Localized Parylene-C bonding with reactive multilayer foils

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Abstract

This paper describes a novel bonding technique using reactive multilayer Ni/Al foils as local heat sources to bond Parylene-C layers to another Parylene-C coating on a silicon wafer. Exothermic reactions in Ni/Al reactive multilayer foils were investigated by x-ray diffraction (XRD) and differential scanning calorimetry. XRD measurements showed that the dominant product after exothermic reaction was ordered B2 AlNi compound. The heat of reaction was calculated to be $-57.9 \, \text{kJ} \, \text{mol}^{-1}$. A numerical model was developed to predict the temperature evolution in the parylene layers and silicon wafers during the bonding process. The simulation results revealed that localized heating occurred during the reactive foil joining process. Our experimental observation showed that the parylene layer was torn when the bond was forcefully broken, indicating a strong bond was achieved. Moreover, leakage test in isopropanol alcohol showed that reactive foil bonds can withstand liquid exposure. This study demonstrated the feasibility of reactive foil joining for broad applications in bio-microelectromechanical systems and microfluidic systems.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Various bonding techniques have been reported for wafer bonding applications including direct bonding and intermediate layer bonding, such as glass-frit bonding, thermocompression bonding, eutectic bonding, solder bonding and adhesive bonding [1–5]. For most of the bonding methods mentioned above except for some types of adhesive bonding, elevated temperatures are required to achieve a successful bond. Temperature sensitive components on the wafers may be damaged or destroyed by the high temperature during the bonding process. Dissimilar materials may debond on cooling due to the mismatch of the coefficients of thermal expansion. Room temperature bonding has been achieved using cold welding [1] and plasma activated surface direct bonding [6]; however, either an ultra clean and smooth surface or a plasma treatment process is required to form a strong bond, while for adhesive bonding, it can be conducted at a relatively low temperature and there is no limitation for the surface of the substrate materials. Many spin-coated polymers such as benzocyclobutene

(BCB) [7], polymethylmethacrylat (PMMA) [8], MYLAR [9] and photoresist [10] have been investigated as adhesive layers for wafer bonding. These conventional polymers, however, have difficulties in spreading uniformly on a wafer surface with tall structures or deep trenches.

Parylene is the generic name for members of a unique polymer series. The most popular one is Parylene-C (glass transition temperature: 382 K), which can be used as a good alternative to the conventional adhesive materials. It can be directly vapour-deposited from a solid powder to a solid film at room temperature providing conformal coating regardless of surface morphology. Parylene-C is also chemically inert and biocompatible. It has been extensively employed in microelectromechanical systems (MEMS) and, recently, began to draw attention as a bonding agent. Kim and Najafi [11] used parylene as an intermediate layer to bond silicon wafers at 503 K for 30 min and achieved a bonding strength of 3.6 MPa. However, this global heating method may damage temperature sensitive components on the wafer. Alternatively, localized heating approaches using micro-heaters have been developed
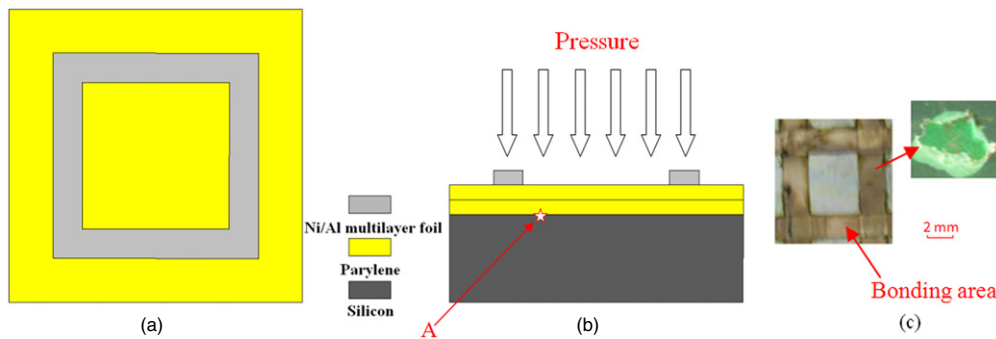
J. Phys. D: Appl. Phys. **42** (2009) 185411

X Qiu *et al*

Figure 1. Schematic showing the reactive bonding geometry of Parylene-C layers with another Parylene-C coating on a silicon wafer under an applied pressure: (*a*) top view; (*b*) cross-sectional view (not drawn to scale); (*c*) a photograph of the bonding assembly. The inset shows the parylene layer was torn when the bond was forcefully broken, indicating a strong bond was achieved.

to solve this problem [9]. However, the use of micro-heaters can introduce complexity to the bonding design and in many cases electrical wiring is not preferred. Meanwhile, parylene bonding has also been accomplished by microwave heating [12]; however, this technique requires complicated equipment and bonding facilities.

