

Bonding silicon wafers with reactive multilayer foils

X. Qiu^a, J. Wang^{a,b,*}

^a Department of Mechanical Engineering, Louisiana State University, Baton Rouge, LA 70803, United States

^b Department of Physics and Tsinghua-Foxconn Nanotechnology Research Center, Tsinghua University, Beijing 100084, China

Received 7 June 2007; received in revised form 5 September 2007; accepted 5 October 2007

Available online 23 October 2007

Abstract

In this study, silicon wafers were bonded using Ni/Al reactive multilayer foils as local heat sources for melting solder layers. Exothermic reactions in Ni/Al reactive multilayer foils were investigated by X-ray diffraction (XRD) and differential scanning calorimetry (DSC). 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 kJ/mol. A numerical model was developed to predict the temperature evolution in silicon wafers during the bonding process. The simulation results showed both localized heating and rapid cooling during the reactive foil joining process. Our experimental observation showed that the bond strength of the silicon wafer joints was estimated to be larger than the failure strength of bulk silicon. Moreover, leakage test in isopropanol alcohol (IPA) showed that reactive foil bonds possessed good hermeticity.

© 2007 Elsevier B.V. All rights reserved.

Keywords: Nickel; Aluminum; Reactive multilayer foil; Wafer bonding; Localized heating; Rapid cooling

1. Introduction

Various bonding methods have been reported for silicon wafer bonding applications, such as anodic bonding, direct bonding, and intermediate layer bonding including glass-frit bonding, thermocompression bonding, eutectic bonding, solder bonding, and adhesive bonding [1–5]. For all of the above bonding methods except for the room temperature adhesive bonding which possesses low bonding strength and nonhermeticity, elevated temperatures are required for bonding silicon wafers. Temperature sensitive components on the wafers may be damaged or destroyed by the global heating during the bonding process. Dissimilar materials may debond on cooling due to the mismatch of coefficient of thermal expansion (CTE). New approaches with localized heating need to be developed to solve the bonding problem, so that high temperature can be locally generated to achieve hermetic and strong bond while keeping low temperature outside the bonding area.

Localized heating can be produced by embedded microheaters instead of global heating furnaces to prevent thermal problems during bonding [6–8]. These microheaters are con-

structed in a way that heating is restricted in a small region that is surrounded by insulation materials, such as silicon dioxide. Thus the bonding process can be conducted locally while the whole wafer is maintained at low temperature. The use of resistive heating from microheaters can introduce complexity to the bonding design and in many cases electrical wiring is not preferred. Nanosecond-pulsed laser welding technique has been reported to overcome this problem [9]. It possesses the advantages of no electrical wiring, fast operation, and localized heating. Another novel wafer bonding method is localized induction heating solder bonding [10], where electroplated magnetic Ni/Co film was heated using induction heating to cause the PbSn solder to flow and form a bond. However, this method is not suitable to bond devices that are sensitive to magnetic field.

In this study, we describe a novel room temperature bonding technique which uses reactive multilayer Ni/Al foils as local heat sources to melt solder layers and thus bond silicon wafers. No external heat sources such as furnace, microheater, or laser are required in this bonding approach. Devices that are sensitive to magnetic field can also be bonded using this method. Reactive multilayer foils contain hundreds of nanoscale Ni and Al bilayers. With a small thermal pulse, these foils can react exothermically and generate a self-propagating reaction. Self-propagating formation reactions in such foils are driven by a reduction in chemical bond energy. This local reduction of

* Corresponding author. Tel.: +86 10 62796007.

E-mail address: jpwang@tsinghua.edu.cn (J. Wang).

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, aluminum, titanium, and metallic glass [11–16]. 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 Ni/Al foils were characterized using X-ray diffraction (XRD) and differential scanning calorimetry (DSC). The bond strength was examined by pull test. Leakage test was also performed on the bonded wafers. Temperature evolutions in silicon wafer specimens during the bonding process were simulated by a numerical method.

