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Adhesion of NCF to oxidized Si wafers after oxygen plasma treatment

Min-Seok Jang [a], Sung Woo Ma [b], Jongsoo Song [c], Myungmo Sung [c], Young-Ho Kim [a,*]

[a] *Division of Materials Science & Engineering, Hanyang University, Seoul 133-791, Republic of Korea*
[b] *Division of Nanoscale Semiconductor Engineering, Hanyang University, Seoul 133-791, Republic of Korea*
[c] *Department of Chemistry, Hanyang University, Seoul 133-791, Republic of Korea*

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ABSTRACT

The effect of oxygen plasma treatment on the adhesion between nonconductive film (NCF) and oxidized Si was investigated. Oxidized Si wafers were treated with oxygen plasma for 5 min and then rinsed in de-ionized water (DIW). The water contact angle was measured by means of the sessile drop technique and the surface roughness was measured by means of atomic force microscopy. The adhesion of the NCF to the oxidized Si wafer was evaluated by means of a single-lap shear test after bonding at 150°C for 5 s. Oxygen plasma treatment decreased the water contact angle. The roughness of the oxidized Si wafer decreased when oxygen plasma treatment was applied alone, but was increased when both oxygen plasma treatment and DIW rinse were applied. Similarly, the shear strength decreased when oxygen plasma treatment was applied alone, but the adhesion of NCF increased when both oxygen plasma treatment and DIW rinse were applied. The increased surface roughness of the oxidized Si wafer played an important role in increasing the adhesion between the NCF and the oxidized Si wafer. The shear strength further increased after post-heat treatment at 170°C for 1 hr or at 280°C for 15 s. Low shear strength observed before post-heat treatment was ascribed to incomplete NCF curing. Differences observed in the adhesion strength between two types of NCF were attributed to differences in their curing degrees and their degrees of surface coverage of the oxidized Si substrates.

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1. Introduction

Flip chip bonding technology has been widely adopted owing to the higher packaging density (high I/O), fast signal response, and low inductance it provides [1–3]. Generally, the flip chip bonding process involves mass reflow for interconnection between solder bumps on a chip and matching metal pads on the substrate, followed by capillary underfilling [3]. However, flip chip bonding formed by means of mass reflow has limitations in fine-pitch packages below 100 μm [4,5]. Due to the tendency of the fine pitch and narrow gap size of the flip chip package, flux residue cleaning and narrow gap underfilling become difficult. Solder bridging failure may occur and voids may become trapped in the joints due to incomplete underfilling [6]. To overcome these issues, the used of pre-applied nonconductive paste (NCP) with thermo-compression (TC) bonding has been developed [7]. Although NCP is normally used as a pre-applied adhesive, maintaining NCP volume control during dispensing is a major challenge [8]. Given the advancements in technology, the pre-applied nonconductive film (NCF) has advantages over the pre-applied NCP for use with TC bonding, including wafer-level process compatibility [9], easy volume control (by regulating film thickness) [10], and lack of overflow into the flip-chip bonder head tool. However, TC bonding has a lower throughput than conventional mass reflow processes due to the use of separate process steps for pick-and-place and joint formation. One method used to improve throughput is to decrease the bonding temperature (below 150–180 °C), because low-temperature bonding decreases the heating and cooling times required in the bonding cycle [9,11]. The other advantages of decreasing the bonding temperature include lower residual stress and less wafer warpage due to thermal mismatch [12] as well as compatibility with heat-sensitive devices. Because NCF in TC bonding plays a similar role as underfill in mass reflow flip chip bonding [13], good adhesion of NCF is important for long-term reliability. Furthermore, the solder joints formed by pre-applied NCF followed by TC bonding at low temperature below the solder melting point are normally maintained by mechanical contact only, without metallurgical bonding. Therefore, strong adhesion between the NCF and the passivation layer on the IC chip is critically required since the joints should be stably maintained by adhesives. Si oxide as well as nitride can be used as passivation layer [14]. Insufficient adhesion of the NCF can lead to delamination which degrades long-term reliability [15]. A number of methods have been developed to improve the adhesion of adhesives. Adhesion of nonconductive adhesive (NCA) can be increased by optimizing the silica content in the NCA, because the mechanical properties of NCA depend upon optimal silica content [2]. Plasma treatment has also been used widely. Chuang et al. [16] studied the effect of argon plasma treatment

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[*] Corresponding author.
E-mail address: kimyh@hanyang.ac.kr (Y.-H. Kim).

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upon the adhesion between NCP and flexible substrate. Adhesion between NCP and flexible substrate was improved after argon plasma treatment. Noh et al. [17] also claimed that plasma treatment increased the adhesion strength between underfill and SiO_2 substrate. However, Luo et al.'s [14] work showed that oxygen plasma treatment did not improve the adhesion between underfill and oxidized Si wafer. Although many studies have been performed to investigate the effect of plasma treatment on the adhesion between various adhesives and substrates, the results remain controversial.

In the present work, we investigated the adhesion of NCF to oxidized Si wafer after bonding at 150 °C for 5 s. The shear strength between NCF and oxidized Si wafer was studied after oxygen plasma treatment and de-ionized water (DIW) rinse. We also investigated the effect of post-heat treatment after low temperature bonding, since NCF may not be completely cured during the bonding process at 150 °C for 5 s.

2. Experimental

Table 1 lists the material properties of the two NCFs used experimentally in the present work. Those NCFs are not commercial products. NCF A had higher melt viscosity and onset temperature than NCF B. Differential scanning calorimetry (DSC) analysis was carried out using a TA Instruments Q100 device to fully understand the curing behavior of the NCF during bonding. NCF samples were held isothermally at 150 °C during DSC measurements.