In this study, we have described a novel bonding technique using reactive multilayer Ni/Al foils as local heat sources to bond Parylene-C layers to another Parylene-C coating on a silicon wafer. Reactive multilayer foils contain thousands of nanoscale Ni and Al bilayers. With a small thermal pulse, they can react exothermically and generate a self-propagating reaction. Self-propagating reactions in these foils are driven by a reduction in chemical bond energy. This local reduction in chemical bond energy produces a large quantity of heat that is conducted down the foil and facilitates more atomic mixing and compound formation. Such exothermic reactions in multilayer foils can be used as local heat sources to melt solders or brazes and thus bond components in a variety of applications, such as bonding stainless steel, aluminium, titanium, metallic glass and silicon wafers [13–19]. With localized heating, temperature sensitive components such as microelectronic devices can be joined without thermal damage. Such bonding can be performed in many environments, such as in vacuum, and can be completed in a second or less. The properties of the reactive multilayer Ni/Al foils were characterized using x-ray diffraction (XRD) and differential scanning calorimetry (DSC). A leakage test was performed on the bonded wafers in isopropanol alcohol (IPA). Temperature evolutions in the parylene layers and silicon wafers during the bonding process were simulated by a numerical method.

2. Experimental procedure

Ni/Al reactive multilayer foils (Ni/Al atom ratio is 1 : 1) (Reactive NanoTechnologies Inc, Hunt Valley, MD) were fabricated by magnetron sputtering. The total thickness of the Ni/Al foils used here was 40 μm and 80 μm, respectively, with a bilayer thickness of 40 nm (Ni: 16 nm; Al: 24 nm). These foils were used as local heat sources to bond Parylene-C layers to another Parylene-C coating on a silicon wafer. Two different thicknesses of foils were used to bond Parylene-C to investigate the impact of difference in heat generation on the bond

quality. The crystallographic phases of the as-deposited Ni/Al multilayer foils and the reaction products were investigated by a Rigaku MiniFlex x-ray diffractometer (XRD) using Cu Kα radiation. The heat of reaction was measured by a Perkin Elmer differential scanning calorimeter (DSC7). In each DSC run, about 10 mg freestanding foils were heated from 323 K to 998 K at a rate of 40 K min^{-1} in flowing N$_2$. A base line was obtained by repeating the heating cycle, which was then subtracted from the heat flow in the first run. By integrating the net heat flow with respect to time, the heat of reaction was obtained.

The geometry for Parylene-C bonding is shown schematically in figure 1. A Parylene-C layer (20 μm thick) was bonded to a silicon wafer (20 mm by 20 mm) with a thin Parylene-C coating (20 μm). Ni/Al multilayer foils were placed on top of the parylene layer as localized heaters. The width of the foil was 2 mm. An Al substrate (1 cm thick) was used to apply pressure (about 1.1 kPa) to the bonding assembly. The reactive foil was ignited by a power supply with a voltage of 2 V. After ignition, a self-propagating reaction occurred in the foil and the heat released penetrated the top parylene layer and formed a bond at the parylene/parylene interface.

A numerical study was performed to predict the temperature evolution in the parylene layers and silicon wafers during the bonding process using the commercial finite element analysis software Fluent [20]. The numerical model was based on a simplified description of the self-propagating reaction and the thermal transport occurring in the bonding assembly. The model assumed one-dimensional motion of the reaction front, which was described using experimentally determined heats of reaction and reaction velocities of the foils [13]. For the foils with a bilayer thickness of 40 nm, the reaction velocity was 6 m s^{-1} [21]. The simulation focused on heat flow into the parylene layers, and temperature evolution within the bonded components. More details of the numerical model can be found in a previous paper [19]. The simulation started when the reactive foil was ignited. The physical properties of different materials used in the simulation are listed in table 1.

