content. However, the photoinitiator content had an effect on the conversion rate. This occurred because the conversion of acrylate bonds is based on the functionality of the monomer: the more radical initiation, the faster the increase in the conversion of the acrylate bonds. Despite this, the conversion rate was not 100%. The remaining C=C bonds may have remained unreacted after the action of the photoinitiator because they were trapped in the crosslinked polymer network.<sup>20</sup> Moreover, the unreacted monomers inhibit the thermal stability of adhesives.

## **Gel Fraction and Swelling Ratio**

Figure 5 shows the relationship between the UV dose and the gel fraction of UV-cured adhesives



Fig. 6. Swelling ratio of UV-curable adhesives with different photoinitiator contents after UV curing.



Fig. 7. Correlation between swelling ratio and gel fraction of UV-curable adhesives.

with different photoinitiator contents. As the UV dose increases, the concentration of free radicals increases, causing the crosslinking density to increase due to a three-dimensional crosslinked insoluble and infusible network. The gel fraction was constant regardless of the photoinitiator content for UV doses higher than  $500 \text{ mJ/cm}^2$ . The gel fraction with 0.5 phr of photoinitiator increased with an increase in the UV dose up to about 63% gel fraction at a UV dose of 2500 mJ/ cm<sup>2</sup>. However, the gel fraction increased to around 83% for 4.0 phr of photoinitiator; That is, at low photoinitiator content, the gel content conspicuously increased, owing to the existence of unreacted monomer, but the rate of increase of the gel fraction decreased with an increase in the UV dose, because the amount of unreacted monomer decreased.<sup>4</sup>

On the other hand, the swelling ratio decreases rapidly with the dose, as shown in Fig. 6, indicating



Fig. 8. Linear shrinkage of UV-curable adhesives during UV irradiation.



Fig. 9. Thermogravimetric analysis of UV-cured adhesives with different UV doses.

that less space between the polymer chains is available for swelling to occur. Figure 7 shows nearly linear dependence between the swelling ratio and gel fraction, suggesting that the processes of crosslinking and chain scission occur at the same time. The variation of the two parameters with the dose may be due to oxidation-induced chain scission and loosening of the network. In high-dose samples, the radical concentration is high and more chain scission reactions are induced, continuing for some time after UV irradiation.<sup>46</sup>

Considering that, for higher radiation doses, scission reactions are supposed to compete with crosslinking, the increase in the extent of cross-linking can be accounted for by the fact that chain scissions lead to entanglement couplings which act as crosslinks.<sup>47</sup> The main chain degradation is ultimately accompanied by crosslinking.

#### Shrinkage

Figure 8 depicts the typical linear shrinkage of UV-curable adhesives with reference to the irradiation time. Initially, the shrinkage of each sample increased sharply regardless of the photoinitiator content. However, as the irradiation time elapsed, the shrinkage increased slightly and then stabilized. Also, the degree of linear shrinkage was dependent on the photoinitiator content. In general, the polymerization shrinkage of monomers and oligomers is due to the conversion of the intermolecular van der Waals forces to covalent single bonds during the polymerization process.<sup>48</sup> Hence, the shrinkage is the consequence of the crosslinking reaction between the multifunctional acrylic oligomer and the monomer.

## **Thermogravimetric Analysis**

Figure 9 shows the thermal stability of UV-cured adhesives at temperatures ranging from 30°C to 500°C.

The weight loss at temperature under 300°C was due to release of trapped volatile materials.<sup>49</sup> The thermal decomposition of the crosslinked PU then started via degradation of the polymer side-chains, occurring at about 300°C. Temperature above 450°C led to complete de-crosslinking and thermal degradation of the cured adhesives.<sup>50</sup> Also, the thermal stability was enhanced with an increase in the UV dose due to the increased density of the crosslinked structures, corresponding to the gel fraction results presented above.

#### CONCLUSIONS

Adhesive wafer bonding is a promising technology that provides unique possibilities for fabrication and improvement of microsystems. Process schemes and parameters for temporary bonding adhesives with fluorinated urethane acrylic functionalities were investigated in this work. The objective is not only to determine the UV curing behavior and thermal stability from different tests but also to establish optimized conditions of photoinitiator content and UV dose. The results show that the degree of thermal stability may be more dependent on the photoinitiator content than on the UV dose, whereas many other factors such as the molecular weight, physical crosslinking density, concentration of free radicals, degree of oxidation, temperature, and humidity influence the relative UV curing of this mechanism. Such factors need to be investigated in future work.

#### ACKNOWLEDGEMENTS

The authors would like to acknowledge financial support from the R&D Convergence Program of the Ministry of Science, ICT, and Future Planning (MSIP) and Korea Research Council for Industrial Science and Technology (ISTK) of the Republic of Korea.

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