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A comparative study of the bonding energy in adhesive wafer bonding

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Abstract

Adhesion energies are determined for three different polymers currently used in adhesive wafer bonding of silicon wafers. The adhesion energies of the polymer off-stoichiometry thiol-ene-epoxy OSTE+ and the nano-imprint resist mr-I 9150XP are determined. The results are compared to the adhesion energies of wafers bonded with benzocyclobutene, both with and without adhesion promoter. The adhesion energies of the bonds are studied by blister tests, consisting of delaminating silicon lids bonded to silicon dies with etched circular cavities, using compressed nitrogen gas. The critical pressure needed for delamination is converted into an estimate of the bond adhesion energy. The fabrication of test dies and the evaluation method are described in detail. The mean bond energies of OSTE+ were determined to be 2.1 and 20 J m⁻² depending on the choice of the epoxy used. A mean bond energy of 1.5 J m⁻² was measured for mr-I 9150XP.

(Some figures may appear in colour only in the online journal)

1. Introduction

Adhesive wafer bonding has become an established technology in microelectromechanical systems (MEMS) integration and packaging applications [1]. It typically consists of spin-coating a polymer adhesive on top of one or both wafers, followed by pressing the wafers together at an elevated temperature. The bond force enables the wafers to come in close contact to each other while the elevated temperature induces a polymerization reaction in the bonding polymer, thus creating a bond. Advantages of adhesive wafer bonding compared to other wafer bonding technologies (fusion, anodic, eutectic etc) [2–5] include better integrated circuit compatibility due to relatively low-temperature processes, insensitivity to particles and surface topology due to plastic reflow and deformation of the polymer adhesive at the bond interface and a wide design window for polymer coating thicknesses [1]. Furthermore, the polymers used in adhesive wafer bonding typically adhere to most materials, which opens up for joining practically any kind of wafer materials to each other [1].

Different polymer adhesives used in adhesive wafer bonding feature different advantages and disadvantages, and a wide

variety have been evaluated, such as polyimides [6, 7], epoxies [8–10], benzocyclobutene (BCB) [11–15], UV-curable epoxies (i.e. SU8) [16–21] and nano-imprint resists [1, 22]. BCB exhibits high bond energies and a high-yielding bonding process [12, 13]. Some BCB-formulations can also be photo-patterned prior to bonding to create 3D structures and cavities for housing MEMS devices [23]. However, BCB forms a chemically inert layer after curing, making it difficult to remove. This limits the application of BCB in MEMS applications in which the adhesive polymer is used as a sacrificial layer [24]. Specific nano-imprint resists have also been proposed as wafer bonding adhesives for heterogeneous integration applications [22]. Their main advantage compared, with BCB, is that nano-imprint resists can be easily removed after curing by dry etching with an oxygen plasma [22, 25, 26]. These nano-imprint resists are thus suitable as sacrificial layers in MEMS fabrication. The drawback of nano-imprint resists is typically low bond strengths compared to BCB [22]. Off-stoichiometry thiol-ene-epoxy, OSTE+, was recently shown to be feasible for adhesive wafer bonding applications [27]. Easy removal after curing, photo-patterning capability, low-temperature processing and a comparably high bond

Figure 1. (A) Burst pressure evaluation method. Adhesively wafer bonded Si lids are pressurized from the backside with compressed air. (B) The lids are delaminated and the critical burst pressure is recorded.

energy make it an attractive material for adhesive wafer bonding [28]. The temperatures needed during the adhesive wafer bonding process differ between these three materials. BCB typically requires a temperature above 200 °C during the curing step while the nano-imprint resist mr-I 9150XP (Micro resist technology GmbH) cures at 160–200 °C [1]. The epoxy-component in OSTE+ starts to cure directly after spin-coating at room temperature and the curing is accelerated at elevated temperatures (typically below 100 °C) [28].

High adhesion energy between adhesive polymers and wafer surfaces is an important requirement irrespective of the adhesive polymer used. The adhesion energy is defined as the energy released per unit of new surface created when separating surfaces from each other [29]. In this work, the adhesion energy is determined for the three polymer systems presented above (OSTE+, mr-I 9150XP and BCB).