The bonding surfaces of thermally oxidized Si wafers (oxide thickness = 300 nm) were subjected to the following surface treatments to investigate the treatments' effects.

(i) Wet cleaning: An oxidized Si wafer was cleaned with acetone and methanol using an ultrasonic cleaner, and then dried with nitrogen gas.
(ii) Oxygen plasma treatment only: An oxidized Si wafer was subjected to wet cleaning and then exposed to oxygen plasma of 120 W input power and 8 Pa pressure for 5 min. These treatment parameters were selected as optimal based upon trial and error optimization.
(iii) Both oxygen plasma treatment and DIW rinse: An oxidized Si wafer was subjected to oxygen plasma treatment as described above and then rinsed in DIW and dried with nitrogen gas.

The surface roughnesses of samples subjected to these three different surface treatments were measured by means of atomic force microscopy (AFM). The oxidized Si wafers were diced into 1 × 1 cm samples to allow them to fit into the AFM instrument, and a 5 × 5 μm area of each sample was scanned in non-contact AFM mode with a silicon nitride tip. All oxidized Si wafers were cut before plasma treatment. The AFM observation was carried out under ambient atmosphere. Root-mean-square (RMS) surface roughnesses were calculated from the AFM roughness profiles thus acquired.

Water contact angles were measured using a contact angle meter (DSA20S) to investigate the surface energies of substrates after plasma treatment. A sample was placed on the stage of the goniometer, and a micro-syringe was used to drop water of 2–3 μm onto the sample surface. Water contact angles were measured under ambient atmosphere by means of the sessile drop technique.

A schematic of the single lap-joint used to conduct lap shear testing is shown in Fig. 1. To prepare joints for testing, two rectangular oxidized Si wafers with the different dimensions of 5 × 20 × 0.65 mm and 10 × 20 × 0.65 mm were bonded at 150 °C for 5 s under 1 MPa. First, NCF was laminated on a 5 × 20 × 0.65 mm size Si wafer. Lamination temperature was varied depending on the NCFs. Lamination temperatures were 60 °C for NCF A and 50 °C for NCF B considering the onset temperature. Then, two Si chips with the different dimensions were aligned and bonded using a flip chip bonder. The fillet of NCF may be formed after bonding. To ensure precision of the bonded area (5 × 5 mm), the fillet of NCF was cut after bonding. Samples subjected to three types of post-heat treatment were prepared: (i) no treatment (as-bonded condition), (ii) post-curing (170 °C, 1 h) and (iii) post-reflow (280 °C, 15 s). The shear strength of the bond between the NCF and the oxidized Si wafer was evaluated using the single-lap shear test, with the crosshead speed of 50 mm/min. We tested more than 10 samples at the same condition. After the single-lap shear test, the failure mode was analyzed by means of scanning electron microscopy (SEM; JEOL JSM-6300).

Table 1
Specification of the NCFs.

Description	NCF A	NCF B
Thickness (μm)	20 ± 2	20 ± 1
Melt viscosity (Pa.s)	6300 ± 400	1200 ± 400
Temperature of melt viscosity (°C)	149 ± 2.5	125 ± 2.5
Onset temperature (°C)	156 ± 5	106 ± 5
Curing speed (°C/min)	23	75

3. Results

3.1. Curing behavior of NCF

DSC analysis is the most widely used method to determine the extent of epoxy curing; accordingly, DSC analysis of both NCF types was conducted. Isothermal DSC curves suggested that bonding at 150 °C for 5 s resulted in incomplete curing of both NCF types (Fig. 2). The curing speed of NCF B was faster than that of NCF A at the same bonding temperature. The onset temperature comparison of NCF A and B given in Table 1 also suggests that NCF B cured faster. The above DSC results indicated that post-heat treatments were needed for both NCFs to ensure complete curing. Since NCF A and B were fully cured at 150 °C in 30 min from the DSC measurement, the heat treatment at 170 °C for 1 h was chosen as the post-curing condition to ensure the complete curing of NCF, as explained in the experimental procedure.

3.2. Surface morphologies of oxidized Si

AFM measurements of the oxidized Si surfaces subjected to various surface treatments were conducted and RMS roughness values were obtained from these measurements (Fig. 3). The RMS roughness of the wet cleaned oxidized Si surface was about 0.248 nm, similar to RMS roughnesses observed in other studies [18,19]. Application of only oxygen plasma treatment smoothened the oxidized Si surface, decreasing the RMS roughness to 0.162 nm. It has been previously reported that oxygen plasma treatment had a smoothing effect upon the surface of oxidized Si [19]. This smoothing effect was attributed to the removal of surface hydrocarbons. Application of both oxygen plasma treatment and DIW rinse induced a rougher surface morphology with the RMS roughness of about 0.331 nm. The RMS roughness after both oxygen plasma treatment and DIW rinse was two times that of after oxygen plasma treatment only. Amirfeiz et al. [20] also reported increased surface roughness of oxidized Si wafer after oxygen plasma treatment and DIW rinse. After both oxygen plasma treatment and DIW rinse, relaxation of the very reactive surface from its excited state to equilibrium may yield a rough surface [21].

3.3. Effect of oxygen plasma treatment upon wettability

Water contact angles were measured on the oxidized Si substrates subjected to oxygen plasma treatment (Fig. 4). This treatment reduced the water contact angle from 61° to 15°, meaning that the oxidized Si surface became more hydrophilic. The application of both oxygen plasma treatment and DIW rinse further reduced the water contact angle on

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