2. Experimental

Ni/Al reactive multilayer foils (Reactive NanoTechnologies Inc., Hunt Valley, MD) were fabricated by magnetron sputtering. The total thickness of the Ni/Al foils is 60 μm and the bilayer thickness is 40 nm. These free-standing foils were used as local heat sources for melting solder layers and joining silicon wafers in this study. The composition of the as-deposited Ni/Al multilayer foils and the reaction products were investigated by a Rigaku MiniFlex X-ray diffractometer 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 free-standing foil was heated from 50 °C to 725 °C at a rate of 40 °C/min in flowing N₂. 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.

Two sheets of AuSn solder (80 wt% Au–20 wt% Sn, Williams Advanced Materials) with thickness of 25 μm and one free-standing reactive Ni/Al foil were stacked between two silicon wafers, as shown schematically in Fig. 1. The silicon wafers were 2 in. in diameter and 300 μm in thickness and were coated with Cr (50 nm) and Au (500 nm) by electron-beam evaporation to enhance wetting between AuSn solder and silicon wafer.

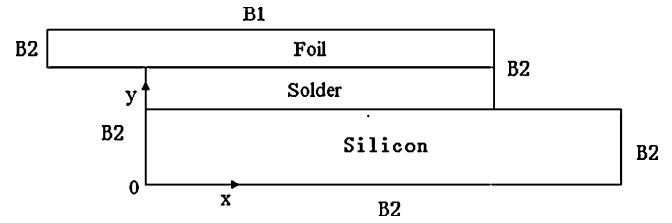


Fig. 2. Boundary conditions used in the simulation.

These silicon wafers were cut by a diamond blade into small pieces with dimension of 25.4 mm × 12.7 mm × 0.3 mm to be used in the bonding experiments. The joint area was approximately 5 mm × 6 mm. Silicon wafer bonding was performed at room temperature in air by igniting the reactive foil with an electrical spark under an applied pressure. One end of the reactive foil was extended out of the bonding package (Fig. 1), which was used as the ignition point. The electrical spark was generated by a dc power supply with a voltage as low as 2 V, and was placed outside the bonding package, so that devices in the bonding area will not be affected. Heat released from the reaction melted the solder layers and thus bonded the silicon wafers together.

A numerical study was performed to predict the temperature evolution in silicon wafers during the bonding process using commercial software, Fluent. The numerical model is based on a simplified description of the self-propagating reaction and the thermal transport and phase evolution occurring in the AuSn solder layers. The model assumes one-dimensional motion of the reaction front, which is described using experimentally determined heats of reaction and velocities of the foils [11]. The computation focuses on simulating heat flow into the solder layers, phase changes in these layers, and temperature evolution within the bonded components. The temperature evolution can be obtained by solving the two-dimensional heat conduction equation:

$$k \frac{\partial^2 T}{\partial x^2} + k \frac{\partial^2 T}{\partial y^2} = \rho C_p \frac{\partial T}{\partial t} \tag{1}$$

where k is thermal conductivity, ρ is density, and C_p is specific heat for different materials used in bonding (values listed in Table 1). Since the bonding geometry is symmetric (Fig. 1),

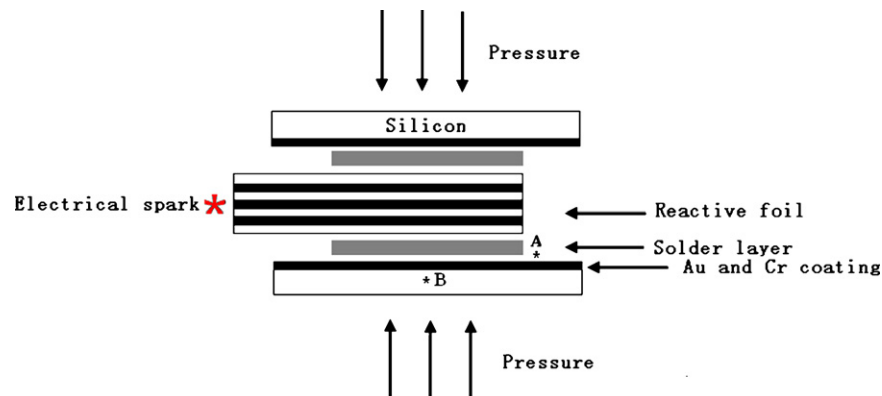


Fig. 1. Schematic showing the reactive bonding of two silicon wafers using a reactive foil and two solder layers under an applied pressure.