2. Experimental details

2.1. Evaluation method

The adhesion energy of an adhesive bond is dependent on the material interfaces at the bond, the use of an adhesion promoter and the layer thickness of the polymer adhesive, among other parameters. The adhesion energies in this work are studied by blister testing, i.e. delamination of silicon lids, either 125 or 300 μm thick, bonded to silicon dies with etched circular cavities. The critical pressure needed for delamination is converted into an estimate of the bond adhesion energy. The method, illustrated in figure 1, consists of pressurizing an adhesively bonded Si lid with compressed nitrogen gas. The critical pressure needed to delaminate the lid from the silicon die that it is bonded to is measured with a pressure sensor, that is connected to a LabView interface. The recorded burst pressure is then transformed into its equivalent bonding energy.

2.2. Method to convert the critical pressure for delamination of lids into its equivalent bonding energy

Dies with etched circular through holes and adhesively bonded Si lids on top were put into a fixture and pressurized from

Figure 2. (A) Defining and etching holes in the SiN layer. (B) KOH Etching through the wafer. (C) Removal of the SiN layer. (D) Defining and etching 100 μm deep circular cavities into the wafer.

the backside with nitrogen gas. The pressure was increased manually in a slow and continuous ramp until the lids delaminated from the prepared dies (figure 1). The pressure at the start of delamination (burst pressure) was converted into a bond energy per unit area according to [29]

$$P_{\text{cr}} = \left(\frac{32E\gamma_a h^3}{3a^4(1-\nu^2)} \right)^{\frac{1}{2}}. \quad (1)$$

Where P_{cr} is the burst pressure, h is the wafer thickness of the lid, a is the radius of the circular cavity, ν is Poisson's ratio, E is Young's modulus for Si and γ_a is the adhesive fracture energy per unit area. Young's modulus was chosen to be 180 GPa, which applies to silicon diaphragms that are deformed under pressure [30]. Poisson's ratio was estimated to be the average of Poisson's ratio in $\langle 1\ 0\ 0 \rangle$ and $\langle 1\ 1\ 0 \rangle$ -directions, i.e. 0.172, since both axes are put under strain [30]. Equation (1) is deduced from Clapeyron's theorem [29] and holds for diaphragms that deform elastically and that are thin compared to the diameter of the circular cavity. The critical pressure needed to delaminate the lid decreases with increased diameter of the circular cavity. The highest pressure needed for delamination is thus reached when delamination of the lid starts, thereby driving the delamination to the edges of the lid, where it is torn off from the die.

2.3. Fabrication of blister test devices

2.3.1. Fabrication of the structured wafer. Sample devices were fabricated by depositing a hard mask protective layer consisting of 60 nm LPCVD silicon nitride (SiN) on 525 μm thick Si wafers. $3 \times 3\ \text{mm}^2$ through holes were photolithographically defined. The wafers were then etched. Firstly, with an anisotropic dry etch to open up holes in the SiN layer (figure 2(A)) and secondly in a 30% KOH solution kept at 60 °C until holes had been etched through the Si wafer (figure 2(B)). The SiN hard mask was then stripped in a phosphoric acid solution kept at 120 °C to retrieve clean silicon surfaces on the wafers (figure 2(C)). The next step consisted of etching 12 mm in diameter circular cavities in the Si wafers. This dimension was chosen to enable lid delamination even

when using moderate pressures (i.e. less than 10 bar). The wafers were photo-patterned and circular cavities with a depth of 100 μm were etched into the Si wafers, using deep reactive ion etching (figure 2(D)). The etched Si wafers were then adhesively bonded to plain Si wafers that had been spin-coated with the bonding polymers that were to be evaluated.

2.3.2. Wafer treatment process before adhesive wafer bonding. All experiments were conducted on 100 mm diameter Si wafers containing a thin native oxide layer.