A leakage test was performed by immersing the bonded wafers in IPA. IPA has better wettability than water and it can more easily penetrate small openings, thus it is more suitable for the leakage test. Red ink was pre-mixed in IPA as a marker for leakage. After testing, the bonded

J. Phys. D: Appl. Phys. **42** (2009) 185411

X Qiu *et al*

Table 1. Thermophysical parameters for the reactive foil, Parylene-C, Al and silicon used in the simulation.

	Thermal conductivity (W mK^{-1})	Heat capacity (J kg^{-1} K^{-1})	Density (kg m^{-3})
Silicon	149	707	2330
Parylene-C	0.08	1000	1289
Al	202.4	871	2719
Foil (as-deposited)	160	830	5500
Foil (reacted)	25	610	5860

wafers were investigated under an optical microscope to see whether red ink had penetrated into the enclosed area. The parylene/parylene bonding interface was examined by scanning electron microscopy (SEM).

3. Results and discussion

XRD traces of the as-deposited Ni/Al multilayer foils and the reaction products are shown in figure 2(*a*). Before the reaction, all major peaks corresponded to Al and Ni, while after the reaction, all major peaks corresponded to the ordered B2 AlNi compound. Thus, the B2 AlNi compound was expected to be the dominant product of the foil during the bonding process. In the DSC experiment of the Ni/Al reactive multilayer foils, three exothermic peaks can be identified in the constant-heating-rate curve, as shown in figure 2(*b*). By integrating the heat flow with respect to time, the heat of reaction was calculated to be −57.9 kJ mol^{-1}. This result was used as input for the numerical study of the temperature distribution in the parylene layers and silicon wafers during the bonding process.

Figure 3 shows the numerical prediction of temperature evolution at the parylene/parylene bonding interface within 100 ms after ignition for the 40 and 80 μm multilayer foils. The highest temperatures experienced at the bonding interface for these foils were 419 K and 545 K, respectively. Both of them were below the melting temperature of Parylene-C, which is around 563 K. From previous research, in order to achieve a uniform bond between two parylene layers, a minimum temperature of 503 K was applied [11], which indicated that in the current bonding approach, 80 μm multilayer foils can be used to bond Parylene-C. The temperature of the bonding interface returned to around room temperature in 100 ms, demonstrating a high cooling rate can also be achieved in this bonding approach.

Leakage testing was performed by immersing the bonded package in IPA. For the packages formed by the 40 μm foil, they failed immediately after immersion, which showed that the 40 μm foils cannot provide enough heat to form a hermetic bond between the parylene layers. In contrast, a good resistance to liquid was shown for the package formed with the 80 μm foil by surviving the IPA leakage test over one week. The quality of the parylene/parylene bonding interface was examined by SEM. Figure 4(*a*) shows the cross-sectional view of the bonding interface formed by the 80 μm foil. It was illustrated that the two parylene layers merged together as one layer without a distinguishable interface. The inset picture
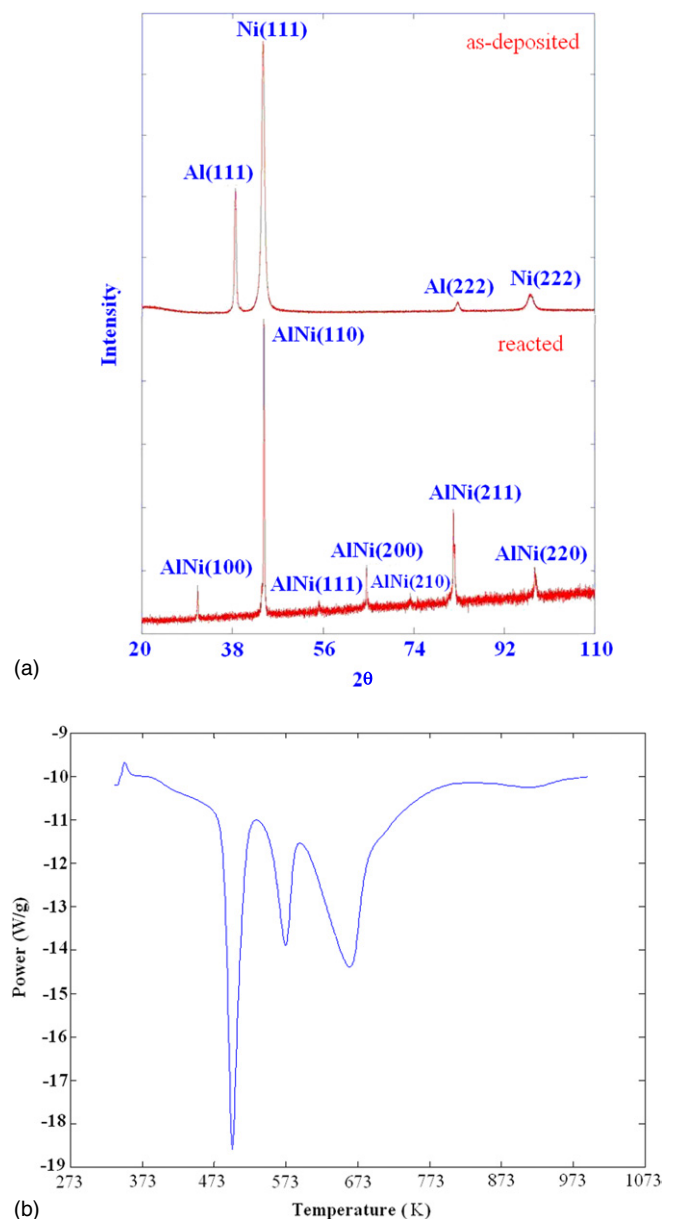