Table 1
Thermophysical parameters for the reactive foil, solder, and silicon used in the simulation

	Silicon	Solder	Foil (reacted)	Foil (unreacted)
Thermal conductivity (W/m K)	149	57	25	160
Heat capacity (J/kg K)	707	170	610	830
Density (g/cm ³)	2.33	14.51	5.86	5.55

only one half of this geometry is used to conduct the simulation. The coordinate system used in this model is shown in Fig. 2. Dimension x ranges from 0 mm to 25.4 mm and dimension y ranges from 0 mm to 0.355 mm. Boundary conditions (B1 and B2) used in the model are also shown in Fig. 2. The boundary condition B1 can be described as:

$$k_f \frac{\partial T}{\partial x} = q \quad (2)$$

where k_f is the thermal conductivity of the unreacted reactive foil (value listed in Table 1), q is the heat flux during the reaction, which can be calculated using the formation enthalpy of AlNi (59 kJ/mol) [18] and the reaction velocity of the foil (6 m/s for foils with bilayer thickness of 40 nm) [19]. Boundary conditions (B2) are convection with a convection coefficient of 35 W/m² K [17] and ambient air temperature of 293 K. The simulation started when the reactive foil was ignited. The thermal transport across the unbonded interfaces is considered using a thermal interface model, which accounts for thermal resistance at the interface between unbonded layers and assumes that the thermal resistance decreases exponentially when wetting occurs at the interface. The thermal conductance, which scales as the inverse of the contact resistance, is used to represent the effect of imperfections in contact between layers. As long as the temperature at the interface is below the melting temperature of the solder, the model assumes that the thermal conductance increases exponentially with temperature and finally becomes infinite at the melting temperature of the solder, corresponding to perfect thermal contact between the layers.

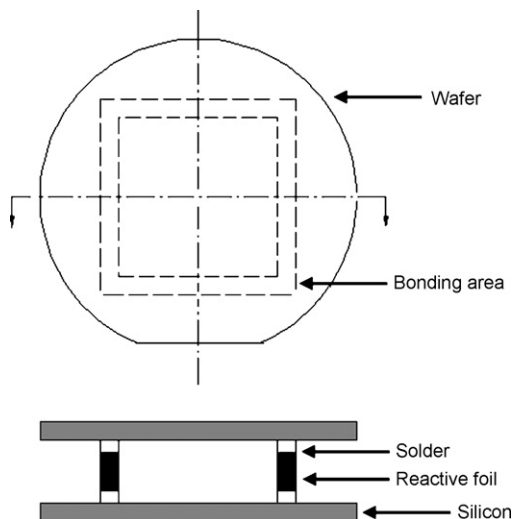


Fig. 3. Geometry of silicon wafers bonding for leakage test.