Two different thiol-ene-epoxy (OSTE+) formulations were evaluated as adhesives for wafer bonding. The first being the commercially available OSTEmer(tm) 321 (Mercene Labs, Sweden), hereafter referred to as blend 1. The other being a thiol-ene-epoxy consisting of PTMP (pentaerythritol tetrakis(2-mercaptoacetate)), TATATO (triallyl-1,3,5-triazine 2,4,6 trione) and BADGE (Bisphenol A diglycidyl ether), in the functional molar proportion PTMP:TATATO:BADGE 1.5:1:0.5, with 0.5% TPO-L as radical polymerization initiator and 0.25% DBN (1,5-Diazabicyclo[4.3.0]non-5-ene) as an anionic polymerization initiator, hereafter referred to as blend 2. All monomers and initiators for blend 2 were obtained from Sigma-Aldrich. The difference between blend 1 and blend 2 is primarily in the epoxy used, where blend 1 uses the epoxy D.E.N. 431 Epoxy Novolac (Enorica GmbH) and blend 2 uses the epoxy BADGE. The polymer blends were diluted 1:1 by weight with toluene (Merck KGaA). Both diluted OSTE+ and undiluted OSTE+ were used to spin-coat 300 μm thick Si wafers at 6000 rpm. For the wafer covered with diluted OSTE+ of blend 1, no increased temperature was used (i.e. soft baking) to avoid unwanted curing of the polymer. Instead, the toluene solvent was removed by exposing the coated wafer to vacuum at room temperature for 10 min. This resulted in a 3.9 μm thick polymer layer. For the wafer covered with diluted OSTE+ of blend 2, a soft baking for 2 min at 50 $^{\circ}\text{C}$ followed by vacuum exposure was used to remove the solvent. OSTE+ has a dual cure mechanism, in which the first curing step is driven by UV exposure and the second curing step is accelerated by the use of elevated bonding temperatures [28]. The first cure of the OSTE+ dual cure system was initiated by a 20 s broadband UV exposure in a mask aligner for a total dose of 400 mJ cm^{-2} . This curing step drives a reaction between the tetrathiol and the triallyl molecules to create a loosely cross-linked polymer network. After this step the polymer coating is a solid containing unreacted thiol and epoxy groups, resulting in a surface that is slightly sticky. The second thermal curing step drives the reaction between the epoxy component in OSTE+ and the unreacted thiol groups left in the polymer after the first UV cure. The epoxy polymerization in the second curing step ensures the adhesion of the polymer to the wafer surface in the subsequent wafer bonding step. At the end of the thermal cure, all thiol and epoxy groups have reacted to form a highly cross-linked polymer network with a high glass transition temperature (T_g) and a high Young's modulus.

Likewise, 300 μm thick Si wafers coated with mr-I 9150XP were prepared. Three different layer thicknesses were applied by spin-coating wafers at 1500, 3000 and 6000 rpm. This resulted in polymer layer thicknesses of

1.2 μm , 1.5 μm and 2.3 μm , respectively. Isocyanate silane (3-(triethoxysilyl)propyl isocyanate, Sigma-Aldrich Chemie GmbH) was evaluated as a potential adhesion promoter for the mr-I 9150XP nano-imprint resist. The adhesion promoter was applied by submerging both a lid wafer and a structured wafer with etched cavities for 10 min in a solution of isocyanate silane dissolved 3% by weight in toluene. This coats the wafers with a thin layer of isocyanate silane. The next step consisted of rinsing the wafers in toluene such that only a monolayer of isocyanate silane remained on the surface. The wafers were then baked for 10 min at 90 $^{\circ}\text{C}$ in a convection oven. The lid wafer with adhesion promoter was then spin-coated with mr-I 9150XP at 3000 rpm, which resulted in a polymer layer thickness of 1.5 μm . A three minute soft bake at 100 $^{\circ}\text{C}$ was used to remove the solvent from the polymer adhesive prior to bonding.

BCB (Cyclotene 3022-46, Dow Chemical Company) was spin-coated on the Si wafers. Lid wafers with a thickness of 125 μm and 306 μm , respectively, were used in the evaluations. In all evaluations a spin speed of 5000 rpm was used, which resulted in a BCB layer thickness of 2.4 μm . The 125 μm lid wafer and a structured wafer were spin-coated at 5000 rpm with AP3000 adhesion promoter (Dow chemical company) before the lid wafer was spin-coated with BCB. No adhesion promoter was applied before spin-coating the 306 μm thick lid wafer. A three minute soft bake at 100 $^{\circ}\text{C}$ was used to remove the solvent from the spin-coated BCB, prior to the adhesive wafer bonding.