(a)

(b)

Figure 2. (*a*) XRD patterns for Ni/Al reactive multilayer foils (both as-deposited and reacted); (*b*) DSC curve of Ni/Al reactive multilayer foil. The heat of reaction was −57.9 kJ mol^{-1}.

shows the bonding region with a higher magnification. Still, no interface in the parylene layer can be identified, indicating a good bond was achieved. The thickness of the new parylene layer was around 23 μm, which was much less than the total thickness of the two parylene layers before bonding (40 μm). This was due to the softening of parylene and the pressure applied during the bonding process. After reaction, the width of the bonded ring was larger than the width of the foil used, indicating some parylene migrated out of the bonding area during the bonding process. The cross section of the bonding interface formed by the 40 μm foil is shown in figure 4(*b*). It was clear that there was an interface between the two parylene layers. At some locations, there were even cracks between these two layers (inset of figure 4(*b*)), which indicated that the 40 μm foil cannot provide enough heat to soften the parylene
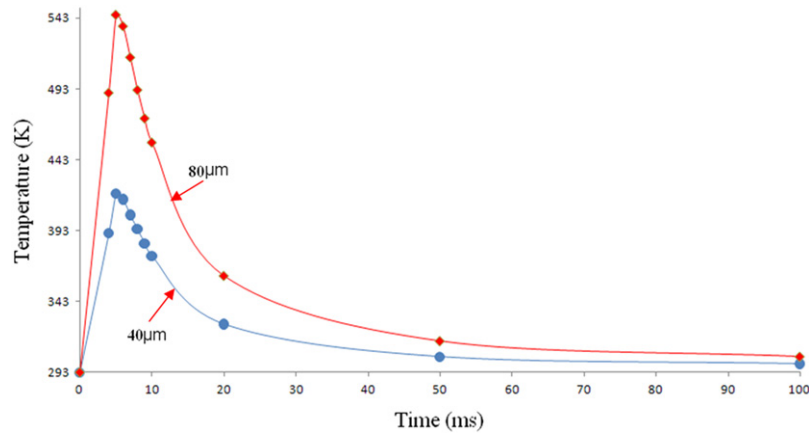
Figure 3. Temperature evolutions at the parylene/parylene bonding interface within 100 ms after ignition for 40 μm and 80 μm multilayer foils. The highest temperatures experienced at the bonding interface for these foils were 419 K and 545 K, respectively.
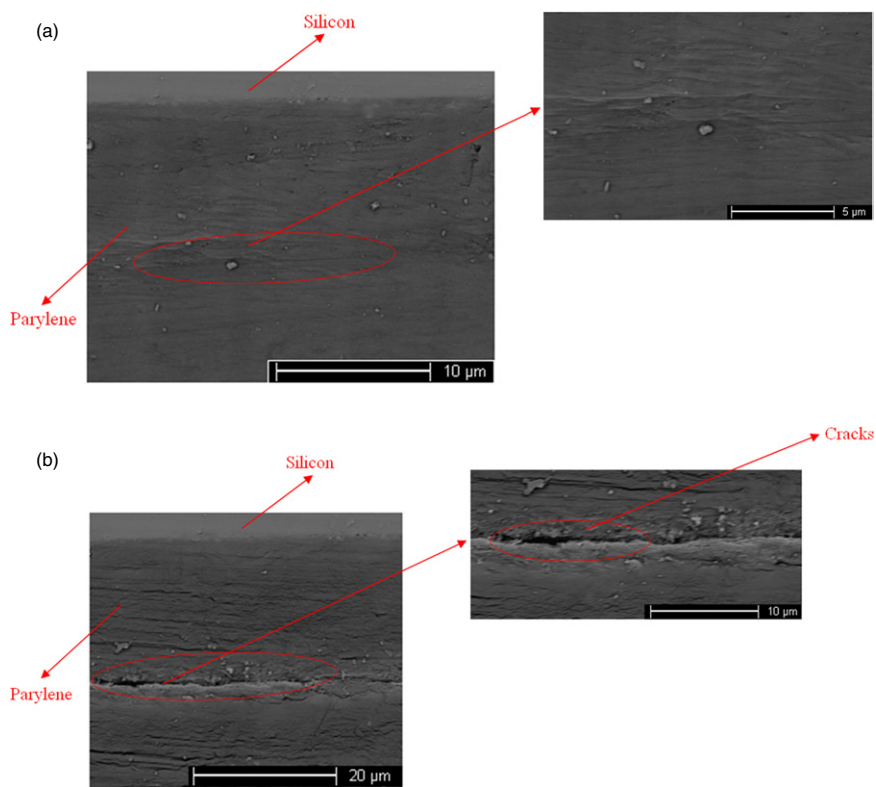