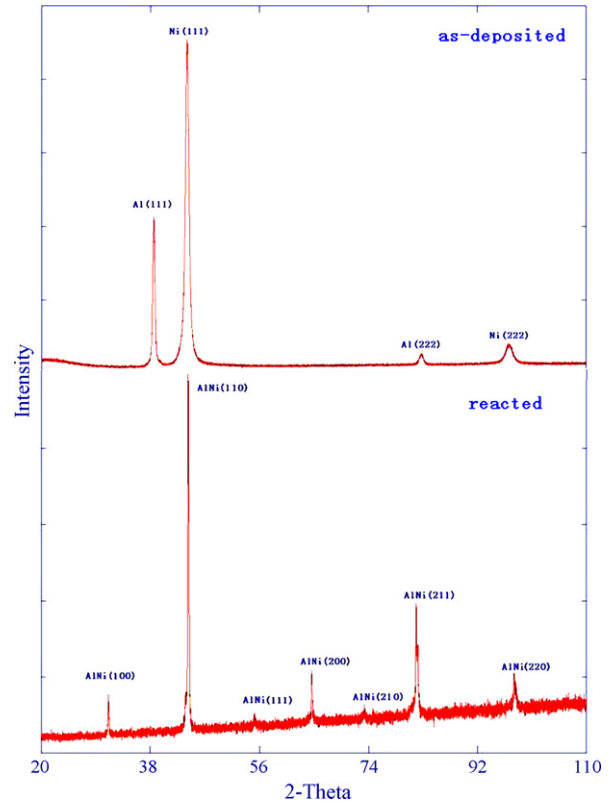


Fig. 4. XRD patterns for Ni/Al reactive multilayer foils (both as-deposited and reacted).

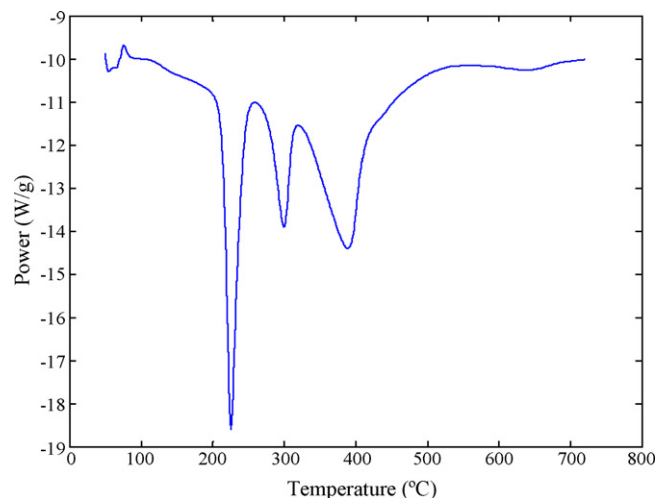


Fig. 5. DSC curve of a Ni/Al reactive multilayer foil.