2.3.3. Wafer bonding process. An SUSS Microtec SB8 wafer bonder was used to bond the wafers. A structured wafer (section 2.3.1), a lid wafer (section 2.3.2) and a 500 μm thick graphite sheet were manually pressed together and fastened in the wafer bonding fixture of the bond tool, where the graphite sheet is used to compensate for wafer and chuck surface imperfections. No spacers are used in the bonding process. The wafer bonding fixture is inserted into the bonder. The bonding processes, irrespective of the adhesive polymer used, start with pumping a vacuum (less than 10^{-4} mbar) in the bond chamber. This is followed by applying a bond force, pressing the wafers together, and a temperature ramp to a specified target temperature. The OSTE+ wafers were bonded with a 5000 N bond force together with a thermal curing time of 1 h at 90 $^{\circ}\text{C}$, using a 10 min temperature ramping from room temperature to 90 $^{\circ}\text{C}$. The nano-imprint resist used a bond force of 12 kN and a bonding temperature of 200 $^{\circ}\text{C}$ for 50 min. The BCB bonded wafers were bonded at a force of 5000 N and a bonding temperature of 250 $^{\circ}\text{C}$ for 1 h to cure the polymer. For both BCB and the nano-imprint resist a 30 min temperature ramp between 50 $^{\circ}\text{C}$ and the target temperature was used. The bonding procedures for the adhesives mr-I 9150XP and BCB were based on parameters prescribed in the literature for the respective polymers to achieve high-quality wafer bonds [12, 22, 31]. Figure 3 illustrates both the spin-coating of the polymers and the bonding process used in the experiments.

2.3.4. Lid and chip dicing. The bonded wafers were diced into individual dies with a wafer dicing saw. This was done in

Figure 3. (A) Spin-coating of lid wafers. (B1) BCB and mr-I 9150XP are soft baked at 100 °C. (B2) Solvent removed by keeping the wafer in vacuum. First curing of OSTE+ by exposing with UV radiation. (C) Wafer bonding of the lid wafer to a wafer with etched circular cavities.

Figure 4. Fabricated samples together with the fixture used to pressurize the die from the through hole, on the backside of the die, until the Si lid delaminated from the etched circular cavity.

two steps. First, the lids were defined by only dicing through the lid wafer and roughly 35 μm into the wafer with etched cavities. Secondly, individual dies were created by dicing through the entire wafer stack. The die surface outside the lid area was used to fixate the die in the chip holder for the pressurizing experiments. The dimensions of the finished dies were $22 \times 22 \text{ mm}^2$ and each bonded wafer resulted in a total of up to 12 dies to be evaluated. Figure 4 shows a photograph of fabricated dies used for the bond energy evaluation together with the die fixture used to pressurize the die lid through the pressure port etched in the Si wafer.

3. Results

Table 1 presents estimated bond energies for the evaluated polymer adhesives. Bond interfaces between lids and bottom

Si structures were inspected following the blister tests. Images of typical dies after the destructive testing can be seen in figure 5. Lids that showed weak bonds (i.e. mr-I 9150XP and OSTE+ blend 2) typically delaminate completely from the bottom die structure. For strongly bonded lids (i.e. BCB and OSTE+ blend 1), delamination typically reaches one of the edges first, creating a freely moving lid edge. The high pressure together with the released edge then tended to tear the Si lids apart, where dies after test had a delamination to at least one edge and a distance around the orifice, leaving a piece of the torn lid still bonded to the bottom die structure, as shown in figure 5.

No voids were visible in the polymer for BCB-bonded dies around the orifices, indicating a high-quality bond. In the case of mr-I 9150XP, micro-voids were visible in microscopy inspections around the orifice. Formation of micro-voids in the nano-imprint resist series mr-I 9000 has been reported in earlier works [22], where it typically occurs in regions between large plane surfaces. Both polymers reflow before cross-linking, evening out surface imperfections. Polymer stripes were visible for wafers bonded with OSTE+ of blend 2. The polymer shape is set for OSTE+ already after UV exposure and the thermal curing starts directly thereafter. This limits the polymer layer quality compared to wafers bonded with polymers that reflow before curing. Inspections of samples of bottom Si structures bonded with OSTE+ of blend 1 tended to be clean Si without any stripes.

4. Discussion

This paper employs blister tests for determining adhesive bond energies between Si wafers in adhesive wafer bonding applications. Using undiluted OSTE+ of blend 1 allows combining a high mean bond energy of 20 J m^{-2} with an easy adhesive removal in oxygen plasma. The bond energy is drastically reduced for samples bonded with diluted OSTE+ of blend 1. A possible reason for this is the incomplete removal of the solvent before the wafer bonding. The standard deviation in the delamination pressure for wafers bonded with diluted OSTE+ of blend 1 was substantial (of the same order as the mean bond energy) and the spread of measured bond energies was large. For example the minimum and maximum bond energy values for diluted OSTE+ bonded samples from the same wafer were 0.3 J m^{-2} and 8.5 J m^{-2} , respectively, indicating some areas that are weakly bonded and other bond areas with a relatively strong bond. Wafers bonded with OSTE+ of blend 2 showed similar bond energies for both undiluted and diluted OSTE+ with acceptable standard deviations of the measured data, although with a weaker bond energy than for blend 1. This indicates that a 2 min soft bake at 50 °C was sufficient to remove the solvent. Wafers bonded with different thicknesses of the nano-imprint resist mr-I 9150XP indicate an increased bond energy for thinner bonding layers in the evaluated thickness regime, although the standard deviation is too large for any firm conclusions. The use of isocyanate silane as adhesion promoter for wafers bonded with the nano-imprint resist mr-I 9150XP decreased the mean bond energy to 0.2 J m^{-2} . This shows that isocyanate silane with the present formulation is an improper choice to