Figure 4. SEM pictures of the parylene/parylene bonding interface. (*a*) the cross-sectional view of the bonding interface formed by 80 μm foil. It illustrates that the two parylene layers merged together as one layer without a distinguishable interface. The inset shows the bonding region with a higher magnification. Still, no interface in the parylene layer can be identified; (*b*) the cross section of the bonding interface formed by 40 μm foil. It is clear that there was an interface between the two parylene layers. At some locations, there were even cracks between these two layers, indicating that 40 μm foil cannot provide enough heat to soften the parylene layers and form a uniform bond between them. The particles in the pictures were due to the gold coating.

layers and form a uniform bond between them. The SEM results were in agreement with the IPA leakage test. The thickness of the parylene layers in figure 4(*b*) was around 34 μm, indicating only a small part of parylene was softened in the bonding process due to less heat generation of the 40 μm foil compared with the 80 μm one. Therefore, the 80 μm foil can be used in the present bonding application and the following experiments and simulations were only conducted for 80 μm foils.

When the bond successfully formed by 80 μm foil was forcefully broken, the Parylene-C layer was torn, indicating a strong bond was achieved (inset in figure 1(*c*)). Figure 5(*a*) shows the simulation results of temperature evolution at the silicon wafer surface within 100 ms after ignition. The highest temperature experienced at the silicon surface was 333 K. Figures 5(*b*) and (*c*) show the contour graphs of the temperature distribution on the silicon surface at 9 ms (when the silicon wafer experienced the highest temperature of 333 K) and
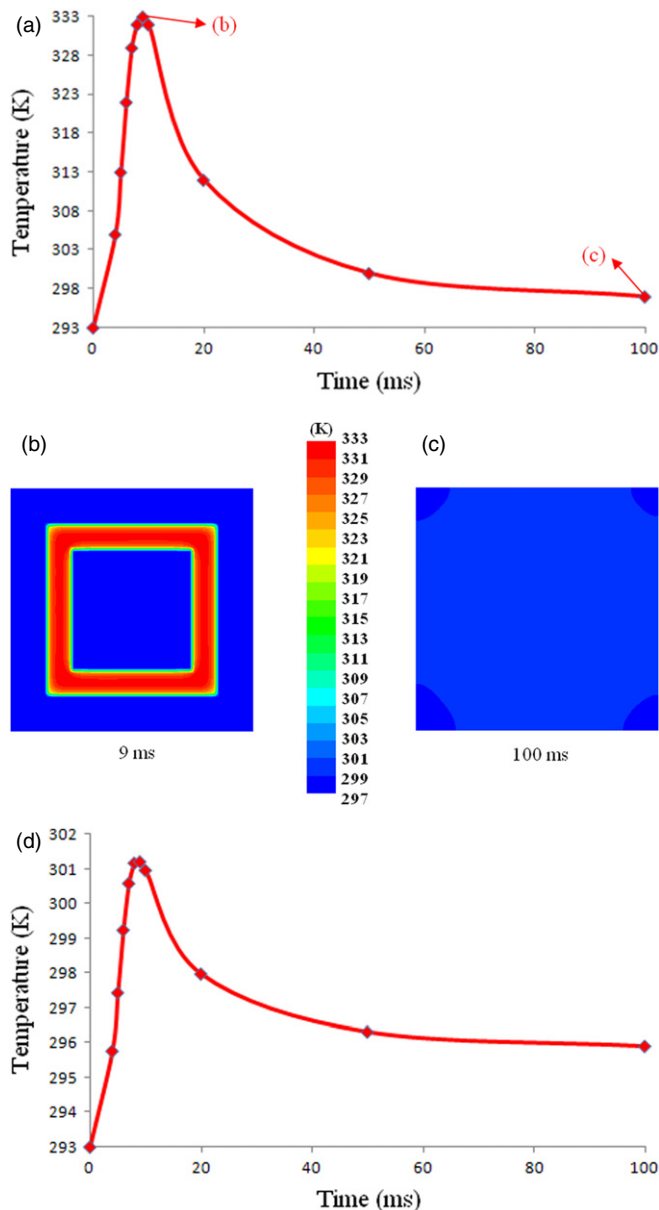
Figure 5. (*a*) the temperature evolution at the silicon wafer surface within 100 ms after ignition. The highest temperature experienced was 333 K; (*b*) and (*c*) the contour graphs of the temperature distribution on the silicon surface at 9 ms and 100 ms after ignition (the positions of (*b*) and (*c*) on curve (*a*) are pointed out by the arrows). The results illustrated