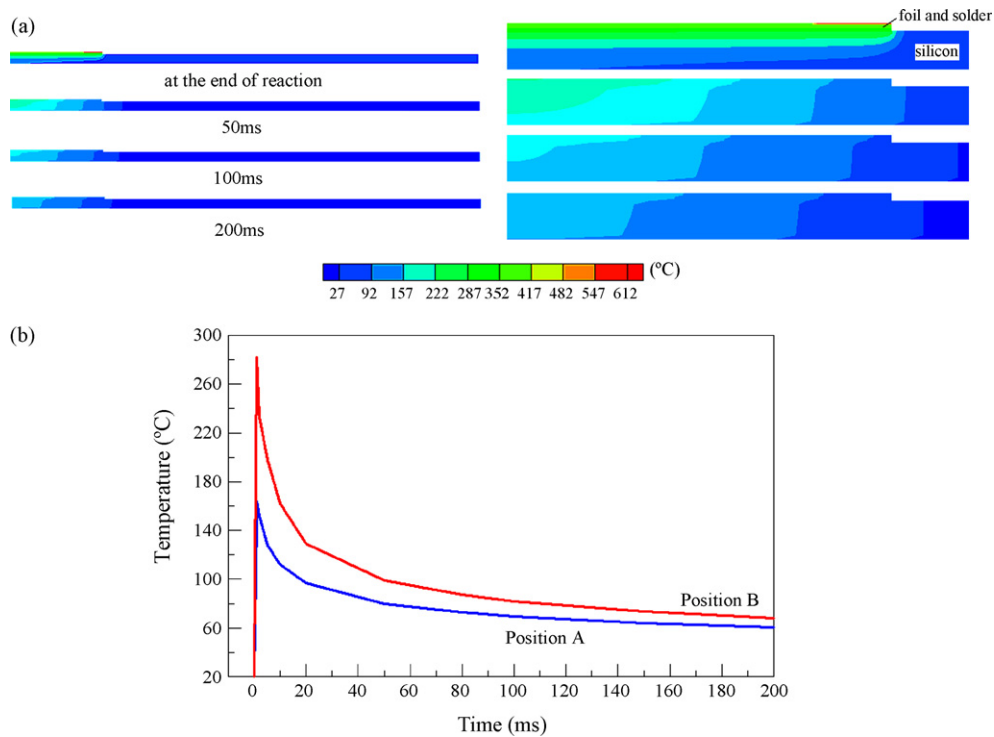


Fig. 6. (a) Temperature distribution in the foil, solder, and silicon wafer at the end of reaction, 50 ms, 100 ms, and 200 ms after reaction; (b) temperature evolutions at positions A and B within 200 ms after ignition.

After reactive bonding, the silicon wafer joints were examined by pull test using an Instron testing machine. During the test, the sample was fixed between two steel bars by epoxy and then loaded uniaxially. In addition, leakage test was performed by immersing the bonded wafers in isopropanol alcohol (IPA), which was pre-mixed with red ink. IPA has better wettability than water and it can more easily penetrate small openings. Thus it is more suitable for leakage test. The bonding geometry of the wafer package used in leakage test is shown in Fig. 3. Four sets of free-standing solder strips and Ni/Al strips with width of

6.35 mm were stacked together to form a square shape between two silicon wafers. The area enclosed by the reactive bond is 12.7 mm × 12.7 mm in dimension.

3. Results and discussion

Ni/Al multilayer foils with total thickness of 60 μm and bilayer thickness of 40 nm were used to bond silicon wafers in this study. Previous studies showed that foils with certain thickness and thus releasing enough heat were required to melt the

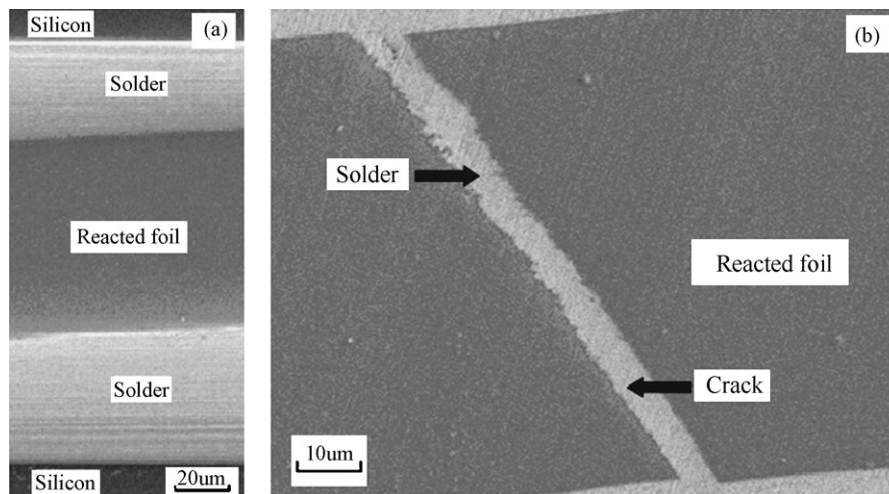


Fig. 7. (a) SEM micrograph of two silicon wafers that were bonded using two pieces of free-standing AuSn solder and one Ni/Al reactive foil; (b) molten solder filled in a crack in the reacted foil formed during the joining process.

entire solder layer and sustain solder melting for at least 0.5 ms in order to form strong bonds [11]. According to our simulation results, 60 μm thick Ni/Al foils are required to satisfy these criteria for bonding silicon wafers with 25 μm thick AuSn solder sheets [20]. For Ni/Al multilayer foils, the reaction velocity decreases with increasing bilayer thickness. The heat of reaction increases as bilayer thickness increases, due to intermixing occurred during deposition [11,19]. In order to achieve a balance between high reaction velocity and high reaction heat, foils with bilayer thickness of 40 nm are used in this study.