Figure 5. (A) Fracture interface of a BCB-bonded chip (prepared with adhesion promoter). The BCB layer is left uniformly on the bottom Si die structure, with the delamination of the bond at the Si lid interface. The lid was delaminated to one of the lid edges, when the rest of the Si lid was torn apart. (B) Fracture interface of an mr-I 9150XP-bonded chip. The delamination of the top chip started between the bottom Si die structure, leaving polymer on the lid. This was followed by delamination between the lid and polymer, approximately 1 mm from the orifice. Complete delamination of lids typically occurred. (C) Fracture interface of OSTE+ blend 1. The polymer separated in the interface between the bottom Si die structure, leaving the polymer on the lid and a clean Si surface on the bottom structure. The lid delaminated around the orifice and was torn apart when one of the lid edges was reached. (D) Fracture interface of OSTE+ blend 2. Delamination happened both at the Si lid and bottom structure interfaces. Stripes of polymer are left on both the lid and bottom Si structure. The lid is delaminated to most of the lid edges, although torn apart toward one of the edges.

Table 1. Adhesive bond energies for different polymers and process parameters.

Specimen	Bond Temperature (°C)	Adhesion promoter	Lid thickness (μm)	Layer thickness (μm)	Number of samples	Mean bond energy (J m ⁻²)	Standard deviation (J m ⁻²)
BCB	250	Yes	125	2.4	10	35	7.6
BCB	250	No	306	2.4	11	5.7	0.4
mr-I 9150XP	200	Yes	304	1.5	11	0.2	0.4
mr-I 9150XP	200	No	302	1.2	10	2.4	0.5
mr-I 9150XP	200	No	300	1.5	11	1.5	0.6
mr-I 9150XP	200	No	303	2.3	8	1.7	0.8
OSTE+ (blend 1) undiluted	90	No	302	Not measured	11	20	6.8
OSTE+ (blend 1) diluted 1:1 by weight with toluene	90	No	132	3.9	11	2.2	2.0
OSTE+ (blend 2) undiluted	90	No	304	4.4	11	2.1	0.5
OSTE+ (blend 2) diluted 1:1 by weight with toluene	90	No	306	2.9	11	2.9	0.9

use as an adhesion promoter for mr-I 9150XP. The measured mean bond energies for BCB-bonded wafers are comparable to values found in the literature [12, 13]. The bond energy of the wafer bonded with the adhesion promoter is (as expected) much higher than that for the wafer bonded without, although

the bond energy even without an adhesion promoter is fairly high. Bond interfaces between bonded surfaces were inspected after the destructive blister tests. No obvious unbounded regions or voids were located in wafers bonded with BCB and mr-I 9150XP (although the nano-imprint resist had the

presence of micro-voids). For OSTE+ of blend 2, polymer layer imperfections (stripes) were visible, indicating a locally uneven polymer layer thickness that could potentially worsen adhesion. This indicates that the use of OSTE+ as a bonding layer requires a tight process control with regards to spin-coating and wafer bonding to achieve acceptable results.

5. Conclusions

Three different adhesives used in adhesive wafer bonding applications have been evaluated with regards to the adhesive bond energy. OSTE+ is suitable for applications that require low-temperature processes due to its ability to cure even at room temperature. BCB is ideal for permanent bonds due to its temperature stability and its chemically inert characteristics. Both the nano-imprint resist mr-I 9150XP and OSTE+ are removable in oxygen plasma, making them ideal as sacrificial polymer layers. OSTE+ formulations based on the epoxy D.E.N. 431 Novolac allow for strong bonds compared to both OSTE+ formulations based on the epoxy BADGE and mr-I 9150XP. All three polymers are suitable for heterogeneous silicon wafer-level integration depending on the application.

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