that the high temperature region was well confined within the bonding area; (*d*) the numerical prediction of temperature evolution within 100 ms after ignition at a location 100 μm away from the bonding region on the silicon surface inside the package. The temperature profile shows that the highest temperature at this location was 301.2 K and it decreased to 295.9 K within 100 ms.

100 ms after ignition. The results illustrated that the high temperature region was well confined within the bonding area. Figure 5(*d*) shows the numerical prediction of temperature evolution within 100 ms after ignition at a location 100 μm away from the bonding region on the silicon surface inside the package (point A in figure 1). The temperature profile demonstrated that the highest temperature at location A was 301.2 K and it decreased to 295.9 K within 100 ms. These

results indicated that the heating area during the reactive bonding process was highly localized and the thermal exposure to the packaged components was very limited. This is a major advantage of the reactive foil bonding process, especially for bonding structures with biological materials on them, since in general, such materials can only survive temperatures up to 318 K for about 2 min [22]. These numerical results were in agreement with previous research on reactive bonding of stainless steel specimens showing that localized heating can be achieved by both numerical prediction and experimental observation [13, 16]. This study demonstrated the feasibility of reactive foil joining for broad applications in bio-MEMS and microfluidic systems.

4. Summary

The Ni/Al reactive multilayer foils were successfully used as local heat sources to bond Parylene-C layers to another Parylene-C coating on a silicon wafer. The parylene layer was torn when the bond was forcefully broken, indicating that a strong bond was achieved. Moreover, leakage testing in IPA showed that the bonds can withstand liquid exposure. Numerical simulation results demonstrated that localized heating occurred during the bonding process, which made reactive foil joining an ideal method for parylene bonding applications.

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