XRD traces of the as-deposited Ni/Al multilayer foils and the reaction products are shown in Fig. 4. Before the reaction, all major peaks correspond to Al and Ni. While after the reaction, all major peaks correspond to the ordered B2 AlNi compound. Thus, during the bonding process, the B2 AlNi compound is expected to be the dominant product for the foil.

In the DSC experiment of the Ni/Al reactive multilayer foil, three exothermic peaks can be identified in the constant-heating-rate curve, as shown in Fig. 5. By integrating the heat flow with respect to time, the heat of reaction was obtained. For the Ni/Al reactive multilayer foil with bilayer thickness of 40 nm, the heat of reaction is -57.9 kJ/mol. This result was used as input for the numerical study of the temperature distribution in silicon wafers during the bonding process.

Fig. 6(a) shows the numerical prediction of temperature distribution in the foil, solder, and silicon wafer at the end of reaction, 50 ms, 100 ms, and 200 ms after reaction (figures at the left side show temperatures across the entire bonding geometry and figures at the right side show temperatures at the bonding area with the most intense temperature gradients). Fig. 6(b) shows the numerical prediction of temperature evolution within 200 ms after ignition at two different positions in the joining geometry. One is 100 μm away from the joining area on the surface of the silicon wafer (point A in Fig. 1). The other is 100 μm under the solder/silicon interface in the middle of the joining area (point B in Fig. 1). These temperature profiles show that the highest temperature at positions A and B are 164 $^{\circ}\text{C}$ and 282 $^{\circ}\text{C}$, respectively; the temperatures at positions A and B decrease to 60 $^{\circ}\text{C}$ and 68 $^{\circ}\text{C}$, respectively within 200 ms after

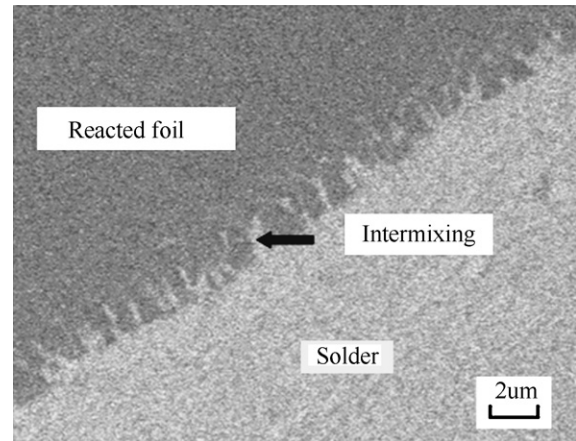


Fig. 8. Microstructure of the solder/foil interface showing chemical intermixing.

ignition. These results indicate that the heating area during the reactive joining process is highly localized and the thermal exposure to the joining components is very limited. The results also show that rapid cooling is achieved in this reactive foil joining method, with estimated cooling rates of 520 $^{\circ}\text{C}/\text{s}$ and 1070 $^{\circ}\text{C}/\text{s}$ for positions A and B, respectively. These numerical results are in agreement with our previous research on reactive bonding of stainless steel specimens showing that localized heating and rapid cooling can be achieved by both numerical prediction and experimental observation (references [11] and [14]). The localized heating and rapid cooling are the advantages of the reactive foil joining method for silicon wafer bonding applications.

Fig. 7(a) shows a cross-sectional scanning electron microscope (SEM) image of two silicon wafers that were bonded using two pieces of free-standing AuSn solder and one Ni/Al reactive foil. Cracking was observed within the reacted foils (Fig. 7(b)) and is attributed to the fact that when the foils react they contract due to densification; they also contract due to cooling from the high reaction temperatures. Both sources of contraction can be constrained by the surrounding material, thereby leading to cracking. Molten AuSn solder typically flowed into these cracks. The microstructure of the solder/foil interface is shown in Fig. 8

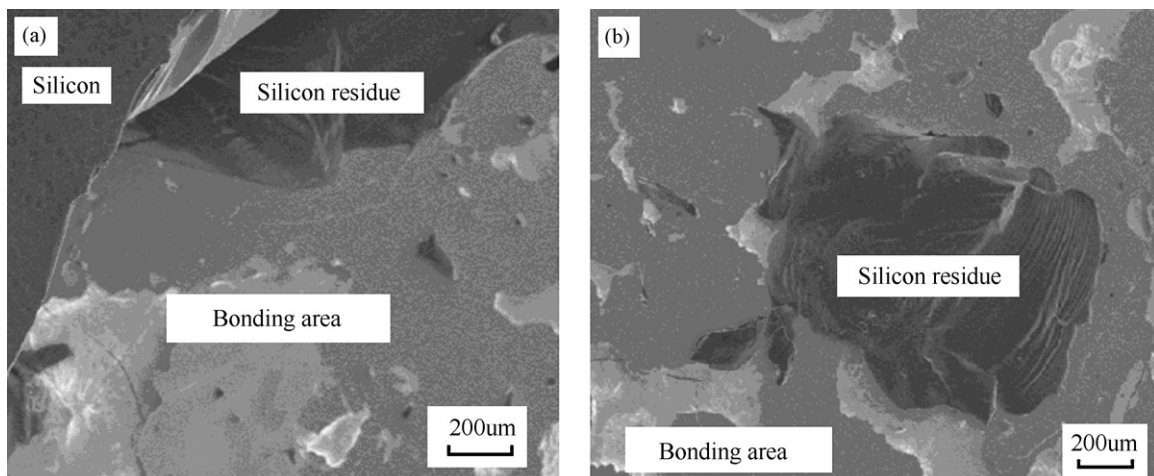


Fig. 9. Fracture surfaces of silicon-to-silicon reactive bond obtained by SEM.

at a higher magnification. It can be observed that there is intermixing between the solder and the reactive foil at the interface.

Fig. 9(a) and (b) shows the SEM images of the fracture surfaces of silicon wafer joints after pull test. Failure occurred at silicon wafer itself in many regions and silicon was stripped off from one wafer and attached to the other wafer, indicating that the bonding interface is stronger than the silicon wafer itself. The reactive bonding strength is estimated to be larger than the failure strength of bulk silicon at approximately 10 MPa [21].

A bonded wafer package was immersed in IPA (pre-mixed with red ink) to conduct the leakage test. After 12 h' immersion in IPA, the bonded package was broken into parts. No red ink can be observed in the enclosed area, suggesting no leakage occurred during the immersion. The hermeticity of the wafer package indicated that this reactive foil bonding approach can be exploited to protect structures enclosed in the package from humidity. A helium leakage test will be performed to further demonstrate the hermeticity of this bonding method.

4. Conclusions

Exothermic reactions in Ni/Al reactive multilayer foils were investigated by XRD and DSC. The dominant product after exothermic reaction was ordered B2 AlNi compound. The heat of reaction was calculated to be -57.9 kJ/mol. The Ni/Al reactive multilayer foils were successfully used as local heat sources to melt AuSn solder layers and bond silicon wafers. Silicon wafer bonds failed at silicon itself during pull test, suggesting that the bond strength is higher than the failure strength of bulk silicon. Moreover, leakage test in IPA suggested that reactive foil joints possessed good hermeticity. A numerical model was developed to predict the temperature evolution in silicon wafers during the bonding process. The simulation results showed both localized heating and rapid cooling during the reactive joining process, which made reactive foil joining an ideal method for silicon wafer bonding applications.

Acknowledgement

This work was supported by the National Science Foundation through Award DMI-0556100.

References

- [1] M.A. Schmidt, Wafer-to-wafer bonding for microstructure formation, *Proc. IEEE* 86 (1998) 1575–1585.

- [2] K.M. Knowles, A.T.J. Van Helvoort, Anodic bonding, *Int. Mater. Rev.* 51 (2006) 273–311.
- [3] C.H. Tsau, S.M. Spearing, M.A. Martin, Characterization of wafer-level thermocompression bonds, *J. Microelectromech. Syst.* 13 (2004) 963–971.
- [4] F. Niklaus, G. Stemme, J.Q. Lu, R.J. Gutmann, Adhesive wafer bonding, *J. Appl. Phys.* 99 (2006) 1101–1128.
- [5] M. Chiao, L. Lin, Device-level hermetic packaging of microresonators by RTP aluminum-to-nitride bonding, *J. Microelectromech. Syst.* 15 (2006) 515–522.
- [6] Y.T. Cheng, W.T. Hsu, K. Najafi, T.C. Nguyen, L. Lin, Vacuum packaging technology using localized aluminum/silicon-to-glass bonding, *J. Microelectromech. Syst.* 11 (2002) 556–565.
- [7] Y.T. Cheng, L. Lin, K. Najafi, Localized silicon fusion and eutectic bonding for MEMS fabrication and packaging, *J. Microelectromech. Syst.* 9 (2000) 3–8.
- [8] Y.C. Su, L. Lin, Localized bonding processes for assembly and packaging of polymeric MEMS, *IEEE Trans. Adv. Pack.* 28 (2005) 635–642.
- [9] C. Luo, L. Lin, The Application of nanosecond-pulsed laser welding technology in MEMS packaging with a shadow mask, *Sens. Actuators A, Phys.* 97–98 (2002) 398–404.
- [10] H. Yang, M. Wu, W. Fang, Localized induction heating solder bonding for wafer level MEMS packaging, *J. Micromech. Microeng.* 15 (2005) 394–399.
- [11] J. Wang, E. Besnoin, A. Duckham, S.J. Spey, M.E. Reiss, O.M. Knio, T.P. Weihs, Joining of stainless-steel specimens with nanostructured Al/Ni foils, *J. Appl. Phys.* 95 (2004) 248–256.
- [12] J. Wang, E. Besnoin, O.M. Knio, T.P. Weihs, Effects of physical properties of components on reactive nanolayer joining, *J. Appl. Phys.* 97 (2005) 4307–4313.
- [13] A. Duckham, S.J. Spey, J. Wang, M.E. Reiss, T.P. Weihs, Reactive nanostructured foil used as a heat source for joining titanium, *J. Appl. Phys.* 96 (2004) 2336–2342.
- [14] J. Wang, E. Besnoin, A. Duckham, S.J. Spey, M. Reiss, O.M. Knio, M. Powers, M. Whitener, T.P. Weihs, Room-temperature soldering with nanostructured foils, *Appl. Phys. Lett.* 83 (2003) 3987–3989.
- [15] A.J. Swiston Jr., T.C. Hufnagel, T.P. Weihs, Joining bulk metallic glass using reactive multilayer foils, *Scripta. Mater.* 48 (2003) 1575–1580.
- [16] J. Wang, E. Besnoin, O.M. Knio, T.P. Weihs, Investigating the effect of applied pressure on reactive multilayer foil joining, *Acta. Mater.* 52 (2004) 5265–5274.
- [17] L. Gardner, K.T. Ng, Temperature development in structural stainless steel sections exposed to fire, *J. Fire. Safe.* 41 (2006) 185–203.
- [18] R. Pretorius, A.M. Vredenberg, F.W. Saris, R. de Reus, Prediction of phase formation sequence and phase stability in binary metal-aluminum thin-film systems using the effective heat of formation rule, *J. Appl. Phys.* 70 (1991) 3636–3646.
- [19] A.J. Gavens, D. Van Heerden, A.B. Mann, M.E. Reiss, T.P. Weihs, Effect of intermixing on self-propagating exothermic reactions in Al/Ni nanolaminate foils, *J. Appl. Phys.* 87 (2000) 1255–1263.
- [20] X.T. Qiu, *Reactive Multilayer Foils and Their Applications in Joining*, MS Thesis, LSU, 2007.
- [21] Robert W. Bower, Mohd S. Ismail, Brian E. Roberds, Low temperature Si₃N₄ direct bonding, *Appl. Phys. Lett.* 62 (1993) 3485–3487.